provide a dry process for removal of SO_X and NO_X as well as other pollutants from a gas stream. It is further desirable to have a dry removal process that eliminates the environmental impacts of the disposal of large volumes of mercury containing solids and particulates and significant amounts of gypsum generated during SO_X wet removal processes.

Wet removal processes can result in significant differential pressures across a removal system. Differential pressures above 30 inches of water column have been observed in wet removal processes. Such large differential pressures are costly because significant energy must be expended to counter the differential pressure and provide a waste gas stream with sufficient energy to flow up and out of a stack. A system and process that can accomplish pollutant removal with minimal or controlled differential pressure across the system therefore would be desirable and cost effective for most industry sectors processing or emitting significant amounts of combustion gases, process gases, and other industrial gases.

The calcium compounds utilized in SO_X wet scrubbing methods form gypsum in the process. They are purchased and consumed in significant quantities and once gypsum is formed the calcium compounds cannot be recovered, at least not cost-effectively. Thus, it would be desirable to have a removal method employing a sorbent that not only can remove pollutants from a gas stream but that can be regenerated, recovered, and then recycled or reused for removal of additional pollutants from a gas stream.

To realize such a system and process, it would need to incorporate process controls and software that can monitor and adjust operational parameters from computer stations onsite or at remote locations through interface with a sophisticated electronics network incorporating an industrial processor. This would allow a technician to monitor and adjust operational parameters in real-time providing controls of such operational parameters as system differential pressure and pollutant capture rates or removal efficiencies. Such a network would be desirable for its real-time control and off-site accessibility.

In light of increased energy demand and recent energy shortages, it would be desirable to be able to return to operational utility idled power plants that have been decommissioned because their gypsum impoundments have reached capacity. This could be accomplished with retrofits of a system employing a regenerable sorbent in a dry removal process that does not require the use of calcium compounds. Such a system would also be readily adapted and incorporated into new power plants that may be coming on line. Utility plants and independent power plants currently in operation could readily be retrofitted with such a system. Further, such a system could be of significant value in enabling emissions

sources to comply with emission standards or air quality permit conditions. With the reductions in emissions of pollutants such as NO_X and SO_X, marketable emissions trading credits could be made available or non-attainment areas for state or national ambient air quality standards may be able to achieve attainment status. Such scenarios would allow for development in areas where regulatory requirements previously prohibited industrial development or expansion.

The systems and processes of the present invention in their various embodiments can achieve and realize the aforementioned advantages, objectives, and desirable benefits.

Summary of the Invention

The invention is directed to an adaptable system for wet removal and combination wet and dry removal of SO_X and/or NO_X and/or other pollutants from gases and to processes employing the system.

In an embodiment of the invention the adaptable system for wet removal of target pollutants from gases with minimal differential pressure across the system is comprised of at least one reaction zone which is a wet scrubber. The wet scrubber is supplied with an acidic aqueous slurry of a sorbent of regenerable oxides of manganese and is configured for introduction of a gas containing at least one target pollutant at a temperature below the boiling point of the slurry. The gas is contacted with the sorbent for a time sufficient to effect capture of the target pollutant at a targeted capture rate set point for the target pollutant. The gas is substantially stripped of the target pollutant through the formation of a reaction product of the target pollutant and the oxides of manganese. The reaction zone is further configured to allow the gas to be vented from the reaction zone. Differential pressure across the system is regulated so that any differential pressure across the system is no greater than a predetermined level.

The system may have a single wet scrubber, or multiple wet scrubber in series for removal of target pollutants. In a dual stage removal system, the two reaction zones of the system may be both wet scrubbers, a wet scrubber followed by a dry scrubber, or a dry scrubber followed by a wet scrubber.

In another embodiment of the invention, the system is utilized in processes the removal of target pollutants from a gas stream. Gas containing a target pollutant is introduced into the reaction zone of the system. The gas is contacted with the sorbent in the sorbent slurry of the system for a time sufficient to effect the capture of the target pollutant at

a targeted capture rate set point for the target pollutant through the formation of a reaction product of the target pollutant and oxides of manganese to substantially strip the gas of the target pollutant;. The gas is vented gas from the reaction zone. These processes can be carried in single reaction zone or in multiple reaction zones of the system.

In another embodiment of the invention a process for the regeneration of oxides of manganese from a solution containing sulfate and nitrate anions and manganese cations formed when the reaction product of the removal of SO_X and NO_X from a gas stream with a sorbent of oxides of manganese, comprising the steps of:

A. providing first and second anion exchangers having an anion exchange resin loaded therein, the anion exchange resin having chloride in the exchange position on the resin;

B. passing a solution containing sulfate and nitrate anions through the first anion exchanger to elute the chloride to form manganese chloride while capturing the sulfate anion on the resin;

C. passing the solution containing nitrate anions through the second anion exchanger to elute the chloride to form manganese chloride while capturing the nitrate anion on the resin;

D. adding a soluble carbonate or hydroxide compound to the solution to precipitate manganese carbonate or manganese hydroxide;

D. separating the manganese carbonate or manganese hydroxide from the solution; and

E. heating the manganese carbonate or manganese hydroxide to form regenerated oxides of manganese.

Brief Description of the Drawings

Figure 1 is a schematic block diagram showing a system according to the invention.

Figure 2 is a schematic block diagram showing a system according to the invention.

Figure 3 is a schematic block diagram showing a system according to the invention.

Figure 4 is a block diagram showing a system according to the invention.

Figure 5 is a block diagram showing a system according to the invention.

Figure 6 is a perspective view of a commercially available bag house.

Figure 7 is an end elevation view of a commercially available bag house.

Figure 8 is a top plan view of a commercially available bag house.

Figure 9 is a side elevation view of a commercially available bag house.

Figure 10 is a sectional view of an inverted bag house according to the invention.

Figure 11 is a top plan view of an inverted bag house according to the invention.

Figure 12 is a flow diagram of a bag house reactor according to the invention.

Figure 13 is a block diagram of a system according to the invention.

Figure 14 is a block diagram of a system according to the invention.

Figure 15 is a block diagram of a system according to the invention.

Figure 16 is a flow diagram an electronic control system useful in the invention.

Figure 17 is electronic control panel display.

Figure 18 is electronic control panel display.

Figure 19 is electronic control panel display.

Figure 20 is a block diagram of a control sub-element according to the invention for regulating differential pressure.

Figure 21 is a control sub-element according to the invention for control of SO_X or NO_X capture rate or sorbent feed rate.

Figure 22 is a control sub-element according to the invention for control of bag house gas inlet temperature.

Figure 23 is a control sub-element according to the invention for control of variable venturi position(s).

Figure 24 is a control sub-element according to the invention for control of SO_X or NO_X capture rate, differential pressure, and sorbent feed rate.

Figure 25 is a control sub-element according to the invention for control of SO_X or NO_X capture rate, differential pressure, sorbent feed rate, and variable venturi position.

Figure 26 is a block diagram of a system and process according to the invention.

Figure 27 is a block diagram of a system and process according to the invention.

Figure 28 is a block diagram of system according to the invention.

Figure 29 is a graph plotting NO_X values over time.

Figure 30 is a graph plotting SO_X values over time.

Detailed Description of the Invention

The invention relates to systems and processes for removal of SO_X and/or NO_X as well as other pollutants, from a gas stream. In the invention, gas containing SO_X and/or NO_X is introduced into a first reaction zone where the gas is contacted with a sorbent of

regenerable oxides of manganese and/or regenerated oxides of manganese. The sorbent may interact with the pollutants in a gas stream as a catalyst, a reactant, an absorbent or an adsorbent. The oxides of manganese react with the SO_X and the NO_X to form, respectively, sulfates of manganese and nitrates of manganese.

"Nitrates of manganese" is used herein to refer to and include the various forms of manganese nitrate, regardless of chemical formula, that may be formed through the chemical reaction between NO_X and the sorbent and includes hydrated forms as well.

Similarly, "sulfates of manganese" is used herein to refer to and include the various forms of manganese sulfate, regardless of chemical formula that may be formed through the chemical reaction between SO_X and the sorbent and includes hydrated forms as well.

"Target pollutant(s)" means the pollutant or pollutants that are targeted for removal in the system.

"Substantially stripped" means that a pollutant has been removed from a gas at about a targeted capture rate whether by interaction with a sorbent or physical removal in a solid-gas separator. With respect to pollutants removed by interaction with a sorbent, it further contemplates that removal up to a targeted capture rate for that pollutant may be commenced in a first reaction zone and completed in a subsequent reaction.

"Reacted sorbent" means sorbent that has interacted with one or more pollutants in a gas whether by chemical reaction, adsorption or absorption. The term does not mean that all reactive or active sites on the sorbent have been utilized since all such sites may not actually be utilized.

"Unreacted sorbent" means virgin sorbent that has not intereacted with pollutants in a gas.

Some of the reaction zones may also serve as solid-gas separators rendering the gas free of solids and particulates, such as sorbent, whether reacted or unreacted, fly ash, and mercury compounds, so as to allow the gas that is substantially stripped of SO_X and/or NO_X or other pollutants to be vented from the reaction zone and passed to another reaction zone or routed up a stack to be vented into the atmosphere. The solids and particulates which include the reacted and unreacted sorbent, fly ash, and the like, are retained within reaction zones that are solid-gas separators and may be subsequently removed for further processing.

Reaction zones may be multi-stage removal systems which would incorporate additional reaction zones. The reaction zones utilized in single stage, dual stage, or multi-stage removal may be a fluidized bed, a pseudo-fluidized bed, a reaction column, a fixed bed,

a pipe/duct reactor, a moving bed, a bag house, an inverted bag house, bag house reactor, serpentine reactor, and a cyclone/multiclone.

The gases that may be processed in the invention are most gases containing SO_X and/or NO_X. Such gases may be generated by the combustion of fossil fuels in power plants, heating plants and various industrial processes, such as the production of taconite pellets by taconite plants, refineries and oil production facilities, gas turbines, and paper mills. Combustion for heating and other process steps at such facilities generate waste or flue gases that contain SO_X and NO_X in various concentrations, typically but not limited to 500 ppm or less for NO_X and 3000 ppm or less for SO_X. Further, the gases may contain other removable pollutants, such as fly ash, and mercury (Hg), as elemental Hg in vapor form or mercury compounds in particulate form, in small concentration, e.g., 0.0047 ppm (4.7 ppb). The gases may further contain hydrogen sulfide and other totally reduced sulfides (TRS) and other pollutants. These gases may typically have temperatures typically ranging from ambient temperature to below the thermal decomposition temperature(s) of nitrates of manganese and to below the thermal decomposition temperature(s) of sulfates of manganese. Gases generally within this temperature range can be processed in the system of the invention.

The primary sorbent useful in the invention are oxides of manganese, which may be found in manganese ore deposits or derived synthetically. Manganese compounds of interest occur in three different oxidation states of +2, +3, and +4; this gives rise to a range of multivalent phases, which provide oxides of manganese with a great diversity of atomic structures and thus mineral forms. Examples of these mineral forms include, but are not limited to, pyrolusite (MnO_2), ramsdellite (MnO_2), manganite (MnOOH or $Mn_2O_3 \cdot H_2O$), groutite (MnOOH), and vernadite ($MnO_2 \cdot nH_2O$) to name a few. This is reported by Jerry E. Post in his article "Manganese Oxide Minerals: Crystal structures and economic and environmental significance," Proc. Nat'l. Acad. Sci, U.S.A., Vol. 96, pp. 3447-3454, March 1999, the disclosure of which is incorporated herein by this reference.

One of the most common of the various forms of oxides of manganese is manganese dioxide, MnO_2. The pyrolusite form of this mineral is often the primary mineral form in manganese deposits. Pyrolusite is composed predominantly of the compound MnO_2. This oxide of manganese exhibits at least two crystalline forms. One is the gamma form, which is nearly amorphous. The other is a beta form that exhibits pronounced crystalline structure. The term "oxides of manganese" as used herein is intended to refer and include the various

forms of manganese oxide, their hydrated forms, and crystalline forms, as well as manganese hydroxide (e.g. $Mn(OH)_2$), etc.

With reference to the removal of SO_X and/or NO_X, the relative capture or removal efficiencies of oxides of manganese may be understood by the below calculation(s) of loading rates. In order to assess the economics of the system and processes of the invention, it is necessary to determine the gas removal efficiencies of the sorbent. Gas capture efficiency based upon test results may be calculated by dividing weight of gas removed by weight of sorbent. This provides an approximate picture of system operations, but does not account for stoichiometry of the reactions or interference between reactive gases in a multiple-gas system. The stoichiometric gas capture ratio is described below.

For the purpose of this assessment the overall reactions believed to occur between the sorbent, oxides of manganese, and sulfur dioxide (SO_2) and nitric oxide (NO) are shown below, with molecular weights shown above each species.

87 64 151

$MnO_{2\,(solid)} + SO_{2\,(gas)} \Rightarrow MnSO_{4\,(solid)}$ (1 mole MnO_2 captures 1 mole SO_2)

87 60 32 179

$MnO_{2\,(solid)} + 2NO_{(gas)} + O_{2\,(gas)} \Rightarrow Mn(NO_3)_{2\,(solid)}$ (1 mole MnO_2 captures 2 moles NO)

These reactions may occur in multiple steps. Molecular weights are shown above each species. Based on these reactions, the theoretical maximum stoichiometric gas capture by weight of MnO_2 sorbent is the ratio of the molecular weights of the products versus the reactants which is 73% for SO_2 or 69% for NO, for systems containing only one reactive gas. For a system containing two reactive gases, depending on reaction characteristics, the maximum stoichiometric gas capture will be lower for both gases. If reaction speeds are assumed to be equal for both reactive gases, maximum stoichiometric gas capture for each gas should be proportional to the percentage of each gas present.

For example, during a 48-hour test, two reactive gases, SO_2 and NO were present at approximately 430 ppm and 300 ppm, respectively. Total weights of reactive inlet gases treated were:

SO_2 = 98.45 lb. NO = 47.02 lb. total = 145.47 lb.

Therefore, SO_2 and NO represented 67.7% and 32.3% respectively, of reactive gases present. If the theoretical maximum stoichiometric gas capture for a single-gas system is corrected to

these reactive gas weight proportions, the theoretical maximum percentage capture for each gas by MnO_2 weight is:

SO_2: (0.73 single-gas) x (0.67 for the 48-hr. test) = 0.489 = 48.9%

NO: (0.69 single-gas) x (0.323 for the 48-hr. test) = 0.223 = 22.3%

Therefore, the theoretical maximum weights of gases captured by 289 lb., for example, of sorbent for the 48-hour test would be:

SO_2: (289 lb. Sorbent) x (0.489) = 141.4 lb. SO_2

NO: (289 lb. Sorbent) x (0.323) = 98.35 lb. NO

Actual gas capture experienced in the 48-hour test was 23.94 lb. of SO_2 and 4.31 lb. of NO. For the 2-gas system, stoichiometric gas capture was:

SO_2: (23.94 lb. captured) / (141.4 lb. SO_2 possible) = 16.9% (of theoretical maximum)

NO: (4.31 lb. captured) / (64.41 lb. possible) = 6.69% (of theoretical maximum)

Oxides of manganese, once reacted with SO_X and NO_X to form sulfates of manganese and nitrates of manganese respectively, can be regenerated. There are essentially two general methods of regeneration, thermal decomposition and chemical decomposition.

In thermal decomposition, the sulfates of manganese and/or nitrates of manganese are heated in an oxidizing atmosphere whereupon manganese oxide is formed and nitrogen dioxide and/or sulfur dioxide are desorbed and captured. The captured nitrogen dioxide or sulfur dioxide can be reacted with other chemicals to produce marketable products.

In the chemical decomposition or regeneration of manganese oxide, the sulfates of manganese and/or nitrates of manganese are dissolved from the used sorbent in a dilute acidic aqueous slurry to which, after separation and recovery of the washed sorbent, other compounds such as alkali or hydroxides or carbonates may be added and manganese oxide is precipitated out of solution and removed. The solution, now free of oxides of manganese, can be routed on for further processing or production of marketable products such as alkali or ammonium sulfates and nitrates. The regeneration of manganese oxide and production of useful or marketable products through thermal or chemical decomposition is further discussed below.

In the process of regeneration, the regenerated oxides of manganese are in particle form and are defined by the chemical formula MnO_X, where X is about 1.5 to 2.0. The regeneration process may be engineered to yield oxides of manganese having a particle size ranging from 0.1 to 500 microns. Oxides of manganese in this range are useful in the

invention. Preferably, the oxides of manganese will have a particle size of less than 300 microns, and more preferably of less than 100 microns. The regenerable oxides of manganese and/or regenerated oxides of manganese are typically fine, powdery, particulate compounds.

Reactivity of dry sorbents may generally be related to its particle surface area. Particles or particulates all have weight, size, and shape, and in most cases they are of inconsistent and irregular shape. In the case of fine powders it is often desirable to know how much surface area a given quantity of powder exhibits, especially for particles that are chemically reactive on particle surfaces, or are used as sorbents, thickeners or fillers. (Usually measurements of surface area properties are done to compare several powders for performance reasons.) Particles may also have microscopic pores, cracks and other features that contribute to surface area.

The BET (Brunauer-Emmett-Teller) method is a widely accepted means for measuring the surface area of powders. A powder sample is exposed to an inert test gas, such as nitrogen, at given temperature and pressures, and because the size of the gas molecules are known at those conditions, the BET method determines how much test gas covers all of the exterior surfaces, exposed pores and cracks with essentially one layer of gas molecules over all of the particles in the powder sample. Optionally, the analyst can use other test gases such as helium, argon or krypton; and can vary from 1 to 3 relative test pressures, or more, for better accuracy. From this, a measure of total surface area is calculated and usually reported in units of square meters of particle surface area per gram of powder sample (m^2/g). Generally, coarse and smooth powders often range in magnitude from 0.001 to 0.1 m^2/g of surface area, and fine and irregular powders range from 1 to 1000 m^2/g. Since the interactions between a sorbent and the pollutant occurs primarily at the surface of sorbent particle, surface area correlates with removal efficiency. The oxides of manganese useful in the invention are fine and irregular powders and thus may have a surface area ranging from 1 to 1000 m^2/g. Preferably the sorbent will have a surface area of greater than 15 m^2/g, and more preferably of greater than 20 m^2/g.

With reference to Figure 1, a system according to the invention is illustrated in block diagram form. The system 10 may be seen as comprised of a feeder 20 and a first reaction zone 30 and a second reaction zone 38. The feeder 20 would contain a supply of sorbent of regenerable oxides of manganese and/or regenerated oxides of manganese. The feeder 20 is configured to handle and feed oxides of manganese, which, upon regeneration, are in particle

form and defined by the chemical formula MnO_X where X is about 1.5 to 2.0. The first reaction zone 30 is configured for introduction of the sorbent in a gas containing SO_X and NO_X. In one embodiment, the first reaction zone 30 may be a section of pipe/duct, possibly configured as a fluidized bed, a pseudo-fluidized bed, a reaction column, a fixed bed, a pipe/duct reactor, a moving bed, a bag house, an inverted bag house, bag house reactor, serpentine reactor, and a cyclone/multiclone. The second reaction zone 38 a fluidized bed, a pseudo-fluidized bed, a reaction column, a fixed bed, a pipe/duct reactor, a moving bed, a bag house, an inverted bag house, bag house reactor, serpentine reactor, and a cyclone/multiclone. Preferably, the second reaction zone is a bag house, such as commercially available bag house, an inverted bag house according to the invention, or a bag house reactor according to the invention.

The gas containing SO_X and NO_X, or other pollutants, comes from a gas source 15 external to the system. The gas is introduced into the first reaction zone 30 and is contacted with sorbent introduced into the first reaction zone 30 from the feeder 20 and is contacted with the sorbent for a time sufficient to primarily effect SO_X capture at a targeted SO_X capture rate. For purpose of discussion, and not wishing to be held to a strict interpretation, with respect to effecting a certain capture, it has been observed that oxides of manganese can more readily capture SO_2 in a gas stream absent of NO, and also can more readily capture NO in a gas stream absent of SO_2, than when the gas stream contains both SO_2 and NO. SO_X capture tends to proceed at a much faster rate than NO_X capture when the two pollutants are present in a gas stream.

The gas and sorbent may be introduced separately or commingled before introduction into a reaction zone. Once the gas and sorbent have been contacted for sufficient time, the SO_X is captured by reacting with the sorbent to form sulfates of manganese to substantially strip the gas of SO_X. The gas substantially stripped of SO_X passes from the first reaction zone 30 into the second reaction zone 38. The second reaction zone 38 is configured for introduction of sorbent and the gas substantially stripped of SO_X. In the second reaction zone 38, the gas is further contacted with sorbent for a time sufficient to primarily effect NO_X capture at a targeted NO_X capture rate. The NO_X is captured by reacting with the sorbent to form nitrates of manganese to substantially strip the gas of NO_X. The second reaction zone 38 is further configured so that the gas which has been substantially stripped of both SO_X and NO_X is rendered free of reacted and unreacted sorbent. The gas may then be vented from the second reaction zone 38 to a stack 40 where the gas is released to the atmosphere.

Differential pressure across the reactor system is regulated by a control sub-element (not shown in Figure 1) so that any differential pressure across the system is no greater than a pre-determined level. As is later described, the control sub-element may control other system parameters such as feeder rate, SO_X and/or NO_X capture rate, and the inlet gas temperature into the reaction zones. Thus, the system of the invention is highly adaptable and, in another embodiment, is generally comprised of a feeder 20, a first reaction zone 30, a second reaction zone 38, and at least one control sub-element for regulating process parameters.

In another embodiment of the invention, the system is comprised of a feeder 20 as previously described and a modular reaction unit 60 comprised of at least three interconnected reaction zones. With reference to Figure 2, where the reaction zones are three interconnected bag houses 62, 64, 66, the modular reaction unit may be understood. The bag houses 62, 64, 66 are connected so that a gas containing SO_X and/or NO_X can be routed through any one of the bag houses, any of the two bag houses in series, or all of the at least three bag houses in series or in parallel or any combination of series or parallel. Each bag house is separately connected to the feeder 20 and to the external gas source 15. Through these connections, sorbent and gas can be introduced into each bag house where SO_X and NO_X capture can occur when the gas is contacted with sorbent for a time sufficient to allow formation of sulfates of manganese, nitrates of manganese, or both. The system in this embodiment may also include control sub-elements 50 (not shown) for regulating various process parameters. The reaction zones of the modular unit 60 are not limited to bag houses and may be any combination of reaction zones useful in the inventory. If the bag houses are operated independently of each other, then the section of pipe or duct (pipe/duct) preceding the bag house and that which is connected to an inlet of each bag house conveys gas into each bag house and is also configured as a first reaction zone 30, a pipe/duct reactor, into which gas containing SO_X and NO_X flows along with the sorbent. The gas is mixed with the sorbent in the pipe/duct reactor for a sufficient time to achieve SO_X capture at a targeted capture rate. In this mode, the system operates as illustrated in Figure 1 with each bag house 62, 64, 66 being a second reaction zone 38 into which the gas that has been substantially stripped of SO_X passes from the first reaction zone 30, pipe/duct reactor.

With reference to Figure 3, another embodiment of the invention is shown. In this embodiment, the system 10 is comprised of a feeder 20, and three bag houses 70, 76, and 78, a common conduit 73 and a diverter valve 74. Gas and sorbent are introduced into the first bag house 70 which serves as a first reaction zone of a two-staged SO_X/NO_X removal system

where primarily SO_X capture occurs. The gas substantially stripped of SO_X then passes from the first bag house 70 into the common conduit 73. As shown in Figure 3, the common conduit 73 is Y-shaped, but may be of any shape that allows gas to flow from the first bag house 72 and to be directed to the second and third bag houses 76, 78 which each function as the second reaction zone of a two-staged SO_X/NO_X removal system.

In the Y-shaped common conduit 73 can be seen a diverter valve 74 illustrated as a dotted line at the fork of the "Y". The diverter valve 74 is positioned in the common conduit 73 so as to direct the flow of gas from the first bag house 70 to the second bag house 76 and/or the third bag house 78. The diverter valve 74 has variable positions, in the first position gas from the first bag house 70 is directed to the second bag house 76, in the second (variable) position gas from the first bag house 70 is directed to both the second and third bag houses 76,78, and in the third position, as illustrated in Figure 3, the gas from the first bag house 70 is directed to the third bag house 78. Gas exiting the second and third bag houses 76 and 78 may be vented and directed for further processing or handling (e.g. directed to stack 40 or directed to a subsequent reactor for Hg removal). The system of this embodiment may incorporate any combination of the reaction zones useful in the invention and is not intended to be limited to bag houses.

However, when the reaction zones are bag houses, the system illustrated in Figure 3 may further comprise an off-line loading circuit 42. The off-line loading circuit 42 is brought into use after the filter bags have been pulsed to clean them of filter cake so reacted sorbent can be removed for recycling or regeneration. There may be more than one off-line loading circuit 42, as shown in Figure 3, each separately connected to a bag house 76 and 78. The off-line loading circuit is connected to a sorbent feeder and a bag house via an off-line loading circuit conduit and incorporates a fan for blowing air commingled with sorbent into the bag houses 76 and 78 in order to pre-load the fabric filter bags in the bag houses by building a filter cake thereon. The air passing through the bags and cake thereon is vented from the bag house. When the bag house is ready to come back on line, the off-line loading circuit can be closed or switched off and the diverter valve 74 moved to a position to permit the flow of process gas through the bag house that is being brought back on line.

When NO_X is captured by the sorbent, the sorbent may not be completely loaded or spent thus having remaining reactive sites. Even though it may no longer be effective as an efficient sorbent for NO_X at this point, the sorbent may have reactive sites that could be utilized efficiently for SO_X capture. Thus, the partially loaded reacted sorbent or NO_X-

reacted sorbent in a second reaction zone of a two-stage SO_X/NO_X removal system could be removed from the second reaction zone and fed into the first reaction zone to allow additional SO_X capture with, or loading onto, the sorbent. This would decrease the frequency at which sorbent regeneration is needed and reduce the amount of virgin or unreacted sorbent that would need to be introduced into the first reaction zone.

With reference to Figure 4 a system according to the invention utilizing counter-flow feed of NO_X-reacted sorbent is illustrated in a block flow diagram. The system 10 is comprised of a first reaction zone 30, a second reaction zone 38, a feeder 20 containing virgin or unreacted sorbent, and a NO_X-reacted sorbent feeder 21. The first reaction zone 30 of system 10 is connected to external gas source 15 and gas flows from the external gas source 15 to the first reaction zone 30, from the first reaction zone 30 to the second reaction zone 38, and from the second reaction zone 38 is either vented to stack 40 or directed on to another system unit such as a mercury-sorbent reactor (not shown). The feeder 20 can feed virgin or unreacted sorbent into the first reaction zone 30 and the second reaction zone 38. NO_X-reacted sorbent is removed from the second reaction zone and is conveyed from the second reaction zone to the first reaction zone via NO_X-reacted sorbent feeder 21 where the NO_X-reacted sorbent with available reaction sites is further contacted with a gas containing both SO_X and NO_X to remove and capture SO_X.

Using reacted sorbent feeders allows sorbent to be recycled to a reaction zone where unreacted sites on the surface of the sorbent can be utilized. Through the mechanical operations of removing reacted sorbent from a reaction zone and returning it to the same or another reaction zone, the amount of virgin or unreacted sorbent that has to be introduced into the system is reduced. A sorbent may be recycled this way several times before regeneration is necessary due to the reduction in available reaction sites on the surface of sorbent particles. This represents significant cost savings and more economical and complete use of the sorbent.

During operation, the surfaces of sorbent particles may become obstructed, for example, by compaction or agglomeration. The physical manipulation and handling of the reacted sorbent re-orients the particles making unexposed surfaces available to capture targeted pollutants.

The recycling of reacted sorbent in this way may proceed as shown in Figure 4 in a counter-flow manner as discussed above. Recycling may also proceed by removing reacted sorbent from a reaction zone conveying it to a reacted sorbent feeder and introducing or re-

introducing the reacted sorbent into the same reaction zone. This is shown in Figure 28, where reacted sorbent feeder 21A receives reacted sorbent conveyed from the first reaction zone 30 and reacted sorbent from reacted sorbent feeder 21A is re-introduced into the first reaction zone 30. Further, reacted sorbent from second reaction zone 38 is conveyed to reacted sorbent feeder 21B and re-introduced into the second reaction zone 38. This may be desirable where a first targeted pollutant is being captured in the first reaction zone and a second targeted pollutant is being captured in the second reaction zone. If, for example, SO_X is being captured in the first reaction zone 30, the SO_X reacted sorbent when it is spent or ceases to be effective for SO_X removal, can then be routed for regeneration and recovery of sulfates as alkali or ammonium sulfate, useful commercial product. Similarly, if NO_X is the pollutant being captured in the second reaction zone 38, the NO_X reacted sorbent can be removed when it ceases to be effective for NO_X removal and directed for regeneration and recovery to produce alkali or ammonium nitrates, again, useful commercial by-products.

Capture rates may be affected by the gas inlet temperature as it enters a reaction zone and may need to be adjusted, cooled or heated to achieve a desired capture rate for SO_X and/or NO_X. This can be accomplished with a heat exchanger. As is illustrated in Figure 5, the system may further include a heat exchanger preceding each reaction zone of a system of the invention. In Figure 5, the system of the invention as illustrated is substantially the same as the illustration of Figure 1, depicting first and second reaction zones 30 and 38, feeder 20, external gas source 15, and stack 40. In Figure 5, heat exchangers 72A, 72B have been introduced into the system before each reaction zone. The heat exchangers 72A, 72B may be utilized to heat or cool the gas stream prior to entry into each reaction zone. As the gas enters into the system, if the gas temperature is above the thermal decomposition temperature(s) of either sulfates of manganese or nitrates of manganese, the heat exchangers 72A, 72B will operate to cool the gas to a desired temperature based upon whether SO_X capture or NO_X capture is the primary pollutant captured in the reaction zone. Similarly, if the gas were below a desired temperature set point, the heat exchangers 72A, 72B will operate to heat the gas to the desired temperature. The heat exchangers 72A, 72B may be a gas-to-gas cooler or a heater unit, or other suitable means for accomplishing heating and cooling of gases to assure that the gas inlet temperature at a targeted temperature or within an acceptable range.

As previously mentioned above, the gases entering the system from external gas source 15 may be any of a variety of process or industrial gases. These gases when generated encompass a range of temperatures. Due to simple economics and the design of various

plants and facilities for efficient use of waste heat which is captured or transferred to provide heat for various processes at a facility, these process gases will typically have a temperature ranging from 250° F. to 350° F or 120° C to 180° C. In less typical situations, these gases may have temperatures upwards of 1000° F, or 540° C. Gases at these temperatures are readily processed in the systems of the invention and the heat exchangers 72A, 72B can be utilized to maintain the gas within these temperature ranges if desired. The system can also process gases at much higher temperatures such as 1000° F. For purposes of SO_X and NO_X capture, the gas temperature should not exceed, respectively, the thermal decomposition temperature(s) of sulfates of manganese and nitrates of manganese. Given that different forms or species of these sulfates and nitrates, the thermal decomposition temperature would depend upon the species formed during capture. It has been reported that that sulfates of manganese may thermally decompose at temperatures approximating 950°C. Similarly, nitrates of manganese are believed to thermally decompose at temperatures ranging up to 260 °C. The system of the invention can process gases approaching these thermal decomposition temperatures. But, more typically, the system in practice will be operated in temperature ranges approximating those of process gases from industrial sources.

Heat or waste heat from the process gases of a facility may be utilized in the regeneration and recovery processes discussed herein below. Further, the waste heat may be utilized for purposes of sorbent preheating which serves to "activate" sorbent prior to introduction into a reaction zone. Although the exact mechanism of activation is not known, it is generally known that oxides of manganese can be "activated" with heat. Thus, as can be seen in Figure 28, a system according to the invention may further include a sorbent preheater 22 which may actually be part of or separate from sorbent feeder 20. The source of heat for the sorbent preheater may be any heat source, but waste heat from facility processes can be economically efficiently utilized for this purpose.

The SO_X and/or NO_X capture rate may be regulated by the amount of sorbent fed into the reaction zones. In order to regulate capture rate, gas measuring devices, such as continuous emission monitors (CEMS), are utilized to measure the composition of the gas at the inlet to the reaction zone and at the outlet of the reaction zone. With reference to Figure 14, the gas flows from the external gas source 15 and past CEMS 80A where the gas composition is measured prior to entry into first reaction zone 30. Another CEMS 80B is provided after the first reaction zone 30 to measure the concentration of the gas substantially stripped of SO_X and/or NO_X as it passes from the first reaction zone 30. As in Figure 1, the

gas may be vented to a stack 40, passed to a second reaction zone 38, or another system unit for further processing.

In the system of the invention, a bag house may serve as a reaction zone and/or as a solid gas separator, since bag houses are solid-gas separators. A conventional, commercially available bag house 82 is depicted in Figures 6 through 9. Figure 6 is a perspective view of a bag house 82. Figure 7 is an end elevation view showing a bag house 82. Figure 8 is a top plan view of a bag house 82. Figure 9 is a side elevation view of a bag house 82. Within the bag house 82 are a plurality of bags 88 also referred to as filter fabric bags shown in Figures 7 through 9. As can be seen in figure 7 through 9, the bag house 82 has a plurality of filter fabric bags 88 suspended therein. Typically, they are suspended from a frame or support structure at the top of the bag house 82. The filter bags 88 may be of various shapes, e.g., conical or pyramidal, and include an internal frame and suitable fabric filter. Those skilled in the art would be able to select suitable filter fabric materials from those commercially available. Gas and entrained sorbent enters the bag house 82 through the bag house inlet 92, shown in Figures 7 through 9, and by virtue of an applied differential pressure, gases are forced through the fabric of the bags 88 and the entrained sorbents are separated from the gas by forming a filter cake on the surface of the bags 88. The filter cake thus formed is a reaction medium where pollutants are contacted with and removed by the sorbent. The commingled gases and sorbents move vertically upward and contact the fabric and/or the filter cake formed thereon. The bags 88 are configured to permit the gases to be directed from the outside to the inside of the bags to a conduit at the top of the bag house 82 and then to the bag house outlet 98, shown in Figures 6 through 9.

While the bag house 82 is in operation, the filter bags 88 may be periodically pulsed or otherwise agitated in order to adjust differential pressure across the bag house 82, which frees some or all of the filter cake and allows gas to flow more freely through the filter cake and the fabric filter bags. If the filter cake is allowed to get too thick, excess differential differential pressure across the bag house or the system of the invention may result. Thus, the pulse intensity or frequency can be utilized to regulate or adjust differential pressure. When the bag house 82 is taken off line, the bags 88 may be pulsed to free the bags 88 of virtually all reacted and unreacted sorbent not otherwise removed during normal operations. The reacted and unreacted sorbent or filter cake fall from the bags 88 by gravity into a hopper 112 (seen in Figures 7 and 9) at the bottom of the bag house 82 for subsequent removal from the

bag house hopper 112. Removal from the hopper 112 may be accomplished with a screw conveyor or by other appropriate means, even manually.

A thicker filter cake will lead to increased removal efficiency, but at the price of extra power required to force the external gas source through the reaction zone. In one example, more power is required for an induction fan to pull exhaust gases through the bag house when the filter cake thickness is greater. The differential pressure may thus be maintained at an optimal level, trading off increased power requirements against the increased pollutant removal. In addition, the thicker the filter cake the longer the residence time of the sorbent material in the system. Longer residence time of the gas in the filter cake results in better removal efficiencies. Higher sorbent loading rates results in less material that will have to be regenerated. This may also be taken into consideration in setting the differential pressure set point.

In Figures 7 and 9, the plurality of filter bags is shown in position within the bag house. Also shown near the top of the bag house 82 is a pulse valve 124 utilized to pulse the fabric bags 88 in order to reduce filter cake thickness or to free the filter cake from the bags 88. The bag house may be provided with a number of pulse valves 124. During operation, these pulse valves 124 may be activated sequentially or randomly in order to pulse the bags 88 in order to regulate and control differential pressure across the bag house 82 or the system as a whole. When the bag house is taken off-line, the bags may be pulsed to free the bags of virtually all filter cake so that reacted and unreacted sorbent may be removed.

The bag house illustrated in Figure 6 through 9 is of a conventional design. In Figures 10 and 11, a novel bag house according to the invention is illustrated. This bag house, which can be utilized in the system of the invention, is referred to as an inverted bag house 140. The inverted bag house 140 eliminates the need for high can velocities, and permits downward, vertical flow of gases and reacted and unreacted sorbent. The inverted bag house 140 is comprised of a bag house housing 142, at least one inlet 145, a plurality of fabric filter bags 88, a support structure 149 for the filter bags, a hopper 152 to receive and collect reacted and unreacted sorbent, an outlet 154, and a conduit 158. The bag house housing permits the introduction of gases and reacted and unreacted sorbent entrained in the gases, has a top and a bottom and is configured for gases to flow vertically downward from the top to the bottom of the bag house. The inlet 145 is located near the top of the bag house housing and is configured for the introduction of gases and reacted and unreacted sorbent entrained in the gases into the bag house. The plurality of fabric filter bags 88 are configured

to allow gas to flow from the outside of the bags 88 to the inside of the bags 88 under an applied differential pressure and to prevent the passage of reacted and unreacted sorbent from the outside to the inside of the bags 88, thereby separating reacted and unreacted sorbent from the gas and forming a filter cake on the bags 88. The support structure 149 is configured to receive and support the fabric filter bags 88 and to provide openings through which reacted and unreacted sorbent may be freely passed downward into the hopper 152 by gravity. The hopper 152 is configured to receive the reacted and unreacted sorbent and to permit the removal of the reacted and unreacted sorbent. The inverted bag house 140 also has an outlet 154 located near the bottom of the housing 142 below the bags 88 and above the hopper 152. The outlet 154 is connected to a conduit 158 located below the fabric filter bags 88 and positioned to receive gas passing through the fabric filter bags. Conduit 158 conveys gas to the outlet so that the gas may be vented or passed from the inverted bag house 140.

In Figure 12, a bag house reactor 150 of the invention is illustrated. This bag house reactor 150 can also be utilized in the system in place of a conventional bag house. The bag house reactor 150 has interior surface 154 and exterior surface 152. It may be viewed as having an upper section 156, central section 157 and lower section 158. Generally located in the central and/or lower sections 157, 158 is a variable venturi 160. The purpose of the variable venturi 160 is to adjust the velocity of gas flowing through the venturi opening within the bag house reactor 150. The variable venturi 160 is configured to adjust the position of the variable venturi by varying the space or distance between the variable venturi 160 and the interior surface 152 of the bag house reactor 150. In order to vary position a variable venturi position detector 367 shown in Figure 23) for determining the position of the variable venturi 160 and a variable venturi positioner 368 (shown in Figure 23) for adjusting the position of the variable venturi 160 are provided.

With the variable venturi 160 contacting the interior surface 154 of the bag house reactor 150, gas cannot flow from the lower section 158 to the central and upper sections 156, 157 of the bag house. By opening the space between the variable venturi 160 and the interior surface 154, gas is allowed to flow through the reactor 150. Gas introduced through gas distribution conduit 164 and the gas distribution port 162 flows from the lower section 158 to above the variable venturi 160 and into the central and upper sections 156, 157, and to the filter bags 88. When the space between the variable venturi 160 and the interior surface 154 is wide, the gas flows at lower velocities which allows some of the sorbent suspended above the variable venturi 160 to fall into the hopper 112.

There is also a sorbent distribution port 166 connected to a sorbent feed conduit 168. The sorbent distribution port 166 is positioned above the variable venturi 160 to allow the introduction of sorbent into the upper section 156 of the bag house reactor 150. The sorbent distribution port 166 is configured to allow introduction of sorbent into the bag house. Port 162 is configured to allow introduction of gas into the bag house reactor.

The bag house reactor 150 has a plurality of fabric filter bags 88 secured therein. The fabric filter bags are mounted in the upper section 156 of the bag house reactor 150 and extend downward into the central section 157. At the bottom of the bag house reactor in the lower section 158, is a sorbent hopper 112 where reacted and unreacted sorbent is collected. The sorbent hopper is connected to outlet 172. Outlet 172 has an outlet valve 176 which in the open position allows for the removal of sorbent from the hopper 112. A vent 180 is located in the top section 156 of the bag house reactor 150. Gases flowing through the bag house reactor 150 pass from the bag house reactor 150 through the vent 180 and may be directed on for further processing or venting to the atmosphere.

Sorbent entrained in gases containing pollutants such as SO_X and NO_X can begin reacting with the sorbent during transport in the sorbent feeder conduit 168. Since SO_X is more reactive than NO_X, the more reactive SO_X is primarily captured while it is being transported to the bag house reactor 150 in the first sorbent feeder conduit 164. At lower gas velocities the larger solids will abrade into finer solids and re-fluidize. The finer solids will travel upward through the opening between the variable venturi 160 and the interior surface 154 where the sorbent is suspended to create a pseudo fluidized-bed above the variable venturi 160 and the finest particules will travel upwards to form a filter cake on the surface of the fabric filter bags 88. By adjusting the position of the variable venturi 160 increasing or decreasing the space between the variable venturi 160 and the interior surface 154 of the bag house reactor 150 gas velocity is correspondingly decreased or increased. In operation, the variable venturi may be positioned to achieve a gas velocity sufficient to suspend a selected coarse fraction sorbent just above the orifice to create a pseudo-fluidized bed which may primarily or preferentially capture SO_X, since SO_X is more reactive than NO_X. Partially stripped gas flows upward from the pseudo-fluidized bed carrying the finer fraction sorbent onto the filter bags. The resulting filter cake provides a reaction medium where "slower" reactions, such as NO_X removal may occur. The variable venturi 160 position may be adjusted to achieve the desired thickness of filter cake on the fabric bags 88 thereby increasing or decreasing the differential pressure across the system also to balance overall

differential pressure by changing the venturi restriction. The fabric filter bags 88 may also be pulsed to partially remove filter cake and thus regulate differential pressure. The gas flow rate entering port 162 can be adjusted to regulate upward gas velocity so that the bags 88 may be pulsed to allow some of the loaded sorbent to fall into the hopper 112 without being reentrained in the gas or redeposited on the bags 88.

Using the variable venturi 160, one can operate the system so that sorbent suspended above the venturi, loaded with the faster reacting gases, can primarily be captured by falling to the hopper before being carried up to the filter bags 88. The fraction of sorbent loaded with faster reacting gases can then be removed from the hopper 112 by opening the outlet valve 176 so that that fraction may be removed from the hopper 112 through the outlet 172. Later the fabric filter bags 88 can be pulsed to release the sorbent loaded with slower reacting gases which would then fall through the variable venturi 160 into the hopper 112. The sorbent loaded with slower reacting gases could then be removed from the hopper through loaded sorbent outlet 172 after the outlet valve 176 has been opened. This could allow for the separate processing of the different loaded sorbent fractions to regenerate the sorbent and produce useful by-products.

Differential pressure, which represents sorbent filter cake thickness, is only one of several process parameters that can be controlled in the system in order to achieve desired levels of SO_X and NO_X removal efficiencies and cost advantages of the system. NO_X and SO_X removal efficiency may be regulated by various processes, including sorbent feeder rate and temperature control at the inlet to the reaction zones of the system. These controls are achieved by the control sub-elements or electronics, which include hardware and software and also are referred to herein below as control loops.

Referring now to Figure 13, a differential pressure control loop 300 is illustrated. External gas source 15 is illustrated feeding first reaction zone 30, which in turn feeds generally an output gas stream 316, which can feed either stack 40 or second reaction zone 38. The differential pressure across first reaction zone 30 may be measured as illustrated as difference in pressure between the inlet pressure 306 and the outlet pressure 304. In the example illustrated, inlet pressure 306 and outlet pressure 304 feed a differential pressure cell 308, which sends a differential pressure signal 310 to a differential pressure controller 302.

Differential pressure controller 302 can be any appropriate controller, including a proportional integral derivative (PID) controller. As used herein, PID controllers may be understood to operate using any combination of the proportional, integral, and derivative

components. Differential pressure controller 302 can accept a set point 312, indicating the desired differential pressure across first reaction zone 30. Set point 312 can be human or computer generated. As discussed below, differential pressure controller 302, and other controllers, may be implemented as a stand-alone controller, distributed control system, as a PID block in a programmable logic controller (PLC), or as a set of discrete calculations within a PLC. Differential pressure controller 302 generates an output signal 314 to control the differential pressure across first reaction zone 30. In embodiments where first reaction zone 30 includes a bag house or uses solids-filtering media, differential pressure controller 302 output signal 314 may control the shaking, pulsing, or other removal of sorbent which has formed a filter cake on the filter medium.

In one embodiment, first reaction zone 30 includes numerous filter bags which can have an exterior containing sorbent material and an interior having a lower pressure, acting to pull the sorbent material against the bag filter media. In one example of the invention, a compressed air jet, pulse valve 124, is periodically discharged within the interior of the filter. In one embodiment, the compressed air pulse is sufficiently strong to dislodge a portion of caked sorbent material from the filter material even during normal operation of the bag house, not requiring the shut down of the bag house. In one embodiment, the individual bags are sequentially pulsed to dislodge a portion of caked sorbent material. The frequency of the pulsing may be increased in order to maintain a thinner filter cake thickness. Thus, increasing the frequency of the periodic pulsing of each filter bag will maintain a smaller filter cake thickness, and thus result in a smaller differential pressure across the bag house as a whole. In one embodiment, filter bags are grouped by row, with each row periodically pulsed at the same instant. In some embodiments, output 314 from differential pressure controller 302 includes a frequency for pulsing filters within a bag house reaction zone. Differential pressure controller 302, in response to a higher differential pressure than set point, may increase the frequency of filter pulsing through output 314. Conversely, in response to a lower differential pressure than set point, differential pressure controller 302 may decrease the frequency of filter pulsing through output 314.

In one embodiment, the individual filter bags are formed of cylindrical filter media disposed about a rigid cylindrical cage, with the compressed air jet, pulse valve 124, disposed within the cylindrical rigid cage. After a period of time, the sorbent material filter cake builds up on the outside of the filter media, forming a thick filter cake. The pulsed air jet can force the filter media momentarily away from the cylindrical rigid cage, thereby cracking the

caked sorbent material and dislodging it, thereby allowing the sorbent material to fall under gravity to be collected and removed from the reaction zone.

A thicker filter cake can lead to increased pollutant removal efficiency, but at the price of extra power required to force the external gas source through the reaction zone. In one example, more power is required for an induction fan to pull exhaust gases through the bag house when the filter cake thickness is greater. The differential pressure may thus be maintained at an optimal level, trading off increased power requirements against the increased pollutant removal. In addition, as the filter cake thickness increases the contact or residence time of the gas with sorbent material in the system increases, resulting in more complete reaction. Therefore less material will have to be regenerated. This may also be taken into consideration in defining the differential pressure set point.

Referring now to Figure 14, an emissions control loop 320 is illustrated. A gas stream may be seen to flow from gas source 15, through a first continuous emission monitor system (CEM) 80A, then to first reaction zone 30, then to a second CEM 80B. A sorbent feeder 20 may be seen to feed material to first reaction zone 30. Feeder 20 may be a screw feeder having a variable speed screw, auger, pneumatic conveyor, or other method to move sorbent, within.

CEM 80A and CEM 80B can represent a NO_X analyzer and or a SO_X analyzer. In one embodiment, CEM 80A is a chemiluminescent monitor, for example, Thermo Electron model 42H. In one embodiment, CEM 80A includes a SO_X monitor such as Bovar Western Research model 921NMP, utilizing a spectrophotometric method. In some embodiments, CEM 80A and CEM 80B include both NO_X and SO_X analyzers. A feed controller 322 may be seen to accept a first input 328 from an outlet CEM signal 325. Controller input 328 may be used as a feedback signal to control the feeder rate. In some embodiments, a feeder controller 322 also has a second input 330 accepting an inlet measurement signal 324, also including pollutant concentration data. Second input 330 may be used to display the incoming gas concentrations and/or to calculate percentage removal set points in the system. Feeder controller 322 also accepts a set point signal 326, indicating the desired feed rate and/or the desired NO_X or SO_X concentration exiting first reaction zone 30. Feeder controller output 332 can be a variable frequency drive signal, among other available signals, to control the speed of feeder 20.

Feeder controller 322 may be any suitable controller, including a PID controller utilizing any combination of its individual modes. In one embodiment, set point 326 is set at

a desired concentration for either NO_X or SO_X, depending on the embodiment. The gas concentration signal 325 from CEM 80B can be used by feeder controller 322 to calculate output signal 332. When the gas concentration is higher than indicated as desirable by set point 326, output 332 can be increased to increase the speed of feeder 20, which will put more sorbent into first reaction zone 30, thereby dropping the pollutant concentration. Conversely, when pollutant gas concentration 325 is lower than required, feeder controller output 332 can be decreased to decrease the rate of sorbent addition from feeder 20 into first reaction zone 30.

Referring now to Figure 15, the gas to be cleaned may be seen to flow from external gas source 15, through a first heat exchanger 72A, through first reaction zone 30, through second heat exchanger 72B, through a second reaction zone 38, and to stack 40. Figure 15 illustrates a system having two reaction zones and two heat exchangers. The temperature to the first reaction zone 30 may be seen to be controlled by a first temperature controller 340, which accepts a set point 344 and a temperature input 342, and generates an output 346 to first heat exchanger 72A. As previously discussed, the maximum desired temperature in the reaction zone may depend on the thermal decomposition temperature(s) of the sulfates of manganese or nitrates of manganese, depending on whether NO_X and/or SO_X are being removed. Lower temperature set points will be above the dew point of the system and adjusted automatically or manually as needed. In one embodiment, the temperature to be controlled is measured at the reaction zone itself, rather than at the outlet from the heat exchanger, in order to more directly measure the temperature in the reaction zone. In one embodiment, temperature controller 340 output 346 may be a variable analog signal or other variable signals used to control a variable speed blower to control the outlet temperature from heat exchanger 72A. Temperature controller 340 may increase/decrease the cooling air passing through heat exchanger 72A when the temperature in first reaction zone 30 is greater/less than set point 344.

A second temperature controller 350 may be seen to accept a temperature input 352 from second reaction zone 38 and a set point 354, and to generate an output 356 for heat exchanger 72B. Second temperature controller 350 may be similar to first temperature controller 340. In one embodiment, heat exchanger 72B is used to cool the incoming gas, using ambient air as the cooling medium. As discussed previously with respect to temperature controller 340, second temperature controller 350 may increase/decrease the

output to a variable speed drive coupled to a blower when the temperature of second reaction zone 38 is greater/less than set point 354.

Figure 15 also illustrates how a first feeder 20A may feed material to first reaction zone 30. A second feeder 20B may be used to feed sorbent material to second reaction zone 38. First feeder 20A and second feeder 20B may be controlled as previously described with respect to feeder 20 in Figure 14.

Referring now to Figure 16, a control and data acquisition system 400 for controlling and monitoring the previously described processes is illustrated. System 400 may be seen to include generally a programmable logic controller (PLC) 402 and a local on-site computer 440. Both PLC 402 and local computer 440 may be coupled to the World Wide Web 424. PLC 402 and local computer 440 may be accessed over World Wide Web 424 by a user PC 428, a hand-held computer such as a Palm Pilot 430, and other devices 426 which can access World Wide Web 424.

PLC 402 may be seen to include a PLC rack 403. In one example, PLC 402 is an Allen Bradley PLC. In one example, the Allen Bradley PLC is a PLC 5. PLC rack 403 may be seen to include a PLC processor module 408, and Ethernet module 410, and a DC power supply 412. PLC 402 may be seen to include an output bus 406, for example a Control net bus 406. Bus 406, in the present example, may be seen to be coupled to numerous input/output cards 404. Input/output cards 404 may be seen to include a discrete I/O cards 404A, mixed discrete and analog I/O cards 404B, discrete I/O cards 404C, discrete and analog I/O cards 404D, more discrete and analog cards I/O 404E, a variable frequency drive card 404F, and a second variable frequency drive card 404G. The discrete I/O may be commonly used to accept inputs from discrete switches such as limit switches, and the output used to open and shut valves and to start and stop motors. The analog I/O may be used to accept input analog measurements from sensors and to control variable position output devices. The variable frequency drive outputs may be used to control variable speed motors, for example, variable speed motors used to control airflow pass the heat exchangers.

PLC 402 may be seen to be coupled to an Ethernet hub 420 via an Ethernet cable 418. In one embodiment, a DSL modem 422 enables Ethernet hub 420 to be accessed from World Wide Web 424. Local computer 440 may also be seen to be coupled to Ethernet hub 420 via an Ethernet cable 444. Ethernet cable 444 can be coupled to an Ethernet card 446. Similarly, local computer phone line 442 may be coupled to a PC modem card 450. The PC modem card can provide access to World Wide Web 424 when a DSL modem line is not available or

is not functioning. Local computer 440 may be seen to include software 448 which can include, for example, Microsoft Windows 2000 as an operating system that is providing both server and terminal functionality. Software component 448 can include an Allen Bradley OLE Process Control (OPC) module 452, as well as an Intellution ® OPC server component 454. The IFIX process monitoring and control package by Intellution is used in one embodiment. An Intellution process database component 456 may also be included. Allen Bradley OPC server 452 can provide communication between local on-site computer and Allen Bradley PLC 402.

Intellution OPC server 454 can provide communication between the Allen Bradley inputs and outputs and the Intellution process monitoring and control system residing within local computer 440. Intellution process database 456 may be used to monitor and control the entire process. Intellution Work Space 458 may be used to allow access to monitor, display, and change current data, and a historical data area 460 may be used to trend historical process data. An Access/Oracle RDB component 462 may also be included to provide database reporting. In one embodiment, a report module, for example, a Microsoft Excel or Crystal report component 464 may also be provided. In some embodiments, an Intellution web server component 466 is provided, as is a Microsoft Internet Information Server (IIS) module 468. In some embodiments, local on-site computer 440 has a local terminal or CRT as well to display, monitor, and change data residing in the Intellution Work Space 458.

In some embodiments, most or all of the controls discussed below in the present application are implemented within control system 400. In one embodiment, most or all controls are implemented within Allen Bradley PLC 402. For example, PID control blocks can be implemented using provided Allen Bradley PID blocks, or the blocks can be created from primitive mathematical operations using ladder logic. Control blocks such as the table blocks and selector blocks of Figures 24 and 25 may be implemented within Allen Bradley PLC 402 using standard blocks. Local on-site computer 440 may be used to store and output values such as PID set points and selector switch values from local computer 440 to registers or control blocks within PLC 402. For example, the set points to heat exchanger, differential pressure, and feed rate control blocks may reside within local computer 440 and be downloaded to PLC 402. The set points may be obtained by local computer 440 from a local terminal and/or from World Wide Web 424 from devices 426, 428, and/or 430, protected by appropriate security. Local computer 440 can be used to provide historical trending, operator interface, alarming, and reporting.

Referring now to Figure 17, a process graphic 450, as displayed on a human-machine interface is displayed. Process graphic 450 may be displayed, for example, on an Intellution IFIX system. Process graphic 450 can be updated in real time and can reside on a personal computer, for example. Process graphic 450 includes a manual switch 458 and an automatic switch 459 for controlling the control mode of the differential pressure across the bag house. Process graphic 450 also includes a table of values 460 including the differential pressure set point, the actual differential pressure and the inlet temperature to the bag house. An output table 462 is also illustrated, including the bag house outlet temperature, the flue gas flow rate, the inlet pressure to the bag house and the outlet pressure from the bag house. A bag house 452 is shown diagrammatically including an inlet 454 and an outlet 456. An outlet emission table 464 is also illustrated, including the SO_2, the NO_X level, and the O_2 level. Process graphic 450 may be used to monitor and control the bag house differential pressure, as previously discussed.

Referring now to Figure 18, a process graphic 470 is illustrated as may be displayed on an Intellution IFIX process graphic. Process graphic 470 can monitor and control the absorbent feeder speed, including an increase button 471 and a decrease button 472. The actual feeder speed in pounds of sorbent per hour is illustrated at feeder speed 483. A scrubber inlet table 473 is illustrated, including a SO_2 level, a NO level, a NO_2 level, a NO_X level, a CO level, and an O_2 level. A scrubber outlet table 474 includes the same levels as the inlet, but at the scrubber outlet. A NO_X control section 475 on the process graphic includes a manual button 476 and an auto button 477, as well as a set point 478. In automatic mode, set point 478 may be used to control the feeder speed using the NO_X set point. Similarly, an SO_2 control section 479 includes a manual control button 480 and an auto control button 481, as well as a set point 482. In automatic mode, set point 479 may be used to control the feeder speed using the SO_2 set point.

Referring now to Figure 19, a process graphic 490 is illustrated, as may be found on a process control and monitoring station. A cooler 491 is illustrated, having an inlet 492 and an outlet 493, with the inlet and outlet temperatures being displayed in real time. Cooler 491 may be a heat exchanger as previously discussed. Process graphic 490 includes a manual button 494 and an auto button 495. The bag house inlet temperature is displayed at 498 as is the cooler set point 497. When in the automatic mode, the fan speed may be controlled by a PID controller using set point 497. Process graphic 490 also includes an outlet emission table 496, including the SO_2 level, the NO_X level, and the O_2 level.

Referring now to Figure 20, differential pressure control loop 300 is illustrated in block diagram form. Differential pressure controller 302 may be seen to accept set point 312 and actual differential pressure 310, and to generate output signal 314 to control the differential pressure across bag house 30. As previously discussed, differential pressure set point 312 may be set taking into account the desired pollutant removal target of the system, the power required to force gas through the filters, and the desired rate of sorbent replenishment.

Referring now to Figure 21, sorbent feeder control loop 320 is illustrated in block diagram form. As previously discussed, feeder control loop 320 can include a reaction zone CEM unit 80B that generates an output signal from the NO_X and/or SO_X emission analyzers. Emissions/Feeder controller 322 can accept the NO_X or SO_X measured emission level through controller input 328, and accepts a set point 326 indicating the desired NO_X and/or SO_X concentration. Controller 322 may also send a controller output 332 to sorbent feeder 20. As previously discussed, sorbent feeder 20 may be a variable speed screw feeder, accepting a variable analog drive signal among others as its input from feeder controller 322. The process trade-offs in setting set point 326 are as previously described.

Figure 22 illustrates a control loop 341 for controlling the temperature of bag house 82. Temperature controller 340 is as previously described with respect to Figure 15. Temperature controller 340 accepts a bag house temperature input 342 and desired bag house input temperature set point 344, generating controller output 346 which can be fed as a fan speed control to heat exchanger 72A. The control scheme rationale is as previously described with respect to Figure 15.

Referring now to Figure 23, a variable venturi control loop 361 is illustrated. Figure 23 illustrates a venturi position controller 360, which accepts a venturi position set point 362 and an actual venturi position input 364, generating a controller output 366 which can be accepted by a variable venturi positioner at 368. The actual position of the variable venturi position may be measured by a position detector 367. In one embodiment, the variable venturi position may be measured in units of 0 to 100%. Venturi set point 362 may be set as a function of one of several desired process parameters.

The variable venturi position may be set to control the space between the variable venturi 160 and interior surface 154, the cross-sectional flow area, available for the bag house inlet gas to flow around the flow occluding devise, variable venturi 160, thereby controlling the fluidization velocity of the gas. When the flow cross-sectional area is decreased, the gas

flow velocity increases, which can be used to support a deeper fluidized bed depth of sorbent material. If the gas flow velocity is made very high, only the densest sorbent particles will be able to descend against the swiftly rising gas and be collected from the system. If the fluid velocity is set very low, even the lightest particles will be able to settle out of the system quickly, thereby increasing the need for regeneration or recycling of material back to the reaction zone for more loading. A higher gas flow velocity will, in effect, create a fluidized bed reactor, having a fluidized bed of sorbent material held in place by the upwardly rising gas stream. A rapidly moving gas stream will also carry more sorbent particles to the fabric bags 88 filter to form a filter cake. Conversely, a slowly moving gas flow around the variable venturi 160 will allow many sorbent particles to fall and be collected prior to becoming caked upon the bags 88. A deeper fluidized bed will create higher differential pressures and a shallow fluidized bed will create lower differential pressures. Removal efficiencies may be taken into consideration when setting SO_X and/or NO_X fluidized bed depth. Variable venturi controller 360 may be any suitable controller, including a PID controller, utilizing any combination of its modes.

Referring now to Figure 24, a control scheme 370 is illustrated for controlling sorbent feeder 20 using one set of inputs selected from the group including NO_X concentration, SO_X concentration, and reactor zone differential pressure. The control of sorbent feeder 20 may be accomplished by selecting one of the aforementioned control inputs, where the selection may be based on the greatest deviation from set point or error.

An error generator 373 may be seen to accept several actual measurement signals 384, as well as several set points 385. The actual signals and set points may be used to generate corresponding errors, for example, using subtraction. Error generator 373 may be seen in this example to output a NO_X error 373A, a SO_X error 373B, and a differential pressure error 373C. The outputs from error generator 373 may be accepted by an error selector gate 374, with one of the input errors selected and output as the error to a controller error input 382. Error selector gate 374 may be operated manually to accept one of the several input errors in some embodiments. In other embodiments, error selector gate 374 may automatically select the largest error or deviation, to control based on the process variable or parameter most requiring attention. For example, sorbent feeder 20 may be controlled based upon the NO_X concentration, the SO_X concentration, or the differential pressure across the reaction zone.

Error selector gate 374 may select the highest deviation, or the highest percent of deviation, of these three error inputs. Error selector gate 374 can generate a selector output

386 which can be used to select which of the inputs a gain selector 372 is to select. Similarly, error selector gate 374 may output a selector output 383 which can be accepted by a set point selector gate 376 to select from various set points provided to the selector gate.

A gain table 371 may be implemented as a table in a fixed database, for example, a series of registers in a PLC. Gain table 371 may be seen to include a NO_X gain 371A, a SO_X gain 371B, and a differential pressure gain 371C. The gains from gain table 371 may be seen to feed gain selector block 372. A gain selector output 377 may be sent to a controller gain input 379.

A set point table 375 may be seen to include a NO_X set point 375A, a SO_X set point 375B, and a differential pressure set point 375C. The set points may be used as inputs to selector gate 376, with selector output 383 being used to select one of the input set points. Selector gate 376 may be seen to output one of the selected set points to controller set point input 380.

Control scheme 370 thus provides a system and method for controlling the sorbent feeder rate based upon any one of the NO_X concentrations, the SO_X concentration or the differential pressure across the reaction zone. This can be accomplished using the selector blocks previously discussed while only requiring a single controller. Controller 378 can be, for example, a PID controller, using any combination of its individual modes.

Referring now to Figure 25, a control scheme 390 is illustrated, similar in some respects to control scheme 370 of Figure 24. Control scheme 390 includes similar control blocks, tables, and outputs as previously described in Figure 24. Control scheme 390 further includes the variable venturi control as one of the possible sets of inputs, gains, and set points to be used to control sorbent feeder 20. Gain table 371 may be seen to include a variable venturi gain 371D. Error generator 373 may be seen to generate a variable venturi error 373D. Set point table 375 may be seen to include a variable venturi set point 375D. Control scheme 390 may thus operate in a manner similar to control scheme 370 of Figure 24, but allowing for control based on the venturi position.

Various components of the system of the invention have been discussed above. Many of the components of the system are commercially available from various original equipment manufacturers and are known to those of ordinary skill in the art. Further, one skilled in the art will recognize and understand that the reaction zones and other units of the system of the invention may be connected by pipes, ducts, and lines , etc. which allow gas and/or sorbent to flow through and within the system and that reaction zones are in flow through

communication in dual and multi stage embodiments of the invention. In addition to the aforementioned system components, the system may further include various hoppers, conveyors, separators, recirculation equipment, horizontal and vertical conveyors, eductors. Further, there may be modulating diverter valves, vibrators associated with feeders, compressors to provide instrument air to pulse filter fabric bags, as well as various meters and sampling ports.

In addition to removing SO_X and NO_X, the system and processes of the invention can be utilized to remove mercury (Hg) and fly ash. Gases emanating from combustion of fuels, which contain mercury and sulfides, include mercury compounds, mercury vapor, ash, SO_X and NO_X. These gases and solids are commingled with oxides of manganese and are transported at a sufficient velocity as a gas-solids mixture to a reactor, which may be a bag house or other reactor/separating device. During transport and during residence in the reactor, oxidation-reduction reactions occur. These reactions cause the conversion of mercury vapor to mercury compound(s), and sorbent and/or alumina adsorb the mercury compound(s). As disclosed above, SO_X and NO_X are removed through reaction with oxides of manganese to form sulfate and nitrate compounds of manganese. These reaction products, unreacted sorbent (if any) alumina, adsorbed mercury, and ash are trapped and collected in the bag house and clean, substantially stripped gases are vented to the stack. Thus, during the processing of gases with the system of the invention, mercury and mercury compounds may also be removed. The reacted and unreacted sorbent when removed from the reaction zones of the system may be further processed to generate useful products and to regenerate the sorbent as described herein below.

The system of the invention in its various embodiments may be utilized in a process for removal of oxides of sulfur and/or oxides of nitrogen, mercury (compounds and vapor), and other pollutants from a gas stream. The processes generally involve providing a system according to the invention, whether single stage, dual-stage, or multi-stage. Gas and sorbent are introduced into a reaction zone and contacted for a time sufficient to effect capture of the targeted pollutant(s) thereby substantially stripping the gas of the targeted pollutant(s). In a single-stage removal process, the reaction zone would need to be a solid-gas separator operating as a reaction zone or else followed by a solid-gas separator in order to render the gas that has been substantially stripped of a target pollutant free of solids so that the gas may either be vented or directed for further processing. In a dual-stage removal process, the second reaction would preferably be a solid-gas separator operating as a reaction zone. And,

in a multi-stage removal process the last reaction zone in the series of reaction zones through which the process gas is directed would need to be a solid-gas separator operating as a reaction zone or else followed by a solid-gas separator in order to render the gas that has been substantially stripped of a target pollutant free of solids so that the gas may either be vented or directed for further processing. Generally, configuring the systems and processes of the invention to incorporate a solid-gas separator as the last reaction zone in a sequence of removal steps would be most economical and efficient.

A process according to the invention is described below using single-stage and dual-stage systems of the invention for purposes of illustration. It should be readily understood by those skilled in the art that the processes as described can be adapted to multi-stage removals and to removal of various targeted pollutants with or without the addition of other sorbent materials or chemical additives, as appropriate.

Removal of SO_X and/or NO_X can be accomplished in a single single-stage removal system. Sorbent and gas containing SO_X and/or NO_X are introduced into a reaction zone 30 where the gas and sorbent are contacted for a time sufficient to substantially strip the gas of SO_X and/or NO_X. If SO_X is the primary target pollutant, the gas may be introduced at temperatures typically ranging from about ambient temperature to below the thermal decomposition temperature(s) of sulfates of manganese. If NO_X is the primary target pollutant, the gas would be introduced at temperatures typically ranging from about ambient temperature to below the thermal decomposition temperature(s) of nitrates of manganese. If both pollutants are present, NO_X will not be captured if the temperature of the gas is above the thermal decomposition temperature of nitrates of manganese. In the reaction zone, the gas would be contacted with the sorbent for a time sufficient to effect capture of the pollutant at a targeted capture rate. If both pollutants are to be captured, the capture rate for the primary targeted pollutant would control or utilize a control sub-element, such as control loop 320 of Figure 14 or control loop 390 of Figure 25. The capture rate for the targeted pollutants can be monitored and adjusted. The reaction zone would preferably be a solid-gas separator that renders the gas free of solids, such as reacted and unreacted sorbent and any other particulate matter in the gas so that the gas may be vented from the reaction zone or directed for further processing, after contacting the gas with sorbent for a sufficient time.

In a dual-stage removal process, a system of the invention having at least two reaction zones, first and second reaction zone 30, 38 as in Figure 1, is provided. It should be understood that the system could be a system of the invention such as the modular reaction

units illustrated in Figures 2 and 3. With reference to Figure 2, any of the bag houses 62, 64, 66 could serve as first and second reaction zones 30, 38 depending upon how the gas is directed through the system. Further, with reference to Figure 3, the first bag house 70 would correspond to first reaction zone 30 and either or both of the second and third bag houses 76, 78 would correspond to second reaction zone 38. Additionally, it is understood that other reaction zones may be substituted for the bag houses of Figures 2 and 3 and the process as described could be carried out.

However, for purposes of illustration, the dual-stage removal process is discussed with reference to Figure 1. In this process of the invention, gas and sorbent are introduced into first reaction zone 30 . The gas is contacted with the sorbent for sufficient time to primarily effect SO_X capture at a targeted capture rate. The gas is rendered free of solids and then vented from the first reaction zone 30. Sorbent and the gas that has been substantially stripped of SO_X are then introduced into second reaction zone 38. In the second reaction zone, the gas is contacted with the sorbent for a sufficient time to primarily effect NO_X capture at a targeted capture rate. The gas is rendered free of solids and then vented from the second reaction zone 38. The vented gas may be directed to stack 40 to be vented or emitted into the atmosphere or directed on for further processing.

With the processes of the invention, other pollutants that can be captured with oxides of manganese can be removed. For example, without being limited or bound by theory, Applicants believe that mercury compounds adsorb onto oxides of manganese. Applicants further believe that, in the system and processes of the invention, elemental mercury is oxidized to form oxides of mercury which also adsorb onto oxides of manganese. Additionally, hydrogen sulfide (H_2S) and other totally reduced sulfides (TRS) can be removed utilizing oxides of manganese. More specifically, Applicants postulate that the sulfur in TRS may be oxidized to form SO_2 which is known to react with oxides of manganese to form sulfates of manganese. Further still, Applicants believe that CO is oxidized to CO_2 which in turn reacts with the sorbent to form carbonated of manganese ($MnCO_3$) from which useful products can be recovered and oxides of manganese regenerated.

It is known that mercury compounds may be removed from gases by adsorption on fly ash and/or alumina. Thus, alumina may be introduced with the sorbent in a reaction zone for purposes of removing mercury compounds and elemental mercury that has be oxidized to form oxides of mercury. Thus, elemental mercury that is not oxidized and therefore not captured by the sorbent in a first or second reaction zone may be captured in a third reaction

zone, which may be referred to as a mercury-alumina reactor or an alumina reactor. With respect to single-stage removal, the mercury compounds may be removed in a reaction zone by contacting the gas with sorbent for a time sufficient for the mercury compounds to adsorb on to the sorbent, and alumina if mixed with the sorbent to thereby substantially strip the gas of mercury. Further, if the reaction zone is a solid-gas separator, mercury compounds adsorbed to fly ash would also be removed, thereby substantially stripping the gas of mercury compounds. In a dual-stage, the mercury compounds would similarly be removed, but depending upon which reaction zone is also a solid gas separator.

Thus, the system and process of the invention are readily understood to include and contemplate the removal of not only SO_X and/or NO_X but other pollutants, such as mercury compounds, elemental mercury, CO, CO_2, TRS, and H_2S.

The system and process of the invention has been tested at several power plants utilizing a SO_X and/or NO_X removal demonstration unit embodying a system according to the invention. The demonstration unit utilized a bag house as the second reaction zone and a pipe/duct as a first reaction zone in a dual stage removal system. The test runs and results are summarized in the following examples.

Example 1

NO_X concentrations were determined using EPA method 7E, chemiluminesent analysis method, and analyzed with a model 42H chemiluminescent instrument manufactured by Thermo Electron Inc. Sulfur dioxide (SO_2) concentrations were measured utilizing, a spectrophotometric analysis method employing a Bovar Western Research Spectrophotometric model 921NMP instrument. In order to obtain accurate and reliable emission concentrations, sampling and reporting was conducted in accordance with US EPA Reference CFR 40, Part 60, Appendix A, Method 6C. Gas flow rates in standard cubic feet per minute (scfm) were measured using AGA method #3, utilizing a standard orifice plate meter run. The demonstration was conducted utilizing a series of test runs on live gas streams from a power plant. Said power plant operates steam boilers which are fired on high sulfur coal. During test runs, NO_X and SO_2 concentration readings were taken continuously alternating from the inlet and the outlet of the demonstration unit. Gas flow rates were measured continuously. The demonstration tests were performed utilizing two different forms of sorbent. The tests conducted utilized various forms of oxides of manganese as sorbent. The tests were performed with and without bag house filter pulsing. The following table summarizes the results and operational parameters:

Range of Operation Parameters	
Range of NO_X Concentrations Processed by the Demonstration Unit	14.14 to 320 ppm
Range of SO_2 Concentrations Processed by the Demonstration Unit	300 to 1800 ppm
Range of Gas Flow through the Demonstration Unit	250 to 2000 scfm
Range of Pressure Across the Bag House	0.5" to 10.0" of H_20
Range of Bag House Temperatures	60° F to 246° F
Maximum NO_X steady state Removal Rate	96.0%
Maximum SO_2 steady state Removal Rate	99.8%

Example 2

A test using the demonstration unit according to the invention, utilizing oxides of manganese as the sorbent was conducted on a simulated gas stream containing varying levels of NO_X. Oxides of manganese powders that were used during this test described generally by 60% of particles less than 45 microns in size and having a BET surface area of approximately 30 m^2/g. Knowing that there is a competition for reaction sites between SO_2 and NO_X, a series of tests was conducted to gather data on the efficiency of NO_X capture in the absence of SO_2. Synthetic NO_X gas was made on site by use of high-concentration bottle gas which was diluted into the inlet gas stream and processed by the demonstration unit. The bag house was pre-loaded with oxides of manganese prior to introduction of test gas by operating the demonstration unit's blower at high speed (typically about 1200 scfm), and feeding the oxides of manganese into the gas stream at a high rate (between 40% and 90% of feeder capacity) in order to form a suitable filter cake on the fabric bags in the bag house. Gas from cylinders containing NO_X, 20% NO, and 20% NO_2, (20,000 ppm) was metered into the bag house inlet through a rotameter-type flow gage. NO_X concentrations were measured at the bag house inlet and outlet on an alternating basis throughout the testing with the demonstration unit's continuous emissions monitoring system (CEMS), utilizing a Thermo Electron model 42H Chemiluminescent instrument. In order to obtain accurate and reliable emission concentrations, sampling and reporting was conducted in accordance with US EPA Reference CFR 40, Part 60, Appendix A, Method 6C.

Tests were performed at varying levels of bag house differential pressure (measured in inches of water column) and flow rates (measured in scfm). The NO_X inlet concentrations ranged from 18.3--376.5 ppm with flow rates ranging from 260-1000. It has been determined that varying levels of filter cake thickness affect the NO_X and SO_2 removal. A thicker filter cake increases the quantity of sorbent exposed to the gas, thus increasing the micro-reaction zone within the filter cake. As a representation of the sorbent filter cake depth, the differential pressure across the bag house (referred to as ΔP) was measured between 2.00"-9.67" of WC (expressed in inches of water column). NO_X concentrations were recorded once the system was in steady state and the readings were stable for up to 20 minutes. The following table illustrates the level of NO_X removal achieved as a function of inlet concentration, gas flow rate, and bag house differential pressure:

Summary of Bottle Gas NO_X Reduction Test

Run No.	Inlet NO_X (ppm)	Outlet NO_X (ppm)	% Reduction	ΔP (in. H_2O)	Flow Rate (scfm)
1	25.5	3.3	87.1	2.00	260
2	140.1	8.5	94.0	3.86	500
3	102.0	10.5	89.7	7.71	1000
4	324.9	17.4	94.7	7.78	1000
5	195.0	15.1	92.3	7.85	1000
6	46.7	8.4	81.9	7.85	1000
7	200.3	32.5	83.8	3.0 to 4.0	1000
8	28.2	6.2	78.0	7.80	500
9	57.8	11.4	80.3	2.10	500
10	84.9	8.9	89.5	3.80	500
11	86.0	8.9	89.7	3.80	500
12	194.5	11.5	94.1	3.80	500
13	317.5	12.7	96.0	3.80	500
14	376.5	26.7	92.9	2.10	500
15	376.5	26.7	92.9	2.10	500
16	18.3	4.0	78.1	4.45	509
17	83.5	8.7	89.6	4.45	509
18	40.1	5.9	85.3	4.45	509
19	83.5	8.7	89.6	4.45	509
20	21.5	4.5	79.2	4.74	500
21	45.7	6.5	85.8	4.75	500
22	92.1	8.6	90.7	4.75	500
23	201.1	11.5	94.3	4.76	500
24	317.5	14.0	95.6	4.79	500
25	52.1	10.0	80.9	9.67	1000
26	82.4	12.0	85.5	9.67	1000
27	105.4	13.2	87.5	9.65	1000
28	224.0	18.5	91.8	9.67	1000
29	328.4	23.1	93.0	9.67	1000
30	100.2	15.0	85.0	9.67	1000

Example 3

A further test of the demonstration unit according to the invention utilizing oxides of manganese as the sorbent, was conducted on a live exhaust gas slipstream from a 170 MW coal fired boiler. The boiler was operating on high sulfur coal of approximately 4-6% sulfur, resulting in emission concentrations of SO_2 in the range of 1200-2000 ppm and NO_X

concentrations in the range of 280-320 ppm. A slipstream averaging 1000 scfm was diverted from the main stack exhaust and routed to the demonstration unit for reaction and sorption by the sorbent oxides of manganese. SO_2 and NO_X concentrations were measured at the scrubber inlet and outlet of the bag house on an alternating basis throughout the testing with the demonstration unit's continuous emissions monitoring system (CEMS). SO_2 concentrations were measured utilizing a Bovar Western Research model 921NMP spectrophotometric analyzer and NO_X concentrations were measured utilizing a Thermo Electron model 42H chemiluminescent instrument. In order to obtain accurate and reliable emission concentrations, sampling and reporting was conducted in accordance with US EPA Reference CFR 40, Part 60, Appendix A, Method 6C.

SO_2 removal efficiencies of 99.8% and NO_X removal efficiencies of 75.3% were achieved while processing on average 1000 scfm of exhaust gas at temperatures typically ranging from 150° F to 250° F. Test runs were conducted with varying levels of bag house differential pressures ranging from 0.5" to 8.6" of WC, which represents various levels of filter cake thickness. Tests were also conducted with different rates of bag house filter bag pulsing and varying levels of oxides of manganese feed rates. Oxides of manganese powders that were used during this test described generally by 60% of particles less than 45 microns in size and having a BET surface area of approximately 30 m^2/g. The following table gives an example of SO_2 and NO_X data collected during a test in which 1000 scfm was processed by the dry scrubber at an inlet temperature of 250 °F, and a differential pressure of 5.75" of WC. Data was collected once the demonstration unit was in a steady state of NO_X and SO_2 removal for a period of 30 minutes. The results are summarized in the below table:

Pollutant	Inlet ppm	Outlet ppm	ppm % Removal
Oxides of Nitrogen (NO_X)	285.9	70.5	75.3%
Sulfur Dioxide (SO_2)	1703	3.9	99.8%

Example 4

An additional series of demonstration tests of the demonstration unit, utilizing oxides of manganese as the sorbent, was conducted on a live exhaust gas slipstream from a 75 MW coal fired boiler. This boiler was operating on Powder River Basin (PRB) coal, resulting in emission concentrations of SO_2 in the range of 340-500 ppm with NO_X concentrations in the range of 250-330 ppm. A slipstream ranging from 500-1000 scfm was diverted from the main stack exhaust and routed to the demonstration unit for reaction and sorption by the

oxides of manganese. Oxides of manganese powder that were used during this test described generally by 60% of particles less than 45 microns in size and having a BET surface area of approximately 30 m^2/g. SO_2 and NO_X concentrations were measured at the bag house inlet and outlet on an alternating basis throughout the test with the demonstration unit's continuous emissions monitoring system (CEMS). SO_2 concentrations were measured utilizing a Bovar Western Research model 921NMP spectrophotometric instrument and NO_X concentrations were measured utilizing a Thermo Electron model 42H chemiluminescent instrument. In order to obtain accurate and reliable emission concentrations, sampling and reporting was conducted in accordance with US EPA Reference CFR 40, Part 60, Appendix A, Method 6C.

SO_2 and NO_X reduction efficiencies were measured at 99.9% and 91.6% respectively. Testing was conducted with varying degrees of differential pressure (ΔP) across the bag house to affect the residence time of the targeted pollutants. Reaction chamber temperatures ranged from 150° F to 280° F. It was determined that longer residence times resulted in improved capture rates for NO_X. However, the fact that the SO_2 reaction occurs so rapidly and completely, the SO_2 reduction efficiency remains nearly complete (99.9%) at even the lowest of residence times. While operating the scrubber at 0.5"-1.0" of WC across the bag house, a pollutant concentration reduction efficiency of 99.8% for SO_2 and 40.0% for NO_X was achieved. It is from these results that the concept for a two stage reaction chamber system develops, whereby the first reaction chamber captures the majority of SO_2 and a small fraction of NO_X, while the second "polishing" stage completes the NO_X removal to desired levels of efficiency, predetermined and controlled by the system operator. Data was collected once the dry scrubber was in a steady state of NO_X and SO_2 removal for a period of 30 minutes. The following table gives an example of SO_2 and NO_X data collected during a testing in which 500 scfm was processed by the demonstration unit at an inlet temperature of 250 °F, and a differential pressure of 8.7" of WC:

Pollutant	Inlet ppm	Outlet ppm	ppm % Removal
Oxides of Nitrogen (NO_X)	268.1	22.4	91.6%
Sulfur Dioxide (SO_2)	434.3	0.5	99.9%

Example 5

In an attempt to determine the effectiveness of SO_2 and NO_X removal, a series of lab-scale tests were conducted utilizing a glass reactor. The reactor was designed to mimic the gas-solid interactions known to be present in the aforementioned demonstration unit. The

glass reactor had a diameter of 2 inches with a length of approximately 24 inches. 50.0 grams of oxides of manganese were suspended in the reactor using a fritted glass filter allowing for flow of the gas stream, while keeping the oxides of manganese suspended. Approximately 3 inches above the fluidized bed of oxides of manganese, a sintered stainless steel filter was arranged to simulate a bag house filter bag. The reactor was heated during the testing to 250°F.and the gas flow rate was metered at a constant 6 liters per minute (lpm). Simulated exhaust gas was produced by use of a calibration gas standard having the following composition: CO_2= 17.35%, NO_X=391 ppm, SO_2=407 ppm, CO=395 ppm, and balance N_2 . The simulated flue gas stream passed through the fluidized bed of oxides of manganese, where the flow carried a portion of the sorbent up onto the filter, thus creating a filter cake, which mimics a bag house reactor chamber.

SO_2 and NO_X concentrations were measured continuously from the reactor outlet utilizing a continuous emissions monitoring system (CEMS). SO_2 concentrations were measured utilizing a Bovar Western Research model 921NMP spectrophotometric instrument and NO_X concentrations were measured utilizing a Thermo Electron model 42H chemiluminescent instrument. In order to obtain accurate and reliable emission concentrations, sampling and reporting was conducted in accordance with US EPA Reference CFR 40, Part 60, Appendix A, Method 6C. Removal efficiencies of 99.9% for SO_2 as well as 99.9% for NO_X were measured and duplicated for several test runs. Inlet temperature was 250° F, with a differential pressure of 2.00" of WC. The following table gives an example of SO_2 and NO_X data collected during testing in which 6 lpm of gas was processed by a glass reactor:

Pollutant	Inlet (ppm)	Outlet (ppm)	Sorbent Weight (g)	% Removal	Flow rate (lpm)	ΔP (in H_2O)	Temp. (° F)	Time with >94% Removal
Oxides of Manganese Type A								
NO_X	391	17.21	50	95.6%	6	2.00	250	29 min
SO_2	407	0.1	50	99.9%	6	2.00	250	>54 min
Oxides of Manganese Type B								
NO_X	391	0.1	50	99.9%	6	2.00	250	60 min
SO_2	407	0.1	50	99.9%	6	2.00	250	>90
Oxides on Manganese Type C								
NO_X	391	0.2	50	99.9%	6	2.00	250	34 min
SO_2	407	0.1	50	99.9%	6	2.00	250	>68 min

The tests of this Example 5 were conducted with three different lots of manganese oxide sorbent. Figures 29 and 30 are, respectively, graphs plotting NO_X and SO_X concentrations at the outlet of the glass reactor versus time. The three different oxides of manganese are represented by the symbols "◇" for type A sorbent, "△" for type B sorbent, and "□" for type C sorbent in Figure 29 and 30. Type A sorbent is an oxide of manganese powder generally at 60% of particles less than 45 microns in size and having a BET surface area of approximately 30 m^2/g. Type B sorbent is an oxide of manganese powder generally at 100% of particles less than 45 microns in size and having a BET surface area of approximately 200 m^2/g. Type C sorbent is an oxide of manganese powder generally at 80% of particles less than 45 microns in size and having a BET surface area of approximately 90 m^2/g. The graph of Figure 30, confirms the above statements regarding near immediate and complete SO_X capture upon contact with the sorbent. The graph of Figure 29 shows a range of capture efficiency over time for NO_X and that different forms of oxide manganese may be able to provide more efficient capture of NO_X. The type B sorbent performed the best before break-through, followed by type C. Useful captures were observed for all three types. With the process controls of the invention a wide variety of oxides of manganese can be utilized to effect removal at targeted capture rates. Further, the graphs of Figures 29 and 30 show that high removal or capture rates can be achieved and sustained over time. The operational parameters of the systems of the invention can be monitored and adjusted to attain and maintain removal or capture rates at these high levels.

As mentioned above, the reacted or loaded sorbent can be recycled and/or regenerated after being removed from a reaction zone. For recycling purposes the reacted sorbent may simply be reintroduced into another reaction zone. For example with reference to Figure 4, the system has first and second reaction zones 30, 38 which are connected to feeder 20 which contains unreacted or virgin sorbent. Gas from external gas source 15 is introduced into first reaction zone 30 along with sorbent fed from feeder 20. The gas is contacted with sorbent for a time sufficient to remove a target pollutant, such as SO_X, and after being rendered free of solids is vented from the first reaction zone 30. The gas is then introduced in the second reaction zone 38 along with sorbent from feeder 20. In the second reaction zone 38, the gas is contacted with gas for a time sufficient to remove another target pollutant, here NO_X. During operation, the level of NO_X loading on the reacted sorbent in second reaction zone 38 reaches the point where the sorbent no longer efficiently removes NO_X. When the point is reached, the NO_X. reacted sorbent is removed from the second reaction zone 38 and conveyed

or transported to NO_X reacted sorbent feeder 21. The NO_X reacted sorbent, which has unused reactive sites available for further SO_X capture, is fed or introduced into the first reaction zone 30 for additional loading or reaction with SO_X in the gas introduced from external gas source 15. When the recycled NO_X reacted sorbent reaches the point where SO_X capture can no longer be achieved at a targeted rate of removal, the now NO_X and SO_X reacted (or loaded) sorbent is removed from the first reaction zone and routed for regeneration. In this way, the amount of virgin or unreacted sorbent that is utilized in the first reaction zone can be reduced and the additional load or reactive sites available on the NO_X reacted sorbent can be utilized.

During a wet regeneration process the reacted surfaces of the sorbent may be removed and the remaining sorbent may be refreshed. This will be understood with reference to Figure 26. In a wet regeneration, reacted sorbent is removed from a reaction zone, a reaction chamber in Figure 26, and washed in an aqueous dilute acid rinse. Since the interaction between pollutants and the sorbent is believed to be a surface-controlled phenomenon, only a small fraction of the oxides of manganese is reacted with the pollutant. It is this small fraction of the sorbent that can be removed by washing or rinsing which thereby "activates" the sorbent by making unreacted surface area available. The solubility in water of nitrates of manganese is greater than the solubility of sulfates of manganese by at least an order of magnitude in cold water and by at least several orders of magnitude in warm to hot water. This differential in solubility can be advantageously utilized in the regeneration process.

The sulfates and nitrates of manganese on the surface of the sorbent particles dissolve off into solution in the dilute acid bath, leaving clean sorbent that can be readily separated from the rinse or bath by known means, such as settling and decanting, filtering, centrifuging or other suitable techniques. As is further discussed below, the clear filtrate or solution containing dissolved sulfates and/or nitrates of manganese are directed to a regeneration vessel for regeneration of sorbent and production of useful by-products. The clean sorbent is then dried in, for example, a kiln to remove excess moisture. The heat for this drying step may be waste heat generated by combustion which is transferred or exchanged from combustion or process gases at an industrial or utility plant. After drying, the clean sorbent may be pulverized as necessary to reduce the clean sorbent to particle sizes useful in the system of the invention. The cleaned or "activated" sorbent is then conveyed or otherwise transported to the unreacted sorbent feeder(s) and thus, recycled.

Again with reference to Figure 26, the regeneration of sorbent and production of useful by-products can be understood. The solution or filtrate containing the dissolved sulfates and nitrates of manganese is passed from the acidic bath to a regeneration vessel to which alkali hydroxides such as potassium hydroxide (KOH) or sodium hydroxide (NaOH), or ammonium hydroxide (NH_4OH) is added. The addition of these hydroxides, yield respectively, a solution containing nitrates and/or sulfates of potassium, sodium, or ammonium and a precipitate of manganese hydroxide ($Mn(OH)_2$). These solutions can be made into fertilizer products or other products such as explosives. Air or oxygen may be bubbled into or otherwise introduced into the reaction vessel to further the regeneration of the sorbent. The precipitate may be removed with or without the prior introduction of air or oxygen and then dried and heated to form oxides of manganese, MnO_X where X is between about 1.5 to 2.0.

Instead of hydroxide compounds, soluble carbonate compounds, e.g., alkali carbonates, such as potassium carbonate (K_2CO_3), sodium carbonate (Na_2CO_3), or ammonium carbonate ($(NH_4)_2CO_3$) may be added to the solution or filtrate in an regeneration vessel. The addition of carbonates will yield a manganese carbonate precipitate and a solution containing nitrates and/or sulfates of potassium, sodium, or ammonium. The precipitate is separated from the solution, dried and heated to thermally decompose the manganese carbonate to form oxides of manganese and CO_2 gas which may be vented or captured and containerized as a marketable product. The oxides of manganese may be further heated in an oxidizing atmosphere to complete the sorbent regeneration, to form oxides of manganese, MnO_X where X is between about 1.5 to 2.0.The oxides of manganese are separated from the solution, much as the cleaned or reactivated sorbent after the acid wash step, and are then dried and pulverized before being conveyed to a virgin or unreacted sorbent feeder. The filtrate from the separation containing useful sulfates and nitrates that can then be further processed into marketable products.

Oxides of manganese may also be regenerated in a dry or thermal regeneration process, taking advantage of the thermal decomposition temperature(s) of nitrates of manganese. This regeneration process may be understood with reference to Figure 27. The process illustrated and discussed herein is based upon a removal process where NO_X is the target pollutant with nitrates of manganese being formed in the removal step in the reaction zone, a reaction chamber in Figure 27. The NO_X reacted sorbent is removed from the reaction chamber and conveyed to a first kiln. In the first kiln, the reacted sorbent is heated

to a temperature at or above the thermal decomposition temperature(s) of nitrates of manganese and NO_2 desorbs or is otherwise driven off. Oxides of manganese, MnO_X where X ranges from about 1.5 to 2.0 are formed in the first kiln which may be heated with waste process heat from the local plant. The regenerated oxides of manganese from the first kiln may be conveyed to a second kiln heated with waste process heat. Air or oxygen are introduced into the second kiln to more completely oxidize the regenerated sorbent so that the X of MnO_X ranges from about 1.5 to 2.0.

If the sorbent was SO_X-reacted the thermal regeneration would proceed much as described for NO_X, except the first kiln would be heated to a temperature at or above the thermal decomposition temperature of sulfates of manganese and SO_2 would desorb or otherwise driven off. With out being bound by theory, Applicants believe that nitrates of manganese thermally decompose at temperatures between about 130° C to about 260° C, while sulfates of manganese tend to liquefy at the temperatures over which nitrates of manganese thermally decompose. Applicants further believe that sulfates of manganese heated to these temperatures in the presence of a reducing agent, e.g., CO, H_2, etc., will decompose to SO_2 and MnO. Thus, if the sorbent were reacted with both SO_X and NO_X, NO_2 could be driven off first by heating reacted sorbent in a kiln to a first temperature at which nitrates of manganese thermally decompose so that NO_2 can be generated and directed for further processing. A reducing agent could then be introduced and the reacted sorbent further heated to desorb SO_2. Alternatively, the reacted sorbent could be heated to a second temperature, the thermal decomposition temperature of sulfates of manganese with SO_2 being desorbed and directed for further processing. The desorbed SO_2 can be directed to a wet scrubber containing water and an optional oxidant to form sulfuric acid. This acid liquor can then be marketed as is or further processed. This further processing would involve the addition of an ammonium or alkali hydroxide solution to form useful sulfates. In either case, the regenerated sorbent is further heated in an oxidizing atmosphere to more completely oxidize the regenerated sorbent so that the X of MnO_X ranges from about 1.5 to 2.0.Referring back to Figure 27, the desorbed NO_2 can be directed to a wet scrubber containing water and an oxidant to form nitric acid. This acid liquor can then be marketed as is or further processed. This further processing would involve the addition of an ammonium or alkali hydroxide solution to form useful nitrates, such as KOH as illustrated in figure 27.

In addition to regeneration of sorbent and production of useful by-products from the sulfates and nitrates of manganese, elemental mercury can be recovered from NO_X, SO_X

reacted sorbent that further has mercury compounds adsorbed thereon can be processed to generate and recover elemental mercury. The reacted sorbent is removed from a reaction zone of a system according to the invention and conveyed to a first kiln, the reacted sorbent is heated to a first temperature to desorb NO_2 which is routed for further processing into marketable products. The reacted sorbent is then heated a second temperature to desorb elemental mercury which is routed to a condenser for recovery as a marketable product. The sorbent is then rinsed to wash away any ash and to dissolve sulfates of manganese into solution to form a liquor. Any ash in the liquor is separated out and the ash routed for further handling. Alkali or ammonium hydroxide is added to the liquor to form an unreacted sorbent precipitate of oxides of manganese and a liquor containing alkali or ammonium sulfates. The liquor contains rinsed sorbent. The rinsed sorbent and unreacted sorbent precipitate and are separated from the liquor and the liquor is routed for further processing into marketable products or for distribution and/or sale as a useful by-product. The rinsed sorbent and sorbent precipitate are dried to form unreacted sorbent which can then be pulverized to de-agglomerate the unreacted sorbent.

Ion exchange can also be utilized as a mechanism for the separation and recovery of useful sulfate and nitrates. The dissolved sulfates and nitrates of manganese in the filtrate or solution left after washing SO_X and/or NO_X reacted sorbent can be processed in anion exchangers, permitting the recovery manganese cations and separation of the sulfate and nitrate anions. To accomplish this separation, the aqueous solution containing dissolved sulfates and nitrates is passed across or through a bed or column of an anion exchange resin that has an affinity for one of the two anions to remove one of the two anions. The resin with absorb the anion, for instance the sulfate, while permitting the nitrate to pass through the bed or column. Additionally, the solution stripped of sulfate can then be passed across or through a second bed or column of yet a second anion exchange resin to remove the nitrate. After the resin is loaded, the vessel containing the resin can be taken off-line and the resin therein stripped of the captured anion and recovered for reuse.

Suitable anion exchange resins and vessels are known to and readily identified by those skilled in the art. For purposes of illustration, the anion exchange resin has a chloride in the exchange position on the resin. The chloride is eluted while capturing the sulfate and/or nitrate anions. The solution, after passing through the anion exchanger or exchangers in series, will contain manganese chloride from which manganese carbonate or manganese hydroxide is precipitated with the addition of a soluble carbonate or hydroxide compound;

and oxides of manganese can be regenerated from the precipitate as discussed above. The sulfates and/or nitrates loaded on the resin can in turn be eluted with a solution containing chlorides of potassium, sodium or ammonium in order to generate useful sulfates and nitrate by-products for marketing or further processing. The filtrate or solution left over after precipitate formation can be utilized for this purpose.

Liquid mercury can also be recovered from mercury adsorbed to alumina in an alumina reactor. The mercury-reacted alumina from the reactor is heated to drive off or desorb mercury. The mercury vapor is then directed to a condenser where it is condensed to form liquid mercury which is a marketable product.

With the system and processes of the invention, CO and CO_2 in a gas stream can also be captured. Applicants believe that CO in a gas stream is oxidized to form CO_2 when contacted with the sorbent. The CO_2 in turn reacts with oxides of manganese to form $MnCO_3$. Thus, in the processes of the invention, manganese carbonate may be formed either during the capture of CO and CO_2 with the sorbent or during a regeneration step in which soluble carbonate compounds are utilized. Manganese carbonate is insoluble in water. Thus, sorbent that has been utilized to capture CO and CO_2 must be thermally regenerated. Sorbent loaded with manganese carbonate must be removed from the system of the invention and heated to thermally regenerate oxides of manganese, releasing CO_2 which may be compressed and containerized for sale or other marketable purposes. The heating of the loaded sorbent may be carried out in either two stages or in two separate heating units or kilns. In the first stage, the sorbent would be heated to thermally decompose the manganese carbonate, driving off CO_2 after which the sorbent would be further heated to complete the sorbent regeneration. In the second stage, the heating would continue either in the same or a second hearting unit or kiln. The second heating stage preferably would be in an oxidizing atmosphere being carried out with the introduction of air or oxygen in order to complete the regeneration of the sorbent to form oxides of manganese, MnO_X where X is between about 1.5 to 2.0.

The above examples of regeneration processes are provided by way of example and are not intended to limit the processes, both known and unknown, for regeneration of oxides of manganese and for recovery of useful and marketable by-products that may be incorporated into the processes of the invention.

The combustion of fossil fuels (e.g., coal, oil, and natural gas) liberates three major air pollutants: (1) particulates (2) sulfur dioxide (SO_2) and (3) oxides of nitrogen (NO_X). Wet scrubbing, electrostatic precipitators and bag houses can remove particulates such as fly ash. Using mechanical filters or electrostatic precipitators does not remove SO_2, SO_3, NO_2, N_2O_4, NO, or N_2O_3. Prior technologies have used wet scrubbing for the process as a means of sorbing SO_X and NO_X. Water is effective as a scrubbing medium for the removal of SO_2; removal efficiencies can be improved by the addition of chemical absorbents such as calcium, magnesium and sodium. However, nitrogen oxide (NO) is essentially insoluble in water, even with the use of sorbtion chemicals. Residence times required and liquid-to-gas surface areas have proven to be impractical where high gas flow rates are encountered such as boiler flue gas.

Some of the economics involved in the wet scrubbing process involve high-energy consumption; on the average 4-5% of a plant gross power generation is consumed in the process. For example: (1) high differential pressure of a venturi / absorber tower requires 30" of WC or a bag house and scrubber combination requires even higher static pressures. (2) Large volumes of high pressure scrubbing liquor injected through nozzles into the scrubbing apparatus. (3) Slurry tanks requiring continual vigorous agitation. (4) High horsepower required to force water-saturated non-buoyant flue gas up the stack.

Environmental drawbacks of existing systems include large quantities of minerals used as sorbents and the insoluble sulfites or sulfate formed from the scrubbing reaction. The precipitate is then taken to landfills or holding ponds. Some other disadvantages of existing systems are fouling of the internal scrubber components with hard scale, increasing operational labor and maintenance costs. Some complex regenerative systems use large quantities of chemicals required to react with the millions of gallons of slurry used every day.

The dry scrubbing process described in this patent is effective in removing nearly all NO_X and SO_X. Differential pressure requirements through the scrubber should typically not exceed 10 inches of water column and residence times within the sorbent cake are typically less than 1 second. Volumes of sorbent used in this invention in comparison to the wet slurry volumes are miniscule and recharging of reaction zones are done periodically. While stack gases remain dry and hot, some waste heat will be used in the drying of washed and re-generated sorbent. Operational costs of the reaction zone(s) are similar to operating an ash bag house; also capital expenditures are estimated to be reasonable requiring standard off-the-shelf equipment and instrumentation.

As a summary, the equipment is used in the dry scrubbing process is much less complex than the wet scrubber process thus requiring lower operational maintenance costs and a reduced operating staff. Chemical and raw material costs are expected to be similar with less waste effluent produced. The major cost savings will be in the reduced power consumption expected to be significantly less than that of a wet scrubbing system, with fan horsepower reduction making up the majority of the savings.

Although economics favor the use of dry scrubbing processes, removal of target pollutants with the sorbent can be accomplished with wet methods or combinations of wet and dry methods. In the system of the invention, wet scrubbers can serve as reaction zones in the place of or in combination with the reaction zones previously described for dry removal. Wet scrubbers useful in the systems and processes of the invention may be of several types, including but not limited to slurry, sprayer, cascading, and others known to those skilled in the art. Whether the wet scrubber is a slurry, sprayer, cascading or other know type of scrubber, an acidic slurry of oxides of manganese is utilized. The acidity serves to enhance the effective removal of the target pollutants. For SO_X and/or NO_X removal, the pH of the slurry is preferably 2.0 or less and more preferably between about 1.5 and about 1.75

The gas should be introduced into the wet scrubber at a temperature below the boiling point of the solution or slurry to prevent excess evaporation of the sorbent slurry. Since gases processed in the system of the invention typically are at elevated temperatures, the gas may be cooled to below the boiling point utilizing a heat exchangers 72A, 72B preceding the reaction zone as is shown in Figure 5. The gas containing target pollutants is introduced into the wet scrubber and contacted with the slurry for a time sufficient to effect capture of a target pollutant at a target capture rate set point for pollutants such as SO_X and/or NO_X, CO and/or CO_2 or TRS, forming respectively, sulfates and/or nitrates of manganese, manganese carbonate, or sulfates of manganese. While the sorbent itself is not soluble in the slurry, reactions products such as sulfates and nitrates of manganese are and dissolve immediately into solution. Manganese carbonate, being insoluble in aqueous solution, does not dissolve.

With respect to the removal of CO and/or CO_2 to manganese carbonate, when the sorbent is no longer effective for pollutant removal at a target capture rate set point, the reacted sorbent must be separated from the slurry for regeneration of the sorbent and recovery of useful by-products. This is accomplished through the thermal decomposition of the manganese carbonate as previously described.

With respect to the sulfates and/or nitrates of manganese formed in wet removal, the sorbent must be periodically separated from the solution. The sorbent, which is by virtue of being in solution, is essentially clean or "activated" and can be returned to the scrubber in a slurry or added to a slurry requiring additional sorbent. The point at which periodic separation would need to be carried out generally depends upon the capacity of the slurry to retain additional solute in solution, the saturation point of the solution. The frequency at which separation must be carried may be affected through temperature adjustment, since generally a saturated solution can dissolve additional solute at increased temperatures. However, as previously noted, the temperature should not be increased to the boiling point of the solution. Further, simply increasing the volume of the slurry with the addition of acidic aqueous solution can decrease the separation frequency, as long as the wet scrubber has sufficient capacity for the increased volume of the slurry. Further still, the periodic separation can be minimized by bleeding of the aqueous solution containing solute from the scrubber and with simultaneous feeding of additional, fresh aqueous solution into the scrubber to maintain the slurry in an unsaturated state. The solution that has been bled from the wet scrubber can be retained in a holding tank, vessel or other suitable container until a sufficient volume has be accumulated and then processed to regenerate oxides of manganese and to recover useful and marketable by-products.

In a single stage wet removal process a single reaction zone, a wet scrubber, is utilized to remove the target pollutants.. The rate of reaction is related to the solubility of the reaction product of target pollutant and the sorbent. For example the solubility of SO_X is greater than the solubility of NO_X in an aqueous solution; and therefore, a longer residence time is required for NO_X removal than for SO_X removal. The gas once substantially stripped of the target pollutant is vented from the scrubber either to a stack or for further processing. A single wet scrubber can be utilized to remove one or more target pollutants; however, the residence time of the gas in the wet scrubber will be driven by a combination of the solubility of the less soluble of the target pollutants and the target capture rate of that target pollutant. In a single-stage wet removal system the gas is introduced into a reaction zone configured for the introduction of the gas and contacted with the sorbent containing slurry for a time sufficient to effect capture of the target pollutant(s) at the target capture rate set point of the target pollutant(s). The target pollutant is captured by reaction with the sorbent to form reaction products. The reaction products may be soluble in the aqueous solution, as with nitrates and sulfates of manganese. Or they may be insoluble as with carbonates of

manganese formed during the removal of CO and/or CO_2. Wet removal methods can be utilized in either case; but are better suited for removal of target pollutants that form soluble reaction products.

Wet removal can also be accomplished in multiple stages with at least two reaction zones in series of which at least one of the reaction zones is a wet scrubbers. This can be illustrated with reference to dual-stage removal of SO_X and NO_X. In dual-stage removal, first and second reaction zones are provided. With both reactions zones being wet scrubber s, gas is introduced into the first reaction zone which is configured for the introduction of a gas containing target pollutants, in this case SO_X and NO_X. In the first reaction zone, the gas is contacted with a sorbent containing slurry for a time sufficient to effect SO_X capture at a target SO_X capture rate set point. The SO_X is captured by reacting with the sorbent to form sulfates of manganese to substantially strip the gas of SO_X. The gas which is substantially stripped of SO_X is vented from the first reaction zone and passed to a second reaction zone, also a wet scrubber, configured for the introduction of the gas substantially stripped of SO_X. In the second wet scrubber the gas is contacted with the sorbent containing slurry for a time sufficient to effect NO_X capture at a target NO_X capture rate set point. The NO_X is captured by reacting with the sorbent to form nitrates of manganese to substantially strip the gas of NO_X. The gas that has been substantially stripped of SO_X and NO_X is vented from the second reaction zone. It is readily understood by those skilled in the art that more than two wet scrubbers could be utilized in series to effect capture of multiple target pollutants and that the sequence of pollutant removal, in a multi-stage removal process, would be determined by the relative solubilities of the reaction products generated from target pollutants with the sorbent.

Dual stage removal may also be carried out with one of the reaction zones being a wet scrubber and the other reaction zone being selected from the group consisting of a fluidized bed, a pseudo-fluidized bed, a reaction column, a fixed bed, a pipe/duct reactor, a moving bed, a bag house, an inverted bag house, bag house reactor, serpentine reactor, and a cyclone/multiclone. Again using SO_X and NO_X for illustrative purposes, the removal can proceed by first wet SO_X removal and dry NOx removal or by first dry SO_X removal and wet NO_X removal. Regardless of the sequence, wet removal and dry removal would proceed as previously described, with the gas substantially stripped of SO_X being directed from the first reaction zone to the second reaction where NO_X removal would occur. In a wet-dry removal system, the first reaction zone would be a wet scrubber; and in dry-wet removal system the second reaction zone would be a wet scrubber. For the dry removal stage, the dry reaction

zone or scrubber , whether the first or second in sequence, is selected from the aforementioned group.

Where the reaction product of the target pollutant is soluble in aqueous solution, the surface area of the oxide of manganese sorbent is not as critical in a wet removal system, i.e., a scrubber, as opposed to a dry removal system. Further, particle size may not be as critical with a liquid medium as opposed to a gas medium; however, particles must be small enough so that the sorbent remains sufficiently mixed in the slurry. Agitators can be used to keep the sorbent sufficiently mixed in the slurry. Generally, oxides of manganese useful as a sorbent for dry removal methods are similarly useful for wet removal methods.

The systems of the invention including those that incorporate wet scrubbers are adaptable; and process parameters, such as differential pressure, inlet gas temperature, and removal efficiency, are monitored and controlled in wet removal systems of the invention with electronic controls just as in dry removal systems according to the invention.

While exemplary embodiments of this invention and methods of practicing the same have been illustrated and described, it should be understood that various changes, adaptations, and modifications might be made therein without departing from the spirit of the invention and the scope of the appended claims.

CLAIMS

1. An adaptable system for wet removal of target pollutants from gases with minimal differential pressure across the system, comprising:

at least one reaction zone, the reaction zone being a wet scrubber supplied with an acidic aqueous slurry of a sorbent of regenerable oxides of manganese, the reaction zone being configured for introduction of a gas containing at least one target pollutant at a temperature below the boiling point of the slurry and contacted with the sorbent therein for a time sufficient to effect capture of the target pollutant at a targeted capture rate set point for the target pollutant, the gas being substantially stripped of the target pollutant through the formation of a reaction product of the target pollutant and the oxides of manganese, the reaction zone being further configured to allow the gas to be vented from the reaction zone; and wherein differential pressure across the system is regulated so that any differential pressure across the system is no greater than a predetermined level

2. Adaptable system as claimed in Claim 1 wherein the system is comprised of two reaction zones, the two reaction zones being a first reaction zone and a second reaction zone.

3. Adaptable system as claimed in Claim 2 wherein the first and second reactions zones are both wet scrubbers.

4. Adaptable system as claimed in Claim 2 wherein the first reaction zone is a dry scrubber selected from the group consisting of a fluidized bed, a pseudo-fluidized bed, a reaction column, a fixed bed, a pipe/duct reactor, a moving bed, a bag house, an inverted bag house, bag house reactor, serpentine reactor, and a cyclone/multiclone and the second reaction zone is a wet scrubber.

5. Adaptable system as claimed in Claim 2 wherein the first reaction zone is a wet scrubber and the second reaction zone is a dry scrubber selected from the group consisting of a fluidized bed, a pseudo-fluidized bed, a reaction column, a fixed bed, a pipe/duct reactor, a moving bed, a bag house, an inverted bag house, bag house reactor, serpentine reactor, and a cyclone/multiclone.

6. Adaptable system as claimed in Claim 1wherein the target pollutant is SO_X and the reaction product formed is sulfates of manganese or the target pollutant is NO_X and the reaction product formed is nitrates of manganese.

7. Adaptable system as claimed in Claim 2 wherein the target pollutants are SO_X and NO_X and SOx is captured in the first reaction zone with sulfates of manganese being

formed as the reaction product and NO_X is captured in the second reaction zone with nitrates of manganese being formed as the reaction product.

8. Adaptable system as claimed in Claim 4 or claim 5 wherein SO_X is captured in the first reaction zone and NO_X is captured in the second reaction zone.

9. Adaptable system of claim 1 wherein the regenerable oxides of manganese, upon regeneration, are in particle form and are defined by the chemical formula MnO_X, where X is about 1.5 to 2.0 and wherein the oxides of manganese have a particle size of less than about 0.1 to about 500 microns and a BET value ranging from about 1 to about $1000m^2/g$.

10. An adaptable system for dry removal of carbon monoxide and/or carbon dioxide from gases with minimal differential pressure across the system, comprising:

A. A feeder containing a supply of sorbent of regenerable oxides of manganese and/or regenerated oxides of manganese; wherein the feeder is configured to handle and feed oxides of manganese which, upon regeneration, are in particle form and are defined by the chemical formula MnO_X, where X is about 1.5 to 2.0 and wherein the oxides of manganese have a particle size of less than about 0.1 to about 500 microns and a BET value ranging from about 1 to about $1000m^2/g$;

B. At least one reaction zone configured for introduction of the sorbent and a gas containing carbon monoxide and/or carbon dioxide where the gas is introduced at temperatures typically ranging from ambient temperature to below the thermal decomposition temperature(s) of carbonates of manganese carbonate and contacted with the sorbent for a time sufficient to effect the capture of carbon monoxide and/or carbon dioxide at a targeted capture rate set point, the carbon monoxide and/or carbon dioxide being captured by reacting with the sorbent to formcarbonates of manganese to substantially strip the gas of carbon monoxide and/or carbon dioxide the reaction zone being further configured to render the gas that has been substantially stripped of carbon monoxide and/or carbon dioxide free of reacted and unreacted sorbent so that the gas may be vented from the reaction zone; and wherein differential pressure within the system is regulated so that any differential pressure across the system is no greater than a predetermined level.

11. A process for the removal of target pollutants from a gas stream with a system incorporating wet removal comprising the steps of:

A. providing a system according to Claim 1;

B. introducing a gas containing a target pollutant into the reaction of the system;

C. contacting the gas with the sorbent in the sorbent slurry of the system for a time sufficient to effect the capture of the target pollutant at a targeted capture rate set point for the target pollutant through the formation of a reaction product of the target pollutant and oxides of manganese to substantially strip the gas of the target pollutant; and

D. venting the gas from the reaction zone.

12. Process for the removal of target pollutants from a gas stream with a system incorporating wet removal as claimed in Claim 11, wherein the target pollutant is SO_X with sulfates of manganese being the reaction product which is dissolved in solution in the slurry, the process further comprising the steps of:

E. separating the sorbent from the slurry to provide a solution containing dissolved sulfates of manganese; and

F. routing the solution for further processing to regenerate oxides of manganese and recover useful sulfate by-products.

13. Process for the removal of target pollutants from a gas stream with a system incorporating wet removal as claimed in Claim 11, wherein the target pollutant is NO_X with nitrates of manganese being the reaction product which is dissolved in solution in the slurry, the process further comprising the steps of:

E. separating the sorbent from the slurry to provide a solution containing dissolved nitrates of manganese; and

F. routing the solution for further processing to regenerate oxides of manganese and recover useful nitrate by-products.

14. A process for the removal of target pollutants from a gas stream with a system incorporating wet removal comprising the steps of :

A. providing a system as claimed in Claims 2, 3 or 4;

B. introducing a gas containing at least two target pollutants into the first reaction zone of the system;

C. contacting the gas in the first reaction zone with the sorbent for a time sufficient to effect capture of a first target pollutant at a targeted capture rate set point for the first target pollutant through the formation of a reaction product of the first target pollutant and oxides of manganese to substantially strip the gas of the first target pollutant;

D. venting the gas from the first reaction zone;

E. introducing the gas vented from the first reaction zone into the second reaction zone of the system;

F. contacting the gas in the second reaction zone with the sorbent for a time sufficient to effect capture of a second target pollutant at a targeted capture rate set point for the second target pollutant through the formation of a reaction product of the second target pollutant and oxides of manganese to substantially strip the gas of the second target pollutant; and

G. venting the gas from the second reaction zone of the system.

15. Process for the regeneration of oxides of manganese from a solution containing sulfate and nitrate anions and manganese cations formed when the reaction product of the removal of SO_X and NO_X from a gas stream with a sorbent of oxides of manganese, comprising the steps of:

A. providing first and second anion exchangers having an anion exchange resin loaded therein, the anion exchange resin having chloride in the exchange position on the resin;

B. passing a solution containing sulfate and nitrate anions through the first anion exchanger to elute the chloride to form manganese chloride while capturing the sulfate anion on the resin;

C. passing the solution containing nitrate anions through the second anion exchanger to elute the chloride to form manganese chloride while capturing the nitrate anion on the resin;

D. adding a soluble carbonate or hydroxide compound to the solution to precipitate manganese carbonate or manganese hydroxide;

D. separating the manganese carbonate or manganese hydroxide from the solution; and

E. heating the manganese carbonate or manganese hydroxide to form regenerated oxides of manganese.

Figure 1

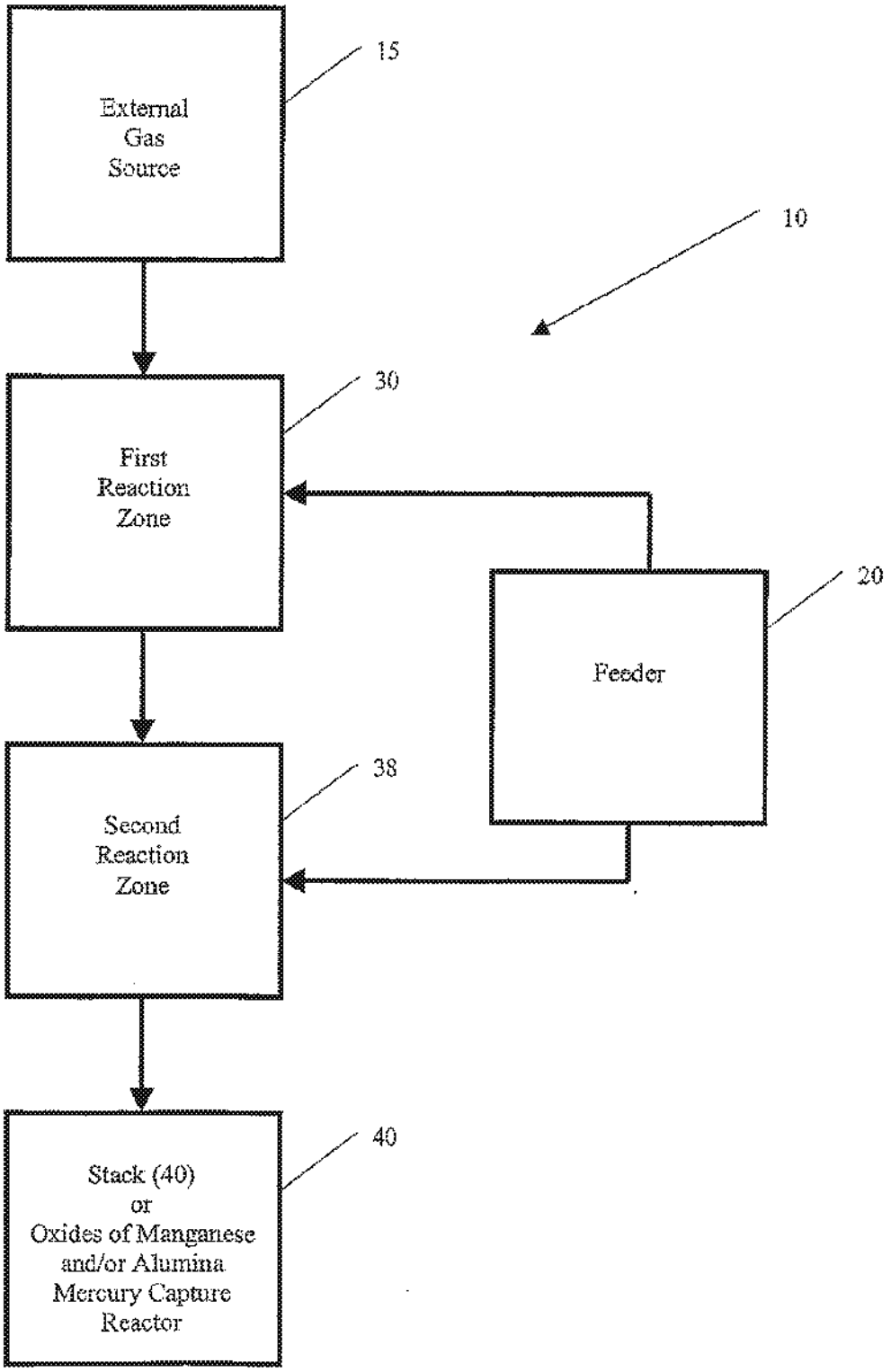

External Gas Source
15
10
First Reaction Zone
30
Feeder
20
Second Reaction Zone
38
Stack (40) or Oxides of Manganese and/or Alumina Mercury Capture Reactor
40

Figure 2

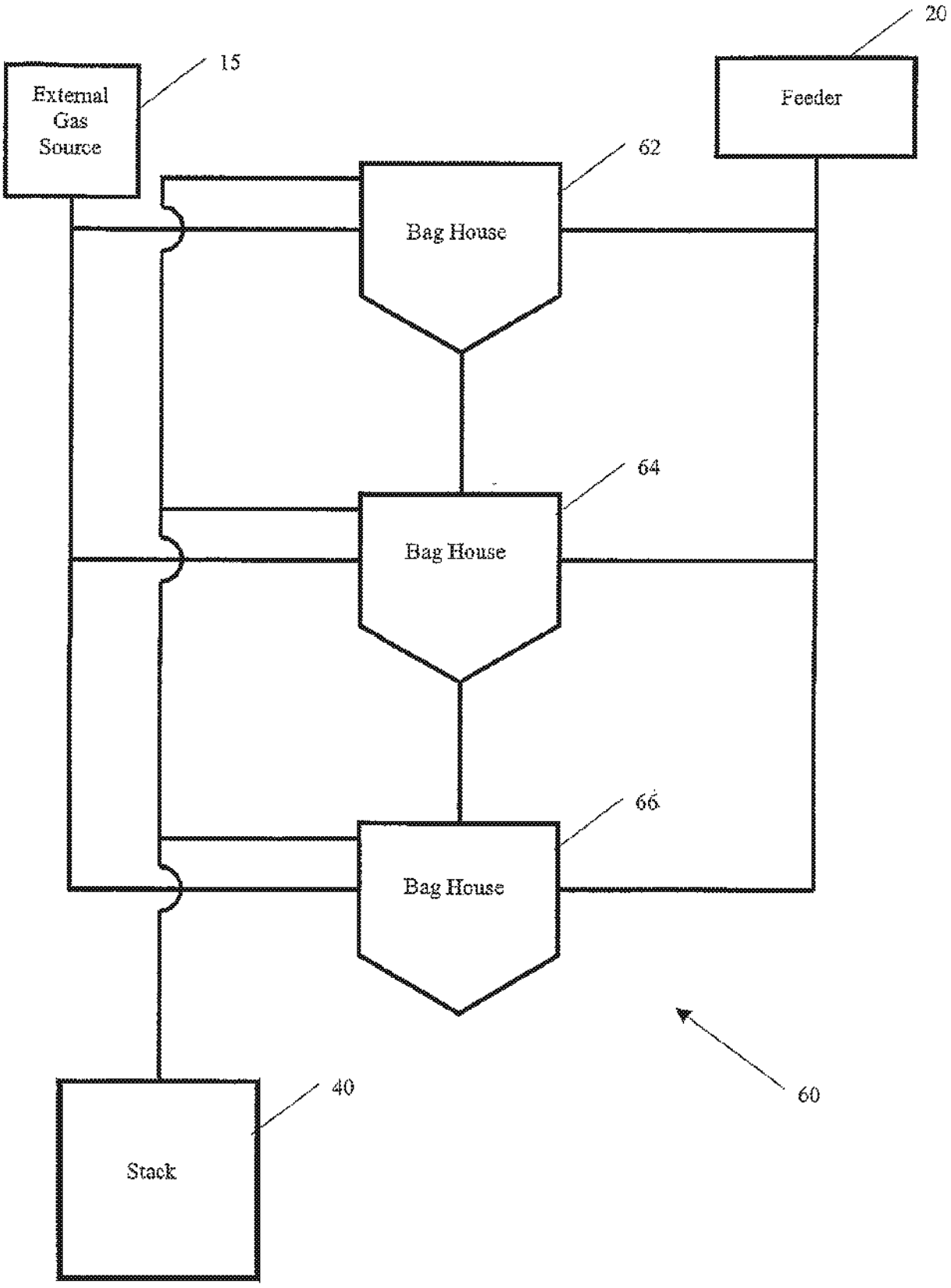
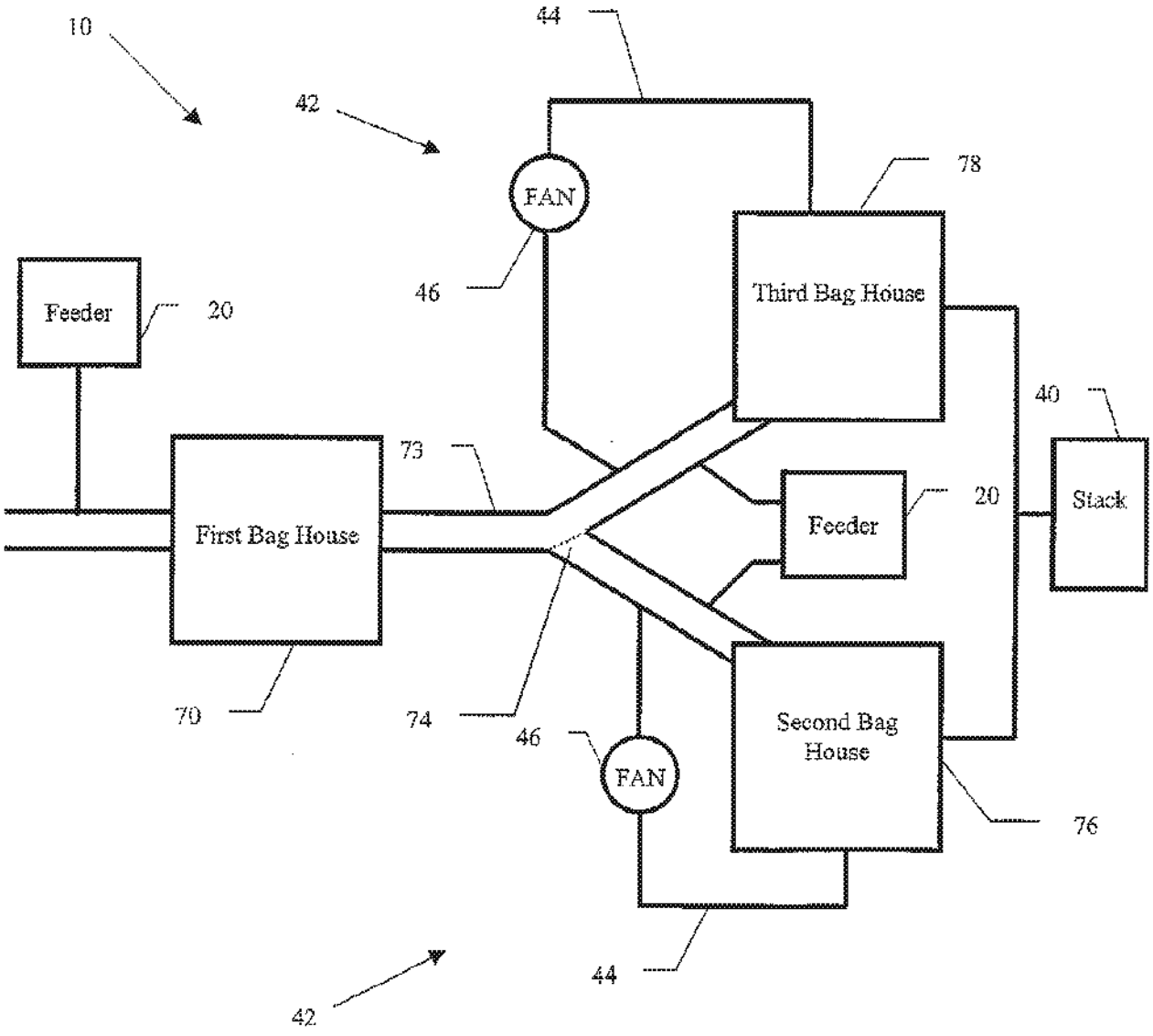

20
15
External Gas Source
Feeder
62
Bag House
64
Bag House
66
Bag House
60
40
Stack

Figure 3


10
42
44
FAN
78
46
Feeder
20
Third Bag House
40
73
First Bag House
Feeder
20
Stack
70
74
46
FAN
Second Bag House
76
44
42

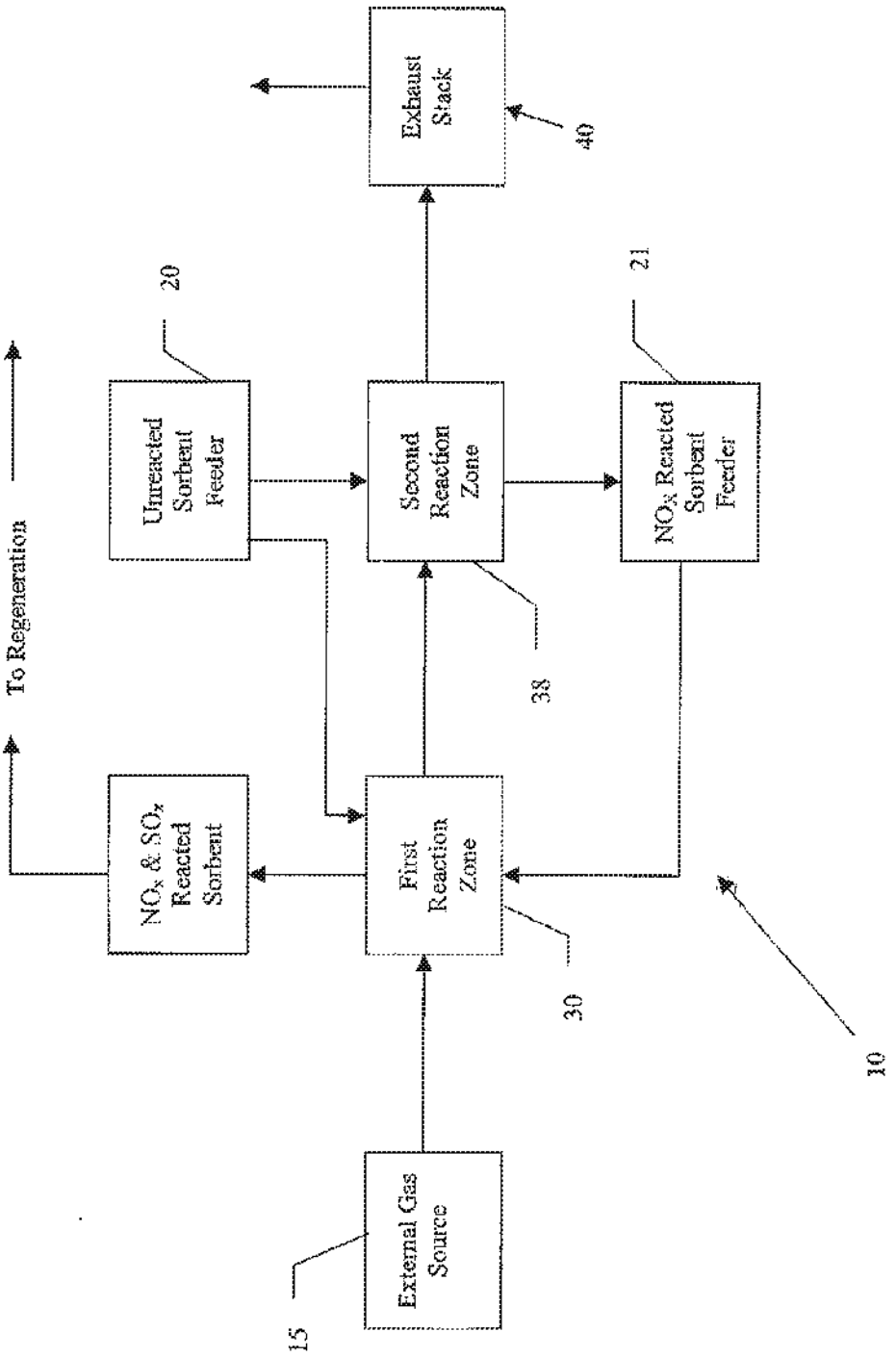
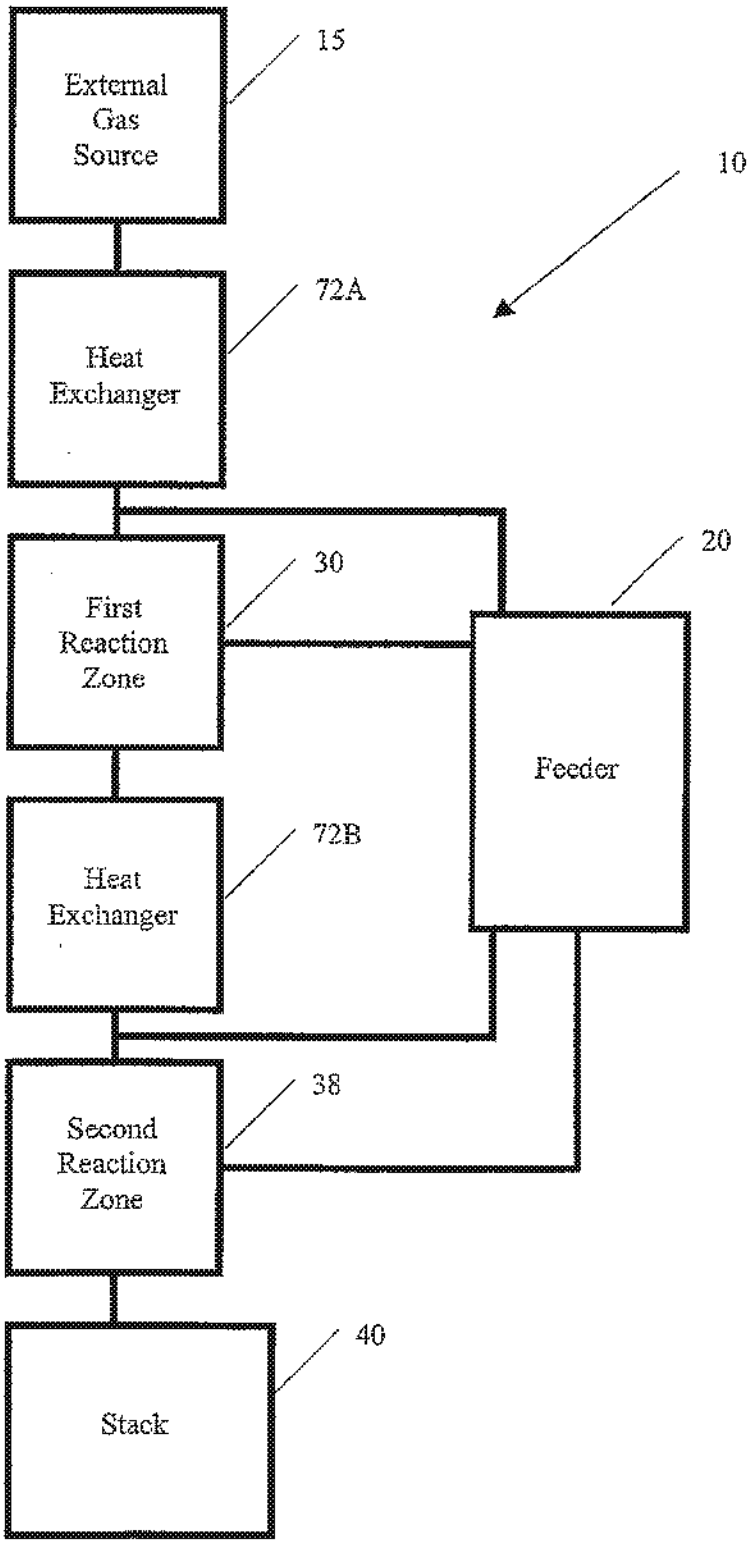

Exhaust Stack
40
20
21
Unreacted Sorbent Feeder
Second Reaction Zone
NO_x Reacted Sorbent Feeder
To Regeneration
38
NO_x & SO_x Reacted Sorbent
First Reaction Zone
30
10
External Gas Source
15

Figure 4

Figure 5


External Gas Source
15
10
72A
Heat Exchanger
First Reaction Zone
30
20
Feeder
72B
Heat Exchanger
Second Reaction Zone
38
Stack
40

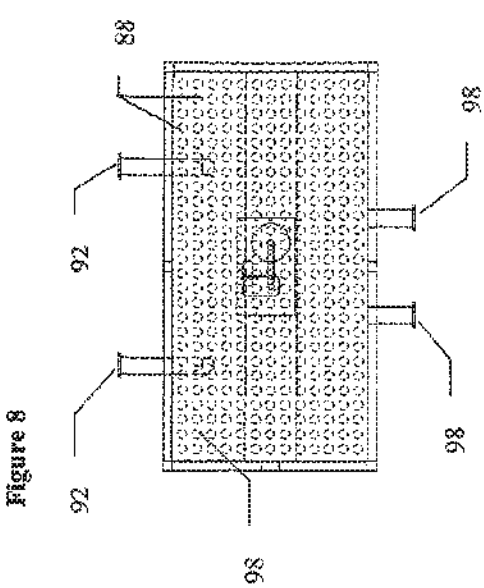


Figure 8

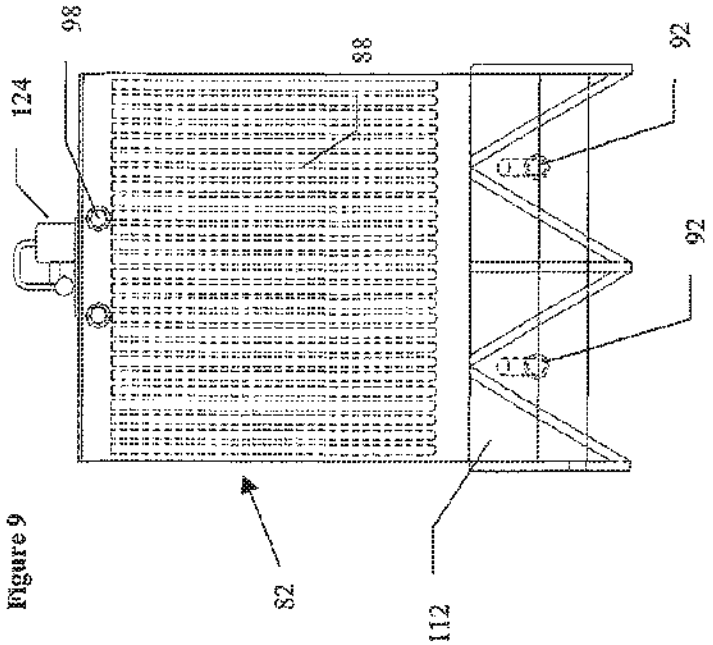


Figure 9

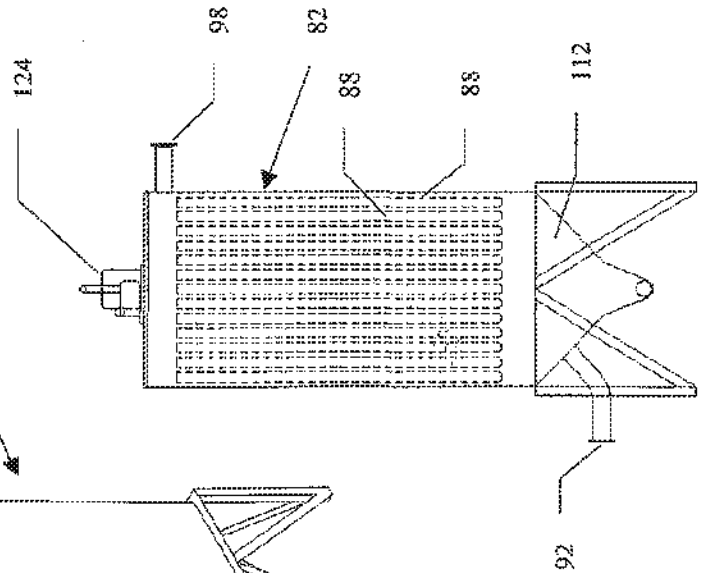


Figure 7

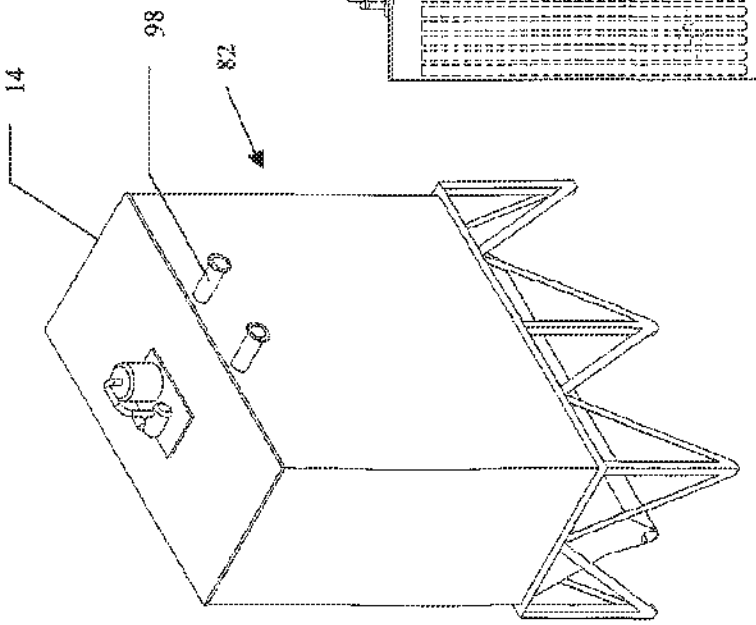


Figure 6

Figure 10

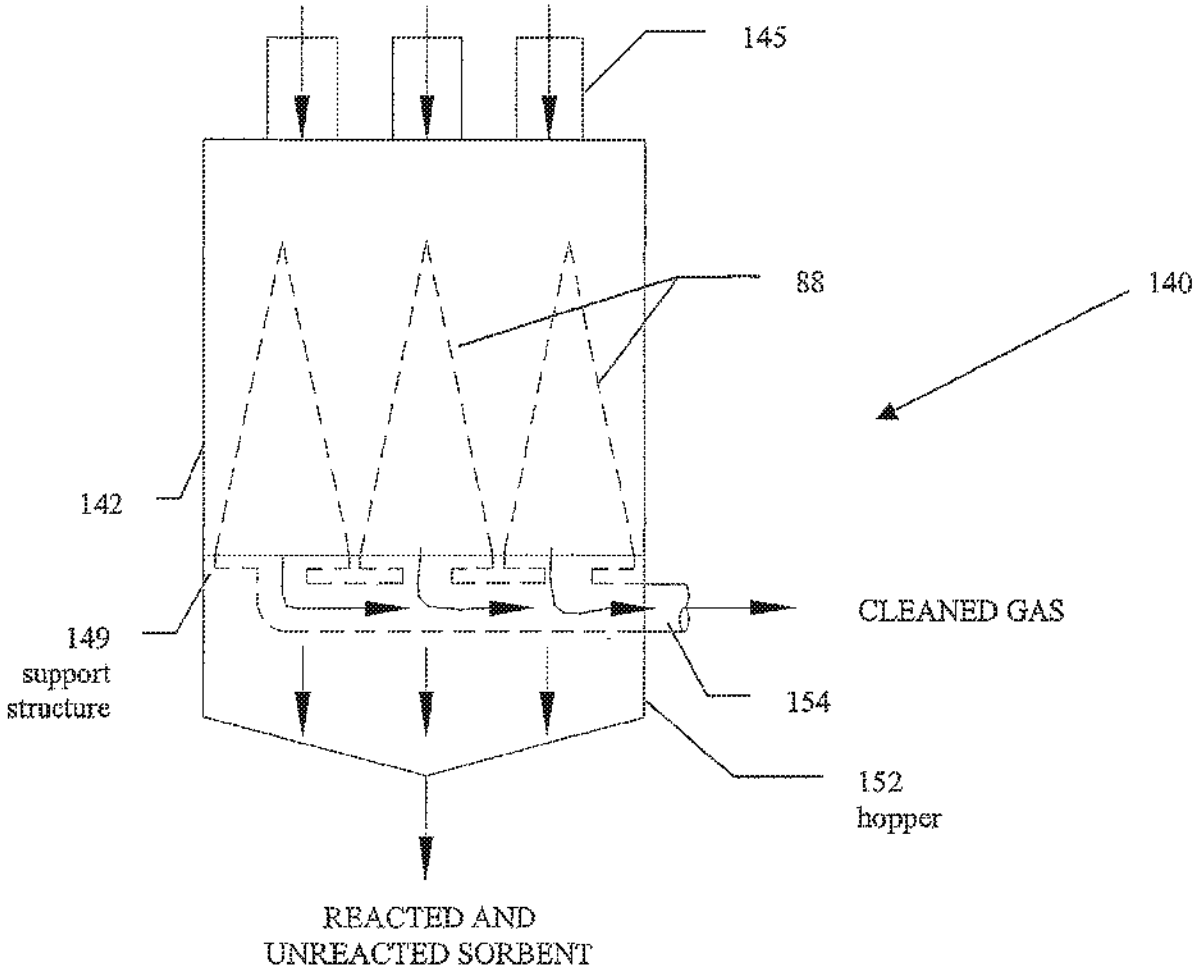


Figure 11

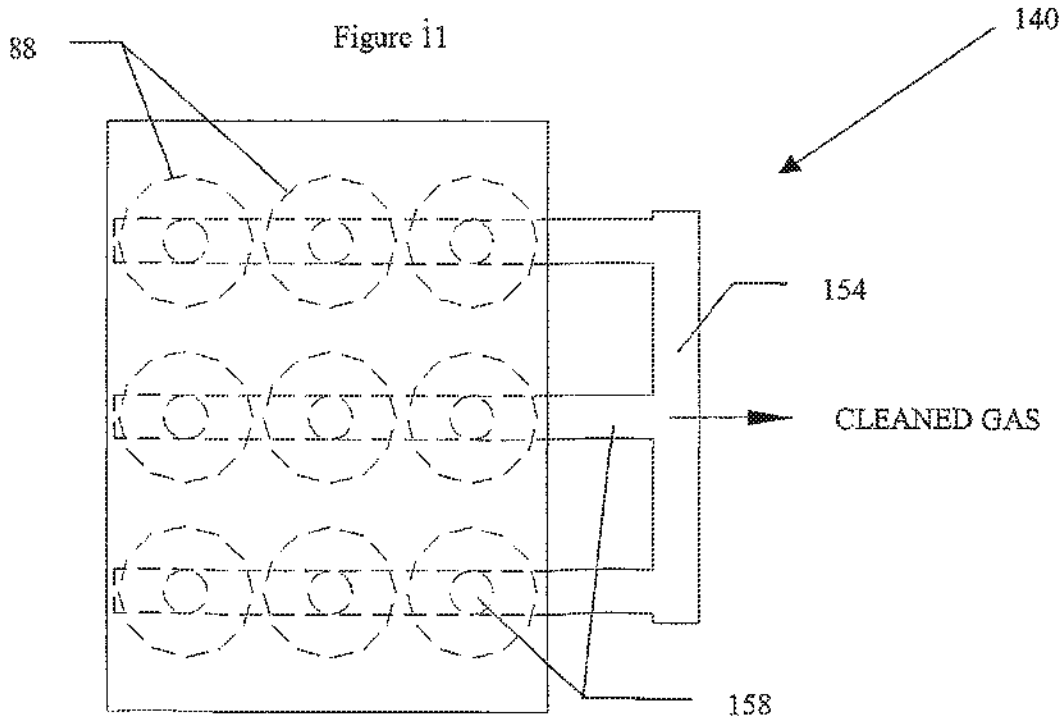


Figure 12

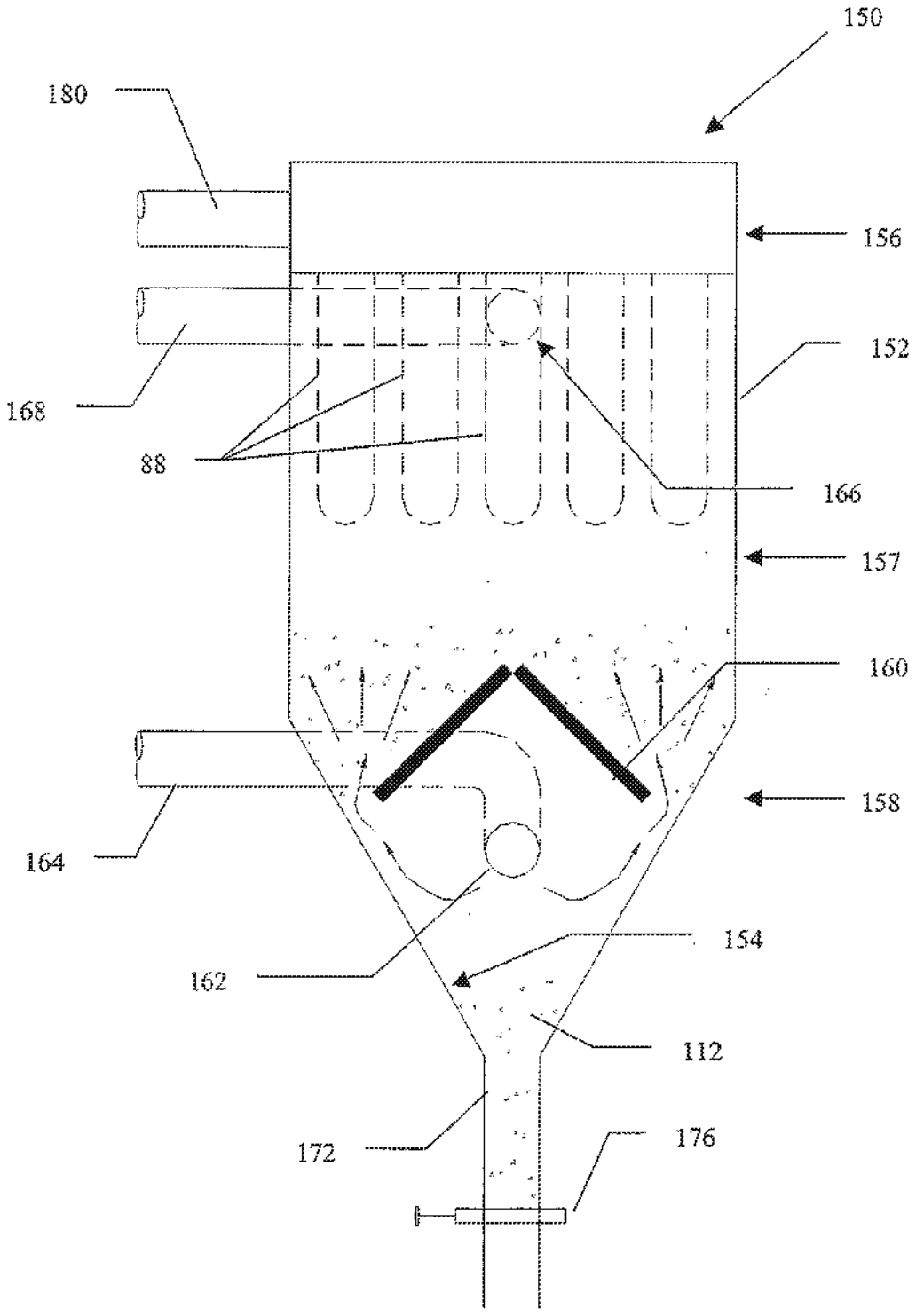


Figure 13

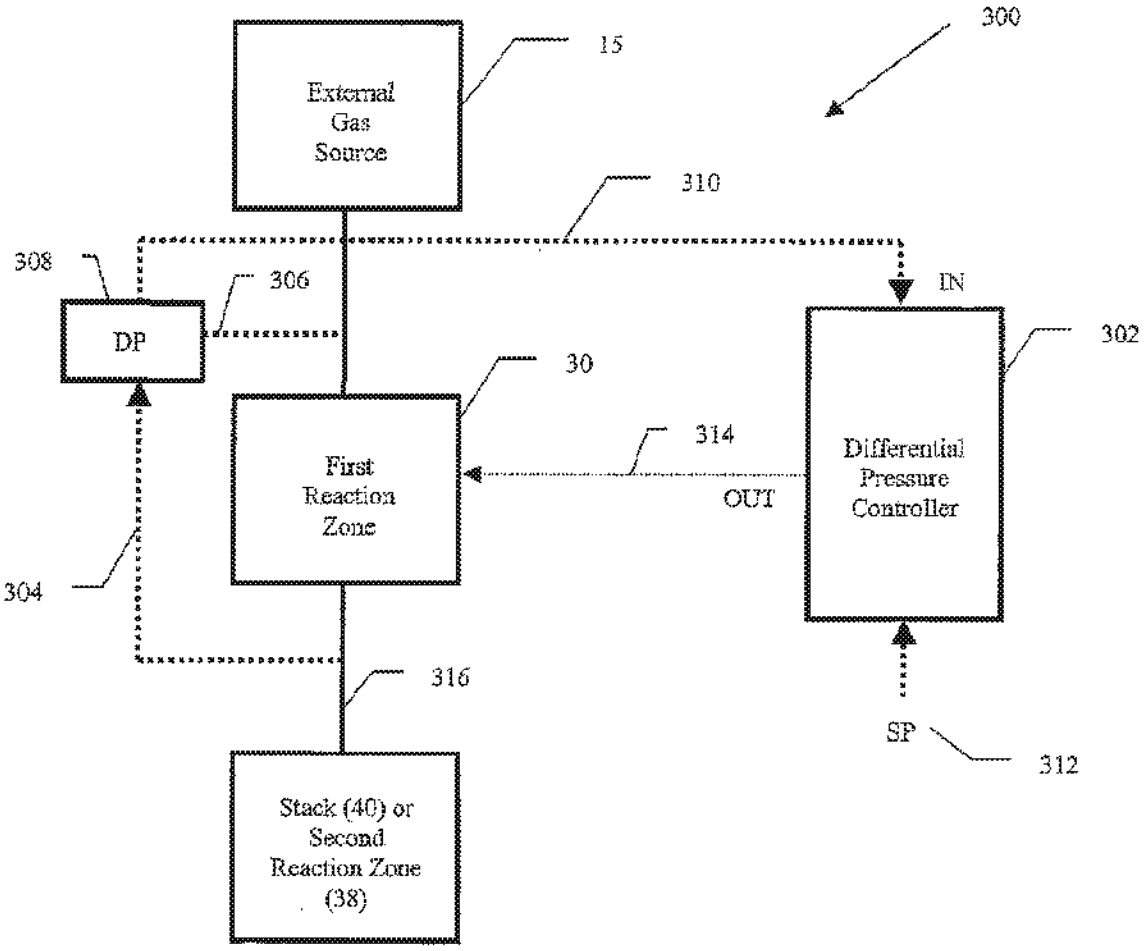

External Gas Source
15
300
310
308
306
IN
DP
302
30
First Reaction Zone
314
Differential Pressure Controller
OUT
304
316
SP
312
Stack (40) or Second Reaction Zone (38)

Figure 14

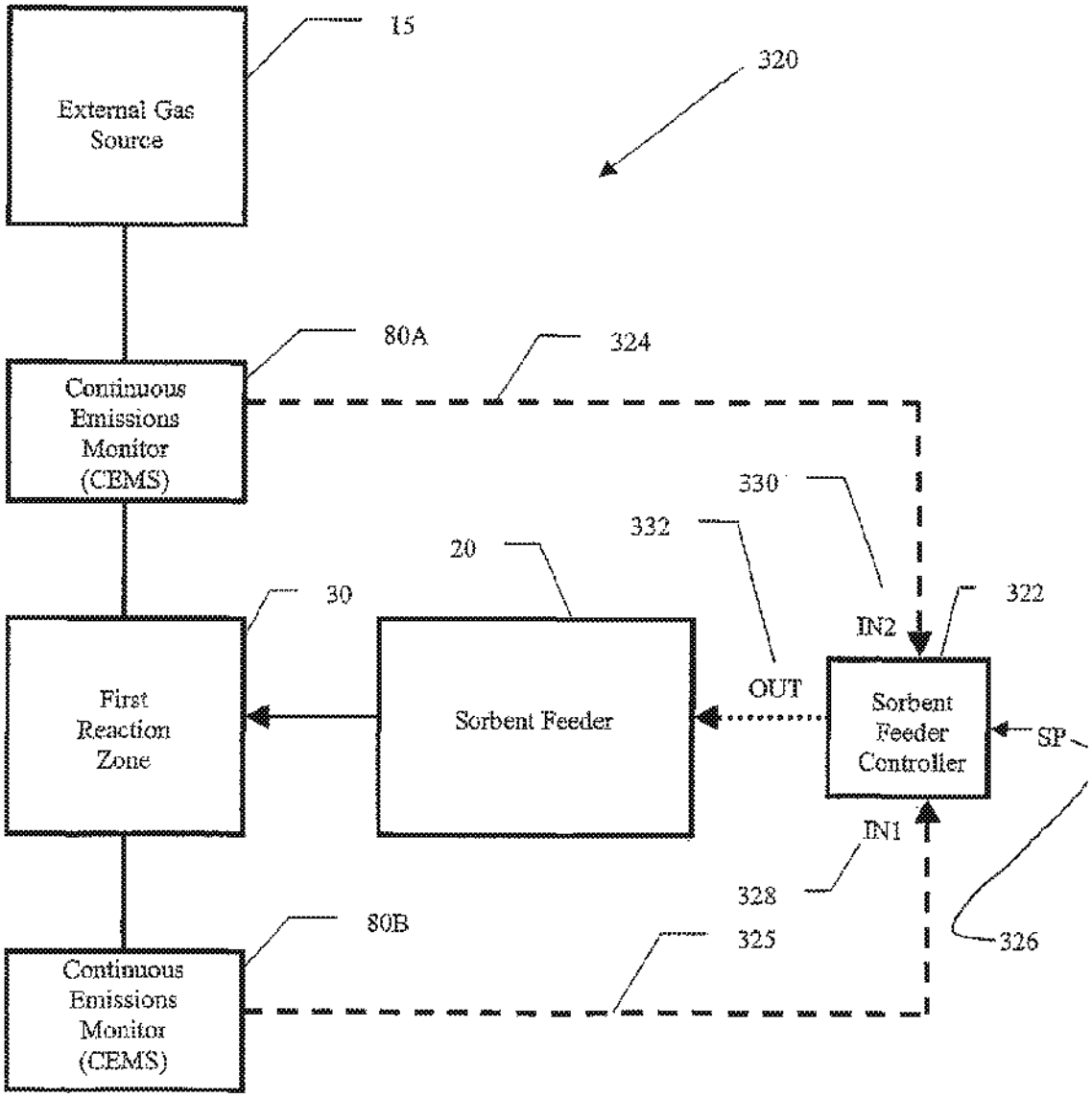

15
320
External Gas Source
80A
324
Continuous Emissions Monitor (CEMS)
330
332
20
30
322
IN2
First Reaction Zone
Sorbent Feeder
OUT
Sorbent Feeder Controller
SP
IN1
328
326
80B
325
Continuous Emissions Monitor (CEMS)

Figure 15

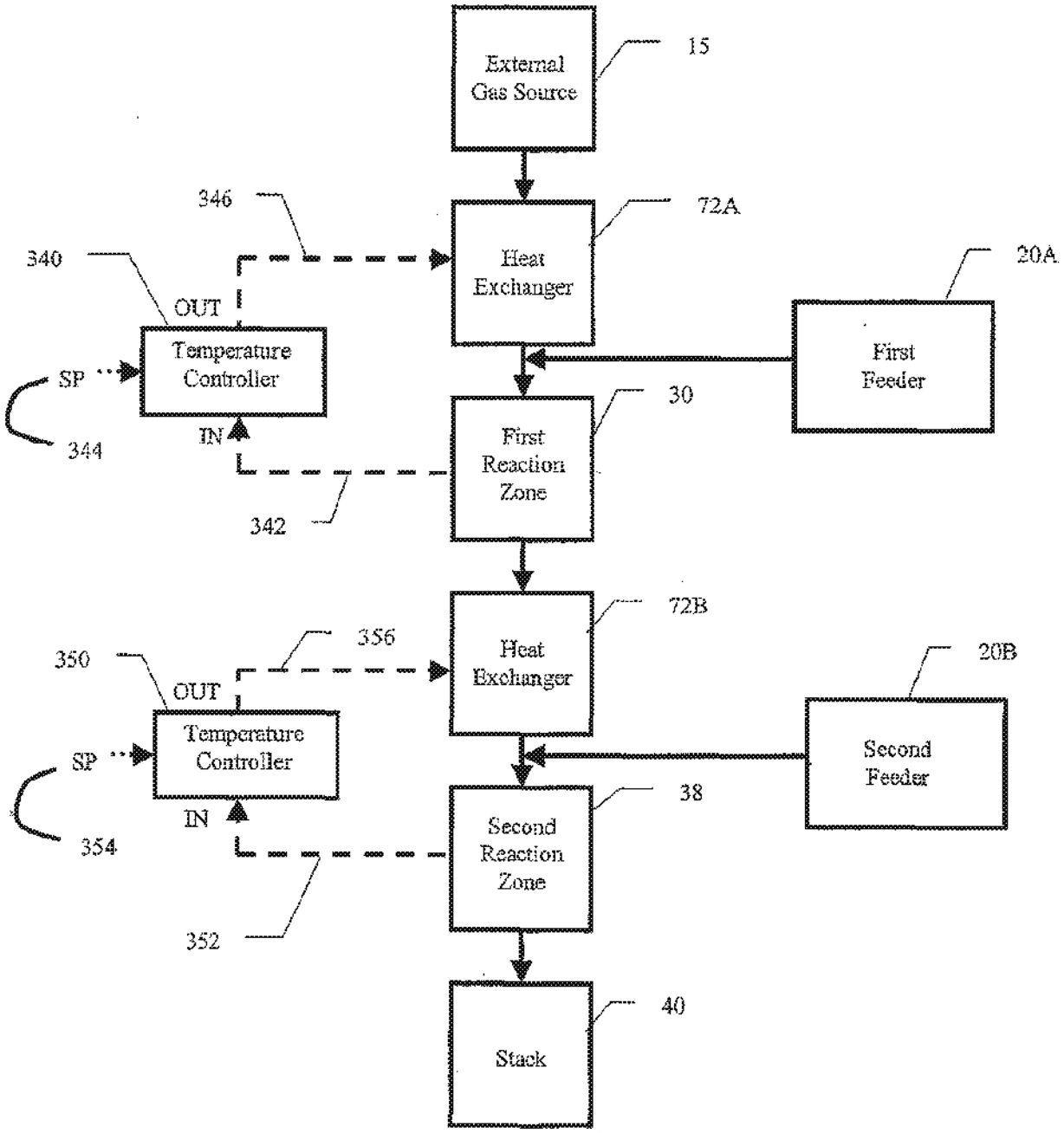

External Gas Source
15
346
72A
340
Heat Exchanger
20A
OUT
Temperature Controller
First Feeder
SP
30
IN
344
First Reaction Zone
342
72B
356
350
Heat Exchanger
20B
OUT
Temperature Controller
Second Feeder
SP
38
IN
354
Second Reaction Zone
352
40
Stack

Figure 16

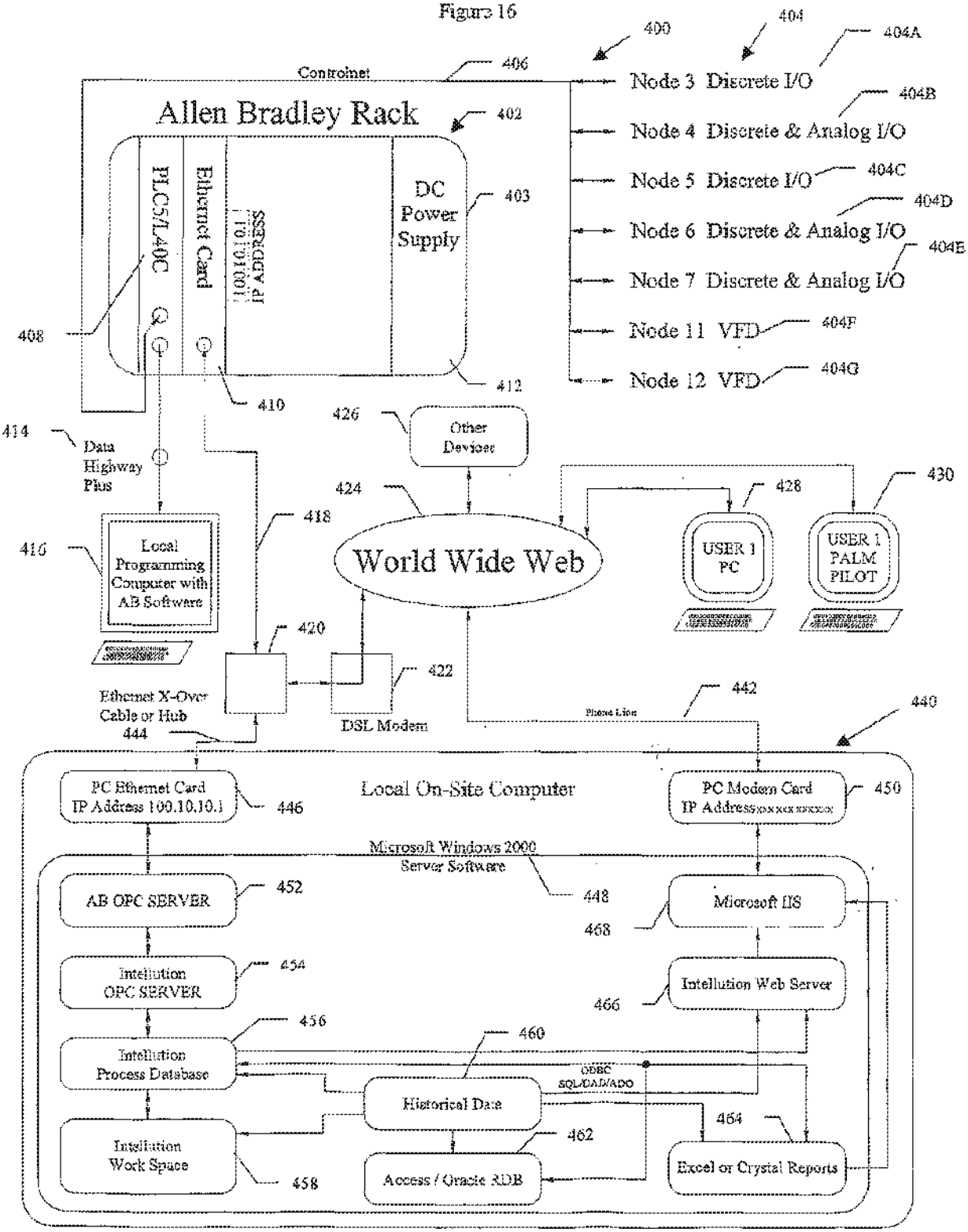

400
404
404A
Controlnet
406
Node 3 Discrete I/O
Allen Bradley Rack
402
404B
Node 4 Discrete & Analog I/O
PLC5/L40C
Ethernet Card
100.10.10.1
IP ADDRESS
DC Power Supply
403
Node 5 Discrete I/O
404C
404D
Node 6 Discrete & Analog I/O
404E
Node 7 Discrete & Analog I/O
Node 11 VFD
404F
Node 12 VFD
404G
408
410
412
414
Data Highway Plus
426
Other Devices
424
418
428
430
416
Local Programming Computer with AB Software
World Wide Web
USER 1 PC
USER 1 PALM PILOT
420
422
442
Ethernet X-Over Cable or Hub
444
DSL Modem
Phone Line
440
PC Ethernet Card
IP Address 100.10.10.1
446
Local On-Site Computer
PC Modem Card
IP Address
450
Microsoft Windows 2000
Server Software
448
AB OPC SERVER
452
Microsoft IIS
468
Intellution OPC SERVER
454
Intellution Web Server
466
456
460
Intellution Process Database
ODBC
SQL/DAI/ADO
Historical Data
464
Intellution Work Space
462
Access / Oracle RDB
Excel or Crystal Reports
458

Figure 17

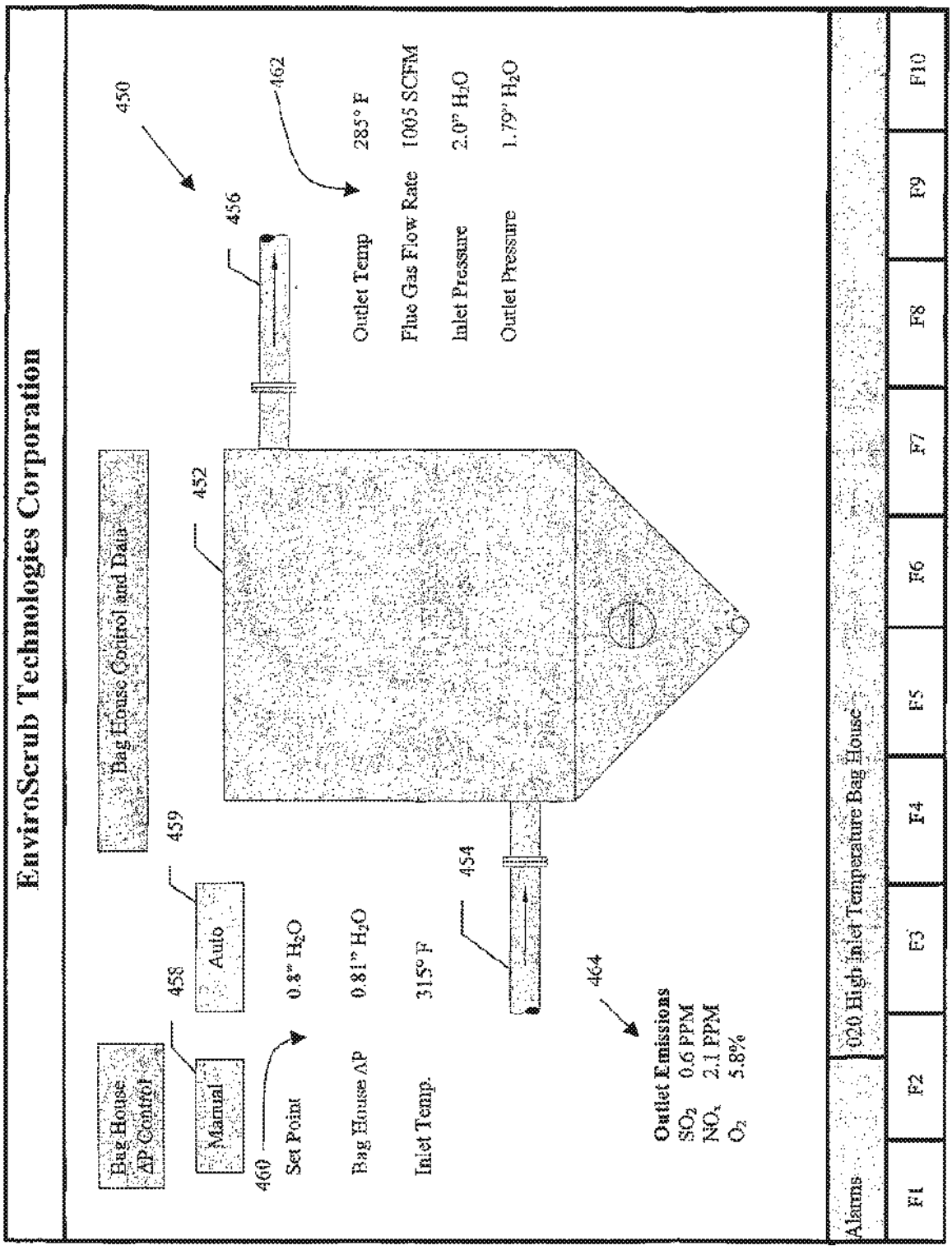

EnviroScrub Technologies Corporation
Bag House ΔP Control
Manual
Auto
458
459
460
Set Point
0.8" H2O
Bag House ΔP
0.81" H2O
Inlet Temp.
315° F
Bag House Control and Data
450
452
454
456
462
464
Outlet Temp
285° F
Flue Gas Flow Rate
1005 SCFM
Inlet Pressure
2.0" H2O
Outlet Pressure
1.79" H2O
Outlet Emissions
SO2 0.6 PPM
NOx 2.1 PPM
O2 5.8%
Alarms
020 High Inlet Temperature Bag House
F1
F2
F3
F4
F5
F6
F7
F8
F9
F10

Figure 18

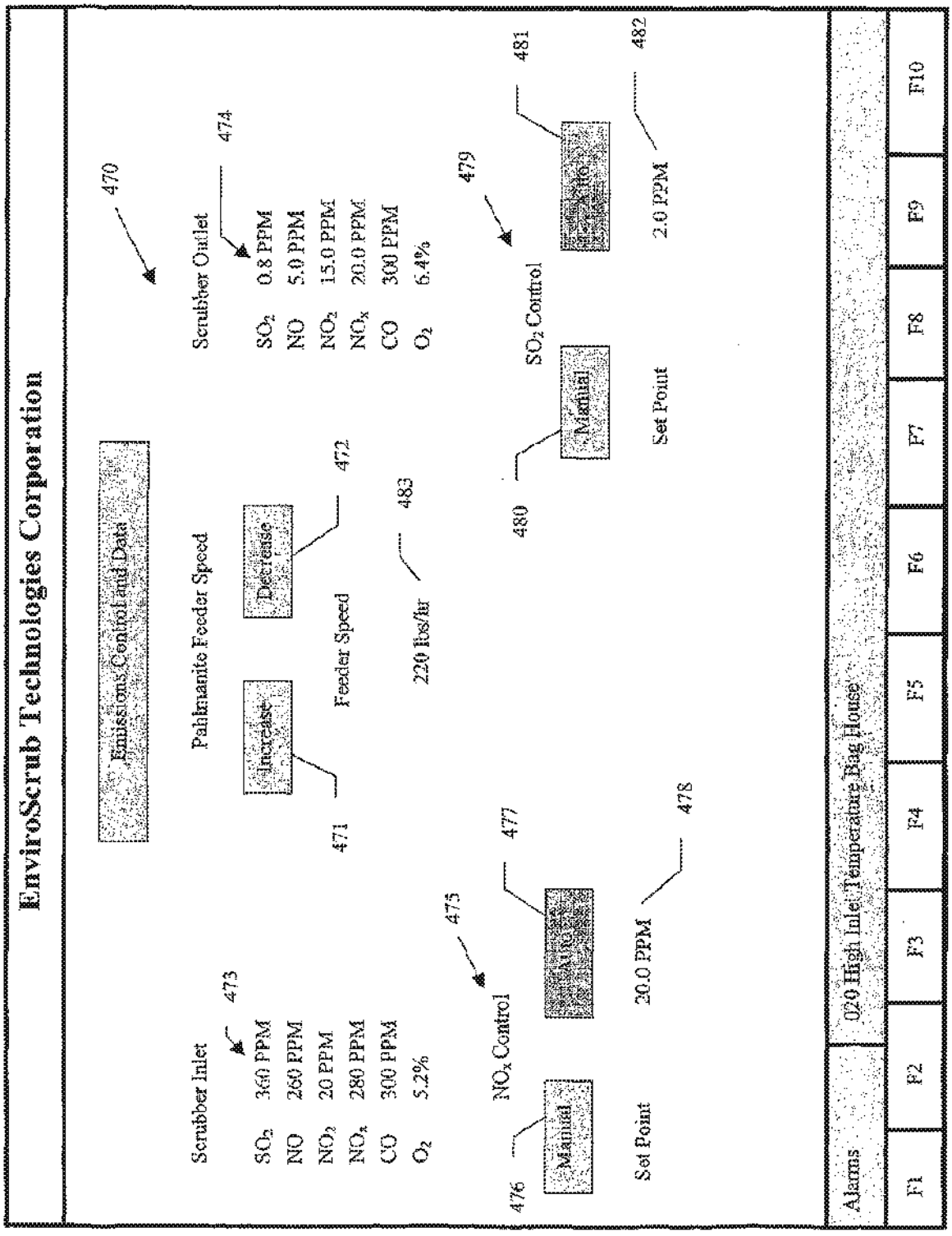

EnviroScrub Technologies Corporation
Emissions Control and Data
470
Scrubber Inlet
473
SO2 360 PPM
NO 260 PPM
NO2 20 PPM
NOx 280 PPM
CO 300 PPM
O2 5.2%
Pahlmanite Feeder Speed
Increase
Decrease
471
472
Feeder Speed
220 lbs/hr
483
Scrubber Outlet
474
SO2 0.8 PPM
NO 5.0 PPM
NO2 15.0 PPM
NOx 20.0 PPM
CO 300 PPM
O2 6.4%
479
480
SO2 Control
Manual
481
Set Point
2.0 PPM
482
475
NOx Control
476
Manual
477
Set Point
20.0 PPM
478
Alarms
026 High Inlet Temperature Bag House
F1
F2
F3
F4
F5
F6
F7
F8
F9
F10

Figure 19

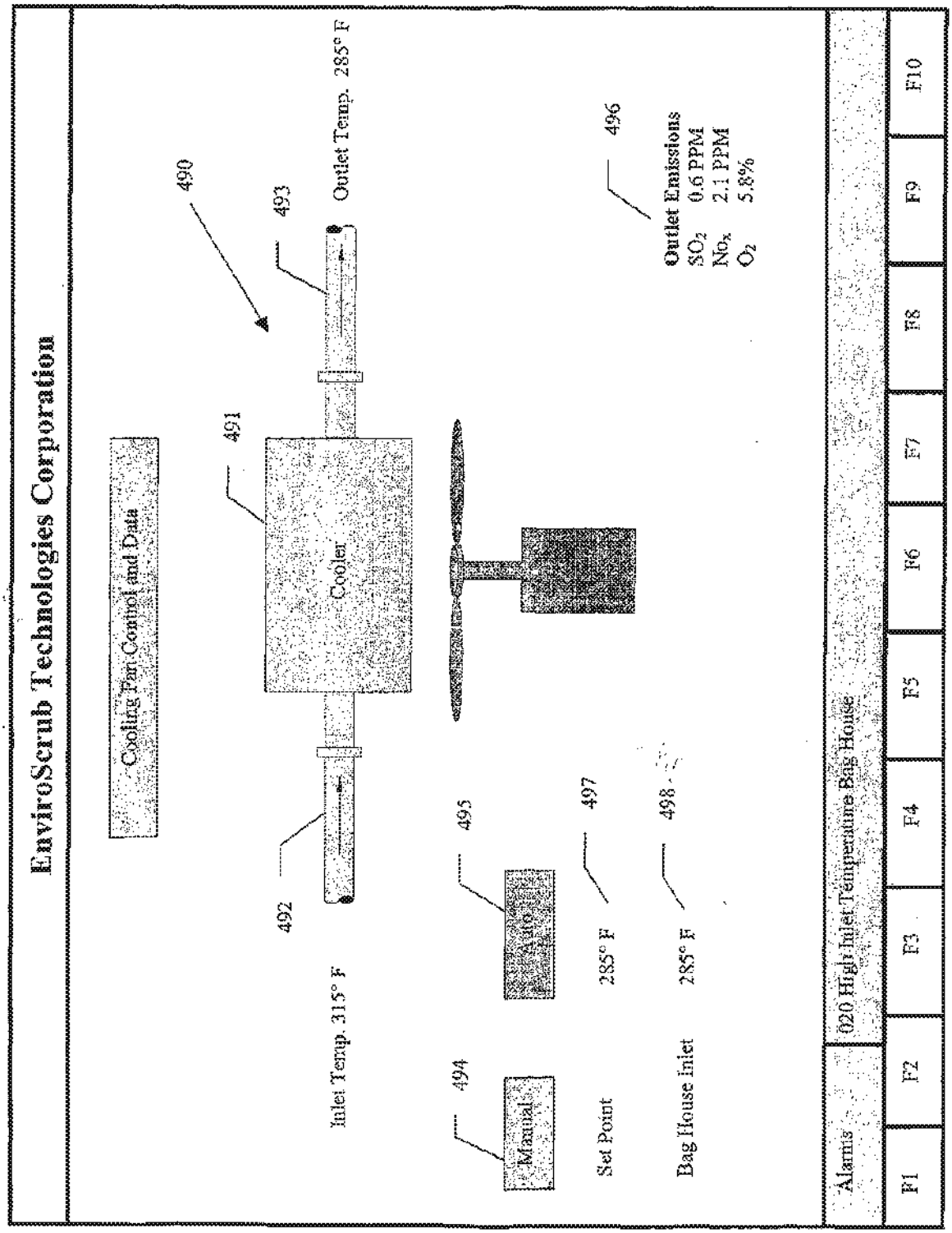

EnviroScrub Technologies Corporation
Cooling Fan Control and Data
490
491
Cooler
492
Inlet Temp. 315° F
493
Outlet Temp. 285° F
494
Manual
495
Auto
496
Outlet Emissions
SO2 0.6 PPM
Nox 2.1 PPM
O2 5.8%
497
Set Point
285° F
498
Bag House Inlet
285° F
Alarms
620 High Inlet Temperature Bag House
F1
F2
F3
F4
F5
F6
F7
F8
F9
F10

Figure 20

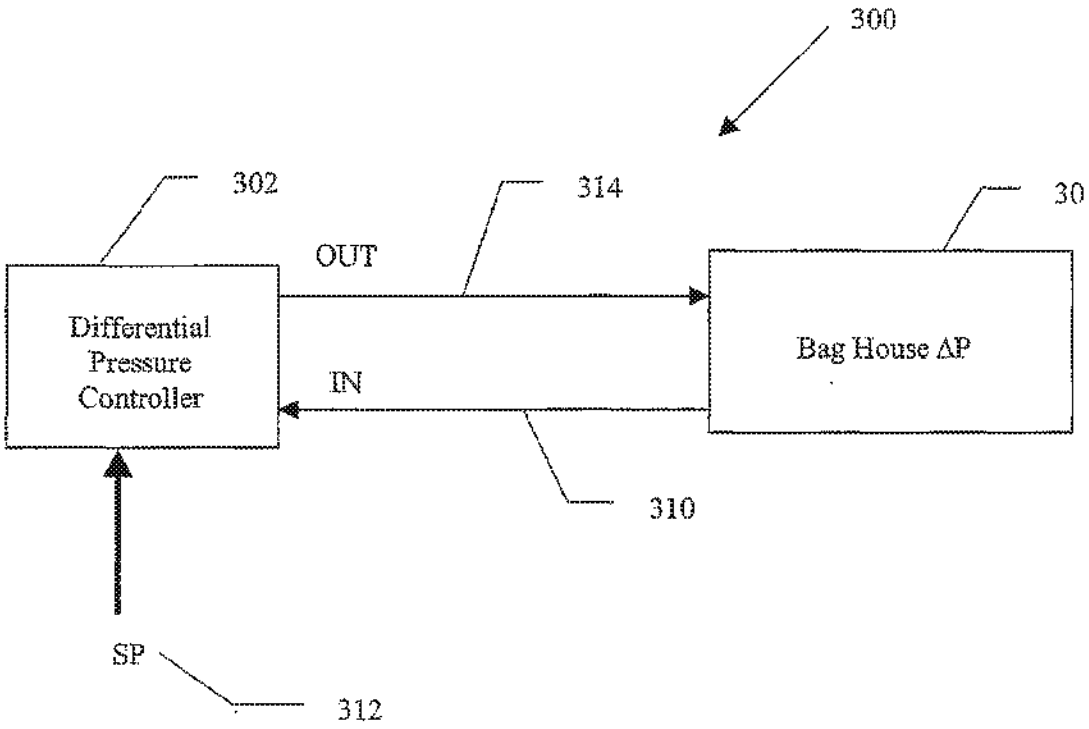

300
302
314
30
OUT
Differential Pressure Controller
Bag House ΔP
IN
310
SP
312

Figure 21

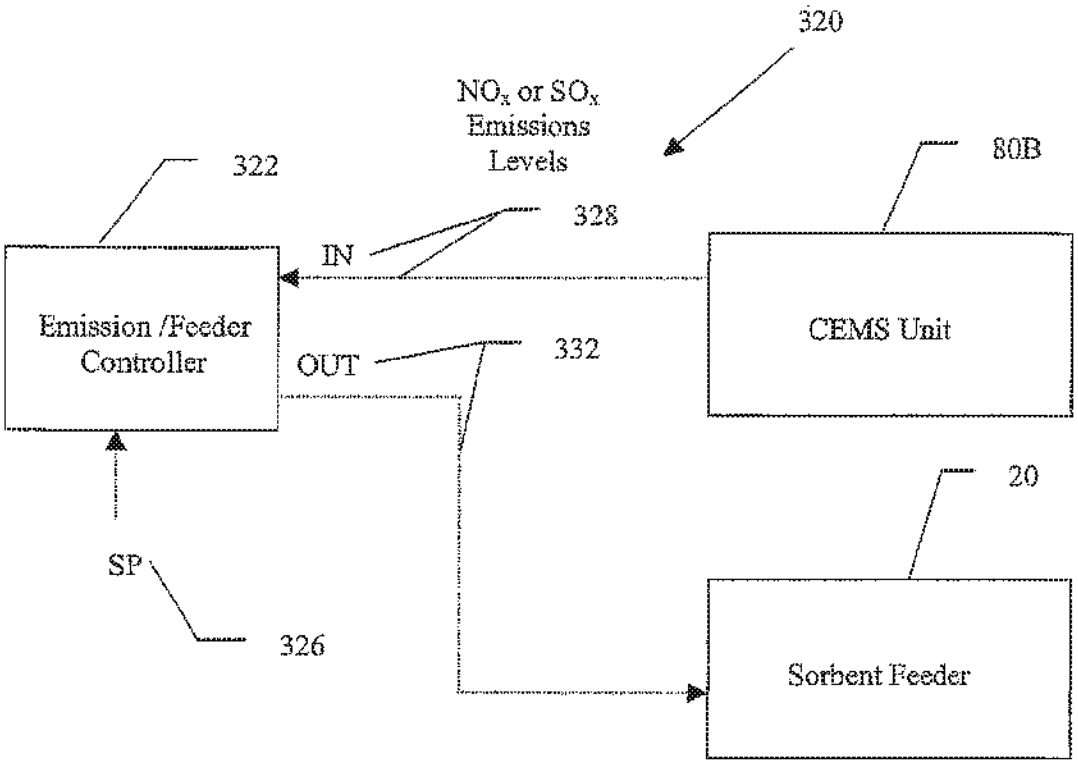

320
NO_x or SO_x Emissions Levels
322
328
80B
IN
Emission /Feeder Controller
CEMS Unit
OUT
332
20
SP
326
Sorbent Feeder

Figure 22

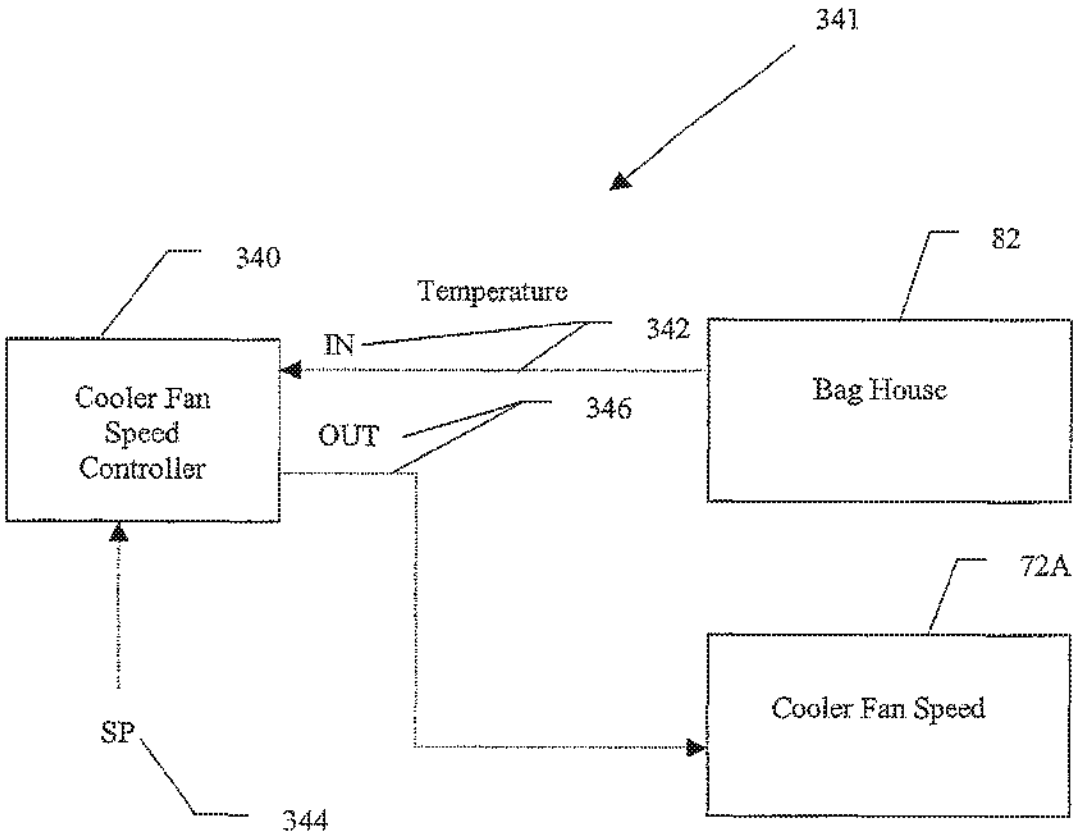

341
340
82
Temperature
342
IN
Cooler Fan Speed Controller
Bag House
346
OUT
72A
Cooler Fan Speed
SP
344

Figure 23

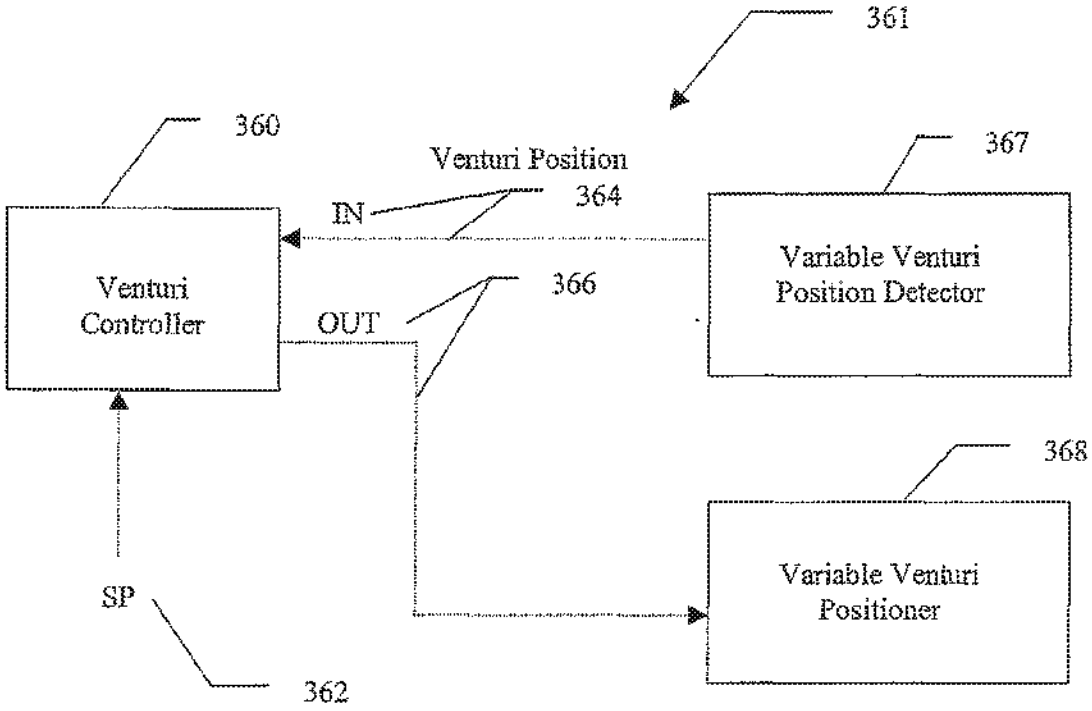

361
360
Venturi Position
367
IN
364
Venturi
Controller
Variable Venturi
Position Detector
366
OUT
368
Variable Venturi
Positioner
SP
362

Figure 24
Software Control Loop for Simultaneous NO_x, SO_x, and ΔP Control
Diagram 3

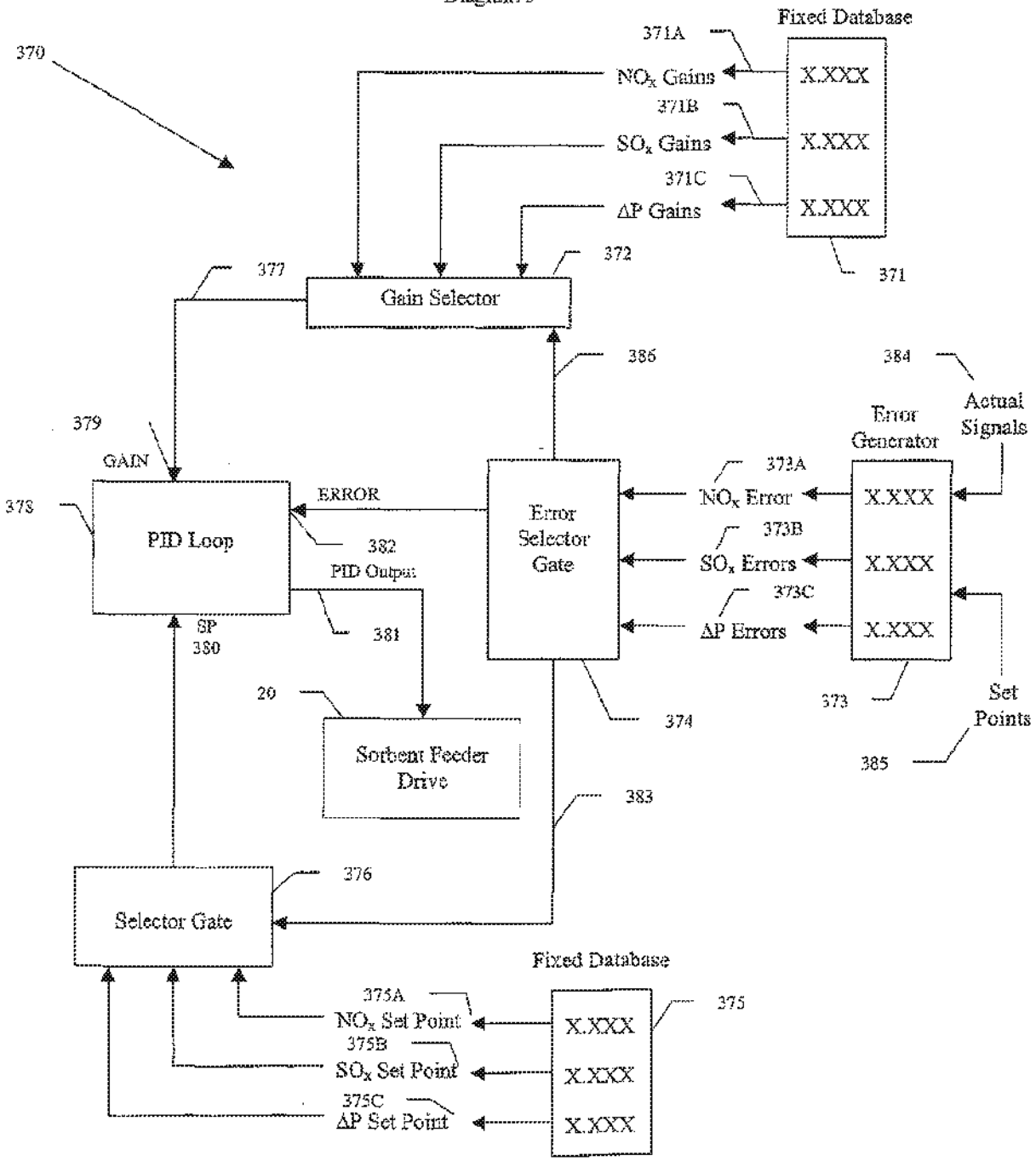
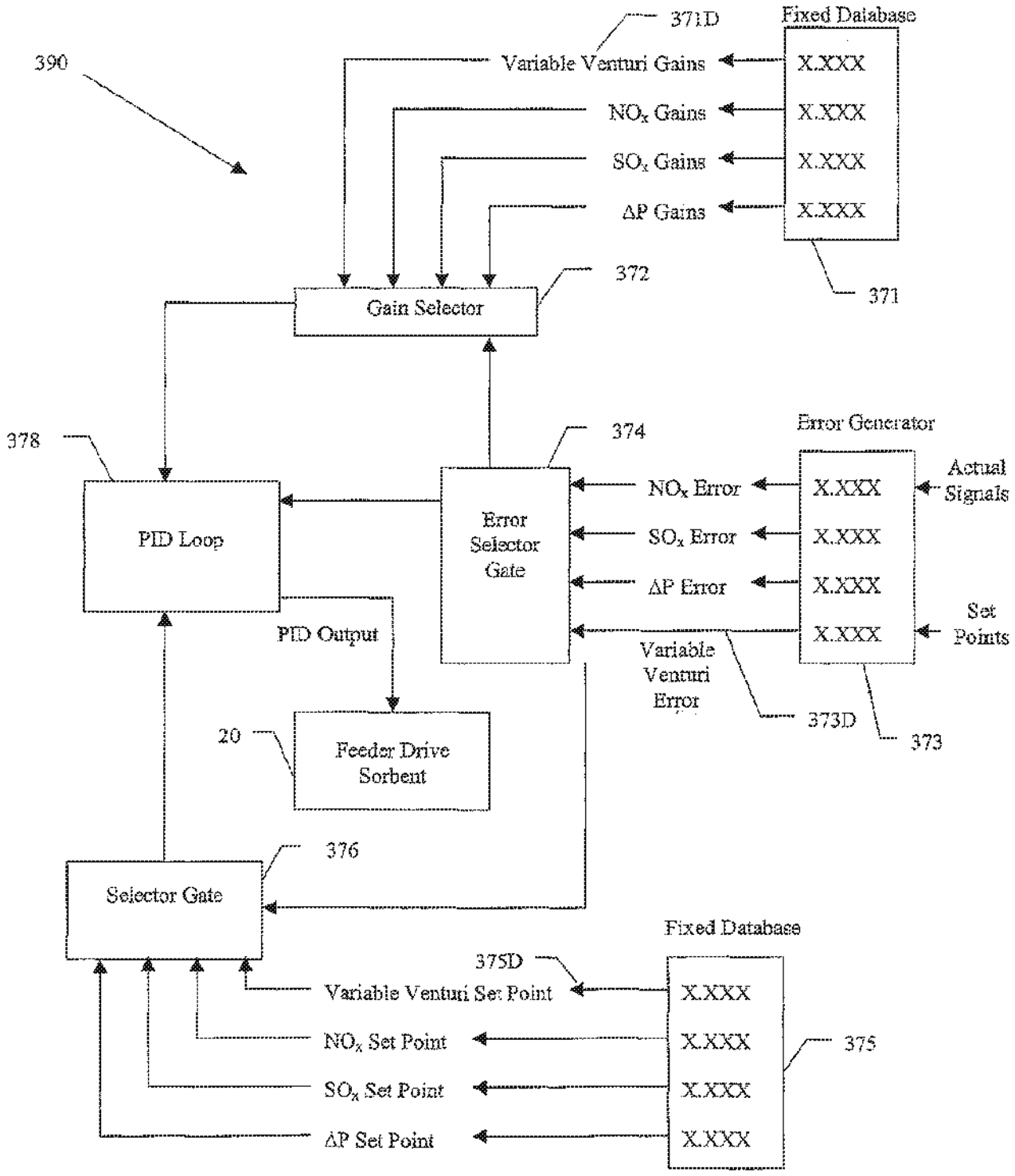


Figure 25
Software Control Loop for Simultaneous NO_x, SO_x, ΔP, and Variable Venturi Position Control
Diagram 6



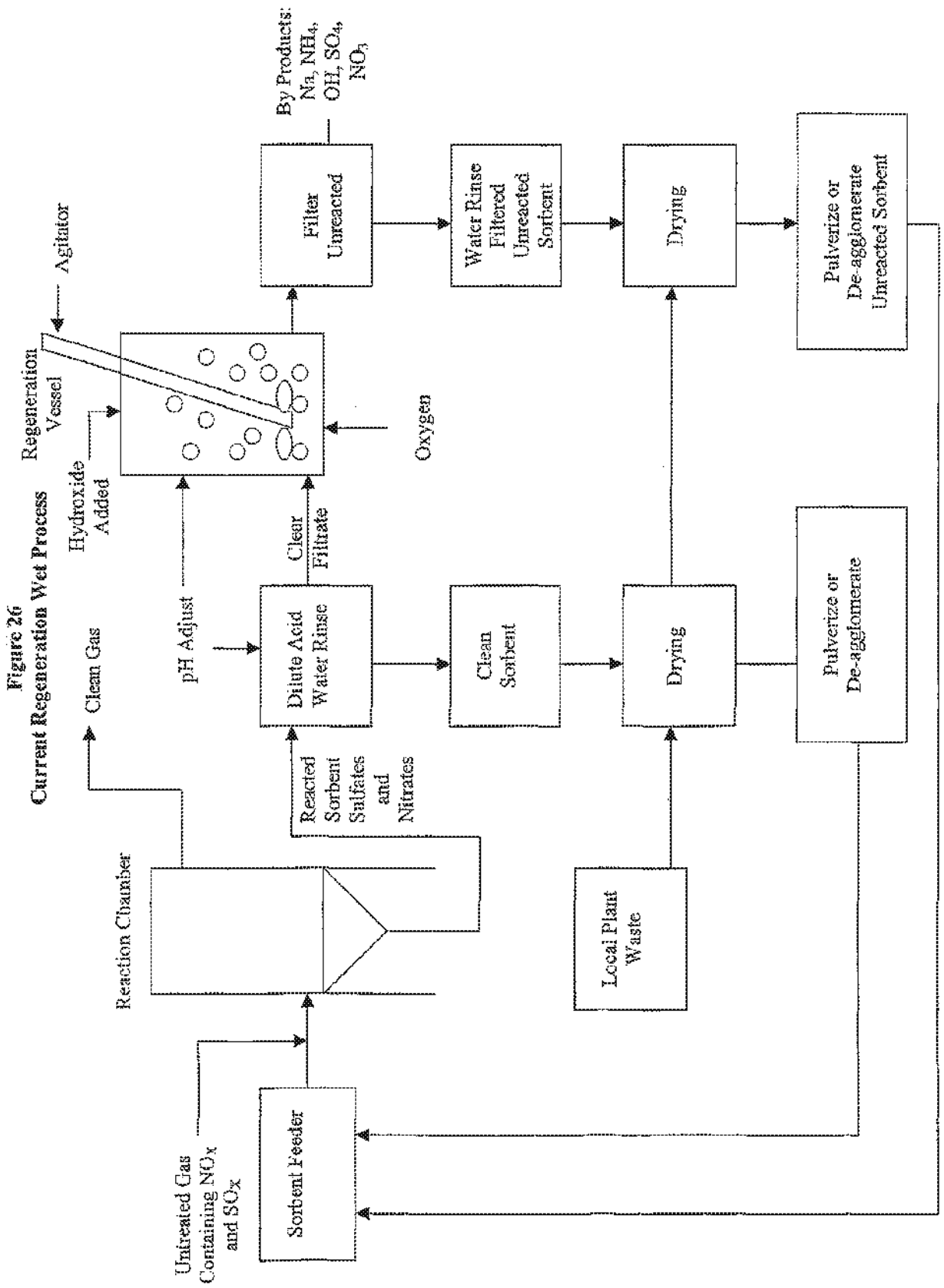


Figure 26
Current Regeneration Wet Process

Figure 27
Current Regeneration Dry Process

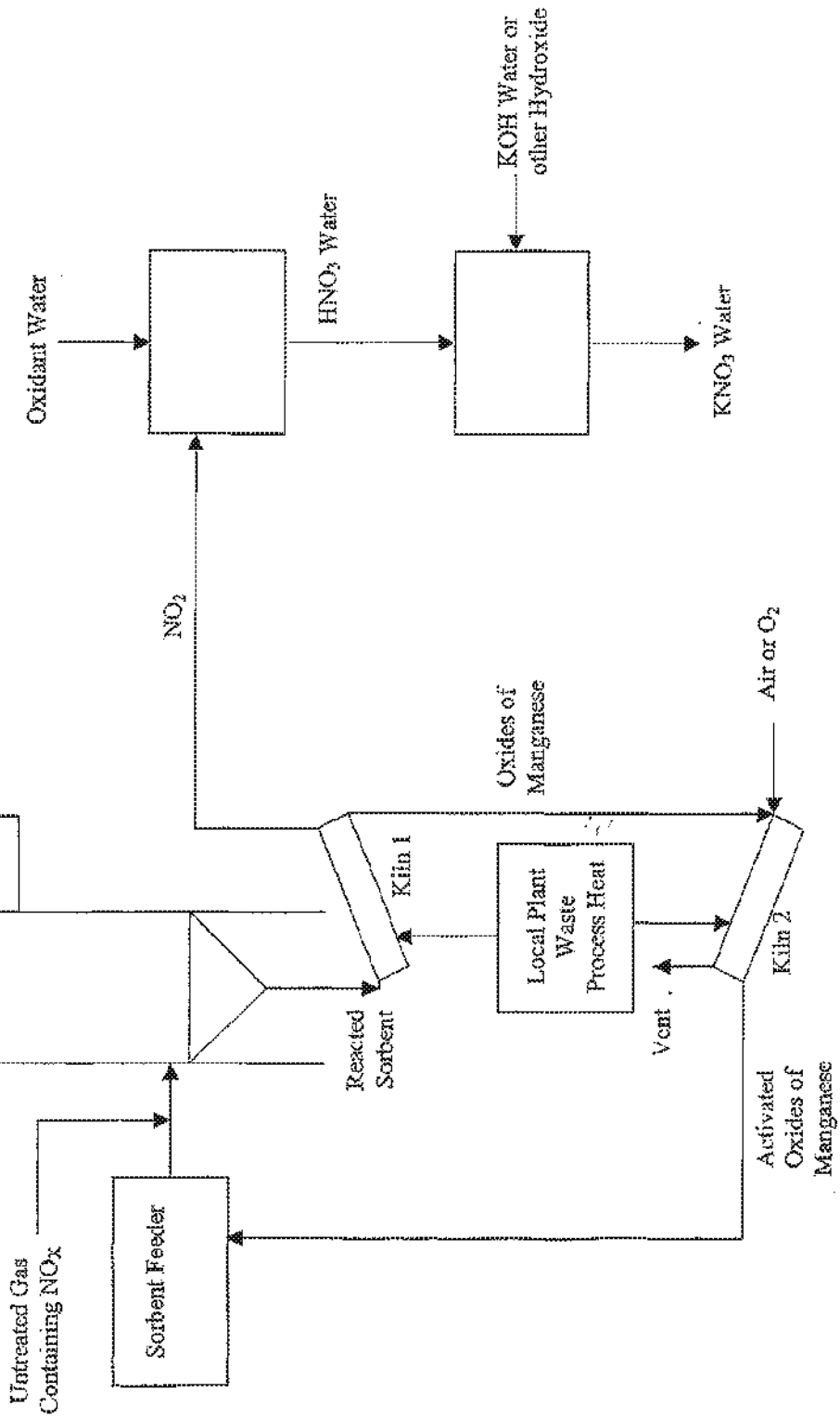
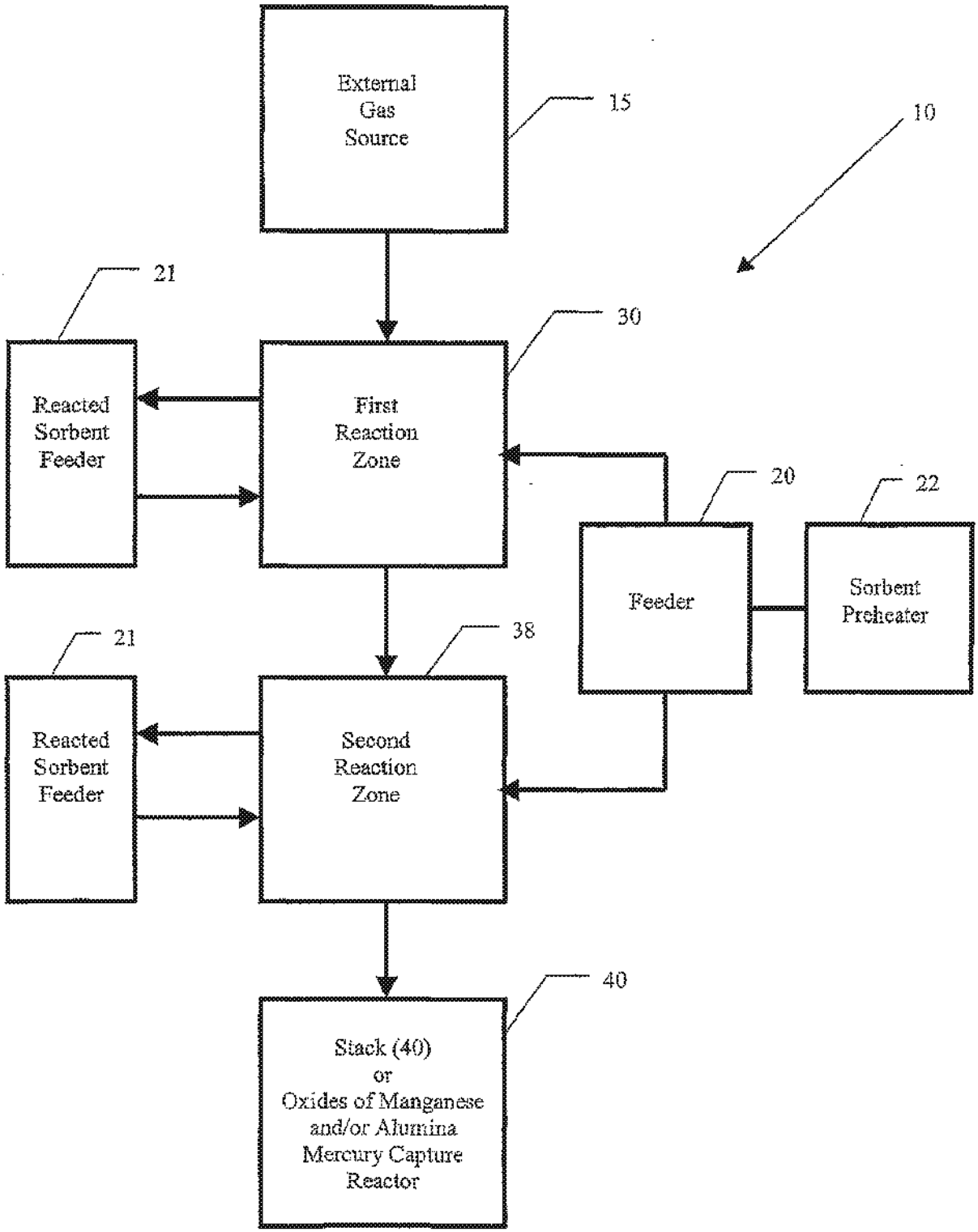
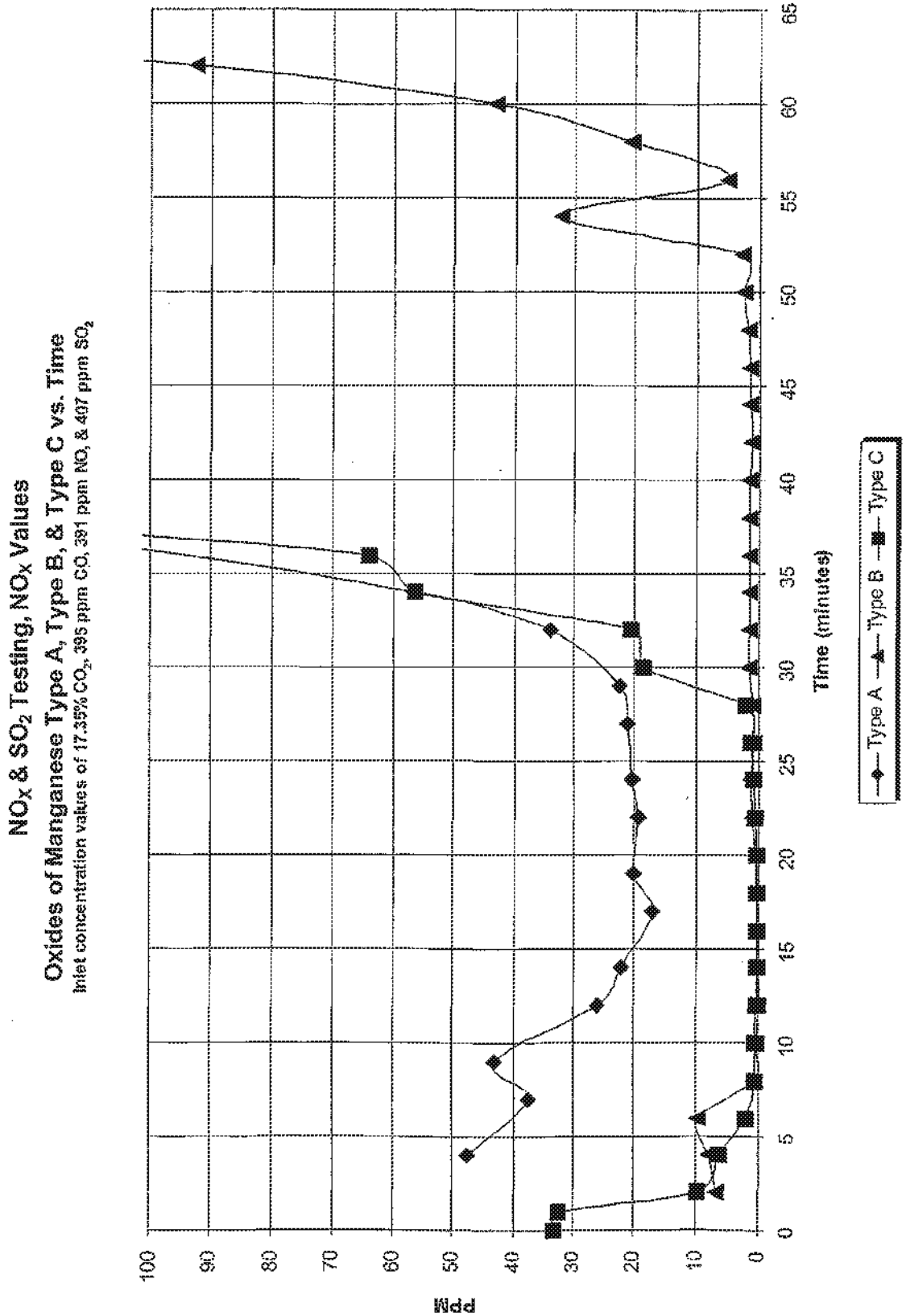
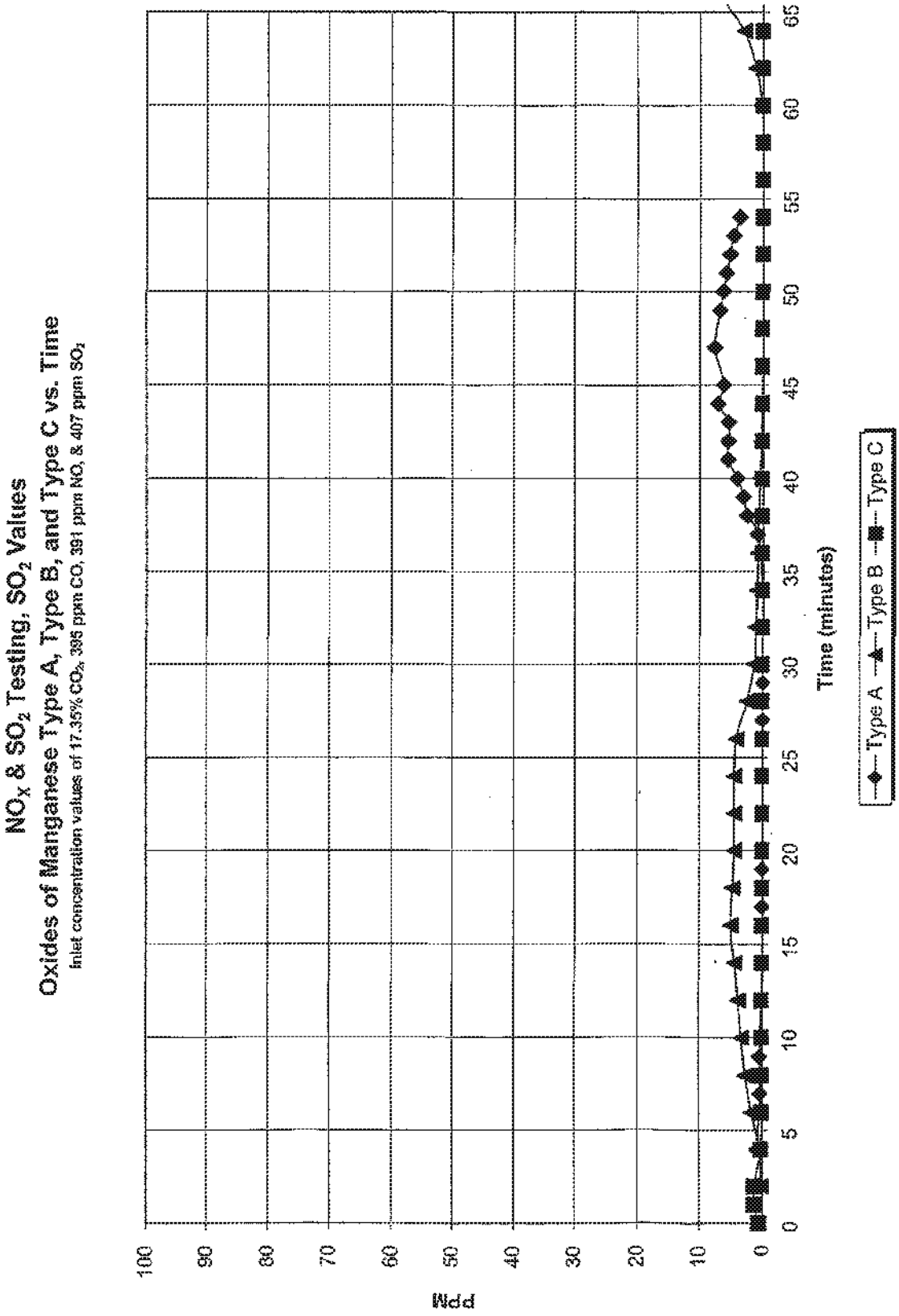


Figure 28


External Gas Source
15
10
21
30
Reacted Sorbent Feeder
First Reaction Zone
20
22
Feeder
Sorbent Preheater
21
38
Reacted Sorbent Feeder
Second Reaction Zone
40
Stack (40) or Oxides of Manganese and/or Alumina Mercury Capture Reactor


NOx & SO2 Testing, NOx Values
Oxides of Manganese Type A, Type B, & Type C vs. Time
Inlet concentration values of 17.35% CO2, 395 ppm CO, 391 ppm NO, & 407 ppm SO2
ppm
Time (minutes)
Type A
Type B
Type C


NO_x & SO_2 Testing, SO_2 Values
Oxides of Manganese Type A, Type B, and Type C vs. Time
Inlet concentration values of 17.35% CO_2, 398 ppm CO, 391 ppm NO, & 407 ppm SO_2
ppm
Time (minutes)
Type A
Type B
Type C

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(72) Inventors; and

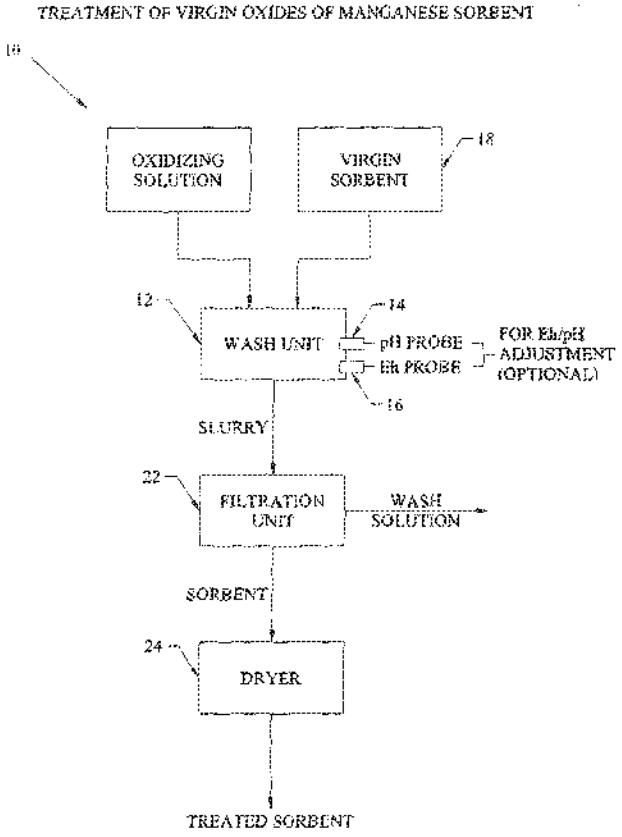
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[Continued on next page]

(54) Title: PRETREATMENT AND REGENERATION OF OXIDES OF MANGANESE



(57) Abstract: The invention relates to systems and processes for treating particulate oxides of manganese useful as oxidizing sorbents for capturing or removing target pollutants from industrial gas streams, including, but not limited to, nitrogen oxides (NO_x), sulfur oxide (SO_x), mercury (Hg), hydrogen sulfide (H_2S), other totally reduced sulfides (TRS), and oxides of carbon (CO and CO_2) gases. Oxides of manganese are washed an aqueous oxidizing solution, which may be adjusted as necessary to maintain the oxidizing solution a desired range of pH (acidity) and Eh (oxidizing potential) within the MnO_2 stability area for aqueous solutions. The resulting treated oxides of manganese are equally or more efficient as sorbents as the oxides of manganese processed with the invention.

WO 03/072241 A1

(84) **Designated States** *(regional)*: ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:

- *with international search report*
- *before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments*

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

PRETREATMENT AND REGENERATION OF OXIDES OF MANGANESE

Related Applications

This application claims priority to US Provisional Application No. 60/342,587 filed December 21, 2001, which is incorporated herein by reference.

Field of the Invention

The invention relates to systems and processes for treating oxides of manganese that may be utilized as a sorbent for capture and removal of target pollutants from industrial and other gas streams. Further, the invention relates to pollution removal systems incorporating such methods and apparatus and to oxides of manganese, oxides of manganese sorbent particles, and oxides of manganese filter cake formed by the processes of the invention.

Background of the Invention

The element manganese (Mn) may exist in six different valence (oxidation) states. Of particular interest and usefulness in the Pahlman™ systems and processes are those oxides of manganese having valence states of +2, +3, and +4, which correspond to the oxides MnO, Mn_2O_3, and MnO_2. The oxide MnO_4 is probably a solid-solution of both the +2 and +3 states.

A characteristic of most oxides of manganese species is non-stoichiometry; that is, most oxides of manganese molecules or MnO_2 species typically contain on average less than the theoretical number of 2 oxygen atoms, with numbers more typically ranging between 1.5 to 2.0. The non-stoichiometry characteristic is thought to result from solid-solution mixtures of two or more oxide species, and exists in all but the beta (β), or pyrolusite, form of manganese dioxide. Oxides of manganese having the formula MnOX where X is about 1.5 to about 2.0 are particularly suitable for dry removal of target pollutants from gas streams. However, the most active types of oxides of manganese for use as a sorbent for target pollutant removal usually have the formula $MnO_{1.7 \text{ to } 1.95}$, which translates into manganese valence states of +3.4 to +3.9, as opposed to the theoretical +4.0 state. It is unusual for average valence states above about 3.9 to exist in most forms of oxides of manganese. The

formula MnO_2, as used herein, symbolically represents all varieties of manganese dioxide including those with valence states ranging from +3 to +4, or $MnO_{1.5-2.0}$.

Oxides of manganese are known to exhibit several identifiable crystal structures, which result from different assembly combinations of their basic molecular structural units. These basic structural "building block" units are MnO_6 octahedra, which consist of one manganese atom at the geometric center, and one oxygen atom at each of the six apex positions of an octahedral geometrical shape. The octahedra may be joined together along their edges and/or corners, to form "chain" patterns, with void spaces ("tunnels"). Regular (and sometimes irregular) three-dimensional patterns consist of layers of such "chains" and "tunnels" of joined octahedra. These crystalline geometries are identified by characteristic x-ray diffraction (XRD) patterns. Most oxides of manganese are classifiable into one or more of the six fundamental crystal structures, which are called alpha (α), beta (β), gamma (γ), delta (δ), epsilon (ε), and ramsdellite. Certain older literature also included rho (ρ) and lambda (λ) structures, which are now thought obsolete, due partly to improvements in XRD technique. Some (amorphous) forms of MnO_2 exhibit no crystalline structure.

Certain characteristics of oxides of manganese probably arise from the size and shape of voids within these crystalline patterns and from certain elements, and compounds, which may occupy the voids and appear to help prevent collapse of certain structures. Applicants believe that these characteristics in addition to the oxidation state may have and affect upon the loading capacity of oxides of manganese sorbent. Further, many oxides of manganese are hydrous, and include structurally bound water. This bound water may also contribute to chemical reactivity and possibly catalytic behavior of the species.

Some oxides of manganese have the ability to absorb oxygen from gas. Manganous oxide (MnO) will oxidize to MnO_2 in the presence of air, for example. Additionally, the dioxides are themselves oxidizers, they readily exchange oxygen in chemical reactions, and they are known to have catalytic properties. This oxygen exchange ability may be related to proton mobility and lattice defects common within most MnO_2 crystal structures. The oxidizing potential of MnO_2 is advantageously utilized in target pollutant removal in the Pahlman™ and other systems and processes. Target pollutants, such as NO_X and SO_2 gases, mercury (Hg) and other pollutants, require oxidation of the species prior to reaction with MnO_2 sorbent to form reaction products, such as manganese sulfates and nitrates, mercury compounds, and other corresponding reaction products, in order for them to be captured and removed from gas stream.

Manganese compounds are soluble in water in the +2 valence state, but not in the +4 state. Therefore Mn^{+2} compounds, including MnO are readily soluble in aqueous solutions, as opposed to MnO_2. During the formation of reaction products such as manganese nitrates and sulfates, the manganese is reduced from about the +4 state to the +2 state. This property allows the reaction products formed on the surface of oxides of manganese sorbent particles to be readily dissolved and removed from the sorbent particles in aqueous solutions by disassociation into sulfate, nitrate, and Mn^{+2} ions.

Manganese dioxides are divided into three origin-based categories, which are: 1) natural (mineral) manganese dioxide (NMD), 2) chemical manganese dioxide (CMD), and 3) electrolytic manganese dioxide (EMD). As implied, NMD occurs naturally as various minerals, which may be purified by mechanical or chemical means. The most common form of NMD is pyrolusite (β-MnO_2), which is inexpensive, but has rather low chemical activity and therefore low pollutant loading capacity. CMD and EMD varieties are synthetic. EMD is produced primarily for the battery industry, which requires relatively high bulk density (which often results from relatively large, compact particles), relatively high purity, and good electrochemical activity. Though useful as a sorbent, characteristics such as low surface area and large compact particle size make EMD somewhat inferior to CMD for gas removal applications, despite its good electrochemical activity. Chemically synthesized oxides of manganese of all kinds falls into the CMD category and includes chemically treated oxides of manganese. In chemical synthesis, a great deal of control is possible over characteristics such as particle size and shape, porosity, composition, surface area, and bulk density in addition to electrochemical or oxidation potential. It is believed that these characteristic contribute the loading capacity of some oxides of manganese.

EnviroScrub Technologies Corporation has developed pollutant removal systems and processes utilizing dry and wet removal techniques and combinations thereof, incorporating the use of oxides of manganese as a sorbent. These systems and processes, commonly known as Pahlman™ systems and processes are the subject of co-pending U.S. Patent Application Numbers 09/919,600, 09/951,697, 10/044,089 and 10/025,270, the disclosures of which are incorporated herein. High target pollutant removal efficiencies have been obtained utilizing the Pahlman™ systems and processes with oxides of manganese as the sorbent. Due to pollutant loading during the removal process, the Pahlman™ system or some of its system components must be periodically taken off-line so that sorbent may be removed for regeneration and recovery of useful by-products. The frequency of such disruptions and

related downtime could be decreased if it were possible to increase the loading capacity of the sorbent. It would therefore be desirable to enhance the loading capacities of the oxides of manganese in order to extend the period of sorbent use and its loading capacity. Applicants have developed methods of treating virgin oxides of manganese and of recycle processing of loaded oxides of manganese with oxidants or oxidizers that result in treated oxides of manganese having enhanced or increased loading capacity.

Brief Description of the Drawings

Figure 1 is a block flow diagram depicting an embodiment of a treatment system and method of the invention.

Figure 2 is a block flow diagram depicting an embodiment of a treatment system and method of the invention.

Figure 3 is a sorbent loading graph.

Figure 4 is a sorbent loading graph.

Figure 5 is a sorbent loading graph.

Figure 6 is a sorbent loading graph.

Figure 7 is a block flow diagram of a treatment system and method of the invention incorporating a controller.

Figure 8 is a block flow diagram of a embodiment of a treatment system and method of the invention incorporating a controller.

Figure 9 is a Pourbaix diagram for an aqueous solution of 1 mole/liter manganese ion concentration.

Figure 10 is a Pourbaix diagram for an aqueous solution of 10^{-6} mole/liter manganese ion concentration.

Summary of the Invention

The invention relates to systems and processes for treatment of oxides of manganese that may be utilized as a sorbent for removal of target pollutants from a gas stream.

An embodiment of a process for pretreatment of virgin oxides of manganese for use as a sorbent to remove target pollutants from a gas stream comprises the steps of washing virgin oxides of manganese in wash unit with an aqueous oxidizing solution, the solution having Eh and pH values within the MnO_2 stability area; and filtering the washed oxides of manganese to form a filtrate and a pretreated oxides of manganese filter cake, the oxides of

manganese of the filter cake having a pollutant loading capacity greater than the pollutant loading capacity of the virgin oxides of manganese.

Another embodiment of a process for pretreatment of virgin oxides of manganese for use as a sorbent to remove target pollutants from a gas stream comprises the steps of washing virgin oxides of manganese in a wash unit with an aqueous oxidizing solution, the solution having Eh and pH values within the MnO_2 stability area; monitoring and adjusting the pH and/or the Eh values of the solution so as to maintain the Eh and pH values of the solution within the MnO_2 stability area; and filtering the washed oxides of manganese to form a filtrate and a pretreated oxides of manganese filter cake, the oxides of manganese of the filter cake having a pollutant loading capacity greater than the pollutant loading capacity of the virgin oxides of manganese. The monitoring and adjusting step may be carried out by a controller capable of individually or simultaneously monitoring and adjusting system operational parameters, the controller providing integrated control of within the wash unit of the temperature, Eh values, and pH values in order to maintain conditions of the aqueous oxidizing solution within the MnO_2 stability area.

An embodiment of a process for pretreatment of virgin oxides of manganese for use as a sorbent to remove target pollutants from a gas stream comprises the steps of washing virgin oxides of manganese in a rapid filtration wash unit with an aqueous oxidizing solution to form a filtrate and a pretreated oxides of manganese filter cake, the solution having Eh and pH values within the MnO_2 stability area; and removing the pretreated oxides of manganese filter cake from the rapid filtration unit, the oxides of manganese of the filter cake having a pollutant loading capacity greater that the pollutant loading capacity of the virgin oxides of manganese.

An embodiment of a process for regeneration of loaded oxides of manganese formed through reaction between unreacted oxides of a manganese and target pollutants in a gas stream comprises the steps of washing loaded oxides of manganese in a rapid filtration wash unit with an aqueous oxidizing solution having Eh and pH values within the MnO_2 stability area to form a filtrate and a regenerated oxides of manganese filter cake; and removing the regenerated oxides of manganese filter cake from the rapid filtration unit, the oxides of manganese of the filter cake having a pollutant loading capacity equal to or greater than the pollutant loading capacity of the unreacted oxides of manganese from which the loaded oxides of manganese are formed.

An embodiment of a process for regeneration of loaded oxides of manganese sorbent particles bearing a reaction product layer of the surface thereof formed during reaction between unreacted oxides of manganese sorbent particles and target pollutants in a gas stream in a pollution removal system comprises the steps of rapidly washing and filtering the loaded oxides of manganese sorbent particles in a rapid filtration wash unit with an aqueous oxidizing solution to dissolve the reaction product layer from the surface of the loaded oxides of manganese sorbent particles into the solution to form a filtrate and a regenerated oxides of manganese filter cake, the solution having Eh and pH values within the MnO_2 stability area; and removing the regenerated oxides of manganese filter cake from the rapid filtration unit, the oxides of manganese of the filter cake having a pollutant loading capacity equal to or greater than the pollutant loading capacity of the unreacted oxides of manganese sorbent particles from which the loaded oxides of manganese sorbent particles are formed.

The above processes may further comprise the steps of drying the filter cake; and comminuting and sizing the dried filter cake to provide oxides of manganese particles. The may alternative further comprise the steps of drying the filter cake; and comminuting and sizing the dried filter cake to provide oxides of manganese particles for introduction into a feeder or reaction chamber of a dry pollution removal system. In a further alternative, they processes may further comprise the steps of drying the filter cake; comminuting and sizing the dried filter cake to provide oxides of manganese particles for introduction into a feeder or reaction chamber of a dry pollution removal system; and introducing the comminuted and sized oxides of manganese particles into the feeder or reaction chamber. In yet a further alternative, the above described processes may further comprise the step of conveying the filter cake to a dryer configured to inject the a wet filter cake or a slurry of oxides of manganese sorbent into the flow of a gas stream prior to introduction into a reaction chamber of a pollutant removal system. In yet another further alternative, the above-described processes may further comprise the steps of conveying the filter cake to a dryer configured to inject the a wet filter cake or a slurry of oxides of manganese sorbent into the flow of a gas stream prior to introduction into a reaction chamber of a pollutant removal system; and injecting the wet filter cake or the slurry into the reaction chamber.

In the above-described processes the aqueous oxidizing solution contains an oxidizer. The oxidizer may be selected from the group consisting of hypochlorites, perchlolates, permanganates, oxygen (O_2), air, ozone, peroxides, persulfates, and combinations thereof.

13 An embodiment of a system for pretreatment of virgin oxides of manganese comprising a wash unit configured to hold and agitate a slurry of virgin oxides of manganese in an aqueous oxidizing solution, the solution have Eh and pH values within the MnO_2 stability area, the wash unit being optionally equipped with a pH probe, an Eh probe, and a temperature probe, the wash unit being further configured to receive the slurry of virgin oxides of manganese or to individually receive virgin oxides of manganese and the aqueous oxidizing solution; a filtration unit configured to receive the slurry from the wash unit and to filter the slurry to form a filtrate and a pretreated oxides of manganese filter cake; an aqueous oxidizing solution vessel containing a supply of aqueous oxidizing solution, the solution having Eh and pH values within the MnO_2 stability area, the vessel having a feeder configured to feed aqueous oxidizing solution to the wash unit and being optionally equipped with an Eh probe; an oxides of manganese vessel containing a supply of virgin oxides of manganese, the vessel having a feeder configured to feed oxides of manganese directly to the wash unit or, in conjunction with the aqueous oxidizing solution feeder to feed a slurry of virgin oxides of manganese to the wash unit; and a pH adjust vessel having a supply of acid or base or separate supplies of acid and base, the pH adjust vessel having a feeder configured to feed acid and/or base to the wash unit. The system of this embodiment may further comprise a controller capable of individually or simultaneously monitoring and adjusting system operational parameters, the controller providing integrated control within the wash unit of the temperature, Eh values, and pH values in order to maintain conditions of the aqueous oxidizing solution within the MnO_2 stability area.

This embodiment of a system for treatment of virgin or loaded oxides of manganese may further comprise a dryer for drying the filter cake; a device for comminuting and sizing the dried filter cake to form oxides of manganese particles; and a reaction chamber of a pollutant removal system into which the oxides of manganese particles are introduced to capture target pollutants from a gas, the reaction chamber being equipped with at least one target pollutant concentration reader for measuring target pollutant concentration of the gas as it exits the reaction chamber, the reader being in electronic communication with the controller for checking the loading performance of the oxides of manganese particles and signaling need for adjustment of operational parameters within the wash unit.

14 Another embodiment of a system for treatment of virgin or loaded oxides of manganese comprising a rapid filtration wash unit, the wash unit being configured to individually receive oxides of manganese and an aqueous oxidizing solution having a Eh and

pH values within the MnO_2 stability area, the wash unit being further configured to rapidly remove the aqueous oxidizing solution from contact with the oxides of manganese forming a filtrate and a treated oxides of manganese filter cake; an aqueous oxidizing solution vessel containing a supply of aqueous oxidizing solution, the solution having Eh and pH values within the MnO_2 stability area, the vessel having a feeder configured to feed aqueous oxidizing solution to the wash unit and being optionally equipped with a probe selected from the group consisting of an Eh probe, a pH probe, a temperature probe, or any combination thereof; an oxides of manganese vessel containing a supply of virgin of loaded oxides of manganese, the vessel having a feeder configured to feed oxides of manganese to the wash unit; optionally, a oxidizer vessel containing a supply of oxidizer, the oxidizer vessel having feeder configured to feed oxidizer to the aqueous oxidizing solution vessel; and optionally, a pH adjust vessel having a supply of acid or base or separate supplies of acid and base, the pH adjust vessel having a feeder configured to feed acid and/or base to the aqueous oxidizing solution vessel. The system of this embodiment may further comprise a controller capable of individually or simultaneously monitoring and adjusting system operational parameters, the controller providing integrated control of the temperature, Eh values, and pH values within the aqueous oxidizing solution vessel in order to maintain conditions of the aqueous oxidizing solution within the MnO_2 stability area.

This embodiment of a system for treatment of virgin or loaded oxides of manganese may further comprise a dryer for drying the filter cake; a device for comminuting and sizing the dried filter cake to form oxides of manganese particles; and a reaction chamber of a pollutant removal system into which the oxides of manganese particles are introduced to capture target pollutants from a gas, the reaction chamber being equipped with at least one target pollutant concentration reader for measuring target pollutant concentration of the gas as it exits the reaction chamber, the reader being in electronic communication with the controller for checking the loading performance of the oxides of manganese particles and signaling need for adjustment of operational parameters within the aqueous oxidizing solution vessel.

Another embodiment of a system for pretreatment of virgin oxides of manganese comprises a wash unit configured to hold and agitate a slurry of virgin oxides of manganese in an aqueous oxidizing solution, the solution have Eh and pH values within the MnO_2 stability area, the wash unit being equipped with a pH probe, an Eh probe, and a temperature probe, the wash unit being further configured to receive the slurry of virgin oxides of manganese or to individually receive virgin oxides of manganese and the aqueous oxidizing

solution; a filtration unit configured to receive the slurry from the wash unit and to filter the slurry to form a filtrate and a pretreated oxides of manganese filter cake; an aqueous oxidizing solution vessel containing a supply of aqueous oxidizing solution, the solution having Eh and pH values within the MnO_2 stability area, the vessel having a feeder configured to feed aqueous oxidizing solution to the wash unit and being equipped with an Eh probe; an oxides of manganese vessel containing a supply of virgin oxides of manganese, the vessel having a feeder configured to feed oxides of manganese directly to the wash unit or, in conjunction with the aqueous oxidizing solution feeder to feed a slurry of virgin oxides of manganese to the wash unit; a pH adjust vessel having a supply of acid or base or separate supplies of acid and base, the pH adjust vessel having a feeder configured to feed acid and/or base to the wash unit; and a controller capable of individually or simultaneously monitoring and adjusting system operational parameters, the controller providing integrated control within the wash unit of the temperature, Eh values, and pH values in order to maintain conditions of the aqueous oxidizing solution within the MnO_2 stability area, the controller being electronic communication with the probes and feeders of the system.

This embodiment of a for pretreatment of virgin oxides of manganese may further comprise a dryer for drying the filter cake; a device for comminuting and sizing the dried filter cake to form oxides of manganese particles; and a reaction chamber of a pollutant removal system into which the oxides of manganese particles are introduced to capture target pollutants from a gas, the reaction chamber being equipped with at least one target pollutant concentration reader for measuring target pollutant concentration of the gas as it exits the reaction chamber, the reader being in electronic communication with the controller for checking the loading performance of the oxides of manganese particles and signaling need for adjustment of operational parameters within the wash unit.

18 Another embodiment of a system for treatment of virgin or loaded oxides of manganese comprises a rapid filtration wash unit, the wash unit being configured to individually receive oxides of manganese and an aqueous oxidizing solution having a Eh and pH values within the MnO_2 stability area, the wash unit being further configured to rapidly remove the aqueous oxidizing solution from contact with the oxides of manganese forming a filtrate and a treated oxides of manganese filter cake; an aqueous oxidizing solution vessel containing a supply of aqueous oxidizing solution, the solution having Eh and pH values within the MnO_2 stability area, the vessel having a feeder configured to feed aqueous oxidizing solution to the wash unit and being equipped with a probe selected from the group

consisting of an Eh probe, a pH probe, a temperature probe, or any combination thereof; an oxides of manganese vessel containing a supply of virgin of loaded oxides of manganese, the vessel having a feeder configured to feed oxides of manganese to the wash unit; a oxidizer vessel containing a supply of oxidizer, the oxidizer vessel having feeder configured to feed oxidizer to the aqueous oxidizing solution vessel; a pH adjust vessel having a supply of acid or base or separate supplies of acid and base, the pH adjust vessel having a feeder configured to feed acid and/or base to the aqueous oxidizing solution vessel; and a controller capable of individually or simultaneously monitoring and adjusting system operational parameters, the controller providing integrated control of the temperature, Eh values, and pH values within the aqueous oxidizing solution vessel in order to maintain conditions of the aqueous oxidizing solution within the MnO_2 stability area, the controller being in electronic communication with the probes and feeders of the system.

This embodiment of a system for treatment of virgin or loaded oxides of manganese may further comprise a dryer for drying the filter cake; a device for comminuting and sizing the dried filter cake to form oxides of manganese particles; and a reaction chamber of a pollutant removal system into which the oxides of manganese particles are introduced to capture target pollutants from a gas, the reaction chamber being equipped with at least one target pollutant concentration reader for measuring target pollutant concentration of the gas as it exits the reaction chamber, the reader being in electronic communication with the controller for checking the loading performance of the oxides of manganese particles and signaling need for adjustment of operational parameters within the aqueous oxidizing solution vessel.

In the above-described systems the aqueous oxidizing solution contains an oxidizer. The oxidizer may be selected from the group consisting of hypochlorites, perchlolates, permanganates, oxygen (O_2), air, ozone, peroxides, persulfates, and combinations thereof.

The invention also relates to oxides of manganese, oxides of manganese particles, and oxides of manganese filter cakes formed by the above-described processes.

Oxides of manganese particles may be formed by a process for pretreatment of virgin oxides of manganese for use as a sorbent to remove target pollutants from a gas stream comprising the steps of washing virgin oxides of manganese in wash unit with an aqueous oxidizing solution, the solution having Eh and pH values within the MnO_2 stability area; filtering the washed oxides of manganese to form a filtrate and a pretreated oxides of manganese filter cake, the oxides of manganese of the filter cake having a pollutant loading capacity greater than the pollutant loading capacity of the virgin oxides of manganese; drying

the filter cake; and comminuting and sizing the dried filter cake to form oxides of manganese particles.

Oxides of manganese particles may be formed by a process for pretreatment of virgin oxides of manganese for use as a sorbent to remove target pollutants from a gas stream, comprising the steps of washing virgin oxides of manganese in a wash unit with an aqueous oxidizing solution, the solution having Eh and pH values within the MnO_2 stability area; monitoring and adjusting the pH and/or the Eh values of the solution so as to maintain the Eh and pH values of the solution within the MnO_2 stability area; filtering the washed oxides of manganese to form a filtrate and a pretreated oxides of manganese filter cake, the oxides of manganese of the filter cake having a pollutant loading capacity greater than the pollutant loading capacity of the virgin oxides of manganese; drying the filter cake; and comminuting and sizing the dried filter cake to form oxides of manganese particles.

Oxides of manganese particles may be formed by a process for pretreatment of virgin oxides of manganese for use as a sorbent to remove target pollutants from a gas stream comprising the steps of washing virgin oxides of manganese in a rapid filtration wash unit with an aqueous oxidizing solution to form a filtrate and a pretreated oxides of manganese filter cake, the solution having Eh and pH values within the MnO_2 stability area; removing the pretreated oxides of manganese filter cake from the rapid filtration unit, the oxides of manganese of the filter cake having a pollutant loading capacity greater that the pollutant loading capacity of the virgin oxides of manganese; drying the filter cake; and comminuting and sizing the dried filter cake to form oxides of manganese particles.

Oxides of manganese particles may formed by a process for regeneration of loaded oxides of manganese formed through reaction between unreacted oxides of a manganese and target pollutants in a gas stream comprising the steps of washing loaded oxides of manganese in a rapid filtration wash unit with an aqueous oxidizing solution having Eh and pH values within the MnO_2 stability area to form a filtrate and a regenerated oxides of manganese filter cake; removing the regenerated oxides of manganese filter cake from the rapid filtration unit, the oxides of manganese of the filter cake having a pollutant loading capacity equal to or greater than the pollutant loading capacity of the unreacted oxides of manganese from which the loaded oxides of manganese are formed; drying the filter cake; and comminuting and sizing the dried filter cake to form oxides of manganese particles.

Oxides of manganese sorbent particles may formed by a process for regeneration of loaded oxides of manganese sorbent particles bearing a reaction product layer of the surface

thereof formed during reaction between unreacted oxides of manganese sorbent particles and target pollutants in a gas stream in a pollution removal system, comprising the steps of rapidly washing and filtering the loaded oxides of manganese sorbent particles in a rapid filtration wash unit with an aqueous oxidizing solution to dissolve the reaction product layer from the surface of the loaded oxides of manganese sorbent particles into the solution to form a filtrate and a regenerated oxides of manganese filter cake, the solution having Eh and pH values within the MnO_2 stability area; removing the regenerated oxides of manganese filter cake from the rapid filtration unit, the oxides of manganese of the filter cake having a pollutant loading capacity equal to or greater than the pollutant loading capacity of the unreacted oxides of manganese sorbent particles from which the loaded oxides of manganese sorbent particles are formed; drying the filter cake; and comminuting and sizing the dried filter cake to form oxides of manganese particles.

Oxides of manganese filter cakes particles may be formed by a process for pretreatment of virgin oxides of manganese for use as a sorbent to remove target pollutants from a gas stream, comprising the steps of washing virgin oxides of manganese in a wash unit with an aqueous oxidizing solution, the solution having Eh and pH values within the MnO_2 stability area; monitoring and adjusting the pH and/or the Eh values of the solution so as to maintain the Eh and pH values of the solution within the MnO_2 stability area; and filtering the washed oxides of manganese to form a filtrate and a pretreated oxides of manganese filter cake, the oxides of manganese of the filter cake having a pollutant loading capacity greater than the pollutant loading capacity of the virgin oxides of manganese.

Oxides of manganese filter cake may be formed by a process for pretreatment of virgin oxides of manganese for use as a sorbent to remove target pollutants from a gas stream, comprising the step of washing virgin oxides of manganese in a rapid filtration wash unit with an aqueous oxidizing solution to form a filtrate and a pretreated oxides of manganese filter cake, the solution having Eh and pH values within the MnO_2 stability area; wherein the oxides of manganese of the pretreated oxides of manganese filter cake have a pollutant loading capacity greater that the pollutant loading capacity of the virgin oxides of manganese.

Oxides of manganese filter cake formed by a process for regeneration of loaded oxides of manganese formed through reaction between unreacted oxides of a manganese and target pollutants in a gas stream comprising the step of washing loaded oxides of manganese in a rapid filtration wash unit with an aqueous oxidizing solution having Eh and pH values

within the MnO_2 stability area to form a filtrate and a regenerated oxides of manganese filter cake; wherein the oxides of manganese of the regenerated oxides of manganese filter cake have a pollutant loading capacity equal to or greater than the pollutant loading capacity of the unreacted oxides of manganese from which the loaded oxides of manganese are formed.

Oxides of manganese filter cake may be formed by a process for regeneration of loaded oxides of manganese sorbent particles bearing a reaction product layer of the surface thereof formed during reaction between unreacted oxides of manganese sorbent particles and target pollutants in a gas stream in a pollution removal system comprising the step of rapidly washing and filtering the loaded oxides of manganese sorbent particles in a rapid filtration wash unit with an aqueous oxidizing solution to dissolve the reaction product layer from the surface of the loaded oxides of manganese sorbent particles into the solution to form a filtrate and a regenerated oxides of manganese filter cake, the solution having Eh and pH values within the MnO_2 stability area; wherein the oxides of manganese in the regenerated oxides of manganese filter cake have a pollutant loading capacity equal to or greater than the pollutant loading capacity of the unreacted oxides of manganese sorbent particles from which the loaded oxides of manganese sorbent particles are formed.

In the processes for formation of the above-described oxides of manganese particles and oxides of manganese filter cakes, the aqueous oxidizing solution contains an oxidizer. The oxidizer may be selected from the group consisting of hypochlorites, perchlolates, permanganates, oxygen (O_2), air, ozone, peroxides, persulfates, and combinations thereof.

Detailed Description of Preferred Embodiments

The following definitions will be useful in understanding the invention disclosed herein:

"Reacted" or "loaded," as used interchangeably herein, refers in conjunction with "oxides of manganese" and/or "sorbent" to oxides of manganese or sorbent that has interacted with one or more target pollutants in a gas whether by chemical reaction, adsorption or absorption. The term does not mean that all reactive or active sites of the sorbent have been utilized as all such sites may not actually be utilized.

"Unreacted" or "virgin," as used interchangeably herein, refers in conjunction with "oxides of manganese" and/or "sorbent" to oxides of manganese or sorbent that has not interacted with target pollutants in a gas or gas stream.

"Nitrates of manganese," as used herein, refers to and includes the various forms of manganese nitrate, regardless of chemical formula, that may be formed through the chemical reaction between NO_X and the sorbent and includes hydrated forms as well.

"Sulfates of manganese," as used herein, refers to and includes the various forms of manganese sulfate, regardless of chemical formula that may be formed through the chemical reaction between SO_X and the sorbent and includes hydrated forms as well.

"Target pollutant," as used herein, refers to the pollutant or pollutants that are or are to be captured and removed from a gas stream.

"MnO_2 stability area," as used herein, refers to the region of thermodynamic stability for manganese dioxide delineated by Eh and pH values for aqueous solutions. More specifically, it refers to the region of thermodynamic stability for manganese dioxide delineated by Eh and pH values for aqueous solutions in an electrochemical stability diagram, such as presented by Pourbaix diagrams.

Oxides of manganese may be utilized for various applications. one such application is in the use as a sorbent for removal of target pollutants in gas streams. Examples of target pollutants that may be removed with an oxide of manganese sorbent, include but are not limited to, NO_X, SO_X, mercury (Hg) and some mercury compounds, H_2S and other totally reduced sulfides (TRS), and oxides of carbon. As previously noted above, certain target pollutant removal systems and processes methods utilizing oxide of manganese sorbents and referred to as Pahlman™ systems and Pahlman™ processes are described and disclosed in co-pending U.S. patent application numbers 09/919,600, 09/951,697, 10/044,089 and 10/025,270, all assigned to EnviroScrub Technologies Corporation, the assignee of this application. The disclosures of these four patent applications are incorporated herein by reference.

Oxides of manganese sorbents are introduced into these and other pollution removal systems and interact with the target pollutants in gas streams routed through the systems as a catalyst, a reactant, an absorbent or an adsorbent.

During such interaction in the process of pollutant removal, the oxidation (or valence) state of the oxides of manganese sorbent is reduced from its original state during reaction with the target pollutants. For example, where the target pollutants are NO_X or SO_X, possibly through overall reactions such as the following:

$SO_2+MnO_2 \rightarrow MnSO_4$ Reaction (1)

$2NO + O_2+MnO_2 \rightarrow Mn(NO_3)_2$ Reaction (2)

In both of the reactions above, manganese (Mn) is reduced from the +4 valence state to +2 valence state during formation of the reaction products shown. It should be noted that the actual reactions may include other steps not shown, and that indicating Reactions 1 and 2 is not in any way intended to limit the scope of the invention, but is used only to illustrate the process.

Additionally, it is believed that reaction products, such as the salts of Reaction (1) and Reaction (2) above, form on the surfaces of the sorbent particles of oxides of manganese. These reactions may extend to some depth inside the sorbent particles. Applicants believe that formation of such reactions products occurs on the surfaces of the oxides of manganese particles, resulting in a layer or coating, which effectively isolates the covered portion of the particle surface and thereby prevents continued rapid reaction with additional target pollutants. Further, the oxidation state and thus the loading capacity of the oxides of manganese below the surface of the reaction product coating may be reduced during the pollutant removal, thus diminishing the loading capacity of sorbent even after the reaction product have been removed or disassociated into an aqueous solution.

Following reaction with the target pollutants, it is desirable for economic reasons to re-use the unreacted portions of the sorbent for subsequent cycles of pollutant gas removal. These unreacted portions may exhibit decreased loading capacity. In order to render reacted oxides of manganese effective for subsequent re-use as a gas sorbent with high removal efficiency, it is necessary to: (1) remove reaction products, such as salts from the sorbent particle surfaces and (2) restore or increase the target pollutant loading capacity of the oxides of manganese sorbent. Applicants have found that washing the loaded oxides of manganese in an aqueous oxidizing solution that has been adjusted to within the MnO_2 stability area can accomplish these requirements. Further, Applicants have found that loading capacity of virgin or unreacted sorbent can be increased by washing with aqueous oxidizing solutions.

Without being bound by theory, Applicants believe that the processing of oxides of manganese according to the invention in aqueous oxidizing solution systems maintained with the MnO_2 stability window may beneficially affect a number of sorbent characteristics. Such characteristics may include particle size and shape, crystalline structure or morphology,

porosity, composition, surface area, bulk density ,electrochemical or oxidation potential or manganese valence states.

Regardless, the increased loading capacity is achieved with aqueous oxidizing solutions. Hypochlorites, such as sodium hypochlorite (NaOCl), have been found to be a suitable oxidizers for use in the aqueous oxidizing solutions, although many other oxidizers may also be suitable for this purpose. Other suitable oxidizers include, but are not limited to chlorates, such as sodium chlorate ($NaClO_3$), perchlorates such as sodium perchlorate (Na_2ClO_4), permanganates, such as potassium permanganate ($KMnO_4$), oxygen (O_2) or air, ozone (O_3), peroxides, such as H_2O_2, and persulfates, such as sodium peroxidisulfate ($Na_2S_2O_8$). The electrochemical potential of the aqueous oxidizing solution, and therefore the effectiveness of the treatment methods of the invention, depends, in part, upon the strength of the oxidizer and/or the concentration of the oxidizer in the solution.

In the treatment methods and systems disclosed herein, the oxidizer used to prepare the aqueous oxidizing solution, must be able to provide the required electrochemical (oxidizing) potential (Eh), within the specified pH range, to provide an Eh-pH combination to achieve stable solution equilibrium, as defined by the MnO_2 stability area as delineated in a Pourbaix diagram, such as those depicted in Figures 9 and 10. In a Pourbaix diagram, the MnO_2 stability area is defined by the thermodynamically stable ranges or boundaries of pH-Eh combinations that promote the existence and formation MnO_2 (Mn having valence state of +4) as the most thermodynamically stable form of manganese in an aqueous solution system. The domain of MnO_2 stability for an aqueous solution varies based upon the various components of the system. For example, changes in dissolved manganese ion concentration, solution temperature, and competing dissolved ions will affect the boundaries of stability for MnO_2. The effects of such changes upon the boundaries of the MnO_2 stability area on a Pourbaix Eh-pH diagram can be determined either by empirical data derived from experimentation or with computer software programs known to those skilled in the art, such as HSC Chemistry distributed by Outokumpu Oy of Finland. Software may also be written to determine the MnO_2 stability area as defined by other diagrams, such as the Latimer Diagram or the Frost Diagram.

In Figure 9, ranges of pH and Eh values for thermodynamically stable aqueous solutions of various manganese compounds are illustrated in graph form for aqueous solution systems at 25° C and a 1mole/liter manganese ion concentration. Figure 10 similarly illustrates ranges of pH and Eh values for aqueous solution systems at 25° C but with a 1.0 x

10^{-6} mole/liter manganese ion concentration. The Pourbaix Window diagrams depicted in Figures 9 and 10 were derived from the diagram presented in Atlas Of Electrochemical Equilibria in Aqueous Solutions," Marcel Pourbaix, pages 286-293, National Association of Corrosion Engineers, Houston, Texas.

The Eh and pH values as plotted on the graphs delineate the boundaries of the MnO_2 stability area for each of the two aqueous solution systems, emphasized with shading in Figures 9 and 10. A comparison of the boundaries of the two shaded areas on Figures 9 and 10 is illustrative of the different stability areas that exist under different system conditions.

Regardless of the specific system conditions, under the thermodynamically stable conditions for MnO_2 at a given pH and corresponding Eh range within the MnO_2 stability area or the given system conditions, the desired manganese valence state (theoretically +4) will exist. Thus, there is no propensity for Mn compounds in the +4 valence state to degrade to +3 or +2 valence states. Applicants have found that oxides of manganese treated in oxidizing solutions maintained within the MnO_2 stability area will exhibit a Mn valence state of close to +4, resulting in a MnO_2 with target pollutant loading capacities equal to and/or greater than (increased) the loading capacities of virgin or loaded oxides of manganese.

From the graph presented in Figure 9 or 10, the thermodynamically stable ranges or boundaries of pH-Eh combinations for the particular aqueous system that will promote the existence and formation of MnO_2 can be identified. Through their understanding of the relationships between these system parameters of the MnO_2 stability area of a Pourbaix Window diagram and application thereof to conditions of a given aqueous system, Applicants are able to treat both virgin and loaded oxides of manganese sorbents so as to yield a treated sorbent, both pretreated and recycled, having equal or increased loading capacity when compared to the untreated sorbent.

Oxidizing solutions having the desired pH-Eh combination can be prepared, maintained or adjusted by increasing or decreasing oxidizer, acid, or base concentrations and/or temperature adjustment, as appropriate, so that the conditions are adjusted to remain within the MnO_2 stability area. With monitoring of Eh and pH data, an operator can make necessary adjustments in order to maintain or return the oxidizing solution to conditions within the MnO_2 stability area. Monitoring and adjusting can also be automated utilizing electronic sensors and controllers.

Virgin (unreacted) oxides of manganese sorbent particles may be treated or pretreated to increase the oxidation state and/or target pollution loading capacity. This can be

accomplished by washing virgin (unreacted) sorbent with a wash solution of volume-to-sample weight ratio (liquid-to-solids ratio) of 2:1 or greater. However, effective treatment can also be accomplished with both lower and higher liquid-to-solids ratios as well. The sorbent powder is added to an aqueous oxidizing solution, for example a hypochlorite solution, stirred or agitated for a time sufficient to achieve an increased oxidation state and/or target pollution loading capacity. The then treated sorbent is separated by filtering, and the wet particles dried, which prepares the sorbent for target pollutant removal.

This method of virgin sorbent treatment may be better understood with reference to Figure 1. Turning to Figure 1 a virgin sorbent pretreatment system 10 is illustrated. The pretreatment system includes a washing unit 12, optionally equipped with pH probe 14 and Eh probe 16. As explained later in the specification, these probes may be utilized to provide information on aqueous solution conditions that may be utilized to provided manual or electronically controlled adjustments to relative to providing and maintaining the desire pH-Eh combination and other operating conditions. For example, temperature probe (not shown) may also optionally be provided. Virgin sorbent from virgin sorbent storage 18 is introduced into wash unit 12 along with an aqueous oxidizing solution prepared to have the desired pH-Eh combination within the MnO_2 stability area. Optionally, adjustments to the slurry pH and Eh can be made using appropriate oxidants for Eh adjustment and mineral acids and bases for pH adjustments.

The solid-liquid mixture or slurry is agitated or stirred and maintained at the desire operating conditions so as to increase the loading capacity and/or the Mn oxidation state. Optionally, adjustments to the slurry pH and Eh can be made using appropriate oxidants for Eh adjustment and mineral acids or bases for pH adjustment, so that the pH/Eh combination of the slurry is maintained within the window of MnO_2 stability for the specific system.

A filtration unit 22 configured for solid-liquid separation is next provided. Suitable filtration units known and readily identified by those skilled in the art may be utilized for filtration unit 22. The slurry from wash unit 12 is conveyed to filtration unit 22 where the treated sorbent is separated from the solution, with the wash solution or filtrate being routed for re-use, other processing or disposal. Since the filtrate may yet contain useful residual oxidizer, it may not be economic or desirable to dispose of the solution after a single use. However, after multiple uses, the solution may contain an accumulation of undesired constituents, such as contaminants and debris, that may be present in a load of virgin sorbent. Such undesired constituents may impact the efficiency or continued usefulness of the filtrate

for subsequent sorbent pretreatment. At that juncture the filtrate may be disposed of or otherwise processed to recover useful values.

In any event, the treated sorbent that has been separated from the slurry is in the form of a wet sorbent cake and may be dried prior to use in a pollutant removal system such as the Pahlman™ system if the sorbent is to be fed dry into the removal system. In some removal systems the sorbent may be fed as a slurry or even as a wet sorbent cake and for such removal systems a drying step would not be required. However, where drying is required or desired prior to introduction into a pollution removal system, the wet sorbent cake is routed to a dryer 24. A variety of dryers known to those skilled in the art may be utilized for this purpose, such as a rotary kiln, heat exchanger, oven, or other suitable dryers. After drying the treated sorbent may require comminution and possible sieving to reduce the sorbent particles to the desired size. Further, the sorbent dryer 24 may be a standard cake or slurry drying system known to those skilled in the art of drying, which could include a fluidized bed dryer or a spray dryer, configured or designed to inject the wet cake or slurry of oxides of manganese sorbent into the flow of a gas stream prior to introduction into a reaction chamber of a pollutant removal system. Whether dried and comminuted or routed to a cake or slurry drying system, the resulting sorbent having increased loading capacity is ready for use in a pollution removal system

Loaded (reacted) sorbent requires a different treatment in order to prepare it for reuse for target pollutant removal. Specifically, applicants have found it advantageous to rapidly remove the aqueous oxidizing solution from contact with the loaded sorbent being treated during washing once dissolved reaction products, such as soluble salts, have disassociated into solution. Applicants believe that such rapid removal of soluble salts is necessary in order to prevent possible re-deposition of a reaction product coating or layer, through precipitation of reaction products, e.g., sulfate and nitrate compounds or their derivatives, upon the washed oxides of manganese sorbent particles. Such coatings are believed to be detrimental to target pollutant removal efficiency of the sorbent. In order to prevent re-deposition of such coatings or layer, the washing or rinsing equipment is configured to prevent buildup of high dissolved salt concentrations and avoids prolonged sorbent contact with the wash solutions used.

Prolonged sorbent-solution contact should be avoided for another reason. As the reaction products are dissolved into solution, the acidity of the wash solution has been observed to increase, perhaps having a pH as low as 2.0-4.0, as a result of sulfate and nitrate salts, for example, combining with wash solution to form weak acids. Such pH shifts during

washing can and oxidizing of loaded sorbent can take the oxidizing solution outside of the MnO_2 stability area At the lower pH ranges, as illustrated in the Pourbaix Window diagram of Figure 9, an upward, sometimes significant, Eh adjustment would be required to remain within the MnO_2 stability area. Base could also be added to accomplish the desired adjustment; however, this will be ineffective if the oxidizer has been depleted, requiring oxidizer addition. As some oxidizers or oxidants are inexpensive, such Eh adjustment can be uneconomic. However, if not otherwise made, a treated sorbent with a lower valance state and/or loading capacity would result. Through avoidance of prolonged contact potentially, costly Eh adjustments and pH adjustments can be avoided or minimized and a treated sorbent with increased loading capacity can be obtained.

Rapid filtration can be accomplished by various methods such as vacuum or pressure filtration. On the laboratory scale, rapid filtration can be accomplished by placing the loaded oxides of manganese sorbent to be washed on a filter media, using an apparatus such as a Beuchner funnel, and maintaining a vacuum to the filter while applying the wash solution onto the loaded oxides of manganese sorbent. This results in rapid filtration of wash solutions through the sorbent, accompanied by rapid transport of dissolved salts away from the washed sorbent.

Loaded oxides of manganese sorbent samples are washed with an oxidizing wash solution of volume-to-sample weight ratio (liquid-to-solids ratio) of between 2:1 and 50:1 or greater, preferably 25:1 to 35:1. Within these liquid-to-solid ratios and with rapid filtration, effective dissolution and rapid removal of reaction products can be achieved, resulting in a treated or recycle processed (sometimes referred to as regenerated) sorbent having an increased oxidation state and/or target pollution loading capacity.

The system and method of recycle processing of loaded sorbent can be understood with reference to Figure 2. Turning to Figure 2 a loaded sorbent treatment or recycle processing system 30 is illustrated. The system 30 includes a combination rapid filtration wash unit 32. The wash unit 32 is configured to receive loaded sorbent from loaded sorbent storage 34 and to aqueous oxidizing solution. The wash unit 32 is further configured and operated so as to avoid and pooling of oxidizing solution or creation of a liquefied slurry or other conditions that would lead to prolonged solid-liquid contact such that the solution no longer remains within the MnO_2 stability area. Devices such as moving belt filters presses, filter presses, and filter drum presses or pressurized or vacuum filters or other such devices

known to those skilled in the art for rapid filtration washing can be used for rapid filtration wash unit 32.

Loaded sorbent is introduced into rapid-wash filtration unit 32 from loaded sorbent storage 34. An aqueous oxidizing solution prepared to have the desired pH-Eh combination within the MnO_2 stability area is then passed over and through the loaded sorbent with rapid filtration. The aqueous oxidizing solution may be monitored and adjusted prior to application to the loaded sorbent and the Eh and pH of the filtrate may be monitored. However, monitoring is not really required as long as there is no pooling so as to cause undesirable prolonged solid-liquid contact. Once the load sorbent had been washed for a time sufficient to restore or increase the loading capacity and/or Mn oxidation state, solution is no longer applied and the wet sorbent cake is routed to dryer 36. The same types and variety of dryers suitable as dryer 24 in the virgin sorbent pretreatment system 10 are suitable for use as dryer 36 in the loaded sorbent treatment system 30. And similarly, the selection will depend upon whether the recycle processed sorbent is to be introduced into a pollutant removal system dry or as a wet sorbent cake. If to be introduced dry, with comminution and possibly sieving, as necessary, the treated or recycle processed loaded sorbent is ready for re-introduction into a pollutant removal system.

It should be noted that the embodiment of the system of the invention in Figure 2 may be utilized for treatment of virgin oxides of manganese as well, even though prolonged liquid-solid contact need not be avoided for pretreatment. Thus, virgin sorbent may be treated in the system depicted in either Figure 1 or Figure 2 and by the same method as loaded sorbent.

Respectively, Figures 7 and 8 illustrate embodiments of the invention incorporating electronic controller to provide integrated simultaneous monitoring and adjustment of operational parameters for treatment of oxides of manganese with option feed back loop for checking the loading capacity of the treated sorbent.

With reference to Figure 7, the pretreatment system 40, illustrated in block flow, has a wash unit 42, a filtration unit 44, and a dryer 46. Wash unit 42 is equipped with temperature probe 47A, pH probe 47B and Eh probe 47C which are in electronic communication with a controller 50. A vessel 52 containing oxidizing solution is configured to feed oxidizing solution to wash unit 42. Virgin sorbent vessel 53 containing virgin sorbent is configured to feed virgin sorbent to wash unit 42. Acid and/or base vessel 54 is configured to feed acid and or base to wash unit 42. The feeders (not shown) of vessels 52, 53 and 54 are in electronic

communication with the controller 50. The controller is also in electronic communication with the Eh probe 55 with which the oxidizing solution vessel 53 is equipped. As illustrated treated sorbent from dryer 46 is routed to reaction chamber 56. Alternatively, sorbent from dryer 46 may be routed for storage or to a sorbent feeder prior to introduction to the reaction chamber 56. Reaction chamber 56 represents a pollution removal system or a reaction zone of a pollution removal system such as a Pahlman™ dry removal system described in co-pending U.S. patent application numbers 09/919,600, 09/951,697, 10/044,089 and 10/025,270 or any other pollution control system that utilizes oxides of manganese as a sorbent for removal of target pollutants from gas streams. Reaction chamber 56 is equipped with optional target pollutant concentration readers or continuous emission monitors (CEMS) for NO_X and SO_X readers 57A and 57B in electronic communication with controller 50.

The controller 50 interfaces with probes 47A-47C, readers 57A, 57B and the feeders of the vessels 52-54 for measurement and adjustment of operational parameters with in the pretreatment system. These parameters are important in determining the proper Eh of the aqueous oxidizing solution prior to addition to the washing unit 42 and the conditions in the washing unit 42 when the virgin sorbent is added. Eh probe 55 is in communication with controller 50 to adjust the Eh of oxidizing solution vessel 52. The controller 50 adds oxidant to vessel 52 until the desired Eh reading is abtained prior to addition to the wash unit 42. The temperature, pH, and Eh of the washing unit are monitored and adjusted so as to maintain conditions within the MnO_2 stability area.

The controller 50 contains a programmable logic controller (PLC) and other hardware components necessary for the operation of the controller such as a power supply, input and output nodules that would communicate with the probes 47A-47C and/or readers 57A, 57B, and the feeders of the vessels 52-54 . The controller 50 receives inputs from the various probes and readers and converts them into ladder logic language that would be used by an internal proportional integral derivative (PID) loop to individually and simultaneously monitor system operational parameters and to reconcile the inputs with predetermined or computer generated calculated set points for the operational parameters, such as temperature, and Eh and pH levels. As determined by computer logic, the controller 50 will send an output as necessary to any of the feeders of vessels 52-53 signaling a feeder to cycle on or to change feeder rate so as to maintain or adjust system operational parameters to within the MnO_2 stability area. The controller 50 may also contain an Ethernet card or other component that allows onsite or offsite remote display and operator interface and control as needed.

The controller 50 would be given a start command and direct the feeders of vessels 52 and 53 to inject, respectively, a predetermined amounts of virgin sorbent and oxidizing solution into the washing unit 42. The controller would signal the feed of a predetermined amount of oxidant into the aqueous oxidizing solution vessel checking and or adjusting the Eh and/or pH of the solution prior to feeding into the washing unit. The Eh of the oxidizing solution in vessel 52 may be adjusted by addition of an oxidizer in sufficient quantity as to raise the Eh to the desired level from an oxidizer vessel, not shown in Figure 7, containing a supply of oxidizer. As determined by programmed controller logic, the controller 50 would also check, based on inputs received from the probes 47A-47C and/or adjust the conditions of the washing unit by adjusting the temperature utilizing a heater or heat exchanger (not shown in Figure 7) to increase or decrease solution temperature; the pH, if needed, by increasing or decreasing the rate of base or acid feed; and the Eh, if needed, by increasing or decreasing the oxidizer concentration of the aqueous oxidizing solution. An optional, final quality control loop may be provided utilizing the readers 57A, 57B checking the loading performance of the treated sorbent by sending, for example, SOx and NOx readings back to the controller 50. As determined by controller logic, the controller 50 would then adjust the washing unit parameters, if needed, to provide treated oxides of manganese having increased loading capacity as compared to the untreated virgin sorbent contained in vessel 53. The same controller may also be used to control the entire operation of both the reaction chamber 56 and the pretreatment system 40, or separate controllers may be provided.

With reference to Figure 8, a recycle processing or regeneration system 60, for treating loaded sorbent is illustrated as a block flow diagram. The regeneration system 60 has a rapid filtration wash unit 62 from which treated sorbent is routed to dryer 64. The filtrate from wash unit 62 is directed for disposal or for other processing to recover useful values therein. Loaded sorbent vessel 66 is configured to feed loaded sorbent to wash unit 62. An oxidizing solution vessel 68 containing oxidizing solution is configured to feed oxidizing solution to wash unit 62. The feeders (not shown) of vessels 66, 68 are in electronic communication with the controller 70. Vessel 68 is equipped with an Eh probe 72A and pH probe 72B which are also in electronic communication with the controller 70. As illustrated, treated sorbent from the dryer 64 is routed to a reaction chamber 56. It being understood that, as in pretreatment system of Figure 7, the treated sorbent may be routed to a sorbent feeder or to a sorbent storage bin. Similarly, optional readers 57A, 57B are in electronic

communication with controller 70, providing a optional control loop to check loading performance of treated sorbent.

The controller 70 interfaces with the Eh probe 72A, pH probe 72B, temperature probe 72C, the readers 57A, 57B, and the feeders of the vessels 66, 68 to monitor and adjust the operational parameters of pH, Eh and temperature within the regeneration system 60. Additionally, the controller 70 interfaces with a vessel (not shown in Figure 8) and the feeder thereof to provide acid or base as necessary to the aqueous oxidizing solution. It is important to determine and adjust, as necessary, the operational parameters prior to introducing the oxidizing solution into wash unit 62 in order to assure that conditions of the oxidizing solution are within the MnO_2 stability area.

The controller 70 contains a programmable logic controller (PLC) and other components that would be necessary for the operation of the controller such as a power supply, input and output modules that would communicate with the probes 72A, 72B, 72C, the readers 57A, 57B and the feeders that supply the loaded sorbent, the aqueous oxidizing solution, and acid or base. The controller 70 receives inputs from the various probes and readers and convert them into ladder logic language that would be used by the internal proportional integral derivative (PID) loop to individually or simultaneously monitor system operational parameters and to reconcile the inputs with predetermined or computer generated calculated set points for the operational parameters. The controller 70 may also contain an Ethernet card or other component that allows onsite or offsite remote display and operator interface and control as needed. Such integrated control of the regeneration system allows a practical alternative to manual adjustments by an operator and lends itself to automation of the regeneration system even from remote locations.

The controller 70 would be given a start command and signal the feeders of the vessels 66, 68, simultaneously or in desired sequence, to inject a predetermined amount of loaded sorbent and oxidizing solution into rapid filtration wash unit 62. In order to maintain conditions of the solution within the MnO_2 stability area, the controller 70 would, as necessary, signal feed of a predetermined amount of oxidizer, acid or base into the aqueous oxidizing solution in vessel 68, regularly monitoring and/or adjusting the Eh and/or pH of the solution prior to being fed into the washing unit. As determined by the controller logic, the controller 70 would also check, based on inputs received from the probes 72A, 72B, 72C, and/or adjust the conditions of the aqueous oxidizing solution in vessel 68 by adjusting the temperature utilizing a heater or heat exchanger (not shown in Figure 8) to increase or

decrease solution temperature; the pH, if needed, by increasing or decreasing the rate of base or acid feed; and the Eh, if needed, by increasing or decreasing the oxidizer concentration of the aqueous oxidizing solution.

As in the pretreatment system, an optional, final quality control loop may be provided utilizing the readers 57A, 57B, checking the loading performance of the treated sorbent by sending, for example, SOx and NOx readings back to the controller 70. As determined by controller logic, the controller 70 would then adjust oxidizing solution parameters, if needed, to provide treated oxides of manganese having increased loading capacity as compared to the untreated loaded sorbent contained in vessel 66. The same controller may also be used to control the entire operation of both the reaction chamber 56 and the treatment system 40, or separate controllers may be provided.

Applicants conducted a series of lab-scale tests utilizing a live slipstream of an actual exhaust gas from a coal-fired combustion source in order to demonstrate the increased loaded capacity achieved with the invention. A glass reactor designed to mimic the gas-solid interactions known to be present in the reaction zones of a Pahlman™ dry target pollutant removal system was utilized for the tests. The glass reactor was a vertically positioned Pyrex™ glass cylinder having an internal diameter of 2 inches and a length of approximately 18 inches. For each test run, 50.0 grams of oxides of manganese were suspended in the reactor using a permeable fritted glass filter positioned approximately 4 inches from the bottom of the reactor, allowing for flow of the gas stream through the reactor while keeping the oxides of manganese suspended. The test reactor was insulated and configured with thermocouples for temperature readings and heating elements for temperature control to maintain a temperature set point, which in the purposes of the conducted testes was 250°F.

A NOx and SO_2 laden gas stream was pumped into the bottom of the test reactor at a flow rate which provided adequate fluidization of the bed of sorbent to promote optimal gas/solids contact. The reactor was heated during the testing to 250°F and the gas flow rate was metered at a constant 6.5 liters per minute (lpm). The slipstream of actual exhaust gas was from a 570 MW tangentially-fired coal-burning boiler operating on Powder River Basin (PRB) western coal.

The composition of the exhaust gas was measured both on the inlet and outlet of the test reactor with appropriate gas analyzers, as one skilled in the art would employ and for the test run examples presented found to be within the following ranges: Oxygen (O_2) 6-7%, carbon dioxide (CO_2) 10-12%, oxides of nitrogen (NOx) 190-250ppm, sulfur dioxide (SO_2)

400-500ppm. The composition of the inlet gas to the test reactor varied slightly from test to test, therefore the data was normalized and presented as pounds (lbs) of NOx or SO_2 into and out of the test reactor. The extent of NOx and SO_2 loading was then calculated to determine the effectiveness of the aqueous oxidizing rinse. The slipstream was passed through the fluidized bed of oxides of manganese, where the flow carried a portion of the sorbent up onto the filter, thus creating a filter cake, which mimics a bag house reaction chamber of a Pahlman™ dry target pollutant removal system.

SO_2 and NO_X concentrations were measured continuously alternating from the reactor inlet and outlet utilizing a continuous emissions monitoring system (CEMS). SO_2 concentrations were measured utilizing a Bovar Western Research model 921NMP spectrophotometric instrument and NO_X concentrations were measured utilizing a Thermo Electron model 42H chemiluminescent instrument. In order to obtain accurate and reliable emission concentrations, sampling and reporting was conducted in accordance with US EPA Reference CFR 40, Part 60, Appendix A, Method 6C. Inlet gas temperature was 250° F, with a differential pressure across the permeable fritted glass filter was 2.00" of WC. Figures 3 and 4 show the results of comparative test runs conducted utilizing 50g of each of virgin EMD type oxides of manganese sorbent, to provide a baseline, and treated virgin EMD type oxides of manganese sorbent prepared according to the invention. The treated virgin EMD type oxides of manganese sorbent sample was prepared by first treating with an aqueous oxidizing solution of 0.01% sodium hypochlorite (NaOCl). The measured pH and Eh values of the starting aqueous oxidizing solution of 0.01% sodium hypochlorite (NaOCl) were 8.5 and 900mV respectively, which provided a solution within the MnO_2 stability area. A quantity of virgin EMD type oxides of manganese sorbent (75.3g) was then added to a mixing beaker of 151g of the aqueous oxidizing solution of 0.01% sodium hypochlorite (NaOCl). The slurry was then continually stirred for a period of 15 minutes while recording data on the changing pH and Eh of the solution. The starting pH of 8.5 quickly dropped to 6.2 within the first minute of mixing and after 15 minutes had dropped to 6.1. The Eh which started at 900mV rose to 1040mV within the first minute of mixing then started slowly dropping in about even increments to a finial value of 885mV after 15 minutes. The slurry was then placed in a paper filter to separate the solids from the filtrate. The now treated EMD type oxides of manganese sorbent was then placed in a drying oven and dried for 5 hrs at 185°F (85°C). The dried sorbent was then gently deagglomerated and 50g were then placed in the laboratory test reactor for subsequent target pollutant loading, the measured data points

are plotted and identified in Figures 3 and 4 as "Virgin Sorbent Following Treatment". Along with the treated virgin EMD type sorbent loading rates, data points from untreated virgin EMD type oxides of manganese sorbent are alos plotted in Figure 3 and 4 as "Virgin Sorbent Baseline" to provide a baseline for loading capacity comparison.

Figure 3 shows the NOx loading curves. Looking at the virgin sorbent baseline, the virgin EMD type sorbent was at least achieving 90% NOx removal on a ppm basis for 24 minutes, during which time an accumulative total of 158 x 10^{-6} pounds of NOx entered into the laboratory test reactor with only 5.6 x 10^{-6} pounds of NOx exiting the reactor, for a total of 152.4 x 10^{-6} pounds of NOx being captured by the virgin EMD type oxides of manganese sorbent. The treated virgin EMD type sorbent is shown to exhibit substantially improved loading rates. NOx removal time at a NO_X removal rate of at least 90% was increased to 47 minutes. For comparison, an accumulative total of 331 x 10^{-6} pounds of NOx entered the laboratory test reactor with only 6.1 x 10^{-6} pounds of NOx exiting the reactor for a total of 324.9 x 10^{-6} pounds of NOx being captured by the treated virgin EMD type oxides of manganese sorbent

Figure 4 shows the SO_2 loading curves. Looking at the virgin sorbent baseline, the virgin EMD type sorbent was at least achieving 99% SO_2 removal on a ppm basis for 24 minutes, during which time an accumulative total of 548 x 10^{-6} pounds of SO_2 entered into the laboratory test reactor with only 1.9 x 10^{-6} pounds of SO_2 exiting the reactor, for a total of 546.1 x 10^{-6} pounds of SO_2 being captured by the virgin EMD type oxides of manganese sorbent. The treated virgin EMD type sorbent is shown to exhibit substantially improved loading rates. SO_2 removal time a a removal rate of at least 99% was increased to 61 minutes. For comparison, an accumulative total of 1266 x 10^{-6} pounds of SO_2 entered the laboratory test reactor with only 0.9 x 10^{-6} pounds of SO_2 exiting the reactor, for a total of 1265.1 x 10^{-6} pounds of SO_2 being captured by the treated virgin EMD type oxides of manganese sorbent.

Figures 5 and 6 show the results of comparative test runs conducted utilizing 50g of each of virgin EMD type oxides of manganese sorbent, to provide a baseline, and treated loaded EMD type oxides of manganese sorbent prepared according to the invention. The treated loaded EMD type sorbent sample was first treated with an aqueous oxidizing solution of 0.01% sodium hypochlorite (NaOCl). The measured pH and Eh values of the starting aqueous oxidizing solution of 0.01% sodium hypochlorite (NaOCl) were 8.9 and 900mV respectively, which provided a solution within the MnO_2 stability area. A quantity of loaded

EMD type oxides of manganese sorbent (98.8g) was evenly distributed on moistened filter paper, which was placed in a Beuchner funnel. After applying vacuum, a first quantity of 1650 ml of aqueous oxidizing solution of 0.01% sodium hypochlorite (NaOCl) was evenly applied to the loaded EMD type sorbent surface at a rate such that no solution pooling was allowed to take place. The filtrate was then collected and the pH and Eh was measured to be 2.8 and 1070mV respectively. A second quantity of 1650 ml of an aqueous oxidizing solution of 0.01% sodium hypochlorite (NaOCl) was again evenly applied to the loaded sorbent surface at a rate such that no solution pooling was allowed to take place. The second filtrate was then found to have a pH of 2.8 and an Eh of 1100mV. The now treated EMD type oxides of manganese sorbent was then placed in a drying oven and dried for 2 hrs at 185°F (85°C). The dried sorbent was then gently deagglomerated and 50g were then placed in the laboratory test reactor for subsequent target pollutant loading, the measured data points are plotted and identified in Figures 5 and 6 as "Loaded Sorbent Following Treatment." The data points from untreated virgin EMD type oxides of manganese sorbent are plotted in Figure 5 and 6 as "Virgin Sorbent Baseline" to provide a baseline for loading capacity comparison.

Figure 5 shows the NOx loading curves. Looking at the virgin sorbent baseline, the EMD type sorbent was at least achieving 90% NOx removal on a ppm basis for 24 minutes, during which time an accumulative total of 158×10^{-6} pounds of NOx entered into the laboratory test reactor with only 5.6×10^{-6} pounds of NOx exiting the reactor, for a total of 152.4×10^{-6} pounds of NOx being captured by the virgin EMD type oxides of manganese sorbent. The loaded EMD type sorbent following treatment is shown to exhibit substantially improved loading rates. NOx removal time at a removal rate of at least 90% was increased to 32 minutes. For comparison, an accumulative total of 222×10^{-6} pounds of NOx entered the laboratory test reactor with 8.4×10^{-6} pounds of NOx exiting the reactor, for a total of 213.6×10^{-6} pounds of NOx being captured by the treated loaded EMD type oxides of manganese sorbent.

Figure 6 shows the SO_2 loading curves. Looking at the virgin sorbent baseline, the EMD type sorbent was at least achieving 99% SO_2 removal on a ppm basis for 24 minutes, during which time an accumulative total of 548×10^{-6} pounds of SO_2 entered into the laboratory test reactor with only 1.9×10^{-6} pounds of SO_2 exiting the reactor, for a total of 546.1×10^{-6} pounds of SO_2 being captured by the virgin EMD type oxides of manganese sorbent. The treated loaded EMD type sorbent is shown to exhibit substantially improved

loading rates. SO_2 removal time at a rate of at least 99% was increased to 47 minutes. For comparison, an accumulative total of 901 x 10^{-6} pounds of SO_2 entered the laboratory test reactor with only 3.2 x 10^{-6} pounds of SO_2 exiting the reactor, for a total of 897.8 x 10^{-6} pounds of SO_2 being captured by the treated loaded EMD type oxides of manganese sorbent.

The data from the lab-scale tests presented in Figure 3-6 illustrate the increased loading capacity that is achievable with the invention.

While exemplary embodiments of this invention and methods of practicing the same have been illustrated and described, it should be understood that various changes, adaptations, and modifications might be made therein without departing from the spirit of the invention and the scope of the appended claims.

WHAT IS CLAIMED IS:

1. A process for pretreatment of virgin oxides of manganese for use as a sorbent to remove target pollutants from a gas stream, comprising the steps of:

a. washing virgin oxides of manganese in wash unit with an aqueous oxidizing solution, the solution having Eh and pH values within the MnO_2 stability area ; and

b. filtering the washed oxides of manganese to form a filtrate and a pretreated oxides of manganese filter cake, the oxides of manganese of the filter cake having a pollutant loading capacity greater than the pollutant loading capacity of the virgin oxides of manganese.

2. A process for pretreatment of virgin oxides of manganese for use as a sorbent to remove target pollutants from a gas stream, comprising the steps of:

a. washing virgin oxides of manganese in a wash unit with an aqueous oxidizing solution, the solution having Eh and pH values within the MnO_2 stability area ;

b. monitoring and adjusting the pH and/or the Eh values of the solution so as to maintain the Eh and pH values of the solution within the MnO_2 stability area; and

c. filtering the washed oxides of manganese to form a filtrate and a pretreated oxides of manganese filter cake, the oxides of manganese of the filter cake having a pollutant loading capacity greater than the pollutant loading capacity of the virgin oxides of manganese.

3. A process for pretreatment of virgin oxides of manganese for use as a sorbent to remove target pollutants from a gas stream, comprising the steps of:

a. washing virgin oxides of manganese in a rapid filtration wash unit with an aqueous oxidizing solution to form a filtrate and a pretreated oxides of manganese filter cake, the solution having Eh and pH values within the MnO_2 stability area; and

b. removing the pretreated oxides of manganese filter cake from the rapid filtration unit, the oxides of manganese of the filter cake having a pollutant loading capacity greater that the pollutant loading capacity of the virgin oxides of manganese.

4. A process for regeneration of loaded oxides of manganese formed through reaction between unreacted oxides of a manganese and target pollutants in a gas stream, comprising the steps of:

a. washing loaded oxides of manganese in a rapid filtration wash unit with an aqueous oxidizing solution having Eh and pH values within the MnO_2 stability area to form a filtrate and a regenerated oxides of manganese filter cake; and

b. removing the regenerated oxides of manganese filter cake from the rapid filtration unit, the oxides of manganese of the filter cake having a pollutant loading capacity equal to or greater than the pollutant loading capacity of the unreacted oxides of manganese from which the loaded oxides of manganese are formed.

5. A process for regeneration of loaded oxides of manganese sorbent particles bearing a reaction product layer of the surface thereof formed during reaction between unreacted oxides of manganese sorbent particles and target pollutants in a gas stream in a pollution removal system, comprising the steps of :

a. rapidly washing and filtering the loaded oxides of manganese sorbent particles in a rapid filtration wash unit with an aqueous oxidizing solution to dissolve the reaction product layer from the surface of the loaded oxides of manganese sorbent particles into the solution to form a filtrate and a regenerated oxides of manganese filter cake, the solution having Eh and pH values within the MnO_2 stability area; and

b. removing the regenerated oxides of manganese filter cake from the rapid filtration unit, the oxides of manganese of the filter cake having a pollutant loading capacity equal to or greater than the pollutant loading capacity of the unreacted oxides of manganese sorbent particles from which the loaded oxides of manganese sorbent particles are formed.

6. The process of any one of claims 1 to 5, further comprising the steps of:
drying the filter cake; and
comminuting and sizing the dried filter cake to provide oxides of manganese particles.

7. The process of any one of claims 1 to 5, further comprising the steps of:
drying the filter cake;

comminuting and sizing the dried filter cake to provide oxides of manganese particles for introduction into a feeder or reaction chamber of a dry pollution removal system.

8. The process of any one of claims 1 to 5, further comprising the steps of:

drying the filter cake;

comminuting and sizing the dried filter cake to provide oxides of manganese particles for introduction into a feeder or reaction chamber of a dry pollution removal system; and

introducing the comminuted and sized oxides of manganese particles into the feeder or reaction chamber.

9. The process of any one of claims 1-5, further comprising the step of:

conveying the filter cake to a dryer configured to inject the a wet filter cake or a slurry of oxides of manganese sorbent into the flow of a gas stream prior to introduction into a reaction chamber of a pollutant removal system.

10. The process of any one of claims 1-5, further comprising the steps of:

conveying the filter cake to a dryer configured to inject the a wet filter cake or a slurry of oxides of manganese sorbent into the flow of a gas stream prior to introduction into a reaction chamber of a pollutant removal system; and

injecting the wet filter cake or the slurry into the reaction chamber.

11. The process of claim 2, wherein the monitoring and adjusting step is carried out by a controller capable of individually or simultaneously monitoring and adjusting system operational parameters, the controller providing integrated control of within the wash unit of the temperature, Eh values, and pH values in order to maintain conditions of the aqueous oxidizing solution within the MnO_2 stability area.

12. The process of anyone of claims 1-5, , wherein the aqueous oxidizing solution contains an oxidizer selected from the group consisting of hypochlorites, chlorates, perchlorates, permanganates, oxygen (O_2), air, ozone, peroxides, persulfates, and combinations thereof.

13. A system for pretreatment of virgin oxides of manganese, comprising:

a. wash unit configured to hold and agitate a slurry of virgin oxides of manganese in an aqueous oxidizing solution, the solution have Eh and pH values within the MnO_2 stability area, the wash unit being optionally equipped with a pH probe, an Eh probe, and a temperature probe, the wash unit being further configured to receive the slurry of virgin oxides of manganese or to individually receive virgin oxides of manganese and the aqueous oxidizing solution;

b. a filtration unit configured to receive the slurry from the wash unit and to filter the slurry to form a filtrate and a pretreated oxides of manganese filter cake;

c. an aqueous oxidizing solution vessel containing a supply of aqueous oxidizing solution, the solution having Eh and pH values within the MnO_2 stability area, the vessel having a feeder configured to feed aqueous oxidizing solution to the wash unit and being optionally equipped with an Eh probe;

d. an oxides of manganese vessel containing a supply of virgin oxides of manganese, the vessel having a feeder configured to feed oxides of manganese directly to the wash unit or, in conjunction with the aqueous oxidizing solution feeder to feed a slurry of virgin oxides of manganese to the wash unit; and

e. a pH adjust vessel having a supply of acid or base or separate supplies of acid and base, the pH adjust vessel having a feeder configured to feed acid and/or base to the wash unit.

14. A system for treatment of virgin or loaded oxides of manganese, comprising:

a. a rapid filtration wash unit, the wash unit being configured to individually receive oxides of manganese and an aqueous oxidizing solution having a Eh and pH values within the MnO_2 stability area, the wash unit being further configured to rapidly remove the aqueous oxidizing solution from contact with the oxides of manganese forming a filtrate and a treated oxides of manganese filter cake;

b. an aqueous oxidizing solution vessel containing a supply of aqueous oxidizing solution, the solution having Eh and pH values within the MnO_2 stability area, the vessel having a feeder configured to feed aqueous oxidizing solution to the wash unit and being optionally equipped with a probe selected from the group consisting of an Eh probe, a pH probe, a temperature probe, or any combination thereof;

c. an oxides of manganese vessel containing a supply of virgin or loaded oxides of manganese, the vessel having a feeder configured to feed oxides of manganese to the wash unit; and

d. optionally, a oxidizer vessel containing a supply of oxidizer, the oxidizer vessel having feeder configured to feed oxidizer to the aqueous oxidizing solution vessel; and

e. optionally, a pH adjust vessel having a supply of acid or base or separate supplies of acid and base, the pH adjust vessel having a feeder configured to feed acid and/or base to the aqueous oxidizing solution vessel.

15. The system of claim 13, further comprising

a controller capable of individually or simultaneously monitoring and adjusting system operational parameters, the controller providing integrated control within the wash unit of the temperature, Eh values, and pH values in order to maintain conditions of the aqueous oxidizing solution within the MnO_2 stability area.

16. The system of claim 14, further comprising

a controller capable of individually or simultaneously monitoring and adjusting system operational parameters, the controller providing integrated control of the temperature, Eh values, and pH values within the aqueous oxidizing solution vessel in order to maintain conditions of the aqueous oxidizing solution within the MnO_2 stability area.

17. A system for pretreatment of virgin oxides of manganese, comprising:

a. a wash unit configured to hold and agitate a slurry of virgin oxides of manganese in an aqueous oxidizing solution, the solution have Eh and pH values within the MnO_2 stability area, the wash unit being equipped with a pH probe, an Eh probe, and a temperature probe, the wash unit being further configured to receive the slurry of virgin oxides of manganese or to individually receive virgin oxides of manganese and the aqueous oxidizing solution;

b. a filtration unit configured to receive the slurry from the wash unit and to filter the slurry to form a filtrate and a pretreated oxides of manganese filter cake;

c. an aqueous oxidizing solution vessel containing a supply of aqueous oxidizing solution, the solution having Eh and pH values within the MnO_2 stability area, the vessel having

a feeder configured to feed aqueous oxidizing solution to the wash unit and being equipped with an Eh probe;

d. an oxides of manganese vessel containing a supply of virgin oxides of manganese, the vessel having a feeder configured to feed oxides of manganese directly to the wash unit or, in conjunction with the aqueous oxidizing solution feeder to feed a slurry of virgin oxides of manganese to the wash unit;

e. a pH adjust vessel having a supply of acid or base or separate supplies of acid and base, the pH adjust vessel having a feeder configured to feed acid and/or base to the wash unit;

f. a controller capable of individually or simultaneously monitoring and adjusting system operational parameters, the controller providing integrated control within the wash unit of the temperature, Eh values, and pH values in order to maintain conditions of the aqueous oxidizing solution within the MnO_2 stability area, the controller being electronic communication with the probes and feeders of the system..

18. A system for treatment of virgin or loaded oxides of manganese, comprising:

a. a rapid filtration wash unit, the wash unit being configured to individually receive oxides of manganese and an aqueous oxidizing solution having a Eh and pH values within the MnO_2 stability area, the wash unit being further configured to rapidly remove the aqueous oxidizing solution from contact with the oxides of manganese forming a filtrate and a treated oxides of manganese filter cake;

b. an aqueous oxidizing solution vessel containing a supply of aqueous oxidizing solution, the solution having Eh and pH values within the MnO_2 stability area, the vessel having a feeder configured to feed aqueous oxidizing solution to the wash unit and being equipped with a probe selected from the group consisting of an Eh probe, a pH probe, a temperature probe, or any combination thereof;

c. an oxides of manganese vessel containing a supply of virgin or loaded oxides of manganese, the vessel having a feeder configured to feed oxides of manganese to the wash unit;

d. a oxidizer vessel containing a supply of oxidizer, the oxidizer vessel having feeder configured to feed oxidizer to the aqueous oxidizing solution vessel;

e. a pH adjust vessel having a supply of acid or base or separate supplies of acid and base, the pH adjust vessel having a feeder configured to feed acid and/or base to the aqueous oxidizing solution vessel; and

f. a controller capable of individually or simultaneously monitoring and adjusting system operational parameters, the controller providing integrated control of the temperature, Eh values, and pH values within the aqueous oxidizing solution vessel in order to maintain conditions of the aqueous oxidizing solution within the MnO_2 stability area, the controller being in electronic communication with the probes and feeders of the system.

19. The system of any one of claims 13, 14, 17, or 18, wherein the aqueous oxidizing solution contains an oxidizer selected from the group consisting of hypochlorites, chlorates, perchlorates, permanganates, oxygen (O_2), air, ozone, peroxides, persulfates, and combinations thereof.

20. The system of claim 15, further comprising,

a dryer for drying the filter cake;

a device for comminuting and sizing the dried filter cake to form oxides of manganese particles;

a reaction chamber of a pollutant removal system into which the oxides of manganese particles are introduced to capture target pollutants from a gas, the reaction chamber being equipped with at least one target pollutant concentration reader for measuring target pollutant concentration of the gas as it exits the reaction chamber, the reader being in electronic communication with the controller for checking the loading performance of the oxides of manganese particles and signaling need for adjustment of operational parameters within the wash unit.

21. The system of claim 16, further comprising:

a dryer for drying the filter cake;

a device for comminuting and sizing the dried filter cake to form oxides of manganese particles;

a reaction chamber of a pollutant removal system into which the oxides of manganese particles are introduced to capture target pollutants from a gas, the reaction chamber being equipped with at least one target pollutant concentration reader for measuring target pollutant concentration of the gas as it exits the reaction chamber, the reader being in electronic communication with the controller for checking the loading performance of the oxides of manganese particles and signaling need for adjustment of operational parameters within the aqueous oxidizing solution vessel.

22. The system of claim 17, further comprising,

a dryer for drying the filter cake;

a device for comminuting and sizing the dried filter cake to form oxides of manganese particles;

a reaction chamber of a pollutant removal system into which the oxides of manganese particles are introduced to capture target pollutants from a gas, the reaction chamber being equipped with at least one target pollutant concentration reader for measuring target pollutant concentration of the gas as it exits the reaction chamber, the reader being in electronic communication with the controller for checking the loading performance of the oxides of manganese particles and signaling need for adjustment of operational parameters within the wash unit.

23. The system of claim 18, further comprising:

a dryer for drying the filter cake;

a device for comminuting and sizing the dried filter cake to form oxides of manganese particles;

a reaction chamber of a pollutant removal system into which the oxides of manganese particles are introduced to capture target pollutants from a gas, the reaction chamber being equipped with at least one target pollutant concentration reader for measuring target pollutant concentration of the gas as it exits the reaction chamber, the reader being in electronic communication with the controller for checking the loading performance of the oxides of manganese particles and signaling need for adjustment of operational parameters within the aqueous oxidizing solution vessel.

24. Oxides of manganese particles formed by a process for pretreatment of virgin oxides of manganese for use as a sorbent to remove target pollutants from a gas stream, comprising the steps of:

a. washing virgin oxides of manganese in wash unit with an aqueous oxidizing solution, the solution having Eh and pH values within the MnO_2 stability area ;

b. filtering the washed oxides of manganese to form a filtrate and a pretreated oxides of manganese filter cake, the oxides of manganese of the filter cake having a pollutant loading capacity greater than the pollutant loading capacity of the virgin oxides of manganese;

c. drying the filter cake; and

d. comminuting and sizing the dried filter cake to form oxides of manganese particles.

25. Oxides of manganese particles formed by a process for pretreatment of virgin oxides of manganese for use as a sorbent to remove target pollutants from a gas stream, comprising the steps of:

a. washing virgin oxides of manganese in a wash unit with an aqueous oxidizing solution, the solution having Eh and pH values within the MnO_2 stability area ;

b. monitoring and adjusting the pH and/or the Eh values of the solution so as to maintain the Eh and pH values of the solution within the MnO_2 stability area;

c. filtering the washed oxides of manganese to form a filtrate and a pretreated oxides of manganese filter cake, the oxides of manganese of the filter cake having a pollutant loading capacity greater than the pollutant loading capacity of the virgin oxides of manganese;

d. drying the filter cake; and

e. comminuting and sizing the dried filter cake to form oxides of manganese particles.

26. Oxides of manganese particles formed by a process for pretreatment of virgin oxides of manganese for use as a sorbent to remove target pollutants from a gas stream, comprising the steps of:

a. washing virgin oxides of manganese in a rapid filtration wash unit with an aqueous oxidizing solution to form a filtrate and a pretreated oxides of manganese filter cake, the solution having Eh and pH values within the MnO_2 stability area;

b. removing the pretreated oxides of manganese filter cake from the rapid filtration unit, the oxides of manganese of the filter cake having a pollutant loading capacity greater than the pollutant loading capacity of the virgin oxides of manganese;

c. drying the filter cake; and

d. comminuting and sizing the dried filter cake to form oxides of manganese particles.

27. Oxides of manganese particles formed by a process for regeneration of loaded oxides of manganese formed through reaction between unreacted oxides of a manganese and target pollutants in a gas stream, comprising the steps of:

a. washing loaded oxides of manganese in a rapid filtration wash unit with an aqueous oxidizing solution having Eh and pH values within the MnO_2 stability area to form a filtrate and a regenerated oxides of manganese filter cake;

b. removing the regenerated oxides of manganese filter cake from the rapid filtration unit, the oxides of manganese of the filter cake having a pollutant loading capacity equal to or greater than the pollutant loading capacity of the unreacted oxides of manganese from which the loaded oxides of manganese are formed;

c. drying the filter cake; and

d. comminuting and sizing the dried filter cake to form oxides of manganese particles.

28. Oxides of manganese sorbent particles formed by a process for regeneration of loaded oxides of manganese sorbent particles bearing a reaction product layer of the surface thereof formed during reaction between unreacted oxides of manganese sorbent particles and target pollutants in a gas stream in a pollution removal system, comprising the steps of:

a. rapidly washing and filtering the loaded oxides of manganese sorbent particles in a rapid filtration wash unit with an aqueous oxidizing solution to dissolve the reaction product layer from the surface of the loaded oxides of manganese sorbent particles into the solution to

form a filtrate and a regenerated oxides of manganese filter cake, the solution having Eh and pH values within the MnO_2 stability area;

b. removing the regenerated oxides of manganese filter cake from the rapid filtration unit, the oxides of manganese of the filter cake having a pollutant loading capacity equal to or greater than the pollutant loading capacity of the unreacted oxides of manganese sorbent particles from which the loaded oxides of manganese sorbent particles are formed;

c. drying the filter cake; and

d. comminuting and sizing the dried filter cake to form oxides of manganese particles.

29. The oxides of manganese particles formed by the process of anyone of claims 24-28, wherein the aqueous oxidizing solution contains an oxidizer selected from the group consisting of hypochlorites, chlorates, perchlorates, permanganates, oxygen (O_2), air, ozone, peroxides, persulfates, and combinations thereof.

30. Oxides of manganese filter cakes particles formed by a process for pretreatment of virgin oxides of manganese for use as a sorbent to remove target pollutants from a gas stream, comprising the steps of:

a. washing virgin oxides of manganese in a wash unit with an aqueous oxidizing solution, the solution having Eh and pH values within the MnO_2 stability area ;

b. monitoring and adjusting the pH and/or the Eh values of the solution so as to maintain the Eh and pH values of the solution within the MnO_2 stability area; and

c. filtering the washed oxides of manganese to form a filtrate and a pretreated oxides of manganese filter cake, the oxides of manganese of the filter cake having a pollutant loading capacity greater than the pollutant loading capacity of the virgin oxides of manganese.

31. Oxides of manganese filter cake formed by a process for pretreatment of virgin oxides of manganese for use as a sorbent to remove target pollutants from a gas stream, comprising the step of:

washing virgin oxides of manganese in a rapid filtration wash unit with an aqueous oxidizing solution to form a filtrate and a pretreated oxides of manganese filter cake, the solution having Eh and pH values within the MnO_2 stability area;

wherein the oxides of manganese of the pretreated oxides of manganese filter cake have a pollutant loading capacity greater that the pollutant loading capacity of the virgin oxides of manganese.

32. Oxides of manganese filter cake formed by a process for regeneration of loaded oxides of manganese formed through reaction between unreacted oxides of a manganese and target pollutants in a gas stream, comprising the step of:

washing loaded oxides of manganese in a rapid filtration wash unit with an aqueous oxidizing solution having Eh and pH values within the MnO_2 stability area to form a filtrate and a regenerated oxides of manganese filter cake;

wherein the oxides of manganese of the regenerated oxides of manganese filter cake have a pollutant loading capacity equal to or greater than the pollutant loading capacity of the unreacted oxides of manganese from which the loaded oxides of manganese are formed.

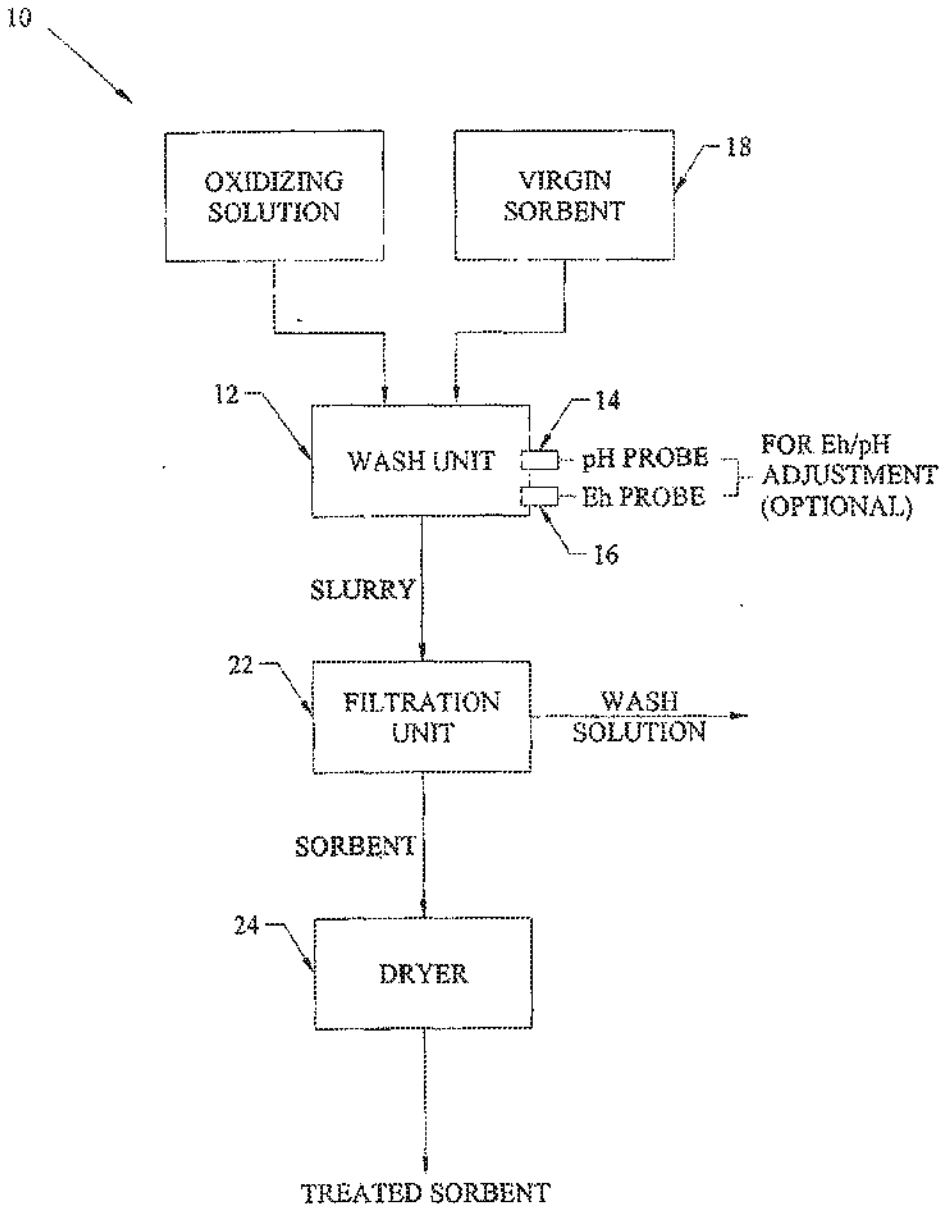
33. Oxides of manganese filter cake formed by a process for regeneration of loaded oxides of manganese sorbent particles bearing a reaction product layer of the surface thereof formed during reaction between unreacted oxides of manganese sorbent particles and target pollutants in a gas stream in a pollution removal system, comprising the step of:

rapidly washing and filtering the loaded oxides of manganese sorbent particles in a rapid filtration wash unit with an aqueous oxidizing solution to dissolve the reaction product layer from the surface of the loaded oxides of manganese sorbent particles into the solution to form a filtrate and a regenerated oxides of manganese filter cake, the solution having Eh and pH values within the MnO_2 stability area;

wherein the oxides of manganese in the regenerated oxides of manganese filter cake have a pollutant loading capacity equal to or greater than the pollutant loading capacity of the unreacted oxides of manganese sorbent particles from which the loaded oxides of manganese sorbent particles are formed.

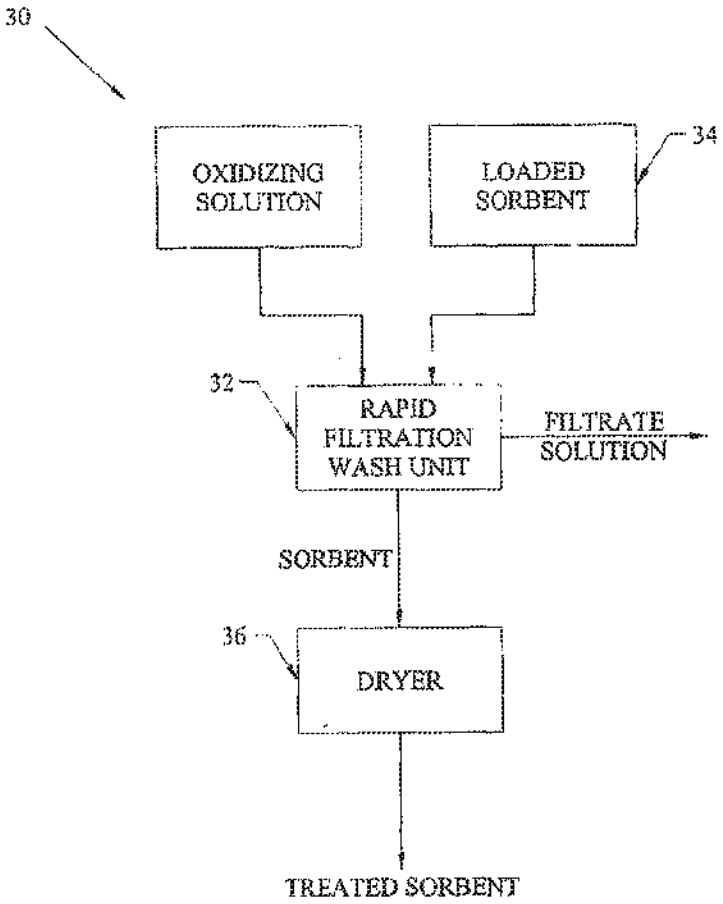
34. The filter cakes of anyone of claims 30-33, wherein the aqueous oxidizing solution contains an oxidizer selected from the group consisting of hypochlorites, chlorates, perchlorates, permanganates, oxygen (O_2), air, ozone, peroxides, persulfates, and combinations thereof.

FIGURE 1
TREATMENT OF VIRGIN OXIDES OF MANGANESE SORBENT



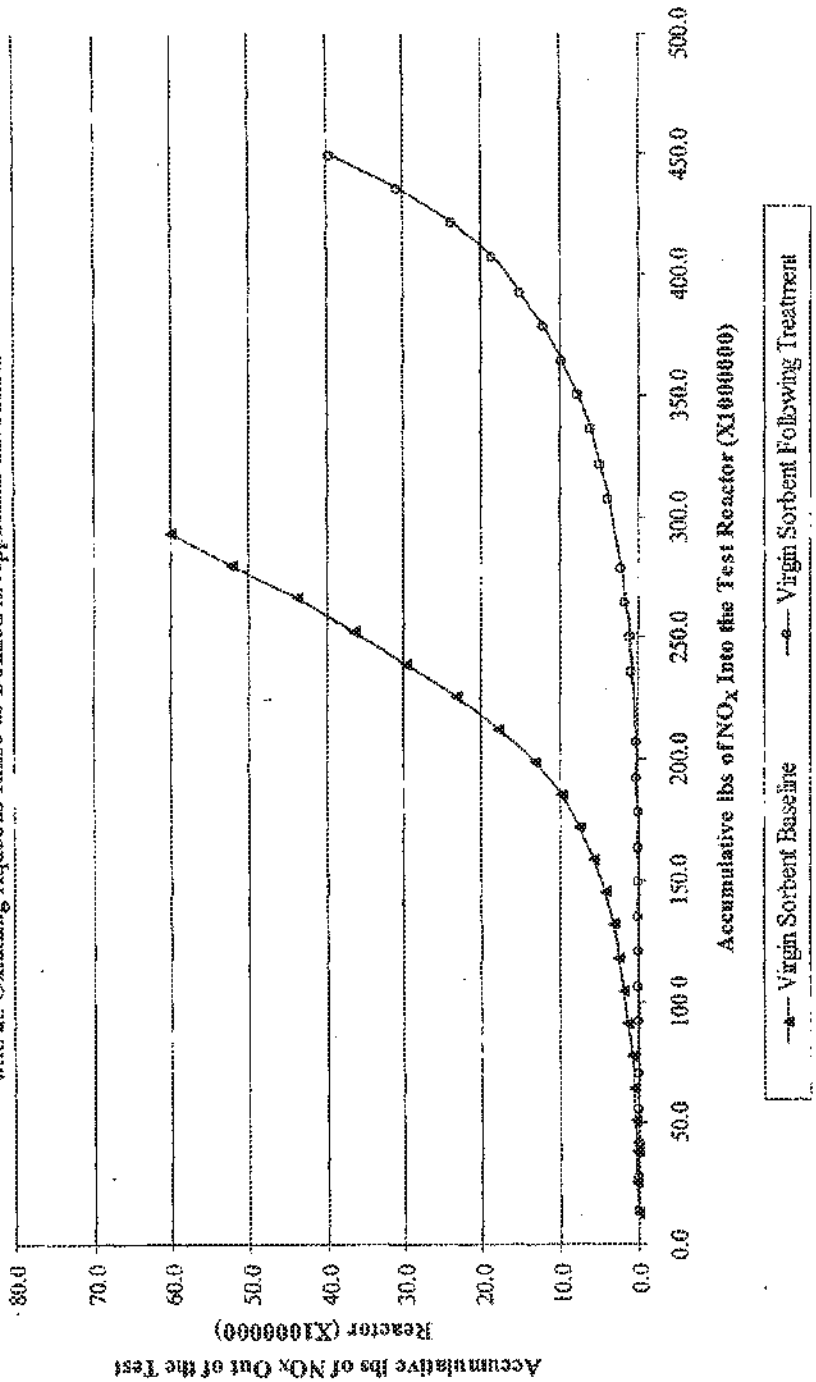
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Page 1506

FIGURE 2
TREATMENT OF LOADED OXIDES OF MANGANESE SORBENT



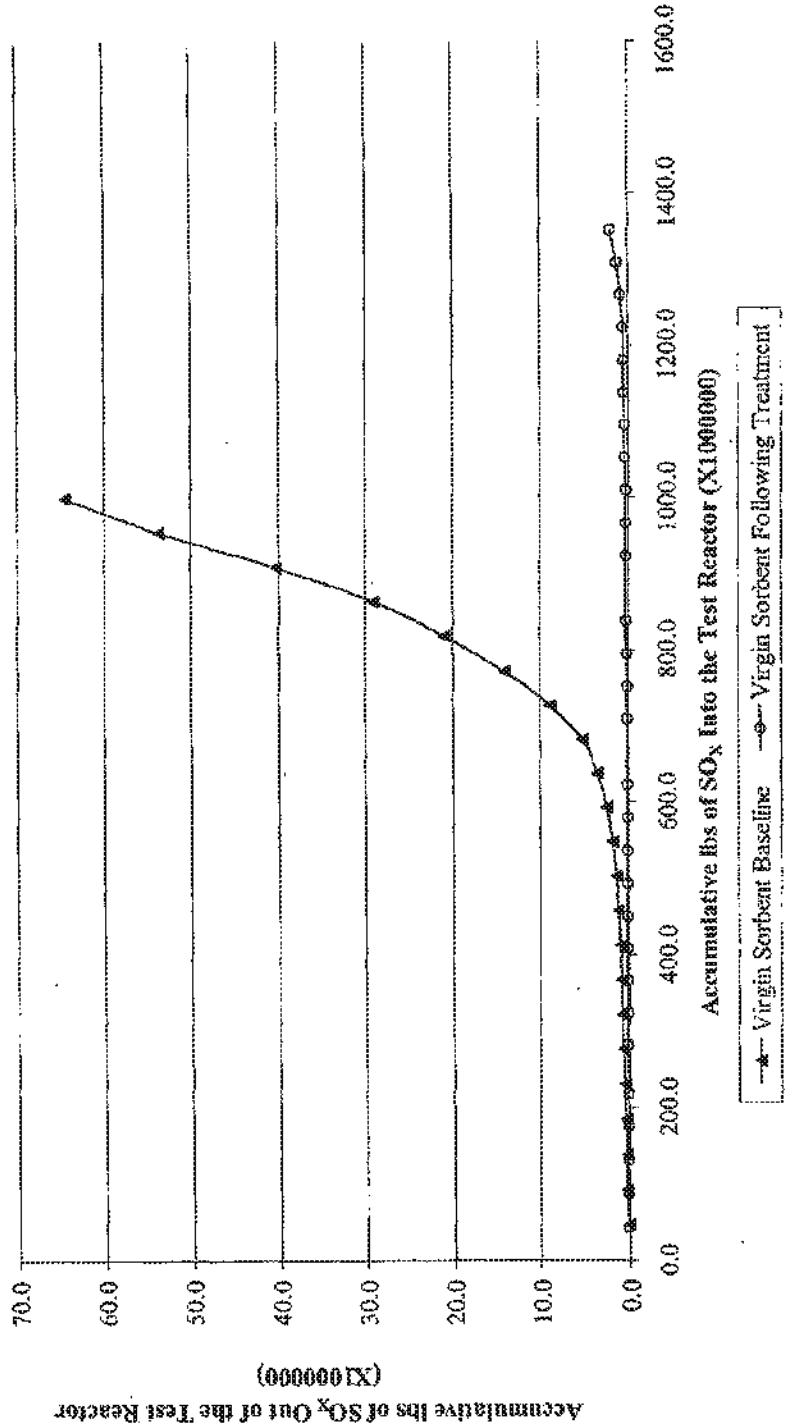
PAC ET AL. EXHIBIT 1023
Page 1507

Figure 3
NO_X Loading of Virgin Unreacted Oxides of Manganese Sorbent Following Treatment with an Oxidizing Aqueous Rinse as Defined in Applicants Invention.



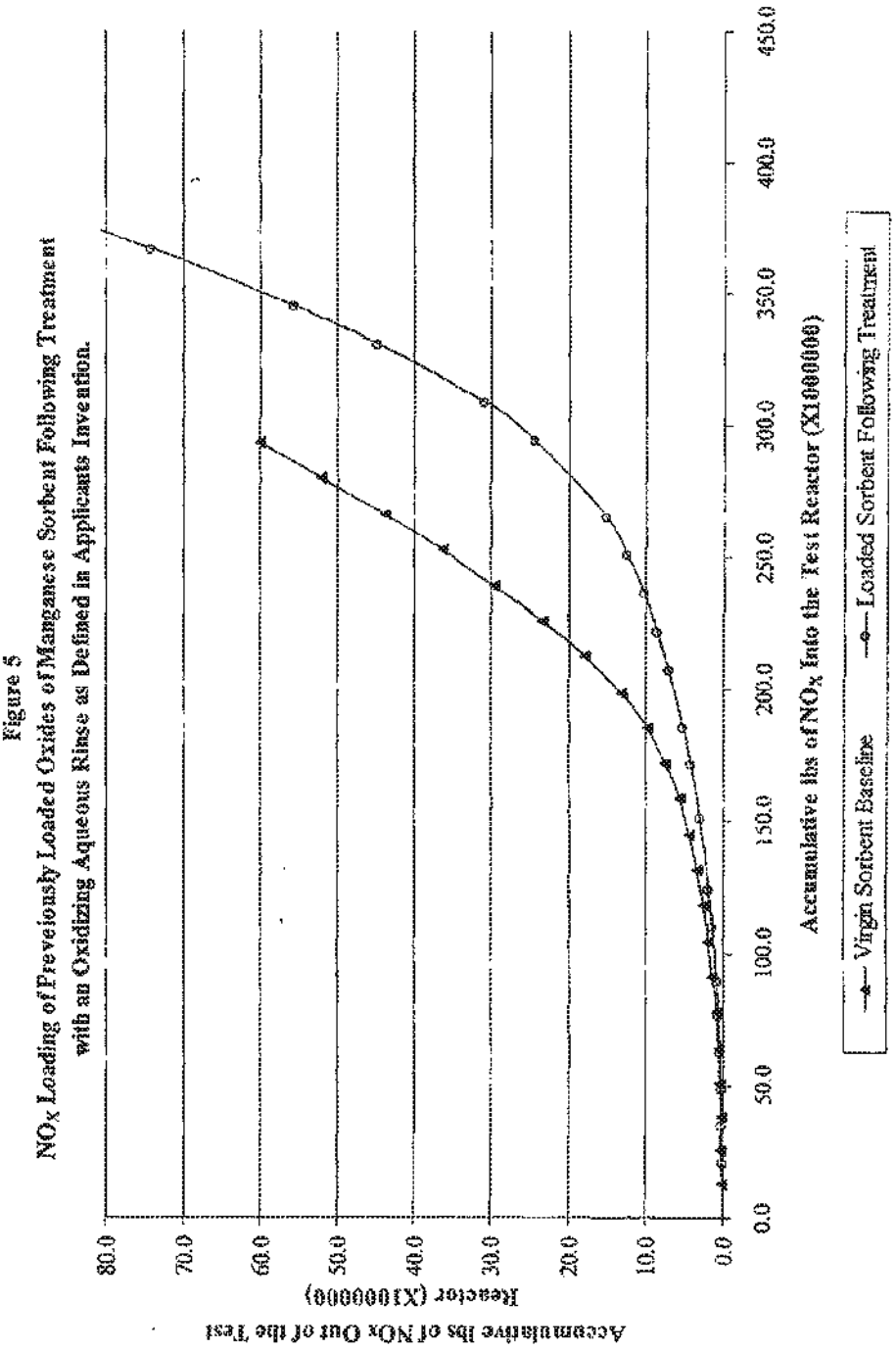
PAC ET AL. EXHIBIT 1023
Page 1508

Figure 4
SO_X Loading of Virgin Unreacted Oxides of Manganese Sorbent Following Treatment with an Oxidizing Aqueous Rinse as Defined in Applicants Invention.



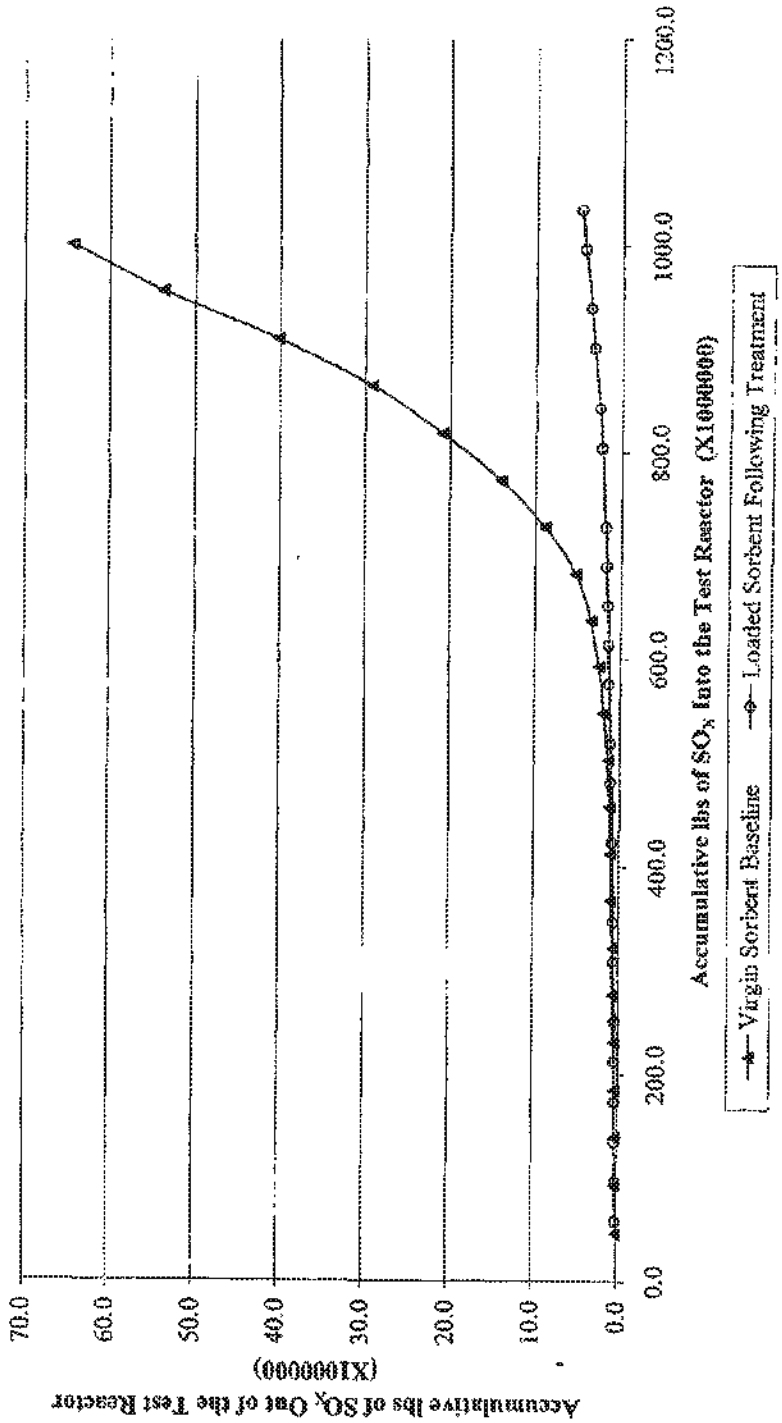
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Page 1509

Figure 5
NO_X Loading of Preveiously Loaded Oxides of Manganese Sorbent Following Treatment with an Oxidizing Aqueous Rinse as Defined in Applicants Invention.



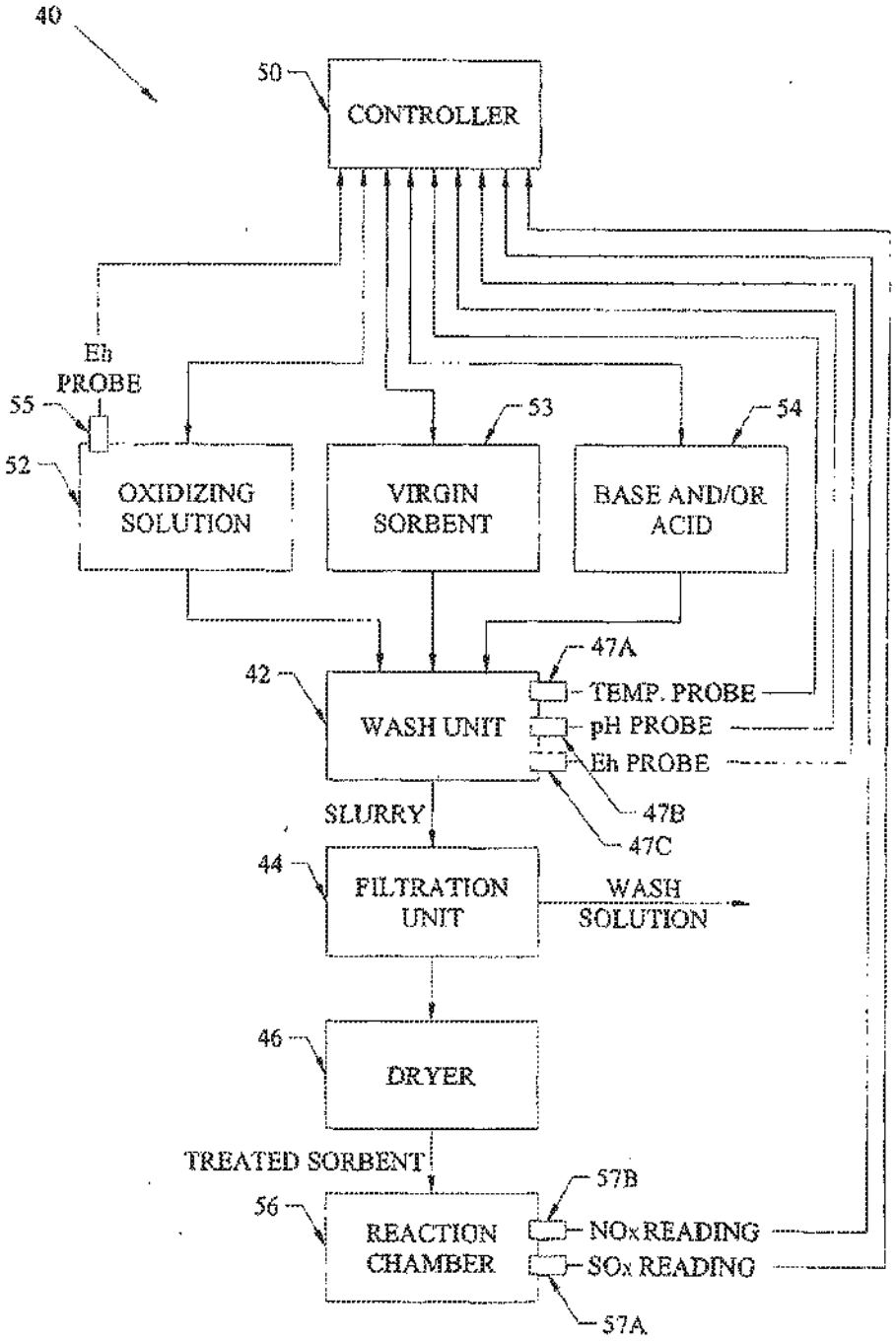
PAC ET AL. EXHIBIT 1023
Page 1510

Figure 6
SO_X Loading of Previously Loaded Oxides of Manganese Sorbent Following Treatment with an Oxidizing Aqueous Rinse as Defined in Applicants Invention.



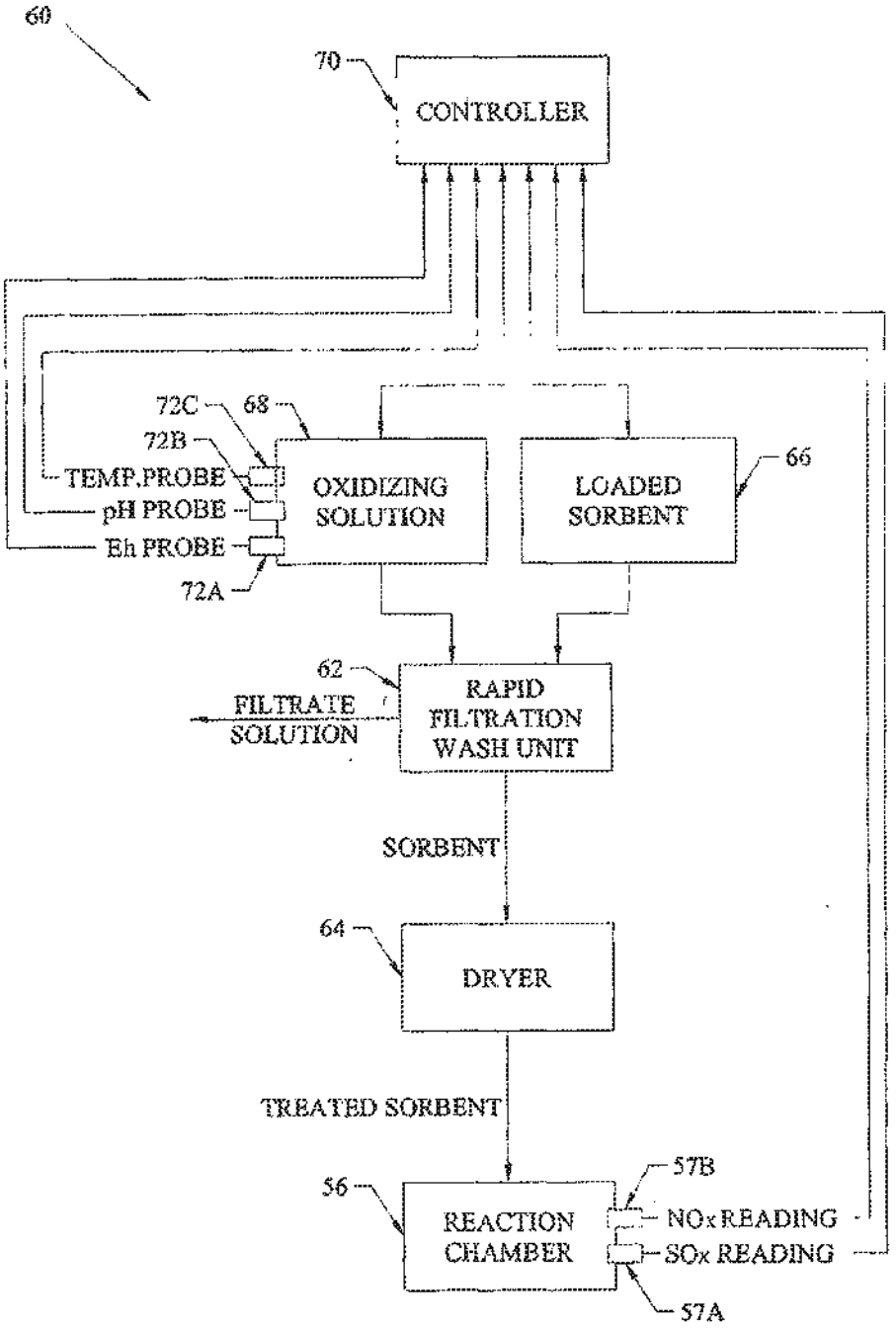
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FIGURE 7
TREATMENT OF VIRGIN OXIDES OF MANGANESE SORBENT



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FIGURE 8
TREATMENT OF LOADED OXIDES OF MANGANESE SORBENT



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Page 1513

FIGURE 9
POURBAIX DIAGRAM AT 25° C WITH $1x10^0$ DISSOLVED MANGANESE CONCENTRATOIN

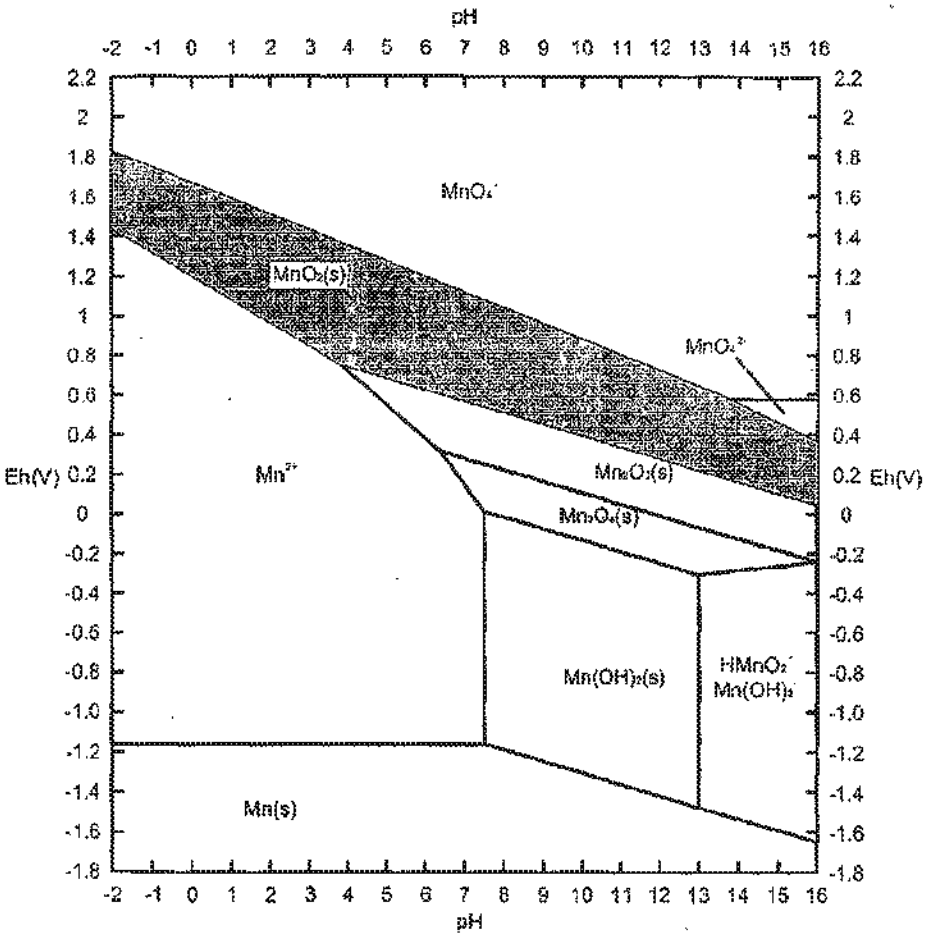
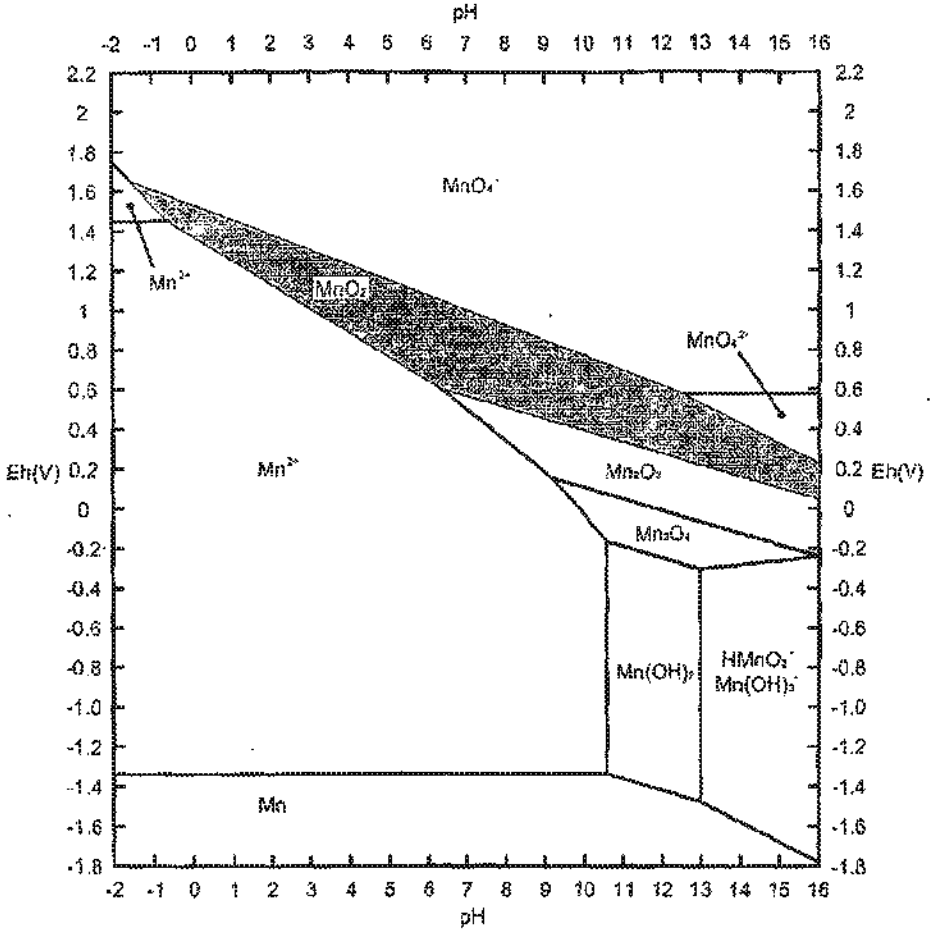


FIGURE 10
POURBAIX DIAGRAM AT 25° C WITH $1x10^{-6}$ DISSOLVED MANGANESE CONCENTRATION



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INTERNATIONAL SEARCH REPORT

International Application No
PCT/US 02/41276

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 B01J20/06 B01J20/34 B01J23/34

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 B01J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 552 734 A (IANNICELLI JOSEPH ET AL) 12 November 1985 (1985-11-12) column 4, line 7 -column 9, line 65; claims; figures; examples	1-34
X	US 5 366 710 A (CHOU CHARLES C ET AL) 22 November 1994 (1994-11-22) column 2, line 53 -column 5, line 68; claims; figures; examples; tables	1-34
X	US 4 164 545 A (SCOTT RICHARD L) 14 August 1979 (1979-08-14) column 1, line 8 -column 4, line 39; claims; figures; examples; tables	1-34

-/--

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
13 June 2003	27/06/2003

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	de Cauwer, R

Form PCT/ISA/210 (second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

International Application No
PCT/US 02/41276

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication,where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 923 688 A (IANNICELLI JOSEPH) 8 May 1990 (1990-05-08) column 2, line 67 -column 8, line 45; claims; figures; examples	1-34
P,X	WO 02 28513 A (ENVIROSCRUB TECHNOLOGIES CORP) 11 April 2002 (2002-04-11) the whole document	1-34
P,X	WO 02 09852 A (ENVIROSCRUB TECHNOLOGIES CORP ;PAHLMAN KATHLEEN S HF (US); AXEN ST) 7 February 2002 (2002-02-07) the whole document	1-34

Form PCT/ISA/210 (continuation of second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US 02/41276

Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This International Search Report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☒ Claims Nos.:
because they relate to parts of the International Application that do not comply with the prescribed requirements to such an extent that no meaningful International Search can be carried out, specifically:
see FURTHER INFORMATION sheet PCT/ISA/210

3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this International Search Report covers all searchable claims.

2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this International Search Report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this International Search Report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

☐ The additional search fees were accompanied by the applicant's protest.

☐ No protest accompanied the payment of additional search fees.

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

Continuation of Box I.2

In view of the large number and also the wording of the claims presently on file, which render it difficult, if not impossible, to determine the matter for which protection is sought, the present application fails to comply with the clarity and conciseness requirements of Article 6 PCT (see also Rule 6.1(a) PCT) to such an extent that a meaningful search is impossible. Consequently, the search has been carried out for those parts of the application which do appear to be clear (and concise), namely a process for the (pre)treatment of oxides of manganese, a system therefore and the products thereof as described in the lab-scale test on page 25 to 29.

The applicant's attention is drawn to the fact that claims, or parts of claims, relating to inventions in respect of which no international search report has been established need not be the subject of an international preliminary examination (Rule 66.1(e) PCT). The applicant is advised that the EPO policy when acting as an International Preliminary Examining Authority is normally not to carry out a preliminary examination on matter which has not been searched. This is the case irrespective of whether or not the claims are amended following receipt of the search report or during any Chapter II procedure.

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/US 02/41276

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 4552734 A	12-11-1985	CA 1216732 A1	20-01-1987
		DK 410584 A	09-03-1985
		GB 2149389 A ,B	12-06-1985
US 5366710 A	22-11-1994	NONE	
US 4164545 A	14-08-1979	NONE	
US 4923688 A	08-05-1990	NONE	
WO 0228513 A	11-04-2002	AU 9080201 A	15-04-2002
		AU 9622101 A	13-02-2002
		EP 1315546 A2	04-06-2003
		WO 0209852 A2	07-02-2002
		WO 0228513 A2	11-04-2002
		US 2002150516 A1	17-10-2002
		US 2002168302 A1	14-11-2002
		AU 9077601 A	15-04-2002
WO 0209852 A	07-02-2002	AU 9622101 A	13-02-2002
		EP 1315546 A2	04-06-2003
		WO 0209852 A2	07-02-2002
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		US 2002168302 A1	14-11-2002
		AU 9080201 A	15-04-2002
		WO 0228513 A2	11-04-2002
		AU 9077601 A	15-04-2002

In Column 5, Line 51, delete "an" and insert --art-- therefor

In Column 10, Line 36, delete "131*h* and 131*c*." and insert --131*b* and 131*c*.-- therefor

In Column 13, Line 34, after "eliminated", insert --.--

In Column 13, Line 43, after "system", insert --.--

In Column 14, Line 20, after "No", insert --.--

In Column 15, Line 7, delete "FGD." and insert --FGD,-- therefor

In Column 15, Line 7, delete "Marshall." and insert --Marshall,-- therefor

In Column 15, Line 49, delete "Miller." and insert --Miller,-- therefor

In Column 16, Line 12, after "sorbent", insert --.--

In Column 20, Lines 65-66, delete "73%-69%°" and insert --73%-69%-- therefor

In Column 20, Line 66, delete "86%-84° %" and insert --86%-84%-- therefor

In Column 34, Line 3, delete "Ib/Macfgave" and insert --Ib/Macf gave-- therefor

In Column 34, Line 19, delete "I993)" and insert --1993)-- therefor

UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
15/951,970	04/12/2018	Edwin S. Olson	4820.001US8	6410

21186 7590 12/14/2021
SCHWEGMAN LUNDBERG & WOESSNER, P.A.
P.O. BOX 2938
MINNEAPOLIS, MN 55402

EXAMINER	
LAWRENCE JR, FRANK M	

ART UNIT	PAPER NUMBER
1776	

NOTIFICATION DATE	DELIVERY MODE
12/14/2021	ELECTRONIC

Please find below and/or attached an Office communication concerning this application or proceeding.

The time period for reply, if any, is set in the attached communication.

Notice of the Office communication was sent electronically on above-indicated "Notification Date" to the following e-mail address(es):

SLW@blackhillsip.com
uspto@slwip.com

Response to the Request for Certificate of Correction	Patent No. 10933370	Applicant(s) Olson et al.
	Issue Date 03/02/2021	Docket No. 4820.001US8

This is in response to the request for a Certificate of Correction filed 03 November 2021

☑ **Request Approval- In- Part** - Consideration has been given to your request for the issuance of a Certificate of Correction under the provisions of 37 CFR 1.322 and/or 37 CFR 1.323. The Request is improper and approved in part for the reason(s) below:

☐ **Supersede/Vacate** - The Certificate of Correction issued in error, in that error(s) was made in identifying the patent number and/or keying text/corrections. See Other Comments below:

1. ☑ Assignees' names and addresses (assignment data) printed in a patent, are based solely on information supplied in the appropriate space for identifying the assignment data on the Issue Fee Transmittal Form (PTOL-85b). Any request for a patent to be corrected to state the name of the assignee, must state that the assignment was submitted for recordation as set forth in in 37 CFR 3.11 before issuance of the patent. Petition under 3.81 is to be filed for consideration of correction to assignee. The petition fee set forth in 37 CFR 1.17(i)(1) (currently $140, $70, $35 for large, small and micro entities, respectively.

2. ☐ The alleged error in _____, is in fact an Amendment and/or Change made by the examiner and considered to be in accordance with the permissible amendments enumerated in the Manual of Patent Examining Procedure (MPEP) Section 1302.04. Applicant did not file objection or amendment under 37 CFR 1.312 prior to payment of the issue fee.

3. ☐ A petition under CFR 1.182 is required to correct the alleged errors in spelling or order of inventor's names, since inventor's names are printed solely in accordance with the type-written names, and in the order of the type-written names on the Application Data Sheet (ADS). The required fee currently under rule 1.17(f) (small entity $200, large entity $400, micro entity fee $100).

4. ☐ With respect to the alleged error in changing the inventor name on the patent due to clerical error in ADS/OATH of related patents. The inventors name is printed in accordance with the OATH/ADS submitted at the time of filing the application. However, your attention is directed to C.F.R. 1.324, wherein a request is being made to change, add or delete inventor(s), after issuance of the patent.

5. ☐ With respect to the alleged error in _____, comparison of the printed patent with the corresponding location in the application file reveals that there is no discrepancy.

6. ☐ With respect to 37 CFR 1.72, the title should be brief but technically accurate and descriptive and should contain fewer than 500 characters. Inasmuch as the words new, improved, improvement of, and improvement in are not considered as part of the title of an invention, these words should not be included at the beginning of the title of the invention and will be deleted when the Office enters the title into the Offices computer records, and when any patent issues.

7. ☐ The fee for correction under 37 CFR 1.323 is set forth in 37 CFR 1.20(a). ☐Partial fee ☐No fee was received with your request. Full fee payment is required before further action is taken on this request.

8. ☐ With respect to the request for corrected Letters Patent (Grant), corrections to the original Letters Patent are made under the provisions of Rule 1.322(b), not Rule1.323, unless a petition is granted.

9. ☑ Other Comments: See Continuation Sheet

Further correspondence concerning this matter should be filed and directed to the Certificates of Correction Branch.

Legal Instrument Examiner: /YOLANDA A MIDDLETON/ LIE, ODM

Phone: (703)756-1282

Certificates of Correction Branch email:CustomerServiceCoC@uspto.gov

CoC Central Phone Number: (703)756-1814

If applicable, information regarding a petition under 37 CFR 1.183 should be directed to the attention of the Commissioner for Patents using the FAX number (571) 273-8300

Continuation of Other Comments: A certificate of correction will be issued to correct the remaining errors noted in your request.

IN UNITED STATES PATENT AND TRADEMARK OFFICE

Patent No.:	10,933,370 B2	Docket No:	4820.001US8
Issue Date:	Mar. 2, 2021	Patentee:	Midwest Energy Emissions Corp.
Customer No.:	21186	Confirmation No.:	6410
Title	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY		

REQUEST FOR CERTIFICATE OF CORRECTION

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450
ATTN: CERTIFICATE OF CORRECTION BRANCH

It is requested that a Certificate of Correction be issued correcting printing errors appearing in the above-identified United States patent. A copy of the text of the Certificate in the suggested form is enclosed.

Authorization is given to charge Deposit Account No. 19-0743 in the amount of $160.00 to cover the Certificate of Correction Fee.

Issuance of the Certificate of Correction would neither expand nor contract the scope of the claims as properly allowed, and re-examination is not required.

The Examiner is authorized to charge any additional fees or credit overpayment to Deposit Account No. 19-0743.

Respectfully Submitted,

SCHWEGMAN, LUNDBERG & WOESSNER, P.A
P.O. Box 2938
Minneapolis, MN 55402
(612) 349-9580

Date : November 3, 2021

By /Nicholas P. Lanzatella/
Nicholas P. Lanzatella
Reg. No: 63,803

PTO/SB/44 (02-01)
Approved for use through 01/31/2004. OMB 0651-0033
U.S. Patent and Trademark Office: U.S. DEPARTMENT OF COMMERCE
Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.
(Also Form PTO-1050)

UNITED STATES PATENT AND TRADEMARK OFFICE
CERTIFICATE OF CORRECTION

PATENT NO : 10,933,370 B2

DATED : Mar. 2, 2021

INVENTOR(S) : Olson et al.

It is certified that error appears in the above-identified patent and that said Letters Patent is hereby corrected as shown below:

On the face page, in field (73), in "Assignee", in column 1, line 1, delete "Corp." and insert -- Corp.,-- therefor

On the face page, in "Related U.S. Application Data", in column 1, line 1, delete "(60)" and insert --(63)-- therefor

On page 5, in column 2, under "Other Publications", line 21, delete "MaY" and insert --May-- therefor

On page 6, in column 2, under "Other Publications", line 59, delete "Actioin" and insert --Action-- therefor

On page 7, in column 1, under "Other Publications", line 67, delete "ay" and insert --May.-- therefor

On page 8, in column 2, under "Other Publications", line 71, delete ""Effectsof" and insert --"Effects of-- therefor

On page 8, in column 2, under "Other Publications", line 72, delete "Reducton" and insert -- Reduction-- therefor

On page 9, in column 2, under "Other Publications", line 5, delete "Removil" and insert --Removal-- therefor

On page 10, in column 2, under "Other Publications", line 62, delete "SHeet"" and insert --Sheet"-- therefor

On page 10, in column 2, under "Other Publications", line 64, delete ""Fluegas" and insert --"Flue gas-- therefor

In column 3, line 13, delete "halides." and insert --halides,-- therefor

MAILING ADDRESS OF SENDER:

SCHWEGMAN, LUNDBERG & WOESSNER, P.A.
P.O. BOX 2938
Minneapolis, MN 55402

Atty Docket No: 4820.001US8

PATENT NO. 10,933,370

No. of additional copies

PTO/SB/44 (02-01)
Approved for use through 01/31/2004. OMB 0651-0033
U.S. Patent and Trademark Office: U.S. DEPARTMENT OF COMMERCE
Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.
(Also Form PTO-1050)

UNITED STATES PATENT AND TRADEMARK OFFICE
CERTIFICATE OF CORRECTION

PATENT NO : 10,933,370 B2

DATED : Mar. 2, 2021

INVENTOR(S) : Olson et al.

It is certified that error appears in the above-identified patent and that said Letters Patent is hereby corrected as shown below:

In column 5, line 51, delete "an" and insert --art-- therefor

In column 10, line 36, delete "131*h* and 131*c*." and insert --131*b* and 131*c*.-- therefor

In column 13, line 34, after "eliminated", insert --.--

In column 13, line 43, after "system", insert --.--

In column 14, line 20, after "No", insert --.--

In column 15, line 7, delete "FGD." and insert --FGD,-- therefor

In column 15, line 7, delete "Marshall." and insert --Marshall,-- therefor

In column 15, line 49, delete "Miller." and insert --Miller,-- therefor

In column 16, line 12, after "sorbent", insert --.--

In column 20, lines 65-66, delete "73%-69%°" and insert --73%-69%-- therefor

In column 20, line 66, delete "86%-84° %" and insert --86%-84%-- therefor

In column 34, line 3, delete "lb/Macfgave" and insert --lb/Macf gave-- therefor

In column 34, line 19, delete "l993)" and insert --1993)-- therefor

MAILING ADDRESS OF SENDER:

SCHWEGMAN, LUNDBERG & WOESSNER, P.A.
P.O. BOX 2938
Minneapolis, MN 55402

Atty Docket No: 4820.001US8

PATENT NO. 10,933,370

No. of additional copies

Electronic Patent Application Fee Transmittal

Application Number:	15951970
Filing Date:	12-Apr-2018
Title of Invention:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY
First Named Inventor/Applicant Name:	Edwin S. Olson
Filer:	Susan Yee/Janine Heitland
Attorney Docket Number:	4820.001US8

Filed as Large Entity

Filing Fees for Utility under 35 USC 111(a)

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Basic Filing:				
Pages:				
Claims:				
Miscellaneous-Filing:				
Petition:				
Patent-Appeals-and-Interference:				
Post-Allowance-and-Post-Issuance:				
CERTIFICATE OF CORRECTION	1811	1	160	160

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Extension-of-Time:				
Miscellaneous:				
			Total in USD ($)	**160**

Electronic Acknowledgement Receipt

EFS ID:	44193051
Application Number:	15951970
International Application Number:	
Confirmation Number:	6410
Title of Invention:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY
First Named Inventor/Applicant Name:	Edwin S. Olson
Customer Number:	21186
Filer:	Susan Yee/Janine Heitland
Filer Authorized By:	Susan Yee
Attorney Docket Number:	4820.001US8
Receipt Date:	03-NOV-2021
Filing Date:	12-APR-2018
Time Stamp:	13:27:31
Application Type:	Utility under 35 USC 111(a)

Payment information:

Submitted with Payment	yes
Payment Type	DA
Payment was successfully received in RAM	$160
RAM confirmation Number	E2021A3D28053039
Deposit Account	190743
Authorized User	Janine Heitland

The Director of the USPTO is hereby authorized to charge indicated fees and credit any overpayment as follows:

37 CFR 1.20 (Post Issuance fees)

File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1		4820001US8COCREQUEST.pdf	184786 9b4fdee44c2bf3d82c92e6ccce063893e615c33c	yes	4

	Multipart Description/PDF files in .zip description		
	Document Description	**Start**	**End**
	Transmittal Letter	1	1
	Request for Certificate of Correction	2	4

Warnings:

Information:

2	Fee Worksheet (SB06)	fee-info.pdf	37939 6ec7ad088641a18c66759af3f6eec7b2f0aaebe4	no	2

Warnings:

Information:

Total Files Size (in bytes):	222725

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Midwest Energy Emissions Corp.

Examiner: Frank M. Lawrence, Jr.

Patent No.: 10,933,370 B2

Group Art Unit: 1776

Issue Date: Mar. 2, 2021

Docket No: 4820.001US8

Title: SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450
ATTN: CERTIFICATE OF CORRECTION BRANCH

We are transmitting herewith the attached:

X Authorization to charge Deposit Account No. 19-0743 in the amount of $160.00 to cover the Certificate of Correction Fee.
X Request for Certificate of Correction. (1 pg.)
X Certificate of Correction Form - PTO-1050 (2 pgs.)

Please charge any additional fees or credit overpayment to Deposit Account No. 19-0743.

SCHWEGMAN, LUNDBERG & WOESSNER, P.A.
Customer No: 21186

By: /Nicholas P. Lanzatella/
Nicholas P. Lanzatella
Reg. No: 63,803

CERTIFICATE UNDER 37 CFR § 1.8: The undersigned hereby certifies that this correspondence is filed using the USPTO's electronic filing system EFS-Web, and is addressed to: Commissioner for Patents, P.O. Box 1450 Alexandria, VA 22313-1450, on this 3rd day of November 2021.

Janine Heitland
Name

/Janine Heitland/
Signature

UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

APPLICATION NO.	ISSUE DATE	PATENT NO.	ATTORNEY DOCKET NO.	CONFIRMATION NO.
15/951,970	03/02/2021	10933370	4820.001US8	6410

21186 7590 02/10/2021

SCHWEGMAN LUNDBERG & WOESSNER, P.A.
P.O. BOX 2938
MINNEAPOLIS, MN 55402

ISSUE NOTIFICATION

The projected patent number and issue date are specified above.

Determination of Patent Term Adjustment under 35 U.S.C. 154 (b)
(application filed on or after May 29, 2000)

The Patent Term Adjustment is 245 day(s). Any patent to issue from the above-identified application will include an indication of the adjustment on the front page.

If a Continued Prosecution Application (CPA) was filed in the above-identified application, the filing date that determines Patent Term Adjustment is the filing date of the most recent CPA.

Applicant will be able to obtain more detailed information by accessing the Patent Application Information Retrieval (PAIR) WEB site (http://pair.uspto.gov).

Any questions regarding the Patent Term Extension or Adjustment determination should be directed to the Office of Patent Legal Administration at (571)-272-7702. Questions relating to issue and publication fee payments should be directed to the Application Assistance Unit (AAU) of the Office of Data Management (ODM) at (571)-272-4200.

APPLICANT(s) (Please see PAIR WEB site http://pair.uspto.gov for additional applicants):

Edwin S. Olson, Grand Forks, ND;
Midwest Energy Emissions Corp., Lewis Center, OH;
Michael J. Holmes, Thompson, ND;
John H. Pavlish, East Grand Forks, MN;

The United States represents the largest, most dynamic marketplace in the world and is an unparalleled location for business investment, innovation, and commercialization of new technologies. The USA offers tremendous resources and advantages for those who invest and manufacture goods here. Through SelectUSA, our nation works to encourage and facilitate business investment. To learn more about why the USA is the best country in the world to develop technology, manufacture products, and grow your business, visit SelectUSA.gov.

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Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
15/951,970	04/12/2018	Edwin S. Olson	4820.001US8	6410

21186 7590 01/28/2021
SCHWEGMAN LUNDBERG & WOESSNER, P.A.
P.O. BOX 2938
MINNEAPOLIS, MN 55402

EXAMINER	
LAWRENCE JR, FRANK M	

ART UNIT	PAPER NUMBER
1776	

NOTIFICATION DATE	DELIVERY MODE
01/28/2021	ELECTRONIC

Please find below and/or attached an Office communication concerning this application or proceeding.

The time period for reply, if any, is set in the attached communication.

Notice of the Office communication was sent electronically on above-indicated "Notification Date" to the following e-mail address(es):

SLW@blackhillsip.com
uspto@slwip.com

Corrected ***Notice of Allowability***	**Application No.** 15/951,970	**Applicant(s)** Olson et al.	
	Examiner FRANK LAWRENCE	**Art Unit** 1776	**AIA (FITF) Status** No

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address--

All claims being allowable, PROSECUTION ON THE MERITS IS (OR REMAINS) CLOSED in this application. If not included herewith (or previously mailed), a Notice of Allowance (PTOL-85) or other appropriate communication will be mailed in due course. **THIS NOTICE OF ALLOWABILITY IS NOT A GRANT OF PATENT RIGHTS.** This application is subject to withdrawal from issue at the initiative of the Office or upon petition by the applicant. See 37 CFR 1.313 and MPEP 1308.

1. ☑ This communication is responsive to the amendment filed November 25, 2020.
   ☐ A declaration(s)/affidavit(s) under **37 CFR 1.130(b)** was/were filed on _____.

2. ☐ An election was made by the applicant in response to a restriction requirement set forth during the interview on _____; the restriction requirement and election have been incorporated into this action.

3. ☑ The allowed claim(s) is/are 1-4 and 8-21 . As a result of the allowed claim(s), you may be eligible to benefit from the **Patent Prosecution Highway** program at a participating intellectual property office for the corresponding application. For more information , please see **http://www.uspto.gov/patents/init_events/pph/index.jsp** or send an inquiry to **PPHfeedback@uspto.gov.**

4. ☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).

   Certified copies:

   a) ☐ All b) ☐ Some *c) ☐ None of the:

   1. ☐ Certified copies of the priority documents have been received.
   2. ☐ Certified copies of the priority documents have been received in Application No. _____ .
   3. ☐ Copies of the certified copies of the priority documents have been received in this national stage application from the International Bureau (PCT Rule 17.2(a)).

   * Certified copies not received: _____ .

Applicant has THREE MONTHS FROM THE "MAILING DATE" of this communication to file a reply complying with the requirements noted below. Failure to timely comply will result in ABANDONMENT of this application.
THIS THREE-MONTH PERIOD IS NOT EXTENDABLE.

5. ☐ CORRECTED DRAWINGS (as "replacement sheets") must be submitted.
   ☐ including changes required by the attached Examiner's Amendment / Comment or in the Office action of Paper No./Mail Date _____ .

   Identifying indicia such as the application number (see 37 CFR 1.84(c)) should be written on the drawings in the front (not the back) of each sheet. Replacement sheet(s) should be labeled as such in the header according to 37 CFR 1.121(d).

6. ☐ DEPOSIT OF and/or INFORMATION about the deposit of BIOLOGICAL MATERIAL must be submitted. Note the attached Examiner's comment regarding REQUIREMENT FOR THE DEPOSIT OF BIOLOGICAL MATERIAL.

Attachment(s)

1. ☐ Notice of References Cited (PTO-892)
2. ☑ Information Disclosure Statements (PTO/SB/08), Paper No./Mail Date _____.
3. ☐ Examiner's Comment Regarding Requirement for Deposit of Biological Material _____.
4. ☐ Interview Summary (PTO-413), Paper No./Mail Date. _____.
5. ☑ Examiner's Amendment/Comment
6. ☐ Examiner's Statement of Reasons for Allowance
7. ☐ Other _____.

/FRANK M LAWRENCE JR/
Primary Examiner, Art Unit 1776

Notice of Pre-AIA or AIA Status

1. The present application is being examined under the pre-AIA first to invent provisions.

Information Disclosure Statement

2. This notice is to reflect a corrected document number in the IDS submitted July 26, 2018.

3. Any inquiry concerning this communication or earlier communications from the examiner should be directed to FRANK LAWRENCE whose telephone number is (571)272-1161. The examiner can normally be reached on Mon-Fri 8:30am-7pm.

Examiner interviews are available via telephone, in-person, and video conferencing using a USPTO supplied web-based collaboration tool. To schedule an interview, applicant is encouraged to use the USPTO Automated Interview Request (AIR) at http://www.uspto.gov/interviewpractice.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Nam Nguyen can be reached on 571-272-1342. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see https://ppair-my.uspto.gov/pair/PrivatePair. Should you have questions on access to the Private

PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free).

If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

/FRANK M LAWRENCE JR/
Primary Examiner, Art Unit 1776

fl

Modified form PTO/SB/08A(04-07) OMB 651-0031
US Patent & Trademark Office: U.S. DEPARTMENT OF COMMERCE
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Substitute for form 1449A/PTO

INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Use as many sheets as necessary)

Complete if Known	
Application Number	15/951,970
Filing Date	April 12, 2018
First Named Inventor	Edwin S. Olson
Group Art Unit	1736
Examiner Name	Unknown

Sheet 1 of 19

Attorney Docket No: 4820.001US8

US PATENT DOCUMENTS

Examiner Initial *	USP Document Number	Publication Date	Name of Patentee or Applicant of cited Document
	US-1,984,164	12/11/1934	Alfred, Stock
	US-2,317,857	4/27/1943	Soday, Frank J
	US-2,456,272A1	2/7/2002	Pahlman, John, et al.
	US-3,194,629	7/13/1965	Dreibelbis, John A, et al.
	US-3,341,185	9/12/1967	Kennedy, Sr, Walter L
	US-3,662,523	5/16/1972	Revoir, William H, et al.
	US-3,786,619	1/22/1974	Melkersson, Karl-axel
	US-3,826,618	7/30/1974	Capuano, Italo A
	US-3,849,267	11/19/1974	Hilgen, Hendrika, et al.
	US-3,876,393	4/8/1975	Kasai, Juichi, et al.
	US-3,935,708	2/3/1976	Harrewijne, A., et al.
	US-4,094,777	6/13/1978	Sugier, Andre, et al.
	US-4,101,631	7/18/1978	Ambrosini, Ronald Frank, et al.
	US-4,196,173	4/1/1980	DeJong, Geert J, et al.
	US-4,338,896	7/13/1982	Papasideris, Stamos I.
	US-4,342,192	8/3/1982	Heyn, K., et al.
	US-4,500,327	2/19/1985	Nishino, Hirosh, et al.
	US-4,530,765	7/23/1985	Sabherwal, Inderjit H
	US-4,708,853	11/24/1987	Matviya, Thomas, et al.
	US-4,772,455	9/20/1988	Izumi, Kazuo, et al.
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INFORMATION DISCLOSURE STATEMENT BY APPLICANT

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Complete if Known	
Application Number	15/951,970
Filing Date	April 12, 2018
First Named Inventor	Edwin S. Olson
Group Art Unit	1736
Examiner Name	Unknown

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	Group Art Unit	1736
	Examiner Name	Unknown
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	Examiner Name	Unknown
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	First Named Inventor	Edwin S. Olson
	Group Art Unit	1736
	Examiner Name	Unknown
Sheet 5 of 19	Attorney Docket No: 4820.001US8	

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Sheet 6 of 19	Attorney Docket No: 4820.001US8	

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	Application Number	15/951,970
	Filing Date	April 12, 2018
	First Named Inventor	Edwin S. Olson
	Group Art Unit	1736
	Examiner Name	Unknown
Sheet 8 of 19	Attorney Docket No: 4820.001US8	

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	"Application Serial No. 12/184,860, Response filed 04-07-11 to Non Final Office Action mailed 01-19-11", 12 pgs	
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	"European Application Serial No. 10767465.7, Intention to Grant mailed 03-17-14", 44 pgs	
	"European Application Serial No. 10767465.7, Intention to Grant mailed 09-30-13", 44 pgs	
	"European Application Serial No. 10767465.7, Response filed 02-03-14 to Intention to Grant mailed 09-30-13", 11 pgs	
	"European Application Serial No. 10767465.7, Response filed 06-20-13 to Extended European Search Report mailed 12-06-12", 18 pgs	
	"European Application Serial No. 11189249.3, Communication Pursuant to Article 94(3) EPC mailed 11-26-12", 4 pgs	
	"European Application Serial No. 11189249.3, Decision to Refuse mailed 12-19-13", 18 pgs	
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	"European Application Serial No. 11189249.3, Office Action mailed 11-04-13", 4 pgs	
	"European Application Serial No. 11189249.3, Response filed 02-04-13 to Communication Pursuant to Article 94(3) EPC mailed 11-26-12", 9 pgs	
	"European Application Serial No. 11189249.3, Response filed 08-28-12 to Extended European Search Report mailed 02-01-12", 9 pgs	
	"European Application Serial No. 11189249.3, Summons to Attend Oral Proceedings mailed 04-15-13", 7 pgs	

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Substitute for form 1449A/PTO **INFORMATION DISCLOSURE STATEMENT BY APPLICANT** (Use as many sheets as necessary)	*Complete if Known*	
	Application Number	15/951,970
	Filing Date	April 12, 2018
	First Named Inventor	Edwin S. Olson
	Group Art Unit	1736
	Examiner Name	Unknown
Sheet 15 of 19	Attorney Docket No: 4820.001US8	

OTHER DOCUMENTS – NON PATENT LITERATURE DOCUMENTS

Examiner Initial *	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	T1
	"European Application Serial No. 11189252.7, Communication Pursuant to Article 94(3) EPC mailed 01-09-13", 4 pgs	
	"European Application Serial No. 11189252.7, Decision to grant mailed 05-02-14", 2 pgs	
	"European Application Serial No. 11189252.7, Extended European Search Report mailed 01-23-12", 6 pgs	
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	"European Application Serial No. 11189252.7, Response filed 02-11-13 to Communication Pursuant to Article 94(3) EPC mailed 01-09-13", 5 pgs	
	"European Application Serial No. 11189252.7, Response filed 10-10-13 to Office Action mailed 04-15-13", 18 pgs	
	"European Application Serial No. 11189252.7, Response filed 11-22-12 to Extended European Search Report mailed 05-09-12", 13 pgs	
	"European Application Serial No. 11189252.7, Summons to Attend Oral Proceedings mailed 04-15-13", 6 pgs	
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	"European Application Serial No. 13719338.9, Office Action mailed 12-19-14", 2 pgs.	
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Sheet 18 of 19	Attorney Docket No: 4820.001US8	

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s):	Edwin S. Olson et al.	Examiner:	HEIDI RIVIERE KELLEY
Serial No.:	15/951,970	Group Art Unit:	1776
Filed:	April 12, 2018	Docket No.:	4820.001US8
Customer No.:	21186	Confirmation No.:	6410
Title:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY		

COMMUNICATION RE: FEE ADDRESS

Mail Stop Issue Fee
Commissioner for Patents
P.O.Box 1450
Alexandria, VA 22313-1450

In response to the Notice of Allowance and Issue Fee Due, please record the Fee Address under the provisions of 37 CFR 1.363 as the following:

Customer Number 21186

Please direct any inquiries to the undersigned attorney at (612) 349-9580.

Respectfully submitted,

SCHWEGMAN LUNDBERG & WOESSNER, P.A.
P.O. Box 2938
Minneapolis, MN 55402
(612) 349-9580

Date January 14, 2021

By /Nicholas P. Lanzatella/
Nicholas P. Lanzatella
Reg. No. 63,803

NPL:CMG:jdh4

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Title:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY		

RESPONSE TO EXAMINER'S REASONS FOR ALLOWANCE

MS Issue Fee
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

In the Notice of Allowance dated January 12, 2021 the Examiner has set forth "Reasons for Allowance." Applicant agrees that the claims are patentable over the references of record, but does not necessarily agree with the reasons provided in the allowance, which are the Examiner's reasons rather than Applicant's and are in no way to be interpreted as admitted by Applicant. Applicant respectfully submits that the subject matter of each of the allowed claims is patentable for its respective recitation of the claimed combination as a whole. That is, the patentability of each claim rests on the combination of recited elements and limitations. As such, Applicant submits that no one element or limitation in particular should be deemed to impart or be required for patentability of the claims. Furthermore, Applicant submits that the dependent claims are allowable not only for their dependence from the allowed independent claims, but also for the additional subject matter recited in each of those dependent claims.

If necessary, please charge any additional fees or credit overpayment to Deposit Account 19-0743.

Respectfully submitted,

SCHWEGMAN LUNDBERG & WOESSNER, P.A.
P.O. Box 2938
Minneapolis, MN 55402
(612) 349-9580

Date January 14, 2021 By /Nicholas P. Lanzatella/
Nicholas P. Lanzatella
Reg. No. 63,803

Electronic Patent Application Fee Transmittal

Application Number:	15951970
Filing Date:	12-Apr-2018
Title of Invention:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY
First Named Inventor/Applicant Name:	Edwin S. Olson
Filer:	Susan Yee/Janine Heitland
Attorney Docket Number:	4820.001US8

Filed as Large Entity

Filing Fees for Utility under 35 USC 111(a)

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Basic Filing:				
Pages:				
Claims:				
Miscellaneous-Filing:				
Petition:				
Patent-Appeals-and-Interference:				
Post-Allowance-and-Post-Issuance:				
UTILITY APPL ISSUE FEE	1501	1	1200	1200

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Extension-of-Time:				
Miscellaneous:				
			Total in USD ($)	**1200**

Electronic Acknowledgement Receipt

EFS ID:	41651852
Application Number:	15951970
International Application Number:	
Confirmation Number:	6410
Title of Invention:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY
First Named Inventor/Applicant Name:	Edwin S. Olson
Customer Number:	21186
Filer:	Susan Yee/Janine Heitland
Filer Authorized By:	Susan Yee
Attorney Docket Number:	4820.001US8
Receipt Date:	14-JAN-2021
Filing Date:	12-APR-2018
Time Stamp:	17:58:26
Application Type:	Utility under 35 USC 111(a)

Payment information:

Submitted with Payment	yes
Payment Type	DA
Payment was successfully received in RAM	$1200
RAM confirmation Number	E20211DH58574623
Deposit Account	190743
Authorized User	Janine Heitland

The Director of the USPTO is hereby authorized to charge indicated fees and credit any overpayment as follows:

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37 CFR 1.17 (Patent application and reexamination processing fees)

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New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s): Edwin S. Olson et al.
Title: SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY

Docket No.:	4820.001US8	Serial No.:	15/951,970
Filed:	April 12, 2018	Due Date:	April 12, 2021
Examiner:	HEIDI RIVIERE KELLEY	Group Art Unit:	1776
Customer No.:	21186	Confirmation No.:	6410

Notice of Allowance Date: January 12, 2021

Mail Stop Issue Fee
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

We are transmitting herewith the following:

X Communication Concerning Prior or Copending Applications (1 pg.)
X Issue Fee Transmittal (Form PTOL-85) (1 pg.)
X Communication Re: Fee Address (1 pg.)
X Response to Examiner's Reasons for Allowance (1 pg.)
X Authorization to charge Deposit 19-0743 in the amount of $1,200.00 to cover the Large Entity Issue Fee Payment.

Please charge any additional required fees or credit overpayment to Deposit Account No. 19-0743.

SCHWEGMAN LUNDBERG & WOESSNER, P.A.
Customer No.: 21186
NPL:CMG:jdh4

By /Nicholas P. Lanzatella/
Nicholas P. Lanzatella
Reg. No. 63,803

CERTIFICATE UNDER 37 CFR 1.8: The undersigned hereby certifies that this correspondence is being filed using the USPTO's electronic filing system EFS-Web, and is addressed to: Mail Stop Issue Fee, Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450 on this 14th day of January, 2021.

Janine Heitland
Name

/Janine Heitland/
Signature

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s):	Edwin S. Olson et al.	Examiner:	HEIDI RIVIERE KELLEY
Serial No.:	15/951,970	Group Art Unit:	1776
Filed:	April 12, 2018	Docket:	4820.001US8
Customer No.:	21186	Confirmation No.:	6410
Title:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY		

COMMUNICATION CONCERNING PRIOR OR COPENDING APPLICATION(S)

Mail Stop Issue Fee
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Pursuant to the guidance of MPEP §§ 2001.06(b) and 2004(9), Applicant would like to bring the following additional application(s) to the Examiner's attention. The identification of these applications is not intended to suggest that the subject matter claimed in any listed application is, or has been, substantially similar to any claim or claims in the present application.

Serial No./ Patent No.	Filing Date	Attorney Docket	Title
17/125,641	December 17, 2020	4820.001U13	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY
16/993,361	August 14, 2020	4820.007US6	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY

Respectfully submitted,

SCHWEGMAN LUNDBERG & WOESSNER, P.A.
P.O. Box 2938
Minneapolis, MN 55402
(612) 349-9580

Date January 14, 2021 By /Nicholas P. Lanzatella/
Nicholas P. Lanzatella
Reg. No. 63,803

PART B - FEE(S) TRANSMITTAL

Complete and send this form, together with applicable fee(s), by mail or fax, or via EFS-Web.

By mail, send to: Mail Stop ISSUE FEE
Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

By fax, send to: (571)-273-2885

INSTRUCTIONS: This form should be used for transmitting the ISSUE FEE and PUBLICATION FEE (if required). Blocks 1 through 5 should be completed where appropriate. All further correspondence including the Patent, advance orders and notification of maintenance fees will be mailed to the current correspondence address as indicated unless corrected below or directed otherwise in Block 1, by (a) specifying a new correspondence address; and/or (b) indicating a separate "FEE ADDRESS" for maintenance fee notifications.

CURRENT CORRESPONDENCE ADDRESS (Note: Use Block 1 for any change of address)

21186 7590 01/12/2021
SCHWEGMAN LUNDBERG & WOESSNER, P.A.
P.O. BOX 2938
MINNEAPOLIS, MN 55402

Note: A certificate of mailing can only be used for domestic mailings of the Fee(s) Transmittal. This certificate cannot be used for any other accompanying papers. Each additional paper, such as an assignment or formal drawing, must have its own certificate of mailing or transmission.

Certificate of Mailing or Transmission

I hereby certify that this Fee(s) Transmittal is being deposited with the United States Postal Service with sufficient postage for first class mail in an envelope addressed to the Mail Stop ISSUE FEE address above, or being transmitted to the USPTO via EFS-Web or by facsimile to (571) 273-2885, on the date below.

Janine Heitland	(Typed or printed name)
/Janine Heitland/	(Signature)
January 14, 2021	(Date)

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
15/951,970	04/12/2018	Edwin S. Olson	4820.001US8	6410

TITLE OF INVENTION: SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY

APPLN. TYPE	ENTITY STATUS	ISSUE FEE DUE	PUBLICATION FEE DUE	PREV. PAID ISSUE FEE	TOTAL FEE(S) DUE	DATE DUE
nonprovisional	UNDISCOUNTED	$1200	$0.00	$0.00	$1200	04/12/2021

EXAMINER	ART UNIT	CLASS-SUBCLASS
LAWRENCE JR, FRANK M	1776	095-134000

1. Change of correspondence address or indication of "Fee Address" (37 CFR 1.363).

☐ Change of correspondence address (or Change of Correspondence Address form PTO/SB/122) attached.

☒ "Fee Address" indication (or "Fee Address" Indication form PTO/SB/47; Rev 03-09 or more recent) attached. **Use of a Customer Number is required.**

2. For printing on the patent front page, list
(1) The names of up to 3 registered patent attorneys or agents OR, alternatively,
(2) The name of a single firm (having as a member a registered attorney or agent) and the names of up to 2 registered patent attorneys or agents. If no name is listed, no name will be printed.

1 Schwegman Lundberg &
2 Woessner, P.A.
3

3. ASSIGNEE NAME AND RESIDENCE DATA TO BE PRINTED ON THE PATENT (print or type)

PLEASE NOTE: Unless an assignee is identified below, no assignee data will appear on the patent. If an assignee is identified below, the document must have been previously recorded, or filed for recordation, as set forth in 37 CFR 3.11 and 37 CFR 3.81(a). Completion of this form is NOT a substitute for filing an assignment.

(A) NAME OF ASSIGNEE: Midwest Energy Emissions Corp

(B) RESIDENCE: (CITY and STATE OR COUNTRY): Corsicana, Texas

Please check the appropriate assignee category or categories (will not be printed on the patent) : ☐ Individual ☒ Corporation or other private group entity ☐ Government

4a. Fees submitted: ☒ Issue Fee ☐ Publication Fee (if required) ☐ Advance Order - # of Copies

4b. Method of Payment: *(Please first reapply any previously paid fee shown above)*

☒ Electronic Payment via EFS-Web ☐ Enclosed check ☐ Non-electronic payment by credit card (Attach form PTO-2038)

☒ The Director is hereby authorized to charge the required fee(s), any deficiency, or credit any overpayment to Deposit Account No. 190743

5. **Change in Entity Status** (from status indicated above)

☐ Applicant certifying micro entity status. See 37 CFR 1.29

☐ Applicant asserting small entity status. See 37 CFR 1.27

☐ Applicant changing to regular undiscounted fee status.

NOTE: Absent a valid certification of Micro Entity Status (see forms PTO/SB/15A and 15B), issue fee payment in the micro entity amount will not be accepted at the risk of application abandonment.
NOTE: If the application was previously under micro entity status, checking this box will be taken to be a notification of loss of entitlement to micro entity status.
NOTE: Checking this box will be taken to be a notification of loss of entitlement to small or micro entity status, as applicable.

NOTE: This form must be signed in accordance with 37 CFR 1.31 and 37 CFR 1.33. See 37 CFR 1.4 for signature requirements and certifications.

Authorized Signature: /Nicholas P. Lanzatella/ Date: January 14, 2021

Typed or printed name: Nicholas P. Lanzatella Registration No. 63,803

PTOL-85 Part B (08-18) Approved for use through 01/31/2020 OMB 0651-0033 U.S. Patent and Trademark Office: U.S. DEPARTMENT OF COMMERCE

PAC ET AL. EXHIBIT 1023
Page 1566

UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

NOTICE OF ALLOWANCE AND FEE(S) DUE

21186 7590 01/12/2021
SCHWEGMAN LUNDBERG & WOESSNER, P.A.
P.O. BOX 2938
MINNEAPOLIS, MN 55402

EXAMINER
LAWRENCE JR, FRANK M

ART UNIT	PAPER NUMBER
1776	

DATE MAILED: 01/12/2021

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
15/951,970	04/12/2018	Edwin S. Olson	4820.001US8	6410

TITLE OF INVENTION: SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY

APPLN. TYPE	ENTITY STATUS	ISSUE FEE DUE	PUBLICATION FEE DUE	PREV. PAID ISSUE FEE	TOTAL FEE(S) DUE	DATE DUE
nonprovisional	UNDISCOUNTED	$1200	$0.00	$0.00	$1200	04/12/2021

THE APPLICATION IDENTIFIED ABOVE HAS BEEN EXAMINED AND IS ALLOWED FOR ISSUANCE AS A PATENT. PROSECUTION ON THE MERITS IS CLOSED. THIS NOTICE OF ALLOWANCE IS NOT A GRANT OF PATENT RIGHTS. THIS APPLICATION IS SUBJECT TO WITHDRAWAL FROM ISSUE AT THE INITIATIVE OF THE OFFICE OR UPON PETITION BY THE APPLICANT. SEE 37 CFR 1.313 AND MPEP 1308.

THE ISSUE FEE AND PUBLICATION FEE (IF REQUIRED) MUST BE PAID WITHIN THREE MONTHS FROM THE MAILING DATE OF THIS NOTICE OR THIS APPLICATION SHALL BE REGARDED AS ABANDONED. THIS STATUTORY PERIOD CANNOT BE EXTENDED. SEE 35 U.S.C. 151. THE ISSUE FEE DUE INDICATED ABOVE DOES NOT REFLECT A CREDIT FOR ANY PREVIOUSLY PAID ISSUE FEE IN THIS APPLICATION. IF AN ISSUE FEE HAS PREVIOUSLY BEEN PAID IN THIS APPLICATION (AS SHOWN ABOVE), THE RETURN OF PART B OF THIS FORM WILL BE CONSIDERED A REQUEST TO REAPPLY THE PREVIOUSLY PAID ISSUE FEE TOWARD THE ISSUE FEE NOW DUE.

HOW TO REPLY TO THIS NOTICE:

I. Review the ENTITY STATUS shown above. If the ENTITY STATUS is shown as SMALL or MICRO, verify whether entitlement to that entity status still applies.

If the ENTITY STATUS is the same as shown above, pay the TOTAL FEE(S) DUE shown above.

If the ENTITY STATUS is changed from that shown above, on PART B - FEE(S) TRANSMITTAL, complete section number 5 titled "Change in Entity Status (from status indicated above)".

For purposes of this notice, small entity fees are 1/2 the amount of undiscounted fees, and micro entity fees are 1/2 the amount of small entity fees.

II. PART B - FEE(S) TRANSMITTAL, or its equivalent, must be completed and returned to the United States Patent and Trademark Office (USPTO) with your ISSUE FEE and PUBLICATION FEE (if required). If you are charging the fee(s) to your deposit account, section "4b" of Part B - Fee(s) Transmittal should be completed and an extra copy of the form should be submitted. If an equivalent of Part B is filed, a request to reapply a previously paid issue fee must be clearly made, and delays in processing may occur due to the difficulty in recognizing the paper as an equivalent of Part B.

III. All communications regarding this application must give the application number. Please direct all communications prior to issuance to Mail Stop ISSUE FEE unless advised to the contrary.

IMPORTANT REMINDER: Maintenance fees are due in utility patents issuing on applications filed on or after Dec. 12, 1980. It is patentee's responsibility to ensure timely payment of maintenance fees when due. More information is available at www.uspto.gov/PatentMaintenanceFees.

PART B - FEE(S) TRANSMITTAL

Complete and send this form, together with applicable fee(s), by mail or fax, or via EFS-Web.

By mail, send to: Mail Stop ISSUE FEE
Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

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CURRENT CORRESPONDENCE ADDRESS (Note: Use Block 1 for any change of address)

21186 7590 01/12/2021
SCHWEGMAN LUNDBERG & WOESSNER, P.A.
P.O. BOX 2938
MINNEAPOLIS, MN 55402

Note: A certificate of mailing can only be used for domestic mailings of the Fee(s) Transmittal. This certificate cannot be used for any other accompanying papers. Each additional paper, such as an assignment or formal drawing, must have its own certificate of mailing or transmission.

Certificate of Mailing or Transmission

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(Typed or printed name)

(Signature)

(Date)

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
15/951,970	04/12/2018	Edwin S. Olson	4820.001US8	6410

TITLE OF INVENTION: SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY

APPLN. TYPE	ENTITY STATUS	ISSUE FEE DUE	PUBLICATION FEE DUE	PREV. PAID ISSUE FEE	TOTAL FEE(S) DUE	DATE DUE
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EXAMINER	ART UNIT	CLASS-SUBCLASS
LAWRENCE JR, FRANK M	1776	095-134000

1. Change of correspondence address or indication of "Fee Address" (37 CFR 1.363).

❑ Change of correspondence address (or Change of Correspondence Address form PTO/SB/122) attached.

❑ "Fee Address" indication (or "Fee Address" Indication form PTO/SB/47; Rev 03-09 or more recent) attached. **Use of a Customer Number is required.**

2. For printing on the patent front page, list
(1) The names of up to 3 registered patent attorneys or agents OR, alternatively,
(2) The name of a single firm (having as a member a registered attorney or agent) and the names of up to 2 registered patent attorneys or agents. If no name is listed, no name will be printed.

1 _____
2 _____
3 _____

3. ASSIGNEE NAME AND RESIDENCE DATA TO BE PRINTED ON THE PATENT (print or type)

PLEASE NOTE: Unless an assignee is identified below, no assignee data will appear on the patent. If an assignee is identified below, the document must have been previously recorded, or filed for recordation, as set forth in 37 CFR 3.11 and 37 CFR 3.81(a). Completion of this form is NOT a substitute for filing an assignment.

(A) NAME OF ASSIGNEE (B) RESIDENCE: (CITY and STATE OR COUNTRY)

Please check the appropriate assignee category or categories (will not be printed on the patent) : ❑ Individual ❑ Corporation or other private group entity ❑ Government

4a. Fees submitted: ❑ Issue Fee ❑ Publication Fee (if required) ❑ Advance Order - # of Copies _____

4b. Method of Payment: *(Please first reapply any previously paid fee shown above)*

❑ Electronic Payment via EFS-Web ❑ Enclosed check ❑ Non-electronic payment by credit card (Attach form PTO-2038)

❑ The Director is hereby authorized to charge the required fee(s), any deficiency, or credit any overpayment to Deposit Account No. _____

5. **Change in Entity Status** (from status indicated above)

❑ Applicant certifying micro entity status. See 37 CFR 1.29

❑ Applicant asserting small entity status. See 37 CFR 1.27

❑ Applicant changing to regular undiscounted fee status.

NOTE: Absent a valid certification of Micro Entity Status (see forms PTO/SB/15A and 15B), issue fee payment in the micro entity amount will not be accepted at the risk of application abandonment.
NOTE: If the application was previously under micro entity status, checking this box will be taken to be a notification of loss of entitlement to micro entity status.
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NOTE: This form must be signed in accordance with 37 CFR 1.31 and 1.33. See 37 CFR 1.4 for signature requirements and certifications.

Authorized Signature _____ Date _____

Typed or printed name _____ Registration No. _____

UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
15/951,970	04/12/2018	Edwin S. Olson	4820.001US8	6410

21186 7590 01/12/2021

SCHWEGMAN LUNDBERG & WOESSNER, P.A.
P.O. BOX 2938
MINNEAPOLIS, MN 55402

EXAMINER	
LAWRENCE JR, FRANK M	
ART UNIT	**PAPER NUMBER**
1776	

DATE MAILED: 01/12/2021

Determination of Patent Term Adjustment under 35 U.S.C. 154 (b)
(Applications filed on or after May 29, 2000)

The Office has discontinued providing a Patent Term Adjustment (PTA) calculation with the Notice of Allowance.

Section 1(h)(2) of the AIA Technical Corrections Act amended 35 U.S.C. 154(b)(3)(B)(i) to eliminate the requirement that the Office provide a patent term adjustment determination with the notice of allowance. See Revisions to Patent Term Adjustment, 78 Fed. Reg. 19416, 19417 (Apr. 1, 2013). Therefore, the Office is no longer providing an initial patent term adjustment determination with the notice of allowance. The Office will continue to provide a patent term adjustment determination with the Issue Notification Letter that is mailed to applicant approximately three weeks prior to the issue date of the patent, and will include the patent term adjustment on the patent. Any request for reconsideration of the patent term adjustment determination (or reinstatement of patent term adjustment) should follow the process outlined in 37 CFR 1.705.

Any questions regarding the Patent Term Extension or Adjustment determination should be directed to the Office of Patent Legal Administration at (571)-272-7702. Questions relating to issue and publication fee payments should be directed to the Customer Service Center of the Office of Patent Publication at 1-(888)-786-0101 or (571)-272-4200.

OMB Clearance and PRA Burden Statement for PTOL-85 Part B

The Paperwork Reduction Act (PRA) of 1995 requires Federal agencies to obtain Office of Management and Budget approval before requesting most types of information from the public. When OMB approves an agency request to collect information from the public, OMB (i) provides a valid OMB Control Number and expiration date for the agency to display on the instrument that will be used to collect the information and (ii) requires the agency to inform the public about the OMB Control Number's legal significance in accordance with 5 CFR 1320.5(b).

The information collected by PTOL-85 Part B is required by 37 CFR 1.311. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 30 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, Virginia 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, Virginia 22313-1450. Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

Privacy Act Statement

The Privacy Act of 1974 (P.L. 93-579) requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1. The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether disclosure of these records is required by the Freedom of Information Act.
2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.
3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.
4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).
5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.
6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).
7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.
8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspection or an issued patent.
9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

Notice of Allowability	Application No. 15/951,970	Applicant(s) Olson et al.	
	Examiner FRANK LAWRENCE	Art Unit 1776	AIA (FITF) Status No

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address--

All claims being allowable, PROSECUTION ON THE MERITS IS (OR REMAINS) CLOSED in this application. If not included herewith (or previously mailed), a Notice of Allowance (PTOL-85) or other appropriate communication will be mailed in due course. **THIS NOTICE OF ALLOWABILITY IS NOT A GRANT OF PATENT RIGHTS.** This application is subject to withdrawal from issue at the initiative of the Office or upon petition by the applicant. See 37 CFR 1.313 and MPEP 1308.

1. ☑ This communication is responsive to the amendment filed November 25, 2020.
   ☐ A declaration(s)/affidavit(s) under **37 CFR 1.130(b)** was/were filed on _____.

2. ☐ An election was made by the applicant in response to a restriction requirement set forth during the interview on _____; the restriction requirement and election have been incorporated into this action.

3. ☑ The allowed claim(s) is/are 1-4 and 8-21 . As a result of the allowed claim(s), you may be eligible to benefit from the **Patent Prosecution Highway** program at a participating intellectual property office for the corresponding application. For more information , please see **http://www.uspto.gov/patents/init_events/pph/index.jsp** or send an inquiry to **PPHfeedback@uspto.gov.**

4. ☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).

   Certified copies:

   a) ☐ All b) ☐ Some *c) ☐ None of the:

   1. ☐ Certified copies of the priority documents have been received.
   2. ☐ Certified copies of the priority documents have been received in Application No. _____ .
   3. ☐ Copies of the certified copies of the priority documents have been received in this national stage application from the International Bureau (PCT Rule 17.2(a)).

   * Certified copies not received: _____ .

Applicant has THREE MONTHS FROM THE "MAILING DATE" of this communication to file a reply complying with the requirements noted below. Failure to timely comply will result in ABANDONMENT of this application.
THIS THREE-MONTH PERIOD IS NOT EXTENDABLE.

5. ☐ CORRECTED DRAWINGS (as "replacement sheets") must be submitted.
   ☐ including changes required by the attached Examiner's Amendment / Comment or in the Office action of Paper No./Mail Date _____ .

   Identifying indicia such as the application number (see 37 CFR 1.84(c)) should be written on the drawings in the front (not the back) of each sheet. Replacement sheet(s) should be labeled as such in the header according to 37 CFR 1.121(d).

6. ☐ DEPOSIT OF and/or INFORMATION about the deposit of BIOLOGICAL MATERIAL must be submitted. Note the attached Examiner's comment regarding REQUIREMENT FOR THE DEPOSIT OF BIOLOGICAL MATERIAL.

Attachment(s)

1. ☐ Notice of References Cited (PTO-892)
2. ☑ Information Disclosure Statements (PTO/SB/08), Paper No./Mail Date _____.
3. ☐ Examiner's Comment Regarding Requirement for Deposit of Biological Material _____.
4. ☑ Interview Summary (PTO-413), Paper No./Mail Date. 12/23/20.
5. ☑ Examiner's Amendment/Comment
6. ☑ Examiner's Statement of Reasons for Allowance
7. ☐ Other _____.

/FRANK M LAWRENCE JR/
Primary Examiner, Art Unit 1776

Notice of Pre-AIA or AIA Status

1. The present application is being examined under the pre-AIA first to invent provisions.

EXAMINER'S AMENDMENT

2. An examiner's amendment to the record appears below. Should the changes and/or additions be unacceptable to applicant, an amendment may be filed as provided by 37 CFR 1.312. To ensure consideration of such an amendment, it MUST be submitted no later than the payment of the issue fee.

Authorization for this examiner's amendment was given in an interview with Mr. Nicholas Lanzatella on December 23, 2020.

3. The application has been amended as follows:

In the Claims:

Claim 1 has been amended as follows:

---1. (Currently Amended) A method for separating mercury from a mercury-containing gas, the method comprising:

combusting a mixture of coal and an additive in a combustion chamber, to form the mercury-containing gas, wherein the additive is chosen from halides, halogens, salts thereof, and combinations thereof;

adding a particulate sorbent material comprising activated carbon into the mercury-containing gas, wherein a weight ratio of ~~I^-, I_2, Br^-, Br_2, Cl^-, Cl_2, or a combination thereof in~~ the additive ~~fed~~added to the coal, added to the combustion chamber, or a combination thereof, to an amount of the sorbent material added to the mercury-containing gas is from about 1:100 to about 30:100;

contacting mercury in the mercury-containing gas with the ~~promoted~~ sorbent material, to form a mercury-sorbent; and

separating the mercury-sorbent from the mercury-containing gas.---

Claim 20 has been amended as follows:

---20. (Currently Amended) A method for separating mercury from a mercury-containing gas, the method comprising:

combusting coal comprising an additive in a combustion chamber, wherein the additive is chosen from halides, halogens, salts thereof, and combinations thereof, to form the mercury-containing gas, wherein the additive comprises a and b, b and c, a and c, or a, b, and c:

a) I^- and/or I_2,

b) Br^- and/or Br_2,

c) Cl^- and/or Cl_2;

injecting a sorbent material comprising activated carbon into the mercury-containing gas, wherein a weight ratio of ~~I^-, I_2, Br^-, Br_2, Cl^-, Cl_2, or a combination thereof in~~ the additive ~~fed~~added to the coal, added to the combustion chamber, or a combination thereof, to an amount of the sorbent material added to the mercury-containing gas is from about 1:100 to about 30:100;

contacting mercury in the mercury-containing gas with the sorbent material, to form a mercury-sorbent; and

separating the mercury-sorbent from the mercury-containing gas.---

Claim 22 has been canceled.

Claim 23 has been canceled.

4. The following is an examiner's statement of reasons for allowance: The examiner agrees with applicant's characterization of how the amended claims distinguish over the prior art of record, which fails to disclose or suggest a motivation for using a weight ratio of additive added to the coal and/or combustion chamber, to an amount of injected sorbent is from about 1:100 to about 30:100, in combination with the other recited process steps. Double patenting over 16/509,071 is withdrawn because it is the last remaining issue before allowance in this earlier filed application.

Any comments considered necessary by applicant must be submitted no later than the payment of the issue fee and, to avoid processing delays, should preferably accompany the issue fee. Such submissions should be clearly labeled "Comments on Statement of Reasons for Allowance."

Information Disclosure Statement

5. The information disclosure statement (IDS) submitted on November 25, 2020 was filed after the mailing date of the Non-final office action on May 27, 2020. The submission is in compliance with the provisions of 37 CFR 1.97. Accordingly, the information disclosure statement is being considered by the examiner.

Any inquiry concerning this communication or earlier communications from the examiner should be directed to FRANK LAWRENCE whose telephone number is (571)272-1161. The examiner can normally be reached on Mon-Fri 8:30am-7pm.

Examiner interviews are available via telephone, in-person, and video conferencing using a USPTO supplied web-based collaboration tool. To schedule an

interview, applicant is encouraged to use the USPTO Automated Interview Request (AIR) at http://www.uspto.gov/interviewpractice.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Nam Nguyen can be reached on 571-272-1342. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see https://ppair-my.uspto.gov/pair/PrivatePair. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

/FRANK M LAWRENCE JR/
Primary Examiner, Art Unit 1776

fl

Examiner-Initiated Interview Summary	Application No. 15/951,970	Applicant(s) Olson et al.	
	Examiner FRANK LAWRENCE	Art Unit 1776	AIA (FITF) Status No

All participants (applicant, applicant's representative, PTO personnel):

(1) FRANK LAWRENCE. (3) _____.

(2) Nicholas Lanzatella. (4) _____.

Date of Interview: 23 December 2020.

Type: ☑ Telephonic ☐ Video Conference
☐ Personal [copy given to: ☐ applicant ☐ applicant's representative]

Exhibit shown or demonstration conducted: ☐ Yes ☑ No.
If Yes, brief description: _____.

Issues Discussed ☐ 101 ☑ 112 ☐ 102 ☐ 103 ☑ Others
(For each of the checked box(es) above, please describe below the issue and detailed description of the discussion)

Claim(s) discussed: 1,20 and 22-23.

Identification of prior art discussed: none.

Substance of Interview
(For each issue discussed, provide a detailed description and indicate if agreement was reached. Some topics may include: identification or clarification of a reference or a portion thereof, claim interpretation, proposed amendments, arguments of any applied references etc...)

Applicant agreed to a proposed examiner's amendment to place the application in condition for allowance, including correcting indefinite terms and typographical errors, and to correct a phrase rasing a new matter issue as detailed in the attached action..

Applicant recordation instructions: It is not necessary for applicant to provide a separate record of the substance of interview.

Examiner recordation instructions: Examiners must summarize the substance of any interview of record. A complete and proper recordation of the substance of an interview should include the items listed in MPEP 713.04 for complete and proper recordation including the identification of the general thrust of each argument or issue discussed, a general indication of any other pertinent matters discussed regarding patentability and the general results or outcome of the interview, to include an indication as to whether or not agreement was reached on the issues raised.

☐ Attachment

/FRANK M LAWRENCE JR/ Primary Examiner, Art Unit 1776	

Search Notes	Application/Control No.	Applicant(s)/Patent Under Reexamination
	15/951,970	Olson et al.
	Examiner	Art Unit
	FRANK LAWRENCE	1776

CPC - Searched*		
Symbol	**Date**	**Examiner**
B01D 53/02,04,10,64, 2253/102,25, 2257/602, 2258/0283	12/23/2020	fl
B01J 20/02,0262,027,04,041,043,10,106,12,20,22,223, 28004,28016,3416, 2220/42	12/23/2020	fl
Y10S 95/901	12/23/2020	fl

CPC Combination Sets - Searched*		
Symbol	**Date**	**Examiner**

US Classification - Searched*			
Class	**Subclass**	**Date**	**Examiner**
95	134, 142	12/23/2020	fl
110	345	12/23/2020	fl
423	210	12/23/2020	fl

* See search history printout included with this form or the SEARCH NOTES box below to determine the scope of the search.

Search Notes		
Search Notes	**Date**	**Examiner**
IDS Search (EAST and DAV)	05/21/2020	AO
Inventor Search (DAV)	05/21/2020	AO
EAST searched for sorbents for mercury from flue gases.	05/21/2020	AO
inventor search, PGPubs, BRS, Derwent, OCR, CPC, global dossier, reviewed related apps, class 96 text	12/23/2020	fl

/AMBER R ORLANDO/ Primary Examiner, Art Unit 1776	

Search Notes	Application/Control No.	Applicant(s)/Patent Under Reexamination
	15/951,970	Olson et al.
	Examiner	Art Unit
	FRANK LAWRENCE	1776

Interference Search			
US Class/CPC Symbol	US Subclass/CPC Group	Date	Examiner
all above	see printout	12/23/2020	fl

/AMBER R ORLANDO/ Primary Examiner, Art Unit 1776	

Issue Classification	Application/Control No.	Applicant(s)/Patent Under Reexamination
	15/951,970	Olson et al.
	Examiner	**Art Unit**
	FRANK LAWRENCE	1776

CPC						
Symbol					**Type**	**Version**
B01D	/	53	/	64	F	2013-01-01
B01D	/	53	/	10	I	2013-01-01
B01J	/	20	/	20	I	2013-01-01
B01J	/	20	/	02	I	2013-01-01
B01J	/	20	/	0262	I	2013-01-01
B01J	/	20	/	027	I	2013-01-01
B01J	/	20	/	04	I	2013-01-01
B01J	/	20	/	041	I	2013-01-01
B01J	/	20	/	043	I	2013-01-01
B01J	/	20	/	10	I	2013-01-01
B01J	/	20	/	106	I	2013-01-01
B01J	/	20	/	12	I	2013-01-01
B01J	/	20	/	223	I	2013-01-01
B01J	/	20	/	28004	I	2013-01-01
B01J	/	20	/	28016	I	2013-01-01
B01J	/	20	/	3416	I	2013-01-01
B01J	/	20	/	22	I	2013-01-01
B01D	/	2253	/	102	A	2013-01-01
B01D	/	2257	/	602	A	2013-01-01
Y10S	/	95	/	901	A	2013-01-01
B01J	/	2220	/	42	A	2013-01-01
B01D	/	2258	/	0283	A	2013-01-01
B01D	/	2253	/	25	A	2013-01-01

CPC Combination Sets							
Symbol					**Type**	**Set**	**Ranking**	**Version**
	/		/					

NONE		**Total Claims Allowed:**	
(Assistant Examiner)	(Date)	18	
/FRANK M LAWRENCE JR/ Primary Examiner, Art Unit 1776	23 December 2020	O.G. Print Claim(s)	O.G. Print Figure
(Primary Examiner)	(Date)	1	11

U.S. Patent and Trademark Office

Part of Paper No.: 20201223

Issue Classification	Application/Control No.	Applicant(s)/Patent Under Reexamination
	15/951,970	Olson et al.
	Examiner	**Art Unit**
	FRANK LAWRENCE	1776

INTERNATIONAL CLASSIFICATION					
CLAIMED					
B01D	/	53	/	10	
B01D	/	53	/	64	

NON-CLAIMED				
	/		/	

US ORIGINAL CLASSIFICATION	
CLASS	**SUBCLASS**
95	134

CROSS REFERENCES(S)						
CLASS	**SUBCLASS (ONE SUBCLASS PER BLOCK)**					

NONE (Assistant Examiner)	(Date)	**Total Claims Allowed:** 18	
/FRANK M LAWRENCE JR/ Primary Examiner, Art Unit 1776 (Primary Examiner)	23 December 2020 (Date)	O.G. Print Claim(s) 1	O.G. Print Figure 11

U.S. Patent and Trademark Office

Part of Paper No.: 20201223

Issue Classification	Application/Control No.	Applicant(s)/Patent Under Reexamination
	15/951,970	Olson et al.
	Examiner	**Art Unit**
	FRANK LAWRENCE	1776

☑ Claims renumbered in the same order as presented by applicant ☐ CPA ☐ T.D. ☐ R.1.47

CLAIMS

Final	Original	Final	Original	Final	Original	Final	Original	Final	Original	Final	Original	Final	Original	Final	Original
1	1	7	10	16	19										
2	2	8	11	17	20										
3	3	9	12	18	21										
4	4	10	13		22										
	5	11	14		23										
	6	12	15												
	7	13	16												
5	8	14	17												
6	9	15	18												

NONE		**Total Claims Allowed:**	
(Assistant Examiner)	(Date)	18	
/FRANK M LAWRENCE JR/ Primary Examiner, Art Unit 1776	23 December 2020	O.G. Print Claim(s)	O.G. Print Figure
(Primary Examiner)	(Date)	1	11

U.S. Patent and Trademark Office

Part of Paper No.: 20201223

EAST Search History

EAST Search History (Prior Art)

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
L1	876	(95/134,142.ccls. or 96/108-154.ccls. or 110/345.ccls. or 423/210.ccls.) and ((mercury or hg) same (furnace or flue or combustion)) and coal	US-PGPUB; USPAT; USOCR	OR	ON	2020/12/21 18:29
L2	1,912	(95/134,142.ccls. or 96/108-154.ccls. or 110/345.ccls. or 423/210.ccls.) and ((mercury or hg))	US-PGPUB; USPAT; USOCR	OR	ON	2020/12/21 18:30
L3	433	2 and ((iodine or iodide or hi or halogen or halide chloride or chlorine or hcl or bromine or bromide or hbr) with (coal or chamber or furnace))	US-PGPUB; USPAT; USOCR	OR	ON	2020/12/21 18:31
L4	346	2 and ((iodine or iodide or hi or halogen or halide chloride or chlorine or hcl or bromine or bromide or hbr) with (coal or chamber or furnace)) and (activated adj carbon)	US-PGPUB; USPAT; USOCR	OR	ON	2020/12/21 18:31
L5	4,166	(b01d53/04,02,10,06,64 or b01d2253/25,102 or b01d2257/602 or b01d2258/0283 or b01j20/02,0262,027,04,041,043,10,106,12,20,22,223,28004,28016,3416 or b01j2220/42 or y10s95/901).cpc. and ((mercury or hg)) not 2	US-PGPUB; USPAT; USOCR	OR	ON	2020/12/21 18:34
L6	395	5 and ((iodine or iodide or hi or halogen or halide chloride or chlorine or hcl or bromine or bromide or hbr) with (coal or chamber or furnace)) and (activated adj carbon)	US-PGPUB; USPAT; USOCR	OR	ON	2020/12/21 18:34
L7	235	(mercury or hg).ti. and ((iodine or iodide or hi or halogen or halide chloride or chlorine or hcl or bromine or bromide or hbr) and (coal or chamber or furnace)) and ((activated adj carbon) or sorbent)	EPO; JPO; DERWENT	OR	ON	2020/12/21 18:35
L8	3,301	(b01d53/04,02,10,06,64 or b01d2253/25,102 or b01d2257/602 or b01d2258/0283 or b01j20/02,0262,027,04,041,043,10,106,12,20,22,223,28004,28016,3416 or b01j2220/42 or y10s95/901).cpc. and ((mercury or hg)) and (furnace or coal)	US-PGPUB; USPAT; USOCR	OR	ON	2020/12/21 18:38
L9	67	8 and ((iodine or iodide or hi or halogen or halide chloride or chlorine or hcl or bromine or bromide or hbr) with ratio with (sorbent or (activated adj carbon)))	US-PGPUB; USPAT; USOCR	OR	ON	2020/12/21 18:38

L10	67	8 and ((iodine or iodide or hi or halogen or halide chloride or chlorine or hcl or bromine or bromide or hbr) with ratio with (sorbent or (activated adj carbon)))	US-PGPUB; USPAT; USOCR	OR	ON	2020/12/21 18:39
L11	309	(95/134,142.ccls. or 96/108-154.ccls. or 110/345.ccls. or 423/210.ccls.) and ((mercury or hg)) and (furnace or coal) not 8	US-PGPUB; USPAT; USOCR	OR	ON	2020/12/21 18:44
L12	1	11 and ((iodine or iodide or hi or halogen or halide chloride or chlorine or hcl or bromine or bromide or hbr) with ratio with (sorbent or (activated adj carbon)))	US-PGPUB; USPAT; USOCR	OR	ON	2020/12/21 18:44
S1	1	"20180229182".pn.	US-PGPUB; USPAT; USOCR	OR	ON	2020/12/21 12:52
S2	61	olson.in. and edwin.in. and mercury and (halide or iodide) and coal and combustion	US-PGPUB; USPAT; USOCR	OR	ON	2020/12/21 14:22
S3	0	holmes.in. and michael.in. and mercury and (halide or iodide) and coal and combustion not S2	US-PGPUB; USPAT; USOCR	OR	ON	2020/12/21 15:08
S4	1	pavlish.in. and john.in. and mercury and (halide or iodide) and coal and combustion not S2	US-PGPUB; USPAT; USOCR	OR	ON	2020/12/21 15:09
S5	7	(("10589225") or ("10596517") or ("10668430") or ("10828596") or ("20070179056") or ("20170056853") or ("20200009532")).PN.	US-PGPUB; USPAT; USOCR	OR	OFF	2020/12/21 15:15
S6	2	S5 not S2	US-PGPUB; USPAT; USOCR	OR	ON	2020/12/21 15:15

EAST Search History (Interference)

<This search history is empty>

12/21/2020 6:45:31 PM

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www.uspto.gov

BIB DATA SHEET

CONFIRMATION NO. 6410

SERIAL NUMBER	FILING or 371(c) DATE	CLASS	GROUP ART UNIT	ATTORNEY DOCKET NO.
15/951,970	04/12/2018 RULE	095	1776	4820.001US8

APPLICANTS

Midwest Energy Emissions Corp., Lewis Center, OH;

INVENTORS

Edwin S. Olson, Grand Forks, ND;
Michael J. Holmes, Thompson, ND;
John H. Pavlish, East Grand Forks, MN;

**** CONTINUING DATA ***************************

This application is a CON of 14/712,558 05/14/2015 PAT 10589225
which is a CON of 14/318,270 06/27/2014 PAT 9757689
which is a CON of 13/966,768 08/14/2013 PAT 8821819
which is a CON of 13/427,665 03/22/2012 PAT 8512655
which is a CON of 12/419,219 04/06/2009 PAT 8168147
which is a CON of 12/201,595 08/29/2008 ABN
which is a DIV of 11/209,163 08/22/2005 PAT 7435286
which claims benefit of 60/605,640 08/30/2004

**** FOREIGN APPLICATIONS ***************************

**** IF REQUIRED, FOREIGN FILING LICENSE GRANTED ****

05/09/2018

Foreign Priority claimed ☐ Yes ☑ No 35 USC 119(a-d) conditions met ☐ Yes ☑ No Verified and Acknowledged /FRANK M LAWRENCE JR/ Examiner's Signature	☐ Met after Allowance Initials	STATE OR COUNTRY	SHEETS DRAWINGS	TOTAL CLAIMS	INDEPENDENT CLAIMS
		ND	11	20	2

ADDRESS

SCHWEGMAN LUNDBERG & WOESSNER, P.A.
P.O. BOX 2938
MINNEAPOLIS, MN 55402
UNITED STATES

TITLE

SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY

FILING FEE RECEIVED		
1880	FEES: Authority has been given in Paper No._____ to charge/credit DEPOSIT ACCOUNT No._____ for following:	☐ All Fees ☐ 1.16 Fees (Filing) ☐ 1.17 Fees (Processing Ext. of time) ☐ 1.18 Fees (Issue) ☐ Other _____ ☐ Credit

BIB (Rev. 05/07).

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US Patent & Trademark Office: U.S. DEPARTMENT OF COMMERCE
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Substitute for form 1449A/PTO **INFORMATION DISCLOSURE STATEMENT BY APPLICANT** (Use as many sheets as necessary)	*Complete if Known*	
	Application Number	15/951,970
	Filing Date	April 12, 2018
	First Named Inventor	Edwin S. Olson
	Group Art Unit	1773
	Examiner Name	HEIDI RIVIERE KELLEY
Sheet 1 of 2	Attorney Docket No: 4820.001US8	

US PATENT DOCUMENTS

	USP Document Number	Publication Date	Name of Patentee or Applicant of cited Document
	US-10,589,225	03/17/2020	Olson, Edwin S, et al.
	US-10,596,517	03/24/2020	Olson, Edwin S, et al.
	US-10,668,430	06/02/2020	Olson, Edwin S, et al.
	US-10,828,596	11/10/2020	Olson, Edwin S
	US-20070179056A1	08/02/2007	Baek, Jeom I, et al.
	US-20170056853A1	03/02/2017	Mimna, Richard A., et al.
	US-20200009532A1	01/09/2020	Olson, Edwin S, et al.

FOREIGN PATENT DOCUMENTS

Examiner Initial *	Foreign Document Number	Publication Date	Name of Patentee or Applicant of cited Document	T 1

EXAMINER /FRANK M LAWRENCE JR/ DATE CONSIDERED 12/21/2020

* EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant. 1 Applicant is to place a check mark here if English language Translation is attached

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	Group Art Unit	1773
	Examiner Name	HEIDI RIVIERE KELLEY
Sheet 2 of 2	Attorney Docket No: 4820.001US8	

OTHER DOCUMENTS – NON PATENT LITERATURE DOCUMENTS		
Examiner Initial *	**Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.**	**T 1**
	"Activated carbon", Wikipedia, [Online] Retrieved from the Internet: <URL: https://en.wikipedia.org/wiki/Activated_carbon>, (Retrieved on 05-11-20), 21 pgs.	
	"Application Serial No. 14/712,558, Notice of Allowance mailed 12-13-19", 7 pgs	
	"Application Serial No. 15/382,114, Non Final Office Action mailed 12-31-19", 33 pgs	
	"Application Serial No. 15/382,114, Notice of Allowance mailed 08-10-20", 11 pgs	
	"Application Serial No. 15/382,114, Response filed 06-30-20 to Non Final Office Action mailed 12-31-19", 21 pgs	
	"Application Serial No. 15/853,029, Response filed 11-20-20 to Restriction Requirement mailed 05-21-20", 10 pgs	
	"Application Serial No. 15/853,029, Restriction Requirement mailed 05-21-20", 8 pgs	
	"Application Serial No. 15/974,343, Non Final Office Action mailed 12-02-19", 12 pgs	
	"Application Serial No. 15/974,343, Notice of Allowance mailed 04-13-20", 7 pgs	
	"Application Serial No. 15/974,343, Response filed 4/2/2020 to Non-Final Office action filed 12/2/2019", 13 pgs.	
	"Application Serial No. 16/130,670, Final Office Action mailed 08-24-20", 11 pgs	
	"Application Serial No. 16/130,670, Non Final Office Action mailed 12-16-19", 8 pgs	
	"Application Serial No. 16/130,670, Response filed 06-16-20 to Non Final Office Action mailed 12-16-19", 14 pgs	
	"Application Serial No. 16/509,071, Final Office Action mailed 12-12-19", 23 pgs	
	"Application Serial No. 16/509,071, Non Final Office Action mailed 05-29-20", 31 pgs	
	"Application Serial No. 16/509,071, Response filed 05-12-20 to Final Office Action mailed 12-12-19", 31 pgs	
	"Application Serial No. 16/509,102, Final Office Action mailed 02-14-20", 13 pgs	
	"Application Serial No. 16/509,146, Final Office Action mailed 02-14-20", 23 pgs	
	"Application Serial No. 16/574,935, Non Final Office Action mailed 09-09-20", 17 pgs	
	"Application Serial No. 16/574,935, Response filed 09-16-20 to Non Final Office Action mailed 09-09-20", 12 pgs	
	"DARCO Hg, Powdered Activated Carbon Data SHeet", CABOT Corp., (24 October 2017), 2 pgs.	
	"Fluegas Properties Calculator", Increase Performance Inc., [Online] Retrieved from the Internet: <URL: http://www.increase-performance.com/calc-flue-gas-prop.html>, (Retrieved on 05-11-20), 1 pg.	
	"STEAM Table 8a, 8b", Steam: its Generation and Use 42nd Ed, (2015), 2 pgs.	

EXAMINER /FRANK M LAWRENCE JR/ DATE CONSIDERED 12/21/2020

* EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant. 1 Applicant is to place a check mark here if English language Translation is attached

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Substitute for form 1449A/PTO

INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Use as many sheets as necessary)

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Sheet 1 of 2

Attorney Docket No: 4820.001US8

US PATENT DOCUMENTS

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	US-10,828,596	11/10/2020	Olson, Edwin S
	US-20070179056A1	08/02/2007	Baek, Jeom I, et al.
	US-20170056853A1	03/02/2017	Mimna, Richard A., et al.
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EXAMINER **DATE CONSIDERED**

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	Examiner Name	HEIDI RIVIERE KELLEY
Sheet 2 of 2	Attorney Docket No: 4820.001US8	

OTHER DOCUMENTS – NON PATENT LITERATURE DOCUMENTS		
Examiner Initial *	**Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.**	**T †**
	"Activated carbon", Wikipedia, [Online] Retrieved from the Internet: <URL: https://en.wikipedia.org/wiki/Activated_carbon>, (Retrieved on 05-11-20), 21 pgs.	
	"Application Serial No. 14/712,558, Notice of Allowance mailed 12-13-19", 7 pgs	
	"Application Serial No. 15/382,114, Non Final Office Action mailed 12-31-19", 33 pgs	
	"Application Serial No. 15/382,114, Notice of Allowance mailed 08-10-20", 11 pgs	
	"Application Serial No. 15/382,114, Response filed 06-30-20 to Non Final Office Action mailed 12-31-19", 21 pgs	
	"Application Serial No. 15/853,029, Response filed 11-20-20 to Restriction Requirement mailed 05-21-20", 10 pgs	
	"Application Serial No. 15/853,029, Restriction Requirement mailed 05-21-20", 8 pgs	
	"Application Serial No. 15/974,343, Non Final Office Action mailed 12-02-19", 12 pgs	
	"Application Serial No. 15/974,343, Notice of Allowance mailed 04-13-20", 7 pgs	
	"Application Serial No. 15/974,343, Response filed 4/2/2020 to Non-Final Office action filed 12/2/2019", 13 pgs.	
	"Application Serial No. 16/130,670, Final Office Action mailed 08-24-20", 11 pgs	
	"Application Serial No. 16/130,670, Non Final Office Action mailed 12-16-19", 8 pgs	
	"Application Serial No. 16/130,670, Response filed 06-16-20 to Non Final Office Action mailed 12-16-19", 14 pgs	
	"Application Serial No. 16/509,071, Final Office Action mailed 12-12-19", 23 pgs	
	"Application Serial No. 16/509,071, Non Final Office Action mailed 05-29-20", 31 pgs	
	"Application Serial No. 16/509,071, Response filed 05-12-20 to Final Office Action mailed 12-12-19", 31 pgs	
	"Application Serial No. 16/509,102, Final Office Action mailed 02-14-20", 13 pgs	
	"Application Serial No. 16/509,146, Final Office Action mailed 02-14-20", 23 pgs	
	"Application Serial No. 16/574,935, Non Final Office Action mailed 09-09-20", 17 pgs	
	"Application Serial No. 16/574,935, Response filed 09-16-20 to Non Final Office Action mailed 09-09-20", 12 pgs	
	"DARCO Hg, Powdered Activated Carbon Data SHeet", CABOT Corp., (24 October 2017), 2 pgs.	
	"Fluegas Properties Calculator", Increase Performance Inc., [Online] Retrieved from the Internet: <URL: http://www.increase-performance.com/calc-flue-gas-prop.html>, (Retrieved on 05-11-20), 1 pg.	
	"STEAM Table 8a, 8b", Steam: its Generation and Use 42nd Ed, (2015), 2 pgs.	

EXAMINER **DATE CONSIDERED**

* EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant. † Applicant is to place a check mark here if English language Translation is attached

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s): Edwin S. Olson et al.	Examiner: HEIDI RIVIERE KELLEY
Serial No.: 15/951,970	Group Art Unit: 1773
Filed: April 12, 2018	Docket: 4820.001US8
Customer No.: 21186	Confirmation No.: 6410
Title: SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY	

SUPPLEMENTAL INFORMATION DISCLOSURE STATEMENT

MS Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

In compliance with the duty imposed by 37 C.F.R. § 1.56, and in accordance with 37 C.F.R. §§ 1.97 *et. seq.*, the enclosed materials are brought to the attention of the Examiner for consideration in connection with the above-identified patent application. Applicant respectfully requests that this Information Disclosure Statement be entered and the documents listed on the attached PTO 1449 Form be considered by the Examiner and made of record. Pursuant to the provisions of MPEP 609, Applicant requests that a copy of the PTO 1449 Form, initialed as being considered by the Examiner, be returned to the Applicant with the next official communication.

Pursuant to 37 C.F.R. § 1.97(c)(2), Applicant hereby authorizes the Commissioner to charge the fee of $260.00 as set forth in 37 C.F.R. § 1.17(p), to Deposit Account No. 19-0743. Please charge any additional fees or deficiencies, or credit any overpayment to Deposit Account No. 19-0743.

Pursuant to 37 C.F.R. § 1.98(a)(2), copies of cited U.S. Patents and Published Applications, and Non-Published Applications identifiable by USPTO Serial Number, are no longer required to be provided to the Office. Applicant acknowledges the requirement to submit copies of foreign patent documents and non-patent literature in accordance with 37 C.F.R § 1.98(a)(2).

The Examiner is invited to contact the undersigned at the telephone number indicated if there are any questions regarding this communication.

Respectfully submitted,

SCHWEGMAN LUNDBERG & WOESSNER, P.A.
P.O. Box 2938
Minneapolis, MN 55402
(612) 349-9580

Date November 25, 2020 By /Nicholas P. Lanzatella/
Nicholas P. Lanzatella
Reg. No. 63,803

NPL:mlr1

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s):	Edwin S. Olson et al.	Examiner:	HEIDI RIVIERE KELLEY
Serial No.:	15/951,970	Group Art Unit:	1773
Filed:	April 12, 2018	Docket:	4820.001US8
Customer No.:	21186	Confirmation No.:	6410
Title:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY		

PETITION FOR A THREE-MONTH EXTENSION OF TIME

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

In accordance with the provision of 37 C.F.R § 1.136(a), it is respectfully requested that a three-month extension of time be granted in which to respond to the Non-Final Office Action mailed May 27, 2020, said period of response being extended from August 27, 2020 to November 27, 2020.

Please charge Deposit Account No. 19-0743 in the amount of $1,480.00 to cover the required extension fee. Please charge any additional fees or credit overpayment to deposit Account No. 19-0743.

Respectfully submitted,

SCHWEGMAN LUNDBERG & WOESSNER, P.A.
P.O. Box 2938
Minneapolis, MN 55402
(612) 349-9580

Date November 25, 2020

By /Nicholas P. Lanzatella/
Nicholas P. Lanzatella
Reg. No. 63,803

REMARKS

This responds to the Non-Final Office Action dated May 27, 2020.

Claims 1, 2, 4, 16-17, and 20 are presently amended. Claims 5-7 are presently canceled, and no claims were canceled previously. New claims 21-23 are presently added. As a result, claims 1-4 and 8-23 are now pending in this application.

Independent claim 1 is presently amended to recite "…wherein a weight ratio of I^-, I_2, Br^-, Br_2, Cl^-, Cl_2, or a combination thereof in the additive fed to the combustion chamber to an amount of the sorbent material added to the mercury-containing gas is from 1:100 to 30:100…" Independent claim 20 is correspondingly amended. Support can be found in the present published U.S. application at paragraphs [0021], [0025], and [0072].

Independent claim 20 is presently amended to recite "…wherein the additive is chosen from halides, halogens, salts thereof, and combinations thereof, wherein the additive comprises a and b, b and c, a and c, or a, b, and c: a) I^- and/or I_2, b) Br^- and/or Br_2, c) Cl^- and/or Cl_2…." Support can be found in the present published U.S. application at paragraphs [0012], [0015], [0022]-[0023], [0073], and [0103].

No new matter is added via the amendments herein.

New dependent claim 21 is added, reciting the "…method of claim 1, wherein the additive comprises a and b, b and c, a and c, or a, b, and c: a) I^- and/or I_2, b) Br^- and/or Br_2, c) Cl^- and/or Cl_2." Support can be found in the present published U.S. application at paragraphs [0012], [0015], [0022]-[0023], [0073], and [0103].

New dependent claim 22 is added, reciting the "…method of claim 1, wherein a weight ratio of I^-, I_2, Br^-, Br_2, or a combination thereof in the additive fed to the combustion chamber to an amount of the sorbent material added to the mercury-containing gas is from 1:100 to 30:100."

New dependent claim 23 is added, reciting the "…method of claim 1, wherein a weight ratio of I^-, Br^-, Br_2, or a combination thereof in the additive fed to the combustion chamber to an amount of the sorbent material added to the mercury-containing gas is from 1:100 to 30:100."

Dependent claims 21-23 are dependent on independent claim 1 and therefore incorporate all the features thereof. Accordingly, dependent claims 21-23 are patentable for at least the reasons given herein for patentability of independent claim 1.

No new matter is added via the new claims herein.

The Rejection of Claims Under § 102

I. The Office Action rejected claims 1-2, 5-10, 14-15, 17, and 20 under 35 U.S.C. § 102(b) over U.S. patent no. 5,435,980 ("Felsvang").

This rejection is respectfully traversed.

Independent claim 1 is presently amended to recite "…wherein a weight ratio of I^-, I_2, Br^-, Br_2, Cl^-, Cl_2, or a combination thereof in the additive fed to the combustion chamber to an amount of the sorbent material added to the mercury-containing gas is from 1:100 to 30:100…." Independent claim 20 is correspondingly amended.

Felsvang is directed to adding chlorine (Cl_2) or chloride (Cl^-) materials to the coal. Felsvang mentions HCl flue gas concentrations of 90 mg/Nm^3, 65 mg/Nm^3, and 142 mg/Nm^3 in Examples 2, 3, and 4, respectively, and Feslvang describes activated carbon injection rates of 1-100 mg/Nm^3 (column 4 lines 16-17). Accordingly, Felsvang mentions a range of the weight of HCl in the flue gas to the weight of activated carbon injected into the flue gas of from 65:100 to 14200:100. Therefore, Felsvang fails to describe or suggest a weight ratio of I^-, I_2, Br^-, Br_2, Cl^-, Cl_2, or a combination thereof in the additive fed to the combustion chamber to an amount of the sorbent material added to the mercury-containing gas of from 1:100 to 30:100. Felsvang fails to describe or suggest all the features of independent claims 1 or 20; therefore, these claims are patentable over Felsvang.

Dependent claims 2, 5-10, 14-15, and 17 are dependent on independent claim 1. As such, these dependent claims incorporate all the features of claim 1. Accordingly, dependent claims 2, 5-10, 14-15, and 17 are patentable for at least the reasons given herein for patentability of independent claim 1.

Applicant therefore respectfully requests reconsideration and withdrawal of the rejection of claims 1-2, 5-10, 14-15, 17, and 20 under 35 U.S.C. § 102(b) over Felsvang.

The Rejection of Claims Under § 103

II. The Office Action rejected claims 3-4 under 35 U.S.C. § 103 over U.S. patent no. 5,435,980 ("Felsvang") in view of U.S. patent publication no. 2004/0086439 ("Vosteen").

This rejection is respectfully traversed.

Independent claim 1 is presently amended to recite "…wherein a weight ratio of I^-, I_2, Br^-, Br_2, Cl^-, Cl_2, or a combination thereof in the additive fed to the combustion chamber to an amount of the sorbent material added to the mercury-containing gas is from 1:100 to 30:100…"

As explained herein at **I**, Felsvang fails to describe or suggest a weight ratio of I^-, I_2, Br^-, Br_2, Cl^-, Cl_2, or a combination thereof in the additive fed to the combustion chamber to an amount of the sorbent material added to the mercury-containing gas of from 1:100 to 30:100$_2$. Similarly, Vosteen fails to mention any specific amount of activated carbon in the emission control system and therefore fails to describe or suggest any specific ratio of any halogen species added to the amount of activated carbon. Felsvang and Vosteen fail to describe or suggest all the features of independent claim 1; therefore, this claim is patentable over this combination of references.

Dependent claims 3-4 are indirectly dependent on independent claim 1. As such, these dependent claims incorporate all the features of claim 1. Accordingly, dependent claims 3-4 are patentable for at least the reasons given herein for patentability of independent claim 1.

Applicant therefore respectfully requests reconsideration and withdrawal of the rejection of claims 3-4 under 35 U.S.C. § 103 over Felsvang in view of Vosteen.

III. The Office Action rejected claim 11 under 35 U.S.C. § 103 over U.S. patent no. 5,435,980 ("Felsvang") in view of U.S. patent publication no. 2004/0086439 ("Vosteen").

This rejection is respectfully traversed.

Independent claim 1 is presently amended to recite "…wherein a weight ratio of I^-, I_2, Br^-, Br_2, Cl^-, Cl_2, or a combination thereof in the additive fed to the combustion chamber to an amount of the sorbent material added to the mercury-containing gas is from 1:100 to 30:100…"

As explained herein at **I**, Felsvang fails to describe or suggest a weight ratio of I^-, I_2, Br^-, Br_2, Cl^-, Cl_2, or a combination thereof in the additive fed to the combustion chamber to an amount of the sorbent material added to the mercury-containing gas of from 1:100 to 30:100. Similarly, Vosteen fails to mention any specific amount of activated carbon in the emission control system and therefore fails to describe or suggest any specific ratio of any halogen species added to coal with respect to the activated carbon used. Felsvang and Vosteen fail to describe or suggest all the

features of independent claim 1; therefore, this claim is patentable over this combination of references.

Dependent claim 11 is dependent on independent claim 1. As such, this dependent claim incorporates all the features of claim 1. Accordingly, dependent claim 11 is patentable for at least the reasons given herein for patentability of independent claim 1.

Applicant therefore respectfully requests reconsideration and withdrawal of the rejection of claim 11 under 35 U.S.C. § 103 over Felsvang in view of Vosteen.

IV. The Office Action rejected claim 12 under 35 U.S.C. § 103 over U.S. patent no. 5,435,980 ("Felsvang") in view of U.S. patent publication no. 2006/0057044 ("Chang").

This rejection is respectfully traversed.

Independent claim 1 is presently amended to recite "…wherein a weight ratio of I^-, I_2, Br^-, Br_2, Cl^-, Cl_2, or a combination thereof in the additive fed to the combustion chamber to an amount of the sorbent material added to the mercury-containing gas is from 1:100 to 30:100…."

As explained herein at **I**, Felsvang fails to describe or suggest a weight ratio of I^-, I_2, Br^-, Br_2, Cl^-, Cl_2, or a combination thereof in the additive fed to the combustion chamber to an amount of the sorbent material added to the mercury-containing gas of from 1:100 to 30:100. Chang fails to describe or suggesting adding halogens or halides to coal, and therefore fails to describe or suggest any specific ratio of any halogen species added to coal with respect to the activated carbon used. Felsvang and Chang fail to describe or suggest all the features of independent claim 1; therefore, this claim is patentable over this combination of references.

Dependent claim 13 is dependent on independent claim 1. As such, this dependent claim incorporates all the features of claim 1. Accordingly, dependent claim 13 is patentable for at least the reasons given herein for patentability of independent claim 1.

Applicant therefore respectfully requests reconsideration and withdrawal of the rejection of claim 12 under 35 U.S.C. § 103 over Felsvang in view of Chang.

V. The Office Action rejected claim 13 under 35 U.S.C. § 103 over U.S. patent no. 5,435,980 ("Felsvang") in view of U.S. patent publication no. 2004/0086439 ("Vosteen") in view of U.S. patent publication no. 2004/0244657 ("Srinivasachar").

This rejection is respectfully traversed.

Independent claim 1 is presently amended to recite "…wherein a weight ratio of I^-, I_2, Br^-, Br_2, Cl^-, Cl_2, or a combination thereof in the additive fed to the combustion chamber to an amount of the sorbent material added to the mercury-containing gas is from 1:100 to 30:100…"

As explained herein at **I**, Felsvang fails to describe or suggest a weight ratio of I^-, I_2, Br^-, Br_2, Cl^-, Cl_2, or a combination thereof in the additive fed to the combustion chamber to an amount of the sorbent material added to the mercury-containing gas of from 1:100 to 30:100. Similarly, Vosteen fails to mention any specific amount of activated carbon in the emission control system and therefore fails to describe or suggest any specific ratio of any halogen species added to coal with respect to the activated carbon used. Srinivasachar describes an activated carbon that includes a halogen species that is prepared outside the flue gas and fails to mention adding halogen species to coal; therefore, Srinivasachar also fails to describe or suggest any ratio of any halogen species added to coal with respect to the activated carbon used. Felsvang, Vosteen, and Srinivasachar fail to describe or suggest all the features of independent claim 1; therefore, claim 1 is patentable over this combination of references.

Dependent claim 13 is dependent on independent claim 1. As such, this dependent claim incorporates all the features of claim 1. Accordingly, dependent claim 13 is patentable for at least the reasons given herein for patentability of independent claim 1.

Applicant therefore respectfully requests reconsideration and withdrawal of the rejection of claim 13 under 35 U.S.C. § 103 over Felsvang in view of Vosteen in view of Srinivasachar".

VI. The Office Action rejected claim 16 under 35 U.S.C. § 103 over U.S. patent no. 5,435,980 ("Felsvang") in view of U.S. patent publication no. 2004/0086439 ("Vosteen") in view of U.S. patent no. 6,136,749 ("Gadkaree").

This rejection is respectfully traversed.

Independent claim 1 is presently amended to recite "…wherein a weight ratio of I^-, I_2, Br^-, Br_2, Cl^-, Cl_2, or a combination thereof in the additive fed to the combustion chamber to an amount of the sorbent material added to the mercury-containing gas is from 1:100 to 30:100…"

As explained herein at **I**, Felsvang fails to describe or suggest a weight ratio of I^-, I_2, Br^-, Br_2, Cl^-, Cl_2, or a combination thereof in the additive fed to the combustion chamber to an amount of the

sorbent material added to the mercury-containing gas of from 1:100 to 30:100. Similarly, Vosteen fails to mention any specific amount of activated carbon in the emission control system and therefore fails to describe or suggest any specific ratio of any halogen species added to coal with respect to the activated carbon used. Gadkaree is directed toward pre-treated activated carbon sorbent and fails to mention adding halogens or halides to coal; therefore, Gadkaree also fails to describe or suggest any ratio of halogen or halide species added to coal with respect to the activated carbon used. Felsvang, Vosteen, and Gadkaree fail to describe or suggest all the features of independent claim 1; therefore, claim 1 is patentable over this combination of references.

Dependent claim 16 is dependent on independent claim 1. As such, this dependent claim incorporates all the features of claim 1. Accordingly, dependent claim 16 is patentable for at least the reasons given herein for patentability of independent claim 1.

Applicant therefore respectfully requests reconsideration and withdrawal of the rejection of claim 16 under 35 U.S.C. § 103 over Felsvang in view of Vosteen in view of Gadkaree.

VII. The Office Action rejected claim 19 under 35 U.S.C. § 103 over U.S. patent no. 5,435,980 ("Felsvang") in view of U.S. patent publication no. 2004/0086439 ("Vosteen") in view of U.S. patent publication no. 2004/0244657 ("Srinivasachar").

This rejection is respectfully traversed.

Independent claim 1 is presently amended to recite "…wherein a weight ratio of I^-, I_2, Br^-, Br_2, Cl^-, Cl_2, or a combination thereof in the additive fed to the combustion chamber to an amount of the sorbent material added to the mercury-containing gas is from 1:100 to 30:100…."

As explained herein at **I**, Felsvang fails to describe or suggest a weight ratio of I^-, I_2, Br^-, Br_2, Cl^-, Cl_2, or a combination thereof in the additive fed to the combustion chamber to an amount of the sorbent material added to the mercury-containing gas of from 1:100 to 30:100. Similarly, Vosteen fails to mention any specific amount of activated carbon in the emission control system and therefore fails to describe or suggest any specific ratio of any halogen species added to coal with respect to the activated carbon used. Srinivasachar describes an activated carbon that includes a halogen species that is prepared outside the flue gas and fails to mention adding halogen species to coal; therefore, Srinivasachar also fails to describe or suggest any ratio of any halogen species added to coal with respect to the activated carbon used. Felsvang, Vosteen, and Srinivasachar fail to

describe or suggest all the features of independent claim 1; therefore, claim 1 is patentable over this combination of references.

Dependent claim 19 is indirectly dependent on independent claim 1. As such, this dependent claim incorporates all the features of claim 1. Accordingly, dependent claim 19 is patentable for at least the reasons given herein for patentability of independent claim 1.

Applicant therefore respectfully requests reconsideration and withdrawal of the rejection of claim 19 under 35 U.S.C. § 103 over Felsvang in view of Vosteen in view of Srinivasachar.

Double Patenting Rejection

VIII(A). The Office Action rejected claims 1-20 for nonstatutory double patenting over claims 1-30 of U.S. patent no. 7,435,286 in view of U.S. patent no. 5,435,980 ("Felsvang") and U.S. patent publication no. 2004/0244657 ("Srinivasachar").

This rejection is respectfully traversed. Applicant does not admit that claims 1-20 are obvious over claims 1-30 of U.S. patent no. 7,435,286 in view of Felsvang and Srinivasachar.

As the current pending claims stand rejected on other grounds and are still subject to amendment, Applicant takes note of these rejections but do not take further action at this time inasmuch as no pending claim has yet to be allowed. Applicant will re-evaluate this rejection, upon an indication of allowable claims.

VIII(B). The Office Action rejected claims 1-20 for nonstatutory double patenting over claims 1-25 of U.S. patent no. 8,168,147 in view of U.S. patent no. 5,435,980 ("Felsvang") and U.S. patent publication no. 2004/0244657 ("Srinivasachar").

This rejection is respectfully traversed. Applicant does not admit that claims 1-20 are obvious over claims 1-25 of U.S. patent no. 8,168,147 in view of Felsvang and Srinivasachar.

As the current pending claims stand rejected on other grounds and are still subject to amendment, Applicant takes note of these rejections but do not take further action at this time inasmuch as no pending claim has yet to be allowed. Applicant will re-evaluate this rejection, upon an indication of allowable claims.

VIII(C). The Office Action rejected claims 1-20 for nonstatutory double patenting over claims 1-60 of U.S. patent no. 8,512,655 in view of U.S. patent no. 5,435,980 ("Felsvang") and U.S. patent publication no. 2004/0244657 ("Srinivasachar").

This rejection is respectfully traversed. Applicant does not admit that claims 1-20 are obvious over claims 1-60 of U.S. patent no. 8,512,655 in view of Felsvang and Srinivasachar.

As the current pending claims stand rejected on other grounds and are still subject to amendment, Applicant takes note of these rejections but do not take further action at this time inasmuch as no pending claim has yet to be allowed. Applicant will re-evaluate this rejection, upon an indication of allowable claims.

VIII(D). The Office Action rejected claims 1-20 for nonstatutory double patenting over claims 1-60 of U.S. patent no. 8,821,819 in view of U.S. patent no. 5,435,980 ("Felsvang") and U.S. patent publication no. 2004/0244657 ("Srinivasachar").

This rejection is respectfully traversed. Applicant does not admit that claims 1-20 are obvious over claims 1-60 of U.S. patent no. 8,821,819 in view of Felsvang and Srinivasachar.

As the current pending claims stand rejected on other grounds and are still subject to amendment, Applicant takes note of these rejections but do not take further action at this time inasmuch as no pending claim has yet to be allowed. Applicant will re-evaluate this rejection, upon an indication of allowable claims.

VIII(E). The Office Action rejected claims 1-20 for nonstatutory double patenting over claims 1-33 of U.S. patent no. 9,757,689 in view of U.S. patent no. 5,435,980 ("Felsvang") and U.S. patent publication no. 2004/0244657 ("Srinivasachar").

This rejection is respectfully traversed. Applicant does not admit that claims 1-20 are obvious over claims 1-33 of U.S. patent no. 9,757,689 in view of Felsvang and Srinivasachar.

As the current pending claims stand rejected on other grounds and are still subject to amendment, Applicant takes note of these rejections but do not take further action at this time inasmuch as no pending claim has yet to be allowed. Applicant will re-evaluate this rejection, upon an indication of allowable claims.

VIII(F). The Office Action rejected claims 1-20 for nonstatutory double patenting over claims 1-20 of U.S. patent no. 9,468,886 in view of U.S. patent no. 5,435,980 ("Felsvang") and U.S. patent publication no. 2004/0244657 ("Srinivasachar").

This rejection is respectfully traversed. Applicant does not admit that claims 1-20 are obvious over claims 1-20 of U.S. patent no. 9,468,886 in view of Felsvang and Srinivasachar.

As the current pending claims stand rejected on other grounds and are still subject to amendment, Applicant takes note of these rejections but do not take further action at this time inasmuch as no pending claim has yet to be allowed. Applicant will re-evaluate this rejection, upon an indication of allowable claims.

VIII(G). The Office Action rejected claims 1-20 for nonstatutory double patenting over claims 1-30 of U.S. patent no. 8,652,235 in view of U.S. patent no. 5,435,980 ("Felsvang") and U.S. patent publication no. 2004/0244657 ("Srinivasachar").

This rejection is respectfully traversed. Applicant does not admit that claims 1-20 are obvious over claims 1-30 of U.S. patent no. 8,652,235 in view of Felsvang and Srinivasachar.

As the current pending claims stand rejected on other grounds and are still subject to amendment, Applicant takes note of these rejections but do not take further action at this time inasmuch as no pending claim has yet to be allowed. Applicant will re-evaluate this rejection, upon an indication of allowable claims.

VIII(H). The Office Action provisionally rejected claims 1-20 for nonstatutory double patenting over claims 1-16 of application no. 15/295,594 in view of U.S. patent no. 5,435,980 ("Felsvang") and U.S. patent publication no. 2004/0244657 ("Srinivasachar").

This rejection is respectfully traversed. Applicant does not admit that claims 1-20 are obvious over claims 1-16 of application no. 15/295,594 in view of Felsvang and Srinivasachar.

As the current pending claims stand rejected on other grounds and are still subject to amendment, Applicant takes note of these rejections but do not take further action at this time inasmuch as no pending claim has yet to be allowed. Applicant will re-evaluate this rejection, upon an indication of allowable claims.

VIII(I). The Office Action provisionally rejected claims 1-20 for nonstatutory double patenting over claims 8-20 of application no. 15/589,359 in view of U.S. patent publication no. 2004/0244657 ("Srinivasachar").

This rejection is respectfully traversed. Applicant does not admit that claims 1-20 are obvious over claims 8-20 of application no. 15/589,359 in view of Srinivasachar.

As the current pending claims stand rejected on other grounds and are still subject to amendment, Applicant takes note of these rejections but do not take further action at this time inasmuch as no pending claim has yet to be allowed. Applicant will re-evaluate this rejection, upon an indication of allowable claims.

VIII(J). The Office Action provisionally rejected claims 1-20 for nonstatutory double patenting over claims 1-20 of application no. 15/853,029 in view of U.S. patent no. 5,435,980 ("Felsvang") and U.S. patent publication no. 2004/0244657 ("Srinivasachar").

This rejection is respectfully traversed. Applicant does not admit that claims 1-20 are obvious over claims 1-20 of application no. 15/853,029 in view of Felsvang and Srinivasachar.

As the current pending claims stand rejected on other grounds and are still subject to amendment, Applicant takes note of these rejections but do not take further action at this time inasmuch as no pending claim has yet to be allowed. Applicant will re-evaluate this rejection, upon an indication of allowable claims.

VIII(K). The Office Action provisionally rejected claims 1-20 for nonstatutory double patenting over claims 1-20 of application no. 14/712,558 in view of U.S. patent publication no. 2004/0244657 ("Srinivasachar").

This rejection is respectfully traversed. Applicant does not admit that claims 1-20 are obvious over claims 1-20 of application no. 14/712,558 in view of Srinivasachar.

As the current pending claims stand rejected on other grounds and are still subject to amendment, Applicant takes note of these rejections but do not take further action at this time inasmuch as no pending claim has yet to be allowed. Applicant will re-evaluate this rejection, upon an indication of allowable claims.

VIII(L). The Office Action provisionally rejected claims 1-20 for nonstatutory double patenting over claims 1-20 of application no. 15/951970 in view of U.S. patent publication no. 2004/0244657 ("Srinivasachar").

This rejection is respectfully traversed. Applicant does not admit that claims 1-20 are obvious over claims 1-20 of application no. 15/951970 in view of Srinivasachar.

As the current pending claims stand rejected on other grounds and are still subject to amendment, Applicant takes note of these rejections but do not take further action at this time inasmuch as no pending claim has yet to be allowed. Applicant will re-evaluate this rejection, upon an indication of allowable claims.

VIII(M). The Office Action provisionally rejected claims 1-20 for nonstatutory double patenting over claims 1-20 of application no. 15/382,114 in view of U.S. patent no. 5,435,980 ("Felsvang") and U.S. patent publication no. 2004/0244657 ("Srinivasachar").

This rejection is respectfully traversed. Applicant does not admit that claims 1-20 are obvious over claims 1-20 of application no. 15/382,114 in view of Felsvang and Srinivasachar.

As the current pending claims stand rejected on other grounds and are still subject to amendment, Applicant takes note of these rejections but do not take further action at this time inasmuch as no pending claim has yet to be allowed. Applicant will re-evaluate this rejection, upon an indication of allowable claims.

VIII(N). The Office Action provisionally rejected claims 1-20 for nonstatutory double patenting over claims 1-20 of application no. 15/853,029 in view of U.S. patent no. 5,435,980 ("Felsvang") and U.S. patent publication no. 2004/0244657 ("Srinivasachar").

This rejection is respectfully traversed. Applicant does not admit that claims 1-20 are obvious over claims 1-20 of application no. 15/853,029 in view of Felsvang and Srinivasachar.

As the current pending claims stand rejected on other grounds and are still subject to amendment, Applicant takes note of these rejections but do not take further action at this time inasmuch as no pending claim has yet to be allowed. Applicant will re-evaluate this rejection, upon an indication of allowable claims.

VIII(O). The Office Action provisionally rejected claims 1-20 for nonstatutory double patenting over claims 1-20 of application no. 15/978,760 in view of U.S. patent no. 5,435,980 ("Felsvang") and U.S. patent publication no. 2004/0244657 ("Srinivasachar").

This rejection is respectfully traversed. Applicant does not admit that claims 1-20 are obvious over claims 1-20 of application no. 15/978,760 in view of Felsvang and Srinivasachar.

As the current pending claims stand rejected on other grounds and are still subject to amendment, Applicant takes note of these rejections but do not take further action at this time inasmuch as no pending claim has yet to be allowed. Applicant will re-evaluate this rejection, upon an indication of allowable claims.

VIII(P). The Office Action provisionally rejected claims 1-20 for nonstatutory double patenting over claims 1-10, 14, and 21-25 of application no. 15/974,343 in view of U.S. patent publication no. 2004/0244657 ("Srinivasachar").

This rejection is respectfully traversed. Applicant does not admit that claims 1-20 are obvious over claims 1-10, 14, and 21-25 of application no. 15/974,343 in view of Srinivasachar.

As the current pending claims stand rejected on other grounds and are still subject to amendment, Applicant takes note of these rejections but do not take further action at this time inasmuch as no pending claim has yet to be allowed. Applicant will re-evaluate this rejection, upon an indication of allowable claims.

VIII(Q). The Office Action provisionally rejected claims 1-20 for nonstatutory double patenting over claims 1, 9, 10, 12, 16, and 18-42 of application no. 15/997,091 in view of U.S. patent publication no. 2004/0244657 ("Srinivasachar").

This rejection is respectfully traversed. Applicant does not admit that claims 1-20 are obvious over claims 1, 9, 10, 12, 16, and 18-42 of application no. 15/997,091 in view of Srinivasachar.

As the current pending claims stand rejected on other grounds and are still subject to amendment, Applicant takes note of these rejections but do not take further action at this time inasmuch as no pending claim has yet to be allowed. Applicant will re-evaluate this rejection, upon an indication of allowable claims.

VIII(R). The Office Action provisionally rejected claims 1-20 for nonstatutory double patenting over claims 1-29 of application no. 16/509,071.

This rejection is respectfully traversed. Applicant does not admit that claims 1-20 are obvious over claims 1-29 of application no. 16/509,071.

As the current pending claims stand rejected on other grounds and are still subject to amendment, Applicant takes note of these rejections but do not take further action at this time inasmuch as no pending claim has yet to be allowed. Applicant will re-evaluate this rejection, upon an indication of allowable claims.

VIII(S). The Office Action provisionally rejected claims 1-20 for nonstatutory double patenting over claims 1-29 of application no. 16/509,146.

This rejection is respectfully traversed. Applicant does not admit that claims 1-20 are obvious over claims 1-29 of application no. 16/509,146.

As the current pending claims stand rejected on other grounds and are still subject to amendment, Applicant takes note of these rejections but do not take further action at this time inasmuch as no pending claim has yet to be allowed. Applicant will re-evaluate this rejection, upon an indication of allowable claims.

CONCLUSION

It is respectfully submitted that the claims are in condition for allowance, and notification to that effect is earnestly requested. The Examiner is invited to telephone the undersigned at (612) 349-9580 to facilitate prosecution of this application.

If necessary, please charge any additional fees or deficiencies, or credit any overpayments to Deposit Account No. 19-0743.

Respectfully submitted,

SCHWEGMAN LUNDBERG & WOESSNER, P.A.
P.O. Box 2938
Minneapolis, MN 55402-0938
(612) 373-6900

Date November 25, 2020

By /Nicholas P. Lanzatella/

Nicholas P. Lanzatella
Reg. No. 63,803

IN THE CLAIMS

Please amend the claims as follows:

1. (Currently Amended) A method for separating mercury from a mercury-containing gas, the method comprising:

combusting a mixture of coal and an additive in a combustion chamber, to form the mercury-containing gas, wherein the additive is chosen from halides, halogens, salts thereof, and combinations thereof;

adding a particulate sorbent material comprising activated carbon into the mercury-containing gas, wherein a weight ratio of I^-, I_2, Br^-, Br_2, Cl^-, Cl_2, or a combination thereof in the additive fed to the combustion chamber to an amount of the sorbent material added to the mercury-containing gas is from 1:100 to 30:100;

~~promoting at least a portion of the sorbent material by chemically reacting the sorbent material with the additive in the mercury-containing gas, to form a promoted sorbent material;~~

~~chemically reacting~~contacting mercury in the mercury-containing gas with the promoted sorbent material, to form a mercury-sorbent; and

separating the mercury-sorbent from the mercury-containing gas.

2. (Currently Amended) The method of claim 1, further comprising ~~mixing~~adding the additive [[and]]to the coal before combusting the coal.

3. (Original) The method of claim 2, wherein the additive mixed with the coal is in a liquid.

4. (Currently Amended) The method of claim 3, wherein the liquid is a hydrocarbon, ~~a chlorinated hydrocarbon,~~ supercritical carbon dioxide, water, salt solution, or a combination thereof.

5. (Canceled)

6. (Canceled)

7. (Canceled)

8. (Original) The method of claim 1, wherein carbocations in the activated carbon of the mercury-sorbent accept electrons from mercury atoms of the mercury-sorbent.

9. (Original) The method of claim 1, wherein the additive is substantially in vapor form in the mercury-containing gas.

10. (Original) The method of claim 1, wherein the additive is chosen from Cl_2, HCl, sodium chloride, calcium chloride, and a combination thereof.

11. (Original) The method of claim 1, wherein the additive is chosen from Br_2, HBr, sodium bromide, calcium bromide, and a combination thereof.

12. (Original) The method of claim 1, wherein the additive is chosen from PBr_3, SCl_2, and a combination thereof.

13. (Original) The method of claim 1, wherein the additive is chosen from I_2, HI, sodium iodide, calcium iodide, potassium iodide, and combinations thereof.

14. (Original) The method of claim 1, wherein the activated carbon is chosen from powdered activated carbon, granular activated carbon, carbon black, carbon fiber, aerogel carbon, pyrolysis char, and combinations thereof.

15. (Original) The method of claim 1, further comprising adding an alkaline component to the mercury-containing gas, the alkaline component chosen from alkali elements, alkaline earth elements, alkali salts, alkaline earth salts, and combinations thereof.

16. (Currently Amended) The method of claim 1, further comprising contacting ~~at least one of~~ the sorbent material ~~and the promoted sorbent material~~ with a mercury-stabilizing reagent chosen from S, Se, H_2S, SO_2, H_2Se, SeO_2, CS_2, P_2S_5, and combinations thereof.

17. (Currently Amended) The method of claim 1, further comprising contacting ~~at least one of~~ the sorbent material ~~and the promoted sorbent material~~ with a secondary component chosen from halogens, hydrogen halides, Group V halides, Group VI halides, and combinations thereof.

18. (Original) The method of claim 17, wherein the secondary component is chosen from halogens, halide salts, HI, HBr, HCl, and combinations thereof.

19. (Original) The method of claim 17, wherein the secondary component is chosen from I_2, HI, and combinations thereof.

20. (Currently Amended) A method for separating mercury from a mercury-containing gas, the method comprising:

combusting ~~coal mixed with an additive~~coal comprising an additive in a combustion chamber, wherein the additive is chosen from halides, halogens, salts thereof, and combinations thereof, to form the mercury-containing gas, wherein the additive comprises a and b, b and c, a and c, or a, b, and c:

a) I^- and/or I_2,

b) Br^- and/or Br_2,

c) Cl^- and/or Cl_2;

injecting a sorbent material comprising activated carbon into the mercury-containing gas, wherein a weight ratio of I^-, I_2, Br^-, Br_2, Cl^-, Cl_2, or a combination thereof in the additive fed to the combustion chamber to an amount of the sorbent material added to the mercury-containing gas is from 1:100 to 30:100;

~~promoting at least a portion of the sorbent material by chemically reacting the sorbent material with the additive in the mercury-containing gas, to form a promoted sorbent material;~~

~~chemically reacting~~contacting mercury in the mercury-containing gas with the ~~promoted~~ sorbent material, to form a mercury-sorbent; and

separating the mercury-sorbent from the mercury-containing gas;

~~wherein the coal and promoter are mixed at a location upstream of a combustion furnace where combustion occurs.~~

21. (New) The method of claim 1, wherein the additive comprises a and b, b and c, a and c, or a, b, and c:

a) I^- and/or I_2,

b) Br^- and/or Br_2,

c) Cl^- and/or Cl_2.

22. (New) The method of claim 1, wherein a weight ratio of I^-, I_2, Br^-, Br_2, or a combination thereof in the additive fed to the combustion chamber to an amount of the sorbent material added to the mercury-containing gas is from 1:100 to 30:100.

23. (New) The method of claim 1, wherein a weight ratio of I^-, Br^-, Br_2, or a combination thereof in the additive fed to the combustion chamber to an amount of the sorbent material added to the mercury-containing gas is from 1:100 to 30:100.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s): Edwin S. Olson et al. Examiner: AMBER ROSE ORLANDO
Application No.: 15/951,970 Group Art Unit: 1776
Filed: April 12, 2018 Docket No.: 4820.001US8
Confirmation No.: 6410
Title: SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY

AMENDMENT & RESPONSE UNDER 37 C.F.R. § 1.111

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Applicant has received the Non-Final Office Action dated May 27, 2020. Please enter the following.

This response is accompanied by a Petition, as well as the appropriate fee, to obtain a three-month extension of the period for responding to the Office Action, thereby moving the deadline for response from August 27, 2020 to November 27, 2020.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s): Edwin S. Olson et al.
Title: SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY

Docket No.:	4820.001US8	Serial No.:	15/951,970
Filed:	April 12, 2018	Due Date:	November 27, 2020
Examiner:	HEIDI RIVIERE KELLEY	Group Art Unit:	1773
Customer No.:	21186	Confirmation No.:	6410

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

We are transmitting herewith the following attached items (as indicated with an "X"):

X Amendment and Response under 37 C.F.R. § 1.111 (19 pgs.)
X Petition for Extension of Time (1 pg.)
X Authorization to charge Deposit Account 19-0743 in the amount of $1480.00 to cover the Extension of Time Fee.
X Supplemental Information Disclosure Statement (2 pgs.), Form 1449 (2 pgs.) Copies of Cited References (23).
X Authorization to charge Deposit Account 19-0743 in the amount of $260.00 to cover the fee for consideration of Information Disclosure Statement under 37 C.F.R. § 1.97(c).

If not provided for in a separate paper filed herewith, please consider this a PETITION FOR EXTENSION OF TIME for sufficient number of months to enter these papers and please charge any additional fees or credit overpayment to Deposit Account No. 19-0743. If applicable, any papers or fees supplied herewith are considered to be timely filed pursuant to 37 C.F.R. § 1.7(a), the response period falling on a Federal Holiday, Saturday or Sunday being extended to the next succeeding business day.

SCHWEGMAN LUNDBERG & WOESSNER, P.A.
Customer No.: 21186

By: /Nicholas P. Lanzatella/
Nicholas P. Lanzatella
Reg. No. 63,803

Electronic Patent Application Fee Transmittal

Application Number:	15951970
Filing Date:	12-Apr-2018
Title of Invention:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY
First Named Inventor/Applicant Name:	Edwin S. Olson
Filer:	Janal M. Kalis/Megan Reed
Attorney Docket Number:	4820.001US8

Filed as Large Entity

Filing Fees for Utility under 35 USC 111(a)

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Basic Filing:				
Pages:				
Claims:				
Miscellaneous-Filing:				
Petition:				
Patent-Appeals-and-Interference:				
Post-Allowance-and-Post-Issuance:				
Extension-of-Time:				

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Extension - 3 months with $0 paid	1253	1	1480	1480
Miscellaneous:				
SUBMISSION- INFORMATION DISCLOSURE STMT	1806	1	260	260
		Total in USD ($)		**1740**

Electronic Acknowledgement Receipt

EFS ID:	41230735
Application Number:	15951970
International Application Number:	
Confirmation Number:	6410
Title of Invention:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY
First Named Inventor/Applicant Name:	Edwin S. Olson
Customer Number:	21186
Filer:	Janal M. Kalis/Megan Reed
Filer Authorized By:	Janal M. Kalis
Attorney Docket Number:	4820.001US8
Receipt Date:	25-NOV-2020
Filing Date:	12-APR-2018
Time Stamp:	16:44:10
Application Type:	Utility under 35 USC 111(a)

Payment information:

Submitted with Payment	yes
Payment Type	DA
Payment was successfully received in RAM	$1740
RAM confirmation Number	E2020AOG44297099
Deposit Account	
Authorized User	

The Director of the USPTO is hereby authorized to charge indicated fees and credit any overpayment as follows:

File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1		SIGNED_4820_001US8_NFOAR espExtSIDS.pdf	307669 660c5b3dce350d02289c24345d09236972d729e4	yes	25

Multipart Description/PDF files in .zip description

Document Description	Start	End
Information Disclosure Statement (IDS) Form (SB08)	24	25
Transmittal Letter	22	23
Extension of Time	21	21
Applicant Arguments/Remarks Made in an Amendment	7	20
Claims	3	6
Amendment/Req. Reconsideration-After Non-Final Reject	2	2
Transmittal Letter	1	1

Warnings:

Information:

2	Non Patent Literature	0001_2_npl_4820001u11_aarf_05122020_f.pdf	513189 229f27aaf52041d21e193f499fee089cf65d0a3d	no	31

Warnings:

Information:

3	Non Patent Literature	0002_2_npl_4820001u11_foar_12122019_f.pdf	996874 b0fb5c78b37d3278470083a50515ead3526559c0	no	23

Warnings:

Information:

4	Non Patent Literature	0003_2_npl_4820001u11_oarn_05292020_f.pdf	1326540 7bfc2b50f8f89c3bf1f7dccfb9e700df350991da	no	31
Warnings:					
Information:					
5	Non Patent Literature	0004_2_npl_4820001u12_foar_02142020_f.pdf	612179 faa201998cd074fe14d2dbcda71582756f0146d0	no	13
Warnings:					
Information:					
6	Non Patent Literature	0005_2_npl_4820001us7_noar_12132019_f.pdf	503008 4a0029b80d0b7773e022b76c30c8c5dfd713fc13	no	7
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7	Non Patent Literature	0006_2_npl_4820001us9_noar_04132020_f.pdf	500080 e65bb9a21496b7867879217ec645582875a4353d	no	7
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Information:					
8	Non Patent Literature	0007_2_npl_4820001us9_nonfinaloaresponse_f.pdf	246734 578d905e83bb040aa71cdf34d38206ad94254005	no	13
Warnings:					
Information:					
9	Non Patent Literature	0008_2_npl_4820001us9_oarnpe_12022019_f.pdf	495911 58240cb34e47d19070653e4849e041264e2b0cb4	no	12
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Information:					
10	Non Patent Literature	0009_2_npl_4820003us3_aarn_09162020_f.pdf	215157 9cbedec05be26e08a94c138e122c7c00c95ffb51	no	12
Warnings:					
Information:					

11	Non Patent Literature	0010_2_npl_4820003us3_oarn_09092020_f.pdf	716156 4c0c8b1ecc498d56f0dc518865a8d8f96ff70a17	no	17
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Information:					
12	Non Patent Literature	0011_2_npl_4820004us1_aarn_06302020_f.pdf	190715 4e883270985 8d61ca335df92ce99552eea22de4f	no	21
Warnings:					
Information:					
13	Non Patent Literature	0012_2_npl_4820004us1_noar_08102020_f.pdf	633874 da84c7bdc54c0f699049255af3b1e47bb6e4ce44	no	11
Warnings:					
Information:					
14	Non Patent Literature	0013_2_npl_4820004us1_oarn_12312019_f.pdf	1599513 d7cdbab178787fd223f659a30d2ae131596cb750	no	33
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Information:					
15	Non Patent Literature	0014_2_npl_4820005us2_aarn_06162020_f.pdf	187372 b6fe36dc0d1b834e0c06e9f9848c50e579aa4e7a	no	14
Warnings:					
Information:					
16	Non Patent Literature	0015_2_npl_4820005us2_foar_08242020_f.pdf	481670 8ccdecfa615973e02b6ee14fba52cae7ea93ca2c	no	11
Warnings:					
Information:					
17	Non Patent Literature	0016_2_npl_4820005us2_oarn_12162019_f.pdf	272067 6f59421deb1f994b116271457037bf764f968019	no	8
Warnings:					
Information:					

18	Non Patent Literature	0017_2_npl_4820007us5_foar_02142020_f.pdf	1035614 d79db5cb806ed3e17e3c9b7fc6581414857f4d3e	no	23
Warnings:					
Information:					
19	Non Patent Literature	0018_2_npl_4820018us1_rerr_05212020_f.pdf	338012 56a717079a66264af8e94d5aa6d1ebed19cd0f88	no	8
Warnings:					
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20	Non Patent Literature	0019_2_npl_4820018us1_rrr1mo_11202020_f.pdf	172137 d5dbec8d1896910d832eaf36f5fe299eea5717a9	no	10
Warnings:					
Information:					
21	Non Patent Literature	0020_2_npl_cabot_activated_carbon_2017_f.pdf	40662 76e8c9d02cdc51f1eb4859b7e22e9f98dbd00eea	no	2
Warnings:					
Information:					
22	Non Patent Literature	0021_2_npl_fluegas_calc_f.pdf	61587 6d8c0744fbcdb5d6d3fd5b68d1ed1fc1bf32da08	no	1
Warnings:					
Information:					
23	Non Patent Literature	0022_2_npl_steam_table8b_f.pdf	423649 409f2627c0e6a82703a20b2485ec6fc96ff7be09	no	2
Warnings:					
The page size in the PDF is too large. The pages should be 8.5 x 11 or A4. If this PDF is submitted, the pages will be resized upon entry into the Image File Wrapper and may affect subsequent processing					
Information:					
24	Non Patent Literature	0023_2_npl_wikipedia_activated_carbon_f.pdf	793611 3451dccef4a16714dc86ff750d9040f6decb25ca	no	21
Warnings:					
Information:					

25	Fee Worksheet (SB06)	fee-info.pdf	32444 5dcb15dd786394588411 00ffe8479f23c37f baff	no	2

Warnings:	
Information:	
Total Files Size (in bytes):	12696424

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
15/951,970	04/12/2018	Edwin S. Olson	4820.001US8	6410

21186 7590 05/27/2020
SCHWEGMAN LUNDBERG & WOESSNER, P.A.
P.O. BOX 2938
MINNEAPOLIS, MN 55402

EXAMINER	
ORLANDO, AMBER ROSE	

ART UNIT	PAPER NUMBER
1776	

NOTIFICATION DATE	DELIVERY MODE
05/27/2020	ELECTRONIC

Please find below and/or attached an Office communication concerning this application or proceeding.

The time period for reply, if any, is set in the attached communication.

Notice of the Office communication was sent electronically on above-indicated "Notification Date" to the following e-mail address(es):

SLW@blackhillsip.com
uspto@slwip.com

Office Action Summary	Application No. 15/951,970	Applicant(s) Olson et al.	
	Examiner AMBER R ORLANDO	Art Unit 1776	AIA (FITF) Status No

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTHS FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

Status

1)☑ Responsive to communication(s) filed on 04/12/2018.

☐ A declaration(s)/affidavit(s) under **37 CFR 1.130(b)** was/were filed on _____.

2a)☐ This action is **FINAL.** 2b) ☑ This action is non-final.

3)☐ An election was made by the applicant in response to a restriction requirement set forth during the interview on _____; the restriction requirement and election have been incorporated into this action.

4)☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

Disposition of Claims*

5) ☑ Claim(s) 1-20 is/are pending in the application.

5a) Of the above claim(s) _____ is/are withdrawn from consideration.

6) ☐ Claim(s) _____ is/are allowed.

7) ☑ Claim(s) 1-20 is/are rejected.

8) ☐ Claim(s) _____ is/are objected to.

9) ☐ Claim(s) _____ are subject to restriction and/or election requirement

* If any claims have been determined allowable, you may be eligible to benefit from the **Patent Prosecution Highway** program at a participating intellectual property office for the corresponding application. For more information, please see http://www.uspto.gov/patents/init_events/pph/index.jsp or send an inquiry to **PPHfeedback@uspto.gov.**

Application Papers

10)☐ The specification is objected to by the Examiner.

11)☑ The drawing(s) filed on 04/12/2018 is/are: a)☑ accepted or b)☐ objected to by the Examiner.

Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).

Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).

Priority under 35 U.S.C. § 119

12)☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).

Certified copies:

a)☐ All b)☐ Some** c)☐ None of the:

1.☐ Certified copies of the priority documents have been received.

2.☐ Certified copies of the priority documents have been received in Application No. _____.

3.☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

** See the attached detailed Office action for a list of the certified copies not received.

Attachment(s)

1) ☑ Notice of References Cited (PTO-892)

2) ☑ Information Disclosure Statement(s) (PTO/SB/08a and/or PTO/SB/08b) Paper No(s)/Mail Date _____.

3) ☐ Interview Summary (PTO-413) Paper No(s)/Mail Date _____.

4) ☐ Other: _____.

Notice of Pre-AIA or AIA Status

The present application is being examined under the pre-AIA first to invent provisions.

DETAILED ACTION

Notice of Pre-AIA or AIA Status

The present application is being examined under the pre-AIA first to invent provisions.

Claim Rejections - 35 USC § 102

In the event the determination of the status of the application as subject to AIA 35 U.S.C. 102 and 103 (or as subject to pre-AIA 35 U.S.C. 102 and 103) is incorrect, any correction of the statutory basis for the rejection will not be considered a new ground of rejection if the prior art relied upon, and the rationale supporting the rejection, would be the same under either status.

The following is a quotation of the appropriate paragraphs of pre-AIA 35 U.S.C. 102 that form the basis for the rejections under this section made in this Office action:

> A person shall be entitled to a patent unless –
>
> (b) the invention was patented or described in a printed publication in this or a foreign country or in public use or on sale in this country, more than one year prior to the date of application for patent in the United States.

Claim(s) 1-2, 5-10, 14-15, 17, and 20 is/are rejected under pre-AIA 35 U.S.C. 102(b) as being anticipated by Felsvang et al. US 5,435,980.

For claims 1, and 20 the Felsvang et al. reference discloses a method for separating mercury from a mercury-containing gas, the method comprising: combusting a mixture of coal and an additive, wherein the additive is chosen from halides, halogens, salts thereof, and combinations thereof to form the mercury-containing gas (column 4, lines 39-50), wherein the additive is chosen from halides, halogens, salts thereof, and combinations thereof (column 4, lines 39-50); adding/injecting a particulate sorbent material comprising activated carbon into the mercury-containing gas (column 6, lines 6-14); promoting at least a portion of the sorbent material by chemically reacting the sorbent material with the

additive in the mercury-containing gas, to form a promoted sorbent material (column 6, lines 6-14 as both the additive and the activated carbon are being added, this promotion would inherently take place); chemically reacting mercury in the mercury-containing gas with the promoted sorbent material, to form a mercury-sorbent; and separating the mercury-sorbent from the mercury-containing gas (column 6, lines 6-14 and figure, objects 2 and 8) separating the mercury –sorbent from the mercury-containing gas (figure objects 2 and 8); wherein the coal and promoter are mixed at a location upstream of a combustion furnace where combustion occurs (column 4, lines 39-50).

For claim 2, the Felsvang et al. reference discloses mixing the additive and the coal before combusting the coal (column 4, lines 39-45).

For claims 5 and 6, one having ordinary skill in the art before the invention was made would recognize that contacting the activated carbon with the additive would inherently produce the same result as that of the present invention. Therefore Felsvang et al. reference discloses the claimed promoted sorbent material (as shown above) and is being made in the same manner as that of the claimed invention, it too would have active sites, wherein the active sites comprise halide anions bound to the activated carbon and comprises active sites and wherein the active sites comprise carbocations bound to halide anions.

For claims 7 and 8, one having ordinary skill in the art before the invention was made would recognize that contacting the activated carbon with the additive would inherently produce the same result as that of the present invention. Therefore Felsvang et al. reference discloses chemically reacting the sorbent material with the additive in the mercury-containing gas comprises chemically reacting carbene species edge sites in the activated carbon with the additive and carbocations in the activated carbon of the mercury-sorbent accept electrons from mercury atoms of the mercury-sorbent.

For claim 9, the Felsvang et al. reference discloses the additive is substantially in vapor form in the mercury-containing gas (column 4, lines 45-62).

For claim 10, the Felsvang et al. reference discloses the additive is chosen from Cl_2, HCl, sodium chloride, calcium chloride, and a combination thereof (column 4 lines 51-58).

For claim 14, the Felsvang et al. reference discloses the activated carbon is chosen from powdered activated carbon, granular activated carbon, carbon black, carbon fiber, aerogel carbon, pyrolysis char, and combinations thereof (column 4, lines 5-12).

For claim 15, the Felsvang et al. reference discloses adding an alkaline component to the mercury-containing gas, the alkaline component chosen from alkali elements, alkaline earth elements, alkali salts, alkaline earth salts, and combinations thereof (claim 3 and column 4, lines 24-34).

For claim 17, the Felsvang et al. reference discloses contacting at least one of the sorbent material and the promoted sorbent material with a secondary component chosen from halogens, hydrogen halides, Group V halides, Group VI halides, and combinations thereof (column 6, lines 11-22).

For claim 18, the Felsvang et al. reference discloses the secondary component is chosen from halogens, halide salts, HI, HBr, HCl, and combinations thereof (column 6, lines 11-22).

Claim Rejections - 35 USC § 103

The following is a quotation of pre-AIA 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

> (a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negatived by the manner in which the invention was made.

The factual inquiries set forth in *Graham v. John Deere Co.*, 383 U.S. 1, 148 USPQ 459 (1966), that are applied for establishing a background for determining obviousness under pre-AIA 35 U.S.C. 103(a) are summarized as follows:

1. Determining the scope and contents of the prior art.

2. Ascertaining the differences between the prior art and the claims at issue.

3. Resolving the level of ordinary skill in the pertinent art.

4. Considering objective evidence present in the application indicating obviousness or nonobviousness.

This application currently names joint inventors. In considering patentability of the claims under pre-AIA 35 U.S.C. 103(a), the examiner presumes that the subject matter of the various claims was commonly owned at the time any inventions covered therein were made absent any evidence to the contrary. Applicant is advised of the obligation under 37 CFR 1.56 to point out the inventor and invention dates of each claim that was not commonly owned at the time a later invention was made in order for the examiner to consider the applicability of pre-AIA 35 U.S.C. 103(c) and potential pre-AIA 35 U.S.C. 102(e), (f) or (g) prior art under pre-AIA 35 U.S.C. 103(a).

Claims 3 and 4 is/are rejected under pre-AIA 35 U.S.C. 103(a) as being unpatentable over Felsvang et al. US 5,435,980 as applied in claim 2 above, and further in view of Vosteen et al. US2004/0086439.

For claims 3 and 4, the Felsvang et al. reference does not explicitly state that the additive mixed with the coal is in a liquid and wherein the liquid is a hydrocarbon, a chlorinated hydrocarbon, supercritical carbon dioxide, water, salt solution, or a combination thereof.

The Felsvang et al. reference does disclose that additive can being in and aqueous solution and wherein the liquid is a hydrocarbon, a chlorinated hydrocarbon, supercritical carbon dioxide, water, salt solution, or a combination thereof (column 4, lines 23-30 and 39-42).

The Vosteen et al. reference discloses the additive is mixed with the coal and is a liquid and wherein the liquid is a hydrocarbon, a chlorinated hydrocarbon, supercritical carbon dioxide, water, salt solution, or a combination thereof (paragraph [0014]).

It would have been obvious to one having ordinary skill in the art before the invention was made to have modifier the Felsvang et al. reference to include the additive is mixed with the coal and is a liquid and wherein the liquid is a hydrocarbon, a chlorinated hydrocarbon, supercritical carbon dioxide, water, salt solution, or a combination thereof (Vosteen et al. paragraph [0014]) so as to allow the additive to effectively contact the component to be burnt.

Claim 11 is/are rejected under pre-AIA 35 U.S.C. 103(a) as being unpatentable over Felsvang et al. US 5,435,980 as applied in claim 1 above, and further in view of Vosteen et al. US2004/0086439.

For claim 11, the Felsvang et al. reference does not disclose the additive is chosen from Br_2, HBr, sodium bromide, calcium bromide, and a combination thereof.

The Vosteen et al. reference discloses the additive is chosen from Br_2, HBr, sodium bromide, calcium bromide, and a combination thereof (paragraph [0010]).

It would have been obvious to one having ordinary skill in the art to before the invention was made to have modified the Felsvang et al. reference to include the additive is chosen from Br_2, HBr, sodium bromide, calcium bromide, and a combination thereof (Vosteen et al. paragraph [0010]) so as to provide a cost efficiency and effective removal means for mercury from flue gas.

Claim 12 is/are rejected under pre-AIA 35 U.S.C. 103(a) as being unpatentable over Felsvang et al. US 5,435,980 as applied in claim 1 above, and further in view of Chang US 2006/0057044.

For claim 12, the Felsvang et al. reference does not disclose the additive is chosen from PBr, SCl_2, and a combination thereof.

The Chang et al. reference discloses an additive is chosen from PBr, SCl_2, and a combination thereof (abstract).

It would have been obvious to one having ordinary skill in the art before the invention was made to have modified the Felsvang et al. reference to include in addition or in place of the chlorine the additive is chosen from PBr, SCl_2, and a combination thereof (Chang et al. abstract) so as to aid in the

removal of mercury from the gas stream. Further the courts have held it to be within the general skill of a worker in the art to select a known material on the basis of its suitability for the intended use as a matter of obvious design choice. *In re Leshin,* 125 USPQ 416.

Claim 13 is/are rejected under pre-AIA 35 U.S.C. 103(a) as being unpatentable over Felsvang et al. US 5,435,980 as applied in claim 1 above, and further in view of Vosteen et al. US2004/0086439 and Srinivasachar et al. US2004/0244657.

For claim 13, the Felsvang et al. reference does not disclose the additive is chosen from I_2, HI, sodium iodide, calcium iodide, potassium iodide, and combinations thereof.

The Vosteen et al. reference recognizes that iodine compounds are more effective than that of chlorine or bromine (paragraph [0017]).

The Srinivasachar et al. reference discloses an additive is chosen from I_2, HI, sodium iodide, calcium iodide, potassium iodide, and combinations thereof (paragraph [0031]).

It would have been obvious to one having ordinary skill in the art before the invention was made to have modified the Felsvang et al. reference to include in addition to or in preplacement of the chloride component an additive chosen from I_2, HI, sodium iodide, calcium iodide, potassium iodide, and combinations thereof (Srinivasachar et al. paragraph [0031] and Vosteen et al. paragraph [0017]) so as to obtain a desired effectiveness as well as cost for the removal of mercury from the flue gas.

Claim 16 is/are rejected under pre-AIA 35 U.S.C. 103(a) as being unpatentable over Felsvang et al. US 5,435,980 as applied in claim 1 above, and further in view of Vosteen et al. US2004/0086439.

For claim 16, the Felsvang et al. does not disclose contacting at least one of the sorbent material and the promoted sorbent material with a mercury-stabilizing reagent chosen from S, Se, ThS, SO2, ThSe, SeCh, CS_2, P_2S_5, and combinations thereof.

The Gadkaree et a. reference discloses at least one of the sorbent material and the promoted sorbent material with a mercury-stabilizing reagent chosen from S, Se, ThS, SO2, ThSe, SeCh, CS_2, P_2S_5, and combinations thereof (abstract and column 2, lines 29-35).

It would have been obvious to one having ordinary skill in the art before the invention was made to have modified the Felsvang et al. reference to include at least one of the sorbent material and the promoted sorbent material with a mercury-stabilizing reagent chosen from S, Se, ThS, SO2, ThSe, SeCh, CS_2, P_2S_5, and combinations thereof (Gadgaree et al. abstract and column 2, lines 29-35) so as to provide an efficient means for removing mercury from flue gas.

Claim 19 is/are rejected under pre-AIA 35 U.S.C. 103(a) as being unpatentable over Felsvang et al. US 5,435,980 as applied in claim 17 above, and further in view of Vosteen et al. US2004/0086439 and Srinivasachar et al. US2004/0244657.

For claim 19, the Felsvang et al. reference does not disclose the secondary component is chosen from I_2, HI, and combinations thereof.

The Vosteen et al. reference recognizes that iodine compounds are more effective than that of chlorine or bromine (paragraph [0017]).

The Srinivasachar et al. reference discloses an additive is chosen from I_2, HI, sodium iodide, calcium iodide, potassium iodide, and combinations thereof (paragraph [0031]).

It would have been obvious to one having ordinary skill in the art before the invention was made to have modified the Felsvang et al. reference to include in addition to or in preplacement of the chloride component an additive chosen from I_2, HI, sodium iodide, calcium iodide, potassium iodide, and combinations thereof (Srinivasachar et al. paragraph [0031] and Vosteen et al. paragraph [0017]) so as to obtain a desired effectiveness as well as cost for the removal of mercury from the flue gas.

Double Patenting

The nonstatutory double patenting rejection is based on a judicially created doctrine grounded in public policy (a policy reflected in the statute) so as to prevent the unjustified or improper timewise extension of the "right to exclude" granted by a patent and to prevent possible harassment by multiple assignees. A nonstatutory double patenting rejection is appropriate where the conflicting claims are not identical, but at least one examined application claim is not patentably distinct from the reference claim(s) because the examined application claim is either anticipated by, or would have been obvious over, the reference claim(s). See, e.g., *In re Berg*, 140 F.3d 1428, 46 USPQ2d 1226 (Fed. Cir. 1998); *In re Goodman*, 11 F.3d 1046, 29 USPQ2d 2010 (Fed. Cir. 1993); *In re Longi*, 759 F.2d 887, 225 USPQ 645 (Fed. Cir. 1985); *In re Van Ornum*, 686 F.2d 937, 214 USPQ 761 (CCPA 1982); *In re Vogel*, 422 F.2d 438, 164 USPQ 619 (CCPA 1970); *In re Thorington*, 418 F.2d 528, 163 USPQ 644 (CCPA 1969).

A timely filed terminal disclaimer in compliance with 37 CFR 1.321(c) or 1.321(d) may be used to overcome an actual or provisional rejection based on nonstatutory double patenting provided the reference application or patent either is shown to be commonly owned with the examined application, or claims an invention made as a result of activities undertaken within the scope of a joint research agreement. See MPEP § 717.02 for applications subject to examination under the first inventor to file provisions of the AIA as explained in MPEP § 2159. See MPEP §§ 706.02(l)(1) - 706.02(l)(3) for applications not subject to examination under the first inventor to file provisions of the AIA. A terminal disclaimer must be signed in compliance with 37 CFR 1.321(b).

The USPTO Internet website contains terminal disclaimer forms which may be used. Please visit www.uspto.gov/patent/patents-forms. The filing date of the application in which the form is filed determines what form (e.g., PTO/SB/25, PTO/SB/26, PTO/AIA/25, or PTO/AIA/26) should be used. A web-based eTerminal Disclaimer may be filled out completely online using web-screens. An eTerminal Disclaimer that meets all requirements is auto-processed and approved immediately upon submission.

For more information about eTerminal Disclaimers, refer to

www.uspto.gov/patents/process/file/efs/guidance/eTD-info-I.jsp.

Claims 1-20 are rejected on the ground of nonstatutory double patenting as being unpatentable over claims 1-30 of U.S. Patent No. US 7,435,286 ('286) in view of Felsvang et al. US 5,435,980 and Srinivasachar et al. US 2004/0244657.

'286 discloses the claimed invention except for mixing the coal and the additive prior to the furnace and the additive comprising I_2, HI, an iodide compound or a combination thereof.

Felsvang et al. and Srinivasachar et al. disclose this limitation as shown above.

It would have been obvious to one having ordinary skill in the art to have modified the '286 reference to include for mixing the coal and the additive prior to the furnace and the additive comprising I_2, HI, an iodide compound or a combination thereof (Felsvang et al. and Srinivasachar et al. as shown above) so as to supplement the halogen content of the system so as to increase the mercury sorption capability and because the halogen compounds are recognized equivalents for the removal of Hg.

This is a provisional nonstatutory double patenting rejection because the patentably indistinct claims have not in fact been patented.

Claims 1-20 are rejected on the ground of nonstatutory double patenting as being unpatentable over claims 1-25 of U.S. Patent No. US 8,168,147 ('147) in view of Felsvang et al. US 5,435,980 and Srinivasachar et al. US 2004/0244657.

'147 discloses the claimed invention except for for mixing the coal and the additive prior to the furnace comprising I_2, HI, an iodide compound or a combination thereof.

Felsvang et al. and Srinivasachar et al. disclose this limitation as shown above.

It would have been obvious to one having ordinary skill in the art to have modified the '147 reference to include for mixing the coal and the additive prior to the furnace and the additive

comprising I_2, HI, an iodide compound or a combination thereof (Felsvang et al. and Srinivasachar et al. as shown above) so as to supplement the halogen content of the system so as to increase the mercury sorption capability and because the halogen compounds are recognized equivalents for the removal of Hg.

This is a provisional nonstatutory double patenting rejection because the patentably indistinct claims have not in fact been patented.

Claims 1-20 are rejected on the ground of nonstatutory double patenting as being unpatentable over claims 1-60 of U.S. Patent No. US 8,512,655 ('655) in view of Felsvang et al. US 5,435,980 and Srinivasachar et al. US 2004/0244657.

'655 discloses the claimed invention except for mixing the coal and the additive prior to the furnace and the additive comprising I_2, HI, an iodide compound or a combination thereof.

Felsvang et al. and Srinivasachar et al. disclose these limitations as shown above.

It would have been obvious to one having ordinary skill in the art to have modified the '655 reference to include for mixing the coal and the additive prior to the furnace and the additive comprising I_2, HI, an iodide compound or a combination thereof (Felsvang et al. and Srinivasachar et al. as shown above) so as to supplement the halogen content of the system so as to increase the mercury sorption capability and because the halogen compounds are recognized equivalents for the removal of Hg.

This is a provisional nonstatutory double patenting rejection because the patentably indistinct claims have not in fact been patented.

Claims 1-20 are rejected on the ground of nonstatutory double patenting as being unpatentable over claims 1-60 of U.S. Patent No. US 8,821,819 ('819) in view of Felsvang et al. US 5,435,980 and Srinivasachar et al. US 2004/0244657.

'819 discloses the claimed invention except for mixing the coal and the additive prior to the furnace and the additive comprising I_2, HI, an iodide compound or a combination thereof.

Felsvang et al. and Srinivasachar et al. disclose this limitation as shown above.

It would have been obvious to one having ordinary skill in the art to have modified the '819 reference to include the coal comprising the additive and the additive comprising I_2, HI, an iodide compound or a combination thereof (Felsvang et al. and Srinivasachar et al. as shown above) so as to supplement the halogen content of the system so as to increase the mercury sorption capability and because the halogen compounds are recognized equivalents for the removal of Hg.

This is a provisional nonstatutory double patenting rejection because the patentably indistinct claims have not in fact been patented.

Claims 1-20 are rejected on the ground of nonstatutory double patenting as being unpatentable over claims 1-33 of U.S. Patent No. US 9,757,689 ('689) in view of Felsvang et al. US 5,435,980 and Srinivasachar et al. US 2004/0244657.

'689 discloses the claimed invention except for for mixing the coal and the additive prior to the furnace and the additive comprising I_2, HI, an iodide compound or a combination thereof.

Felsvang et al. and Srinivasachar et al. disclose this limitation as shown above.

It would have been obvious to one having ordinary skill in the art to have modified the '689 reference to include for mixing the coal and the additive prior to the furnace and the additive comprising I_2, HI, an iodide compound or a combination thereof (Felsvang et al. and Srinivasachar et al.) so as to supplement the halogen content of the system so as to increase the mercury sorption capability and because the halogen compounds are recognized equivalents for the removal of Hg.

This is a provisional nonstatutory double patenting rejection because the patentably indistinct claims have not in fact been patented.

Claims 1-20 are rejected on the ground of nonstatutory double patenting as being unpatentable over claims 1-20 of U.S. Patent No. US 9,468,886 ('886) in view of Felsvang et al. US 5,435,980 and Srinivasachar et al. US 2004/0244657.

'886 discloses the claimed invention except for for mixing the coal and the additive prior to the furnace and the additive comprising I_2, HI, an iodide compound or a combination thereof.

Felsvang et al. and Srinivasachar et al. disclose this limitation as shown above.

It would have been obvious to one having ordinary skill in the art to have modified the '886 reference to include the coal comprising the additive and the additive comprising I_2, HI, an iodide compound or a combination thereof (Felsvang et al. and Srinivasachar et al. as shown above) so as to supplement the halogen content of the system so as to increase the mercury sorption capability and because the halogen compounds are recognized equivalents for the removal of Hg.

This is a provisional nonstatutory double patenting rejection because the patentably indistinct claims have not in fact been patented.

Claims 1-20 are rejected on the ground of nonstatutory double patenting as being unpatentable over claims 1-30 of U.S. Patent No. 8,652,235 ('235) in view of Felsvang et al. US 5,435,980 and Srinivasachar et al. US 2004/0244657.

'235 discloses the claimed invention except for mixing the coal and the additive prior to the furnace and the additive comprising I_2, HI, an iodide compound or a combination thereof.

Felsvang et al. and Srinivasachar et al. disclose this limitation as shown above.

It would have been obvious to one having ordinary skill in the art to have modified the '235 reference to include for mixing the coal and the additive prior to the furnace and the additive comprising I_2, HI, an iodide compound or a combination thereof (Felsvang et al. and Srinivasachar et al. as shown above) so as to supplement the halogen content of the system so as to increase the mercury

sorption capability and because the halogen compounds are recognized equivalents for the removal of Hg.

This is a provisional nonstatutory double patenting rejection because the patentably indistinct claims have not in fact been patented.

Claims 1-20 are provisionally rejected on the ground of nonstatutory double patenting as being unpatentable over claims 1-16 of copending Application No. 15/295594 ('594) in view of Felsvang et al. US 5,435,980 and Srinivasachar et al. US 2004/0244657.

'594 discloses the claimed invention except for mixing the coal and the additive prior to the furnace and the additive comprising I_2, HI, an iodide compound or a combination thereof.

Felsvang et al. and Srinivasachar et al. disclose this limitation as shown above.

It would have been obvious to one having ordinary skill in the art to have modified the '594 reference to include for mixing the coal and the additive prior to the furnace and the additive comprising I_2, HI, an iodide compound or a combination thereof (Felsvang et al. and Srinivasachar et al. as shown above) so as to supplement the halogen content of the system so as to increase the mercury sorption capability and because the halogen compounds are recognized equivalents for the removal of Hg.

This is a provisional nonstatutory double patenting rejection because the patentably indistinct claims have not in fact been patented.

Claims 1-20 are provisionally rejected on the ground of nonstatutory double patenting as being unpatentable over claims 8-20 of copending Application No. 15/589359 ('359) in view of Srinivasachar et al. US 2004/0244657.

'359 discloses the claimed invention except for the additive comprising I_2, HI, an iodide compound or a combination thereof.

Srinivasachar et al. discloses this limitation as shown above.

It would have been obvious to one having ordinary skill in the art to have modified the '359 reference to include the additive comprising I_2, HI, an iodide compound or a combination thereof (Srinivasachar as shown above) as the halogen compounds are recognized equivalents for the removal of Hg.

This is a provisional nonstatutory double patenting rejection because the patentably indistinct claims have not in fact been patented.

Claims 1-20 are provisionally rejected on the ground of nonstatutory double patenting as being unpatentable over claims 1-20 of copending Application No. 15/853029 ('029) in view of Felsvang et al. US 5,435,980 and Srinivasachar et al. US 2004/0244657.

'029 discloses the claimed invention except for mixing the coal and the additive prior to the furnace and the additive comprising I_2, HI, an iodide compound or a combination thereof.

Felsvang et al. and Srinivasachar et al. disclose this limitation as shown above.

It would have been obvious to one having ordinary skill in the art to have modified the '029 reference to include for mixing the coal and the additive prior to the furnace and the additive comprising I_2, HI, an iodide compound or a combination thereof (Felsvang et al. and Srinivasachar et al. as shown above) so as to supplement the halogen content of the system so as to increase the mercury sorption capability and because the halogen compounds are recognized equivalents for the removal of Hg.

This is a provisional nonstatutory double patenting rejection because the patentably indistinct claims have not in fact been patented.

Claims 1-20 are provisionally rejected on the ground of nonstatutory double patenting as being unpatentable over claims 1-20 of copending Application No. 14/712558 ('558) in view of and Srinivasachar et al. US 2004/0244657.

'558 discloses the claimed invention except for the additive comprising I_2, HI, an iodide compound or a combination thereof.

Srinivasachar et al. and disclose this limitation as shown above.

It would have been obvious to one having ordinary skill in the art to have modified the '558 reference to include the additive comprising I_2, HI, an iodide compound or a combination thereof (Srinivasachar as shown above) as the halogen compounds are recognized equivalents for the removal of Hg.

This is a provisional nonstatutory double patenting rejection because the patentably indistinct claims have not in fact been patented.

Claims 1-20 are provisionally rejected on the ground of nonstatutory double patenting as being unpatentable over claims 1-20 of copending Application No. 15/951970 ('970) in view of Srinivasachar et al. US 2004/0244657.

'970 discloses the claimed invention except for the additive comprising I_2, HI, an iodide compound or a combination thereof.

Srinivasachar et al. discloses this limitation as shown above.

It would have been obvious to one having ordinary skill in the art to have modified the '970 reference to include the additive comprising I_2, HI, an iodide compound or a combination thereof (Srinivasachar as shown above) as the halogen compounds are recognized equivalents for the removal of Hg.

This is a provisional nonstatutory double patenting rejection because the patentably indistinct claims have not in fact been patented.

Claims 1-30 are provisionally rejected on the ground of nonstatutory double patenting as being unpatentable over 1-20 of copending Application No. 15/382114 ('114) in view of Felsvang et al. US 5,435,980 and Srinivasachar et al. US 2004/0244657.

'114 discloses the claimed invention except for mixing the coal and the additive prior to the furnace and the additive comprising I_2, HI, an iodide compound or a combination thereof.

Felsvang et al. and Srinivasachar et al. disclose this limitation as shown above.

It would have been obvious to one having ordinary skill in the art to have modified the '114 reference to include for mixing the coal and the additive prior to the furnace and the additive comprising I_2, HI, an iodide compound or a combination thereof (Felsvang et al. and Srinivasachar et al. as shown above) so as to supplement the halogen content of the system so as to increase the mercury sorption capability and because the halogen compounds are recognized equivalents for the removal of Hg.

This is a provisional nonstatutory double patenting rejection because the patentably indistinct claims have not in fact been patented.

Claims 1-20 are provisionally rejected on the ground of nonstatutory double patenting as being unpatentable over 1-20 of copending Application No. 15/853029 ('029) in view of Felsvang et al. US 5,435,980 and Srinivasachar et al. US 2004/0244657.

'029 discloses the claimed invention except for mixing the coal and the additive prior to the furnace and the additive comprising I_2, HI, an iodide compound or a combination thereof.

Felsvang et al. and Srinivasachar et al. disclose this limitation as shown above.

It would have been obvious to one having ordinary skill in the art to have modified the '029 reference to include for mixing the coal and the additive prior to the furnace and the additive comprising I_2, HI, an iodide compound or a combination thereof (Felsvang et al. and Srinivasachar et al. as shown above) so as to supplement the halogen content of the system so as to increase the mercury sorption capability and because the halogen compounds are recognized equivalents for the removal of Hg.

This is a provisional nonstatutory double patenting rejection because the patentably indistinct claims have not in fact been patented.

Claims 1-20 are provisionally rejected on the ground of nonstatutory double patenting as being unpatentable over 1-20 of copending Application No. 15/978,760 ('760) in view of Felsvang et al. US 5,435,980 and Srinivasachar et al. US 2004/0244657.

'760 discloses the claimed invention except for mixing the coal and the additive prior to the furnaceand the additive comprising I_2, HI, an iodide compound or a combination thereof.

Felsvang et al. and Srinivasachar et al. disclose this limitation as shown above.

It would have been obvious to one having ordinary skill in the art to have modified the '760 reference to include for mixing the coal and the additive prior to the furnace and the additive comprising I_2, HI, an iodide compound or a combination thereof (Felsvang et al. column 5, 60-65 and Srinivasachar et al. as shown above) so as to supplement the halogen content of the system so as to increase the mercury sorption capability and because the halogen compounds are recognized equivalents for the removal of Hg.

This is a provisional nonstatutory double patenting rejection because the patentably indistinct claims have not in fact been patented.

Claims 1-20 are provisionally rejected on the ground of nonstatutory double patenting as being unpatentable over 1-10, 14 and 21-25 of copending Application No. 15/974,343 ('343) and Srinivasachar et al. US 2004/0244657.

'343 discloses the claimed invention except for the additive comprising I_2, HI, an iodide compound or a combination thereof.

Srinivasachar et al. discloses this limitation as shown above.

It would have been obvious to one having ordinary skill in the art to have modified the '343 reference to include the additive comprising I_2, HI, an iodide compound or a combination thereof

(Srinivasachar as shown above) as the halogen compounds are recognized equivalents for the removal of Hg.

This is a provisional nonstatutory double patenting rejection because the patentably indistinct claims have not in fact been patented.

Claims 1-20 are provisionally rejected on the ground of nonstatutory double patenting as being unpatentable over 1,9,10,12,16 and 18-42 of copending Application No. 15/997,091 ('091) in view of Srinivasachar et al. US 2004/0244657.

'091 discloses the claimed invention except for the additive comprising I_2, HI, an iodide compound or a combination thereof.

Felsvang et al. and disclose this limitation as shown above.

It would have been obvious to one having ordinary skill in the art to have modified the '091 reference to include the additive comprising I_2, HI, an iodide compound or a combination thereof (Srinivasachar as shown above) as the halogen compounds are recognized equivalents for the removal of Hg.

This is a provisional nonstatutory double patenting rejection because the patentably indistinct claims have not in fact been patented.

Claims 1-20 are provisionally rejected on the ground of nonstatutory double patenting as being unpatentable over claims 1-29 of copending Application No. 16/509,071('071).

'071 discloses essentially the same invention with little to no patentability distinct differences.

This is a provisional nonstatutory double patenting rejection because the patentably indistinct claims have not in fact been patented.

Claims 1-20 are provisionally rejected on the ground of nonstatutory double patenting as being unpatentable over claims 1-29 of copending Application No. 16/509,146 ('146).

'146 discloses essentially the same invention with little to no patentability distinct differences.

This is a provisional nonstatutory double patenting rejection because the patentably indistinct claims have not in fact been patented.

Conclusion

Any inquiry concerning this communication or earlier communications from the examiner should be directed to AMBER ROSE ORLANDO whose telephone number is (571)270-3149. The examiner can normally be reached on Monday-Thursday 7:00-5:30.

Examiner interviews are available via telephone, in-person, and video conferencing using a USPTO supplied web-based collaboration tool. To schedule an interview, applicant is encouraged to use the USPTO Automated Interview Request (AIR) at http://www.uspto.gov/interviewpractice.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Duane Smith can be reached on (571)272-1166. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see https://ppair-my.uspto.gov/pair/PrivatePair. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

AMBER ROSE. ORLANDO
Primary Examiner
Art Unit 1776

/AMBER R ORLANDO/
Primary Examiner, Art Unit 1776

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Search Notes	Application/Control No.	Applicant(s)/Patent Under Reexamination
	15/951,970	Olson et al.
	Examiner	Art Unit
	AMBER R ORLANDO	1776

CPC - Searched*		
Symbol	Date	Examiner

CPC Combination Sets - Searched*		
Symbol	Date	Examiner

US Classification - Searched*			
Class	Subclass	Date	Examiner

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Search Notes		
Search Notes	Date	Examiner
IDS Search (EAST and DAV)	05/21/2020	AO
Inventor Search (DAV)	05/21/2020	AO
EAST searched for sorbents for mercury from flue gases.	05/21/2020	AO

Interference Search			
US Class/CPC Symbol	US Subclass/CPC Group	Date	Examiner

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INFORMATION DISCLOSURE STATEMENT BY APPLICANT

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Complete if Known	
Application Number	15/951,970
Filing Date	April 12, 2018
First Named Inventor	Edwin S. Olson
Group Art Unit	1736
Examiner Name	Unknown

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Attorney Docket No: 4820.001US8

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/AMBER R ORLANDO/

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Group Art Unit	1736
Examiner Name	Unknown

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	Examiner Name	Unknown
Sheet 4 of 19	Attorney Docket No: 4820.001US8	

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	Application Number	15/951,970
	Filing Date	April 12, 2018
	First Named Inventor	Edwin S. Olson
	Group Art Unit	1736
	Examiner Name	Unknown
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	Application Number	15/951,970
	Filing Date	April 12, 2018
	First Named Inventor	Edwin S. Olson
	Group Art Unit	1736
	Examiner Name	Unknown
Sheet 11 of 19	Attorney Docket No: 4820.001US8	

OTHER DOCUMENTS – NON PATENT LITERATURE DOCUMENTS

Examiner Initial *	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	T1
	"Application Serial No. 14/712,558, Examiner Interview Summary mailed 03-05-18", 3 pgs	
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	"DARCO® FGD- Powdered Activated Carbon", Norit Americas Inc. Datasheet No. 1100, (May 2009), 1 pg	
	"DATABASE WPI Week 197450", Thomson Scientific, London, GB; AN 1974-86199V XP002514926	
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	"European Application Serial No. 05814011.2, Third Party Observations submitted 12-04-07", 5 pgs.	
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	"European Application Serial No. 10767465.7, Intention to Grant mailed 03-17-14", 44 pgs	
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	"European Application Serial No. 13719338.9, Office Action mailed 12-19-14", 2 pgs.	
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Substitute for form 1449A/PTO **INFORMATION DISCLOSURE STATEMENT BY APPLICANT** (Use as many sheets as necessary)	*Complete if Known*	
	Application Number	15/951,970
	Filing Date	April 12, 2018
	First Named Inventor	Edwin S. Olson
	Group Art Unit	1736
	Examiner Name	Unknown
Sheet 18 of 19	Attorney Docket No: 4820.001US8	

OTHER DOCUMENTS – NON PATENT LITERATURE DOCUMENTS

Examiner Initial *	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	T1
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S/N 15/951,970 PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s):	Edwin S. Olson et al.	Examiner:	AMBER ROSE ORLANDO
Serial No.:	15/951,970	Group Art Unit:	1776
Filed:	April 12, 2018	Docket:	4820.001US8
Customer No.:	21186	Confirmation No.:	6410
Title:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY		

COMMUNICATION CONCERNING PRIOR OR COPENDING APPLICATION(S)

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Pursuant to the guidance of MPEP §§ 2001.06(b) and 2004(9), Applicant would like to bring the following additional application(s) to the Examiner's attention. The identification of these applications is not intended to suggest that the subject matter claimed in any listed application is, or has been, substantially similar to any claim or claims in the present application.

Serial No./ Patent No.	Filing Date	Attorney Docket	Title
16/574,935	September 18, 2019	4820.003US3	ACTIVATED CARBON SORBENT INCLUDING NITROGEN AND METHODS OF USING THE SAME

Respectfully submitted,

SCHWEGMAN LUNDBERG & WOESSNER, P.A.
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Date 11/26/2019 By /Nicholas P. Lanzatella/
Nicholas P. Lanzatella
Reg. No. 63,803

/AMBER R ORLANDO/ 05/21/2020

ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /A.R.O/

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Examiner Name	AMBER ROSE ORLANDO

Sheet 1 of 2 — Attorney Docket No: 4820.001US8

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Examiner Initial *	USP Document Number	Publication Date	Name of Patentee or Applicant of cited Document
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Examiner Initial *	Foreign Document Number	Publication Date	Name of Patentee or Applicant of cited Document	T 1

OTHER DOCUMENTS – NON PATENT LITERATURE DOCUMENTS

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Sheet 2 of 2	Attorney Docket No: 4820.001US8	

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EAST Search History

EAST Search History (Prior Art)

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
L1	91	"7435286"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2020/05/21 15:54
S1	45	(95/134.ccls. or 95/142.ccls. or 110/345.ccls. or 423/210.cds.) and activated carbon and promoter	US-PGPUB; USPAT	ADJ	ON	2013/09/09 09:19
S2	0	(95/134.ccls. or 95/142.ccls. or 110/345.ccls. or 423/210.cds.) and sorben and promoter and activated carbond and mercury	US-PGPUB; USPAT	ADJ	ON	2013/09/09 09:19
S3	0	(95/134.ccls. or 95/142.ccls. or 110/345.ccls. or 423/210.cds.) and sorbent and promoter and activated carbond and mercury	US-PGPUB; USPAT	ADJ	ON	2013/09/09 09:20
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S8	13	"11209163"	US-PGPUB; USPAT	ADJ	ON	2015/03/17 12:28
S9	3	"11209163" and non carbon	US-PGPUB; USPAT	ADJ	ON	2015/03/17 12:28
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S11	0	(95/134.ccls. or 95/142.ccls. or 110/345.ccls. or 423/210.cds.) and sorbent and promoter and activated carbond and mercury	US-PGPUB; USPAT	ADJ	ON	2015/03/17 13:50
S12	116	(95/134.ccls. or 95/142.ccls. or 110/345.ccls. or 423/210.cds.) and (halogen or halide of HBr) with sorbent	US-PGPUB; USPAT	ADJ	ON	2015/03/17 13:50
S13	172	(B01D53/64, B01D2257/602, B01D2253/104, B01D2253/10, B01D53/10).cpc. and (halogen or halide of HBr) with sorbent	US-PGPUB; USPAT	ADJ	ON	2015/08/20 22:04
S14	67	(95/134.ccls. or 95/142.ccls. or 110/345.ccls. or 423/210.cds.) and activated carbon and promoter	US-PGPUB; USPAT	ADJ	ON	2015/08/20 22:04
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S16	20	"684374"	US-PGPUB; USPAT	ADJ	ON	2016/05/27 09:12
S17	1	"20040003716"	US-PGPUB; USPAT	ADJ	ON	2016/05/27 09:12
S18	68	"6848374"	US-PGPUB; USPAT	ADJ	ON	2016/05/27 09:12

S19	41	"7435286"	US-PGPUB; USPAT	ADJ	ON	2016/05/27 09:13
S20	13	"8168147"	US-PGPUB; USPAT	ADJ	ON	2016/05/27 09:13
S21	69	(95/134.ccls. or 95/142.ccls. or 110/345.ccls. or 423/210.ccls.) and activated carbon and promoter	US-PGPUB; USPAT	ADJ	ON	2016/06/07 09:06
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S25	28	"5266280"	US-PGPUB; USPAT	ADJ	ON	2016/06/07 13:41
S26	281	sorbent with mercury and (diameter or size) with ("30" or "40" or "50" or "60" or "70") with (sorbent or carbon)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2016/09/29 15:01
S27	148	sorbent with mercury and (diameter or size) with ("30" or "40" or "50" or "60" or "70") with (sorbent or carbon) and halogen	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2016/09/29 15:02
S28	159	sorbent with mercury and (diameter or size) with ("30" or "40" or "50" or "60" or "70") with (sorbent or carbon) and halogen$4	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2016/09/29 16:18
S29	206	(sorbent or activated carbon) with mercury and (diameter or size) with ("30" or "40" or "50" or "60" or "70") with (sorbent or carbon) and halogen$4 and activated carbon	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2016/09/29 16:22
S30	195	(B01D53/64, B01D2257/602, B01D2253/104, B01D2253/10,	US-PGPUB; USPAT	ADJ	ON	2016/09/30 13:48

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S34	49	"2004003716"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2016/11/16 10:57
S35	81	"6848374"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2016/11/16 10:58
S36	56	"7435286"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2016/11/16 11:02
S37	7	olson.in. and ("40" microns).clm.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2016/11/16 11:04
S38	0	("40" microns and halocarbon sorbent).clm.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2016/11/16 11:05
S39	0	(halocarbon sorbent).clm. and "40" microns and olson.in.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO;	ADJ	ON	2016/11/16 11:05

			DERWENT; IBM_TDB			
S40	65	"40" microns and olson.in.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2016/11/16 11:06
S41	3907	("40" microns).clm.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2016/11/16 11:06
S42	29	("40" microns and sorbent).clm.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2016/11/16 11:06
S43	1	(halocarbon sorbent).clm. and olson.in.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2016/11/16 11:08
S44	36	(sorbent).clm. and olson.in.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2016/11/16 11:08
S45	0	(sorbent).clm. and olson.in. and "40" micron	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2016/11/16 11:08
S46	17	"11209163"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO;	ADJ	ON	2016/11/16 11:09

			DERWENT; IBM_TDB			
S47	2	"20110233133"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2016/11/16 11:27
S48	7	"20110023427"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2016/11/16 11:28
S49	15	"2004025274"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2016/11/16 11:32
S50	11	"20040025274"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2016/11/16 11:32
S51	15	"2004025274"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2016/11/16 11:33
S52	40	"6312489"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2016/11/16 11:33
S53	30	("20030024872" | "20050045553" | "20050144916" | "4008060" | "4640779" | "4655921" | "4673503" | "4738778" | "4746432" | "4799944" | "5043000" |	US-PGPUB; USPAT; USOCR	ADJ	ON	2016/11/16 11:36

		"5051118" | "5120296" | "5174896" | "5320657" | "5512075" | "5674302" | "6152979" | "6312489" | "6315130" | "6336946" | "6398836" | "6585793" | "6598749" | "7648546").PN. OR ("7931723").URPN.				
S54	33	("4200444" | "4640779" | "5968215" | "6059851").PN. OR ("6312489").URPN.	US-PGPUB; USPAT; USOCR	ADJ	ON	2016/11/16 11:40
S55	5	"8518142"	US-PGPUB; USPAT; USOCR	ADJ	ON	2016/11/16 11:54
S56	4	"8747505"	US-PGPUB; USPAT; USOCR	ADJ	ON	2016/11/16 11:57
S57	2	"14318270"	US-PGPUB; USPAT; USOCR	ADJ	ON	2016/11/16 12:06
S58	13	pac with size with sorbent	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2016/11/16 12:13
S59	197	(B01D53/64, B01D2257/602, B01D2253/104, B01D2253/10, B01D53/10).cpc. and (halogen or halide of HBr) with sorbent	US-PGPUB; USPAT	ADJ	ON	2016/11/16 15:23
S60	0	(B01D53/64, B01D2257/602, B01D2253/104, B01D2253/10, B01D53/10).cpc. and (halogen or halide of HBr) with sorbent "4198388"	US-PGPUB; USPAT	ADJ	ON	2016/11/17 12:39
S61	10	"4198388"	US-PGPUB; USPAT	ADJ	ON	2016/11/17 12:39
S62	0	"2010083696"	US-PGPUB; USPAT	ADJ	ON	2016/11/17 13:32
S63	2	"20100083696"	US-PGPUB; USPAT	ADJ	ON	2016/11/17 13:32

S64	2	absorp$5 with mea with methanol with acid	US-PGPUB; USPAT	ADJ	ON	2016/11/17 13:36
S65	2	"20070283813"	US-PGPUB; USPAT	ADJ	ON	2016/11/17 13:39
S66	21	"4469596"	US-PGPUB; USPAT	ADJ	ON	2016/11/21 08:29
S67	4	"2003229245"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2017/05/10 10:36
S68	2	"20030229245"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2017/05/10 10:36
S69	45	"5,338,458"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2017/05/10 10:38
S70	45	"5338458"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2017/05/10 10:38
S71	213	(B01D53/64, B01D2257/602, B01D2253/104, B01D2253/10, B01D53/10).cpc. and (halogen or halide of HBr) with sorbent	US-PGPUB; USPAT	ADJ	ON	2017/05/10 11:02
S72	64	("1786483" | "1984164" | "2317857" | "20010002387" | "20010003116" | "20020134242" | "20020150516" | "20030136509" | "20040003716" | "20040013589" | "20050019240" | "20060048646" | "20060191835" | "20090062119" | "20100047146" |	US-PGPUB; USPAT	ADJ	ON	2017/05/10 11:20

		"20120183458" | "20140056787" | "3194629" | "3662523" | "3786619" | "3826618" | "3849267" | "4196173" | "4500327" | "4814152" | "4820318" | "4889698" | "4959162" | "5300137" | "5409522" | "5462908" | "5505766" | "5575982" | "5607496" | "5672323" | "5695726" | "5827352" | "5891324" | "6027551" | "6080281" | "6136072" | "6136749" | "6214304" | "6258334" | "6372187" | "6375909" | "6383981" | "6471936" | "6528030" | "6818043" | "6848374" | "6878358" | "6942840" | "6953494" | "7101631" | "7435286" | "7479263" | "7514052" | "7563311" | "7611564" | "8007749" | "8168147" | "8168149" | "8512655").PN.				
S73	3	("20090235848" | "20110076210" | "20150246315").PN.	US-PGPUB; USPAT	ADJ	ON	2017/05/10 11:31
S79	3	("20090235848" | "20110076210" | "20140224121").PN.	US-PGPUB; USPAT	ADJ	ON	2017/06/27 09:36
S80	96	("1984164" | "2317857" | "20010002387" | "20010003116" | "20020043496" | "20020134242" | "20020150516" | "20030104937" | "20030136509" | "20040003716" | "20040013589" | "20040076570" | "20050019240" | "20060048646" | "20060191835" | "20080182747" | "20080292512" | "20090062119" | "20090136401" | "20090320678" | "20100047146" | "20110168018" | "20120183458" | "20130280156" | "20140056787" | "20140255279" | "20140308188" | "20150098878" | "3194629" | "3662523" | "3786619" | "3826618" | "3849267" | "3876393" | "4094777" | "4101631" | "4196173" | "4338896" | "4500327" | "4708853" | "4786483" | "4814152" | "4820318" | "4889698" | "4956162" | "5300137" | "5336835" | "5409522" | "5462908" | "5505766" | "5575982" | "5607496" | "5672323" | "5695726" | "5827352" | "5891324" | "6027551" | "6080281" | "6136072" | "6136749" | "6214304" | "6258334" | "6372187" |	US-PGPUB; USPAT	ADJ	ON	2017/06/27 09:41

		"6375909" | "6383981" | "6471936" | "6528030" | "6638485" | "6808692" | "6818043" | "6848374" | "6878358" | "6942840" | "6953494" | "6960329" | "7081434" | "7211707" | "7435286" | "7479263" | "7514052" | "7521032" | "7544338" | "7563311" | "7611564" | "7622092" | "7722843" | "7767174" | "7780765" | "8007749" | "8168147" | "8168149" | "8216535" | "8512655" | "8652235" | "8821819" | "9011805").PN.				
S81	593	honeycomb with plug$4 and plug$4 with length	US-PGPUB; USPAT	ADJ	ON	2017/06/27 14:45
S82	0	honeycomb with plug$4 and plug$4 with length with less than with "5"	US-PGPUB; USPAT	ADJ	ON	2017/06/27 14:45
S83	0	honeycomb with plug$4 and plug$4 with length with less than	US-PGPUB; USPAT	ADJ	ON	2017/06/27 14:45
S84	127	honeycomb with plug$4 and plug$4 with length with "5"	US-PGPUB; USPAT	ADJ	ON	2017/06/27 14:46
S85	241	honeycomb with plug$4 and plug$4 with length with mm	US-PGPUB; USPAT	ADJ	ON	2017/06/27 14:47
S86	41	honeycomb with plug$4 and plug$4 with length with "3 mm "	US-PGPUB; USPAT	ADJ	ON	2017/06/27 14:49
S87	28	honeycomb with plug$4 and plug$4 with length with "3 mm " and plug$4 with porosity	US-PGPUB; USPAT	ADJ	ON	2017/06/27 14:52
S88	28	honeycomb and plug$4 with length with "3 mm " and plug$4 with porosity	US-PGPUB; USPAT	ADJ	ON	2017/06/27 14:53
S89	43	honeycomb and plug$4 with porosity with "100"	US-PGPUB; USPAT	ADJ	ON	2017/06/27 15:06
S90	20	honeycomb and plug$4 with pore (size or diameter) with "100"	US-PGPUB; USPAT	ADJ	ON	2017/06/27 15:07
S91	8	saw with honeycomb with filter	US-PGPUB; USPAT	ADJ	ON	2017/06/29 13:31
S92	230	(B01D53/64, B01D2257/602, B01D2253/104, B01D2253/10,	US-PGPUB; USPAT	ADJ	ON	2017/12/01 14:36

		B01D53/10).cpc. and (halogen or halide of HBr) with sorbent				
S93	2	"20060260468"	US-PGPUB; USPAT	ADJ	ON	2018/07/03 10:34
S94	19	engine with air with inlet with filter$4 with cone	US-PGPUB; USPAT	ADJ	ON	2018/07/03 15:26
S95	36	engine with air with inlet with filter$4 with (cone or conical or (diameter with (increas$4 or decreas$4)))	US-PGPUB; USPAT	ADJ	ON	2018/07/03 15:27
S96	24	("3771638" | "3870495" | "3872012" | "4157902" | "4702754" | "5125940" | "5522909" | "5549724" | "5632793").PN. OR ("5888260").URPN.	US-PGPUB; USPAT; USOCR	ADJ	ON	2018/07/03 15:31
S97	41	("0153535" | "0439091" | "1398685" | "1530163" | "1887052" | "2087688" | "4003836" | "4018580" | "4032455" | "4039308" | "4065276" | "4261710" | "4410341" | "4414110" | "4699715" | "5106397" | "5178656" | "5522909" | "5549722" | "5549724" | "5888260" | "6397842" | "6508052" | "D401597" | "D401942" | "D403414" | "D403416").PN. OR ("6808552").URPN.	US-PGPUB; USPAT; USOCR	ADJ	ON	2018/07/03 15:31
S98	15	("D403416").URPN.	USPAT	ADJ	ON	2018/07/03 15:33
S99	0	"20030089233"	USPAT	ADJ	ON	2018/07/04 12:29
S100	3	"20030089233"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/07/04 12:29
S101	26	"6833023"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/07/04 12:29
S102	2	"20070168213"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/07/09 13:08

S103	14	carbocations with sorbent with bromine	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/07/09 13:14
S104	0	a side forming a cross sectional shape of each of the first	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/07/09 13:47
S105	0	non of side forming	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/07/09 13:56
S106	0	none of side forming	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/07/09 13:56
S107	0	none of sides forming	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/07/09 13:56
S109	13	olson.in. and (alkali and promoter and earth).clm.	US-PGPUB; USPAT	ADJ	ON	2018/07/17 16:22
S110	0	olson.in. and (alkali and promoter and earth with vapor).clm.	US-PGPUB; USPAT	ADJ	ON	2018/07/17 16:22
S111	10	olson.in. and (alkali and promoter with vapor).clm.	US-PGPUB; USPAT	ADJ	ON	2018/07/17 16:22
S112	0	"2015008283"	US-PGPUB; USPAT	ADJ	ON	2018/07/23 06:26
S113	2	"20150082833"	US-PGPUB; USPAT	ADJ	ON	2018/07/23 06:26
S114	24	olson.in. and liquid.clm. and promoted	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/07/23 07:50
S115	75	remov$4 with mercury and (br or bromine) with liquid with carbon	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/07/23 07:54

S116	0	separating greater than "70" wt% of the mercury	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/07/23 09:17
S117	0	((separating greater) with (of the mercury)).clm.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/07/23 09:17
S118	20	"11209163"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/07/23 09:43
S119	40	(during with after with mercury with sorbent)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/01 17:38
S120	304	sorbent with mercury and sorbent with boiler	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/01 17:39
S121	2	"20070168213"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/01 17:41
S122	18	before with burn$4 with coal with sorbent	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/01 17:42
S123	204	("0174348" | "0202092" | "0208011" | "0224649" | "0229159" | "0298727" | "0346765" | "0347078" | "0367014" | "0537926" | "0537998" | "0541025" | "0625754" | "0647622" | "0685719" | "0688782" | "0700888" | "0744908" | "0846338" | "0894110" | "0896876" | "0911960" | "0945331" | "0945846" | "1112547" | "1167471" | "1167472" | "1183445" | "1788466" | "1984164" | "20020065581" | "20020114749" | "20020184817" | "20030088370" | "20030103882" | "20040003716" | "20040013589" | "20040086439" |	US-PGPUB; USPAT; USOCR	ADJ	ON	2018/10/01 17:42

		"20040219083" | "20050039598" | "20060047526" | "20060210463" | "2016821" | "2059388" | "2089599" | "2511288" | "3194629" | "3288576" | "3437476" | "3599610" | "3662523" | "3725530" | "3764496" | "3823676" | "3838190" | "3849267" | "3849537" | "3956458" | "3961020" | "3974254" | "4040802" | "4075282" | "4094777" | "4101631" | "4115518" | "4148613" | "4174373" | "4196173" | "4226601" | "4233274" | "4272250" | "4280817" | "4305726" | "4322218" | "4377599" | "4387653" | "4394354" | "4440100" | "4472278" | "4474896" | "4519807" | "4519995" | "4555392" | "4582936" | "4602918" | "4629721" | "4693731" | "4716137" | "4741278" | "4758418" | "4764219" | "4786483" | "4804521" | "4807542" | "4824441" | "4830829" | "4873930" | "4886519" | "4892567" | "4915818" | "4933158" | "4936047" | "4964889" | "5013358" | "5024171" | "5049163" | "5116793" | "5126300" | "5137854" | "5162598" | "5190566" | "5238488" | "5350728" | "5368617" | "5409522" | "5435980" | "5447703" | "5460643" | "5505746" | "5505766" | "5571490").PN. OR ("5587003" | "5618508" | "5635150" | "5659100" | "5670122" | "5733516" | "5738834" | "5787823" | "5810910" | "5897688" | "5910292" | "5989506" | "6024931" | "6083289" | "6240859" | "6258334" | "6372187" | "6375909" | "6475451" | "6521021" | "6528030" | "6533842" | "6558454" | "6610263" | "6613110" | "6719828" | "6732055" | "6737031" | "6746531" | "6790420" | "6808692" | "6848374" | "6878358" | "6942840" | "6953494" | "6962617" | "6974564" | "6975975" | "7468170" | "7507083" | "7674442" | "7758827" | "7776301").PN.				
S124	1	"15573128"	US-PGPUB; USPAT; USOCR	ADJ	ON	2018/10/02 12:07

S125	2	"20110207597"	US-PGPUB; USPAT; USOCR	ADJ	ON	2018/10/02 12:08
S126	2	"20070168213"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/02 17:30
S127	71	sorbent with before with coal and remov$4 with mercury	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/02 17:30
S128	234	"6953494"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/02 17:38
S129	121	"5435980"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/02 17:40
S130	35	promoter with sorbent and "30" g	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/02 22:19
S131	28	promoter with sorbent with mercury and "30" g	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/02 22:19
S132	4	promoter with sorbent with mercury and ("30" g).clm.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/02 22:20
S133	4	promot$4 with sorbent with mercury and ("30" g).clm.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/02 22:23
S134	4	promot$4 with sorbent with mercury and pre.clm.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/02 22:41

S135	2	promot$4 with sorbent with mercury and pre-heater	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/02 22:42
S136	2	promot$4 with sorbent with mercury and pre heater	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/03 08:36
S137	57	promot$4 with sorbent with mercury and pre	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/03 08:36
S138	24	sorbent with preheat$4 with coal	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/03 08:39
S139	121	"5435980"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/03 08:47
S140	23	"11209163"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/03 11:28
S141	5	"14102896"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/03 11:48
S142	7	"12429058"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/03 12:09
S143	4	"12202595"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/03 12:23
S144	20	"12201595"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/03 12:24

S145	3	"14564860"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/03 12:27
S146	1	"15978760"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/03 12:28
S147	1	"15589359"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/03 12:32
S148	1	"15853029"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/03 12:37
S149	8	"15997091"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/03 12:42
S150	6	"14712558"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/03 12:42
S151	2	"15951970"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/03 15:18
S152	4	"15382114"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/03 15:47
S153	1	"15853029"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/03 15:51
S154	2	"20070168213"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/03 18:18

S155	69	"4279873"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/03 18:42
S156	75	"7435286"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/04 07:59
S157	4	"15295594"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/04 08:08
S158	1	("20180257030").PN.	US-PGPUB; USPAT	ADJ	ON	2018/10/04 09:31
S159	86	"5569436"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/04 09:32
S160	6	"5785935"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/04 09:33
S161	8	"6475471"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/04 09:33
S162	53	"6103205"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/04 11:46
S163	0	pyrolysis with char with mercury with activated	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/04 11:49
S164	17	pyrolysis with char with mercury	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/04 11:49

S165	121	pyrolysis with char with activated carbon	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/04 11:50
S166	0	pyrolysis with char with activated carbon with mercury with sorbent	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/04 11:50
S167	43	pyrolysis with char with activated carbon and mercury with sorbent	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/04 11:50
S168	118	pyrolysis with char with tire	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/04 12:04
S169	58	pyrolysis with char with activated carbon and mercury	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/04 12:04
S170	8	"20040081724"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/04 15:07
S171	4	"14128179"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/04 15:10
S172	84	activat$4 carbon with boiler with coal and remov$4 with mercury	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/15 13:27
S173	88	activat$4 carbon with boiler with coal and (remov$4 or filter$4 or absorb$4 or adsorb$4) with mercury	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/15 13:27
S174	0	mercury with stabiliz$4 with sulfer dioxide	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/15 14:23

S175	75	"7435286"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/15 15:07
S176	6	"9468886"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/16 06:48
S177	17	"8821819"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/16 06:51
S178	15	"8652235"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/16 06:55
S179	93	"6136749"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/24 09:08
S180	4	"9757689"	US-PGPUB; USPAT	ADJ	ON	2018/10/24 12:37
S181	9	"8939388"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/29 09:59
S182	669	urea with inject$4 with filter$4 with exhaust	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/29 10:15
S183	2	urea with inject$4 with filter$4 with exhaust and urea with quench$4	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/29 10:16
S184	13	"3615165"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/29 10:35

S185	136	urea with quench$4 with remov$4	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/29 10:39
S186	48	urea dust with scrub$4	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/29 11:25
S187	38	"6221155"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/29 12:04
S188	6	"9468886"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/10/29 14:12
S189	773	ammonium with remov$4 with mercury	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/12/03 14:14
S190	108	ammonium with salt with remov$4 with mercury	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/12/03 14:15
S191	45	ammonium with salt with remov$4 with mercury and activated carbon	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/12/03 14:15
S192	42	"5556447"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/12/03 14:21
S193	174	"4500327"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/12/03 14:21
S194	72	"4500327" and ammonium	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/12/03 14:23

S195	3	anionic couterion	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/12/03 14:32
S196	3480	anionic counterion	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/12/03 14:32
S197	2422	anionic counterion and ammonium with salt	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/12/03 14:32
S198	9	anionic counterion and ammonium with salt and remov$4 with mercury	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/12/03 14:32
S199	52	"6960329"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/12/03 14:50
S200	4	"20040003716"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2018/12/06 11:23
S201	1	"45219176".FMID.	US-PGPUB; USPAT; FPRS	ADJ	ON	2018/12/06 13:32
S202	0	pyrolysis with char with activated carbon with mercury with sorbent	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/01/07 09:41
S203	284	(sorbent or activated carbon) with mercury and (diameter or size) with ("30" or "40" or "50" or "60" or "70") with (sorbent or carbon) and halogen$4 and activated carbon	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	ADJ	ON	2019/04/15 14:29
S204	88	activat$4 carbon with boiler with coal and (remov$4 or filter$4 or absorb$4 or adsorb$4) with mercury	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/04/15 14:29

S205	42	"6132790"	US-PGPUB; USPAT	ADJ	ON	2019/06/12 16:06
S206	19	remov$4 with mercury and calcium nitrate with sorbent	US-PGPUB; USPAT	ADJ	ON	2019/06/12 16:55
S207	1	remov$4 with mercury with calcium nitrate	US-PGPUB; USPAT	ADJ	ON	2019/06/12 16:57
S208	1	"20040146613"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/06/13 12:59
S209	71	("6372187").URPN.	USPAT	ADJ	ON	2019/06/18 10:24
S210	48	("6372187").URPN. and (iodide or HI)	USPAT	ADJ	ON	2019/06/18 10:25
S211	61	(iodide or HI) with inject$4 and mercury with remov$4	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/06/18 10:30
S212	85	(iodide or HI) with coal and mercury with remov$4	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/06/18 10:35
S213	131	"6558454"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/06/18 10:41
S214	5	"15295594"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/07/30 12:48
S215	82	"7435286"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/07/30 16:29
S216	65	"4013516"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/07/30 21:01

S217	24	"4699896"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/07/30 21:01
S218	2	"20120100053"	US-PGPUB; USPAT	ADJ	ON	2019/08/17 11:40
S219	1	"20130157845"	US-PGPUB; USPAT	ADJ	ON	2019/08/17 11:40
S220	204	coal with combust$4 and sorbent with (iodine or iodide or HI)	US-PGPUB; USPAT	ADJ	ON	2019/09/04 12:15
S221	184	coal with combust$4 and sorbent with (iodine or HI)	US-PGPUB; USPAT	ADJ	ON	2019/09/04 12:18
S222	184	coal with combust$4 and sorbent with (iodine or HI) and (iodine or HI)	US-PGPUB; USPAT	ADJ	ON	2019/09/04 12:20
S223	0	electric utility coal combustion chamber	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/09/04 14:06
S224	12	electric with coal combustion chamber	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/09/04 14:07
S225	1755	electric with coal with combustion	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/09/04 14:08
S226	219	electric with coal with combustion with chamber	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/09/04 14:09
S227	89	electric with coal with combustion with chamber	US-PGPUB; USPAT; USOCR	ADJ	ON	2019/09/04 14:09
S228	20	subbituminous with coal with chamber	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/09/04 19:42

S229	191	subbituminous with coal with combustion	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/09/04 19:43
S230	1009	lignite with coal with combustion	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/09/04 20:09
S231	0	lignite with coal with combustion and mercury with sorbet	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/09/04 20:10
S232	54	lignite with coal with combustion and mercury with sorbent	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/09/04 20:10
S233	4	"14917980"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/09/05 12:48
S234	246	"6878358"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/09/05 13:31
S250	25	"2012035853"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/09/19 14:19
S251	326	(B01D53/025).cpc.	US-PGPUB; USPAT	ADJ	ON	2019/12/04 15:09
S252	2	"20050274307"	US-PGPUB; USPAT	ADJ	ON	2019/12/09 08:26
S253	11	"9221013"	US-PGPUB; USPAT	ADJ	ON	2019/12/09 09:06
S254	36	"5556447"	US-PGPUB; USPAT	ADJ	ON	2019/12/09 09:15
S255	42	"7361209"	US-PGPUB; USPAT	ADJ	ON	2019/12/09 09:23

S256	16	"20020114749"	US-PGPUB; USPAT	ADJ	ON	2019/12/09 09:23
S257	1	"20030113239"	US-PGPUB; USPAT	ADJ	ON	2019/12/09 09:25
S258	1	"20040244657"	US-PGPUB; USPAT	ADJ	ON	2019/12/09 09:26
S259	1	"20070259781"	US-PGPUB; USPAT	ADJ	ON	2019/12/09 09:26
S260	3	"10343114"	US-PGPUB; USPAT	ADJ	ON	2019/12/09 10:11
S261	134	"5435980"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/12/09 12:08
S262	1	"20040244657"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/12/09 12:13
S263	4	("10471412" | "20050274307" | "20190329179" | "20190329215").PN.	US-PGPUB; USPAT	ADJ	ON	2019/12/09 12:21
S264	2	"20050274307"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/12/23 23:35
S265	107	"6848374"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2019/12/24 00:10
S266	112	"6848374"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2020/05/20 22:32
S267	84	phosphorus (tribromide or tri bromide) and remov$4 with mercury	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2020/05/20 23:36

S268	93	phosphorus with (tribromide or tri bromide) and remov$4 with mercury	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2020/05/20 23:37
S269	259	(tribromide or tri bromide) and remov$4 with mercury	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2020/05/20 23:37
S270	62	(tribromide or tri bromide) and remov$4 with mercury and coal with flue	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2020/05/20 23:37
S271	10	sulfur chloride and remov$4 with mercury and coal with flue	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2020/05/20 23:38
S272	0	sulfur wiht chloride and remov$4 with mercury and coal with flue	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2020/05/20 23:40
S273	304	sulfur with chloride and remov$4 with mercury and coal with flue	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2020/05/20 23:40
S274	82	phosphorus with (tribromide or tri bromide) and sorbent	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2020/05/20 23:47
S275	41	phosphorus with (tribromide or tri bromide) and sorbent and (mercury or Hg)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2020/05/20 23:48
S276	23	sulfur dichloride and sorbent and (mercury or Hg)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO	ADJ	ON	2020/05/20 23:50

EAST Search History (Interference)

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
S4	0	(activated carbon and non-carbon sorbent and carbon sorbent and mercury and halogen and promoter).clm.	USPAT	ADJ	ON	2013/09/09 09:20
S5	1	(activated carbon and carbon sorbent and mercury and halogen and promoter).clm.	USPAT	ADJ	ON	2013/09/09 09:20
S6	1	(carbon sorbent and mercury and halogen and promoter).clm.	USPAT	ADJ	ON	2013/09/09 09:20
S23	1	(activated carbon and non-carbon sorbent and carbon sorbent and mercury and halogen and promoter).clm.	USPAT	ADJ	ON	2016/06/07 09:06
S24	2	(carbon sorbent and mercury and halogen and promoter).clm.	USPAT	ADJ	ON	2016/06/07 09:06
S31	1	(activated carbon and non-carbon sorbent and carbon sorbent and mercury and halogen and promoter).clm.	USPAT	ADJ	ON	2016/09/30 13:48
S32	2	(carbon sorbent and mercury and halogen and promoter).clm.	USPAT	ADJ	ON	2016/09/30 13:48
S74	0	(sorbent and halocarbond and mean particle size).clm.	USPAT	ADJ	ON	2017/05/10 11:03
S75	0	(sorbent and halocarbon and mean particle size).clm.	USPAT	ADJ	ON	2017/05/10 11:03
S76	1	(sorbent and mean particle size and breakthrough).clm.	USPAT	ADJ	ON	2017/05/10 11:03
S77	5	(sorbent and halocarbon).clm.	USPAT	ADJ	ON	2017/05/10 11:05
S78	17	olson.in. and halocarbon	USPAT	ADJ	ON	2017/05/10 11:06
S108	0	none of sides forming	USPAT	ADJ	ON	2018/07/09 13:56

5/21/2020 4:15:26 PM
C:\Users\amillerharris\Documents\EAST\Workspaces\12429058.wsp

S/N 15/951,970 PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s):	Edwin S. Olson et al.	Examiner:	Unknown
Serial No.:	15/951,970	Group Art Unit:	1736
Filed:	April 12, 2018	Docket:	4820.001US8
Customer No.:	21186	Confirmation No.:	6410

Title: SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY

COMMUNICATION CONCERNING PRIOR OR COPENDING APPLICATION(S)

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Pursuant to the guidance of MPEP §§ 2001.06(b) and 2004(9), Applicant would like to bring the following additional application(s) to the Examiner's attention. The identification of these applications is not intended to suggest that the subject matter claimed in any listed application is, or has been, substantially similar to any claim or claims in the present application.

Serial No./ Patent No.	Filing Date	Attorney Docket	Title
16/509,071	July 11, 2019	4820.001U11	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY
16/509,102	July 11, 2019	4820.001U12	TREATMENT OF COAL WITH MERCURY CONTROL ADDITIVES
16/509,146	July 11, 2019	4820.007US5	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY

Respectfully submitted,

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Date 08/01/2019 By /Nicholas P. Lanzatella/
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/AMBER R ORLANDO/ 05/21/2020

Bibliographic Data

Application No: 15/951,970

Foreign Priority claimed: ○ Yes ◉ No

35 USC 119 (a-d) conditions met: ☐ Yes ☑ No ☐ Met After Allowance

Verified and Acknowledged: /AMBER R ORLANDO/ Examiner's Signature | Initials

Title: SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY

FILING or 371(c) DATE	CLASS	GROUP ART UNIT	ATTORNEY DOCKET NO.
04/12/2018	095	1776	4820.001US8
RULE			

APPLICANTS

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Michael J. Holmes Thompson, ND, UNITED STATES

John H. Pavlish East Grand Forks, MN, UNITED STATES

CONTINUING DATA

This application is a CON of 14712558 05/14/2015 PAT 10589225

14712558 is a CON of 14318270 06/27/2014 PAT 9757689

14318270 is a CON of 13966768 08/14/2013 PAT 8821819

13966768 is a CON of 13427665 03/22/2012 PAT 8512655

13427665 is a CON of 12419219 04/06/2009 PAT 8168147

12419219 is a CON of 12201595 08/29/2008ABN

12201595 is a DIV of 11209163 08/22/2005 PAT 7435286

11209163 has PRO of 60605640 08/30/2004

FOREIGN APPLICATIONS

IF REQUIRED, FOREIGN LICENSE GRANTED**

05/09/2018

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$1,880

S/N 15/951,970 PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s):	Edwin S. Olson et al.	Examiner:	Unknown
Serial No.:	15/951,970	Group Art Unit:	1736
Filed:	April 12, 2018	Docket:	4820.001US8
Customer No.:	21186	Confirmation No.:	6410
Title:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY		

COMMUNICATION CONCERNING PRIOR OR COPENDING APPLICATION(S)

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

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16/509,071	July 11, 2019	4820.001U11	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY
16/509,102	July 11, 2019	4820.001U12	TREATMENT OF COAL WITH MERCURY CONTROL ADDITIVES
16/509,146	July 11, 2019	4820.007US5	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY

Respectfully submitted,

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Date 08/01/2019 By /Nicholas P. Lanzatella/
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Reg. No. 63,803

/AMBER R ORLANDO/ 05/21/2020

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Substitute for form 1449A/PTO

INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Use as many sheets as necessary)

Complete if Known	
Application Number	15/951,970
Filing Date	April 12, 2018
First Named Inventor	Edwin S. Olson
Group Art Unit	1736
Examiner Name	Unknown

Sheet 1 of 2 — Attorney Docket No: 4820.001US8

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	Application Number	15/951,970
	Filing Date	April 12, 2018
	First Named Inventor	Edwin S. Olson
	Group Art Unit	1736
	Examiner Name	Unknown
Sheet 2 of 2	Attorney Docket No: 4820.001US8	

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INFORMATION DISCLOSURE STATEMENT BY APPLICANT

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Complete if Known	
Application Number	15/951,970
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First Named Inventor	Edwin S. Olson
Group Art Unit	1736
Examiner Name	Unknown

Sheet 1 of 2 | Attorney Docket No: 4820.001US8

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Examiner Initial *	USP Document Number	Publication Date	Name of Patentee or Applicant of cited Document
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	First Named Inventor	Edwin S. Olson
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	Group Art Unit	1736
	Examiner Name	Unknown
Sheet 1 of 1	Attorney Docket No: 4820.001US8	

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Application Number	15/951,970
Filing Date	April 12, 2018
First Named Inventor	Edwin S. Olson
Group Art Unit	1776
Examiner Name	AMBER ROSE ORLANDO

Sheet 1 of 2 | Attorney Docket No: 4820.001US8

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	First Named Inventor	Edwin S. Olson
(Use as many sheets as necessary)	**Group Art Unit**	1776
	Examiner Name	AMBER ROSE ORLANDO
Sheet 2 of 2	Attorney Docket No: 4820.001US8	

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EFS ID:	37871651
Application Number:	15951970
International Application Number:	
Confirmation Number:	6410
Title of Invention:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY
First Named Inventor/Applicant Name:	Edwin S. Olson
Customer Number:	21186
Filer:	Garrett M. Hall/Jamie Johnson
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Information:					
16	Non Patent Literature	0015_2_npl_4820003us2_cnoa_10112019_f.pdf	101037 c6c6b14ccc599f8c850c0c4527d0394cb6711059	no	2
Warnings:					
Information:					
17	Non Patent Literature	0016_2_npl_4820004us1_amendmentresponsefinalofficeaction_f.pdf	484739 9ffad1f9e97ca8fb6089fc35083da23da8b4322a	no	23
Warnings:					
Information:					
18	Non Patent Literature	0017_2_npl_4820007us3_foar_08022019_f.pdf	772710 38862044ffd8a299ce2559a58b5ede4ee1463aba	no	18
Warnings:					
Information:					
19	Non Patent Literature	0018_2_npl_4820007us5_oarn_10172019_f.pdf	1057697 0cd0c6e9ee724e6b903e637ae9dc47b733659e4d	no	24
Warnings:					
Information:					
20	Non Patent Literature	0019_2_npl_me2c_aep_answer_counterclaim_10092019_f.pdf	472359 4de5590625f32862bfd2676ee6ddc73d28907a06	no	71
Warnings:					
Information:					

21	Non Patent Literature	0020_2_npl_me2c_aep_plaintiff_answer_10302019_f.pdf	359806 82e115bf4d3da3db743bd36155d861ddf044351	no	37
Warnings:					
Information:					
22	Non Patent Literature	0021_2_npl_me2c_vistra_complaint_07172019_f.pdf	233926 7466264d22236b45d077bce4092c3e23b99d035e	no	31
Warnings:					
Information:					
23	Non Patent Literature	0022_2_npl_olson_chemicalmechanisms_2003_f.pdf	3555311 e13cd0ffe179a215ffe2bcf47742335c5a0f2b99	no	6
Warnings:					
Information:					
24	Non Patent Literature	0023_2_npl_olson_improvedmodel_2003_f.pdf	710676 8f2574c45d05b01ab6854135a119deda085fdece	no	8
Warnings:					
Information:					
25	Non Patent Literature	0024_2_npl_olson_multiplesite_2003_f.pdf	503809 fc27fab265437e5cd6978fa38fc21fd7f5347dd1	no	3
Warnings:					
The page size in the PDF is too large. The pages should be 8.5 x 11 or A4. If this PDF is submitted, the pages will be resized upon entry into the Image File Wrapper and may affect subsequent processing					
Information:					
26	Non Patent Literature	0025_2_npl_olson_multiplesite_abstract_2003_f.pdf	1478989 c4701bf7f11136b13359470b6a80ddc5fe7cd44b	no	3
Warnings:					
Information:					
27	Non Patent Literature	0026_2_npl_steam_42_ch_35_2015_f.pdf	4031937 2513a2c9d373f4ff36402a7ce17e59376ed0c026	no	24
Warnings:					
Information:					

Total Files Size (in bytes):	21684299

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s): Edwin S. Olson et al.
Title: SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY

Docket No.:	4820.001US8	Serial No.:	15/951,970
Filed:	April 12, 2018	Due Date:	N/A
Examiner:	AMBER ROSE ORLANDO	Group Art Unit:	1776
Customer No.:	21186	Confirmation No.:	6410

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

We are transmitting herewith the following attached items (as indicated with an "X"):

X Communication Concerning Prior and Copending Applications (1 pg.)
X Supplemental Information Disclosure Statement (2 pgs.), Form 1449 (2 pgs.) Copies of Cited References (26).

If not provided for in a separate paper filed herewith, please consider this a PETITION FOR EXTENSION OF TIME for sufficient number of months to enter these papers and please charge any additional fees or credit overpayment to Deposit Account No. 19-0743. If applicable, any papers or fees supplied herewith are considered to be timely filed pursuant to 37 C.F.R. § 1.7(a), the response period falling on a Federal Holiday, Saturday or Sunday being extended to the next succeeding business day.

SCHWEGMAN LUNDBERG & WOESSNER, P.A.
Customer No.: 21186

By /Nicholas P. Lanzatella/
Nicholas P. Lanzatella
Reg. No. 63,803

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s):	Edwin S. Olson et al.	Examiner:	AMBER ROSE ORLANDO
Serial No.:	15/951,970	Group Art Unit:	1776
Filed:	April 12, 2018	Docket:	4820.001US8
Customer No.:	21186	Confirmation No.:	6410
Title:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY		

COMMUNICATION CONCERNING PRIOR OR COPENDING APPLICATION(S)

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Pursuant to the guidance of MPEP §§ 2001.06(b) and 2004(9), Applicant would like to bring the following additional application(s) to the Examiner's attention. The identification of these applications is not intended to suggest that the subject matter claimed in any listed application is, or has been, substantially similar to any claim or claims in the present application.

Serial No./ Patent No.	Filing Date	Attorney Docket	Title
16/574,935	September 18, 2019	4820.003US3	ACTIVATED CARBON SORBENT INCLUDING NITROGEN AND METHODS OF USING THE SAME

Respectfully submitted,

SCHWEGMAN LUNDBERG & WOESSNER, P.A.
P.O. Box 2938
Minneapolis, MN 55402
(612) 349-9580

Date 11/26/2019 By /Nicholas P. Lanzatella/

Nicholas P. Lanzatella
Reg. No. 63,803

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s):	Edwin S. Olson et al.	Examiner:	AMBER ROSE ORLANDO
Serial No.:	15/951,970	Group Art Unit:	1776
Filed:	April 12, 2018	Docket:	4820.001US8
Customer No.:	21186	Confirmation No.:	6410
Title:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY		

SUPPLEMENTAL INFORMATION DISCLOSURE STATEMENT

MS Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

In compliance with the duty imposed by 37 C.F.R. § 1.56, and in accordance with 37 C.F.R. §§ 1.97 *et. seq.*, the enclosed materials are brought to the attention of the Examiner for consideration in connection with the above-identified patent application. Applicant respectfully requests that this Information Disclosure Statement be entered and the documents listed on the attached PTO 1449 Form be considered by the Examiner and made of record. Pursuant to the provisions of MPEP 609, Applicant requests that a copy of the PTO 1449 Form, initialed as being considered by the Examiner, be returned to the Applicant with the next official communication.

Pursuant to 37 C.F.R. § 1.97(b), it is believed that no fee or statement is required with the Information Disclosure Statement. However, if an Office Action on the merits has been mailed after filing of the application or after the filing of the most recent RCE, the Commissioner is hereby authorized to charge the required fees to Deposit Account No. 19-0743 in order to have this Information Disclosure Statement considered.

Pursuant to 37 C.F.R. § 1.98(a)(2), copies of cited U.S. Patents and Published Applications, and Non-Published Applications identifiable by USPTO Serial Number, are no longer required to be provided to the Office. Applicant acknowledges the requirement to submit copies of foreign patent documents and non-patent literature in accordance with 37 C.F.R § 1.98(a)(2).

The Examiner is invited to contact the undersigned at the telephone number indicated if there are any questions regarding this communication.

Respectfully submitted,

SCHWEGMAN LUNDBERG & WOESSNER, P.A.
P.O. Box 2938
Minneapolis, MN 55402
(612) 349-9580

Date 11/26/2019 By /Nicholas P. Lanzatella/
Nicholas P. Lanzatella
Reg. No. 63,803

NPL:jj

UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
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APPLICATION NUMBER	FILING or 371(c) DATE	GRP ART UNIT	FIL FEE REC'D	ATTY.DOCKET.NO	TOT CLAIMS	IND CLAIMS
15/951,970	04/12/2018	1776	1880	4820.001US8	20	2

CONFIRMATION NO. 6410

CORRECTED FILING RECEIPT

21186
SCHWEGMAN LUNDBERG & WOESSNER, P.A.
P.O. BOX 2938
MINNEAPOLIS, MN 55402

OC000000011910877

Date Mailed: 10/17/2019

Receipt is acknowledged of this non-provisional utility patent application. The application will be taken up for examination in due course. Applicant will be notified as to the results of the examination. Any correspondence concerning the application must include the following identification information: the U.S. APPLICATION NUMBER, FILING DATE, NAME OF FIRST INVENTOR, and TITLE OF INVENTION. Fees transmitted by check or draft are subject to collection.

Please verify the accuracy of the data presented on this receipt. If an error is noted on this Filing Receipt, please submit a written request for a corrected Filing Receipt, including a properly marked-up ADS showing the changes with strike-through for deletions and underlining for additions. If you received a "Notice to File Missing Parts" or other Notice requiring a response for this application, please submit any request for correction to this Filing Receipt with your reply to the Notice. When the USPTO processes the reply to the Notice, the USPTO will generate another Filing Receipt incorporating the requested corrections provided that the request is grantable.

Inventor(s)

Edwin S. Olson, Grand Forks, ND;
Michael J. Holmes, Thompson, ND;
John H. Pavlish, East Grand Forks, MN;

Applicant(s)

Midwest Energy Emissions Corp., Lewis Center, OH;

Power of Attorney: The patent practitioners associated with Customer Number 21186

Domestic Priority data as claimed by applicant

This application is a CON of 14/712,558 05/14/2015
which is a CON of 14/318,270 06/27/2014 PAT 9757689
which is a CON of 13/966,768 08/14/2013 PAT 8821819
which is a CON of 13/427,665 03/22/2012 PAT 8512655
which is a CON of 12/419,219 04/06/2009 PAT 8168147
which is a CON of 12/201,595 08/29/2008 ABN
which is a DIV of 11/209,163 08/22/2005 PAT 7435286
which claims benefit of 60/605,640 08/30/2004

Foreign Applications for which priority is claimed (You may be eligible to benefit from the **Patent Prosecution Highway** program at the USPTO. Please see http://www.uspto.gov for more information.) - None.
Foreign application information must be provided in an Application Data Sheet in order to constitute a claim to foreign priority. See 37 CFR 1.55 and 1.76.

Permission to Access Application via Priority Document Exchange: Yes

Permission to Access Search Results: Yes

Applicant may provide or rescind an authorization for access using Form PTO/SB/39 or Form PTO/SB/69 as appropriate.

If Required, Foreign Filing License Granted: 05/09/2018

The country code and number of your priority application, to be used for filing abroad under the Paris Convention, is **US 15/951,970**

Projected Publication Date: Not Applicable

Non-Publication Request: No

Early Publication Request: No

Title

SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY

Preliminary Class

095

Statement under 37 CFR 1.55 or 1.78 for AIA (First Inventor to File) Transition Applications: No

PROTECTING YOUR INVENTION OUTSIDE THE UNITED STATES

Since the rights granted by a U.S. patent extend only throughout the territory of the United States and have no effect in a foreign country, an inventor who wishes patent protection in another country must apply for a patent in a specific country or in regional patent offices. Applicants may wish to consider the filing of an international application under the Patent Cooperation Treaty (PCT). An international (PCT) application generally has the same effect as a regular national patent application in each PCT-member country. The PCT process **simplifies** the filing of patent applications on the same invention in member countries, but **does not result** in a grant of "an international patent" and does not eliminate the need of applicants to file additional documents and fees in countries where patent protection is desired.

Almost every country has its own patent law, and a person desiring a patent in a particular country must make an application for patent in that country in accordance with its particular laws. Since the laws of many countries differ in various respects from the patent law of the United States, applicants are advised to seek guidance from specific foreign countries to ensure that patent rights are not lost prematurely.

Applicants also are advised that in the case of inventions made in the United States, the Director of the USPTO must issue a license before applicants can apply for a patent in a foreign country. The filing of a U.S. patent application serves as a request for a foreign filing license. The application's filing receipt contains further information and guidance as to the status of applicant's license for foreign filing.

Applicants may wish to consult the USPTO booklet, "General Information Concerning Patents" (specifically, the section entitled "Treaties and Foreign Patents") for more information on timeframes and deadlines for filing foreign patent applications. The guide is available either by contacting the USPTO Contact Center at 800-786-9199, or it can be viewed on the USPTO website at http://www.uspto.gov/web/offices/pac/doc/general/index.html.

For information on preventing theft of your intellectual property (patents, trademarks and copyrights), you may wish to consult the U.S. Government website, http://www.stopfakes.gov. Part of a Department of Commerce initiative, this website includes self-help "toolkits" giving innovators guidance on how to protect intellectual property in specific countries such as China, Korea and Mexico. For questions regarding patent enforcement issues, applicants may call the U.S. Government hotline at 1-866-999-HALT (1-866-999-4258).

LICENSE FOR FOREIGN FILING UNDER

Title 35, United States Code, Section 184

Title 37, Code of Federal Regulations, 5.11 & 5.15

GRANTED

The applicant has been granted a license under 35 U.S.C. 184, if the phrase "IF REQUIRED, FOREIGN FILING LICENSE GRANTED" followed by a date appears on this form. Such licenses are issued in all applications where the conditions for issuance of a license have been met, regardless of whether or not a license may be required as set forth in 37 CFR 5.15. The scope and limitations of this license are set forth in 37 CFR 5.15(a) unless an earlier license has been issued under 37 CFR 5.15(b). The license is subject to revocation upon written notification. The date indicated is the effective date of the license, unless an earlier license of similar scope has been granted under 37 CFR 5.13 or 5.14.

This license is to be retained by the licensee and may be used at any time on or after the effective date thereof unless it is revoked. This license is automatically transferred to any related applications(s) filed under 37 CFR 1.53(d). This license is not retroactive.

The grant of a license does not in any way lessen the responsibility of a licensee for the security of the subject matter as imposed by any Government contract or the provisions of existing laws relating to espionage and the national security or the export of technical data. Licensees should apprise themselves of current regulations especially with respect to certain countries, of other agencies, particularly the Office of Defense Trade Controls, Department of State (with respect to Arms, Munitions and Implements of War (22 CFR 121-128)); the Bureau of Industry and Security, Department of Commerce (15 CFR parts 730-774); the Office of Foreign AssetsControl, Department of Treasury (31 CFR Parts 500+) and the Department of Energy.

NOT GRANTED

No license under 35 U.S.C. 184 has been granted at this time, if the phrase "IF REQUIRED, FOREIGN FILING LICENSE GRANTED" DOES NOT appear on this form. Applicant may still petition for a license under 37 CFR 5.12, if a license is desired before the expiration of 6 months from the filing date of the application. If 6 months has lapsed from the filing date of this application and the licensee has not received any indication of a secrecy order under 35 U.S.C. 181, the licensee may foreign file the application pursuant to 37 CFR 5.15(b).

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UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
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Alexandria, Virginia 22313-1450
www.uspto.gov

APPLICATION NUMBER	FILING OR 371(C) DATE	FIRST NAMED APPLICANT	ATTY. DOCKET NO./TITLE
15/951,970	04/12/2018	Edwin S. Olson	4820.001US8

CONFIRMATION NO. 6410

21186
SCHWEGMAN LUNDBERG & WOESSNER, P.A.
P.O. BOX 2938
MINNEAPOLIS, MN 55402

POA ACCEPTANCE LETTER

OC000000111910904

Date Mailed: 10/17/2019

NOTICE OF ACCEPTANCE OF POWER OF ATTORNEY

This is in response to the Power of Attorney filed 10/11/2019.

The Power of Attorney in this application is accepted. Correspondence in this application will be mailed to the above address as provided by 37 CFR 1.33.

Questions about the contents of this notice and the requirements it sets forth should be directed to the Office of Data Management, Application Assistance Unit, at **(571) 272-4000** or **(571) 272-4200** or **1-888-786-0101**.

/hteffera/

Amended PTO/AIA/82B(07-13)
Approved for use through 11/30/2014. OMB 0651-0035
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POWER OF ATTORNEY BY APPLICANT

I hereby revoke all previous powers of attorney given in the application identified in either the attached transmittal letter or the boxes below.

Application Number	Filing Date

☒ I hereby appoint the Patent Practitioner(s) associated with the following Customer Number as my/our attorney(s) or agent(s), and to transact all business in the United States Patent and Trademark Office connected therewith for the application referenced in the attached transmittal letter (form PTO/AIA/82A) or identified above: **21186**

OR

☐ I hereby appoint Practitioner(s) named in the attached list (form PTO/AIA/82C) as my/our attorney(s) or agent(s), and to transact all business in the United States Patent and Trademark Office connected therewith for the patent application referenced in the attached transmittal letter (form PTO/AIA/82A) or identified above. (Note: Complete form PTO/AIA/82C.)

Please recognize or change the correspondence address for the application identified in the attached transmittal letter to:

☒ The address associated with the above mentioned Customer Number.

OR

☐ The address associated with Customer Number:

OR

☐ Firm or Individual Name					
Address					
City		State		Zip	
Country					
Telephone		Email			

I am the Applicant (if the Applicant is a juristic entity, list the Applicant name in the box):

Midwest Energy Emissions Corp.

☐ Inventor or Joint Inventor (title not required below)

☐ Legal Representative of a Deceased or Legally Incapacitated Inventor (title not required below)

☒ Assignee or Person to Whom the Inventor is Under an Obligation to Assign (provide signer's title if applicant is a juristic entity)

☐ Person Who Otherwise Shows Sufficient Proprietary Interest (*e.g.*, a petition under 37 CFR 1.46(b)(2) was granted in the application or is concurrently being filed with this document) (provide signer's title if applicant is a juristic entity)

SIGNATURE of Applicant for Patent

The undersigned (whose title is supplied below) is authorized to act on behalf of the applicant (e.g., where the applicant is a juristic entity).

Signature	John Pavlish	Date (Optional)	6/9/2017
Name	John Pavlish		
Title	Chief Technology Officer		

NOTE: Signature - This form must be signed by the applicant in accordance with 37 CFR 1.33. See 37 CFR 1.4 for signature requirements and certifications. If more than one applicant, use multiple forms.

☒ *Total of 1 forms are submitted.

This collection of information is required by 37 CFR 1.31, 1.32 and 1.33. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 3 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

Amended PTO/AIA/96 (08-12)
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U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE
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STATEMENT UNDER 37 CFR 3.73(c)

Applicant/Patent Owner: Midwest Energy Emissions Corp.

Application No./Patent No.: 15/951,970 Filed/Issue Date: April 12, 2018

Titled: SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY

Midwest Energy Emissions Corp. a Corporation
(Name of Assignee) (Type of Assignee, e.g., corporation, partnership, university, government agency, etc.)

states that, for the patent application/patent identified above, it is (choose **one** of options 1, 2, 3 or 4 below):

1. ☒ The assignee of the entire right, title, and interest.

2. ☐ An assignee of less than the entire right, title, and interest (check applicable box):

   ☐ The extent (by percentage) of its ownership interest is _____%. Additional Statement(s) by the owners holding the balance of the interest must be submitted to account for 100% of the ownership interest.

   ☐ There are unspecified percentages of ownership. The other parties, including inventors, who together own the entire right, title and interest are:

   Additional Statement(s) by the owner(s) holding the balance of the interest must be submitted to account for the entire right, title, and interest.

3 ☐ The assignee of an undivided interest in the entirety (a complete assignment from one of the joint inventors was made). The other parties, including inventors, who together own the entire right, title, and interest are:

   Additional Statement(s) by the owner(s) holding the balance of the interest must be submitted to account for the entire right, title, and interest.

4. ☐ The recipient, via a court proceeding or the like (e.g., bankruptcy, probate), of an undivided interest in the entirety (a complete transfer of ownership interest was made). The certified document(s) showing the transfer is attached.

The interest identified in option 1, 2 or 3 above (not option 4) is evidenced by either (choose **one** of options A or B below):

A. ☐ An assignment from the inventor(s) of the patent application/patent identified above. The assignment was recorded in the United States Patent and Trademark Office at Reel , Frame, or for which a copy thereof is attached.

B. ☒ A chain of title from the inventor(s), of the patent application/patent identified above, to the current assignee as follows:

1. From: PAVLISH, JOHN H. To: ENERGY AND ENVIRONMENTAL RESEARCH CENTER FOUNDATION

   The document was recorded in the United States Patent and Trademark Office at

   Reel 050462, Frame 0111, or for which a copy thereof is attached.

2. From: OLSON, EDWIN S. To: ENERGY AND ENVIRONMENTAL RESEARCH CENTER FOUNDATION

   The document was recorded in the United States Patent and Trademark Office at

   Reel 050461, Frame 0076, or for which a copy thereof is attached.

This collection of information is required by 37 CFR 3.73(b). The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 12 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

Amended PTO/AIA/96 (08-12)
Approved for use through 01/31/2013. OMB 0651-0031
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE
Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

STATEMENT UNDER 37 CFR 3.73(c)

3. From: HOLMES, MICHAEL J. To: ENERGY AND ENVIRONMENTAL RESEARCH CENTER FOUNDATION

The document was recorded in the United States Patent and Trademark Office at

Reel 050461, Frame 0741, or for which a copy thereof is attached.

4. From: ENERGY AND ENVIRONMENTAL RESEARCH CENTER FOUNDATION To: MIDWEST ENERGY EMISSIONS CORP.

The document was recorded in the United States Patent and Trademark Office at

Reel 050462, Frame 0607, or for which a copy thereof is attached.

5. From: _____ To: _____

The document was recorded in the United States Patent and Trademark Office at

Reel _____, Frame _____, or for which a copy thereof is attached.

6. From: _____ To: _____

The document was recorded in the United States Patent and Trademark Office at

Reel _____, Frame _____, or for which a copy thereof is attached.

☐ Additional documents in the chain of title are listed on a supplemental sheet(s).

☐ As required by 37 CFR 3.73(c)(1)(i), the documentary evidence of the chain of title from the original owner to the assignee was, or concurrently is being, submitted for recordation pursuant to 37 CFR 3.11.

[NOTE: A separate copy (i.e., a true copy of the original assignment document(s)) must be submitted to Assignment Division in accordance with 37 CFR Part 3, to record the assignment in the records of the USPTO. See MPEP 302.08]

The undersigned (whose title is supplied below) is authorized to act on behalf of the assignee.

/Nicholas P. Lanzatella/

Signature

10/11/2019

Date

Nicholas P. Lanzatella

Printed or Typed Name

USPTO Reg. No. 63,803

Title or Registration Number

Electronic Acknowledgement Receipt

EFS ID:	37436359
Application Number:	15951970
International Application Number:	
Confirmation Number:	6410
Title of Invention:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY
First Named Inventor/Applicant Name:	Edwin S. Olson
Customer Number:	21186
Filer:	James Lee Shands/Diana Johnson
Filer Authorized By:	James Lee Shands
Attorney Docket Number:	4820.001US8
Receipt Date:	11-OCT-2019
Filing Date:	12-APR-2018
Time Stamp:	15:02:46
Application Type:	Utility under 35 USC 111(a)

Payment information:

Submitted with Payment	no

File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1		4820001US8UADSPOADecSigned.pdf	5648295 ed3cba42e0664aaedf797f4f7566ad80ba650e22	yes	15

	Multipart Description/PDF files in .zip description		
	Document Description	**Start**	**End**
	Transmittal Letter	1	1
	Miscellaneous Incoming Letter	2	2
	Oath or Declaration filed	3	5
	Request for Corrected Filing Receipt	6	6
	Application Data Sheet	7	12
	Power of Attorney	13	13
	Assignee showing of ownership per 37 CFR 3.73	14	15
Warnings:			
Information:			
	Total Files Size (in bytes):	5648295	

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s): Edwin S. Olson et al.
Title: SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY

Docket No.:	4820.001US8		
Filed:	April 12, 2018	Serial No.:	15/951,970
Examiner:	AMBER ROSE ORLANDO	Group Art Unit:	1736
Customer No.:	21186	Confirmation No.:	6410

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

We are transmitting herewith the attached:

X Communication Re: Late Submission of Declaration (1 pg.).
X Signed Declaration (3 pgs.).
X Communication Re: Update to Application Data Sheet (1 Pg.).
X Marked Up Application Data Sheet (6 Pgs.)
X Power of Attorney (1 pg.)
X Statement Under 37 CFR 3.73 (c) (2 pgs.)

It is believed that no additional fee is required. However, if necessary, please charge any additional required fees or credit overpayment to Deposit Account No. 19-0743.

Schwegman Lundberg & Woessner, P.A.
Customer No.: 21186

By /Nicholas P. Lanzatella/
Nicholas P. Lanzatella
Reg. No. 63,803

CERTIFICATE UNDER 37 CFR 1.8: The undersigned hereby certifies that this correspondence is being filed using the USPTO's electronic filing system EFS-Web, and is addressed to: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450 on this 11th day of October, 2019.

Diana Johnson
Name

/Diana Johnson/
Signature

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s):	Edwin S. Olson et al.	Examiner:	AMBER ROSE ORLANDO
Serial No.:	15/951,970	Group Art Unit:	1736
Filed:	April 12, 2018	Docket:	4820.001US8
Customer No.:	21186	Confirmation No.:	6410
Title:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY		

COMMUNICATION RE: LATE SUBMISSION OF DECLARATION

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

In compliance with 37 CFR 1.63, we submit the Signed Declaration. Corresponding fees with regard to the late submission were paid with the filing of the application on April 12, 2018 therefore applicants believe no additional fees are due at this time.

Applicants assume the application is now in proper order and in condition for allowance. Please direct any inquiries to the undersigned at (612) 349-9580. If necessary, please charge any additional fees or credit overpayment to Deposit Account 19-0743.

Respectfully submitted,

Schwegman Lundberg & Woessner, P.A.
P.O. Box 2938
Minneapolis, MN 55402
(612) 349-9580

Date 10/11/2019 By /Nicholas P. Lanzatella/

Nicholas P. Lanzatella
Reg. No. 63,803

Amended PTO/AIA/01 (06-12)
Approved for use through 01/31/2014. OMB 0651-0032
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE
Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

DECLARATION (37 CFR 1.63) FOR UTILITY OR DESIGN APPLICATION USING AN APPLICATION DATA SHEET (37 CFR 1.76)

Title of Invention	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY

As the below named inventor, I hereby declare that:

This declaration is directed to:

☐ The attached application, or

☑ United States application or PCT international application number 13/966,768, filed on August 14, 2013.

The above-identified application was made or authorized to be made by me.

I believe that I am the original inventor or an original joint inventor of a claimed invention in the application.

I hereby acknowledge that any willful false statement made in this declaration is punishable under 18 U.S.C. 1001 by fine or imprisonment of not more than (5) years, or both.

WARNING:

Petitioner/applicant is cautioned to avoid submitting personal information in documents filed in a patent application that may contribute to identity theft. Personal information such as social security numbers, bank account numbers, or credit card numbers (other than a check or credit card authorization form PTO-2038 submitted for payment purposes) is never required by the USPTO to support a petition or an application. If this type of personal information is included in documents submitted to the USPTO, petitioners/applicants should consider redacting such personal information from the documents before submitting them to the USPTO. Petitioner/applicant is advised that the record of a patent application is available to the public after publication of the application (unless a non-publication request in compliance with 37 CFR 1.213(a) is made in the application) or issuance of a patent. Furthermore, the record from an abandoned application may also be available to the public if the application is referenced in a published application or an issued patent (see 37 CFR 1.14). Checks and credit card authorization forms PTO-2038 submitted for payment purposes are not retained in the application file and therefore are not publicly available.

LEGAL NAME OF INVENTOR

Inventor: Edwin S. Olson Date (Optional): _____

Signature: E. S. Olson

Note: An application data sheet (PTO/SB/14 or equivalent), including naming the entire inventive entity, must accompany this form. Use an additional PTO/AIA/01 form for each additional inventor.

This collection of information is required by 35 U.S.C. 115 and 37 CFR 1.63. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 1 minute to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

Amended PTO/AIA/01 (08-12)
Approved for use through 01/31/2014. OMB 0651-0032
U.S. Patent and Trademark Office: U.S. DEPARTMENT OF COMMERCE
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DECLARATION (37 CFR 1.63) FOR UTILITY OR DESIGN APPLICATION USING AN APPLICATION DATA SHEET (37 CFR 1.76)

Title of Invention	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY

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The above-identified application was made or authorized to be made by me.

I believe that I am the original inventor or an original joint inventor of a claimed invention in the application.

I hereby acknowledge that any willful false statement made in this declaration is punishable under 18 U.S.C. 1001 by fine or imprisonment of not more than (5) years, or both.

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LEGAL NAME OF INVENTOR

Inventor: Michael J. Holmes Date (Optional): Oct. 24, 2013

Signature: Michael J. Holmes

Note: An application data sheet (PTO/SB/14 or equivalent), including naming the entire inventive entity, must accompany this form. Use an additional PTO/AIA/02 form for each additional inventor.

This collection of information is required by 35 U.S.C. 115 and 37 CFR 1.63. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 1 minute to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing the burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

Amended PTO/AIA/01 (09-12)
Approved for use through 01/31/2014. OMB 0651-0032
U.S. Patent and Trademark Office: U.S. DEPARTMENT OF COMMERCE
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DECLARATION (37 CFR 1.63) FOR UTILITY OR DESIGN APPLICATION USING AN APPLICATION DATA SHEET (37 CFR 1.76)

Title of Invention	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY

As the below named inventor, I hereby declare that:

This declaration is directed to:

☐ The attached application, or

☒ United States application or PCT international application number 13/966,768, filed on August 14, 2013.

The above-identified application was made or authorized to be made by me.

I believe that I am the original inventor or an original joint inventor of a claimed invention in the application.

I hereby acknowledge that any willful false statement made in this declaration is punishable under 18 U.S.C. 1001 by fine or imprisonment of not more than (5) years, or both.

WARNING:

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LEGAL NAME OF INVENTOR

Inventor: John Henry Pavlish Date (Optional): _____

Signature: John Pavlish

Note: An application data sheet (PTO/SB/14 or equivalent), including naming the entire inventive entity, must accompany this form. Use an additional PTO/AIA/03 form for each additional inventor.

This collection of information is required by 35 U.S.C. 115 and 37 CFR 1.63. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 1 minute to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s):	Edwin S. Olson et al.	Examiner:	AMBER ROSE ORLANDO
Serial No.:	15/951,970	Group Art Unit:	1736
Filed:	April 12, 2018	Docket:	4820.001US8
Customer No.:	21186	Confirmation No.:	6410
Title:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY		

COMMUNICATION RE: UPDATE TO APPLICATION DATA SHEET

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Applicants hereby request that the information provided on the Application Data Sheet filed in the above-referenced application on April 12, 2018, be updated with the information as provided on the enclosed marked up Application Data Sheet.

We therefore submit a marked up copy of the Application Data Sheet which shows an update to the Applicant Information.

Once this request is entered, it is respectfully requested that the USPTO update their records and provide confirmation of the above request by way of an Updated Filing Receipt.

It is believed that no fee is required to fulfill this request, however, if necessary, please charge any additional required fees or credit overpayment to Deposit Account No. 19-0743.

Respectfully submitted,

Schwegman Lundberg & Woessner, P.A.
P.O. Box 2938
Minneapolis, MN 55402
(612) 349-9580

Date 10/11/2019 By /Nicholas P. Lanzatella/

Nicholas P. Lanzatella
Reg. No. 63,803

Modified PTO/AIA/14 (12-13)
Approved for use through 04/30/2017. OMB 0651-0032
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE
Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

Application Data Sheet 37 CFR 1.76	Attorney Docket Number	4820.001US8
	Application Number	15/951,970
Title of Invention	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY	

The application data sheet is part of the provisional or nonprovisional application for which it is being submitted. The following form contains the bibliographic data arranged in a format specified by the United States Patent and Trademark Office as outlined in 37 CFR 1.76.
This document may be completed electronically and submitted to the Office in electronic format using the Electronic Filing System (EFS) or the document may be printed and included in a paper filed application.

Secrecy Order 37 CFR 5.2:

☐ Portions or all of the application associated with this Application Data Sheet may fall under a Secrecy Order pursuant to 37 CFR 5.2 (Paper filers only. Applications that fall under Secrecy Order may not be filed electronically.)

Inventor Information:

Inventor:

Legal Name

Prefix	Given Name	Middle Name	Family Name	Suffix

Residence Information (Select One) ☐ US Residency ☐ Non US Residency ☐ Active US Military Service

City		State/Province		Country of Residence	

Mailing Address of Inventor:

Address 1			
Address 2			
City		State/Province	
Postal Code		Country	

Correspondence Information:

Enter either Customer Number or complete the Correspondence Information section below.
For further information see 37 CFR 1.33(a).

☐ An Address is being provided for the correspondence Information of this application.

Customer Number	
Email Address	

Application Information:

Title of the Invention			
Attorney Docket Number		Small Entity Status Claimed ☐	
Application Type			
Subject Matter			
Suggested Class (if any)		Sub Class (if any)	
Suggested Technology Center (if any)			
Total Number of Drawing Sheets (if any)		Suggested Figure for Publication (if any)	

Application Data Sheet 37 CFR 1.76	Attorney Docket Number	4820.001US8
	Application Number	15/951,970
Title of Invention	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY	

The application data sheet is part of the provisional or nonprovisional application for which it is being submitted. The following form contains the bibliographic data arranged in a format specified by the United States Patent and Trademark Office as outlined in 37 CFR 1.76.
This document may be completed electronically and submitted to the Office in electronic format using the Electronic Filing System (EFS) or the document may be printed and included in a paper filed application.

Filing By Reference:

Only complete this section when filing an application by reference under 35 U.S.C. 111(c) and 37 CFR 1.57(a). Do not complete this section if application papers including a specification and any drawings are being filed. Any domestic benefit or foreign priority information must be provided in the appropriate section(s) below (i.e., "Domestic Benefit/National Stage Information" and "Foreign Priority Information").

For the purposes of a filing date under 37 CFR 1.53(b), the description and any drawings of the present application are replaced by this reference to the previously filed application, subject to conditions and requirements of 37 CFR 1.57(a).

Application number of the previously filed application	Filing date (YYYY-MM-DD)	Intellectual Property Authority or Country Publication

Publication Information:

☐ **Request Early Publication (Fee required at time of Request 37 CFR 1.219)**

☐ **Request Not to Publish. I hereby request that the attached application not be published under 35 U.S.C. 122(b) and certify that the invention disclosed in the attached application has not and will not be the subject of an application filed in another country, or under a multilateral international agreement, that requires publication at eighteen months after filing.**

Representative Information:

Representative information should be provided for all practitioners having a power of attorney in the application. Providing this information in the Application Data Sheet does not constitute a power of attorney in the application (see 37 CFR 1.32). Enter either Customer Number or complete the Representative Name section below. If both sections are completed the Customer Number will be used for the Representative Information during processing.

Please Select One:	☐ Customer	☐ US Patent Practitioner	☐ Limited Recognition (37 CFR 11.9)
Customer Number			

Domestic Benefit/National Stage Information:

This section allows for the applicant to either claim benefit under 35 U.S.C. 119(e), 120, 121, or 365(c) or indicate National Stage entry from a PCT application. Providing this information in the application data sheet constitutes the specific reference required by 35 U.S.C. 119(e) or 120, and 37 CFR 1.78(a)(2) or CFR 1.78(a)(4), and need not otherwise be made part of the specification.

Prior Application Status			
Application Number	Continuity Type	Prior Application Number	Filing or 371(c) Date (YYYY-MM-DD)

Foreign Priority Information:

Modified PTO/AIA/14 (12-13)
Approved for use through 04/30/2017. OMB 0651-0032
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE
Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

Application Data Sheet 37 CFR 1.76	Attorney Docket Number	4820.001US8
	Application Number	15/951,970
Title of Invention	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY	

The application data sheet is part of the provisional or nonprovisional application for which it is being submitted. The following form contains the bibliographic data arranged in a format specified by the United States Patent and Trademark Office as outlined in 37 CFR 1.76.
This document may be completed electronically and submitted to the Office in electronic format using the Electronic Filing System (EFS) or the document may be printed and included in a paper filed application.

This section allows for the applicant to claim priority to a foreign application. Providing this information in the application data sheet constitutes the claim for priority as required by 35 U.S.C. 119(b) and 37 CFR 1.55(d). When priority is claimed to a foreign application that is eligible for retrieval under the priority document exchange program (PDX)i the information will be used by the Office to automatically attempt retrieval pursuant to 37 CFR 1.55(h)(1) and (2). Under the POX program, applicant bears the ultimate responsibility for ensuring that a copy of the foreign application is received by the Office from the participating foreign intellectual property office, or a certified copy of the foreign priority application is filed, within the time period specified in 37 CFR 1.55(g)(1).

Application Number	Country	Parent Filing Date (YYYY-MM-DD)	Access Code (if applicable)

Modified PTO/AIA/14 (12-13)
Approved for use through 04/30/2017. OMB 0651-0032
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE
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Application Data Sheet 37 CFR 1.76	Attorney Docket Number	4820.001US8
	Application Number	15/951,970
Title of Invention	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY	

The application data sheet is part of the provisional or nonprovisional application for which it is being submitted. The following form contains the bibliographic data arranged in a format specified by the United States Patent and Trademark Office as outlined in 37 CFR 1.76.
This document may be completed electronically and submitted to the Office in electronic format using the Electronic Filing System (EFS) or the document may be printed and included in a paper filed application.

Authorization or Opt-Out of Authorization to Permit Access:

When this Application Data Sheet is properly signed and filed with the application, applicant has provided written authority to permit a participating foreign intellectual property (IP) office access to the instant application-as-filed (see paragraph A in subsection 1 below) and the European Patent Office (EPO) access to any search results from the instant application (see paragraph B in subsection 1 below).

Should applicant choose not to provide an authorization identified in subsection 1 below, applicant must opt-out of the authorization by checking the corresponding box A or B or both in subsection 2 below.

NOTE: This section of the Application Data Sheet is ONLY reviewed and processed with the INITIAL filing of an application. After the initial filing of an application, an Application Data Sheet cannot be used to provide or rescind authorization for access by a foreign IP office(s). Instead, Form PTO/SB/39 or PTO/SB/69 must be used as appropriate.

1. Authorization to Permit Access by a Foreign Intellectual Property Office(s)

A. Priority Document Exchange (PDX) - Unless box A in subsection 2 (opt-out of authorization) is checked, the undersigned hereby grants the USPTO authority to provide the European Patent Office (EPO), the Japan Patent Office (JPO), the Korean Intellectual Property Office (KIPO), the State Intellectual Property Office of the People's Republic of China (SIPO), the World Intellectual Property Organization (WIPO), and any other foreign intellectual property office participating with the USPTO in a bilateral or multilateral priority document exchange agreement in which a foreign application claiming priority to the instant patent application is filed, access to: (1) the instant patent application-as-filed and its related bibliographic data, (2) any foreign or domestic application to which priority or benefit is claimed by the instant application and its related bibliographic data, and (3) the date of filing of this Authorization. See 37 CFR 1.14(h)(1).

B. Search Results from U.S. Application to EPO - Unless box B in subsection 2 (opt-out of authorization) is checked, the undersigned hereby grants the USPTO authority to provide the EPO access to the bibliographic data and search results from the instant patent application when a European patent application claiming priority to the instant patent application is filed. See 37 CFR 1.14(h)(2).

The applicant is reminded that the EPO's Rule 141(1) EPC (European Patent Convention) requires applicants to submit a copy of search results from the instant application without delay in a European patent application that claims priority to the instant application.

2. Opt-Out of Authorizations to Permit Access by a Foreign Intellectual Property Office(s)

☐ **A. Applicant DOES NOT authorize the USPTO to permit a participating foreign IP office access to the instant application-as-filed. If this box is checked, the USPTO will not be providing a participating foreign IP office with any documents and information identified in subsection 1A above.**

☐ **B. Applicant DOES NOT authorize the USPTO to transmit to the EPO any search results from the instant patent application. If this box is checked, the USPTO will not be providing the EPO with search results from the instant application.**

NOTE: Once the application has published or is otherwise publicly available, the USPTO may provide access to the application in accordance with 37 CFR 1.14.

Modified PTO/AIA/14 (12-13)
Approved for use through 04/30/2017. OMB 0651-0032
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE
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Application Data Sheet 37 CFR 1.76	Attorney Docket Number	4820.001US8
	Application Number	15/951,970
Title of Invention	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY	

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This document may be completed electronically and submitted to the Office in electronic format using the Electronic Filing System (EFS) or the document may be printed and included in a paper filed application.

Applicant Information:

Providing assignment information in this section does not substitute for compliance with any requirement of part 3 of Title 37 of CFR to have an assignment recorded by the Office.

Applicant: ~~Midwest Energy Emissions Corp~~ Midwest Energy Emissions Corp.

If the applicant is the inventor (or the remaining joint inventor or inventors under 37 CFR 1.45), this section should not be completed. The information to be provided in this section is the name and address of the legal representative who is the applicant under 37 CFR 1.43; or the name and address of the assignee, person to whom the inventor is under an obligation to assign the invention, or person who otherwise shows sufficient proprietary interest in the matter who is the applicant under 37 CFR 1.46. If the applicant is an applicant under 37 CFR 1.46 (assignee, person to whom the inventor is obligated to assign, or person who otherwise shows sufficient proprietary interest) together with one or more joint inventors, then the joint inventor or inventors who are also the applicant should be identified in this section.

☒ Assignee	☐ Legal Representative under 35 U.S.C. 117	☐ Joint Inventor
☐ Person to whom the inventor is obligated to assign	☐ Person who shows sufficient proprietary interest	

If applicant is the legal representative, indicate the authority to file the patent application, the inventor is:

☐ Deceased	☐ Legally incapacitated

Name of the Deceased or Legally Incapacitated Inventor :

If the Applicant is an Organization check here: ☒

Organization Name: ~~Midwest Energy Emissions Corp~~ Midwest Energy Emissions Corp.

Prefix	Given Name	Middle Name	Family Name	Suffix

Mailing Address Information:

Address 1	670 D Enterprise Drive		
Address 2			
City	Lewis Center	State/Province	OH
Country	United States of America	Postal Code	43035
Phone Number			
Email Address			

Modified PTO/AIA/14 (12-13)
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Application Data Sheet 37 CFR 1.76	Attorney Docket Number	4820.001US8
	Application Number	15/951,970
Title of Invention	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY	

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Assignee Information including Non-Applicant Assignee Information:

Providing assignment information in this section does not substitute for compliance with any requirement of part 3 of Title 37 of CFR to have an assignment recorded by the Office.

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Signature	/Nicholas J. Lanzatella/			Date (YYYY-MM-DD)	2019-10-11
First Name	Nicholas	Last Name	Lanzatella	Registration Number	63,803

This collection of information is required by 37 CFR 1.76. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 23 minutes to complete, including gathering, preparing, and submitting the completed application data sheet form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-14**

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s):	Edwin S. Olson et al.	Examiner:	Unknown
Serial No.:	15/951,970	Group Art Unit:	1736
Filed:	April 12, 2018	Docket:	4820.001US8
Customer No.:	21186	Confirmation No.:	6410
Title:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY		

SUPPLEMENTAL INFORMATION DISCLOSURE STATEMENT

MS Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

In compliance with the duty imposed by 37 C.F.R. § 1.56, and in accordance with 37 C.F.R. §§ 1.97 *et. seq.*, the enclosed materials are brought to the attention of the Examiner for consideration in connection with the above-identified patent application. Applicant respectfully requests that this Information Disclosure Statement be entered and the documents listed on the attached PTO 1449 Form be considered by the Examiner and made of record. Pursuant to the provisions of MPEP 609, Applicant requests that a copy of the PTO 1449 Form, initialed as being considered by the Examiner, be returned to the Applicant with the next official communication.

Pursuant to 37 C.F.R. § 1.97(b), it is believed that no fee or statement is required with the Information Disclosure Statement. However, if an Office Action on the merits has been mailed after filing of the application or after the filing of the most recent RCE, the Commissioner is hereby authorized to charge the required fees to Deposit Account No. 19-0743 in order to have this Information Disclosure Statement considered.

Pursuant to 37 C.F.R. § 1.98(a)(2), copies of cited U.S. Patents and Published Applications, and Non-Published Applications identifiable by USPTO Serial Number, are no longer required to be provided to the Office. Applicant acknowledges the requirement to submit copies of foreign patent documents and non-patent literature in accordance with 37 C.F.R § 1.98(a)(2).

The Examiner is invited to contact the undersigned at the telephone number indicated if there are any questions regarding this communication.

Respectfully submitted,

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Substitute for form 1449A/PTO **INFORMATION DISCLOSURE STATEMENT BY APPLICANT** (Use as many sheets as necessary)	*Complete if Known*	
	Application Number	15/951,970
	Filing Date	April 12, 2018
	First Named Inventor	Edwin S. Olson
	Group Art Unit	1736
	Examiner Name	Unknown
Sheet 1 of 1	Attorney Docket No: 4820.001US8	

US PATENT DOCUMENTS

Examiner Initial *	USP Document Number	Publication Date	Name of Patentee or Applicant of cited Document
	US-10,343,114	7/9/2019	Olson, Edwin S, et al.
	US-20120100053A1	4/26/2012	Durham, Michael D, et al.
	US-20130157845A1	6/20/2013	Nalepa, Christopher J, et al.

FOREIGN PATENT DOCUMENTS

Examiner Initial *	Foreign Document Number	Publication Date	Name of Patentee or Applicant of cited Document	T1
	WO-2012030560A1	3/8/2012	Nalepa, Christopher J, et al.	

OTHER DOCUMENTS – NON PATENT LITERATURE DOCUMENTS

Examiner Initial *	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	T1
	"Application Serial No. 14/712,558, Response filed 07-10-19 to Final Office Action mailed - 1-11-19", 57 pgs.	
	"Application Serial No. 15/295,594, Response filed 4-23-19 to Non Final Office Action mailed 10-23-18", 45 pgs.	
	"Application Serial No. 15/382,114, Final Office Action mailed 06-21-19", 31 pgs.	
	"Application Serial No. 15/589,359, Advisory Action mailed 05-22-19", 3 pgs.	
	"Application Serial No. 15/589,359, Non Final Office Action mailed 06-21-19", 7 pgs.	
	"Application Serial No. 15/589,359, Notice of Allowance mailed 07-31-19", 7 pgs.	
	"Application Serial No. 15/589,359, Response filed 04-30-19 to Final Office Action mailed 03-01-19", 11 pgs.	
	"Application Serial No. 15/589,359, Response filed 07-16-19 to Non Final Office Action mailed 06-21-19", 7 pgs.	
	"Application Serial No. 15/974,343, Response Filed 07-10-2019 to Final Office Action mailed 01-14-19", 81 pgs.	
	"Application Serial No. 15/978,760, Ex Parte Quayle Action mailed 04-19-19", 13 pgs.	
	"Application Serial No. 15/978,760, Notice of Allowability mailed 05-30-19", 3 pgs.	
	"Application Serial No. 15/978,760, Notice of Allowance mailed 05-08-19", 7 pgs.	
	"Application Serial No. 15/978,760, Response filed 4-23-19 to Ex Parte Quayle Action mailed 04-19-19", 15 pgs.	
	"Application Serial No. 15/997,091, Response filed 07-10-19 to Final Office Action mailed 01-11-19", 80 pgs.	

EXAMINER **DATE CONSIDERED**

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Declarations under Rule 4.17:

— *as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii))*

— *as to the applicant's entitlement to claim the priority of the earlier application (Rule 4.17(iii))*

— *of inventorship (Rule 4.17(iv))*

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(54) Title: IMPROVED SORBENTS FOR REMOVING MERCURY FROM EMISSIONS PRODUCED DURING FUEL COMBUSTION

(57) Abstract: Activated carbon is rendered more thermally stable by exposure to a non- halogenated additive, and optionally to a halogen and/or a halogen-containing compound. Such treated carbon is suitable for use in mitigating the content of hazardous substances in flue gases, especially flue gases having a temperature within the range of from about 100 °C to about 420 °C.

WO 2012/030560 A1

IMPROVED SORBENTS FOR REMOVING MERCURY FROM EMISSIONS PRODUCED DURING FUEL COMBUSTION

BACKGROUND

[0001] It has become both desirable and necessary to reduce the hazardous substance content of industrial flue gasses. The hazardous substances can have a deleterious affect on the public health and the environment. Industry and government have been working to reduce the emissions of such substances with good progress being made. Special focus has been on flue gas from coal-fired boilers, such as that found in electric generation plants. Recent focus has also been on emissions from cement kilns. But there is more to do. Hazardous substances include particulates, e.g. fly ash, acid gases, e.g. SOx, NOx, as well as dioxins, furans, heavy metals and the like.

[0002] The methods used to mitigate the emission of hazardous substances depend on the nature of the hazardous substance, the minimum emission level sought, the volume of emitted gas to be treated per unit time and the cost of the mitigating method. Some hazardous substances lend themselves to removal from gaseous effluent by mechanical means, e.g. capture and removal with electrostatic precipitators (ESP), fabric filters (FF) or wet scrubbers. Other substances do not lend themselves to direct mechanical removal.

[0003] Hazardous gaseous substances that are present in a gaseous effluent present interesting challenges, given that direct mechanical removal of any specific gaseous component from a gas stream is problematic. However, it is known, and an industrial practice, to remove hazardous gaseous components from a gaseous effluent by dispersing a fine particulate adsorbent evenly in the effluent to contact and capture, in flight, the targeted gaseous component. This is followed by mechanical removal of the adsorbent with its adsorbate from the effluent vapor by ESP, FF or wet scrubbers. A highly efficacious adsorbent is carbon, e.g., cellulosic-based carbons and coal-based carbons in a form such as powdered activated carbon (PAC). Such PACs, for example, can be used with or without modification. Modified PACs may enhance capture of the target hazardous substance by enhancing adsorption efficiency. PAC modification is exemplified by US 4,427,630; US 5,179,058; US 6,514,907; US 6,953,494; US 2001/0002387; US 2006/0051270; and US 2007/0234902. Cellulosic-

based carbons include, without limitation, carbons derived from woody materials, coconut shell materials, or other vegetative materials. Coal-based PACs include, without limitation, carbons derived from peat, lignite, bituminous, anthracite, or other similar sources.

[0004] A problem with the use of carbons in industrial applications, is their unreliable thermal stability, that is, the lack of assurance that they are resistant to self-ignition. Self-ignition is especially problematic when the carbon is used in the treatment of warm or hot gaseous effluents or when packaged or collected in bulk amounts. For example, bulk PAC is encountered (i) when the PAC is packaged, such as in super-sacks or (ii) when formed as a filter cake in an FF unit or is collected in silos or hoppers associated with an ESP, TOXECON unit, and baghouse. Self-ignition results from unmitigated oxidation of the carbon and can lead to its smoldering or burning. Self-ignition is exacerbated by the carbon being warm or hot, as could be the case when used in treating coal-fired boiler effluents. If oxygen (air) is not denied to the oxidation site or if the site is not cooled, the heat from the initial oxidation will propagate until the carbon smolders or ignites. Such an ignition can be catastrophic. Utility plants are especially sensitive about self-ignition as smoldering or fire within the effluent line can cause a plant shut-down with widespread consequences to served customers.

[0005] Further information on PAC thermal stability can be found in US 6,843,831, "Process for the Purification of Flue Gas." Some carbons are more resistant to self-ignition than others. For example, in the US, the use of coal-derived PACs is often employed for utility flue gas treatment, in part because of the generally recognized good thermal stability of coal-derived PACs.

[0006] It would be advantageous if PACs of lesser thermal stability, such as those derived from certain cellulosic-based carbons could be modified to be more thermally stable so that the practitioner could enjoy the benefit of the excellent adsorption qualities of cellulosic-based carbons. It would also be advantageous to improve the thermal stability of certain coal-based PACs, such as, those that are lignite-based, since even these carbons have been associated with self-ignition and smoldering events.

THE INVENTION

[0007] This invention meets the above-described needs by providing an activated carbon that has been exposed to a non-halogenated additive comprising sulfur, sulfuric acid, sulfamic acid, boric acid, phosphoric acid, ammonium sulfate, urea, ammonium sulfamate, monoammonium phosphate, diammonium phosphate, melamine, melamine phosphate, boric acid/borate combination, silica gel/sodium carbonate, or urea/formaldehyde and, optionally to a halogen and/or a halogen-containing compound, and that has at least one of the following: (i) a temperature of initial energy release that is greater than the temperature of initial energy release for the same activated carbon without the exposure to the non-halogenated additive and, optionally, to the halogen and/or the halogen-containing compound; (ii) a self-sustaining ignition temperature that is greater than the self-sustaining ignition temperature for the same activated carbon without the exposure; or (iii) an early stage energy release value that is less than the early stage energy release value for the same activated carbon without the exposure. It is believed that any one or more of the qualities recited in (i), (ii) and (iii) is indicative of an enhancement of the thermal stability of an activated carbon exposed to one or more non-halogenated additives, and optionally to a halogen and/or a halogen-containing compound, according to this invention as compared to the same activated carbon without the exposure. This invention also relates to a process for enhancing the thermal stability of activated carbon. The process comprises exposing the activated carbon to a non-halogenated additive comprising sulfur, sulfamic acid, boric acid, phosphoric acid, ammonium sulfate, urea, ammonium sulfamate, monoammonium phosphate, diammonium phosphate, melamine, melamine phosphate, boric acid/borate combination, silica gel/sodium carbonate, or urea/formaldehyde and, optionally, to a halogen and/or a halogen-containing compound, at a temperature and for a time sufficient so that the exposed activated carbon has at least one of the following: (i) a temperature of initial energy release that is greater than the temperature of initial energy release for the same activated carbon without the exposure to the non-halogenated additive and, optionally to the halogen and/or the halogen-containing compound; (ii) a self-sustaining ignition temperature that is greater than the self-sustaining ignition temperature for the same activated carbon without the exposure; or (iii) an early stage energy release value that is less than the early stage energy release value for the same activated carbon without the exposure. This invention also relates

to a process for mitigating the atmospheric release of gaseous hazardous substances from flue gases containing such substances, the process comprising contacting the flue gas with activated carbon that has been exposed to a non-halogenated additive comprising sulfur, sulfamic acid, boric acid, phosphoric acid, ammonium sulfate, urea, ammonium sulfamate, monoammonium phosphate, diammonium phosphate, melamine, melamine phosphate, boric acid/borate combination, silica gel/sodium carbonate, or urea/formaldehyde and, optionally, to a halogen and/or a halogen-containing compound, and that has at least one of the following: (i) a temperature of initial energy release that is greater than the temperature of initial energy release for the same activated carbon without the exposure to the non-halogenated additive and, optionally to the halogen and/or the halogen-containing compound; (ii) a self-sustaining ignition temperature that is greater than the self-sustaining ignition temperature for the same activated carbon without the exposure; or (iii) an early stage energy release value that is less than the early stage energy release value for the same activated carbon without the exposure.

[0008] The activated carbons of this invention can be, as before noted, derived from both cellulosic-based and coal-based materials.

[0009] The production of activated cellulosic-based carbons, e.g., wood-based PACs, is well known and generally entails either a thermal activation or chemical activation process. For more details see, *Kirk-Othmer Encyclopedia of Chemical Technology,* 4th Edition, Volume 4, pages 1015-1037 (1992). The activated wood-based carbon can be produced from any woody material, such as sawdust, woodchips, coconut shell materials, or other vegetative materials. The production of activated coal-based carbons, e.g., lignite-based PACs, are produced by similar processes.

[0010] Activated cellulosic-based carbons are commercially available. For example, activated wood-based carbons can be obtained from MeadWestvaco Corporation, Specialty Chemical Division. Activated coal-based carbons are also commercially available. Activated lignite-based carbons can be obtained from Norit Americas, Inc., whilst activated bituminous-based carbons can be obtained from Calgon Corporation. Activated carbons can be characterized by their particle size distribution (D^{10}, D^{50} and D^{90}); average particle size; BET surface area; Iodine No.; total pore volume; pore volume distribution (macro/meso and micro pores); elemental analysis; moisture content; and ash speciation and content. Particularly useful activated carbons have one or more of the following characteristics:

Characteristic	General Range	Specific Range
D^{10}	1-10 μm	2-5 μm
D^{50}	5-35 μm	10-20 μm
D^{90}	20-100 μm	30-60 μm
Average Particle Size:	10-50 μm	5-25 μm
BET:	>300 m^2/g	>500 m^2/g
Iodine No.:	300-1200 mg/g	>600 mg/g
Total Pore Volume:	0.10-1.20 cc/g	0.15-0.8 cc/g
Macro/Meso Pore Volume:	0.05-0.70cc/g	0.05-0.40cc/g
Micro Pore Volume:	0.05-0.50 cc/g	0.10-0.40 cc/g
Ash Content:	0-15 wt%	<10 wt%
Moisture Content:	0-15 wt%	<5 wt%

[0011] A non-halogenated additive comprising sulfur, sulfamic acid, boric acid, phosphoric acid, ammonium sulfate, urea, ammonium sulfamate, monoammonium phosphate, diammonium phosphate, melamine, melamine phosphate, boric acid/borate combination, silica gel/sodium carbonate, or urea/formaldehyde can be used in treating carbons in accordance with this invention.

[0012] The halogen and/or the halogen-containing compound optionally used in treating cellulosic-derived carbons in accordance with this invention can comprise bromine, chlorine, fluorine, iodine, ammonium bromide, other nitrogen-containing halogen salts, sodium bromide, calcium bromide, potassium bromine, other inorganic halides, etc.

[0013] The non-halogenated additive and, optionally, the halogen and/or halogen-containing compound treatment of the carbons can be affected by batch or continuous methods. A suitable batch process feeds the carbon to a tumble reactor/dryer whereupon it is mixed with the non-halogen compound. The non-halogen compound can be added as a crystalline material, dry powder, slurry or solution depending upon the physical and/or solubility properties of the non-halogen compound. Upon completion of the feed of non-halogen compound, the treated carbon material can be dried as needed, especially if its moisture content exceeds 5 wt% based on the total weight of the fed carbon. In one application, gaseous Br_2, at its boiling point temperature, is optionally fed to the reactor/dryer at an initial temperature of from about

75 °C to about 82 °C. The reactor/dryer pressure is conveniently kept at around ambient pressure. The dryer is run in the tumble mode during and after the feed. The post-feed tumble period is from about 30 minutes to an hour. Quantitatively, the amount of Br_2 fed corresponds identically or nearly identically with the desired bromine content of self-ignition resistant carbon. For example, if a self-ignition resistant carbon having a bromine content of about 5 wt% is desired, then the amount of Br_2 fed is 5 parts Br_2 per 95 parts of treated carbon. The Br_2 feed rate is essentially uniform throughout the Br_2 feed period. After the post feed tumble period, the self-ignition resistant carbon is removed from the reactor/dryer to storage or packaging.

[0014] A suitable continuous process for treating carbon features a separate feed of non-halogenated additive, and optionally, the halogen and/or halogen-containing compound, and the carbon to a continuous reactor. The non-halogenated additive and the optional halogen and/or halogen-containing compound can be co-fed as well. The particulate carbon is conveniently transported to and through the continuous reactor by a gas such as air and/or nitrogen. To enhance mixing, a downstream eductor can be used to insure turbulent mixing. Quantitatively, the same proportions used as in the batch method are used in the continuous method.

[0015] In both the batch and continuous modes it may be preferable, depending upon the properties of the non-halogenated additive, to introduce the optional halogen and/or halogen-containing compound prior to introduction of the non-halogenated additive by methods described above.

[0016] In both the described batch and continuous methods, all of the optional halogen and/or halogen-containing compound is incorporated in the self-ignition resistant carbon material. Thus, it is convenient to refer to the amount of Br_2 in the self-ignition resistant carbon material by reference to the amounts of Br_2 and treated carbon fed to the reactor. A 5 kg feed of Br_2 and a 95 kg feed of treated will be deemed to have produced a gaseous bromine treated self-ignition resistant carbon material containing 5 wt% bromine. However, if a practitioner should desire to directly measure the incorporated bromine, such measure can be affected by Schöniger Combustion followed by silver nitrate titration.

[0017] The optional halogen and/or halogen-containing self-ignition resistant carbon material can contain from about 2 to about 20 wt% halogen, the wt% being based on the total weight of the self-ignition resistant carbon. A wt% halogen value within the

range of from about 5 to about 15 wt% is especially useful when treating flue gas from coal-fired boilers.

[0018] Several techniques exist for determining the thermal properties of materials. For example, one can determine (i) the temperature of initial energy release; (ii) the self-sustaining ignition temperature; and/or (iii) the early stage energy release values. For these determinations it is useful to have a Differential Scanning Calorimetry (DSC) trace of the heat flow values vs temperature (°C) of the treated and untreated activated cellulosic-based carbon samples as they are controllably heated. The DSC conditions can be as follows: the sample size is about 10 mg; the carrier gas is air at a flow rate of 100 ml/minute; the temperature ramp rate is 10 degrees centigrade/ minute from ambient temperature to 850 °C. The DSC can be run on a TA Instruments Thermal Analyst 5000 Controller with Model 2960 DSC/TGA module. The DSC traces created from the DSC test results can be analyzed with TA Instruments Universal Analysis Software, version 4.3.0.6. The sample can be dried thoroughly before being submitted to DSC testing. Thermal drying is acceptable, e.g., drying a 0.5 to 5.0 gram sample at a temperature of 110 °C for 1 hour. The values obtained from the DSC testing can be traced on a Heat Flow (watts/gram) versus Temperature (°C) graph.

[0019] The thermal stability of a substance can be assessed, e.g., via the temperature of initial energy release, a.k.a., the point of initial oxidation (PIO) of the substance. As used in this specification, including the claims, the PIO of compositions and/or sorbents of this invention is defined as the temperature at which the heat flow, as determined by DSC, has increased by 1.0 W/g with the baseline corrected to zero at 100 °C. PIO has been found to be a good predictor of thermal stability, especially when compared to values for PACs known to generally have suitable thermal stability, i.e. "benchmark carbons." One such a benchmark carbon is exemplified by the lignite coal derived PAC impregnated with NaBr marketed by Norit Americas, Inc., designated DARCO Hg-LH, which coated PAC has been found to have a PIO value of 343 °C.

[0020] Another thermal stability assessment method of comparison is the self-sustaining ignition temperature (SIT). The SIT is usually defined as the intersection of the baseline and the slope at the inflection point of the heat flow as a function of temperature curve. The inflection point can be determined using TA Instruments Universal Analysis Software. Generally, the inflection point is defined in differential calculus as a point on a curve at which the curvature changes sign. The curve changes

from being concave upwards (positive curvature) to concave downwards (negative curvature), or vice versa.

[0021] One final thermal stability assessment method involves determining the early stage energy release values by integration of the DSC trace between 125 °C to 425 °C and between 125 °C to 375 °C. The values from these two integrations are each compared against the same values obtained for PACs that are known to generally have suitable thermal stability, i.e. "benchmark carbons." Such a benchmark carbon is again exemplified by the lignite coal derived PAC designated as DARCO Hg-LH, which has been found to have an early stage energy release values (125 °C to 425 °C) of 1,378 joules/gram and 370 joules/gram for 125 °C to 375 °C.

EXAMPLES

[0022] The following examples, summarized in Table 1, are illustrative of the principles of this invention. It is understood that this invention is not limited to any one specific embodiment exemplified herein, whether in the examples or the remainder of this patent application. The general procedure used to prepare the samples comprised blending a solution of non-halogenated additive with activated carbon. Certain non-halogenated additives (e.g., elemental sulfur), due to their special handling and solubility properties, are more preferably blended as a dry powder with the carbon. The activated carbon mixture was dried overnight in a recirculating air oven to provide a treated carbon. The treated carbon was optionally brominated with elemental bromine according to the process disclosed in US 6953494 or blended with other halogen sources, such as sodium bromide, potassium bromide, calcium bromide, hydrogen bromide, and/or ammonium bromide.

Examples 1-56.

[0023] The following table lists PIO values for a series of samples. The PAC designations are as follows:

-DARCO Hg LH – commercially-available lignite-based powdered activated carbon treated with sodium bromide; particle size, avg. = 18.1 μm.

-TWPAC – thermally-activated wood-based powdered activated carbon, from MeadWestvaco; particle size = 15.4 μm; surface area = 756 m^2/g; pore diameter, avg. = 21.0 Å.

-CCN – activated coconut-based powdered activated carbon, from Jacobi; particle size, avg. = 20.7 μm.

-CWPAC – chemically-activated wood-based powdered activated carbon, from MeadWestvaco; particle size = 16.2 μm.

Table 1. Thermal Properties of Cellulosic PACs Treated with Non-Halogenated Additives and (Optionally) Sources of Halogen

Example	Activated Carbon	Treatment	PIO (°C)
1 (Comparative)	Lignite	DARCO Hg-LH	343
2 (Comparative)	TWPAC	None	266
3 (Comparative)	TWPAC	Br_2 (5%)	356
4 (Comparative)	TWPAC	HCl (3.5%)	310
5 (Comparative)	TWPAC	HNO3 (3.5%)	300
6	TWPAC	Sulfamic Acid (3%)	384
7	TWPAC	Sulfamic Acid (10%)	416
8	TWPAC	Sulfamic Acid (3%); Br_2 (5%)	392
9	TWPAC	Sulfamic Acid (1.5%); Br_2 (5%)	388
10	TWPAC	Sulfur (5%)	402
11	TWPAC	Sulfur (2.5%)	397
12	TWPAC	Sulfur (2.5%); Br_2 (5%)	376
13	TWPAC	Sulfur (1.2%); Br_2 (5%)	378
14	TWPAC	Sulfuric Acid (3%)	309
15	TWPAC	Sulfuric Acid (3%); Br_2 (5%)	386
16	TWPAC	Sulfuric Acid (1.5%); Br_2 (5%)	375
17	TWPAC	Boric Acid (5%)	338
18	TWPAC	Boric Acid (5%); Br_2 (5%)	411
19	TWPAC	Phosphoric Acid (5%)	373
20	TWPAC	Phosphoric Acid (5%); Br_2 (5%)	403
21	TWPAC	Ammonium Sulfate (5%)	399
22	TWPAC	Ammonium Sulfate (3.4%)	384
23	TWPAC	Ammonium Sulfate (5%); Br_2 (5%)	395
24	TWPAC	Urea (5%)	306
25	TWPAC	Urea (5%); Br_2 (5%)	377
26 (Comparative)	TWPAC	Br_2 (10%)	370
27	TWPAC	Sulfuric Acid (15%); Br_2 (10%)	413
28	TWPAC	Br_2 (10%); Sulfuric Acid (15%)	421
29 (Comparative)	TWPAC	NaBr (10%)	287
30 (Comparative)	TWPAC	NaBr (5%)	282
31	TWPAC	NaBr (5%); S (2.5%)	372
32	TWPAC	NaBr (5%); Ammonium Sulfate (1.2%)	363
33	TWPAC	NaBr (5%); Sulfamic Acid (5%)	392
34	TWPAC	NaBr (5%); Sulfamic Acid (1.5%)	358
35 (Comparative)	TWPAC	KBr (10%)	276
36 (Comparative)	TWPAC	KBr (5%)	270
37	TWPAC	KBr (5%); Sulfamic Acid (5%)	394
38	TWPAC	KBr (5%); Sulfamic Acid (1.5%)	343
39 (Comparative)	TWPAC	$CaBr_2$ (10%)	307
40 (Comparative)	TWPAC	$CaBr_2$ (5%)	347

Example	Activated Carbon	Treatment	PIO (°C)
41	TWPAC	$CaBr_2$ (5%); Sulfamic Acid (5%)	362
42	TWPAC	$CaBr_2$ (5%); Sulfamic Acid (1.5%)	320
43 (Comparative)	TWPAC	aq. HBr (10%)	305
44 (Comparative)	TWPAC	aq. HBr (5%)	338
45	TWPAC	aq. HBr (5%); Sulfamic Acid (5%)	390
46	TWPAC	aq. HBr (5%); Sulfamic Acid (1.5%)	335
47 (Comparative)	TWPAC	NH_4Br (10%)	398
48 (Comparative)	TWPAC	NH4Br (5%)	368
49	TWPAC	NH_4Br (5%); Sulfamic Acid (5%)	401
50	TWPAC	NH_4Br (5%); Sulfamic Acid (1.5%)	386
51 (Comparative)	CCN	None	320
52	CCN	Sulfamic Acid (5%)	430
53	CCN	Sulfamic Acid (5%); Br_2 (5%)	447
54	CCN	Sulfuric Acid (5%)	433
55	CCN	Sulfuric Acid (5%); Br_2 (5%)	417
56	CCN	Boric Acid	463
57	CCN	Boric Acid; Br_2 (5%)	455
58	CCN	Sulfur (2.5%)	438
59	CCN	Sulfur (5%)	441
60	CCN	Sulfur (2.5%); Br_2 (5%)	443
61 (Comparative)	CCN	NaBr (5%)	354
62 (Comparative)	CWPAC	None	353
63 (Comparative)	CWPAC	Br_2 (5%)	300
64	CWPAC	Boric Acid (5%)	371
65	CWPAC	Boric Acid (5%); Br_2 (5%)	353
66	CWPAC	Sulfamic Acid (5%)	389
67	CWPAC	Sulfamic Acid (5%); Br_2 (5%)	360
68	CWPAC	Phosphoric Acid (5%)	363
69	CWPAC	Phosphoric Acid (5%); Br_2 (5%)	342
70	CWPAC	Sulfur (5%)	378
71	CWPAC	Sulfur (2.5%)	375
72	CWPAC	Sulfur (2.5%(; Br_2 (5%)	342
73	Lignite	None	392
74	Lignite	Br_2 (5%)	358
75	Lignite	Boric Acid (5%)	452
76	Lignite	Boric Acid (5%); Br_2 (5%)	416
77	Lignite	Sulfamic Acid (5%)	421
78	Lignite	Sulfamic Acid (5%); Br_2 (5%)	382
79	Lignite	Phosphoric Acid (5%)	423
80	Lignite	Phosphoric Acid (5%); Br_2 (5%)	383
81	Lignite	Sulfur (5%)	410
82	Lignite	Sulfur (5%); Br_2 (5%)	398

[0024] The following data indicate that the processes of this invention not only improve the thermal properties of brominated and non-brominated activated carbons but also provide good mercury capture results as well. These data were obtained using the mercury capture device described in US 6953494.

Table 2. Mercury Capture Data for Treated PACs of Examples 2, 3, 8, 10, 12, 15, 18, 20, 23, 25, 26, 27, 28, 29, 30, 33, 36, 40, 47, 48

Brominated PAC	Mercury Capture, (%, Avg)
Example 2 (Comparative)	46
Example 3 (Comparative)	72
Example 8	75
Example 10	50
Example 12	77
Example 15	75
Example 18	76
Example 20	73
Example 23	70
Example 25	71
Example 26 (Comparative)	79
Example 27	76
Example 28	53
Example 29 (Comparative)	71
Example 30 (Comparative)	69
Example 33	59
Example 36 (Comparative)	61
Example 40 (Comparative)	68
Example 47 (Comparative)	74
Example 48 (Comparative)	69

[0025] It is to be understood that the reactants and components referred to by chemical name or formula anywhere in the specification or claims hereof, whether referred to in the singular or plural, are identified as they exist prior to being combined with or coming into contact with another substance referred to by chemical name or chemical type (e.g., another reactant, a solvent, or etc.). It matters not what chemical changes, transformations and/or reactions, if any, take place in the resulting combination or solution or reaction medium as such changes, transformations and/or reactions are the natural result of bringing the specified reactants and/or components together under the conditions called for pursuant to this disclosure. Thus the reactants

and components are identified as ingredients to be brought together in connection with performing a desired chemical reaction or in forming a combination to be used in conducting a desired reaction. Accordingly, even though the claims hereinafter may refer to substances, components and/or ingredients in the present tense ("comprises", "is", etc.), the reference is to the substance, component or ingredient as it existed at the time just before it was first contacted, combined, blended or mixed with one or more other substances, components and/or ingredients in accordance with the present disclosure. Whatever transformations, if any, which occur *in situ* as a reaction is conducted is what the claim is intended to cover. Thus the fact that a substance, component or ingredient may have lost its original identity through a chemical reaction or transformation during the course of contacting, combining, blending or mixing operations, if conducted in accordance with this disclosure and with the application of common sense and the ordinary skill of a chemist, is thus wholly immaterial for an accurate understanding and appreciation of the true meaning and substance of this disclosure and the claims thereof. As will be familiar to those skilled in the art, the terms "combined", "combining", and the like as used herein mean that the components that are "combined" or that one is "combining" are put into a container, e.g., a combustion chamber, a pipe, etc. with each other. Likewise a "combination" of components means the components having been put together in such a container.

[0026] While the present invention has been described in terms of one or more preferred embodiments, it is to be understood that other modifications may be made without departing from the scope of the invention, which is set forth in the claims below.

CLAIMS

What is claimed is:

1. An activated carbon that has been exposed to a non-halogenated additive comprising sulfur, sulfuric acid, sulfamic acid, boric acid, phosphoric acid, ammonium sulfate, urea, ammonium sulfamate, monoammonium phosphate, diammonium phosphate, melamine, melamine phosphate, boric acid/borate combination, silica gel/sodium carbonate, or urea/formaldehyde and, optionally to a halogen and/or a halogen-containing compound and that has at least one of the following: (i) a temperature of initial energy release that is greater than the temperature of initial energy release for the same activated carbon without exposure to the non-halogenated additive and, optionally to the halogen and/or the halogen-containing compound; (ii) a self-sustaining ignition temperature that is greater than the self-sustaining ignition temperature for the same activated carbon without the exposure; or (iii) an early stage energy release value that is less than the early stage energy release value for the same activated carbon without the exposure.

2. The activated carbon of claim 1 wherein the halogen and/or the halogen-containing compound comprises bromine, chlorine, fluorine, iodine, ammonium bromide, other nitrogen-containing halogen salts, or sodium bromide, potassium bromide, calcium bromide, or other inorganic bromide salts.

3. A process for enhancing the thermal stability of activated carbon, which process comprises exposing the activated carbon to a non-halogenated additive comprising sulfur, sulfuric acid, sulfamic acid, boric acid, phosphoric acid, ammonium sulfate, urea, ammonium sulfamate, monoammonium phosphate, diammonium phosphate, melamine, melamine phosphate, boric acid/borate combination, silica gel/sodium carbonate, or urea/formaldehyde and, optionally to a halogen and/or a halogen-containing compound at a temperature and for a time sufficient so that activated carbon that has been exposed to the non-halogenated additive and, optionally to the halogen and/or the halogen-containing compound has at least one of the following: (i) a temperature of initial energy release greater than the temperature of initial energy release for the same activated carbon prior to exposure to the non-halogenated additive

and, optionally to the halogen and/or the halogen-containing compound; (ii) a self-sustaining ignition temperature that is greater than the self-sustaining ignition temperature for the same activated carbon prior to the exposure; or (iii) an early stage energy release value that is less than the early stage energy release value for the same activated carbon prior to the exposure.

4. The process of claim 3 wherein the halogen and/or the halogen-containing compound comprises bromine, chlorine, fluorine, iodine, ammonium bromide, other nitrogen-containing halogen salts, or sodium bromide, potassium bromide, calcium bromide, or other inorganic bromide salts.

5. A non-halogenated additive comprising sulfur, sulfuric acid, sulfamic acid, boric acid, phosphoric acid, ammonium sulfate, urea, ammonium sulfamate, monoammonium phosphate, diammonium phosphate, melamine, melamine phosphate, boric acid/borate combination, silica gel/sodium carbonate, or urea/formaldehyde and, optionally a halogen and/or a halogen-containing compound exposed, activated carbon that contains from about 2 to about 20 wt% halogen and has at least one of the following: (i) a temperature of initial energy release that is greater than the temperature of initial energy release for the same activated carbon prior to exposure to the non-halogenated additive and, optionally the halogen and/or the halogen-containing compound; (ii) a self-sustaining ignition temperature that is greater than the self-sustaining ignition temperature for the same activated carbon prior to the exposure; or (iii) an early stage energy release value that is less than the early stage energy release value for the same activated carbon prior to the exposure.

6. The activated carbon of claim 5 wherein the halogen and/or the halogen-containing compound comprises bromine, chlorine, fluorine, iodine, ammonium bromide, other nitrogen-containing halogen salts, or sodium bromide, potassium bromide, calcium bromide or other inorganic bromide salts.

7. A process for mitigating the atmospheric release gaseous hazardous substances from flue gases containing such substances, the process comprising contacting the flue gas with an activated carbon that has been exposed to a non-halogenated additive comprising sulfur, sulfuric acid, sulfamic acid, boric acid, phosphoric acid, ammonium

sulfate, urea, ammonium sulfamate, monoammonium phosphate, diammonium phosphate, melamine, melamine phosphate, boric acid/borate combination, silica gel/sodium carbonate, or urea/formaldehyde and, optionally to a halogen and/or a halogen-containing compound and that has at least one of the following: (i) a temperature of initial energy release that is greater than the temperature of initial energy release for the same activated carbon prior to exposure to the non-halogenated additive and, optionally to the halogen and/or the halogen-containing compound; (ii) a self-sustaining ignition temperature that is greater than the self-sustaining ignition temperature for the same activated carbon prior to the exposure; or (iii) an early stage energy release value that is less than the early stage energy release value for the same activated carbon prior to the exposure.

8. The process of claim 7 wherein the flue gas has a temperature within the range of from about 100 °C to about 420 °C.

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Page 1758

INTERNATIONAL SEARCH REPORT

International application No
PCT/US2011/048454

A. CLASSIFICATION OF SUBJECT MATTER
INV. B01J20/20 B01D53/64 C01B31/08
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
B01J B01D C01B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	GB 2 088 719 A (TAKEDA CHEMICAL INDUSTRIES LTD) 16 June 1982 (1982-06-16) cited in the application claims 1,5-9 page 1, line 51 - line 55 page 2, line 53 - line 57 examples 4-6 tables 3-5	1-8
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[X] Further documents are listed in the continuation of Box C. [X] See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier document but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
14 November 2011	23/11/2011

Name and mailing address of the ISA/	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Kaluza, Nicoleta

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Form PCT/ISA/210 (second sheet) (April 2005)

PACET AL. EXHIBIT 1023
Page 1759

INTERNATIONAL SEARCH REPORT

International application No
PCT/US2011/048454

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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X,P	WO 2010/114985 A1 (ALBEMARLE CORP [US]; O'DWYER JONATHAN P [US]; ZHOU QUNHUI [US]; LAMBET) 7 October 2010 (2010-10-07) claims 1-10	1-8
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2

Form PCT/ISA/210 (continuation of second sheet) (April 2005)

PACET AL. EXHIBIT 1023
Page 1760

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/US2011/048454

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Electronic Acknowledgement Receipt

EFS ID:	36760365
Application Number:	15951970
International Application Number:	
Confirmation Number:	6410
Title of Invention:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY
First Named Inventor/Applicant Name:	Edwin S. Olson
Customer Number:	21186
Filer:	David L. Buck/Jamie Johnson
Filer Authorized By:	David L. Buck
Attorney Docket Number:	4820.001US8
Receipt Date:	01-AUG-2019
Filing Date:	12-APR-2018
Time Stamp:	17:22:33
Application Type:	Utility under 35 USC 111(a)

Payment information:

Submitted with Payment	no

File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1		15951970_SIDS_08-01-2019.pdf	216224 5c0178438a79b21d752e557ea0f1be0aeeb1abcb	yes	5

	Multipart Description/PDF files in .zip description		
	Document Description	**Start**	**End**
	Miscellaneous Incoming Letter	1	1
	Miscellaneous Incoming Letter	2	2
	Information Disclosure Statement (IDS) Form (SB08)	3	4
	Transmittal Letter	5	5

Warnings:					
Information:					
2	Foreign Reference	0001_1_for_wo_2012030560_a1_f.pdf	955221 a9b55a1cd1d4236988228ccab1abfff359db140c	no	19
Warnings:					
Information:					
3	Non Patent Literature	0002_2_npl_15295594_aarn_42319_f.pdf	4161904 aef25fd006d688db57755b0a665603624a77571c	no	45
Warnings:					
Information:					
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9	Non Patent Literature	0008_2_npl_4820003us2_adar_05222019_f.pdf	161044 3f8ded966b1c69427808e8d6fab34edc83e81a31	no	3
Warnings:					
Information:					
10	Non Patent Literature	0009_2_npl_4820003us2_noar_07312019_f.pdf	513833 7cbd19e3cec6d426d6a593d2fbca6750a486ce08	no	7
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Information:					
11	Non Patent Literature	0010_2_npl_4820003us2_oarn_06212019_f.pdf	284875 18a980621b7872ae763578f6a09609fd76d04016	no	7
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Information:					
13	Non Patent Literature	0012_2_npl_4820007us4_epqa_04192019_f.pdf	436348 fc7473975818732e5e13870eb59f1cc641b2ab50	no	13
Warnings:					
Information:					

14	Non Patent Literature	0013_2_npl_4820007us4_nall_05302019_f.pdf	150885 a489f8e9b415d9d0006c35c1eaf1d00bf683b093	no	3
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Information:					
15	Non Patent Literature	0014_2_npl_4820007us4_noar_05082019_f.pdf	398773 50b90bfae06ecd8bb48c255f57b9fd45a7600468	no	7
Warnings:					
Information:					
16	Non Patent Literature	0015_2_npl_4820_007us3_nfoaresp_f.pdf	4162347 f46b4ae7778dd76a4fdcb62328b220ff04b8e232	no	45
Warnings:					
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		Total Files Size (in bytes):	29981065		

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New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s): Edwin S. Olson et al.
Title: SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY

Docket No.:	4820.001US8	Serial No.:	15/951,970
Filed:	April 12, 2018	Due Date:	N/A
Examiner:	Unknown	Group Art Unit:	1736
Customer No.:	21186	Confirmation No.:	6410

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

We are transmitting herewith the following attached items (as indicated with an "X"):

X Communication Concerning Prior and Copending Applications (1 pg.)
X Supplemental Information Disclosure Statement (2 pgs.), Form 1449 (1 pg.) Copies of Cited References (15).

If not provided for in a separate paper filed herewith, please consider this a PETITION FOR EXTENSION OF TIME for sufficient number of months to enter these papers and please charge any additional fees or credit overpayment to Deposit Account No. 19-0743. If applicable, any papers or fees supplied herewith are considered to be timely filed pursuant to 37 C.F.R. § 1.7(a), the response period falling on a Federal Holiday, Saturday or Sunday being extended to the next succeeding business day.

SCHWEGMAN LUNDBERG & WOESSNER, P.A.
Customer No.: 21186

By /Nicholas P. Lanzatella/
Nicholas P. Lanzatella
Reg. No. 63,803

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s):	Edwin S. Olson et al.	Examiner:	Unknown
Serial No.:	15/951,970	Group Art Unit:	1736
Filed:	April 12, 2018	Docket:	4820.001US8
Customer No.:	21186	Confirmation No.:	6410
Title:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY		

COMMUNICATION CONCERNING PRIOR OR COPENDING APPLICATION(S)

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Pursuant to the guidance of MPEP §§ 2001.06(b) and 2004(9), Applicant would like to bring the following additional application(s) to the Examiner's attention. The identification of these applications is not intended to suggest that the subject matter claimed in any listed application is, or has been, substantially similar to any claim or claims in the present application.

Serial No./ Patent No.	Filing Date	Attorney Docket	Title
16/509,071	July 11, 2019	4820.001U11	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY
16/509,102	July 11, 2019	4820.001U12	TREATMENT OF COAL WITH MERCURY CONTROL ADDITIVES
16/509,146	July 11, 2019	4820.007US5	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY

Respectfully submitted,

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Date 08/01/2019 By /Nicholas P. Lanzatella/
Nicholas P. Lanzatella
Reg. No. 63,803

Modified form PTO/SB/08A(04-07) OMB 651-0031
US Patent & Trademark Office: U.S. DEPARTMENT OF COMMERCE
Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

Substitute for form 1449A/PTO **INFORMATION DISCLOSURE STATEMENT BY APPLICANT** (Use as many sheets as necessary)	*Complete if Known*	
	Application Number	15/951,970
	Filing Date	April 12, 2018
	First Named Inventor	Edwin S. Olson
	Group Art Unit	1736
	Examiner Name	Unknown
Sheet 1 of 2	Attorney Docket No: 4820.001US8	

US PATENT DOCUMENTS

Examiner Initial *	USP Document Number	Publication Date	Name of Patentee or Applicant of cited Document
	US-20190009247A1	1/10/2019	Olson, Edwin S, et al.

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Examiner Initial *	Foreign Document Number	Publication Date	Name of Patentee or Applicant of cited Document	T 1

OTHER DOCUMENTS – NON PATENT LITERATURE DOCUMENTS

Examiner Initial *	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	T 1
	"Application Serial No. 14/712,558, Amendment and Response filed 12-03-18 to Non Final Office Action mailed 10-19-18", 28 pgs	
	"Application Serial No. 14/712,558, Final Office Action mailed 01-11-19", 27 pgs	
	"Application Serial No. 15/382,114, Non Final Office Action mailed 12-12-18", 46 pgs	
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	"Application Serial No. 15/589,359, Final Office Action mailed 03-01-19", 10 pgs	
	"Application Serial No. 15/974,343, Amendment and Response filed 12-03-18 to Non Final Office Action mailed 10-29-18", 37 pgs	
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	"Application Serial No. 15/978,760, Final Office Action mailed 12-26-18", 21 pgs	
	"Application Serial No. 15/978,760, Response filed 03-20-19 to Final Office Action mailed 12-26-18", 89 pgs.	
	"Application Serial No. 15/997,091, Final Office Action mailed 01-11-19", 20 pgs	
	"Application Serial No. 15/997,091, Response filed 12-03-18 Non Final Office Action mailed 11-02-18", 40 pgs	
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	"Control of Mercury Emissions from Coal Fired Electric Utility Boilers: An Update", US EPA Air Pollution Prevention and Control Division, National Risk Management Research Laboratory, Office of Research and Development, (February 18, 2005), 59 pgs	

EXAMINER **DATE CONSIDERED**

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Modified form PTO/SB/08A(04-07) OMB 651-0031
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Substitute for form 1449A/PTO **INFORMATION DISCLOSURE STATEMENT BY APPLICANT** (Use as many sheets as necessary)	*Complete if Known*	
	Application Number	15/951,970
	Filing Date	April 12, 2018
	First Named Inventor	Edwin S. Olson
	Group Art Unit	1736
	Examiner Name	Unknown
Sheet 2 of 2	Attorney Docket No: 4820.001US8	

OTHER DOCUMENTS – NON PATENT LITERATURE DOCUMENTS		
Examiner Initial *	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	T 1
	"Sorbent Enhancement Additives for Mercury Control", US DOE Office of Fossil Energy, National Energy Technology Laboratory, (June 2008), 4 pgs	
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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s):	Edwin S. Olson et al.	Examiner:	Unknown
Serial No.:	15/951,970	Group Art Unit:	1736
Filed:	April 12, 2018	Docket:	4820.001US8
Customer No.:	21186	Confirmation No.:	6410
Title:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY		

SUPPLEMENTAL INFORMATION DISCLOSURE STATEMENT

MS Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

In compliance with the duty imposed by 37 C.F.R. § 1.56, and in accordance with 37 C.F.R. §§ 1.97 *et. seq.*, the enclosed materials are brought to the attention of the Examiner for consideration in connection with the above-identified patent application. Applicant respectfully requests that this Information Disclosure Statement be entered and the documents listed on the attached PTO 1449 Form be considered by the Examiner and made of record. Pursuant to the provisions of MPEP 609, Applicant requests that a copy of the PTO 1449 Form, initialed as being considered by the Examiner, be returned to the Applicant with the next official communication.

Pursuant to 37 C.F.R. § 1.97(b), it is believed that no fee or statement is required with the Information Disclosure Statement. However, if an Office Action on the merits has been mailed after filing of the application or after the filing of the most recent RCE, the Commissioner is hereby authorized to charge the required fees to Deposit Account No. 19-0743 in order to have this Information Disclosure Statement considered.

Pursuant to 37 C.F.R. § 1.98(a)(2), copies of cited U.S. Patents and Published Applications, and Non-Published Applications identifiable by USPTO Serial Number, are no longer required to be provided to the Office. Applicant acknowledges the requirement to submit copies of foreign patent documents and non-patent literature in accordance with 37 C.F.R § 1.98(a)(2).

The Examiner is invited to contact the undersigned at the telephone number indicated if there are any questions regarding this communication.

Respectfully submitted,

SCHWEGMAN LUNDBERG & WOESSNER, P.A.
P.O. Box 2938
Minneapolis, MN 55402
(612) 349-9580

Date 03/27/2019 By /Nicholas P. Lanzatella/

Nicholas P. Lanzatella
Reg. No. 63,803

NPL:jj

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s): Edwin S. Olson et al.
Title: SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY

Docket No.:	4820.001US8	Serial No.:	15/951,970
Filed:	April 12, 2018	Due Date:	N/A
Examiner:	Unknown	Group Art Unit:	1736
Customer No.:	21186	Confirmation No.:	6410

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

We are transmitting herewith the following attached items (as indicated with an "X"):

X Supplemental Information Disclosure Statement (2 pgs.), Form 1449 (2 pgs.) Copies of Cited References (29).

If not provided for in a separate paper filed herewith, please consider this a PETITION FOR EXTENSION OF TIME for sufficient number of months to enter these papers and please charge any additional fees or credit overpayment to Deposit Account No. 19-0743. If applicable, any papers or fees supplied herewith are considered to be timely filed pursuant to 37 C.F.R. § 1.7(a), the response period falling on a Federal Holiday, Saturday or Sunday being extended to the next succeeding business day.

SCHWEGMAN LUNDBERG & WOESSNER, P.A.
Customer No.: 21186

By: /Nicholas P. Lanzatella/
Nicholas P. Lanzatella
Reg. No. 63,803

Electronic Acknowledgement Receipt

EFS ID:	35549695
Application Number:	15951970
International Application Number:	
Confirmation Number:	6410
Title of Invention:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY
First Named Inventor/Applicant Name:	Edwin S. Olson
Customer Number:	21186
Filer:	David L. Buck/Jamie Johnson
Filer Authorized By:	David L. Buck
Attorney Docket Number:	4820.001US8
Receipt Date:	27-MAR-2019
Filing Date:	12-APR-2018
Time Stamp:	17:11:55
Application Type:	Utility under 35 USC 111(a)

Payment information:

Submitted with Payment	no

File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1		15951970_SIDS_03-27-2019.pdf	275300 8a38960e8bc524c89d54796e871bef55a4d4e350	yes	5

	Multipart Description/PDF files in .zip description		
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	Transmittal Letter	2	3
	Miscellaneous Incoming Letter	1	1

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If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

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INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Use as many sheets as necessary)

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Application Number	15/951,970
Filing Date	April 12, 2018
First Named Inventor	Edwin S. Olson
Group Art Unit	1736
Examiner Name	Unknown

Sheet 1 of 2

Attorney Docket No: 4820.001US8

US PATENT DOCUMENTS

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	US-4,013,516	3/22/1977	Greenfield, Charles, et al.
	US-4,699,896	10/13/1987	Sing, Kenneth S, et al.
	US-5,569,436	10/29/1996	Lerner, B. J.
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	US-20180257031A1	9/13/2018	Olson, Edwin S., et al.
	US-20180280870A1	10/4/2018	Olson, Edwin S, et al.

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	Examiner Name	Unknown
Sheet 2 of 2	Attorney Docket No: 4820.001US8	

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Examiner Initial *	**Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.**	**T 1**
	"Application Serial No. 15/589,359, Response filed 07-26-18 to Non Final Office Action mailed 06-04-18", 14 pgs.	
	"Application Serial No. 15/589,359, Response filed 09-14-18 to Final Office Action mailed 09-12-18", 18 pgs.	
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	PIETRZAK, et al., "Preparation of nitrogen-enriched activated carbons from brown coal", Energy and Fuels 20.3, (2006), 1275-1280	

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Serial No.:	15/951,970	Group Art Unit:	1736
Filed:	April 12, 2018	Docket:	4820.001US8
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Title:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY		

SUPPLEMENTAL INFORMATION DISCLOSURE STATEMENT

MS Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

In compliance with the duty imposed by 37 C.F.R. § 1.56, and in accordance with 37 C.F.R. §§ 1.97 *et. seq.*, the enclosed materials are brought to the attention of the Examiner for consideration in connection with the above-identified patent application. Applicant respectfully requests that this Information Disclosure Statement be entered and the documents listed on the attached PTO 1449 Form be considered by the Examiner and made of record. Pursuant to the provisions of MPEP 609, Applicant requests that a copy of the PTO 1449 Form, initialed as being considered by the Examiner, be returned to the Applicant with the next official communication.

Pursuant to 37 C.F.R. § 1.97(b), it is believed that no fee or statement is required with the Information Disclosure Statement. However, if an Office Action on the merits has been mailed after filing of the application or after the filing of the most recent RCE, the Commissioner is hereby authorized to charge the required fees to Deposit Account No. 19-0743 in order to have this Information Disclosure Statement considered.

Pursuant to 37 C.F.R. § 1.98(a)(2), copies of cited U.S. Patents and Published Applications, and Non-Published Applications identifiable by USPTO Serial Number, are no longer required to be provided to the Office. Applicant acknowledges the requirement to submit copies of foreign patent documents and non-patent literature in accordance with 37 C.F.R § 1.98(a)(2).

The Examiner is invited to contact the undersigned at the telephone number indicated if there are any questions regarding this communication.

Respectfully submitted,

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Date 11/13/2018 By /Nicholas P. Lanzatella/
Nicholas P. Lanzatella
Reg. No. 63,803

NPL:jj

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s):	Edwin S. Olson et al.	Examiner:	Unknown
Serial No.:	15/951,970	Group Art Unit:	1736
Filed:	April 12, 2018	Docket:	4820.001US8
Customer No.:	21186	Confirmation No.:	6410
Title:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY		

COMMUNICATION CONCERNING PRIOR OR COPENDING APPLICATION(S)

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Pursuant to the guidance of MPEP §§ 2001.06(b) and 2004(9), Applicant would like to bring the following additional application(s) to the Examiner's attention. The identification of these applications is not intended to suggest that the subject matter claimed in any listed application is, or has been, substantially similar to any claim or claims in the present application.

Serial No./ Patent No.	Filing Date	Attorney Docket	Title
16/130,670	September 13, 2018	4820.005US2	SORBENT COMPRISING CARBON AND NITROGEN AND METHODS OF USING THE SAME

Respectfully submitted,

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Date 11/13/2018 By /Nicholas P. Lanzatella/

Nicholas P. Lanzatella
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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s): Edwin S. Olson et al.
Title: SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY

Docket No.:	4820.001US8	Serial No.:	15/951,970
Filed:	April 12, 2018	Due Date:	N/A
Examiner:	Unknown	Group Art Unit:	1736
Customer No.:	21186	Confirmation No.:	6410

Mail Stop Amendment
Commissioner for Patents
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Alexandria, VA 22313-1450

We are transmitting herewith the following attached items (as indicated with an "X"):

X Communication Concerning Prior and Copending Applications (1 pg.)
X Supplemental Information Disclosure Statement (2 pgs.), Form 1449 (2 pgs.) Copies of Cited References (19).

If not provided for in a separate paper filed herewith, please consider this a PETITION FOR EXTENSION OF TIME for sufficient number of months to enter these papers and please charge any additional fees or credit overpayment to Deposit Account No. 19-0743. If applicable, any papers or fees supplied herewith are considered to be timely filed pursuant to 37 C.F.R. § 1.7(a), the response period falling on a Federal Holiday, Saturday or Sunday being extended to the next succeeding business day.

SCHWEGMAN LUNDBERG & WOESSNER, P.A.
Customer No.: 21186

By: /Nicholas P. Lanzatella/
Nicholas P. Lanzatella
Reg. No. 63,803

Electronic Acknowledgement Receipt

EFS ID:	34299318
Application Number:	15951970
International Application Number:	
Confirmation Number:	6410
Title of Invention:	SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY
First Named Inventor/Applicant Name:	Edwin S. Olson
Customer Number:	21186
Filer:	Jordan Rivard Golomb/Jamie Johnson
Filer Authorized By:	Jordan Rivard Golomb
Attorney Docket Number:	4820.001US8
Receipt Date:	13-NOV-2018
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Time Stamp:	17:44:48
Application Type:	Utility under 35 USC 111(a)

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File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1		15951970_SIDS_11-13-2018.pdf	279612 09a533939af004f15747ebe04f28457a9c9365a9	yes	6

	Multipart Description/PDF files in .zip description		
	Document Description	**Start**	**End**
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	Transmittal Letter	3	4
	Miscellaneous Incoming Letter	2	2
	Miscellaneous Incoming Letter	1	1

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2	Non Patent Literature	0001_2_npl_4820001u10_oarnpe_07272018_f.pdf	566382 51d545c5ffcb8a1daf1ba9534d703e3d59f6eff9	no	13
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New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

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APPLICATION NUMBER	FILING OR 371(C) DATE	FIRST NAMED APPLICANT	ATTY. DOCKET NO./TITLE
15/951,970	04/12/2018	Edwin S. Olson	4820.001US8

CONFIRMATION NO. 6410

21186
SCHWEGMAN LUNDBERG & WOESSNER, P.A.
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PUBLICATION NOTICE

OC000000101719755

Title:SORBENTS FOR THE OXIDATION AND REMOVAL OF MERCURY

Publication No.US-2018-0229182-A1
Publication Date:08/16/2018

NOTICE OF PUBLICATION OF APPLICATION

The above-identified application will be electronically published as a patent application publication pursuant to 37 CFR 1.211, et seq. The patent application publication number and publication date are set forth above.

The publication may be accessed through the USPTO's publically available Searchable Databases via the Internet at www.uspto.gov. The direct link to access the publication is currently http://www.uspto.gov/patft/.

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ABSTRACT DE202012003747

Nitrogen-modified and / or nitrogen-functionalized activated carbon, in particular having catalytic and / or reactive properties, preferably activated carbon with nitrogen as catalytically active heteroatom, obtainable by a process, wherein in the process an activated carbon ("starting activated carbon") used as starting material first of all surface oxidation using and / or in the presence of at least one oxidation reagent ("OR") and the thus obtained, surface oxidized activated carbon following equipment, in particular modification and / or functionalization, with nitrogen, in particular with nitrogen-containing functional groups, using and / or or in the presence of at least one nitrogen modifying reagent ("SMR").

(19) Deutsches Patent- und Markenamt

(10) **DE 20 2012 003 747 U1** 2013.01.10

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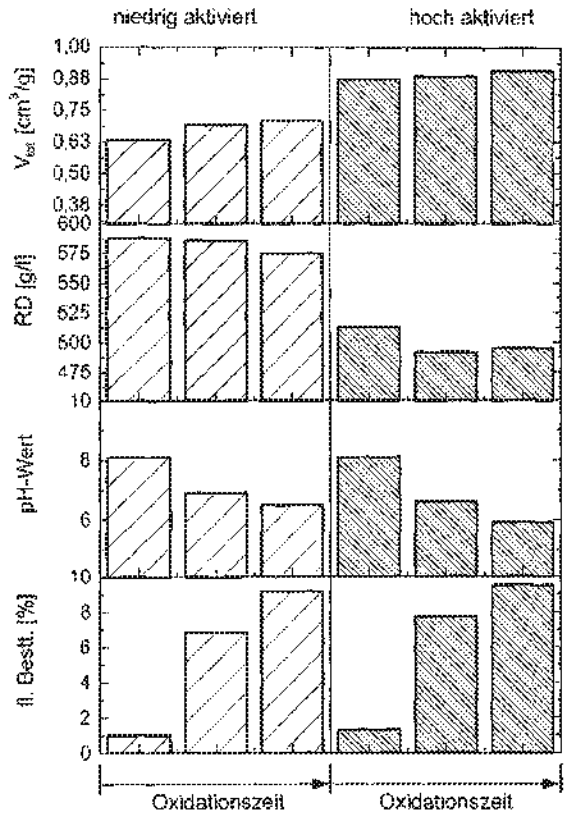
(73) Name und Wohnsitz des Inhabers:
Blücher GmbH, 40699, Erkrath, DE

(74) Name und Wohnsitz des Vertreters:
Patentanwälte Gesthuysen, von Rohr & Eggert, 45128, Essen, DE

Die folgenden Angaben sind den vom Anmelder eingereichten Unterlagen entnommen

(54) Bezeichnung: **Oberflächenmodifizierte Adsorbentien**

(57) Hauptanspruch: Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle, insbesondere mit katalytischen und/oder reaktiven Eigenschaften, vorzugsweise Aktivkohle mit Stickstoff als katalytisch aktivem Heteroatom, erhältlich durch ein Verfahren, wobei bei dem Verfahren eine als Ausgangsmaterial eingesetzte Aktivkohle ("Ausgangsaktivkohle") zunächst einer Oberflächenoxidation unter Verwendung und/oder in Gegenwart mindestens eines Oxidationsreagenzes ("OR") unterzogen wird und die auf diese Weise erhaltene, an ihrer Oberfläche oxidierte Aktivkohle nachfolgend einer Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, mit Stickstoff, insbesondere mit stickstoffhaltigen funktionellen Gruppen, unter Verwendung und/oder in Gegenwart mindestens eines Stickstoffmodifizierungsreagenzes ("SMR") unterzogen wird.



Beschreibung

[0001] Die vorliegende Erfindung betrifft das technische Gebiet adsorptiver Materialien auf Basis von Aktivkohle, welche insbesondere katalytische und/oder reaktive Eigenschaften, insbesondere auf Basis einer Stickstoffmodifizierung und/oder Stickstofffunktionalisierung, vorzugsweise mit Stickstoff als katalytisch aktivem Heteroatom, aufweisen.

[0002] Insbesondere betrifft die vorliegende Erfindung eine stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle, insbesondere mit katalytischen und/oder reaktiven Eigenschaften, vorzugsweise eine Aktivkohle mit Stickstoff als katalytisch aktivem Heteroatom, welche durch ein Verfahren zu deren Herstellung erhältlich ist.

[0003] Des Weiteren betrifft die vorliegende Erfindung Schutzmaterialien, welche unter Verwendung der Aktivkohle nach der Erfindung hergestellt sind bzw. die Aktivkohle nach der Erfindung aufweisen.

[0004] Zudem betrifft die vorliegende Erfindung auch Filter bzw. Filtermaterialien, welche unter Verwendung der Aktivkohle nach der Erfindung hergestellt sind bzw. welche die erfindungsgemäße Aktivkohle aufweisen.

[0005] Aktivkohle ist aufgrund ihrer recht unspezifischen adsorptiven Eigenschaften das am meisten angewendete Adsorbens. Gesetzliche Auflagen, aber auch das steigende Bewusstsein der Verantwortung für die Umwelt, führen zu einem steigenden Bedarf an Aktivkohle.

[0006] Aktivkohle wird im Allgemeinen durch Carbonisierung (synonym auch als Schwelung, Pyrolyse, Abbrand etc. bezeichnet) und anschließende Aktivierung kohlenstoffhaltiger Ausgangsverbindungen erhalten. Die Beschaffenheit der erzeugten Aktivkohle – fein- oder grobporig, fest oder brüchig, feinteilig oder kornförmig etc. – hängt dabei insbesondere auch vom Ausgangsmaterial ab – neben den Verfahrensbedingungen für die Carbonisierung und Aktivierung. Übliche Ausgangsmaterialien sind insbesondere Kokosnussschalen, Holzkohle und Holz (z. B. Holzabfälle), Torf, Steinkohle, Peche, aber auch besondere Kunststoffe bzw. organische Polymere, die unter anderem bei der Herstellung von Aktivkohlegeweben eine gewisse Rolle spielen. Für weitergehende Einzelheiten zur Aktivkohleherstellung kann insbesondere verwiesen werden auf H. v. Kienle und E. Bäder, "Aktivkohle und ihre industrielle Anwendung", Enke Verlag Stuttgart, 1980.

[0007] Aktivkohle wird in verschiedenen Formen verwendet: Pulverkohle, Splitterkohle bzw. Kornkohle, Formkohle und seit Ende der 1970er Jahre auch kugelförmige Aktivkohle ("Kugelkohle"). Kugelförmige Aktivkohle hat gegenüber anderen Formen von Aktivkohle, wie Pulver-, Splitter-, Korn- und Formkohle und dergleichen, eine Reihe von Vorteilen, die sie für bestimmte Applikationen wertvoll oder sogar unverzichtbar macht: Sie ist rieselfähig, abriebfest bzw. staubfrei und hart. Kugelkohle ist wegen ihrer speziellen Form, aber auch wegen der hohen Abriebfestigkeit beispielsweise für besondere Einsatzgebiete sehr gefragt.

[0008] Kugelkohle wird heute noch meist durch mehrstufige und sehr aufwendige Verfahren hergestellt. Das bekannteste Verfahren besteht in der Herstellung von Kügelchen aus Steinkohlenteerpech und geeigneten asphaltartigen Rückständen der Erdölchemie, welche oxidiert – damit sie unschmelzbar werden – und nachfolgend geschwelt und aktiviert werden. Beispielsweise kann die Kugelkohle auch in einem mehrstufigen Verfahren ausgehend von Bitumen hergestellt werden. Diese mehrstufigen Verfahren sind sehr kostenintensiv, und der damit verbundene hohe Preis dieser Kugelkohle verhindert viele Anwendungen, bei denen die Kugelkohle aufgrund ihrer Eigenschaften eigentlich bevorzugt werden müsste.

[0009] In der WO 98/07655 A1 wird ein Verfahren zur Herstellung von Aktivkohlekügelchen beschrieben, bei dem zunächst eine Mischung, die einen aus der Diisocyanatherstellung stammenden Destillationsrückstand, einen kohlenstoffhaltigen Verarbeitungshilfsstoff und gegebenenfalls einen oder mehrere weitere Zusatzstoffe umfasst, zu rieselförmigen Kügelchen verarbeitet wird und anschließend die auf diese Weise erhaltenen Kügelchen carbonisiert und dann aktiviert werden.

[0010] Aus dem Stand der Technik bekannt ist ferner die Herstellung von Kugelkohle durch Schwelung und anschließende Aktivierung von neuen oder gebrauchten Ionenaustauschern, die Sulfonsäuregruppen enthalten, bzw. durch Schwelung von Ionenaustauschervorstufen in Gegenwart von Schwefelsäure und anschließende Aktivierung, wobei die Sulfonsäuregruppen bzw. die Schwefelsäure die Funktion eines Vernetzers haben. Solche Verfahren sind beispielsweise in der DE 43 28 219 A1 und in der DE 43 04 026 A1 sowie in der DE 196 00 237 A1 einschließlich der deutschen Zusatzanmeldung DE 196 25 069 A1 beschrieben.

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[0011] Weiterhin sind aus dem Stand der Technik Verfahren bekannt, bei denen die Herstellung von Aktivkohle, insbesondere Kugelkohle, durch Schwelung und anschließende Aktivierung sulfonierter divinylbenzolvernetzter Polystyrole (d. h. sulfonierte Styrol(Divinylbenzol-Copolymere) erfolgt (vgl. z. B. DE 10 2007 050 971 A1).

[0012] Bei speziellen Anwendungen ist aber nicht nur die Geometrie bzw. die äußere Gestalt der Aktivkohle von entscheidender Bedeutung, sondern auch deren Porosität, insbesondere das Gesamtporenvolumen und die Adsorptionskapazität einerseits und die Verteilung der Poren, d. h. der Anteil an Mikro-, Meso- und Makroporen in Bezug auf das Gesamtporenvolumen, andererseits; insbesondere kann die Porosität durch die Auswahl der Ausgangsmaterialien sowie die Verfahrensbedingungen gezielt gesteuert werden. Im Rahmen der vorliegenden Erfindung bezeichnet der Begriff der Mikroporen solche Poren mit Porendurchmessern von weniger als 2 nm, wohingegen der Begriff der Mesoporen solche Poren mit Porendurchmessern im Bereich von 2 nm (d. h. 2 nm einschließlich) bis 50 nm einschließlich bezeichnet und der Begriff der Makroporen solche Poren mit Porendurchmessern von mehr als 50 nm (d. h. > 50 nm) bezeichnet.

[0013] Aufgrund ihrer guten adsorptiven Eigenschaften kommt Aktivkohle für eine Vielzahl von Anwendungen zum Einsatz: So wird Aktivkohle beispielsweise in der Medizin oder Pharmazie eingesetzt, aber auch in der Lebensmittelindustrie. Auch für Filteranwendungen findet Aktivkohle weitreichende Anwendungen (z. B. Filtration von Gasen und Flüssigkeiten, Entfernung von unerwünschten oder schädlichen bzw. toxischen Gasen etc.).

[0014] Insbesondere kann Aktivkohle in Adsorptionsfiltermaterialien, insbesondere auch speziell in Schutzmaterialien gegenüber Giften, wie chemisch-biologischen Kampfstoffen, wie beispielsweise ABC-Schutzbekleidung, zum Einsatz kommen. Zu diesem Zweck sind insbesondere luft- und wasserdampfdurchlässige Schutzanzüge gegen chemische Kampfstoffe bekannt; derartige luft- und wasserdampfdurchlässige Schutzanzüge besitzen oftmals eine Adsorptionsfilterschicht mit Aktivkohle, welche die chemischen Gifte adsorbieren.

[0015] Zur Steigerung der Adsorptionsleistung wird Aktivkohle oft mit einer katalytisch aktiven oder reaktiven Komponente ausgerüstet, indem die Aktivkohle beispielsweise mit einem biozid bzw. biostatisch wirkenden Katalysator, insbesondere auf Basis von Metallen oder Metallverbindungen, imprägniert wird. Eine derartige Aktivkohle kann insbesondere in Schutzmaterialien zum Einsatz kommen, wie sie beispielsweise beschrieben sind in der DE 195 19 869 A1, welche ein mehrlagiges, textiles, gasdurchlässiges Filtermaterial mit einer Adsorptionsschicht auf Basis von Aktivkohle, insbesondere in Form von karbonisierten Fasern, enthält, die mit einem Katalysator aus der Gruppe von Kupfer, Cadmium, Platin, Palladium, Quecksilber und Zink in Mengen von 0,05 bis 12 Gew.-%, bezogen auf das Aktivkohlematerial, imprägniert ist.

[0016] Eine spezielle, in diesem Zusammenhang zum Einsatz kommende Imprägnierung ist beispielsweise eine so genannte ABEK-Imprägnierung, welche gegenüber spezifischen toxischen Substanzen eine katalytische bzw. abbauende Wirkung aufweist. In diesem Zusammenhang bezieht sich Typ A auf bestimmte organische Gase und Dämpfe mit einem Siedepunkt > 65°C, beispielsweise Cyclohexan. Typ B bezieht sich auf bestimmte anorganische Gase und Dämpfe, beispielsweise Cyanwasserstoff. Typ E bezieht sich auf eine abbauende bzw. schützende Wirkung gegenüber Schwefeldioxid und anderen sauren Gasen und Dämpfen. Typ K schließlich bezieht sich auf eine Schutzfunktion gegenüber Ammoniak und organischen Ammoniakderivaten. Für weiterführende Informationen kann auf die diesbezügliche Europäische Norm EN 14387 (Januar 2004) verwiesen werden.

[0017] Nachteilig bei herkömmlichen Imprägnierungen von Aktivkohle mit Metallen bzw. Metallsalzen ist insbesondere die Tatsache, dass durch die Imprägnierung ein Teil der Adsorptionskapazität der Aktivkohle, welche für die Adsorption und somit für die Unschädlichmachung von chemischen Schadstoffen benötigt wird, verlorengeht. Durch die aus dem Stand der Technik bekannten Imprägnierprozesse der Aktivkohle mit Metallen bzw. Metallsalzen wird somit die Leistungsfähigkeit der Aktivkohle nachteilig beeinträchtigt. Des Weiteren wird durch eine herkömmliche Imprägnierung nicht immer die gewünschte Wirksamkeit erreicht. Auch das Problem des Durchschlagens von Gift- bzw. Kampfstoffen bei hohen Konzentrationen wird durch dieses Prinzip nicht immer gelöst; andererseits wird bei sehr geringen Konzentrationen an zu entfernenden Schadstoffen oder störenden Gasen (z. B. bei der Luftaufbereitung für Reinraumbedingungen) oft nicht die gewünschte Effizienz erreicht, da eine Adsorption erst bei höheren Konzentrationen einsetzt. Schließlich benötigt der herkömmliche Imprägnierprozess mit Metallen bzw. Metallsalzen relativ große Mengen des Imprägniermaterials, da ein großer Teil des als Feststoff vorliegenden Imprägniermaterials bei der Adsorption nicht zur Verfügung steht, insbesondere dann nicht, wenn größere Schichtdicken des Imprägniermaterials im Porensystem der Aktivkohle, insbesondere in Form von Kristalliten, vorliegen.

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[0018] Ein weiterer Nachteil der Ausrüstung von Aktivkohle mit Metallen bzw. Metallsalzen besteht darin, dass herkömmliche Aktivkohle an ihrer Oberfläche unpolar bzw. hydrophob ausgebildet ist und insbesondere keine signifikante Affinität gegenüber den eingesetzten Imprägniermitteln besitzt, welche zur reaktiven bzw. katalytischen Ausrüstung der Aktivkohle eingesetzt werden. Infolgedessen muss ein großer Überschuss an Imprägniermitteln eingesetzt werden, um überhaupt eine gewisse Beladung der Aktivkohle zu gewährleisten. Die Imprägniermittel haften im Allgemeinen nur über rein physikalische Wechselwirkungen und können bei Kontakt mit Flüssigkeiten unerwünschtermaßen zumindest zu einem überwiegenden Teil wieder entfernt werden.

[0019] Darüber hinaus ist es im Stand der Technik insbesondere im Hinblick auf die Adsorption saurer Gase, wie Schwefeldioxid und dergleichen, vorgesehen, eine nachträgliche Imprägnierung der Aktivkohle mit insbesondere basischen stickstoffhaltigen Imprägnierreagenzien, insbesondere Aminen, durchzuführen. Hierbei treten gleichermaßen die zuvor geschilderten Nachteile auf, insbesondere was den Einsatz eines großen Überschusses an Imprägniermittel, die schlechte Beständigkeit der Imprägnierung und das Verstopfen des Porensystems der Aktivkohle anbelangt.

[0020] Darüber hinaus wurde im Stand der Technik auch versucht, eine Ausrüstung bzw. Imprägnierung auf Basis von stickstoffhaltigen Imprägnierreagenzien insofern zu realisieren, als das Imprägnierreagenz im Rahmen des Herstellungsprozesses der Aktivkohle, insbesondere bei der Aktivierung, dem zu behandelnden Substrat zugegeben wird. Diesbezüglich wird im Stand der Technik Ammoniak bzw. NH_3 als entsprechendes Imprägnierreagenz eingesetzt, insbesondere in Form von Ammoniakwasser. Die zugrundeliegende Vorgehensweise, wonach nämlich das Imprägnierreagenz im Rahmen des Aktivierungsprozesses und somit während der Herstellung der Aktivkohle als solche zugegeben wird, ist jedoch insbesondere dahingehend nachteilig, dass mit einem großen Überschuss an Imprägnierreagenz gearbeitet werden muss, da ein überwiegender Teil des Imprägnierreagenes nicht zur eigentlichen Imprägnierung verwendet wird, sondern vielmehr Bestandteil des Aktivierungsprozesses ist. Weiterhin ist bei dieser Vorgehensweise nachteilig, dass der eigentliche Imprägnierprozess nur schlecht kontrolliert werden kann, da die Zugabe des Imprägnierreagenzes im Rahmen der eigentlichen Aktivierung stattfindet, bei welcher nämlich gleichzeitig auch ein Abbrand der Aktivkohle erfolgt, was einer homogenen Beladung der Aktivkohle mit hohen Mengen des Imprägnierungsreagenz abträglich ist. In diesem Zusammenhang ist auch anzuführen, dass die Gegenwart des Imprägnierungsreagenzes gleichermaßen auch eine kontrollierte und exakt reproduzierbare Durchführung des Aktivierungsprozesses erschwert bzw. verhindert, insbesondere was die Ausbildung einer definierten Porenstruktur und dergleichen anbelangt. Weiterhin ist es im Hinblick auf die zuvor geschilderte Vorgehensweise mit der Zugabe des Imprägnierreagenzes bzw. -mittels während des Aktivierungsprozesses nicht möglich, eine chemische Anbindung bzw. Integration der Stickstoffkomponente an das Aktivkohlegerüst zu erreichen, insbesondere da das zugrundeliegende Aktivkohlesubstrat hinsichtlich seiner Oberflächenbeschaffenheit unpolar bzw. hydrophob ist, was einem Inkontaktbringen bzw. einer Wechselwirkung mit dem Imprägnierreagenz abträglich ist. Auf Basis dieser Vorgehensweise ist es somit im Ergebnis nicht möglich, eine effektive Imprägnierung mit einer katalytisch aktiven Komponente zu ermöglichen, da sowohl die Ausrüstung mit der zugrundeliegenden reaktiven Komponente weder dauerhaft noch in großen Mengen bzw. homogen erfolgt und darüber hinaus gleichzeitig auch der Aktivierungsprozess als solcher nachteilig beeinflusst ist.

[0021] Es ist daher eine Aufgabe der vorliegenden Erfindung, adsorptive Materialien mit reaktiven und/oder katalytischen Eigenschaften in Form von Aktivkohle bereitzustellen, wobei die zuvor geschilderten Nachteile des Standes der Technik zumindest weitgehend vermieden oder aber wenigstens abgeschwächt werden sollen.

[0022] Insbesondere soll im Rahmen der vorliegenden Erfindung eine Aktivkohle bereitgestellt werden, welche eine reaktive bzw. katalytische Ausrüstung auf Basis von Stickstoff als katalytisch aktivem Heteroatom aufweist und welche sich insbesondere zur Adsorption bzw. Unschädlichmachung von insbesondere sauren Schadstoffen bzw. Schadgasen eignet, wobei die erfindungsgemäß bereitgestellte Aktivkohle gegenüber dem Stand der Technik weiterführend verbessert sein soll und insbesondere auch chemisorptive Eigenschaften aufweisen soll. Insbesondere soll im Rahmen der vorliegenden Erfindung ein effizientes Verfahren eingesetzt werden, welches die Bereitstellung einer Aktivkohle ermöglicht, die in effizienter Weise mit einer reaktiven bzw. katalytisch aktiven Komponente in Form von Stickstoff ausgerüstet ist, wobei gleichzeitig gewährleistet sein soll, dass die zugrundeliegende Ausrüstung mit der katalytisch aktiven Komponente dauerhaft ist und zu hervorragenden reaktiven bzw. katalytischen Eigenschaften der resultierenden Aktivkohle führt.

[0023] Wie die Anmelderin nun in völlig überraschender Weise herausgefunden hat, kann die zuvor angeführte und der vorliegenden Erfindung zugrundeliegende Aufgabe in nicht erwarteter Weise dadurch gelöst werden, dass im Rahmen der vorliegenden Erfindung eine stickstoffmodifizierte bzw. stickstofffunktionalisierte

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Aktivkohle bereitgestellt wird, welche durch ein Verfahren erhältlich ist, wonach als Ausgangsmaterial Aktivkohle als solche eingesetzt wird, welche zunächst einer Oberflächenoxidation unter Einsatz mindestens eines Oxidationsreagenzes ("OR") einer Oberflächenoxidation unterzogen wird und wonach die so erhaltene, an ihre Oberfläche oxidierte Aktivkohle nachfolgend einer Ausrüstung mit Stickstoff bzw. mit stickstoffhaltigen funktionellen Gruppen unter Einsatz mindestens eines Stickstoffmodifizierungsreagenzes ("SMR") unterzogen wird.

[0024] Zur Lösung der zuvor geschilderten Aufgabe schlägt die vorliegende Erfindung somit – gemäß einem ersten Aspekt der vorliegenden Erfindung – die erfindungsgemäße stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohle, insbesondere mit katalytischen bzw. reaktiven Eigenschaften, vorzugsweise eine Aktivkohle mit Stickstoff als katalytisch aktivem Heteroatom, welche mit einem Verfahren zu ihrer Herstellung bereitgestellt ist, gemäß Anspruch 1 vor. Weitere, insbesondere vorteilhafte Ausgestaltungen des erfindungsgemäß durchgeführten Verfahrens sind Gegenstand der diesbezüglichen Unteransprüche.

[0025] Weiterer Gegenstand der vorliegenden Erfindung – gemäß einem zweiten Aspekt der vorliegenden Erfindung – ist die stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohle nach der Erfindung, welche insbesondere katalytische bzw. reaktive Eigenschaften und welche vorzugsweise Stickstoff als katalytisch aktives Heteroatom aufweist, gemäß den diesbezüglich unabhängigen Ansprüchen. Weitere, insbesondere vorteilhafte Ausgestaltungen der erfindungsgemäßen Aktivkohle sind Gegenstand der entsprechenden Unteransprüche.

[0026] Darüber hinaus sind weiterer Gegenstand der vorliegenden Erfindung – gemäß einem dritten Aspekt der vorliegenden Erfindung – die erfindungsgemäßen Schutzmaterialien, insbesondere für den zivilen bzw. militärischen Bereich, insbesondere Schutzbekleidung, gemäß dem diesbezüglich unabhängigen Anspruch.

[0027] Schließlich sind weiterer Gegenstand der Erfindung – gemäß einem vierten Aspekt der vorliegenden Erfindung – Filter und Filtermaterialien, insbesondere zur Entfernung von Schad-, Geruchs- und Giftstoffen aller Art, gemäß dem betreffenden unabhängigen Anspruch.

[0028] Es versteht sich von selbst, dass bei der nachfolgenden Beschreibung der vorliegenden Erfindung solche Ausgestaltungen, Ausführungsformen, Vorteile, Beispiele oder dergleichen, welche nachfolgend – zu Zwecken der Vermeidung unnötiger Wiederholungen – nur zu einem einzelnen Erfindungsaspekt ausgeführt werden, selbstverständlich auch in Bezug auf die übrigen Erfindungsaspekte entsprechend gelten, ohne dass dies einer ausdrücklichen Erwähnung bedarf.

[0029] Weiterhin versteht es sich von selbst, dass bei nachfolgenden Angaben von Werten, Zahlen und Bereichen die diesbezüglichen Werte-, Zahlen- und Bereichsangaben nicht beschränkend zu verstehen sind; es versteht sich dabei für den Fachmann von selbst, dass einzelfallbedingt oder anwendungsbezogen von den angegebenen Bereichen bzw. Angaben abgewichen werden kann, ohne dass der Rahmen der vorliegenden Erfindung verlassen ist.

[0030] Zudem gilt, dass alle im Folgenden genannten Werte- bzw. Parameterangaben oder dergleichen grundsätzlich mit genormten bzw. standardisierten oder explizit angegebenen Bestimmungsverfahren oder aber andernfalls mit dem Fachmann auf diesem Gebiet an sich geläufigen Bestimmungs- bzw. Messmethoden ermittelt bzw. bestimmt werden können.

[0031] Im Übrigen gilt, dass bei allen nachstehend aufgeführten relativen bzw. prozentualen, insbesondere gewichtsbezogenen Mengenangaben, zu beachten ist, dass diese Angaben im Rahmen der vorliegenden Erfindung vom Fachmann derart auszuwählen bzw. zu kombinieren sind, dass in der Summe – gegebenenfalls unter Einbeziehung weiterer Komponenten bzw. Inhaltsstoffen, insbesondere wie nachfolgend definiert – stets 100% bzw. 100 Gew.-% resultieren. Dies versteht sich für den Fachmann aber von selbst.

[0032] Dies vorausgeschickt, wird im Folgenden die vorliegende Erfindung näher beschrieben:
Gemäß einem ersten Aspekt der vorliegenden Erfindung betrifft die vorliegende Erfindung eine stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohle, welche durch ein Verfahren zu deren Herstellung erhältlich ist, wobei die mit dem erfindungsgemäß eingesetzten Verfahren hergestellte Aktivkohle katalytische bzw. reaktive Eigenschaften aufweist. Bei der mit dem erfindungsgemäß eingesetzten Verfahren hergestellten Aktivkohle handelt es sich insbesondere um eine Aktivkohle mit Stickstoff als katalytisch aktivem Heteroatom. Das erfindungsgemäß durchgeführte Verfahren zeichnet sich dadurch aus, dass eine als Ausgangsmaterial eingesetzte Aktivkohle ("Ausgangsaktivkohle") zunächst einer Oberflächenoxidation unter Verwendung bzw. in Gegenwart mindestens eines Oxidationsreagenzes ("OR") unterzogen wird und die auf diese Weise erhaltene, an ih-

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rer Oberfläche oxidierte Aktivkohle ("Intermediat") nachfolgend einer Ausrüstung, insbesondere Modifizierung bzw. Funktionalisierung, mit Stickstoff, insbesondere mit stickstoffhaltigen funktionellen Gruppen, unter Verwendung bzw. in Gegenwart mindestens eines Stickstoffmodifizierungsreagenzes ("SMR") unterzogen wird.

[0033] Wie zuvor angeführt, besteht die grundlegende Idee der vorliegenden Erfindung somit darin, eine Aktivkohle auf Basis eines Verfahrens bereitzustellen, auf dessen Basis es möglich ist, eine Aktivkohle als Ausgangsmaterial in optimierter Weise mit Stickstoff zu modifizieren bzw. zu funktionalisieren, wobei Stickstoff als katalytisch aktives Heteroatom fungiert und wobei Stickstoff in gezielter Weise insbesondere an die Oberflächenstruktur der eingesetzten Aktivkohle gekoppelt bzw. chemisch gebunden bzw. dauerhaft immobilisiert wird, und zwar in Form von stickstoffhaltigen funktionellen Gruppen einerseits oder aber nach Art einer Dotierung mit Stickstoff bzw. einer N-Dotierung unter Integration bzw. Einbau in das Kohlenstoffgerüst der Aktivkohle (d. h. unter Immobilisierung bzw. Einbindung des Stickstoffs in bzw. an das Kohlenstoffgerüsts des Endprodukts) andererseits.

[0034] In diesem Zusammenhang ist es im Rahmen der vorliegenden Erfindung völlig überraschend, dass durch die der eigentlichen Stickstofffixierung bzw. -modifizierung vorgeschaltete Oberflächenoxidation der als Ausgangsmaterial eingesetzten Aktivkohle eine signifikante Verbesserung der Stickstoffmodifizierung bzw. Stickstofffunktionalisierung der Aktivkohle erreicht wird. Ohne sich auf diese Theorie beschränken bzw. festlegen zu wollen, wird durch die gezielte und zweckgerichtete Oxidation der Oberfläche der eingesetzten Aktivkohle unter Verwendung spezieller Oxidationsreagenzien ("OR") die Oberflächenstruktur der Aktivkohle im Hinblick auf die Modifizierung bzw. Funktionalisierung mit Stickstoff insofern optimiert, als zum einen hydrophile Strukturen geschaffen bzw. die Hydrophilität der Aktivkohle als solche gesteigert wird, was die Interaktion mit dem Stickstoffmodifizierungsreagenz ("SMR") verbessert. Zum anderen werden durch die gezielte Oxidation der Oberfläche der Aktivkohle insbesondere solche funktionellen chemischen Gruppen geschaffen, welche mit dem Stickstoffmodifizierungsreagenz ("SMR") insbesondere unter Ausbildung chemischer Bindungen zu interagieren bzw. zu reagieren imstande sind, was die chemische Bindung bzw. Integration des Stickstoffs in Bezug auf die Aktivkohle verbessert bzw. ermöglicht. Auf Basis der erfindungsgemäß durchgeführten Oxidation im Sinne einer Vorbehandlung der Aktivkohle wird die Affinität der Aktivkohle zum Stickstoffmodifizierungsreagenz ("SMR") erhöht.

[0035] Wie nachfolgend noch im Einzelnen geschildert, ist es aufgrund der speziellen Verfahrensführung möglich, die resultierende stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohle bzw. die mit Stickstoff ausgerüstete Aktivkohle individuell anzupassen bzw. maßzuschneidern, was sowohl auf Ebene der Oxidation der eingesetzten (Ausgangs-)Aktivkohle als auch im Hinblick auf die nachfolgende Stickstoffmodifizierung möglich ist, so dass im Ergebnis auf Basis des erfindungsgemäß durchgeführten Verfahrens Aktivkohlen bereitgestellt werden, welche insgesamt einem breiten Einsatzspektrum zugänglich sind bzw. im Speziellen für die Adsorption bzw. Unschädlichmachung spezieller toxischer Substanzen optimiert sind.

[0036] Ein weiterer zentraler Vorteil der erfindungsgemäß durchgeführten Verfahrensführung ist darin zu sehen, dass eine Vielzahl kommerziell erhältlicher bzw. handelsüblicher Aktivkohlen als Ausgangsmaterial für die erfindungsgemäß vorgesehene Verfahrensführung bzw. Modifizierung eingesetzt werden können, so dass das erfindungsgemäß durchgeführte Verfahren gewissermaßen universell im Hinblick auf die Ausgangsmaterialien auf Basis von Aktivkohle, beispielsweise mit variabler Porosität oder dergleichen, eingesetzt werden kann. Aufgrund der sozusagen nachgeschalteten Modifizierung bzw. Ausrüstung mit Stickstoff werden im Rahmen der Aktivkohleherstellung als solche vorliegende Prozesse, wie Carbonisierung und Aktivierung, nicht beeinflusst.

[0037] Aufgrund der erfindungsgemäß realisierten Verfahrensführung mit der Funktionalisierung der Aktivkohle mit stickstoffhaltigen funktionellen Gruppen bzw. mit der gezielten Integration von Stickstoff in die Kohlenstoffoberfläche bzw. in das Gerüst der Aktivkohle werden somit spezielle stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohlen erhalten, welche eine katalytische Aktivität bzw. Reaktivität und somit chemisorptive Eigenschaften gegenüber unterschiedlichen Substanzen, insbesondere toxischen gasförmigen Substanzen, aufweisen. Diesbezüglich weisen die mit dem erfindungsgemäß durchgeführten Verfahren erhaltenen stickstoffmodifizierten bzw. stickstofffunktionalisierten Aktivkohlen eine hohe Eignung für den insbesondere katalytischen bzw. reaktiven Abbau verschiedener toxischer Gase auf. Hierbei kann es sich beispielsweise und in nicht beschränkender Weise um sogenannte Chemical Warfare Agents (CWAs), Toxic Industrial Materials (TIMs), wie größere Moleküle, beispielsweise Ethylenoxid oder dergleichen, um Toxic Industrial Chemicals (TICs), wie NH_3, SO_2, H_2S, Cl_2, HCl oder dergleichen, handeln. Insbesondere eignet sich die mit dem erfindungsgemäß durchgeführten Verfahren erhaltene stickstofffunktionalisierte bzw. stickstoffmodifizierte Aktivkohle bzw. die mit Stickstoff ausgerüstete Aktivkohle zur Adsorption bzw. Unschädlichmachung von insbesondere sauren toxischen Gasen, wie H_2S, SO_2 bzw. HCl.

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[0038] Ohne sich auf diese Theorie beschränken bzw. festlegen zu wollen, ist insbesondere ein Abbau auch von solchen toxischen Gasen bzw. Schadgasen über eine Oxidation an der Kohlenstoffoberfläche der modifizierten Aktivkohle möglich, wie es beispielsweise in nicht beschränkender Weise für SO_2, H_2S, AsH_3 sowie DMS, möglich ist. Dies ist insbesondere dann der Fall, wenn der Stickstoff, wie zuvor geschildert, in das Kohlenstoffgerüst der erfindungsgemäß modifizierten Aktivkohle nach Art einer N-Dotierung integriert bzw. inkorporiert ist, d. h. der Stickstoff sozusagen immobilisiert ist, insbesondere durch chemische und/oder kovalente Bindung in das Kohlenstoffgerüst der erfindungsgemäß modifizierten Aktivkohle.

[0039] Darüber hinaus weist die auf Basis des erfindungsgemäß eingesetzten Verfahrens erhältliche stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohle – gleichermaßen ohne sich auf diese Theorie beschränken bzw. festlegen zu wollen – eine katalytische Aktivität bzw. reaktive Eigenschaften gegenüber solchen toxischen Substanzen bzw. Gasen auf, welche mit den ausgebildeten stickstoffhaltigen funktionellen Gruppen auf der Oberfläche der erfindungsgemäß modifizierten Aktivkohle zu reagieren imstande sind, wie beispielsweise Formaldehyd und NO_x-Verbindungen.

[0040] Auf Basis der der vorliegenden Erfindung zugrundeliegenden Konzeption mit der speziellen Ausrüstung von Aktivkohle mit Stickstoff wird somit in völlig überraschender Weise eine Aktivkohle nach der Erfindung bereitgestellt, welche über hervorragende Eigenschaften hinsichtlich der (Chemi-)Sorption bzw. des katalytischen und/oder reaktiven Abbaus von unterschiedlichen Schadstoffen bzw. toxischen Gasen, wie chemischen Gift- bzw. Kampfstoffen, verfügt. Beispielsweise eignet sich die so ausgerüstete Aktivkohle auch für den insbesondere oxidativen Abbau von insbesondere schwefelhaltigen Komponenten eignen. Gleichermaßen können die so erhaltenen Aktivkohlen durch eine zweckgerichtete Optimierung der diesbezüglichen Ausrüstungen für Breitbandanwendungen, beispielsweise im Rahmen von Atemschutzanwendungen, eingesetzt werden. In diesem Zusammenhang kommt auch ein Einsatz in ABEK-Filtern, insbesondere in Kombination mit weiteren Adsorbentien in Betracht.

[0041] Im Allgemeinen eignet sich die erfindungsgemäße Aktivkohle beispielsweise zu Gasadsorptionen und insbesondere zur Adsorption von Schwefel enthaltenden Gasen (wie Schwefeloxiden und/oder Schwefelwasserstoff), von stickstoffenthaltenden Gasen (wie Stickoxiden und/oder Stickstoffwasserstoffen, wie Ammoniak) sowie von halogenhaltigen Gasen (wie Chlorwasserstoffgas). Gleichermaßen eignet sich die erfindungsgemäße Aktivkohle auch zur Adsorption von organisch basierten schädlichen Substanzen, wie beispielsweise Formaldehyd oder dergleichen. Insbesondere weist die erfindungsgemäße Aktivkohle besonders gute Eigenschaften im Hinblick auf die Sorption, insbesondere Physisorption und/oder Chemisorption, von Schwefel- und Stickstoffoxiden, Halogenwasserstoffen, Cyangas, Cyanwasserstoff, Schwefelwasserstoff, Chlorgas, Arsenwasserstoff und/oder Ammoniak auf.

[0042] Aufgrund der erfindungsgemäßen Konzeption des erfindungsgemäß eingesetzten Verfahrens mit der sozusagen vorgeschalteten Oberflächenoxidation bzw. der der Oberflächenoxidation nachgeschalteten Ausrüstung mit Stickstoff der zu behandelnden Aktivkohle als Ausgangsmaterial wird eine besonders homogene bzw. gleichmäßige Ausrüstung bzw. Beladung der Aktivkohle – und zwar insbesondere über das gesamte Porenspektrum – mit dem zu inkorporierenden Stickstoff erreicht, da das Stickstoffmodifizierungsreagenz ("SMR") besonders gut in das Porensystem eindringen kann und dort aufgrund der vorgeschalteten Oxidation mit der hohen Affinität der Reaktionsparameter zueinander eine gleichmäßige Beladung der Aktivkohle unter Ausbildung stickstoffhaltiger funktioneller Gruppen bzw. unter Integration bzw. Immobilisierung von Stickstoff in das Kohlenstoffgerüst gewährleistet.

[0043] Das erfindungsgemäß durchgeführte Verfahren zeichnet sich dabei durch eine gewissermaßen zweistufige Verfahrensführung aus, wobei nämlich zunächst herkömmliche Aktivkohle als Ausgangsmaterial einer Oberflächenoxidation unterzogen wird und nachfolgend die so erhaltene, an ihrer Oberfläche oxidierte Aktivkohle nachfolgend in zweckgerichteter Weise unter Verwendung eines Stickstoffmodifizierungsreagenzes ("SMR") mit Stickstoff ausgerüstet wird, und zwar auf Basis von stickstoffhaltigen funktionellen Gruppen und/oder auf Basis der Inkorporation bzw. des Einbaus von Stickstoff im Sinne einer Stickstoffdotierung (N-Dotierung) in das Kohlenstoffgerüst, insbesondere in das die äußere und innere Oberfläche bildende Kohlenstoffgerüst.

[0044] In diesem Zusammenhang ist es erfindungsgemäß zudem völlig überraschend, dass das Porensystem der als Ausgangsmaterial eingesetzten Aktivkohle zumindest im Wesentlichen nicht durch die Ausrüstung mit dem Stickstoff beeinflusst wird. Mit anderen Worten ist es erfindungsgemäß gewährleistet, dass das Porensystem der Aktivkohle nicht bzw. zumindest im Wesentlichen nicht nachteilig hinsichtlich der Adsorption von Schadstoffen beeinflusst wird. Die auf Basis des erfindungsgemäß durchgeführten Verfahrens erhältliche Ak-

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tivkohle weist somit auch nach Ausrüstung bzw. Funktionalisierung mit Stickstoff z. B. eine hohe, der Adsorption von Schadstoffen zugängliche BET-Oberfläche auf, so dass die grundlegenden Eigenschaften der Aktivkohle als solche auch bei Ausrüstung mit Stickstoff erhalten bleiben, was nicht vorhersehbar war. Somit ist die erfindungsgemäße Vorgehensweise im Gegensatz zu der im Stand der Technik bekannten Imprägnierung mit dem Vorteil verbunden, dass kein Verlust an spezifischer Oberfläche bzw. Gesamtporenvolumen vorliegt und auch die Porenstruktur als solche im Hinblick auf ihre Verteilung in Form von Mikro-, Meso- und Makroporen zumindest im Wesentlichen nicht verändert wird.

[0045] Diesbezüglich war es im Rahmen der vorliegenden Erfindung völlig überraschend, dass durch die gezielte Ausrüstung der Aktivkohle mit Stickstoff – im grundlegenden Unterschied zu den Verfahren des Standes der Technik – nicht zu Verstopfungen des Porensystems führt, da die Stickstoffkomponente dauerhaft unter Ausbildung chemischer Bindungen in die Kohlenstoffstruktur der Aktivkohle integriert ist, und zwar entweder auf Basis stickstoffhaltiger funktioneller Gruppen oder unter Integration in die Kohlenstoffmatrix der behandelten bzw. modifizierten Aktivkohle. Insbesondere wird aufgrund der erfindungsgemäß realisierten Verfahrensführung eine Kristallbildung bzw. eine Auskristallisation innerhalb des Porensystems verhindert, was die Leistungsfähigkeit der erfindungsgemäßen Aktivkohle noch weiter verbessert. Auch hierin ist ein entscheidender Unterschied zu dem im Stand der Technik bekannten Imprägnierverfahren zu sehen, welche insbesondere bei mikroporöser Aktivkohle zu einem Auftreten von Verstopfungen im Hinblick auf die Porenstruktur der Aktivkohle führt.

[0046] Aufgrund der gezielten Ausrüstung der Aktivkohle mit Stickstoff als katalytisch aktives Heteroatom weist die mit dem erfindungsgemäß durchgeführten Verfahren hergestellte Aktivkohle, wie zuvor angeführt, sozusagen eine reaktive bzw. katalytische Oberfläche in Ergänzung zu den chemisorptiven und/oder physisorptiven Eigenschaften der Aktivkohle als solche auf. Die mit dem erfindungsgemäß durchgeführten Verfahren erhältliche Aktivkohle vereint folglich chemisorptive wie physisorptive Eigenschaften in einem einzigen Material und ist somit imstande, die Sorbate bzw. die zu sorbierenden Substanzen – im Unterschied zu nichtkatalytischer bzw. nichtreaktiver Aktivkohle – auch zuverlässig und irreversibel bzw. dauerhaft zu binden bzw. abzubauen.

[0047] Auch durch die besonders homogene bzw. gleichmäßige Ausrüstung der mit dem erfindungsgemäß durchgeführten Verfahren erhältlichen Aktivkohle mit der Stickstoffkomponente wird die Leistungsfähigkeit der so erhaltenen stickstoffmodifizierten bzw. stickstofffunktionalisierten Aktivkohle im Hinblick auf die Sorption bzw. den Abbau zu entfernender Substanzen nochmals gesteigert, insbesondere da die so erhältliche Aktivkohle gewissermaßen über einen großen Teil ihrer Oberfläche über kombinierte Eigenschaften der Physisorption einerseits und der Chemisorption auf Basis reaktiver bzw. katalytischer Eigenschaften andererseits verfügt, welche sich zudem über die Summe der Einzelmaßnahmen hinaus verstärken, was als Indiz für das Vorliegen eines synergistischen Effekts gewertet werden muss.

[0048] Im Rahmen der vorliegenden Erfindung wird somit ein leistungsfähiges Verfahren eingesetzt, welches zu einem optimierten Adsorptionssystem mit kombinierten und sich ergänzenden Eigenschaften von Physisorption einerseits und Chemisorption andererseits führt.

[0049] Wie zuvor angeführt, resultiert erfindungsgemäß eine funktionalisierte Aktivkohle, welche im Unterschied zum Stand der Technik jedoch zumindest im Wesentlichen keine signifikante Beeinträchtigung anderer physikochemischer Parameter, wie beispielsweise Porosität, Porenvolumen, Porenverteilung und dergleichen, aufweist.

[0050] Im Rahmen der vorliegenden Erfindung ist es zudem völlig überraschend, dass sich Aktivkohle als solche überhaupt in effektiver Weise hinsichtlich der erfindungsgemäß vorgesehenen Stickstoffmodifizierung bzw. Stickstofffunktionalisierung eignet. Denn Aktivkohle weist also solche grundsätzlich hydrophobe Eigenschaften auf, was einer effizienten Behandlung bzw. Interaktion mit dem Stickstoffmodifizierungsreagenz ("SMR") zur Ausbildung funktioneller Gruppen bzw. zur Integration von Stickstoff grundsätzlich abträglich ist. Insbesondere ist es jedoch aufgrund der speziellen erfindungsgemäß durchgeführten Verfahrensweise mit der zweckgerichteten vorgeschalteten Oberflächenoxidation der als Ausgangsmaterial eingesetzten Aktivkohle in völlig überraschender und nicht vorhersehbarer Weise gelungen, die Kompatibilität von Aktivkohle einerseits und Stickstoffmodifizierungsreagenz ("SMR") andererseits zu erhöhen, um Aktivkohle als solche in effektiver Weise mit Stickstoff zu funktionalisieren bzw. zu modifizieren, was zu den zuvor angeführten positiven Eigenschaften hinsichtlich der Sorption von Schadstoffen führt.

[0051] Zusammenfassend zeichnet sich die erfindungsgemäß realisierte Verfahrensführung somit dadurch aus, dass eine speziell mit Stickstoff modifizierte bzw. funktionalisierte Aktivkohle erhalten wird, welche über

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hervorragende Adsorptionseigenschaften auf Basis einer Kombination von Physisorption und Chemisorption verfügt. Insbesondere tritt eine bei herkömmlicher Aktivkohle, z. B. bei Erschöpfung oder übermäßiger Beladung häufig vorkommende Freisetzung bzw. Resorption bereits sorbierter Substanzen bei der erfindungsgemäßen Aktivkohle nicht bzw. nicht in nennenswertem Maße auf. Aufgrund der erfindungsgemäßen Konzeption der Bereitstellung einer Aktivkohle, welche neben ihren inhärenten adsorptiven Eigenschaften gleichermaßen reaktive bzw. katalytische Eigenschaften aufweist, wird eine dauerhaft und im Allgemeinen irreversible Bindung bzw. ein effizienter Abbau sorbierter Substanzen gewährleistet, was gleichermaßen zu einer verringerten Desorption der in Rede stehenden Substanzen und zu einer Steigerung der Gesamtadsorptionskapazität führt. Aufgrund der festen chemischen Anbindung von Stickstoff an die Aktivkohlestruktur liegt zudem eine dauerhafte und beständige Modifizierung der Aktivkohle mit Stickstoff vor. Insbesondere kann der dauerhaft immobilisierte Stickstoff nicht durch Auswaschen oder dergleichen wieder von der Aktivkohle entfernt werden.

[0052] Erfindungsgemäß kann das eingesetzte Verfahren weiter zusammenfassend insofern charakterisiert werden, als zunächst im Rahmen der Verfahrensdurchführung eine Ausgangsaktivkohle bzw. ein Basisadsorbens einer chemischen bzw. thermischen Oxidation zum Erhalt einer an ihrer Oberfläche oxidierten Aktivkohle (oxidiertes Basisadsorbens) unterzogen wird, wobei das so erhaltene Intermediat nachfolgend bzw. in einem sich der Oxidation anschließenden Schritt bzw. in einem separaten Schritt mit Stickstoff modifiziert bzw. funktionalisiert wird. Hierzu wird zunächst das oxidative Intermediat mit dem Stickstoffmodifizierungsreagenz ("SMR") in Kontakt gebracht, beispielsweise unter Verwendung einer Harnstofflösung, wobei in einem solchen Fall das oxidative Intermediat im Rahmen einer Tauchmethode oder dergleichen mit dem Stickstoffmodifizierungsreagenz ("SMR") in Kontakt gebracht werden kann. Gleichermaßen ist ein Inkontaktbringen auch im Rahmen eines Luftstromes, beispielsweise unter Verwendung von Ammoniak (NH_3) oder dergleichen, möglich. Es schließt sich eine entsprechende thermische Behandlung zur Ausbildung von stickstoffhaltigen Funktionalisierungen bzw. zur Dotierung des Aktivkohlegerüstes mit Stickstoff (d. h. zur Immobilisierung des Stickstoffs) an.

[0053] Was den Begriff "Oberflächenoxidation", wie er erfindungsgemäß verwendet wird, anbelangt, so ist hierunter insbesondere eine Oxidation solcher Oberflächen der als Ausgangsmaterial eingesetzten Aktivkohle zu verstehen, welche in Kontakt mit der insbesondere das Oberflächenreagenz ("OR") enthaltenden Umgebung stehen, bzw. welche sozusagen von außen für das erfindungsgemäß eingesetzte Oxidationsreagenz ("OR") zugänglich sind. Hierbei handelt es sich insbesondere auch um das Porensystem der Aktivkohle auf Basis von Makro-, Meso- und Mikroporen.

[0054] Ohne sich auf diese Theorie beschränken bzw. festlegen zu wollen, resultiert auf Basis der erfindungsgemäß realisierten Verfahrensführung gewissermaßen eine oxidierte Schicht im Hinblick auf die Aktivkohle, welche im Allgemeinen über die nachfolgend noch angeführten sauerstoffhaltigen funktionellen Gruppen verfügt. Die sich der Oberflächenoxidation anschließende Ausrüstung mit dem Stickstoff erfolgt dann – gleichermaßen ohne sich auf diese Theorie beschränken bzw. festlegen zu wollen – insbesondere im Bereich der oxidierten (Grenz-)Schicht, wobei die sauerstoffhaltigen funktionellen Gruppen gewissermaßen als Eindungs- bzw. Ankerstellen bzw. Reaktionspartner für das erfindungsgemäß eingesetzte Stickstoffmodifizierungsreagenz ("SMR") fungieren.

[0055] Durch die sich der Oxidation anschließende Ausrüstung der Aktivkohle im Rahmen des durchgeführten Verfahrens mit Stickstoff als katalytisch aktivem Heteroatom werden somit Aktivkohlen mit reaktiven bzw. katalytisch aktiven Oberflächen generiert.

[0056] Was das erfindungsgemäß durchgeführte Verfahren weiterhin anbelangt, so kann es vorgesehen sein, dass die Oberflächenoxidation unter Ausbildung einer hydrophilen Oberfläche der Aktivkohle durchgeführt wird. Erfindungsgemäß ist es gleichermaßen möglich, dass die Oberflächenoxidation zu einer Ausbildung von sauerstoffhaltigen funktionellen Gruppen an der Oberfläche der Aktivkohle führt. In diesem Zusammenhang können die sauerstoffhaltigen funktionellen Gruppen ausgewählt werden aus sauren und basischen sauerstoffhaltigen funktionellen Gruppen sowie deren Kombinationen, insbesondere sauren und basischen Oberflächenoxiden. Insbesondere können die sauerstoffhaltigen funktionellen Gruppen ausgewählt werden aus der Gruppe von Hydroxyl-, Carboxyl-, Carbonyl-, Anhydrid-, Lacton-, Chinon-, Pyron-, Chromen- und Ethergruppen sowie deren Kombinationen. Derartige Gruppen eigenen sich in besonderer Weise zur Interaktion mit dem Stickstoffmodifizierungsreagenz ("SMR").

[0057] Wie zuvor angeführt, führen die an der Oberfläche der Aktivkohle insbesondere durch Reaktion des Oxidationsreagenzes ("OR") mit der als Ausgangsmaterial eingesetzten Aktivkohle ("Ausgangsaktivkohle") entstehenden sauerstoffhaltigen funktionellen Gruppen zu einer Erhöhung der Hydrophilität bzw. der hydrophilen Eigenschaften der so behandelten Aktivkohle, was die Interaktion mit dem nachfolgend eingesetzten Stickstoff-

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modifizierungsreagenz ("SMR") verbessert und in entsprechender Weise zu einer verbesserten Funktionalisierung bzw. Modifizierung der Aktivkohle mit Stickstoff führt. Insbesondere können die auf diese Weise generierten sauerstoffhaltigen funktionellen Gruppen als Reaktionspartner für das Stickstoffmodifizierungsreagenz ("SMR") fungieren, um so die chemische Bindung bzw. den Einbau bzw. Integration von Stickstoff in die zu modifizierende Aktivkohle zu verbessern bzw. zu ermöglichen. Die erfindungsgemäß durchgeführte und vorgeschaltete Oberflächenoxidation dient somit gewissermaßen zur Vorbereitung der nachfolgenden Stickstoffmodifizierung bzw. -funktionalisierung der Aktivkohle. In diesem Zusammenhang wird durch die erfindungsgemäß realisierte Oberflächenoxidation insbesondere auch die Oberflächenhydrophilie des Kohlenstoffgerüsts der zu behandelnden Aktivkohle erhöht, was zu den vorgenannten Vorteilen führt.

[0058] Was das erfindungsgemäß durchgeführte Verfahren weiterhin anbelangt, so kann durch die zweckgerichtete Oberflächenoxidation auch der pH-Wert, insbesondere der Oberflächen-pH-Wert, der an ihrer Oberfläche oxidierten Aktivkohle eingestellt werden, und zwar im Vergleich zu der Ausgangsaktivkohle um einen Wert von mindestens 0,25, insbesondere mindestens 0,5, vorzugsweise mindestens 0,75, bevorzugt mindestens 1, besonders bevorzugt mindestens 1,5, verändert werden. Insbesondere kann es vorgesehen sein, dass die Acidität der an ihrer Oberfläche oxidierten Aktivkohle erhöht und/oder der pH-Wert, insbesondere der Oberflächen-pH-Wert, der an ihrer Oberfläche oxidierten Aktivkohle durch die vorgenommene Oxidation der Aktivkohle verringert wird, insbesondere um einen Wert von mindestens 0,25, insbesondere mindestens 0,5, vorzugsweise mindestens 0,75, bevorzugt mindestens 1, besonders bevorzugt mindestens 1,5, oder dass durch die vorgenommene Oxidation der Aktivkohle die Basizität der an ihrer Oberfläche oxidierten Aktivkohle erhöht und/oder der pH-Wert, insbesondere der Oberflächen-pH-Wert, der an ihrer Oberfläche oxidierten Aktivkohle erhöht wird, insbesondere um einen Wert von mindestens 0,25, insbesondere mindestens 0,5, vorzugsweise mindestens 0,75, bevorzugt mindestens 1, besonders bevorzugt mindestens 1,5. Durch die zweckgerichtete Einstellung des pH-Werts auf Basis der erfindungsgemäß durchzuführenden Oberflächenoxidation der Ausgangsaktivkohle kann insbesondere die Affinität zum nachfolgend eingesetzten Stickstoffmodifizierungsreagenz ("SMR") weiter optimiert werden, was zu einer noch weitergehenden Effizienzsteigerung hinsichtlich der Ausrüstung der auf Basis des erfindungsgemäß durchgeführten Verfahrens erhältlichen Aktivkohle mit Stickstoff führt. Insbesondere resultiert bei Verwendung von Ausgangsaktivkohlen mit relativ hohem pH-Wert bzw. basischen Ausgangsaktivkohlen eine durch die Oberflächenoxidation hervorgerufene Verringerung des Oberflächen-pH-Wertes.

[0059] Erfindungsgemäß kann es auch vorgesehen sein, dass die Oberflächenoxidation durchgeführt wird derart, dass die auf diese Weise erhaltene, an ihrer Oberfläche oxidierte Aktivkohle einen Gehalt an sauerstoffhaltigen funktionellen Gruppen, berechnet und/oder ausgedrückt als Gehalt an flüchtigen Bestandteilen ("fB") und bezogen auf das Trockengewicht der an ihrer Oberfläche oxidierten Aktivkohle, von mindestens 1 Gew.-%, insbesondere mindestens 2 Gew.-%, vorzugsweise mindestens 3 Gew.-%, bevorzugt mindestens 4 Gew.-%, und/oder im Bereich von 1 Gew.-% bis 30 Gew.-%, insbesondere 1,5 Gew.-% bis 25 Gew.-%, vorzugsweise 2 Gew.-% bis 20 Gew.-%, bevorzugt 3 Gew.-% bis 15 Gew.-%, aufweist.

[0060] In diesem Zusammenhang kann im Rahmen der erfindungsgemäß realisierten Verfahrensführung der Gehalt an sauerstoffhaltigen funktionellen Gruppen beispielsweise durch entsprechende Wahl der Temperatur und/oder Zeitdauer und/oder Art und/oder Konzentration an Oxidationsreagenz ("OR") eingestellt werden. Auf diese Weise kann eine Maßschneiderung des Oxidationsprozesses insbesondere im Hinblick auf die nachfolgende Stickstofffunktionalisierung bzw. -modifizierung erfolgen. Im Rahmen der erfindungsgemäß realisierten Verfahrensführung wird somit der Sauerstoffgehalt der an ihrer Oberfläche oxidierten Aktivkohle im Vergleich zu der eingesetzten Ausgangsaktivkohle – welche im Allgemeinen einen Sauerstoffgehalt von weniger als 1 Gew.-%, ausgedrückt als Gehalt an flüchtigen Bestandteilen ("fB") und bezogen auf das Trockengewicht der Ausgangsaktivkohle, aufweist – signifikant erhöht.

[0061] Der Gehalt an flüchtigen Bestandteilen ("fB") fungiert dabei im Allgemeinen als Maß für die Oxidation und bezieht sich insbesondere auf durch die Oxidation gebildete Oberflächenoxide. Insbesondere kann der Gehalt an flüchtigen Bestandteilen auf Basis der ISO 562:1981 bestimmt werden. Insbesondere kann der Gehalt an flüchtigen Bestandteilen ("fB") an der zuvor getrockneten, an ihrer Oberfläche oxidierten Aktivkohle bei einer entsprechenden Erwärmung für einen Zeitraum von 7 Minuten auf 900°C unter inerten Bedingungen bestimmt werden.

[0062] Durch die Einstellung des Gehalts an sauerstoffhaltigen funktionellen Gruppen kann auch die Menge des nachfolgend einzubauenden Stickstoffs vorgegeben bzw. beeinflusst werden, insbesondere durch eine entsprechende Vorgabe der Anzahl an Reaktionspartnern. Die aufgenommene Menge an Stickstoff der erfindungsgemäß bereitgestellten stickstoffmodifizierten bzw. stickstofffunktionalisierten Aktivkohle korreliert somit

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mit dem Gehalt bzw. der Menge an Oberflächenoxiden bzw. mit den flüchtigen Bestandteilen der oxidierten Basis in Form der an ihrer Oberfläche oxidierten Aktivkohle.

[0063] Was das im Rahmen der Oberflächenoxidation eingesetzte Oxidationsreagenz ("OR") anbelangt, so können hierzu eine Vielzahl an Reagenzien bzw. Verbindungen eingesetzt werden. Grundsätzlich erfolgt dabei der Oxidationsvorgang, wie nachfolgend geschildert, für einen definierten Zeitraum insbesondere unter Wärme- bzw. Temperatureinfluss.

[0064] Im Allgemeinen kann das Oxidationsreagenz ("OR") ausgewählt werden aus der Gruppe von Sauerstoff, Ozon, anorganischen oder organischen Oxiden und Peroxiden sowie anorganischen oder organischen Säuren und Persäuren und deren Kombinationen. In erfindungsgemäß bevorzugter Weise wird das Oxidationsreagenz ausgewählt aus der Gruppe von Sauerstoff, Wasserstoffperoxid (H_2O_2), Salpetersäure (HNO_3) und Ammoniumperoxodisulfat ($(NH4)_2S_2O_8$) sowie deren Kombinationen. Im Rahmen der vorliegenden Erfindung wird insbesondere bei der Verwendung von Sauerstoff, welcher der zu oxidierenden Aktivkohle insbesondere in Form von atmosphärischer Luft zugeführt werden kann, auch von einer sogenannten thermischen Oxidation gesprochen, da diese in der Regel unter Erwärmung stattfindet, wohingegen bei Verwendung der weiteren Oxidationsreagenzien, insbesondere von H_2O_2 bzw. HNO_3 erfindungsgemäß im Allgemeinen auch von einer chemischen Oxidation gesprochen wird, das in diesem Fall in Abhängigkeit von dem eingesetzten Reagenz mitunter keine zusätzliche Erwärmung erforderlich ist, obwohl diese im Allgemeinen auch hier durchgeführt werden kann. Insbesondere können die zuvor genannten Oxidationsreagenzien ("OR") in gasförmiger und/oder in flüssiger Form, insbesondere in Form von vorzugsweise wässrigen Lösungen oder dergleichen eingesetzt werden, was dem Fachmann als solches grundsätzlich bekannt ist.

[0065] Im Allgemeinen kann die Oberflächenoxidation unter Erwärmen erfolgen. Hierbei kann die Oberflächenoxidation bei solchen Temperaturen erfolgen, dass eine Reaktion des Oxidationsreagenzes ("OR") mit der Aktivkohle unter Ausbildung von sauerstoffhaltigen funktionellen Gruppen an der Oberfläche der Aktivkohle erfolgt. Insbesondere kann in diesem Zusammenhang die Oberflächenoxidation im Temperaturbereich von –20°C bis 1.000°C, insbesondere 0°C bis 800°C, vorzugsweise 20°C bis 600°C, bevorzugt 30°C bis 500°C, durchgeführt werden. Insbesondere kann in diesem Zusammenhang die Oberflächenoxidation für eine Zeitdauer im Bereich von 1 Minute bis 600 Minuten, insbesondere 5 Minuten bis 500 Minuten, bevorzugt 10 Minuten bis 400 Minuten, durchgeführt werden. Hinreichend kann das Maß der Oberflächenoxidation, insbesondere die Anzahl bzw. der Gehalt an sauerstoffhaltigen Gruppen auf der Aktivkohleoberfläche, vorgegeben werden.

[0066] Gemäß einer ersten erfindungsgemäß besonders bevorzugten Ausführungsform kann das Oxidationsreagenz ("OR") ausgewählt werden aus der Gruppe von anorganischen oder organischen Oxiden und Peroxiden sowie anorganischen oder organischen Säuren und Persäuren und deren Kombinationen, bevorzugt aus der Gruppe von Wasserstoffperoxid (H_2O_2) und Salpetersäure (HNO_3), vorzugsweise Salpetersäure (HNO_3). Insbesondere kann dabei die Oberflächenoxidation dann im Temperaturbereich von –20°C bis 500°C, insbesondere –10°C bis 400°C, vorzugsweise 0°C bis 350°C, bevorzugt 10°C bis 300°C, besonders bevorzugt 15°C bis 250°C, ganz besonders bevorzugt 20°C bis 200°C, durchgeführt werden. Hierbei handelt es sich, wie zuvor beschrieben, sozusagen um eine chemische Oxidation, welche grundsätzlich und in Abhängigkeit des eingesetzten Oxidationsreagenzes auch bei Raumtemperatur durchgeführt werden kann.

[0067] Gemäß einer zweiten erfindungsgemäß besonders bevorzugten Ausführungsform kann das Oxidationsreagenz ("OR") ausgewählt werden aus der Gruppe von Sauerstoff und Ozon, vorzugsweise Sauerstoff. Insbesondere sollte dabei die Oberflächenoxidation dann im Temperaturbereich von 100°C bis 1.000°C, insbesondere 150°C bis 900°C, vorzugsweise 200°C bis 750°C, bevorzugt 225°C bis 600°C, besonders bevorzugt 250°C bis 500°C, ganz besonders bevorzugt 250°C bis 450°C, durchgeführt werden. Wie zuvor angeführt handelt es sich hierbei gewissermaßen um eine thermische Oxidation.

[0068] Was die erfindungsgemäß vorgesehene Oberflächenoxidation der Aktivkohle anbelangt, so handelt es sich hierbei zusammenfassend zum einen gezielt regelbaren bzw. einstellbaren Prozess, welcher auf Basis der Art und Weise des einzusetzenden Oxidationsreagenzes ("OR"), der Oxidationszeit und/oder der Temperatur gesteuert werden kann. Wie zuvor angeführt, wird durch die gezielte Oxidation die auf diese Weise an ihrer Oberfläche oxidierte Aktivkohle für die nachfolgende Funktionalisierung bzw. Modifizierung mit Stickstoff optimiert. So kann beispielsweise in Abhängigkeit von der Oxidationszeit der Gehalt an flüchtigen Bestandteilen als Maß für den Oxidationsgrad erhöht werden. Zudem kann der pH-Wert, insbesondere der Oberflächen-pH-Wert der an ihrer Oberfläche oxidierten Aktivkohle durch Wahl der Oxidationsbedingungen eingestellt bzw. maßgeschneidert werden, beispielsweise über die Dauer der Oxidation. So ist bei der zuvor beschriebenen chemischen Oxidation insbesondere die Bildung saurer Oberflächenoxide bevorzugt, wohingegen bei

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der zuvor beschriebenen thermischen Oxidation insbesondere die Bildung basischer Oberflächenoxide bevorzugt ist. Gleichermaßen hat eine zunehmende Oxidationszeit in Bezug auf die an ihrer Oberfläche oxidierte Aktivkohle einen Einfluss auf die sogenannte Rütteldichte der Aktivkohle, welche im Allgemeinen mit zunehmender Oxidationszeit zumindest geringfügig abnimmt. Gleichermaßen hat die Oxidation auch einen Einfluss auf das Gesamtporenvolumen, insbesondere das Gesamtporenvolumen nach Gurvich, der Aktivkohle, welche beispielsweise mit zunehmender Oxidationszeit zumindest geringfügig vergrößert wird. Im Ergebnis kann somit auf Basis der Oxidation die Oberflächeneigenschaften der Aktivkohle in gezielter Weise hinsichtlich der nachfolgenden Modifizierung mit Stickstoff eingestellt bzw. maßgeschneidert werden.

[0069] Im Rahmen der erfindungsgemäß realisierten Verfahrensführung kann es zudem vorgesehen sein, dass im Anschluss an die Oberflächenoxidation und insbesondere vor der Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, mit Stickstoff eine Aufreinigung bzw. Trocknung der an ihrer Oberfläche oxidierten Aktivkohle erfolgt. In diesem Zusammenhang kann die Aufreinigung mittels mindestens eines Waschvorgangs in einer Flüssigkeit, insbesondere Wasser, erfolgen. Gleichermaßen kann die Trocknung unter Erwärmung der an ihrer Oberfläche oxidierten Aktivkohle, insbesondere auf Temperaturen im Bereich von 40°C bis 200°C, insbesondere 50°C bis 150°C, vorzugsweise 60°C bis 120°C, erfolgen. Durch die gezielte Trocknung der an ihrer Oberfläche oxidierten Aktivkohle wird das so erhaltene Zwischenprodukt bzw. Intermediat weiterführend für die nachfolgende Modifizierung bzw. Funktionalisierung mit Stickstoff optimiert, insbesondere da die Zugänglichkeit für das Stickstoffmodifizierungsreagenz ("SMR") verbessert wird.

[0070] Was die sich der Oberflächenoxidation der Aktivkohle anschließende Ausrüstung, insbesondere Modifizierung bzw. Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff anbelangt, so sollte diese ein Inkontaktbringen der nach der Oberflächenoxidation erhaltenen Aktivkohle mit dem Stickstoffmodifizierungsreagenz ("SMR"), insbesondere gefolgt von einer thermischen Behandlung, umfassen. Auf diese Weise wird die chemische Anbindung bzw. Integration bzw. die Ausbildung stickstoffhaltiger funktioneller Gruppen an der Oberfläche der zuvor oxidierten Aktivkohle gewährleistet. Wie zuvor angeführt, interagiert das Stickstoffmodifizierungsreagenz ("SMR") insbesondere mit den sauerstoffhaltigen Oberflächenfunktionalitäten, welche im Rahmen des vorangehenden Oxidationsschrittes in gezielter Weise auf der Aktivkohle generiert worden sind.

[0071] Durch das Inkontaktbringen der an ihrer Oberfläche oxidierten Aktivkohle mit dem Stickstoffmodifizierungsreagenz ("SMR") wird insbesondere auch das mit der Umgebung in Verbindung stehende Porensystem der Aktivkohle mit dem Stickstoffmodifizierungsreagenz gefüllt bzw. zusammengebracht, was auf diese Weise die nachfolgenden chemischen Reaktionen insbesondere mit den sauerstoffhaltigen funktionellen Gruppen der an ihrer Oberfläche oxidierten Aktivkohle ermöglicht.

[0072] Insbesondere erfolgt im Rahmen der Modifizierung bzw. Funktionalisierung mit Stickstoff eine Reaktion des Stickstoffmodifizierungsreagenzes ("SMR") mit den sauerstoffhaltigen funktionellen Gruppen, wie zuvor definiert, wobei diesbezüglich ein zentraler Vorteil darin zu sehen ist, dass das Porenvolumen der Aktivkohle nicht signifikant oder allenfalls nur geringfügig verändert wird. Erfindungsgemäß liegt gewissermaßen eine chemische Anbindung von Stickstoff an den sauerstoffhaltigen funktionellen Gruppen vor, welche gegebenenfalls weiterführend modifiziert werden kann, wie nachfolgend noch angeführt.

[0073] Was das erfindungsgemäß für die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff eingesetzte Stickstoffmodifizierungsreagenz ("SMR") anbelangt, so kann dieser erfindungsgemäß ausgewählt werden aus der Gruppe von Harnstoff, Ammoniak, Melamin und Aminosäuren sowie deren Kombinationen, insbesondere Harnstoff und Ammoniak sowie deren Kombinationen, vorzugsweise Harnstoff.

[0074] Gemäß einer erfindungsgemäß bevorzugten Ausführungsform kann als das für die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff eingesetzte Stickstoffmodifizierungsreagenz ("SMR") Harnstoff verwendet werden. In diesem Zusammenhang kann der Harnstoff in Form einer vorzugsweise wässrigen Lösung, bevorzugt in Form einer zumindest im Wesentlichen gesättigten Harnstofflösung, eingesetzt werden. Im Allgemeinen kann in Abhängigkeit von dem gewünschten Grad der Stickstofffunktionalisierung die Menge und Konzentration der Harnstofflösung entsprechend variiert werden, was im fachüblichen Können des Fachmanns liegt. In diesem Zusammenhang kann erfindungsgemäß beispielsweise derart vorgegangen werden, dass die an ihrer Oberfläche oxidierte Aktivkohle zunächst in die Harnstofflösung eingetaucht wird, die überflüssige Harnstofflösung dann abdekantiert wird und nachfolgend eine thermische Behandlung insbesondere in einem Inertgasstrom erfolgt.

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[0075] Gemäß einer weiteren erfindungsgemäßen Ausführungsform kann es auch vorgesehen sein, dass als Stickstoffmodifizierungsreagenz ("SMR") Ammoniak (NH_3) verwendet wird. In diesem Zusammenhang kann Ammoniak in Form des Gases und/oder als Ammoniakwasser, insbesondere in Form des Gases, eingesetzt werden. In diesem Zusammenhang kann die zu behandelnde und an ihrer Oberfläche oxidierte Aktivkohle einem Ammoniak- bzw. NH_3-Gasstrom bzw. einem NH_3-haltigen Gasstrom unter entsprechender Erwärmung ausgesetzt werden.

[0076] Was die Ausrüstung, insbesondere Modifizierung bzw. Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff weiterhin anbelangt, so kann diese im Rahmen des erfindungsgemäß durchgeführten Verfahrens durch Reaktion, insbesondere chemische Reaktion, vorzugsweise unter Ausbildung kovalenter Bindungen, des Stickstoffmodifizierungsreagenzes ("SMR") mit der an ihrer Oberfläche oxidierten Aktivkohle, insbesondere mit den sauerstoffhaltigen funktionellen Gruppen an der Oberfläche der Aktivkohle, erfolgen. In diesem Zusammenhang kann es erfindungsgemäß vorgesehen sein, dass die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff unter Ausbildung stickstoffhaltiger funktioneller Gruppen, insbesondere ausgewählt aus der Gruppe von Aminen, insbesondere primären, sekundären, tertiären und quartären Aminen, Iminen und Azinen sowie deren Kombinationen erfolgt, insbesondere können Amin-, Imin-, Azin-, Pyrrol-, Nitril-, Nitro-, Nitroso-, Pyridin-, Imid-, Amid-, Lactam- und/oder Pyridongruppen sowie deren Kombinationen, ausgebildet werden. Erfindungsgemäß kann somit zumindest zunächst eine chemische Anbindung von Stickstoff unter Ausbildung stickstoffhaltiger funktionellen Gruppen vorliegen, wobei die chemisch Anbindung insbesondere an der Oberfläche der zuvor oxidierten Aktivkohle lokalisiert ist und insbesondere aufgrund einer chemischen Reaktion unter Ausbildung kovalenter Bindungen mit den zuvor gebildeten sauerstoffhaltigen funktionellen Gruppen resultiert.

[0077] Erfindungsgemäß kann es aber auch vorgesehen sein, dass die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff unter Integration und/oder Einbau, insbesondere unter Ausbildung chemischer Bindungen, vorzugsweise unter Ausbildung kovalenter chemischer Bindungen, insbesondere zwischen Stickstoff einerseits und Kohlenstoff andererseits, in das Kohlenstoffgerüst und/oder in die Kohlenstoffmatrix der an ihrer Oberfläche oxidierten Aktivkohle erfolgt. Auf diese Weise wird der Stickstoff sozusagen direkt in das Kohlenstoffgerüst der Aktivkohle integriert bzw. eingebaut. Dies entspricht einer Dotierung mit Stickstoff (N-Dotierung). Die Integration von Stickstoff in das Kohlenstoffgerüst kann dabei ausgehend von der zuvor beschriebenen Anbindung des Stickstoffs bzw. des Stickstoffmodifizierungsreagenzes ("SMR") an die sauerstoffhaltigen funktionellen Gruppen erfolgen, insbesondere im Rahmen einer nachfolgend noch spezifizierten thermischen Behandlung, welche ausgehend von den resultierenden stickstoffhaltigen funktionellen Gruppen zu einer Integration des Stickstoffs in das Kohlenstoffgerüst der Aktivkohle insbesondere auf Basis einer Pyrolyse führt.

[0078] Was die erfindungsgemäß realisierte Verfahrensführung weiterhin anbelangt, so kann es im Rahmen der vorliegenden Erfindung vorgesehen sein, dass die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff unter Erwärmen erfolgt, insbesondere auf Temperaturen oberhalb der Zersetzungstemperatur des Stickstoffmodifizierungsreagenzes ("SMR"). In diesem Zusammenhang kann die Ausrüstung, insbesondere Modifizierung bzw. Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff unter solchen Temperaturen erfolgen, dass eine Reaktion des Stickstoffmodifizierungsreagenzes ("SMR") mit der an ihrer Oberfläche oxidierten Aktivkohle, insbesondere mit den sauerstoffhaltigen funktionellen Gruppen an der Oberfläche der Aktivkohle, erfolgt. Insbesondere kann diesbezüglich derart vorgegangen werden, dass die Ausrüstung, insbesondere Modifizierung bzw. Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff im Temperaturbereich von 200°C bis 1.200°C, insbesondere 300°C bis 1.000°C, vorzugsweise 500°C bis 950°C, durchgeführt wird. Was die Zeitdauer der Wärmebehandlung anbelangt, so kann es erfindungsgemäß zudem vorgesehen sein, dass die Ausrüstung, insbesondere Modifizierung bzw. Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff für eine Zeitdauer im Bereich von 0,1 Stunden bis 15 Stunden, insbesondere 0,2 Stunden bis 10 Stunden, vorzugsweise 0,5 Stunden bis 8 Stunden, durchgeführt wird. Insbesondere kann in Bezug auf die Verwendung von Harnstoff als Stickstoffmodifizierungsreagenz ("SMR") bei Temperaturen oberhalb von 180°C verfahren werden, wobei die vorgenannte Temperatur der Zersetzungstemperatur von Harnstoff entspricht.

[0079] Was die zur Anbindung des Stickstoffmodifizierungsreagenzes ("SMR") an die sauerstoffhaltigen Gruppen zur Ausbildung der stickstoffhaltigen funktionellen Gruppen bzw. zur Integration des Stickstoffs in das Kohlenstoffgerüst bzw. -gitter durchgeführte Wärmebehandlung der an ihrer Oberfläche oxidierten Aktivkohle gemäß einer weiteren bevorzugten Ausführungsform weiterhin anbelangt, so kann es erfindungsgemäß vorgesehen sein, dass die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff unter Erwärmen in Form einer einstufigen Wärmebehandlung erfolgt.

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[0080] Diesbezüglich kann es insbesondere vorgesehen sein, dass die Ausrüstung, insbesondere Modifizierung bzw. Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff unter Erwärmen auf einen Temperaturbereich von 300°C bis 700°C, insbesondere 400°C bis 600°C, vorzugsweise 400°C bis 550°C, durchgeführt wird, insbesondere für eine Zeitdauer im Bereich von 0,1 Stunden bis 15 Stunden, bevorzugt 0, 2 Stunden bis 10 Stunden, vorzugsweise 0,5 Stunden bis 8 Stunden. Auf diese Weise werden insbesondere stickstoffhaltige funktionelle Gruppen, wie sie zuvor definiert worden sind, ausgebildet.

[0081] Erfindungsgemäß kann es auch vorgesehen sein, dass die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff unter Erwärmen auf einen Temperaturbereich von 750°C bis 1.200°C, insbesondere 800°C bis 1.100°C, vorzugsweise 850°C bis 1.050°C, durchgeführt wird. Diesbezüglich kann insbesondere für eine Zeitdauer im Bereich von 0,1 Stunden bis 15 Stunden, insbesondere 0,2 Stunden bis 10 Stunden, vorzugsweise 0,5 Stunden bis 8 Stunden, verfahren werden. Im Rahmen der zuvor beschriebenen Wärmebehandlung erfolgt insbesondere eine Integration bzw. ein Einbau des Stickstoffs in das Kohlenstoffgerüst bzw. die Kohlenstoffmatrix der an ihrer Oberfläche oxidierten Aktivkohle, insbesondere wie zuvor definiert. Auf Basis dieser Verfahrensführung mit einer Wärmebehandlung unter höheren Temperaturen erfolgt somit vorrangig eine Immobilisierung des Stickstoffs aufgrund einer chemischen Anbindung bzw. Integration von Stickstoff an bzw. in das Kohlenstoffgerüst bzw. Kohlenstoffgitter der Aktivkohle, wobei auch diesbezüglich – ohne sich auf diese Theorie beschränken bzw. festlegen zu wollen – zunächst stickstoffhaltige funktionelle Gruppen gebildet werden können, welche nachfolgend zur Überführung des Stickstoffs in die Kohlenstoffstruktur gewissermaßen thermisch bzw. auf Basis von Pyrolyse zersetzt werden, so dass im Endprodukt reaktiver bzw. katalytischer Stickstoff direkt in das Kohlenstoffgerüst der so behandelten Aktivkohle eingebaut bzw. inkorporiert vorliegt.

[0082] Gemäß einer weiteren erfindungsgemäß bevorzugten Ausführungsform kann die Ausrüstung, insbesondere Modifizierung bzw. Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff auch unter Erwärmen in Form einer mehrstufigen, insbesondere zweistufigen Wärmebehandlung erfolgen.

[0083] In diesem Zusammenhang kann es erfindungsgemäß insbesondere vorgesehen sein, dass die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff in einer ersten Wärmebehandlung unter Erwärmen auf einen Temperaturbereich von 300°C bis 700°C, insbesondere 400°C bis 600°C, vorzugsweise 400°C bis 550°C, und einer nachfolgenden zweiten Wärmebehandlung unter Erwärmen auf einen Temperaturbereich von 750°C bis 1.200°C, insbesondere 800°C bis 1.100°C, vorzugsweise 850°C bis 1.050°C, durchgeführt wird. Insbesondere können dabei die jeweiligen Wärmebehandlungen, unabhängig voneinander, für eine Zeitdauer im Bereich von 0,1 Stunden bis 15 Stunden, insbesondere 0,2 Stunden bis 10 Stunden, vorzugsweise 0,5 Stunden bis 8 Stunden, durchgeführt werden. Insbesondere werden auf diese Weise im ersten Schritt die stickstoffhaltigen funktionellen Gruppen gebildet, welche dann im zweiten Schritt unter Pyrolyse zersetzt werden, wobei der Stickstoff in das Kohlenstoffgerüst überführt bzw. eingebaut wird.

[0084] Die Temperatur der Wärmebehandlung hat somit einen signifikanten Einfluss auf die chemische Konstitution des in die Aktivkohle eingebrachten Stickstoffs. Bei niedrigen Temperaturen der Wärmebehandlung, wie zuvor definiert, resultiert vorrangig die Bildung von stickstoffhaltigen funktionellen Gruppen, und bei höheren Temperaturen, wie zuvor definiert, erfolgt insbesondere bzw. vorrangig ein Einbau bzw. eine Integration des Stickstoffs in die Gerüststruktur der Aktivkohle.

[0085] In diesem Zusammenhang kann es auch vorgesehen sein, dass im Anschluss an die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, mit Stickstoff eine Aufreinigung und/oder Trocknung der erhaltenen Aktivkohle erfolgt. In diesem Zusammenhang kann die Aufreinigung mittels mindestens eines Waschvorganges in einer Flüssigkeit, insbesondere Wasser, erfolgen. Zudem kann eine Trocknung unter Erwärmung der mit Stickstoff ausgerüsteten Aktivkohle, insbesondere auf Temperaturen im Bereich von 40°C bis 200°C, insbesondere 50°C bis 150°C, vorzugsweise 60°C bis 120°C, erfolgen können. Durch den Aufreinigungs- bzw. Waschvorgang werden insbesondere auch Verbrennungsrückstände von der mit Stickstoff modifizierten Aktivkohle entfernt.

[0086] Aufgrund der erfindungsgemäß realisierten Verfahrensführung kann der Stickstoffgehalt der behandelten Aktivkohle signifikant gesteigert werden, wobei für das erhaltene und mit Stickstoff modifizierte Produkt der Gehalt an flüchtigen Bestandteile ("fB") der Aktivkohle als Maß auch für den Gehalt an Stickstoffgruppen nach erfolgter Stickstoffmodifizierung bzw. -funktionalisierung herangezogen werden kann, insbesondere im Hinblick auf die zuvor definierten stickstoffhaltigen funktionellen Gruppen: So kann die mit Stickstoff ausgerüstete Aktivkohle einen Gehalt an flüchtigen Bestandteilen ("tB"), bezogen auf das Trockengewicht der mit

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Stickstoff ausgerüsteten Aktivkohle, von mindestens 0,1 Gew.-%, insbesondere mindestens 0,2 Gew.-%, vorzugsweise mindestens 0,3 Gew.-%, bevorzugt mindestens 0,5 Gew.-%, besonders bevorzugt mindestens 1 Gew.-%, ganz besonders bevorzugt mindestens 2 Gew.-%, und/oder im Bereich von 0,1 Gew.-% bis 20 Gew.-%, insbesondere 0,2 Gew.-% bis 15 Gew.-%, vorzugsweise 0,3 Gew.-% bis 12 Gew.-%, bevorzugt 0,5 Gew.-% bis 10 Gew.-%, besonders bevorzugt 1 Gew.-% bis 10 Gew.-%, aufweisen. Auch diesbezüglich können die flüchtigen Bestandteile ("fB") auf Basis der Norm ISO 562:1981 bzw. unter Erwärmen für 7 Minuten auf eine Temperatur von 900°C unter Inertatmosphäre bestimmt werden, wobei insbesondere von der Trockensubstanz ausgegangen wird.

[0087] Darüber hinaus kann die mit Stickstoff ausgerüstete Aktivkohle einen Stickstoffgehalt (d. h. Gesamtstickstoffgehalt), bezogen auf das Trockengewicht der mit Stickstoff ausgerüsteten Aktivkohle, von mindestens 0,1 Gew.-%, insbesondere mindestens 0,2 Gew.-%, vorzugsweise mindestens 0,3 Gew.-%, bevorzugt mindestens 0,5 Gew.-%, besonders bevorzugt mindestens 1 Gew.-%, ganz besonders bevorzugt mindestens 2 Gew.-%, und/oder im Bereich von 0,1 Gew.-% bis 30 Gew.-%, insbesondere 0,2 Gew.-% bis 25 Gew.-%, vorzugsweise 0,3 Gew.-% bis 20 Gew.-%, bevorzugt 0,5 Gew.-% bis 15 Gew.-%, besonders bevorzugt 1 Gew.-% bis 15 Gew.-%, aufweisen. In diesem Zusammenhang sollten mindestens 50%, insbesondere mindestens 60%, vorzugsweise mindestens 70%, bevorzugt mindestens 80% des Stickstoffs, bezogen auf den Gesamtstickstoffgehalt, in der mit Stickstoff ausgerüsteten Aktivkohle chemisch gebunden und/oder integriert vorliegen und/oder nicht in Form von flüchtigen Bestandteilen ("fB") in der mit Stickstoff ausgerüsteten Aktivkohle vorliegen. Gleichermaßen sollten in diesem Zusammenhang 50% bis 99,9%, insbesondere 60% bis 99%, vorzugsweise 70% bis 95%, bevorzugt 80% bis 90%, des Stickstoffs, bezogen auf den Gesamtstickstoffgehalt, in der mit Stickstoff ausgerüsteten Aktivkohle chemisch gebunden und/oder integriert vorliegen und/oder nicht in Form von flüchtigen Bestandteilen ("fB") in der mit Stickstoff ausgerüsteten Aktivkohle vorliegen. Mit anderen Worten sollte der Stickstoff auf Basis der obigen Ausführungen bzw. der obigen Mengen in das Kohlenstoffgerüst bzw. -gitter der Aktivkohle integriert vorliegen, wobei dann der restliche Anteil an Stickstoff in Form von stickstoffhaltigen funktionellen Gruppen vorliegt, insbesondere wie zuvor definiert. Hierdurch wird eine besonders gute katalytische Aktivität bzw. Reaktivität bei gleichzeitig hervorragender Beständigkeit der Ausrüstung mit Stickstoff erreicht.

[0088] In diesem Zusammenhang werden im Allgemeinen die höheren Werte für den in das Kohlenstoffgerüst bzw. -gitter der Aktivkohle integrierten Stickstoff erreicht, wenn die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff unter Erwärmen auf einen Temperaturbereich von 750°C bis 1.200°C, insbesondere wie zuvor definiert, durchgeführt wird. Der Stickstoff liegt dann allenfalls nur zu einem geringen Anteil in Form von stickstoffhaltigen funktionellen Gruppen vor. Dagegen werden im Allgemeinen geringere Werte für den in das Kohlenstoffgerüst bzw. -gitter der Aktivkohle integrierten Stickstoff erreicht, wenn die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff unter Erwärmen auf einen Temperaturbereich von 300°C bis 700°C, insbesondere wie zuvor definiert, durchgeführt wird. Wie zuvor erläutert, liegt der Stickstoff dann zu einem größeren Anteil in Form von stickstoffhaltigen funktionellen Gruppen vor. Hierdurch kann insbesondere eine verbesserte Abbauleistung gegenüber bestimmten Substanzen, wie insbesondere Formaldehyd, erreicht werden.

[0089] Weiterhin kann die mit Stickstoff ausgerüstete erfindungsgemäße Aktivkohle einen Gehalt an in das Kohlenstoffgerüst eingebautem bzw. integriertem (d. h. sozusagen immobilisiertem) Stickstoff, bezogen auf das Trockengewicht der mit Stickstoff ausgerüsteten Aktivkohle, von mindestens 0,05 Gew.-%, insbesondere mindestens 0,1 Gew.-%, vorzugsweise mindestens 0,2 Gew.-%, bevorzugt mindestens 0,3 Gew.-%, besonders bevorzugt mindestens 0,5 Gew.-%, ganz besonders bevorzugt mindestens 1 Gew.-%, bzw. im Bereich von 0,05 Gew.-% bis 30 Gew.-%, insbesondere 0,1 Gew.-% bis 25 Gew.-%, vorzugsweise 0,2 Gew.-% bis 20 Gew.-%, bevorzugt 0,5 Gew.-% bis 15 Gew.-%, aufweisen.

[0090] In diesem Zusammenhang kann der jeweilige Stickstoffgehalt der erfindungsgemäßen Aktivkohlen mit an sich bekannten Methoden bestimmt werden. Der Gesamtstickstoffgehalt kann mittels an sich bekannter Elementaranalyse bestimmt werden, während die Ermittlung der flüchtigen Bestandteile ("fB") gemäß der ISO 562:1981 erfolgen kann. Aus der Differenz von mittels Elementaranalyse bestimmtem Gesamtstickstoffgehalt einerseits und gemäß der ISO 562:1981 bestimmtem Gehalt an flüchtigen Bestandteilen (d. h. flüchtigen stickstoffhaltigen funktionellen Gruppen) kann dann der Gehalt an immobilisiertem bzw. in das Kohlenstoffgerüst eingebautem Stickstoff ermittelt werden.

[0091] Nachfolgend werden weitere charakteristische Eigenschaften bzw. Parameter der mit dem erfindungsgemäß durchgeführten Verfahren erhältlichen Aktivkohle definiert. Die nachfolgenden Parameterangaben be-

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züglich der im Rahmen des erfindungsgemäß durchgeführten Verfahrens erhältlichen stickstoffmodifizierten bzw. stickstofffunktionalisierten Aktivkohle werden dabei mit genormten oder explizit angegebenen Bestimmungsverfahren oder mit dem Fachmann an sich geläufigen Bestimmungsmethoden bestimmt. Insbesondere die Parameterangaben betreffend die Charakterisierung der Porosität, der Porengrößenverteilung und anderer Adsorptionseigenschaften ergeben sich im Allgemeinen jeweils aus dem betreffenden Stickstoffsorptionsisothermen der betreffenden Aktivkohle bzw. der vermessenen Produkte.

[0092] Insbesondere wird im Rahmen der vorliegenden Erfindung eine mit Stickstoff ausgerüstete Aktivkohle bzw. eine stickstoffmodifizierte oder stickstofffunktionalisierte Aktivkohle erhalten, welche ein Gesamtporenvolumen, insbesondere ein Gesamtporenvolumen nach Gurvich, im Bereich von 0,4 bis 4 cm^3/g, insbesondere 0,5 bis 3,5 cm^3/g, vorzugsweise 0,6 bis 3 cm^3/g, besonders bevorzugt 0,7 bis 2,5 cm^3/g, aufweist. In diesem Zusammenhang sollten 30 bis 99%, insbesondere 40% bis 99%, vorzugsweise 50% bis 95%, des Gesamtporenvolumens, insbesondere des Gesamtporenvolumens nach Gurvich, der mit Stickstoff ausgerüsteten Aktivkohle, durch Poren mit Porendurchmessern von ≤ 50 nm, insbesondere durch Mikro- und/oder Mesoporen, gebildet werden. Gleichermaßen sollten in diesem Zusammenhang 10% bis 85%, insbesondere 20% bis 80%, vorzugsweise 30% bis 75%, des Gesamtporenvolumens, insbesondere des Gesamtporenvolumens nach Gurvich, der mit Stickstoff ausgerüsteten Aktivkohle, durch Poren mit Porendurchmessern im Bereich von 2 nm bis 50 nm, insbesondere durch Mesoporen, gebildet werden.

[0093] Was die Bestimmung des Gesamtporenvolumens nach Gurvich anbelangt, so handelt es sich um eine dem Fachmann auf diesem Gebiet an sich bekannte Mess-Bestimmungsmethode. Zu weitergehenden Einzelheiten bezüglich der Bestimmung des Gesamtporenvolumens nach Gurvich kann beispielsweise verwiesen werden auf L. Gurvich (1915), J. Phys. Chem. Soc. Russ. 47, 805, sowie auf S. Lowell et al., Characterization of Porous Solids and Powders: Surface Area Pore Size and Density, Kluwer Academic Publishers, Article Technology Series, Seiten 111 ff.

[0094] Insbesondere kann es im Rahmen der vorliegenden Erfindung vorgesehen sein, dass die mit Stickstoff ausgerüstete Aktivkohle ein durch Poren mit Porendurchmessern ≤ 2 nm gebildetes Porenvolumen, insbesondere Mikroporenvolumen nach Carbon Black, insbesondere nach Auswertung mittels t-plot, im Bereich von 0,05 bis 2 cm^3/g, insbesondere 0,1 bis 1,5 cm^3/g, vorzugsweise 0,2 bis 1,1 cm^3/g, besonders bevorzugt 0,3 bis 1 cm^3/g, aufweist. Gleichermaßen kann es vorgesehen sein, dass 10% bis 95%, insbesondere 20% bis 95%, vorzugsweise 30% bis 90%, des Gesamtporenvolumens der mit Stickstoff ausgerüsteten Aktivkohle durch Poren mit Porendurchmessern von ≤ 2 nm, insbesondere durch Mikroporen, gebildet werden.

[0095] Die Bestimmungsmethode nach Carbon Black ist dem Fachmann an sich bekannt, so dass es diesbezüglich keiner weitergehenden Einzelheiten bedarf. Zudem kann zu weitergehenden Einzelheiten der Bestimmung der Porenoberfläche und des Porenvolumens nach Carbon Black beispielsweise verwiesen werden auf R. W. Magee, Evaluation of the External Surface Area of Carbon Black by Nitrogen Adsorption, Presented at the Meeting of the Rubber Division of the American Chem. Soc., October 1994, z. B. referiert in: Quantachrome Instruments, AUTOSORB-1, AS1 WinVersion 1.50, Operating Manual, OM, 05061, Quantachrome Instruments 2004, Florida, USA, Seiten 71 ff.

[0096] Im Rahmen der vorliegenden Erfindung ist es gleichermaßen vorteilhaft, wenn die stickstoffmodifizierte bzw. stickstofffunktionalisierte mit Stickstoff ausgerüstete Aktivkohle einen mittleren Porendurchmesser im Bereich von 1 bis 60 nm, insbesondere 1 bis 55 nm, bevorzugt 1,5 bis 50 nm, besonders bevorzugt 2 bis 45 nm, aufweist.

[0097] Um die Sorptionseigenschaften der mit dem erfindungsgemäß durchgeführten Verfahren erhältlichen mit Stickstoff ausgerüsteten Aktivkohle zu erhalten bzw. zu gewährleisten, sollte die mit Stickstoff ausgerüstete Aktivkohle eine spezifische BET-Oberfläche im Bereich von 500 m^2/g bis 3.500 m^2/g, insbesondere 600 bis 3.000 m^2/g, vorzugsweise 700 bis 2.750 m^2/g, besonders bevorzugt 800 bis 2.500 m^2/g, aufweisen.

[0098] Die Bestimmung der spezifischen Oberfläche gemäß BET ist dem Fachmann grundsätzlich als solche bekannt, so dass diesbezüglich keine weitergehenden Einzelheiten ausgeführt zu werden brauchen. Sämtliche BET-Oberflächenangaben beziehen sich auf die Bestimmung gemäß ASTM D6556-04. Im Rahmen der vorliegenden Erfindung wird zur Bestimmung der BET-Oberfläche – im Allgemeinen und sofern nachfolgend nicht ausdrücklich abweichend angegeben – die so genannte MultiPoint-BET-Bestimmungsmethode (MP-BET) in einem Partialdruckbereich p/p_0 von 0,05 bis 0,1 angewendet.

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[0099] In Bezug auf weitergehende Einzelheiten zu der Bestimmung der BET-Oberfläche bzw. zu der BET-Methode kann auf die vorgenannte ASTM D6556-04 sowie auf Römpp Chemielexikon, 10. Auflage, Georg Thieme Verlag, Stuttgart/New York, Stichwort: "BET-Methode", einschließlich der dort referierten Literatur, und auf Winnacker-Küchler (3. Auflage), Band 7, Seiten 93 ff. sowie auf Z. Anal. Chem. 238, Seiten 187 bis 193 (1968), verwiesen werden.

[0100] Des Weiteren ist es von Vorteil, wenn die stickstoffmodifizierte bzw. stickstofffunktionalisierte bzw. die mit Stickstoff ausgerüstete Aktivkohle eine durch Mikroporen gebildete Oberfläche von 400 bis 2.500 m^2/g, insbesondere 500 bis 2.300 m^2/g, vorzugsweise 600 bis 2.100 m^2/g, besonders bevorzugt 700 bis 1.900 m^2/g, aufweist.

[0101] Die stickstoffmodifizierte bzw. stickstofffunktionalisierte bzw. die mit Stickstoff ausgerüstete Aktivkohle sollte zudem eine Dichte von 1,1 bis 3,5 g/ml, insbesondere von 1,5 bis 3 g/ml, vorzugsweise von 1,75 bis 2,75 g/ml, bevorzugt von 2 bis 2,5 g/ml, aufweisen.

[0102] Gleichermaßen sollte die stickstoffmodifizierte bzw. stickstofffunktionalisierte bzw. die mit Stickstoff ausgerüstete Aktivkohle eine Schüttdichte von 0,1 bis 1,5 g/ml, insbesondere von 0,15 bis 1 g/ml, vorzugsweise von 0,2 bis 0,8 g/ml, bevorzugt von 0,3 bis 0,6 g/ml, aufweisen.

[0103] Zudem ist es von Vorteil, wenn die stickstoffmodifizierte bzw. stickstofffunktionalisierte bzw. mit Stickstoff ausgerüstete Aktivkohle eine Druck- und/oder Berstfestigkeit (Gewichtsbelastbarkeit) pro Aktivkohlekorn, insbesondere pro Aktivkohlekügelchen, von mindestens 5 Newton, insbesondere mindestens 10 Newton, vorzugsweise mindestens 15 Newton, bevorzugt mindestens 20 Newton, aufweist und/oder dass die Aktivkohle eine Druck- und/oder Berstfestigkeit (Gewichtsbelastbarkeit) pro Aktivkohlekorn, insbesondere pro Aktivkohlekügelchen, im Bereich von 5 bis 50 Newton, insbesondere 10 bis 45 Newton, vorzugsweise 15 bis 40 Newton, aufweist.

[0104] Was darüber hinaus die im Rahmen des erfindungsgemäß durchgeführten Verfahrens eingesetzte Aktivkohle bzw. Ausgangsaktivkohle anbelangt, welche zunächst an ihrer Oberfläche oxidiert und nachfolgend mit Stickstoff modifiziert bzw. ausgerüstet wird, anbelangt, so kann diesbezüglich eine Vielzahl unterschiedlicher Aktivkohlen, beispielsweise mit unterschiedlicher Porenstruktur im Hinblick auf die Verteilung von Mikro-, Meso- und Makroporen, eingesetzt werden. Insbesondere können kommerziell erhältliche bzw. handelsübliche Aktivkohlen eingesetzt werden. Im Rahmen der vorliegenden Erfindung zum Einsatz kommende Aktivkohlen werden beispielsweise von der Blücher GmbH, Erkrath, Deutschland oder der AdsorTech GmbH, Premnitz, Deutschland, vertrieben.

[0105] Was die erfindungsgemäß eingesetzte Aktivkohle bzw. Ausgangsaktivkohle weiterhin anbelangt, so werden diesbezüglich insbesondere eine kornförmige, insbesondere kugelförmige Aktivkohle verwendet. Die eingesetzte Aktivkohle bzw. Ausgangsaktivkohle, insbesondere Kornkohle oder Kugelkohle, kann dabei nach bekannten Verfahren des Standes erhalten werden: Üblicherweise werden zu diesem Zweck korn- bzw. kugelförmig sulfonierte organische Polymere, insbesondere auf Basis von divinylbenzolvernetztem Polystyrol, carbonisiert und anschließend zu der betreffenden Aktivkohle aktiviert. Für weitergehende diesbezügliche Einzelheiten kann beispielsweise auf die Druckschriften DE 43 28 219 A1, DE 43 04 026 A1, DE 196 00 237 A1 sowie EP 1 918 022 A1 bzw. auf die parallele, zu derselben Patentfamilie gehörende US 7,737,038 B2 verwiesen werden. Gleichermaßen kann auch Aktivkohle auf Basis anderer Ausgangsmaterialien, wie Pech oder dergleichen, zum Einsatz kommen. Derartige Aktivkohlen sind dem Fachmann als solche bekannt.

[0106] Insbesondere wird im Rahmen der vorliegenden Erfindung eine Aktivkohle als Ausgangsmaterial eingesetzt, welche Partikelgrößen, insbesondere Teilchendurchmesser, im Bereich von 0,001 bis 2 mm, insbesondere 0,01 bis 1 mm, bevorzugt 0,05 bis 0,8 mm, vorzugsweise 0,1 bis 0,7 mm, besonders bevorzugt 0,15 bis 0,6 mm, aufweist. Dabei sollten, bezogen auf die Aktivkohle, mindestens 80 Gew.-%, insbesondere mindestens 90 Gew.-%, vorzugsweise mindestens 95 Gew.-% der eingesetzten Aktivkohle Partikelgrößen, insbesondere Teilchendurchmesser, in den vorgenannten Bereichen aufweisen. Weiterhin ist es erfindungsgemäß vorteilhaft, wenn die als Ausgangsmaterial eingesetzte Aktivkohle mittlere Partikelgrößen (D50), insbesondere mittlere Teilchendurchmesser (D50), im Bereich von 0,01 bis 1 mm, insbesondere 0,05 bis 0,8 mm, bevorzugt 0,1 bis 0,6 mm, vorzugsweise 0,15 bis 0,5 mm, besonders bevorzugt 0,2 bis 0,4 mm, aufweist. Die entsprechenden Teilchengrößen bzw. -durchmesser können beispielsweise auf Basis der Methode nach ASTM D2862-97/04 bestimmt werden. Zudem können die vorgenannten Größen mit Bestimmungsmethoden auf Basis einer Siebanalyse, auf Basis von Röntgenbeugung, Laserdiffraktometrie oder dergleichen bestimmt werden. Die jeweiligen Bestimmungsmethoden sind dem Fachmann als solche wohlbekannt, so dass es diesbe-

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züglich keiner weiteren Ausführungen bedarf. Die angegebenen Parameter der Ausgangaktivkohle finden sich in entsprechender Weise auch in der stickstoffmodifizierten bzw. stickstofffunktionalisierten Aktivkohle wieder.

[0107] Das erfindungsgemäß durchgeführte Verfahren ist mit einer Vielzahl von Vorteilen verbunden, wie sie bereits zuvor angeführt sind. Aufgrund der herausragenden Sorptionseigenschaften der mit dem erfindungsgemäß durchgeführten Verfahren erhaltenen bzw. erhältlichen Aktivkohle ist ein breites Einsatz- bzw. Verwendungsspektrum möglich, wie es nachfolgend noch im Detail angeführt ist.

[0108] Weiterer Gegenstand der vorliegenden Erfindung – gemäß einem zweiten Aspekt der vorliegenden Erfindung – ist die erfindungsgemäße stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohle als solche, insbesondere mit katalytischen bzw. reaktiven Eigenschaften, vorzugsweise mit Stickstoff als katalytisch aktivem Heteroatom.

[0109] Die stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohle nach der Erfindung ist durch Oberflächenoxidation einer als Ausgangsmaterial eingesetzten Aktivkohle ("Ausgangsaktivkohle") unter Verwendung bzw. in Gegenwart mindestens eines Oxidationsreagenzes ("OR") und nachfolgender Ausrüstung, insbesondere Modifizierung bzw. Funktionalisierung, mit Stickstoff der auf diese Weise erhaltenen, an ihrer Oberfläche oxidierten Aktivkohle unter Verwendung bzw. in Gegenwart mindestens eines Stickstoffmodifizierungsreagenzes ("SMR") erhältlich.

[0110] Somit betrifft die vorliegende Erfindung gemäß diesem Erfindungsaspekt auch die stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohle als solche nach der Erfindung, insbesondere mit katalytischen bzw. reaktiven Eigenschaften, vorzugsweise mit Stickstoff als katalytisch aktivem Heteroatom. Die erfindungsgemäße Aktivkohle zeichnet sich dadurch aus, dass Stickstoff unter Ausbildung chemischer Bindungen, vorzugsweise unter Ausbildung kovalenter chemischer Bindungen, an die Aktivkohle gebunden ist.

[0111] Was die erfindungsgemäße Aktivkohle bzw. die mit Stickstoff ausgerüstete Aktivkohle nach der Erfindung als solche weiterhin anbelangt, so kann es erfindungsgemäß vorgesehen sein, dass Stickstoff in Form mindestens einer stickstoffhaltigen funktionellen Gruppe, insbesondere ausgewählt aus der Gruppe von Aminen, insbesondere primären, sekundären, tertiären und quartären Aminen, Iminen und Azinen sowie deren Kombinationen erfolgt. Insbesondere kann der Stickstoff in Form von Amin-, Imin-, Azin-, Pyrrol-, Nitril-, Nitro-, Nitroso-, Pyridin-, Imid-, Amid-, Lactam- und Pyridongruppen sowie deren Kombinationen, vorliegen. Dabei ist der Stickstoff vorzugsweise an der stickstoffmodifizierten und/oder stickstofffunktionalisierten Aktivkohle gebunden, vorzugsweise chemisch und/oder kovalent gebunden.

[0112] Gleichermaßen kann es erfindungsgemäß vorgesehen sein, dass in Bezug auf die stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach der Erfindung Stickstoff in das Kohlenstoffgerüst bzw. in die Kohlenstoffmatrix der stickstoffmodifizierten bzw. stickstofffunktionalisierten Aktivkohle vorzugsweise unter Ausbildung kovalenter chemischer Bindungen, insbesondere zwischen Stickstoff einerseits und Kohlenstoff andererseits, integriert bzw. eingebaut ist.

[0113] Im Rahmen der vorliegenden Erfindung kann es vorgesehen sein, dass die stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle einen Gehalt an flüchtigen Bestandteilen ("fB"), bezogen auf das Trockengewicht der mit Stickstoff ausgerüsteten Aktivkohle, von mindestens 0,1 Gew.-%, insbesondere mindestens 0,2 Gew.-%, vorzugsweise mindestens 0,3 Gew.-%, bevorzugt mindestens 0,5 Gew.-%, besonders bevorzugt mindestens 1 Gew.-%, ganz besonders bevorzugt mindestens 2 Gew.-%, und/oder im Bereich von 0,1 Gew.-% bis 20 Gew.-%, insbesondere 0,2 Gew.-% bis 15 Gew.-%, vorzugsweise 0,3 Gew.-% bis 12 Gew.-%, bevorzugt 0,5 Gew.-% bis 10 Gew.-%, besonders bevorzugt 1 Gew -% bis 10 Gew.-%, aufweist. Wie zuvor angeführt, können die flüchtigen Bestandteile ("fB") als Maß für den Gehalt an Stickstoff im Hinblick auf das Vorliegen in Form von stickstoffhaltigen funktionellen Gruppen, insbesondere wie zuvor definiert, herangezogen werden. Für die diesbezüglichen Bestimmungsverfahren kann auf obige Ausführungen verwiesen werden.

[0114] Weiterhin ist es im Rahmen der vorliegenden Erfindung von Vorteil, wenn die stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohle einen Stickstoffgehalt, bezogen auf das Trockengewicht der mit Stickstoff ausgerüsteten Aktivkohle, von mindestens 0,1 Gew.-%, insbesondere mindestens 0,2 Gew.-%, vorzugsweise mindestens 0,3 Gew.-%, bevorzugt mindestens 0,5 Gew.-%, besonders bevorzugt mindestens 1 Gew.-%, ganz besonders bevorzugt mindestens 2 Gew.-%, und/oder im Bereich von 0,1 Gew.-% bis 30 Gew.-%, insbesondere 0,2 Gew.-% bis 25 Gew.-%, vorzugsweise 0,3 Gew.-% bis 20 Gew.-%, bevorzugt 0,5 Gew.-% bis 15 Gew.-%, besonders bevorzugt 1 Gew.-% bis 15 Gew.-%, aufweist. Diesbezüglich sollten mindestens 50%, insbesondere mindestens 60%, vorzugsweise mindestens 70%, bevorzugt mindestens 80%, des Stick-

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stoffs, bezogen auf den Gesamtstickstoffgehalt, in der stickstoffmodifizierten bzw. stickstofffunktionalisierten Aktivkohle chemisch gebunden bzw. integriert vorliegen und/oder nicht in Form von flüchtigen Bestandteilen ("fB") in der stickstoffmodifizierten und/oder stickstofffunktionalisierten Aktivkohle vorliegen. In diesem Zusammenhang sollten 50% bis 99,9%, insbesondere 60% bis 99%, vorzugsweise 70% bis 95%, bevorzugt 80% bis 90%, des Stickstoffs, bezogen auf den Gesamtstickstoffgehalt, in der stickstoffmodifizierten bzw. stickstofffunktionalisierten Aktivkohle chemisch gebunden bzw. integriert vorliegen und/oder nicht in Form von flüchtigen Bestandteilen ("fB") in der stickstoffmodifizierten und/oder stickstofffunktionalisierten Aktivkohle vorliegen.

[0115] Weiterhin kann die mit Stickstoff ausgerüstete erfindungsgemäße Aktivkohle einen Gehalt an in das Kohlenstoffgerüst eingebautem bzw. integriertem (d. h. sozusagen immobilisiertem) Stickstoff, bezogen auf das Trockengewicht der mit Stickstoff ausgerüsteten Aktivkohle, von 0,05 Gew.-%, insbesondere mindestens 0,1 Gew.-%, vorzugsweise mindestens 0,2 Gew.-%, bevorzugt mindestens 0,3 Gew.-%, besonders bevorzugt mindestens 0,5 Gew.-%, ganz besonders bevorzugt mindestens 1 Gew.-%, bzw. im Bereich von 0,05 Gew.-% bis 30 Gew.-%, insbesondere 0,1 Gew.-% bis 25 Gew.-%, vorzugsweise 0,2 Gew.-% bis 20 Gew.-%, bevorzugt 0,5 Gew.-% bis 15 Gew.-%, aufweisen.

[0116] Insbesondere zeichnet sich die erfindungsgemäße Aktivkohle mit Stickstoffmodifizierung bzw. Stickstofffunktionalisierung auch durch die folgenden Parameter aus; für die diesbezüglichen Bestimmungsverfahren kann gleichermaßen auf obige Ausführungen verwiesen werden:
Erfindungsgemäß sollte die stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle ein Gesamtporenvolumen, insbesondere ein Gesamtporenvolumen nach Gurvich, im Bereich von 0,4 bis 4 cm^3/g, insbesondere 0,5 bis 3,5 cm^3/g, vorzugsweise 0,6 bis 3 cm^3/g, besonders bevorzugt 0,7 bis 2,5 cm^3/g, aufweisen.

[0117] Diesbezüglich sollten mindestens 30% bis 99%, insbesondere 40% bis 99%, vorzugsweise 50% bis 95%, des Gesamtporenvolumens, insbesondere des Gesamtporenvolumens nach Gurvich, der stickstoffmodifizierten und/oder stickstofffunktionalisierten Aktivkohle, durch Poren mit Porendurchmessern von ≤ 50 nm, insbesondere durch Mikro- und/oder Mesoporen, gebildet sein. Insbesondere können dabei 10% bis 85%, insbesondere 20% bis 80%, vorzugsweise 30% bis 75%, des Gesamtporenvolumens, insbesondere des Gesamtporenvolumens nach Gurvich, der stickstoffmodifizierten und/oder stickstofffunktionalisierten Aktivkohle, durch Poren mit Porendurchmessern im Bereich von 2 nm bis 50 nm, insbesondere durch Mesoporen, gebildet sein.

[0118] Weiterhin sollte die stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle erfindungsgemäß ein durch Poren mit Porendurchmessern ≤ 2 nm gebildetes Porenvolumen, insbesondere Mikroporenvolumen nach Carbon Black, insbesondere nach Auswertung mittels t-plot, im Bereich von 0,05 bis 2 cm^3/g, insbesondere 0,1 bis 1,5 cm^3/g, vorzugsweise 0,2 bis 1,1 cm^3/g, besonders bevorzugt 0,3 bis 1 cm^3/g, aufweisen. Dabei sollten 10% bis 95%, insbesondere 20% bis 95%, vorzugsweise 30% bis 90%, des Gesamtporenvolumens der stickstoffmodifizierten und/oder stickstofffunktionalisierten Aktivkohle durch Poren mit Porendurchmessern von ≤ 2 nm, insbesondere durch Mikroporen, gebildet sein.

[0119] Des Weiteren sollte die stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach der Erfindung einen mittleren Porendurchmesser im Bereich von 1 bis 60 nm, insbesondere 1 bis 55 nm, bevorzugt 1,5 bis 50 nm, besonders bevorzugt 2 bis 45 nm, aufweisen.

[0120] Erfindungsgemäß sollte die stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach der Erfindung zudem eine spezifische BET-Oberfläche im Bereich von 500 m^2/g bis 3.500 m^2/g, insbesondere 600 bis 3.000 m^2/g, vorzugsweise 700 bis 2.750 m^2/g, besonders bevorzugt 800 bis 2.500 m^2/g, aufweisen.

[0121] Des Weiteren sollte die mit Stickstoff ausgerüstete Aktivkohle eine durch Mikroporen gebildete Oberfläche von 400 bis 2.500 m^2/g, insbesondere 500 bis 2.300 m^2/g, vorzugsweise 600 bis 2.100 m^2/g, besonders bevorzugt 700 bis 1.900 m^2/g, aufweisen.

[0122] Darüber hinaus sollte die mit Stickstoff ausgerüstete Aktivkohle nach der Erfindung eine Dichte von 1,1 bis 3,5 g/ml, insbesondere von 1,5 bis 3 g/ml, vorzugsweise von 1,75 bis 2,75 g/ml, bevorzugt von 2 bis 2,5 g/ml, aufweisen.

[0123] Weiterhin sollte die mit Stickstoff ausgerüstete Aktivkohle nach der Erfindung eine Schüttdichte von 0,1 bis 1,5 g/ml, insbesondere von 0,15 bis 1 g/ml, vorzugsweise von 0,2 bis 0,8 g/ml, bevorzugt von 0,3 bis 0,6 g/ml, aufweisen.

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[0124] Erfindungsgemäß sollte die mit Stickstoff ausgerüstete Aktivkohle zudem eine Druck- und/oder Berstfestigkeit (Gewichtsbelastbarkeit) pro Aktivkohlekorn, insbesondere pro Aktivkohlekügelchen, von mindestens 5 Newton, insbesondere mindestens 10 Newton, vorzugsweise mindestens 15 Newton, bevorzugt mindestens 20 Newton, aufweisen. Zudem sollte die Aktivkohle eine Druckund/oder Berstfestigkeit (Gewichtsbelastbarkeit) pro Aktivkohlekorn, insbesondere pro Aktivkohlekügelchen, im Bereich von 5 bis 50 Newton, insbesondere 10 bis 45 Newton, vorzugsweise 15 bis 40 Newton, aufweisen.

[0125] Was die stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle weiterhin anbelangt, so kann diese selbsttragend sein bzw. in Form einer insbesondere losen Schüttung vorliegt.

[0126] Alternativ ist es aber auch möglich, dass die Aktivkohle auf einem Trägermaterial aufgebracht ist.

[0127] In diesem Zusammenhang kann das Trägermaterial gasdurchlässig, insbesondere luftdurchlässig, ausgebildet sein.

[0128] Was in diesem Zusammenhang das Trägermaterial als solches anbelangt, so sollte dieses eine Gasdurchlässigkeit, insbesondere Luftdurchlässigkeit, von mindestens 10 $l \cdot m^{-2} \cdot s^{-1}$, insbesondere mindestens 30 $l \cdot m^{-2} \cdot s^{-1}$, vorzugsweise mindestens 50 $l \cdot m^{-2} \cdot s^{-1}$, besonders bevorzugt mindestens 100 $l \cdot m^{-2} \cdot s^{-1}$, ganz besonders bevorzugt mindestens 500 $l \cdot m^{-2} \cdot s^{-1}$, und/oder eine Gasdurchlässigkeit, insbesondere Luftdurchlässigkeit, von bis zu 10.000 $l \cdot m^{-2} \cdot s^{-1}$, insbesondere bis zu 20.000 $l \cdot m^{-2} \cdot s^{-1}$, bei einem Strömungswiderstand von 127 Pa aufweisen.

[0129] Gemäß einer Ausführungsform der vorliegenden Erfindung kann das erfindungsgemäß eingesetzte Trägermaterial eine dreidimensionale Struktur aufweisen. Insbesondere kann das erfindungsgemäß eingesetzte Trägermaterial gemäß dieser Ausführungsform als vorzugsweise offenporiger Schaumstoff, besonders bevorzugt als Polyurethanschaumstoff, ausgebildet sein.

[0130] Gemäß einer alternativen Ausführungsform dagegen kann das erfindungsgemäß eingesetzte Trägermaterial eine zweidimensionale bzw. flächige Struktur aufweisen. Insbesondere kann bei dieser Ausführungsform das erfindungsgemäß eingesetzte Trägermaterial als vorzugsweise textiles Flächengebilde ausgebildet sein. Beispielsweise kann das Trägermaterial als ein textiles Flächengebilde, vorzugsweise ein luftdurchlässiges Textilmaterial, bevorzugt ein Gewebe, Gewirke, Gestricke, Gelege oder Textilverbundstoff, insbesondere Vlies (Nonwoven), ausgebildet sein. Gleichermaßen kann das Trägermaterial ein Flächengewicht von 5 bis 1.000 g/m^2, insbesondere 10 bis 500 g/m^2, bevorzugt 25 bis 450 g/m^2, aufweisen.

[0131] Bei dieser Ausführungsform kann das Trägermaterial ein natürliche Fasern und/oder synthetische Fasern (Chemiefasern) enthaltendes oder hieraus bestehendes textiles Flächengebilde sein. In diesem Zusammenhang können die natürlichen Fasern aus der Gruppe von Wollfasern und Baumwollfasern (CO) ausgewählt sein. Zudem können die synthetischen Fasern ausgewählt sein aus der Gruppe von Polyester (PES) ; Polyolefinen, insbesondere Polyethylen (PE) und/oder Polypropylen (PP); Polyvinylchloriden (CLF); Polyvinylidenchloriden (CLF); Acetaten (CA); Triacetaten (CTA); Polyacryl (PAN); Polyamiden (PA), insbesondere aromatischen, vorzugsweise flammfesten Polyamiden; Polyvinylalkoholen (PVAL); Polyurethanen; Polyvinylestern; (Meth-)Acrylaten; Polymilchsäuren (PLA); Aktivkohle; sowie deren Mischungen.

[0132] Üblicherweise ist die stickstofffunktionalisierte bzw. stickstoffmodifizierte Aktivkohle nach der Erfindung an bzw. auf dem Trägermaterial fixiert. Dies kann beispielsweise mittels Verklebung (z. B. mittels eines Klebstoffs) oder infolge von Eigenklebrigkeit oder Eigenadhäsion, insbesondere des Trägermaterials verwiesen werden.

[0133] Die erfindungsgemäße Aktivkohle mit der Stickstoffmodifizierung bzw. Stickstofffunktionalisierung ist mit einer Vielzahl von Vorteilen verbunden, wie sie bereits zuvor angeführt sind.

[0134] Die Aktivkohle nach der Erfindung kann für zahlreiche Verwendungen eingesetzt werden.

[0135] So kann die stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohle zur Herstellung von Filtern und Filtermaterialien aller Art, insbesondere zur Entfernung von Schad-, Geruchs- und Giftstoffen aller Art, insbesondere aus Luft- und/oder Gasströmen, wie ABC-Schutzmaskenfiltern, Geruchsfiltern, Flächenfiltern, Luftfiltern, insbesondere Filtern für die Raumluftreinigung, adsorptionsfähigen Trägerstrukturen und Filtern für den medizinischen Bereich, verwendet werden.

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[0136] Zudem kann die erfindungsgemäße stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohle zur Herstellung von Schutzmaterialien aller Art, insbesondere von Schutzbekleidung, insbesondere für den zivilen oder militärischen Bereich, wie Schutzanzügen, Schutzhandschuhe, Schutzschuhwerk, Schutzsocken, Kopfschutzbekleidung und dergleichen, und von Schutzabdeckungen aller Art, vorzugsweise alle vorgenannten Schutzmaterialien für den ABC-Einsatz, verwendet werden.

[0137] Dabei weist die erfindungsgemäße stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohle ausgezeichnete Sorptionseigenschaften auf, insbesondere in Bezug auf schwefelhaltige bzw. stickstoffhaltige Gase, wie Schwefelwasserstoffe, insbesondere Schwefelwasserstoff (H_2S), sowie gegenüber Ammoniak (NH_3).

[0138] Insbesondere eignet sich die erfindungsgemäße stickstoffmodifizierte bzw. stickstofffunktionalierte Aktivkohle auch zur Verwendung als Sorptionsspeicher für Gase oder Flüssigkeiten. Zudem kann die erfindungsgemäße Aktivkohle mit Stickstofffunktionalisierung bzw. Stickstoffmodifizierung als Katalysator oder Katalysatorträger Verwendung finden. Für die vorgenannten Anwendungen ist die hohe spezifische Oberfläche und die ausgezeichnete Reaktivität bzw. katalytische Aktivität der erfindungsgemäßen stickstoffmodifizierten bzw. stickstofffunktionalisierten Aktivkohle von entscheidender Bedeutung.

[0139] Weiterhin kann die erfindungsgemäße stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohle für die chemische Katalyse, insbesondere zur Katalyse chemischer Prozesse und Reaktionen, eingesetzt werden.

[0140] Eine weitere Anwendungsmöglichkeit der erfindungsgemäßen stickstoffmodifizierten bzw. stickstofffunktionalisierten Aktivkohle liegt in der Verwendung in oder als Gassensoren oder in Brennstoffzellen.

[0141] Darüber hinaus liegt eine weitere Anwendungsmöglichkeit der Aktivkohle nach der Erfindung mit Stickstoffmodifizierung bzw. Stickstofffunktionalisierung in der Verwendung für sorptive, insbesondere adsorptive Anwendungen, insbesondere als vorzugsweise reaktives und/oder katalytisches Adsorbens.

[0142] Darüber hinaus eignet sich die erfindungsgemäße Aktivkohle mit Stickstoffmodifizierung bzw. Stickstofffunktionalisierung zur Verwendung für die Gasreinigung bzw. für die Gasaufbereitung.

[0143] Eine weitere Verwendung der stickstoffmodifizierten bzw. stickstofffunktionalisierten Aktivkohle nach der Erfindung ist im Rahmen der Entfernung von Schadstoffen, insbesondere gasförmigen Schadstoffen, oder von umweltschädlichen, gesundheitsschädlichen oder toxischen Substanzen oder Gasen zu sehen.

[0144] Schließlich eignet sich die erfindungsgemäße stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohle auch für die Verwendung zur Aufbereitung und/oder Bereitstellung von Reinraumatmosphären, insbesondere für die Elektroindustrie, insbesondere für die Halbleiter- oder Chipherstellung.

[0145] Darüber hinaus betrifft die vorliegende Erfindung – gemäß einem dritten Aspekte der vorliegenden Erfindung – Schutzmaterialien, insbesondere für den zivilen oder militärischen Bereich, insbesondere Schutzbekleidung, wie Schutzanzüge, Schutzhandschuhe, Schutzschuhwerk, Schutzsocken Schutzbekleidung und dergleichen, sowie Schutzabdeckungen, vorzugsweise alle vorgenannten Schutzmaterialien für den ABC-Einsatz, welche unter Verwendung der zuvor definierten stickstoffmodifizierten bzw. stickstofffunktionalisierten Aktivkohle nach der Erfindung hergestellt sind bzw. welche die zuvor definierte stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohle nach der Erfindung aufweisen.

[0146] Darüber hinaus sind weiterer Gegenstand der vorliegenden Erfindung – gemäß einem vierten Aspekt der vorliegenden Erfindung – Filter und Filtermaterialien, insbesondere zur Entfernung von Schad-, Geruchs- und Giftstoffen aller Art, insbesondere aus Luft- und/oder Gasströmen, wie ABC-Schutzmaskenfilter, Geruchsfilter, Flächenfilter, Luftfilter, insbesondere Filter für die Raumluftreinigung, adsorptionsfähige Trägerstrukturen und Filter für den medizinischen Bereich, hergestellt unter Verwendung der zuvor definierten Aktivkohle nach der Erfindung mit Stickstoffmodifizierung bzw. Stickstofffunktionalisierung bzw. aufweisend die zuvor definierte stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohle nach der Erfindung.

[0147] Im Folgenden wird die vorliegende Erfindung anhand von bevorzugte Ausführungsbeispiele darstellenden Zeichnungen bzw. Figurendarstellungen näher erläutert. Im Zusammenhang mit der Erläuterung dieser bevorzugten Ausführungsbeispiele der vorliegenden Erfindung, welche jedoch in Bezug auf die vorliegende Erfindung keinesfalls beschränkend sind, werden auch weitergehende Vorteile, Eigenschaften, Aspekte und Merkmale der vorliegenden Erfindung beschrieben.

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[0148] In den Figurendarstellungen zeigt:

[0149] Fig. 1a eine grafische Darstellung des Einflusses für die thermische Oxidation der als Ausgangsmaterial eingesetzten Aktivkohle gewählten Oxidationszeit auf verschiedene Parameter bzw. auf die texturellen Eigenschaften der als Intermediat erhaltenen, an ihrer Oberfläche oxidierten Aktivkohle;

[0150] Fig. 1b eine grafische Darstellung der Abhängigkeit verschiedener Parameter bzw. der texturellen Eigenschaften der an ihrer Oberfläche oxidierten Aktivkohle von dem Gehalt an flüchtigen Bestandteilen ("fB") der an ihrer Oberfläche oxidierten Aktivkohle als Maß für den Gehalt an sauerstoffhaltigen funktionellen Gruppen bzw. als Maß für den Oxidationsgrad der als Intermediat erhaltenen an ihrer Oberfläche oxidierten Aktivkohle;

[0151] Fig. 1c eine grafische Darstellung zur Analyse der chemischen Beschaffenheit der an ihrer Oberfläche oxidierten Aktivkohle im Hinblick auf Basizität und Azidität der gebildeten Oberflächenoxide, wobei die zugrundeliegende Bestimmung mittels potentiometrischer Titration durchgeführt worden ist. Die Figurendarstellung zeigt dabei die Ausbildung von Oberflächenoxiden unter Einsatz von (Luft-)Sauerstoff als Oxidationsreagenz ("OR") unter variablen Oxidationstemperaturen (thermische Oxidation). In der Figur bezeichnet der Buchstabe "a" das basische Oberflächenoxide indizierende Kurvenmaximum;

[0152] Fig. 1d eine grafische Darstellung zur weiteren Analyse der chemischen Beschaffenheit der gebildeten Oberflächenoxide bzw. der sauerstoffhaltigen funktionellen Gruppen an der Oberfläche der Aktivkohle nach Maßgabe der Ausführungen in Fig. 1c. Als Oxidationsreagenz ("OR") wurden jedoch unterschiedlich konzentrierte Harnstofflösungen bzw. Wasserstoffperoxid in unterschiedlichen Konzentrationen eingesetzt (chemische Oxidation). In der Figur bezeichnet der Buchstabe "b" das saure Oberflächenoxide indizierende Kurvenmaximum;

[0153] Fig. 1e eine grafische Darstellung zur Analyse der chemischen Beschaffenheit im Hinblick auf Basizität bzw. Azidität der im Rahmen der Oxidation erhaltenen sauerstoffhaltigen funktionellen Gruppen, wobei die Daten mittels TPD-MS (Temperaturprogrammierte Desorption, gekoppelt mit Massenspektroskopie) ermittelt worden sind. Die Desorption von Kohlendioxid (CO_2) indiziert dabei die während der Oxidation vorliegende Bildung saurer Oberflächenoxide, wogegen die Desorption von Kohlenmonoxid (CO) die im Rahmen der Oxidation hervorgerufene Bildung basischer Oberflächenoxide Gruppen indiziert. Die Oxidation wurde auf Basis der zuvor in Fig. 1d angeführten chemischen Oxidation durchgeführt.

[0154] Fig. 1f eine grafische Darstellung nach Maßgabe von Fig. 1e, wobei sich die erhaltenen Daten bzw. Messwerte im Gegensatz hierzu auf eine mittels thermischer Oxidation erhaltenen, an ihrer Oberfläche oxidierten Aktivkohle, wie in Fig. 1c angeführt, beziehen;

[0155] Fig. 2a eine grafische Darstellung der Wasserdampfisothermen von an ihrer Oberfläche oxidierten Aktivkohlen, welche im Rahmen von Oxidation mittels (Luft-)Sauerstoff bei variablen Temperaturen bzw. Zeitdauern gewonnen wurden (thermische Oxidation), im Vergleich zu einem nichtoxidierten Ausgangsmaterial bzw. zu einer Ausgangsaktivkohle;

[0156] Fig. 2b eine grafische Darstellung von Wasserdampfisothermen von an ihrer Oberfläche oxidierten Aktivkohlen, wobei als diesbezügliches Oxidationsreagenz ("OR") Wasserstoffperoxid bzw. Salpetersäure (HNO_3) bei jeweils variabler Oxidationsdauer eingesetzt wurde (chemische Oxidation) im Vergleich zu einer nichtoxidierten Ausgangsaktivkohle;

[0157] Fig. 3a eine grafische Darstellung der Wasserdampfisothermen von stickstoffmodifizierten bzw. stickstofffunktionalisierten Aktivkohlen mit dem Einfluss der bei der Stickstoffausrüstung gewählten Temperatur auf das Endprodukt, wobei der jeweils rechte Adsorptionsast der untersuchten Aktivkohle die Adsorption und der jeweils linke Adsorptionsast der untersuchten Aktivkohle die Desorption von Wasser darstellt;

[0158] Fig. 3b grafische Darstellungen des Einflusses der durchgeführten Oxidation mit der nachfolgenden Stickstofffunktionalisierung bzw. -modifizierung auf die jeweiligen Parameter bzw. texturellen Eigenschaften der resultierenden Aktivkohle und des diesbezüglichen Einflusses der bei der Funktionalisierung bzw. Modifizierung mit Stickstoff gewählten Temperatur, wobei in der grafischen Darstellung das Symbol ① die Basis- bzw. Ausgangsaktivkohle, das Symbol ② die nach Oberflächenoxidation erhaltene, an ihrer Oberfläche oxidierte Aktivkohle (Intermediat), welche somit noch nicht stickstoffmodifiziert bzw. -funktionalisiert ist, und das Symbol ③ die mit Stickstoff funktionalisierte bzw. modifizierte Aktivkohle in Form des Endprodukts darstellen;

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[0159] Fig. 4a einen dem erfindungsgemäß durchgeführten Verfahren zugrundeliegenden, das erfindungsgemäß durchgeführte Verfahren jedoch in nichtbeschränkender Weise charakterisierenden Reaktionsablauf zum Erhalt einer stickstoffmodifizierten bzw. stickstofffunktionalisierten Aktivkohle, ausgehend von einer an ihrer Oberfläche oxidierten Aktivkohle unter Einsatz von Harnstoff als Stickstoffmodifizierungsreagenz ("SMR");

[0160] Fig. 4b ein XPS-Spektrum (X-Ray Photoelectron Spectroscopy; Röntgen-Fotoelektronenspektroskopie) zum Nachweis stickstoffhaltiger funktioneller Gruppen für verschiedene Wärmebehandlungsschritte zum Erhalt der stickstofffunktionalisierten bzw. stickstoffmodifizierten Aktivkohlen;

[0161] Fig. 5 eine grafische Darstellung in Form eines Balkendiagramms mit den für verschiedene Aktivkohlen ermittelten Durchbruchzeiten in Bezug auf verschiedene Sorptionssubstanzen (NH_3, H_2S sowie SO_2), wobei in Fig. 5 das Symbol ① eine nichtoxidierte Ausgangaktivkohle darstellt und die weiteren Aktivkohlen gemäß den Symbolen ② bis ④ jeweils auf Basis von Harnstoff stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohlen darstellen. In diesem Zusammenhang stellen die Symbole ② bzw. ③ stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohlen dar, welche auf Basis einer mit (Luft-)Sauerstoff bei Temperaturen von 400°C bzw. 450°C an ihrer Oberfläche oxidierten Aktivkohlen erhalten wurden (thermische Oxidation). Das Symbol ③ stellt zudem eine stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohle dar, welche auf Basis einer mittels Salpetersäure (HNO_3) an ihrer Oberfläche oxidierten Aktivkohle erhalten wurde (chemische Oxidation);

[0162] Fig. 6a eine grafische Darstellung von Durchbruchexperimenten unter Verwendung von Schwefeldioxid (SO_2) als zu sorbierendes Gas auf Basis eines Vergleichs von Ausgangsaktivkohle ("Ausgangsmaterial") einerseits und einer stickstoffmodifizierten bzw. stickstofffunktionalisierten Aktivkohle ("Endprodukt") andererseits;

[0163] Fig. 6b eine grafische Darstellung von Durchbruchexperimenten nach Maßgabe der Ausführungen zu Fig. 6a, wobei im Unterschied hierzu Schwefelwasserstoff (H_2S) als zu sorbierendes Gas eingesetzt wurde;

[0164] Fig. 7a ein XPS-Spektrum eines C 1 s-Bereichs-Scans einer an ihrer Oberfläche oxidierten, jedoch noch nicht stickstoffmodifizierten bzw. stickstofffunktionalisierten Aktivkohle mit der Darstellung der erhaltenen Messwerte ("Messdaten") und der entsprechenden Datenanpassung zu Zwecken der Oberflächenanalyse bzw. der chemischen Struktur der Oberfläche der oxidierten Aktivkohle als Intermediat des erfindungsgemäß durchgeführten Verfahrens;

[0165] Fig. 7b ein weiteres XPS-Spektrum eines O 1 s-Bereichs-Scans mit den auf diese Weise ermittelten Messdaten ("Messdaten") und der entsprechenden Datenanpassung für eine an ihrer Oberfläche oxidierten, jedoch noch nicht stickstoffmodifizierten bzw. stickstofffunktionalisierten Aktivkohle.

[0166] Die Figurendarstellung gemäß Fig. 1a veranschaulicht den Einfluss der Oxidationszeit auf verschiedene Parameter bzw. auf die texturellen Eigenschaften der auf Basis der erfindungsgemäß durchgeführten Oxidation erhaltenen, an ihrer Oberfläche oxidierten Aktivkohle, wobei (Luft-)Sauerstoff als Oxidationsreagenz ("OR") eingesetzt wurde (thermische Oxidation) und wobei als Ausgangsaktivkohle zum einen eine niedrig aktivierte Aktivkohle und zum anderen eine hoch aktivierte Aktivkohle eingesetzt worden ist. Die Figur zeigt, dass mit zunehmender Oxidationszeit der Gehalt an flüchtigen Bestandteilen ("fB") für beide Aktivkohlen signifikant ansteigt. Wie zuvor angeführt, stellt der Gehalt an flüchtigen Bestandteilen der an ihrer Oberfläche aktivierten Aktivkohle ein Maß für den Gehalt an sauerstoffhaltigen funktionellen Gruppen dar, wie sie im Rahmen der Oxidation gebildet werden. Für beide Aktivkohlen wird somit mit zunehmender Oxidationszahl ein Anstieg des Gehaltes an sauerstoffhaltigen funktionellen Gruppen beobachtet, welche im weiteren Verlauf der erfindungsgemäß realisierten Verfahrensführung als Reaktionspartner für das Stickstoffmodifizierungsreagenz ("SMR") fungieren können. Fig. 1a veranschaulicht weiterhin, dass unter den gegebenen Verfahrensbedingungen mit zunehmender Oxidationszeit der Oberflächen-pH-Wert der jeweiligen Aktivkohle von einem leicht basischen Bereich in einen leicht sauren Bereich absinkt. Schließlich zeigt die grafische Darstellung gemäß Fig. 1a, dass das Gesamtporenvolumen (V_{tot}) sowie die Rütteldichte (RD) für beide Aktivkohlen im Rahmen der thermischen Oxidation geringfügig verändert werden, wobei in Bezug auf das Gesamtporenvolumen sogar eine geringfügige Erhöhung vorliegt.

[0167] Fig. 1b zeigt zur weiteren Veranschaulichung die Abhängigkeit des Oberflächen-pH-Werts der an ihrer Oberfläche oxidierten Aktivkohle einerseits sowie der Rütteldichte und des Gesamtporenvolumens andererseits von dem Gehalt an flüchtigen Bestandteilen als Maß für den Gehalt an sauerstoffhaltigen funktionellen Gruppen der an ihrer Oberfläche oxidierten Aktivkohle als Intermediat der erfindungsgemäß umgesetzten Ver-

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fahrensführung. Fig. 1b zeigt gleichermaßen, dass mit Zunahme des Gehalts an sauerstoffhaltigen funktionellen Gruppen und damit des Oxidationsgrads der pH-Wert und die Rütteldichte der an ihrer Oberfläche oxidierten Aktivkohle leicht abnimmt, während in Bezug auf das Porenvolumen eine Zunahme zu beobachten ist.

[0168] Weiterhin verdeutlicht die Figurendarstellung gemäß Fig. 1c, dass im Rahmen der thermischen Oxidation unter Einsatz von (Luft-)Sauerstoff als Oxidationsreagenz ("OR") (chemische Oxidation) die Bildung basischer Oberflächenoxide bevorzugt ist. Demgegenüber zeigt die Figurendarstellung gemäß Fig. 1d, dass unter Verwendung von Harnstoff bzw. Wasserstoffperoxid (chemische Oxidation) gleichermaßen auch saure Oberflächenoxide gebildet werden. In diesem Zusammenhang bestätigen die Figurendarstellungen gemäß Fig. 1e und Fig. 1f die zuvor angeführten experimentellen Befunde. So zeigt Fig. 1f, dass die zuvor angeführte thermische Oxidation maßgeblich zur Desorption von Kohlenmonoxid (CO) als Maß für die Bildung basischer Oberflächenoxide führt, während die Bildung saurer Oberflächenoxide, welche durch die Desorption von Kohlendioxid (CO_2) angezeigt wird, nur in geringfügigem Maße erfolgt. In diesem Zusammenhang zeigt Fig. 1e, dass unter chemischen Oxidationsbedingungen im Rahmen der erfindungsgemäß realisierten Verfahrensführung als Intermediat eine an ihrer Oberfläche oxidierte Aktivkohle erhalten wird, welche einen erhöhten Gehalt an sauren Oberflächenoxiden aufweist.

[0169] Insgesamt verdeutlicht die Figurendarstellung gemäß Fig. 1a bis Fig. 1e somit, dass es bei der erfindungsgemäß umgesetzten Verfahrensführung mit der gezielten Auswahl bzw. Abstimmung der Oxidationsbedingungen zur Bildung der an ihrer Oberfläche oxidierten Aktivkohle um einen steuerbaren bzw. individuell einstellbaren Prozess handelt, auf dessen Basis das so erhaltene Intermediat individuell ausgebildet werden kann, insbesondere im Hinblick auf die nachfolgende Ausrüstung bzw. Modifizierung mit Stickstoff. Als diesbezüglich im Rahmen der Oxidation variable Verfahrensparameter kommen insbesondere zum einen das eingesetzte Oxidationsreagenz ("OR") sowie zum anderen Temperatur und Oxidationszeit infrage. Die im Rahmen der erfindungsgemäß realisierten Verfahrensführung vorgesehene oxidative Vorbehandlung zum Erhalt der an ihrer Oberfläche oxidierten Aktivkohle als Intermediat stellt somit einen einstellbaren bzw. individuell abstimmbaren Prozess dar, wobei insbesondere die Intensität bzw. das Maß der Oxidation durch Prozessparameter wie Oxidationszeit, Oxidationstemperatur und Konzentration des eingesetzten Oxidationsreagenzes ("OR") und die Art der gebildeten Oberflächenoxide über die Auswahl des Oxidationsmittels gesteuert bzw. maßgeschneidert werden kann.

[0170] Weiterhin zeigen die Figurendarstellungen gemäß Fig. 2a und Fig. 2b die Wasserdampfisothermen von auf verschiedene Art und Weise an ihrer Oberfläche oxidierter Aktivkohle im Vergleich zu einer nichtoxidierten Ausgangsaktivkohle, wobei Fig. 2a auf eine thermische Oxidation unter Verwendung von (Luft-)Sauerstoff als Oxidationsreagenz ("OR") unter verschiedenen Temperaturen (thermische Oxidation) und Fig. 2b auf den Einsatz von Oxidationsreagenzien ("OR") auf Basis von Wasserstoffperoxid und Salpetersäure bei unterschiedlichen Oxidationszeiten (chemische Oxidation) abstellen. Dabei ist in beiden Figuren zu erkennen, dass die durchgeführte Oxidation zu einer Verschiebung der Adsorption von Wasser in Richtung kleinerer p/p_0-Werte führt, was einer Erhöhung der Kapazität infolge der durchgeführten Oxidation unter Schaffung hydrophiler Oberflächen entspricht, was wiederum im Hinblick der nachfolgenden Anbindung des Stickstoffmodifizierungsreagenzes ("SMR") von Relevanz ist. In diesem Zusammenhang zeigt Fig. 2a, dass eine Erhöhung der Temperatur einerseits und der Oxidationszeit und -dauer andererseits zu einer höheren Kapazität bzw. zu einer vermehrten Bildung hydrophiler Oberflächen der so behandelten Aktivkohle führt. Entsprechend kann Fig. 2b entnommen werden, dass unter den vorliegenden Bedingungen Salpetersäure zu einer stärkeren Kapazitätserhöhung bzw. zu einem höheren Gehalt hydrophiler Oberflächen im Vergleich zu Wasserstoffperoxid führt, wobei auch hier eine weitere Erhöhung der entsprechenden Werte bei Zunahme der Oxidationsdauer zu beobachten ist.

[0171] Die Figurendarstellung gemäß Fig. 3a zeigt die für eine unter Verwendung von Ammoniak bei verschiedenen Temperaturen stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohle erhaltenen Wasserdampfisothermen, wobei bei einer geringeren Funktionalisierungstemperatur von 500°C im Vergleich zu einer höheren Funktionalisierungstemperatur von 920°C eine größere Verschiebung der Adsorption zu kleineren p/p_0-Werten zu beobachten ist, was auf eine höhere Funktionalität, insbesondere auch im Hinblick auf die Ausbildung hydrophiler Oberflächen, der erhaltenen Aktivkohle bei niedrigeren Temperaturen bzw. eine Integration von Stickstoff in das Kohlenstoffgerüst bei höheren Temperaturen spricht. In diesem Zusammenhang zeigt die Figurendarstellung gemäß Fig. 3b, dass auf Basis der erfindungsgemäßen umgesetzten Verfahrensführung mit der Oxidation von Ausgangsaktivkohle und der nachfolgenden Stickstoffmodifizierung bzw. -funktionalisierung die zugrundeliegenden Parameter bzw. texturellen Eigenschaften der erhaltenen Aktivkohle nicht negativ beeinflusst werden. Vielmehr ist sowohl in Bezug auf das Gesamtporenvolumen nach Gurvich (V_{tot}), das Mikroporenvolumen (V_{mic}) und der MP-BET-Oberfläche zu beobachten, dass die durchgeführte Oxidati-

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on zur Ausbildung funktioneller sauerstoffhaltiger Gruppen an der Oberfläche der Aktivkohle sogar zu einem leichten Anstieg und die nachfolgende Stickstoffmodifizierung bzw. -funktionalisierung allenfalls zu einer geringfügigen Abnahme der in Rede stehenden Werte führt, welche jedoch immer noch oberhalb der Werte des entsprechenden Ausgangsmaterials liegen. Die erfindungsgemäß realisierte Verfahrensführung führt somit in keiner Weise zu einer Verschlechterung der angeführten Parameter der zugrundeliegenden Aktivkohle, wobei diesbezüglich auch keine erheblichen Unterschiede hinsichtlich der bei der Stickstofffunktionalisierung bzw. -modifizierung gewählten Temperatur vorliegen. Die in Fig. 3b untersuchten Aktivkohlen wurden gleichermaßen unter Verwendung von Ammoniak mit Stickstoff ausgerüstet.

[0172] Die Figurendarstellung gemäß Fig. 4a wiederum zeigt einen beispielhaften, dem erfindungsgemäß durchgeführten Verfahren zugrundeliegenden Reaktionsablauf, wobei Abbildung A das Kohlenstoffgerüst der an ihrer Oberfläche oxidierten Aktivkohle (Intermediat) mit den ausgebildeten sauerstoffhaltigen funktionellen Gruppen darstellt. Unter Zugabe von Harnstoff als Stickstoffmodifizierungsreagenz ("SNR") wird nachfolgend das in Abbildung B veranschaulichte Zwischenprodukt mit angebundener Harnstoffkomponente erhalten. Abbildung C zeigt das bei einer Temperaturbehandlung von 500°C erhaltene (Zwischen-)Produkt mit der Umsetzung des Harnstoffs zu den entsprechenden stickstoffhaltigen funktionellen Gruppen an der Oberfläche der Kohlenstoffmatrix, wobei diesbezüglich zyklische Harnstoffderivate, Nitrile, Amide bzw. primäre Amine ausgebildet werden. Durch eine weitere Temperaturbehandlung bei 920°C erfolgt eine Integration des Stickstoffs in das Kohlenstoffgerüst bzw. in die Kohlenstoffmatrix der stickstoffmodifizierten bzw. stickstofffunktionalisierten Aktivkohle unter Abbau der stickstoffhaltigen funktionellen Gruppe, wie in Abbildung D dargestellt.

[0173] In diesem Zusammenhag veranschaulicht die Figurendarstellung gemäß Fig. 4b auf Basis eines XPS-Spektrums das Vorhandensein verschiedener Spezies von stickstoffhaltigen funktionellen Gruppen zu verschiedenen Zeitpunkten bzw. bei verschiedenen Temperaturen während des Vorgangs der Stickstoffmodifizierung bzw. -funktionalisierung. Während bei niedrigen Temperaturen insbesondere stickstoffhaltige funktionelle Gruppen auf Basis von Amiden bzw. Pyrrolen vorliegen, erfolgt bei höheren Temperaturen von 500°C eine Umwandlung bzw. eine Verlagerung zu insbesondere quaternären stickstoffhaltigen Gruppen, während bei noch höheren Temperaturen von 920°C der Gehalt an den vorgenannten Gruppen signifikant zurückgeht, was auf eine Integration von Stickstoff in das Kohlenstoffgerüst bzw. in die Kohlenstoffmatrix der so behandelten Aktivkohle hindeutet.

[0174] Was die Figurendarstellung gemäß Fig. 5 anbelangt, so zeigt diese einen Vergleich der Durchbruchzeiten von drei verschiedenen erfindungsgemäßen Aktivkohlen in Bezug auf die Sorption von Ammoniak (NH_3), Schwefelwasserstoff (H_2S) und Schwefeldioxid (SO_2) im Vergleich zu einer Ausgangsaktivkohle. Hierzu wurden die entsprechenden Aktivkohlen unter definierten Bedingungen für einen bestimmten Zeitraum einem kontinuierlichen Volumenstrom einer mit dem jeweiligen Schadgas angereicherten Atmosphäre ausgesetzt und die jeweiligen Durchbruchzeiten bestimmt. Sämtliche erfindungsgemäßen Aktivkohlen zeigen deutlich über die Ausgangsaktivkohle hinausgehende Sorptionsleisturgen sowohl in Bezug auf Ammoniak (NH_3), Schwefelwasserstoff (H_2S) als auch Schwefeldioxid (SO_2). Insgesamt ist das Sorptionsverhalten der erfindungsgemäßen Aktivkohlen als hervorragend einzustufen.

[0175] Weiterhin verdeutlichen die Figurendarstellungen gemäß Fig. 6a und Fig. 6b das signifikant verbesserte Sorptionsverhalten einer erfindungsgemäß ausgerüsteten Aktivkohle (Endprodukt) im Vergleich zu dem eingesetzten Ausgangsmaterial bzw. der eingesetzten Ausgangsaktivkohle. So zeigt die auf Basis des erfindungsgemäß durchgeführten Verfahrens hergestellte und mit Stickstoff ausgerüstete Aktivkohle ein signifikant verbessertes Durchbruchverhalten gegenüber den zu sorbierenden Gasen Schwefeldioxid (SO_2) (Fig. 6a) einerseits und Schwefelwasserstoff (H_2S) (Fig. 6b) andererseits. Zur Ermittlung der Durchbruchdaten wurden die jeweiligen Aktivkohlen unter definierten Bedingungen einem konstanten Volumenstrom einer mit dem zu sorbierenden Gas angereicherten Atmosphäre für einen definierten Zeitraum ausgesetzt, wobei die in den jeweiligen Figuren angegebenen Konzentrationen des zu sorbierenden Gases eingesetzt wurden.

[0176] Schließlich zeigen die Figurendarstellungen gemäß Fig. 7a und Fig. 7b das Vorhandensein von sauerstoffhaltigen funktionellen Gruppen einer für die nachfolgende Stickstoffmodifizierung bzw. Stickstofffunktionalisierung eingesetzten an ihrer Oberfläche oxidierten Aktivkohle. Durch Oxidation unter Einbezug eines Oxidationsreagenzes ("OR") werden zahlreiche sauerstoffhaltige funktionelle Gruppen erhalten, wie z. B. Carboxyl- und Carbonylgruppen, welche als Reaktionspartner bzw. Aminogruppen für die nachfolgende Modifizierung bzw. Funktionalisierung mit dem Stickstoffmodifizierungsreagenz ("SMR") fungieren.

[0177] Zusammenfassend ist es im Rahmen der vorliegenden Erfindung durch die Umsetzung eines speziellen Verfahrens erstmalig gelungen, eine Funktionalisierung von Aktivkohle bzw. von polymerbasierter sphä-

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rischer Aktivkohle (PBSAC) mit stickstoffhaltigen funktionellen Gruppen bzw. von Aktivkohle mit Integration von Stickstoff in die Kohlenstoffoberfläche (N-Dotierung) zu erhalten. Die so erhaltenen Aktivkohlen bzw. polymerbasierten sphärischen Aktivkohlen (PBSACs) weisen dabei eine Reaktivität gegenüber unterschiedlichen Zielgasen auf. Als entsprechende Zielgase für die auf Basis des erfindungsgemäß durchgeführten Verfahrens erhaltenen Aktivkohlen können Gase in Betracht kommen, deren Abbau über eine Oxidation an der Kohlenstoffoberfläche möglich ist, wie es beispielsweise für SO_2, H_2S, ASH_3 sowie DMS der Fall ist.

[0178] Die im Rahmen des erfindungsgemäß durchgeführten Verfahrens erhaltenen Aktivkohlen können gleichermaßen stickstoffhaltige funktionelle Gruppen auf der Oberfläche aufweisen, welche mit organischen Gasen bzw. Dämpfen Bindungen eingehen können, wie Formaldehyd und gegebenenfalls NO_x-Verbindungen. Durch die Stickstofffunktionalisierung kann im Allgemeinen die Basizität der Oberfläche der Aktivkohle (im Gegensatz zur Oxidation) erhöht werden. Die Ausbildung von stickstoffhaltigen funktionellen Gruppen bzw. die Dotierung der Aktivkohle mit Stickstoff kann im Rahmen der erfindungsgemäß realisierten Verfahrensführung in nicht beschränkender Weise durch das Füllen der Poren mit einer stickstoffhaltigen organischen Verbindung (wie z. B. Harnstoff) insbesondere aus wässrigen Lösungen und einem sich anschließenden Pyrolyseprozess erfolgen.

[0179] Nach dem eigentlichen Herstellungsprozess kann sich ein Aufreinigungsschritt, insbesondere ein Waschen mit Wasser, anschließen, insbesondere um etwaige Pyrolyse- bzw. Verbrennungsrückstände zu entfernen. Die bei der Stickstofffunktionalisierung bzw. -modifizierung gewählte Temperatur beeinflusst im Rahmen der erfindungsgemäß umgesetzten Verfahrensführung die chemische Konstitution des eingebrachten Stickstoffs. In diesem Zusammenhang führen niedrige Temperaturen, insbesondere im Bereich von 500°C, insbesondere zur Ausbildung von stickstoffhaltigen funktionellen Gruppen, während höhere Temperaturen, insbesondere im Bereich von 920°C vorrangig zu den zuvor beschriebenen N-dotierten Aktivkohlen führt.

[0180] Im Rahmen der vorliegenden Erfindung hat es sich als besonders vorteilhaft gezeigt, dass im Rahmen des erfindungsgemäß durchgeführten Verfahrens eine vorgeschaltete Oxidation der Aktivkohle durchgeführt wird, da diese die Aufnahme von Stickstoff in signifikanten Mengen ermöglicht. In diesem Zusammenhang korreliert die Menge an aufgenommenem bzw. integriertem Stickstoff mit den flüchtigen Bestandteilen der oxidierten Aktivkohle als Maß für die Menge bzw. den Gehalt an Oberflächenoxiden auf der Aktivkohle.

AUSFÜHRUNGSBEISPIELE:

1. Herstellung einer erfindungsgemäß ausgerüsteten Aktivkohle mit Stickstofffunktionalisierung bzw. -modifizierung:

[0181] Im Rahmen der im Folgenden beschriebenen Herstellung von erfindungsgemäßen Aktivkohlen wurde als Ausgangsmaterial eine kugelförmige Aktivkohle als Ausgangsaktivkohle eingesetzt, welche auf Basis von divinylbenzolvernetztem Polystyrol hergestellt worden ist. Eine derartige Ausgangsaktivkohle ist von der Fa. Blücher GmbH, Erkrath (Deutschland) erhältlich.

a) Zur Herstellung der erfindungsgemäßen Aktivkohle A wurden 500 g der Ausgangsaktivkohle mit 1.000 g einer HNO_3-Lösung (50%) zusammengeführt, und die resultierende Mischung wurde für 240 min gerührt. Anschließend wurde die überschüssige Salpetersäure abdekantiert, und die auf diese Weise erhaltene oxidierte Aktivkohle wurde mit destilliertem Wasser gewaschen und anschließend getrocknet. Eine Menge von 500 g der an ihrer Oberfläche oxidierten Aktivkohle wurden dann mit 1.000 g einer gesättigten Harnstofflösung zusammengeführt, und die resultierende Mischung wurde für 2,5 h gerührt. Anschließend wurde die überschüssige Harnstofflösung abdekantiert. Die mit der Harnstofflösung ausgerüstete Aktivkohle wurde anschließend im Inertgasstrom bei 500°C für 240 min behandelt. Die erhaltene stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohle wurde dann mit destilliertem Wasser gewaschen und getrocknet, um etwaige Verbrennungs- bzw. Pyrolyserückstände zu entfernen.

b) Zur Herstellung der erfindungsgemäßen Aktivkohle B wurden 500 g der Ausgangsaktivkohle mit 1.000 g einer HNO_3-Lösung (50%) zusammengeführt und die resultierende Mischung für 240 min gerührt. Anschließend wurde überschüssige HNO_3-Lösung abdekantiert, und die so erhaltene oxidierte Aktivkohle wurde mit destilliertem Wasser gewaschen und getrocknet. Anschließend wurden 500 g der oxidierten Aktivkohle mit 1.000 g einer gesättigten Harnstofflösung zusammengeführt, und die resultierende Mischung wurde für 2,5 h gerührt. Die überschüssige Harnstofflösung wurde abdekantiert. Die mit der Harnstofflösung ausgerüstete Aktivkohle wurde im Inertgasstrom bei 920°C für 240 min behandelt. Die so erhaltene stickstofffunktionalisierte bzw. stickstoffmodifizierte Aktivkohle wurde mit destilliertem Wasser gewaschen und anschließend getrocknet, um Verbrennungsrückstände zu entfernen.

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c) Zur Herstellung der erfindungsgemäßen Aktivkohle C wurden 500 g der Ausgangsaktivkohle im Drehrohrofen im Luftstrom bei 450°C für 120 min behandelt. Die so erhaltene oxidierte Aktivkohle wurde mit 1.000 g einer gesättigten Harnstofflösung zusammengeführt, und die resultierende Mischung wurde für 2,5 h gerührt. Anschließend wurde die überschüssige Harnstofflösung abdekantiert. Die mit der Harnstofflösung ausgerüstete Aktivkohle wurde anschließend im Inertgasstrom bei 500°C für 240 min behandelt. Die resultierende stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohle wurde schließlich gewaschen und getrocknet, um etwaige Verbrennungsrückstände zu entfernen.
d) Zur Herstellung der erfindungsgemäßen Aktivkohle D wurden 50 g der Ausgangsaktivkohle im Drehrohrofen im Luftstrom bei 450°C für 120 min behandelt. 500 g der so erhaltenen Aktivkohle wurden mit 1.000 g einer gesättigten Harnstofflösung zusammengeführt, und die resultierende Mischung wurde für 2,5 h gerührt. Anschließend wurde die überschüssige Harnstofflösung abdekantiert, und die mit der Harnstofflösung ausgerüstete Aktivkohle wurde mit Inertgasstrom bei 920°C für 240 min behandelt. Die auf diese Weise erhaltene stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohle wurde schließlich mit destilliertem Wasser gewaschen und anschließend getrocknet, um etwaige Verbrennungsrückstände zu entfernen.

[0182] Die erfindungsgemäße Aktivkohlen A bis D weisen jeweils signifikant erhöhte Stickstoffgehalte auf, und die chemisorptiven Eigenschaften sind jeweils als hervorragend einzustufen.

[0183] In diesem Zusammenhang wurde der jeweilige Stickstoffgehalt der erfindungsgemäßen Aktivkohlen mit an sich bekannten Methoden bestimmt. Der Gesamtstickstoffgehalt kann mittels an sich bekannter Elementaranalyse bestimmt werden, während die Ermittlung der flüchtigen Bestandteile ("fB") gemäß der ISO 562:1981 erfolgen kann. Aus der Differenz von mittels Elementaranalyse bestimmtem Gesamtstickstoffgehalt einerseits und gemäß der ISO 562:1981 bestimmtem Gehalt an flüchtigen Bestandteilen (d. h. flüchtigen stickstoffhaltigen funktionellen Gruppen) kann dann der Gehalt an immobilisiertem bzw. in das Kohlenstoffgerüst eingebautem Stickstoff ermittelt werden.

[0184] Im Einzelnen ergeben sich für die erfindungsgemäßen Aktivkohlen A bis D die folgenden Stickstoffgehalte: So weist die erfindungsgemäße Aktivkohle A einen Gesamtstickstoffgehalt von 13% auf, bezogen auf das Trockengewicht der stickstoffmodifizierten bzw. stickstofffunktionalisierten Aktivkohle, wobei hiervon etwa 12%, bezogen auf den Gesamtstickstoffgehalt, in Form von flüchtigen Bestandteilen ("fB") vorliegt. Für die erfindungsgemäße Aktivkohle B ergibt sich ein entsprechender Gesamtstickstoffgehalt von 18 Gew.-%, bezogen auf das Trockengewicht der stickstoffmodifizierten bzw. stickstofffunktionalisierten Aktivkohle, wobei diesbezüglich lediglich 5% des Stickstoffs, bezogen auf den Gesamtstickstoffgehalt, in Form von flüchtigen Bestandteilen ("fB") vorliegt, d. h. 95% des Stickstoffs liegen in der Kohlenstoffmatrix integriert vor.

[0185] Weiterhin ergibt sich für die erfindungsgemäße Aktivkohle C ein Gesamtstickstoffgehalt von 15 Gew.-%, bezogen auf die stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohle. In diesem Fall beträgt der Anteil des in Form von flüchtigen Bestandteilen ("fB") vorliegenden Stickstoffs, bezogen auf den Gesamtstickstoffgehalt, 14%.

[0186] Schließlich ergibt sich für die erfindungsgemäße Aktivkohle D ein Gesamtstickstoffgehalt, bezogen auf die stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohle von 19,5 Gew.-%, wobei in diesem Zusammenhang 7% des Stickstoffs in Form von flüchtigen Bestandteilen ("fB") vorliegt.

[0187] Die Ergebnisse veranschaulichen den hohen Gehalt an Stickstoff in Bezug auf die erfindungsgemäßen Aktivkohlen, welche auf Basis des erfindungsgemäß durchgeführten Verfahrens erhalten werden, wobei diesbezüglich gleichermaßen gewährleistet ist, dass ein hoher Anteil des Stickstoffs fest in das Kohlenstoffgerüst integriert vorliegt. Die diesbezügliche Integration bzw. Inkorporierung des Stickstoffs in das Kohlenstoffgerüst ist bei höheren Temperaturen während der Stickstoffmodifizierung bzw. -funktionalisierung stärker ausgebildet, während bei niedrigen Temperaturen der Anteil an stickstoffhaltigen funktionellen Gruppen erhöht ist.

[0188] In Bezug auf die erfindungsgemäßen Aktivkohlen wurden zudem Durchbruchuntersuchungen gegenüber Schwefelwasserstoff (H_2S) durchgeführt. In diesem Zusammenhang wurde eine definierte Schüttung der jeweiligen Aktivkohlen A bis D, welche jeweils eine Höhe von 2 cm aufwies, bei Temperaturen von 30°C einem konstanten Volumenstrom mit konstanter Strömungsgeschwindigkeit ausgesetzt, welcher 20 ppm H_2S enthielt. Die erfindungsgemäßen Aktivkohlen wiesen dabei folgende Durchbruchzeiten auf: So konnte für die erfindungsgemäße Aktivkohle A eine Durchbruchzeit von 112 min und für die erfindungsgemäße Aktivkohle B eine Durchbruchszeit von 123 min ermittelt werden. Weiterhin ergab sich für die erfindungsgemäße Aktivkohle C eine Durchbruchzeit von 112 min, und für die erfindungsgemäße Aktivkohle D konnte eine Durchbruchzeit von 119 min ermittelt werden. Eine als Vergleich eingesetzte unbehandelte Ausgangsaktivkohle wies eine

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Durchbruchzeit von lediglich 75 min auf. Die vorgenannten Ergebnisse belegen die signifikante Verbesserung der Adsorptionsleistung der erfindungsgemäßen Aktivkohle in Bezug auf das zu sorbierende Schadgas.

[0189] Die zuvor geschilderten Herstellungsbeispiele wurden wiederholt, wobei jedoch als Stickstoffmodifizierungsreagenz Melamin ("Aktivkohle E"), gasförmiges Ammoniak ("Aktivkohle F"), Ammoniakwasser ("Aktivkohle G") oder Aminosäuren ("Aktivkohle H") eingesetzt wurden; es wurden vergleichbare Ergebnisse erhalten.

2. Weitere Ausführungsbeispiele und experimentelle Untersuchungen:

[0190] Im Folgenden wird auf die den bereits zuvor beschriebenen Figurendarstellungen zugrundeliegenden Untersuchungen bzw. Ausführungsbeispiele weiterführend eingegangen:

a) Wie zuvor angeführt, liegt der Figurendarstellung gemäß Fig. 1a eine Untersuchung des Einflusses der durchgeführten Oxidation der Ausgangsaktivkohle auf bestimmte Parameter der erhaltenen, an ihrer Oberfläche oxidierten Aktivkohle zugrunde. Diesbezüglich wurde eine niedrig aktivierte Ausgangsaktivkohle mit relativ geringem Gesamtporenvolumen einerseits und eine hoch aktivierte Ausgangsaktivkohle mit relativ hohem Gesamtporenvolumen andererseits als für die nachfolgende Oxidation eingesetztes Material verwendet. Die Oxidation wurde unter Verwendung von (Luft-)Sauerstoff als Oxidationsreagenz ("OR") im Sinne der zuvor definierten thermischen Oxidation durchgeführt, wobei in diesem Zusammenhang die in Fig. 1a dargestellten Parameter in Abhängigkeit von der Oxidationszeit bestimmt wurden. Dabei zeigt sich, dass für beide Ausgangsaktivkohlen mit zunehmender Oxidationszeit eine Erhöhung der flüchtigen Bestandteile ("fB") vorliegt, was einer Generierung von sauerstoffhaltigen funktionellen Gruppen an der Oberfläche der jeweiligen Aktivkohle entspricht.

Gleichermaßen zeigt die Figurendarstellung gemäß Fig. 1a, dass mit zunehmender Oxidationszeit der Oberflächen-pH-Wert und die Rütteldichte der oxidierten, jedoch noch nicht einer Stickstoffmodifizierung bzw. Stickstofffunktionalisierung unterzogenen Aktivkohle sinken. Dahingehen ist in Bezug auf das Gesamtporenvolumen eine leichte Erhöhung zu beobachten. Die diesbezüglich aufgefundenen Abhängigkeiten der in Rede stehenden Parameter von der zugrundeliegenden Verfahrensdurchführung wird gleichermaßen durch die Figurendarstellung gemäß Fig. 1b bestätigt. Für weitere diesbezügliche Ausführungen kann auf obige Ausführungen zu den entsprechenden Figurendarstellungen verwiesen werden.

Zusammenfassend ist somit festzustellen, dass durch die gezielte Auswahl von Prozessparametern, wie z. B. der Oxidationszeit, im Rahmen der dem erfindungsgemäß durchgeführten Verfahren zugrundeliegenden Oxidation von Ausgangsaktivkohle, die entsprechenden Eigenschaften der an ihrer Oberfläche oxidierten Aktivkohle als Intermediat der erfindungsgemäß umgesetzten Verfahrensführung gezielt eingestellt bzw. maßgeschneidert werden können.

b) Die zuvor aufgezeigte Einflussnahme von Prozessparametern auf die texturellen Eigenschaften der an ihrer Oberfläche oxidierten Aktivkohle wird auch durch den der Figurendarstellung gemäß Fig. 1c bis Fig. 1f zugrundeliegenden Untersuchungskomplex, welcher auf die Einflussnahme der Oxidationsbedingungen auf die Basizität bzw. Azidität der ausgebildeten Oberflächenoxide abstellt, bestätigt. In diesem Zusammenhang wurden entsprechende Ausgangsaktivkohlen zum einen einer thermischen Oxidation unter Einsatz von (Luft-)Sauerstoff als Oxidationsreagenz ("OR") und andererseits einer chemischen Oxidation unter Einsatz von Salpetersäure bzw. Wasserstoffperoxid als Oxidationsreagenz ("OR") unterzogen. Eine nachfolgende Analyse der so erhaltenen an ihrer Oberfläche oxidierten Aktivkohlen zeigt dabei, dass für thermische Oxidationsbedingungen vorrangig basische Oberflächenoxide gebildet werden, wogegen für chemische Oxidationsbedingungen auch saure Oberflächenoxide gebildet werden. Auf dieser Basis kann somit die chemische Struktur der Oberfläche der oxidierten Aktivkohle gezielt eingestellt bzw. maßgeschneidert werden. Für weitere Ausführungen kann zur Vermeidung unnötiger Wiederholungen auf die obigen Ausführungen zu den entsprechenden Figurendarstellungen verwiesen werden.

Im Ergebnis zeigen die zugrundeliegenden Untersuchungen somit, dass durch die gezielte Auswahl der Oxidationsbedingungen die Basizität bzw. Azidität durch Bildung spezieller Oberflächenoxide gesteuert werden kann.

c) Im Rahmen eines weiteren Untersuchungskomplexes wurden entsprechende Ausgangsmaterialien einerseits einer thermischen Oxidation unter Einfluss von (Luft-)Sauerstoff und andererseits einer chemischen Oxidation unter Einsatz von Wasserstoffperoxid bzw. Salpetersäure als jeweilige Oxidationsreagenzien ("OR") unterzogen. Die so erhaltenen an ihrer Oberfläche oxidierten Aktivkohlen (Intermediat) wurden anschließend hinsichtlich der Adsorption bzw. Resorption von Wasserdampf im Vergleich zu einer nichtbehandelten Ausgangsaktivkohle (Ausgangsmaterial) untersucht. Wie in den Figurendarstellungen gemäß Fig. 2a und Fig. 2b dargestellt, erfolgt hierbei für die behandelten Aktivkohlen deutliche Verschiebung zu kleineren p/p_0-Werten. Diesbezüglich kann auch auf die obigen Ausführungen zu Fig. 2a und Fig. 2b verwiesen werden.

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Im Ergebnis zeigen die in den Figurendarstellungen gemäß Fig. 2a und Fig. 2b in Bezug genommenen Untersuchungen, dass die Wasserdampfisothermen durch die erfindungsgemäß vorgesehene Oxidation der Aktivkohle signifikant beeinflusst werden, was insbesondere als Maß für das Vorliegen einer erhöhten Hydrophilie der Oberfläche der Aktivkohle infolge der generierten sauerstoffhaltigen funktionellen Gruppen herangezogen werden kann.

d) Was die der Figurendarstellung gemäß Fig. 3a und Fig. 3b zugrundeliegenden Untersuchungen anbelangt, so fokussieren diese auf Versuchsparameter und Analyseergebnisse für zwei auf verschiedene Weise stickstofffunktionalisierte bzw. stickstoffmodifizierte Aktivkohlen nach der Erfindung (N-PBSAC), welche jeweils ausgehend von Ausgangsaktivkohle auf Basis von karbonisierten und aktivierten Polymeren hergestellt wurden (PBSAC). Das im Rahmen des erfindungsgemäß durchgeführten Verfahrens hergestellte oxidierte Basisadsorbenz (Intermediat) enthielt dabei 13,7 Gew.-% flüchtige Bestandteile ("fB"), bezogen auf das Trockengewicht des Intermediats.

In Bezug auf eine erste an ihrer Oberfläche oxidierten Aktivkohle wurde anschließend die Stickstoffmodifizierung bzw. -funktionalisierung bei einer Temperatur von T = 500°C durchgeführt, wobei diesbezüglich die folgenden Verfahrensschritte durchgeführt worden sind. Die an ihrer Oberfläche aktivierte Aktivkohle wurde in einem NH_3-Volumenstrom (5 l/h) mit einer Aufheizrate von 36 K/min auf 100°C erwärmt, und die Temperatur wurde für 30 min gehalten. Anschließend wurde mit einer Aufheizrate von 26 K/min auf 400°C erwärmt. Anschließend wurde im Stickstoffstrom mit einem Volumenstrom von 6 l/h mit 36 K/min auf 500°C aufgeheizt, wobei die Temperatur für 240 min gehalten wurde. Anschließend erfolgte eine Abkühlung über Nacht unter Stickstoffatmosphäre.

In Bezug auf die zweite Aktivkohle wurde wie zuvor beschrieben verfahren, jedoch mit der Maßgabe dass unter Stickstoffatmosphäre nicht eine Aufheizung auf 500°C, sondern auf 920°C erfolgte.

Die so erhaltenen funktionalisierten Aktivkohlen wiesen die folgenden Eigenschaften auf:

[0191]

Stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohle	T = 500°C	T = 920°C
N-Gehalt [Gew.-%]	4,11	6,49
flüchtige Bestandteile ("fB")	10,2	0,86
RD [g/l]	547	541
pH-Wert	8,5	9,9
V_{tot} [cm^3/g]	0,7397	0,7166
V_{mic} [cm^3/g]	0,5846	0,5870
Mesoporenanteil [%]	21,0	18,1
MP-BET [m^2/g]	1476	1453

Die obige Tabelle zeigt insbesondere, dass bei der höheren Temperaturen während der Stickstoffmodifizierung bzw. Stickstofffunktionalisierung eine Verringerung des Anteils an flüchtigen Bestandteilen ("fB") vorliegt, was einem Einbau bzw. einer Integration des Stickstoffs in das Kohlenstoffgerüst bzw. die Kohlenstoffmatrix der so behandelten Aktivkohle entspricht. Demgegenüber ist für niedrigere Temperaturen festzustellen, dass der Anteil an flüchtigen Bestandteilen höher ausfällt, was als Beleg für das Vorliegen des Stickstoffs in Form von stickstoffhaltigen funktionellen Gruppen zu werten ist.

Zudem zeigt eine Analyse der entsprechenden Wasserdampfisothermen (Fig. 3a), dass bei niedrigen Temperaturen (T = 500°C) die Funktionalisierung der behandelten Aktivkohle höher ist, was im Vergleich zu der bei höheren Temperaturen (T = 920°C) funktionalisierten Aktivkohle zu einer stärkeren Verschiebung der entsprechenden Adsorptions- bzw. Desorptionsäste zu geringen p/p_0-Werten führt. Zudem zeigt sich, wie in Fig. 3b dargestellt, dass die oxidierte Basis bzw. die an ihrer Oberfläche oxidierten Aktivkohlen im Hinblick auf die Parameter gemäß dem Gesamtporenvolumen, Mikroporenvolumen und der MP-BET-Oberfläche eine Erhöhung vorliegt, wobei diese Werte durch die nachfolgende Stickstofffunktionalisierung nur geringfügig verringert werden und immer noch oberhalb der Ausgangswerte auf Basis der eingesetzten Ausgangsaktivkohle liegen. Diesbezüglich kann auch auf obige Ausführungen zu den Figurendarstellungen gemäß Fig. 3a und Fig. 3b verwiesen werden.

Im Ergebnis zeigen die vorliegenden Untersuchungen, dass höhere Funktionalisierungstemperaturen zu einer vermehrten Integration bzw. Inkorporation von Stickstoff in das Kohlenstoffgerüst der so ausgerüsteten Aktivkohle führen, während niedrigere Temperaturen bei der Funktionalisierung zur Ausbildung stickstoff-

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haltiger funktioneller Gruppen führen. Zudem kann gezeigt werden, dass die erfindungsgemäße realisierte Verfahrensführung zu keinerlei negativen Beeinträchtigung der Produktparameter führt.

e) Wie zuvor angeführt, zeigt die Figurendarstellung gemäß Fig. 4a einen dem erfindungsgemäß durchgeführten Verfahren beispielhaft zugrundeliegenden Reaktionsablauf mit der Integration von Stickstoff in das Kohlenstoffgerüst. Eine Analyse auf Basis eines XPS-Spektrums zeigt, wie in Fig. 4b dargestellt, dass bei niedrigen Temperaturen im Rahmen der Stickstoffmodifizierung bzw. Stickstofffunktionalisierung vorrangig stickstoffhaltige funktionelle Gruppen auf Basis von Amiden bzw. Pyrrolen vorliegen, während mit zunehmender Temperatur eine Verschiebung zu stickstoffhaltigen funktionellen Gruppen auf Basis von quaternären Stickstoffverbindungen vorliegt. Bei einer weiterführenden Temperaturerhöhung nehmen die entsprechenden XPS-Signale der jeweiligen funktionellen Gruppen ab, was als Beleg für die Integration von Stickstoff in das Kohlenstoffgerüst bzw. in die Kohlenstoffmatrix der so behandelten Aktivkohle heranzuziehen ist. Für weitere Ausführungen kann auf die entsprechenden Ausführungen zu den Figurendarstellungen gemäß Fig. 4a und Fig. 4b verwiesen werden.

f) Im Rahmen der der Figurendarstellung gemäß Fig. 5 zugrundeliegenden Untersuchungen wurden die Durchbruchzeiten von drei verschiedenen erfindungsgemäßen Aktivkohlen für die Schadgase NH_3, H_2S und SO_2 im Vergleich zu einer unbehandelten Ausgangsaktivkohle ermittelt. In Bezug auf die Durchbruchzeiten für Ammoniak (NH_3) ergibt sich hierbei für die Ausgangsaktivkohle ein Wert von lediglich 14 min, während für die erfindungsgemäßen Aktivkohlen mit der Stickstoffmodifizierung bzw. Ausrüstung ein jeweiliger Wert von 20 min, 22 min und 33 min erhalten wird. In Bezug auf die Durchbruchzeit gegenüber Schwefelwasserstoff (H_2S) wird für das unbehandelte Ausgangsmaterial ein Wert von 80 min erreicht, während demgegenüber für die erfindungsgemäßen Aktivkohlen Werte von 120 min, 110 min bzw. nochmals 120 min erhalten werden. Schließlich wurde in Bezug auf die Durchbruchzeit gegenüber Schwefeldioxid (SO_2) für das unbehandelte Ausgangsmaterial ein Wert von 30 min ermittelt, während für die drei erfindungsgemäßen Aktivkohlen Werte von 78 min, 47 min sowie 58 min ermittelt wurde. Diesbezüglich kann auch auf obige Ausführungen zu Fig. 5 verwiesen werden.

Im Ergebnis zeigen die durchgeführten Untersuchungen, dass die erfindungsgemäßen Aktivkohlen im Vergleich zu dem Ausgangsmaterial signifikant erhöhte Durchbruchzeiten für die in Rede stehenden Schadstoffen aufweisen, so dass die erfindungsgemäßen Aktivkohlen gegenüber dem Ausgangsmaterial hinsichtlich ihrer Leistungsfähigkeit erheblich verbessert sind.

g) Diese Befunde werden auch durch die in der Figurendarstellung gemäß Fig. 6a und Fig. 6b in Bezug genommenen Untersuchungen bestätigt: So ist sowohl in Bezug auf Schwefeldioxid (SO_2) als auch in Bezug auf Schwefelwasserstoff (H_2S) jeweils eine signifikante Verbesserung des Durchbruchverhaltens im Vergleich zu einer unbehandelten Ausgangsaktivkohle zu beobachten. In Bezug auf die der Figurendarstellung gemäß Fig. 6a zugrundeliegenden Versuchsparameter wurde ein Schüttbett der zu untersuchenden Aktivkohle mit einer Betthöhe von 1,5 cm und einer Bettfläche von 1,3 cm^2 hergestellt, wobei das Schüttbett bei einer relativen Feuchte von 70% einem SO_2-Volumenstrom mit 1.000 ppm SO_2 bei 220 ml/min ausgesetzt wurde. Bei dem untersuchten Endprodukt handelt es sich um eine bei Temperaturen von 920°C stickstoffmodifizierte bzw. stickstofffunktionalisierte Aktivkohle nach der Erfindung. In Bezug auf Fig. 6b wurde die untersuchte Aktivkohle in Form eines Schüttbetts mit einer Betthöhe von 1 cm eingesetzt. Bei einer relativen Feuchtigkeit von 50% wurde das Aktivkohlebett einem H_2S-Volumenstrom mit 20 ppm H_2S bei 0,2 m/s ausgesetzt. Bei der eingesetzten Aktivkohle handelt es sich gleichermaßen um die bereits in Fig. 6a in Bezug genommene Aktivkohle.

Die zugrundeliegenden Untersuchungen zeigen somit, dass auf Basis der erfindungsgemäßen Stickstofffunktionalisierung eine signifikante Verbesserung hinsichtlich der Chemisorption der zuvor beschriebenen Schadgase resultiert, was sich in deutlich vergrößerten Durchbruchzeiten widerspiegelt.

h) Im Rahmen eines weiteren Untersuchungskomplexes wurden Strukturanalysen an einer im Rahmen des erfindungsgemäß durchgeführten Verfahrens zum Erhalt des Intermediats an ihrer Oberfläche oxidierten Aktivkohle durchgeführt. Wie bereits zuvor angeführt, werden durch die Oxidation unter Einbezug eines Oxidationsreagenzes ("OR") verschiedene sauerstoffhaltige funktionelle Gruppen bzw. Spezies generiert, welche nachfolgend gewissermaßen als Ankergruppen bzw. Reaktionspartner für die Anbindung mit dem Stickstoffmodifizierungsreagenz ("SMR") fungieren. Gleichermaßen beeinflussen die auf diese Weise hergestellten sauerstoffhaltigen funktionellen Gruppen die Oberflächeneigenschaften der so oxidierten Aktivkohle, insbesondere im Hinblick auf den pH-Wert; die Hydrophilie und dergleichen, was insbesondere auch zu einer Optimierung der nachfolgenden Interaktion mit dem Stickstoffmodifizierungsreagenz führt. Diesbezüglich kann auch auf obige Ausführungen zu den Figurendarstellungen gemäß Fig. 7a und Fig. 7b verwiesen werden.

i) Es wurden weiteren erfindungsgemäße Aktivkohlen unter Verwendung von Harnstofflösungen in der oben beschriebenen Verfahrensweise hergestellt, wobei die Harnstoffbehandlung mit nachfolgender Wärmebehandlung bei T = 900°C erfolgte. Die vorangehende Oberflächenoxidation erfolgte in Gegenwart von Luftsauerstoff. Die erhaltenen Aktivkohle wurden für Durchbruchmessungen an einem Schüttbett (Durchmes-

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ser: 50 mm; Höhe: 20 mm) für verschiedene Gase (H_2S und SO_2) getestet (Strom mit jeweils 1.000 ppm Schadgas; Strömungsgeschwindigkeit: 0,1 m/s; 70% relative Feuchte; Messtemperatur: 20°C; Durchbruch bei 10 ppm für H_2S und 5 ppm für SO_2). Als Vergleich dienten eine unbehandelte Aktivkohle (Vergleich I) und eine herkömmlich basisch imprägnierte Aktivkohle (Vergleich II). Für H_2S bzw. SO_2 konnte bei der erfindungsgemäßen Aktivkohle eine Durchbruchzeit von 122 min bzw. 133 min ermittelt werden, während für die Vergleichskohle I die Werte bei < 1 min bzw. 4 min und für die Vergleichskohle II bei 57 min bzw. 46 min lagen. Die erfindungsgemäßen Aktivkohlen weisen somit zwei- bis dreifach erhöhte Durchbruchzeiten im Vergleich zu herkömmlich imprägnierten Aktivkohlen auf; auch für Cyclohexan und Chlor werden sehr lange Durchbruchzeiten erhalten.

3. Vergleich einer erfindungsgemäßen Aktivkohle mit Stickstoffmodifizierung zu Vergleichsaktivkohlen:

[0192] Im Rahmen der zugrundeliegenden Untersuchungen wurde eine erfindungsgemäße Aktivkohle, wie sie auf Basis der Ausführungen zu der unter 1.) hergestellten erfindungsgemäßen Aktivkohle B erhalten wurde, mit Vergleichsaktivkohlen verglichen, welche auf unterschiedliche Art und Weise mit Stickstoff ausgerüstet wurden: So wurde eine erste Vergleichskohle I erhalten, indem ausgehend von entsprechenden Polymeren ein zweistufiger Prozess zur Herstellung der Aktivkohle auf Basis einer Carbonisierung mit nachfolgender Aktivierung durchgeführt wurde, wobei die Aktivierung in Gegenwart von Kohlendioxid, Wasserdampf und Ammoniakgas durchgeführt wurde; das stickstoffhaltige Mittel in Form von NH_3 wurde somit ihm Rahmen der Aktivkohleherstellung bei der Aktivierung zugegeben.

[0193] Des Weiteren wurde eine zweite Vergleichsaktivkohle II erhalten, indem eine fertige Aktivkohle (d. h. nach Carbonisierung und Aktivierung) als solche einer herkömmlichen Harnstoffimprägnierung unterzogen wurde, wobei diesbezüglich eine Harnstofflösung mit der Aktivkohle gemischt und die Aktivkohle auf diese Weise imprägniert bzw. getränkt wurde. Die so erhaltene Aktivkohle wurde anschließend getrocknet. Schließlich wurde eine Vergleichsaktivkohle III erhalten, indem eine Aktivkohle ohne zwischengeschaltete Oxidation direkt einer Stickstoffmodifizierung unterzogen wurde, indem eine Harnstofflösung mit der Aktivkohle in Kontakt gebracht wurde und die auf diese Weise behandelte Aktivkohle einer nachfolgenden Wärmebehandlung unterzogen wurde. Die Vergleichsaktivkohle III unterscheidet sich somit von der erfindungsgemäßen Aktivkohle dadurch, dass eine Aktivkohle als solche und somit keine an ihrer Oberfläche oxidierte Aktivkohle einer nachfolgenden Behandlung zur Stickstoffmodifizierung unterzogen wurde. Die entsprechende Aktivkohle wies somit in Bezug auf das Stickstoffmodifizierungsreagenz keine Ankergruppen auf.

[0194] An den so erhaltenen Untersuchungen wurden entsprechende Analysen des Stickstoffgehalts und weiterführende Untersuchungen in Bezug auf das Durchbruchverhalten von Schwefelwasserstoff (H_2S) durchgeführt. Bezüglich der Analyse des Stickstoffgehalts und des Vorliegens von Stickstoff in der Aktivkohle kann auf obige Ausführungen verwiesen werden. Die Durchbruchexperimente wurden auf Basis einer Schüttung der jeweiligen Aktivkohle mit einer Höhe von 2 cm in einem konstanten Volumenstrom bei einer Temperatur von T = 30°C durchgeführt, wobei der mit einer konstanten Strömungsgeschwindigkeit durch die Schüttung geführte Volumenstrom mit 20 ppm H_2S versehen wurde. Die nachfolgende Tabelle zeigt die erhaltenen Ergebnisse.

Aktivkohle	B (Erf.)	I (Vgl.)	II (Vgl.)	III (Vgl.)
Stickstoffgehalt N-Gehalt [Gew.-%]	18	1	4	8
Gehalt an Stickstoff nicht in Form von flüchtigen Bestandteilen (Gew.-%)	95	≤ 0,1	≤ 0,1	≤ 0,5
Durchbruchszeit gegenüber H_2S [min]	≥ 100	54,5	62,5	81

[0195] Die Tabelle zeigt, dass in Bezug auf die erfindungsgemäße Aktivkohle B sowohl der Gehalt an in das Gerüst gebundenem (d. h. nicht in Form von flüchtigen Bestandteilen vorliegendem) Stickstoff in der Aktivkohle erhöht ist, wobei im Gegensatz zu den Vergleichsaktivkohlen ein erheblich größerer Anteil des Stickstoffs in Form von nichtflüchtigen Stickstoffbestandteilen vorliegt, was als Maß für die Integration des Stickstoffs in die Aktivkohlematrix bzw. das Aktivkohlegerüst zu verstehen ist.

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[0196] Gleichermaßen weist die erfindungsgemäße Aktivkohle B ein deutlich verbessertes Durchbruchverhalten gegenüber Schwefelwasserstoff und somit eine signifikant verbesserte Leistungsfähigkeit im Vergleich zu den Vergleichsaktivkohlen I bis III auf.

[0197] Im Rahmen einer weiterführenden Untersuchung wurden die zugrundeliegenden Aktivkohlen, nämlich die erfindungsgemäße Aktivkohle B und die Vergleichsaktivkohlen I bis III, einem Waschvorgang unter Verwendung von 1 N Salzsäure für eine Zeitdauer von einer Stunde unterzogen. Anschließend wurden die Aktivkohlen neutralgewaschen und nachfolgend getrocknet. Mit den so behandelten Aktivkohlen wurden erneut Durchbruchmessungen in Bezug auf Schwefelwasserstoff (H_2S), wie zuvor beschrieben, durchgeführt. Während für die erfindungsgemäße Aktivkohle B eine Durchbruchzeit von weiterhin mehr als 100 min ermittelt werden konnte, betrugen die entsprechenden Durchbruchzeiten für die Vergleichsaktivkohlen I bis III 15 min, 18 min bzw. 25 min. Dies ist als Beleg dafür zu werten, dass aufgrund des Waschvorgangs mit Salzsäure hinsichtlich der Vergleichsaktivkohle I bis III eine erhebliche Menge an Stickstoff entfernt wurde, was für die erfindungsgemäße Aktivkohle B nicht der Fall ist. Dies bedeutet, dass in Bezug auf die Vergleichsaktivkohlen I bis III der Stickstoff zumindest im Wesentlichen nicht in immobilisierter Form vorliegt und somit leicht entfernt werden kann. Im Gegensatz hierzu ist aufgrund der Inkorporation des Stickstoffs in das Kohlenstoffgerüst gemäß Aktivkohle B der Stickstoff immobilisiert bzw. fest verankert, was zu der hohen Beständigkeit auch gegenüber Säuren führt.

[0198] Im Ergebnis zeigen die durchgeführten Untersuchungen die hohe Leistungsfähigkeit und die positiven Eigenschaften der auf Basis des erfindungsgemäß durchgeführten Verfahrens erhältlichen stickstoffmodifizierten bzw. stickstofffunktionalisierten Aktivkohlen.

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ZITATE ENTHALTEN IN DER BESCHREIBUNG

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Schutzansprüche

1. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle, insbesondere mit katalytischen und/oder reaktiven Eigenschaften, vorzugsweise Aktivkohle mit Stickstoff als katalytisch aktivem Heteroatom, erhältlich durch ein Verfahren, wobei bei dem Verfahren eine als Ausgangsmaterial eingesetzte Aktivkohle ("Ausgangsaktivkohle") zunächst einer Oberflächenoxidation unter Verwendung und/oder in Gegenwart mindestens eines Oxidationsreagenzes ("OR") unterzogen wird und die auf diese Weise erhaltene, an ihrer Oberfläche oxidierte Aktivkohle nachfolgend einer Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, mit Stickstoff, insbesondere mit stickstoffhaltigen funktionellen Gruppen, unter Verwendung und/oder in Gegenwart mindestens eines Stickstoffmodifizierungsreagenzes ("SMR") unterzogen wird.

2. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach Anspruch 1, dadurch gekennzeichnet, dass die Oberflächenoxidation unter Ausbildung einer hydrophilen Oberfläche der Aktivkohle durchgeführt wird und/oder dass die Oberflächenoxidation unter Ausbildung von sauerstoffhaltigen funktionellen Gruppen an der Oberfläche der Aktivkohle durchgeführt wird und/oder dass die Oberflächenoxidation zu einer Ausbildung von sauerstoffhaltigen funktionellen Gruppen an der Oberfläche der Aktivkohle führt, insbesondere wobei die sauerstoffhaltigen funktionellen Gruppen ausgewählt werden aus sauren und basischen sauerstoffhaltigen funktionellen Gruppen sowie deren Kombinationen, insbesondere sauren und basischen Oberflächenoxiden, und/oder insbesondere wobei die sauerstoffhaltigen funktionellen Gruppen ausgewählt werden aus Hydroxyl-, Carboxyl-, Carbonyl-, Anhydrid-, Lacton-, Chinon-, Pyron-, Chromen- und Ethergruppen sowie deren Kombinationen.

3. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach Anspruch 1 oder 2, dadurch gekennzeichnet,
dass durch die Oberflächenoxidation der pH-Wert, insbesondere der Oberflächen-pH-Wert, der an ihrer Oberfläche oxidierten Aktivkohle im Vergleich zu der Ausgangsaktivkohle um einen Wert von mindestens 0,25, insbesondere mindestens 0,5, vorzugsweise mindestens 0,75, bevorzugt mindestens 1, besonders bevorzugt mindestens 1,5, verändert wird; und/oder
dass die Acidität der an ihrer Oberfläche oxidierten Aktivkohle erhöht und/oder der pH-Wert, insbesondere der Oberflächen-pH-Wert, der an ihrer Oberfläche oxidierten Aktivkohle verringert wird, insbesondere um einen Wert von mindestens 0,25, insbesondere mindestens 0,5, vorzugsweise mindestens 0,75, bevorzugt mindestens 1, besonders bevorzugt mindestens 1,5, oder aber dass die Basizität der an ihrer Oberfläche oxidierten Aktivkohle erhöht und/oder der pH-Wert, insbesondere der Oberflächen-pH-Wert, der an ihrer Oberfläche oxidierten Aktivkohle erhöht wird, insbesondere um einen Wert von mindestens 0,25, insbesondere mindestens 0,5, vorzugsweise mindestens 0,75, bevorzugt mindestens 1, besonders bevorzugt mindestens 1,5.

4. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass die Oberflächenoxidation durchgeführt wird derart, dass die auf diese Weise erhaltene, an ihrer Oberfläche oxidierte Aktivkohle einen Gehalt an sauerstoffhaltigen funktionellen Gruppen, berechnet und/oder ausgedrückt als Gehalt an flüchtigen Bestandteilen ("fB") und bezogen auf das Trockengewicht der an ihrer Oberfläche oxidierten Aktivkohle, von mindestens 1 Gew.-%, insbesondere mindestens 2 Gew.-%, vorzugsweise mindestens 3 Gew.-%, bevorzugt mindestens 4 Gew.-%, und/oder im Bereich von 1 Gew.-% bis 30 Gew.-%, insbesondere 1,5 Gew.-% bis 25 Gew.-%, vorzugsweise 2 Gew.-% bis 20 Gew.-%, bevorzugt 3 Gew.-% bis 15 Gew.-%, aufweist, insbesondere wobei der Gehalt an sauerstoffhaltigen funktionellen Gruppen durch Temperatur und/oder Zeitdauer und/oder Art und/oder Konzentration an Oxidationsreagenz ("OR") eingestellt wird.

5. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass das Oxidationsreagenz ("OR") ausgewählt wird aus der Gruppe von Sauerstoff, Ozon, anorganischen oder organischen Oxiden und Peroxiden sowie anorganischen oder organischen Säuren und Persäuren und deren Kombinationen, bevorzugt aus der Gruppe von Sauerstoff, Wasserstoffperoxid (H_2O_2), Salpetersäure (HNO_3) und Ammoniumperoxodisulfat ($(NH_4)_2S_2O_8$) sowie deren Kombinationen.

6. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass die Oberflächenoxidation unter Erwärmen erfolgt, insbesondere wobei die Oberflächenoxidation unter solchen Temperaturen erfolgt, dass eine Reaktion des Oxidationsreagenzes ("OR") mit der Aktivkohle unter Ausbildung von sauerstoffhaltigen funktionellen Gruppen an der Oberfläche der Aktivkohle erfolgt, und/oder insbesondere wobei die Oberflächenoxidation im Temperaturbereich von –20°C bis 1.000°C, insbesondere 0°C bis 800°C, vorzugsweise 20°C bis 600°C, bevorzugt 30°C bis 500°C,

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durchgeführt wird und/oder insbesondere wobei die Oberflächenoxidation für eine Zeitdauer im Bereich von 1 Minute bis 600 Minuten, insbesondere 5 Minuten bis 500 Minuten, bevorzugt 10 Minuten bis 400 Minuten, durchgeführt wird.

7. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass das Oxidationsreagenz ("OR") ausgewählt wird aus der Gruppe von anorganischen oder organischen Oxiden und Peroxiden sowie anorganischen oder organischen Säuren und Persäuren und deren Kombinationen, bevorzugt aus der Gruppe von Wasserstoffperoxid (H_2O_2) und Salpetersäure (HNO_3), vorzugsweise Salpetersäure (HNO_3), insbesondere wobei die Oberflächenoxidation im Temperaturbereich von –20°C bis 500°C, insbesondere –10°C bis 400°C, vorzugsweise 0°C bis 350°C, bevorzugt 10°C bis 300°C, besonders bevorzugt 15°C bis 250°C, ganz besonders bevorzugt 20°C bis 200°C durchgeführt wird.

8. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass das Oxidationsreagenz ("OR") ausgewählt wird aus der Gruppe von Sauerstoff und Ozon, vorzugsweise Sauerstoff, insbesondere wobei die Oberflächenoxidation im Temperaturbereich von 100°C bis 1.000°C, insbesondere 150°C bis 900°C, vorzugsweise 200°C bis 750°C, bevorzugt 225°C bis 600°C, besonders bevorzugt 250°C bis 500°C, ganz besonders bevorzugt 250°C bis 450°C, durchgeführt wird.

9. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass im Anschluss an die Oberflächenoxidation und insbesondere vor der Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, mit Stickstoff eine Aufreinigung und/oder Trocknung der an ihrer Oberfläche oxidierten Aktivkohle erfolgt, insbesondere wobei die Aufreinigung mittels mindestens eines Waschvorgangs in einer Flüssigkeit, insbesondere Wasser, erfolgt und/oder insbesondere wobei die Trocknung unter Erwärmung der an ihrer Oberfläche oxidierten Aktivkohle, insbesondere auf Temperaturen im Bereich von 40°C bis 200°C, insbesondere 50°C bis 150°C, vorzugsweise 60°C bis 120°C, erfolgt.

10. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, mit Stickstoff ein Inkontaktbringen der nach der Oberflächenoxidation erhaltenen Aktivkohle und/oder der an ihrer Oberfläche oxidierten Aktivkohle mit dem Stickstoffmodifizierungreagenz ("SMR"), gefolgt von einer thermischen Behandlung, umfasst.

11. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass das für die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff eingesetzte Stickstoffmodifizierungsreagenz ("SMR") ausgewählt wird aus der Gruppe von Harnstoff, Ammoniak, Melamin und Aminosäuren sowie deren Kombinationen, insbesondere Harnstoff und Ammoniak sowie deren Kombinationen, vorzugsweise Harnstoff.

12. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass als das für die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff eingesetzte Stickstoffmodifizierungsreagenz ("SMR") Harnstoff verwendet wird, insbesondere wobei der Harnstoff in Form einer vorzugsweise wässrigen Lösung, bevorzugt in Form einer zumindest im Wesentlichen gesättigten Harnstofflösung, eingesetzt wird.

13. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass als das für die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff eingesetzte Stickstoffmodifizierungsreagenz ("SMR") Ammoniak verwendet wird, insbesondere wobei das Ammoniak in Form des Gases und/oder als Ammoniakwasser, insbesondere in Form des Gases, eingesetzt wird.

14. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff durch Reaktion, insbesondere chemische Reaktion, vorzugsweise unter Ausbildung kovalenter Bindungen, des Stickstoffmodifizierungsreagenzes ("SMR") mit der an ihrer Oberfläche oxidierten Aktivkohle, insbesondere mit den sauerstoffhaltigen funktionellen Gruppen an der Oberfläche der Aktivkohle, erfolgt.

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15. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff unter Ausbildung stickstoffhaltiger funktioneller Gruppen, insbesondere ausgewählt aus der Gruppe von Aminen, insbesondere primären, sekundären, tertiären und quartären Aminen, Iminen und Azinen sowie deren Kombinationen und/oder insbesondere ausgewählt aus der Gruppe von Amin-, Imin-, Azin-, Pyrrol-, Nitril-, Nitro-, Nitroso-, Pyridin-, Imid-, Amid-, Lactam- und Pyridongruppen sowie deren Kombinationen, erfolgt.

16. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff unter Integration und/oder Einbau, insbesondere unter Ausbildung chemischer Bindungen, vorzugsweise unter Ausbildung kovalenter chemischer Bindungen, insbesondere zwischen Stickstoff einerseits und Kohlenstoff andererseits, in das Kohlenstoffgerüst und/oder die Kohlenstoffmatrix der an ihrer Oberfläche oxidierten Aktivkohle erfolgt.

17. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff unter Erwärmen erfolgt, insbesondere auf Temperaturen oberhalb der Zersetzungstemperatur des Stickstoffmodifizierungsreagenzes ("SMR"), insbesondere wobei die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff unter solchen Temperaturen erfolgt, dass eine Reaktion des Stickstoffmodifizierungsreagenzes ("SMR") mit der an ihrer Oberfläche oxidierten Aktivkohle, insbesondere mit den sauerstoffhaltigen funktionellen Gruppen an der Oberfläche der Aktivkohle, erfolgt, und/oder insbesondere wobei die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff im Temperaturbereich von 200°C bis 1.200°C, insbesondere 300°C bis 1.000°C, vorzugsweise 500°C bis 950°C, durchgeführt wird und/oder insbesondere wobei die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff für eine Zeitdauer im Bereich von 0,1 Stunden bis 15 Stunden, insbesondere 0,2 Stunden bis 10 Stunden, vorzugsweise 0,5 Stunden bis 8 Stunden, durchgeführt wird.

18. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff unter Erwärmen in Form einer einstufigen Wärmebehandlung erfolgt.

19. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff unter Erwärmen auf einen Temperaturbereich von 300°C bis 700°C, insbesondere 400°C bis 600°C, vorzugsweise 400°C bis 550°C, durchgeführt wird, insbesondere für eine Zeitdauer im Bereich von 0,1 Stunden bis 15 Stunden, bevorzugt 0,2 Stunden bis 10 Stunden, vorzugsweise 0,5 Stunden bis 8 Stunden, insbesondere wobei stickstoffhaltige funktionelle Gruppen, wie in Anspruch 15 definiert, ausgebildet werden.

20. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff unter Erwärmen auf einen Temperaturbereich von 750°C bis 1.200°C, insbesondere 800°C bis 1.100°C, vorzugsweise 850°C bis 1.050°C, durchgeführt wird, insbesondere für eine Zeitdauer im Bereich von 0,1 Stunden bis 15 Stunden, bevorzugt 0,2 Stunden bis 10 Stunden, vorzugsweise 0,5 Stunden bis 8 Stunden, insbesondere wobei eine Integration und/oder ein Einbau des Stickstoffs in das Kohlenstoffgerüst und/oder die Kohlenstoffmatrix der an ihrer Oberfläche oxidierten Aktivkohle erfolgt, insbesondere wie in Anspruch 16 definiert.

21. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff unter Erwärmen in Form einer mehrstufigen, insbesondere zweistufigen Wärmebehandlung, erfolgt.

22. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, der an ihrer Oberfläche oxidierten Aktivkohle mit Stickstoff in einer ersten Wärmebehandlung unter Er-

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wärmen auf einen Temperaturbereich von 300°C bis 700°C, insbesondere 400°C bis 600°C, vorzugsweise 400°C bis 550°C, und einer nachfolgenden zweiten Wärmebehandlung unter Erwärmen auf einen Temperaturbereich von 750°C bis 1.200°C, insbesondere 800°C bis 1.100°C, vorzugsweise 850°C bis 1.050°C, durchgeführt wird, insbesondere wobei die jeweiligen Wärmebehandlungen, unabhängig voneinander, für eine Zeitdauer im Bereich von 0,1 Stunden bis 15 Stunden, insbesondere 0,2 Stunden bis 10 Stunden, vorzugsweise 0,5 Stunden bis 8 Stunden, durchgeführt werden.

23. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass im Anschluss an die Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, mit Stickstoff eine Aufreinigung und/oder Trocknung der erhaltenen Aktivkohle erfolgt, insbesondere wobei die Aufreinigung mittels mindestens eines Waschvorgangs in einer Flüssigkeit, insbesondere Wasser, erfolgt und/oder insbesondere wobei die Trocknung unter Erwärmung der mit Stickstoff ausgerüsteten Aktivkohle, insbesondere auf Temperaturen im Bereich von 40°C bis 200°C, insbesondere 50°C bis 150°C, vorzugsweise 60°C bis 120°C, erfolgt.

24. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass die mit Stickstoff ausgerüstete Aktivkohle einen Gehalt an flüchtigen Bestandteilen ("fB"), bezogen auf das Trockengewicht der mit Stickstoff ausgerüsteten Aktivkohle, von mindestens 0,1 Gew.-%, insbesondere mindestens 0,2 Gew.-%, vorzugsweise mindestens 0,3 Gew.-%, bevorzugt mindestens 0,5 Gew.-%, besonders bevorzugt mindestens 1 Gew.-%, ganz besonders bevorzugt mindestens 2 Gew.-%, und/oder im Bereich von 0,1 Gew.-% bis 20 Gew.-%, insbesondere 0,2 Gew.-% bis 15 Gew.-%, vorzugsweise 0,3 Gew.-% bis 12 Gew.-%, bevorzugt 0,5 Gew.-% bis 10 Gew.-%, besonders bevorzugt 1 Gew.-% bis 10 Gew.-%, aufweist.

25. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass die mit Stickstoff ausgerüstete Aktivkohle einen Stickstoffgehalt, bezogen auf das Trockengewicht der mit Stickstoff ausgerüsteten Aktivkohle, von mindestens 0,1 Gew.-%, insbesondere mindestens 0,2 Gew.-%, vorzugsweise mindestens 0,3 Gew.-%, bevorzugt mindestens 0,5 Gew.-%, besonders bevorzugt mindestens 1 Gew.-%, ganz besonders bevorzugt mindestens 2 Gew.-%, und/oder im Bereich von 0,1 Gew.-% bis 30 Gew.-%, insbesondere 0,2 Gew.-% bis 25 Gew.-%, vorzugsweise 0,3 Gew.-% bis 20 Gew.-%, bevorzugt 0,5 Gew.-% bis 15 Gew.-%, besonders bevorzugt 1 Gew.-% bis 15 Gew.-%, aufweist, insbesondere wobei mindestens 50%, insbesondere mindestens 60%, vorzugsweise mindestens 70%, bevorzugt mindestens 80%, des Stickstoffs, bezogen auf den Gesamtstickstoffgehalt, in der mit Stickstoff ausgerüsteten Aktivkohle chemisch gebunden und/oder integriert vorliegen und/oder nicht in Form von flüchtigen Bestandteilen ("fB") in der mit Stickstoff ausgerüsteten Aktivkohle vorliegen und/oder insbesondere wobei 50% bis 99,9%, insbesondere 60% bis 99%, vorzugsweise 70% bis 95%, bevorzugt 80% bis 90%, des Stickstoffs, bezogen auf den Gesamtstickstoffgehalt, in der mit Stickstoff ausgerüsteten Aktivkohle chemisch gebunden und/oder integriert vorliegen und/oder nicht in Form von flüchtigen Bestandteilen ("fB") in der mit Stickstoff ausgerüsteten Aktivkohle vorliegen.

26. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass die mit Stickstoff ausgerüstete Aktivkohle ein Gesamtporenvolumen, insbesondere ein Gesamtporenvolumen nach Gurvich, im Bereich von 0,4 bis 4 cm^3/g, insbesondere 0,5 bis 3,5 cm^3/g, vorzugsweise 0,6 bis 3 cm^3/g, besonders bevorzugt 0,7 bis 2,5 cm^3/g, aufweist, insbesondere wobei 30% bis 99%, insbesondere 40% bis 99%, vorzugsweise 50% bis 95%, des Gesamtporenvolumens, insbesondere des Gesamtporenvolumens nach Gurvich, der mit Stickstoff ausgerüsteten Aktivkohle, durch Poren mit Porendurchmessern von ≤ 50 nm, insbesondere durch Mikro- und/oder Mesoporen, gebildet werden und/oder insbesondere wobei 10% bis 85%, insbesondere 20% bis 80%, vorzugsweise 30% bis 75%, des Gesamtporenvolumens, insbesondere des Gesamtporenvolumens nach Gurvich, der mit Stickstoff ausgerüsteten Aktivkohle, durch Poren mit Porendurchmessern im Bereich von 2 nm bis 50 nm, insbesondere durch Mesoporen, gebildet werden; und/oder
dass die mit Stickstoff ausgerüstete Aktivkohle ein durch Poren mit Porendurchmessern ≤ 2 nm gebildetes Porenvolumen, insbesondere Mikroporenvolumen nach Carbon Black, im Bereich von 0,05 bis 2 cm^3/g, insbesondere 0,1 bis 1,5 cm^3/g, vorzugsweise 0,2 bis 1,1 cm^3/g, besonders bevorzugt 0,3 bis 1 cm^3/g, aufweist und/oder dass 10% bis 95%, insbesondere 20% bis 95%, vorzugsweise 30% bis 90%, des Gesamtporenvolumens der mit Stickstoff ausgerüsteten Aktivkohle durch Poren mit Porendurchmessern von ≤ 2 nm, insbesondere durch Mikroporen, gebildet werden und/oder
dass die mit Stickstoff ausgerüstete Aktivkohle einen mittleren Porendurchmesser im Bereich von 1 bis 60 nm, insbesondere 1 bis 55 nm, bevorzugt 1,5 bis 50 nm, besonders bevorzugt 2 bis 45 nm, aufweist.

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27. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass als eingesetzte Aktivkohle ("Ausgangsaktivkohle") kornförmige, insbesondere kugelförmige Aktivkohle verwendet wird und/oder dass als eingesetzte Aktivkohle ("Ausgangsaktivkohle") eine derartige Aktivkohle verwendet wird, welche durch Carbonisierung eines Ausgangsmaterials, insbesondere von sulfonierten organischen Polymeren, vorzugsweise auf Basis von divinylbenzolvernetztem Polystyrol, und anschließende Aktivierung des carbonisierten Ausgangsmaterials erhalten wird.

28. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet,
dass die mit Stickstoff ausgerüstete Aktivkohle eine spezifische BET-Oberfläche im Bereich von 500 m^2/g bis 3.500 m^2/g, insbesondere 600 bis 3.000 m^2/g, vorzugsweise 700 bis 2.750 m^2/g, besonders bevorzugt 800 bis 2.500 m^2/g, aufweist und/oder
dass die mit Stickstoff ausgerüstete Aktivkohle eine durch Mikroporen gebildete Oberfläche von 400 bis 2.500 m^2/g, insbesondere 500 bis 2.300 m^2/g, vorzugsweise 600 bis 2.100 m^2/g, besonders bevorzugt 700 bis 1.900 m^2/g, aufweist und/oder
dass die mit Stickstoff ausgerüstete Aktivkohle eine Dichte von 1,1 bis 3,5 g/ml, insbesondere von 1,5 bis 3 g/ml, vorzugsweise von 1,75 bis 2,75 g/ml, bevorzugt von 2 bis 2,5 g/ml, aufweist und/oder
dass die mit Stickstoff ausgerüstete Aktivkohle eine Schüttdichte von 0,1 bis 1,5 g/ml, insbesondere von 0,15 bis 1 g/ml, vorzugsweise von 0,2 bis 0,8 g/ml, bevorzugt von 0,3 bis 0,6 g/ml, aufweist und/oder
dass die mit Stickstoff ausgerüstete Aktivkohle eine Druck- und/oder Berstfestigkeit (Gewichtsbelastbarkeit) pro Aktivkohlekorn, insbesondere pro Aktivkohlekügelchen, von mindestens 5 Newton, insbesondere mindestens 10 Newton, vorzugsweise mindestens 15 Newton, bevorzugt mindestens 20 Newton, aufweist und/oder dass die Aktivkohle eine Druck- und/oder Berstfestigkeit (Gewichtsbelastbarkeit) pro Aktivkohlekorn, insbesondere pro Aktivkohlekügelchen, im Bereich von 10 bis 50 Newton, insbesondere 10 bis 45 Newton, vorzugsweise 15 bis 40 Newton, aufweist.

29. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle, insbesondere mit katalytischen und/oder reaktiven Eigenschaften, vorzugsweise mit Stickstoff als katalytisch aktivem Heteroatom, wobei die stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle durch Oberflächenoxidation einer als Ausgangsmaterial eingesetzten Aktivkohle ("Ausgangsaktivkohle") unter Verwendung und/oder in Gegenwart mindestens eines Oxidationsreagenzes ("OR") und nachfolgender Ausrüstung, insbesondere Modifizierung und/oder Funktionalisierung, mit Stickstoff der auf diese Weise erhaltenen, an ihrer Oberfläche oxidierten Aktivkohle unter Verwendung und/oder in Gegenwart mindestens eines Stickstoffmodifizierungsreagenzes ("SMR") erhältlich ist.

30. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle, insbesondere mit katalytischen und/oder reaktiven Eigenschaften, vorzugsweise mit Stickstoff als katalytisch aktivem Heteroatom, dadurch gekennzeichnet, dass Stickstoff unter Ausbildung chemischer Bindungen, vorzugsweise unter Ausbildung kovalenter chemischer Bindungen, an die Aktivkohle gebunden ist.

31. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach Anspruch 30, dadurch gekennzeichnet, dass Stickstoff in Form mindestens einer stickstoffhaltigen funktionellen Gruppe, insbesondere ausgewählt aus der Gruppe von Aminen, insbesondere primären, sekundären, tertiären und quartären Aminen, Iminen und Azinen sowie deren Kombinationen und/oder insbesondere ausgewählt aus der Gruppe von Amin-, Imin-, Azin-, Pyrrol-, Nitril-, Nitro-, Nitroso-, Pyridin-, Imid-, Amid-, Lactam- und Pyridongruppen sowie deren Kombinationen, vorliegt, insbesondere an die stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle gebunden ist, vorzugsweise chemisch und/oder kovalent gebunden ist.

32. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach Anspruch 30 oder 31, dadurch gekennzeichnet, dass Stickstoff in das Kohlenstoffgerüst und/oder die Kohlenstoffmatrix der stickstoffmodifizierten und/oder stickstofffunktionalisierten Aktivkohle, vorzugsweise unter Ausbildung kovalenter chemischer Bindungen, insbesondere zwischen Stickstoff einerseits und Kohlenstoff andererseits, integriert und/oder eingebaut ist.

33. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der Ansprüche 30 bis 32, dadurch gekennzeichnet, dass die stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle einen Gehalt an flüchtigen Bestandteilen ("fB"), bezogen auf das Trockengewicht der mit Stickstoff ausgerüsteten Aktivkohle, von mindestens 0,1 Gew.-%, insbesondere mindestens 0,2 Gew.-%, vorzugsweise mindestens 0,3 Gew.-%, bevorzugt mindestens 0,5 Gew.-%, besonders bevorzugt mindestens 1 Gew.-%, ganz besonders bevorzugt mindestens 2 Gew.-%, und/oder im Bereich von 0,1 Gew.-% bis 20 Gew.-%, insbesondere 0,2 Gew.-

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% bis 15 Gew.-%, vorzugsweise 0,3 Gew.-% bis 12 Gew.-%, bevorzugt 0,5 Gew.-% bis 10 Gew.-%, besonders bevorzugt 1 Gew.-% bis 10 Gew.-%, aufweist.

34. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der Ansprüche 30 bis 33, dadurch gekennzeichnet, dass die stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle einen Stickstoffgehalt, bezogen auf das Trockengewicht der mit Stickstoff ausgerüsteten Aktivkohle, von mindestens 0,1 Gew.-%, insbesondere mindestens 0,2 Gew.-%, vorzugsweise mindestens 0,3 Gew.-%, bevorzugt mindestens 0,5 Gew.-%, besonders bevorzugt mindestens 1 Gew.-%, ganz besonders bevorzugt mindestens 2 Gew.-%, und/oder im Bereich von 0,1 Gew.-% bis 30 Gew.-%, insbesondere 0,2 Gew.-% bis 25 Gew.-%, vorzugsweise 0,3 Gew.-% bis 20 Gew.-%, bevorzugt 0,5 Gew.-% bis 15 Gew.-%, besonders bevorzugt 1 Gew.-% bis 15 Gew.-%, aufweist, insbesondere wobei mindestens 50%, insbesondere mindestens 60%, vorzugsweise mindestens 70%, bevorzugt mindestens 80%, des Stickstoffs, bezogen auf den Gesamtstickstoffgehalt, in der stickstoffmodifizierten und/oder stickstofffunktionalisierten Aktivkohle chemisch gebunden und/oder integriert vorliegen und/oder nicht in Form von flüchtigen Bestandteilen ("fB") in der stickstoffmodifizierten und/oder stickstofffunktionalisierten Aktivkohle vorliegen und/oder insbesondere wobei 50% bis 99,9%, insbesondere 60% bis 99%, vorzugsweise 70% bis 95%, bevorzugt 80% bis 90%, des Stickstoffs, bezogen auf den Gesamtstickstoffgehalt, in der stickstoffmodifizierten und/oder stickstofffunktionalisierten Aktivkohle chemisch gebunden und/oder integriert vorliegen und/oder nicht in Form von flüchtigen Bestandteilen ("fB") in der stickstoffmodifizierten und/oder stickstofffunktionalisierten Aktivkohle vorliegen.

35. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der Ansprüche 30 bis 34, dadurch gekennzeichnet,
dass die stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle ein Gesamtporenvolumen, insbesondere ein Gesamtporenvolumen nach Gurvich, im Bereich von 0,4 bis 4 cm^3/g, insbesondere 0,5 bis 3,5 cm^3/g, vorzugsweise 0,6 bis 3 cm^3/g, besonders bevorzugt 0,7 bis 2,5 cm^3/g, aufweist, insbesondere wobei 30% bis 99%, insbesondere 40% bis 99%, vorzugsweise 50% bis 95%, des Gesamtporenvolumens, insbesondere des Gesamtporenvolumens nach Gurvich, der stickstoffmodifizierten und/oder stickstofffunktionalisierten Aktivkohle, durch Poren mit Porendurchmessern von ≤ 50 nm, insbesondere durch Mikro- und/oder Mesoporen, gebildet werden und/oder insbesondere wobei 10% bis 85%, insbesondere 20% bis 80%, vorzugsweise 30% bis 75%, des Gesamtporenvolumens, insbesondere des Gesamtporenvolumens nach Gurvich, der stickstoffmodifizierten und/oder stickstofffunktionalisierten Aktivkohle, durch Poren mit Porendurchmessern im Bereich von 2 nm bis 50 nm, insbesondere durch Mesoporen, gebildet sind; und/oder
dass die stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle ein durch Poren mit Porendurchmessern ≤ 2 nm gebildetes Porenvolumen, insbesondere Mikroporenvolumen nach Carbon Black, im Bereich von 0,05 bis 2 cm^3/g, insbesondere 0,1 bis 1,5 cm^3/g, vorzugsweise 0,2 bis 1,1 cm^3/g, besonders bevorzugt 0,3 bis 1 cm^3/g, aufweist und/oder dass 10% bis 95%, insbesondere 20% bis 95%, vorzugsweise 30% bis 90%, des Gesamtporenvolumens der stickstoffmodifizierten und/oder stickstofffunktionalisierten Aktivkohle durch Poren mit Porendurchmessern von ≤ 2 nm, insbesondere durch Mikroporen, gebildet sind und/oder
dass die stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle einen mittleren Porendurchmesser im Bereich von 1 bis 60 nm, insbesondere 1 bis 55 nm, bevorzugt 1,5 bis 50 nm, besonders bevorzugt 2 bis 45 nm, aufweist.

36. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der Ansprüche 30 bis 35, dadurch gekennzeichnet,
dass die stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle eine spezifische BET-Oberfläche im Bereich von 500 m^2/g bis 3.500 m^2/g, insbesondere 600 bis 3.000 m^2/g, vorzugsweise 700 bis 2.750 m^2/g, besonders bevorzugt 800 bis 2.500 m^2/g, aufweist und/oder dass die stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle eine durch Mikroporen gebildete Oberfläche von 400 bis 2.500 m^2/g, insbesondere 500 bis 2.300 m^2/g, vorzugsweise 600 bis 2.100 m^2/g, besonders bevorzugt 700 bis 1.900 m^2/g, aufweist und/oder
dass die stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle eine Dichte von 1,1 bis 3,5 g/ml, insbesondere von 1,5 bis 3 g/ml, vorzugsweise von 1,75 bis 2,75 g/ml, bevorzugt von 2 bis 2,5 g/ml, aufweist und/oder
dass die stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle eine Schüttdichte von 0,1 bis 1,5 g/ml, insbesondere von 0,15 bis 1 g/ml, vorzugsweise von 0,2 bis 0,8 g/ml, bevorzugt von 0,3 bis 0,6 g/ml, aufweist und/oder
dass die stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle eine Druck- und/oder Berstfestigkeit (Gewichtsbelastbarkeit) pro Aktivkohlekorn, insbesondere pro Aktivkohlekügelchen, von mindestens 5 Newton, insbesondere mindestens 10 Newton, vorzugsweise mindestens 15 Newton, bevorzugt mindestens 20 Newton, aufweist und/oder dass die Aktivkohle eine Druck- und/oder Berstfestigkeit (Gewichtsbelastbarkeit)

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pro Aktivkohlekorn, insbesondere pro Aktivkohlekügelchen, im Bereich von 10 bis 50 Newton, insbesondere 10 bis 45 Newton, vorzugsweise 15 bis 40 Newton, aufweist.

37. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der Ansprüche 30 bis 36, dadurch gekennzeichnet, dass die stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle selbsttragend ist und/oder in Form einer insbesondere losen Schüttung vorliegt.

38. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der Ansprüche 30 bis 37, dadurch gekennzeichnet, dass die stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle auf einem Trägermaterial aufgebracht ist, insbesondere wobei das Trägermaterial gasdurchlässig, insbesondere luftdurchlässig, ausgebildet ist und/oder insbesondere wobei das Trägermaterial eine Gasdurchlässigkeit, insbesondere Luftdurchlässigkeit, von mindestens 10 $l \cdot m^{-2} \cdot s^{-1}$, insbesondere mindestens 30 $l \cdot m^{-2} \cdot s^{-1}$, vorzugsweise mindestens 50 $l \cdot m^{-2} \cdot s^{-1}$, besonders bevorzugt mindestens 100 $l \cdot m^{-2} \cdot s^{-1}$, ganz besonders bevorzugt mindestens 500 $l \cdot m^{-2} \cdot s^{-1}$, und/oder eine Gasdurchlässigkeit, insbesondere Luftdurchlässigkeit, von bis zu 10.000 $l \cdot m^{-2} \cdot s^{-1}$, insbesondere bis zu 20.000 $l \cdot m-2 \cdot s^{-1}$, bei einem Strömungswiderstand von 127 Pa aufweist.

39. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach Anspruch 38, dadurch gekennzeichnet, dass das Trägermaterial eine dreidimensionale Struktur aufweist, insbesondere wobei das Trägermaterial als vorzugsweise offenporiger Schaumstoff, besonders bevorzugt Polyurethanschaumstoff, ausgebildet ist.

40. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach Anspruch 38, dadurch gekennzeichnet, dass das Trägermaterial eine zweidimensionale und/oder flächige Struktur aufweist, insbesondere wobei das Trägermaterial als vorzugsweise textiles Flächengebilde ausgebildet ist.

41. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach Anspruch 40, dadurch gekennzeichnet, dass das Trägermaterial als ein textiles Flächengebilde, vorzugsweise ein luftdurchlässiges Textilmaterial, bevorzugt ein Gewebe, Gewirke, Gestricke, Gelege oder Textilverbundstoff, insbesondere Vlies (Nonwoven), ausgebildet ist und/oder dass das Trägermaterial ein Flächengewicht von 5 bis 1.000 g/m^2, insbesondere 10 bis 500 g/m^2, bevorzugt 25 bis 450 g/m^2, aufweist.

42. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach Anspruch 40 oder 41, dadurch gekennzeichnet, dass das Trägermaterial ein natürliche Fasern und/oder synthetische Fasern (Chemiefasern) enthaltendes oder hieraus bestehendes textiles Flächengebilde ist, insbesondere wobei die natürlichen Fasern aus der Gruppe von Wollfasern und Baumwollfasern (CO) ausgewählt sind und/oder insbesondere wobei die synthetischen Fasern ausgewählt sind aus der Gruppe von Polyestern (PES); Polyolefinen, insbesondere Polyethylen (PE) und/oder Polypropylen (PP); Polyvinylchloriden (CLF); Polyvinylidenchloriden (CLF); Acetaten (CA); Triacetaten (CTA); Polyacryl (PAN); Polyamiden (PA), insbesondere aromatischen, vorzugsweise flammfesten Polyamiden; Polyvinylalkoholen (PVAL); Polyurethanen; Polyvinylestern; (Meth-)-Acrylaten; Polymilchsäuren (PLA); Aktivkohle; sowie deren Mischungen.

43. Stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der Ansprüche 38 bis 42, dadurch gekennzeichnet, dass die Aktivkohle an und/oder auf dem Trägermaterial fixiert ist, vorzugsweise mittels Verklebung, insbesondere mittels eines Klebstoffs oder infolge von Eigenklebrigkeit oder Eigenadhäsion.

44. Schutzmaterialien, insbesondere für den zivilen oder militärischen Bereich, insbesondere Schutzbekleidung, wie Schutzanzüge, Schutzhandschuhe, Schutzschuhwerk, Schutzsocken, Kopfschutzbekleidung und dergleichen, sowie Schutzabdeckungen, vorzugsweise alle vorgenannten Schutzmaterialien für den ABC-Einsatz, hergestellt unter Verwendung einer stickstoffmodifizierten und/oder stickstofffunktionalisierten Aktivkohle nach einem der Ansprüche 30 bis 43 und/oder aufweisend eine stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der Ansprüche 30 bis 43.

45. Filter und Filtermaterialien, insbesondere zur Entfernung von Schad-, Geruchs- und Giftstoffen aller Art, insbesondere aus Luft- und/oder Gasströmen, wie ABC-Schutzmaskenfilter, Geruchsfilter, Flächenfilter, Luftfilter, insbesondere Filter für die Raumluftreinigung, adsorptionsfähige Trägerstrukturen und Filter für den medizinischen Bereich, hergestellt unter Verwendung einer stickstoffmodifizierten und/oder stickstofffunktionalisierten Aktivkohle nach einem der Ansprüche 30 bis 43 und/oder aufweisend eine stickstoffmodifizierte und/oder stickstofffunktionalisierte Aktivkohle nach einem der Ansprüche 30 bis 43.

Es folgen 10 Blatt Zeichnungen

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Anhängende Zeichnungen

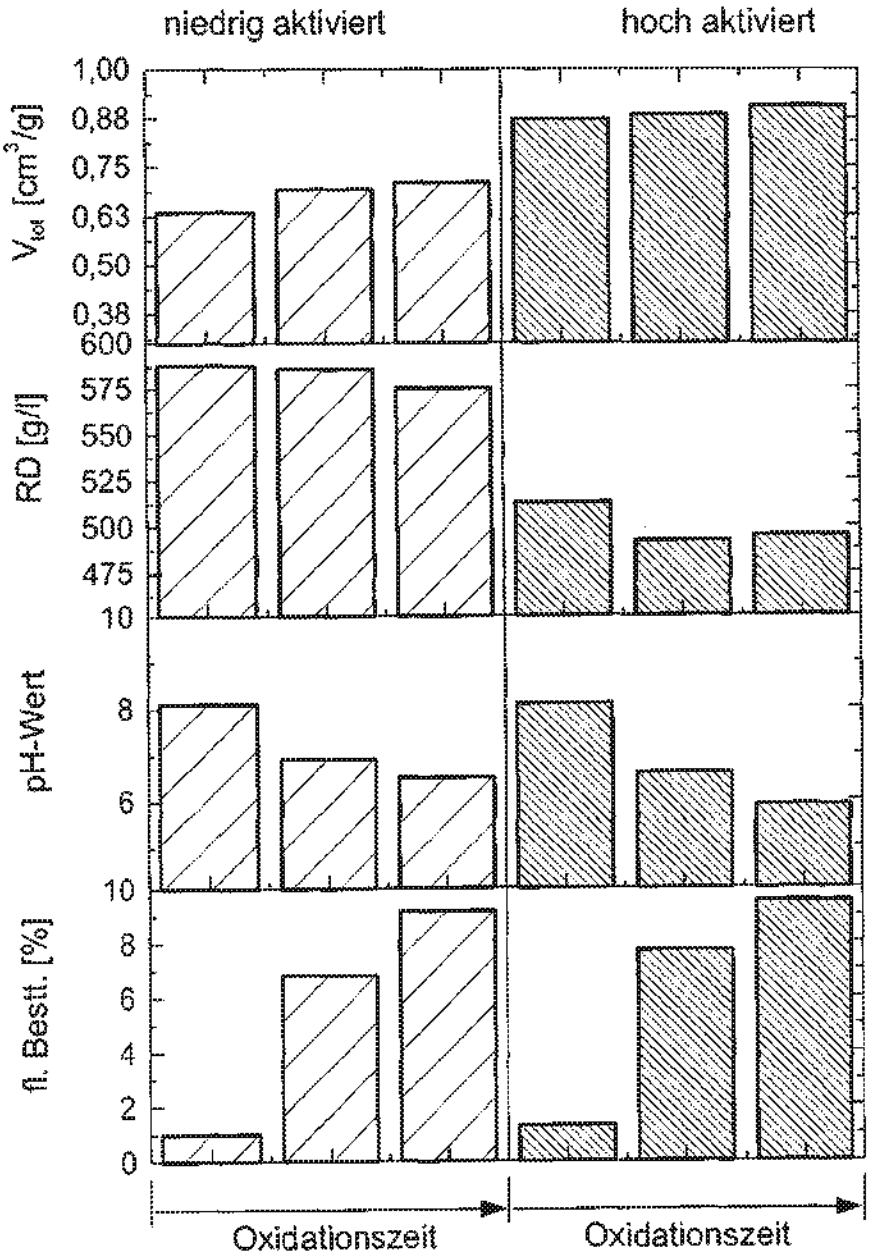


Fig. 1a

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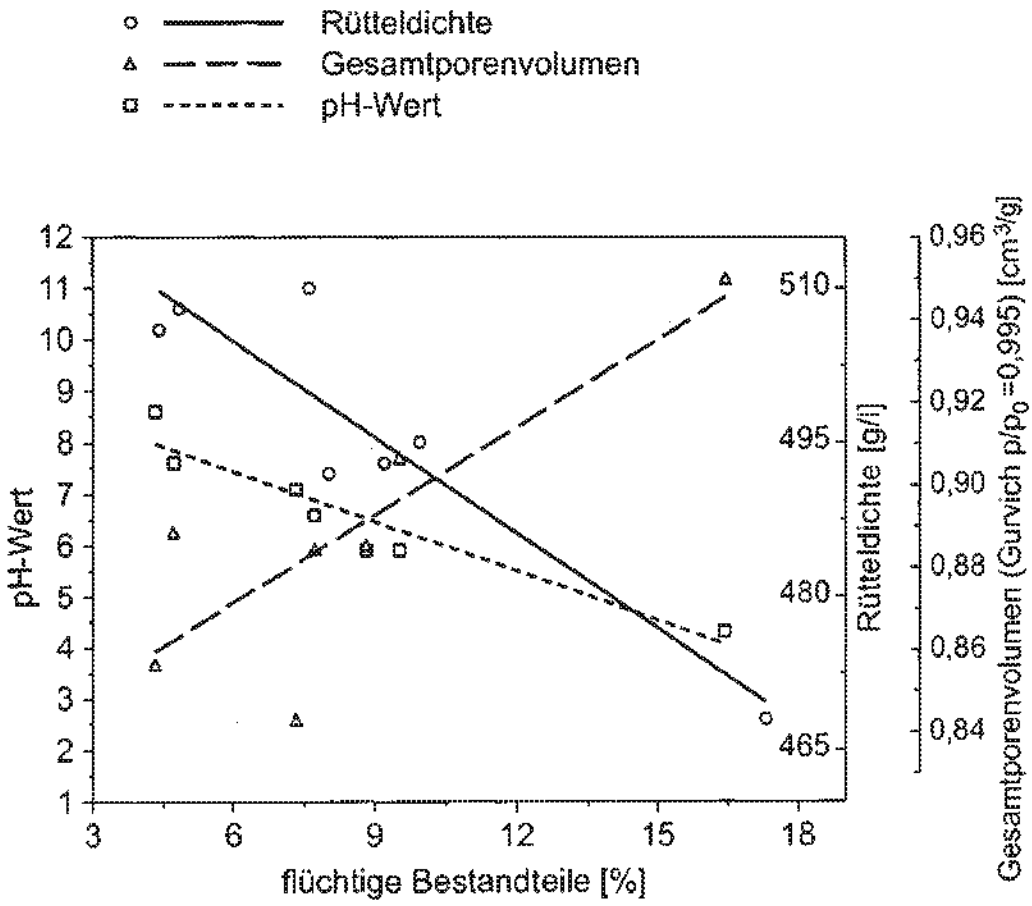


Fig. 1b

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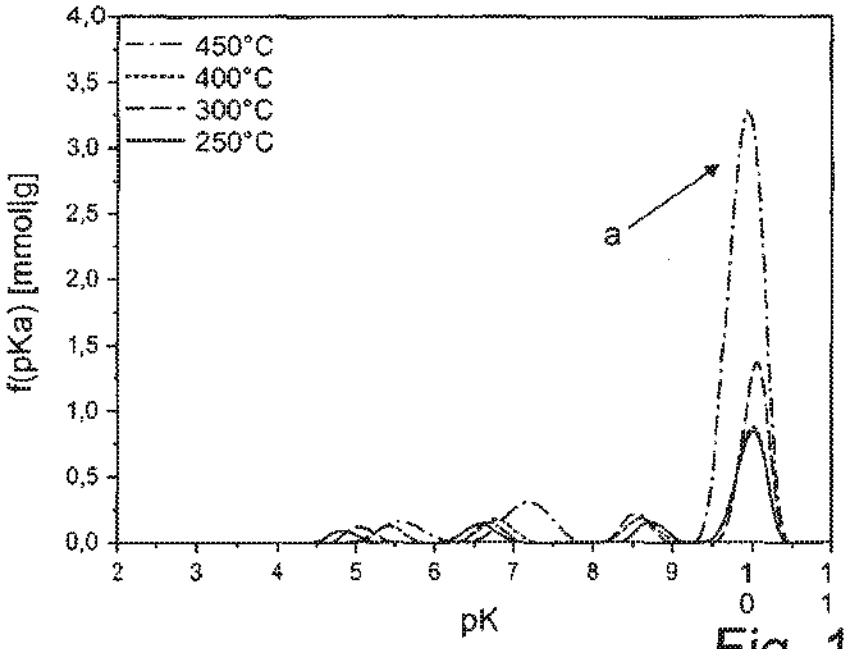


Fig. 1c

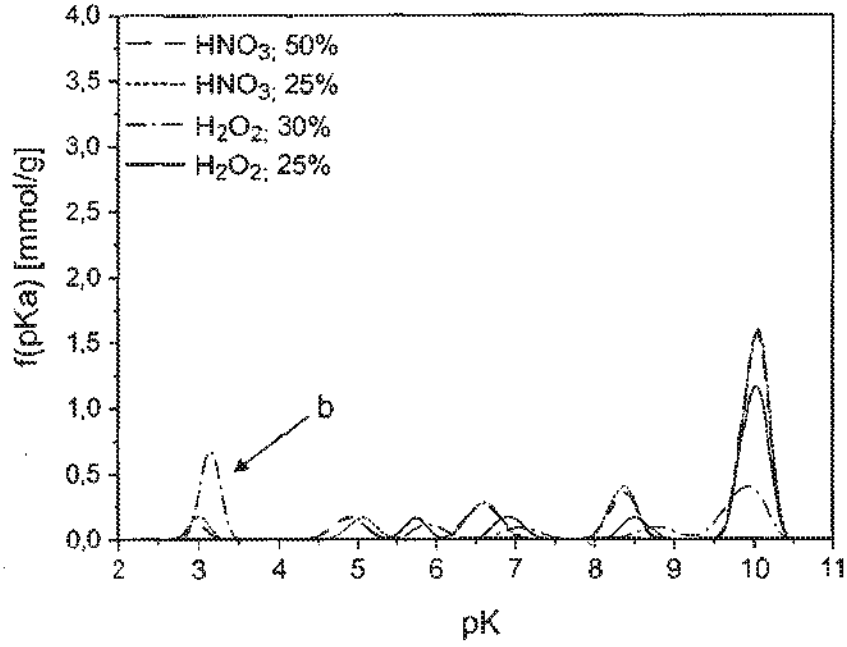


Fig. 1d

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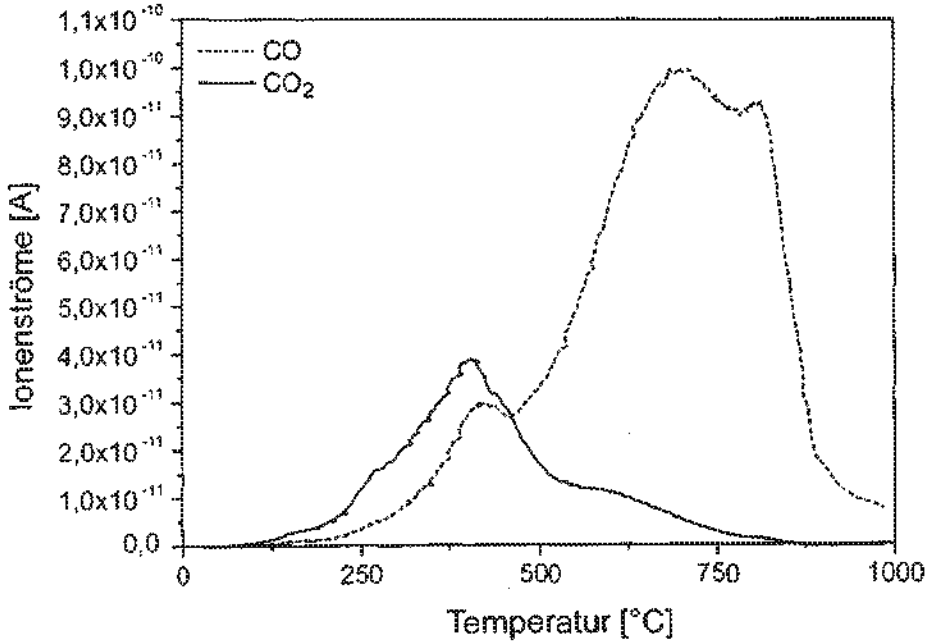

1,1x10⁻¹⁰
1,0x10⁻¹⁰
9,0x10⁻¹¹
8,0x10⁻¹¹
7,0x10⁻¹¹
6,0x10⁻¹¹
5,0x10⁻¹¹
4,0x10⁻¹¹
3,0x10⁻¹¹
2,0x10⁻¹¹
1,0x10⁻¹¹
0,0
Ionenströme [A]
CO
CO2
0
250
500
750
1000
Temperatur [°C]

Fig. 1e

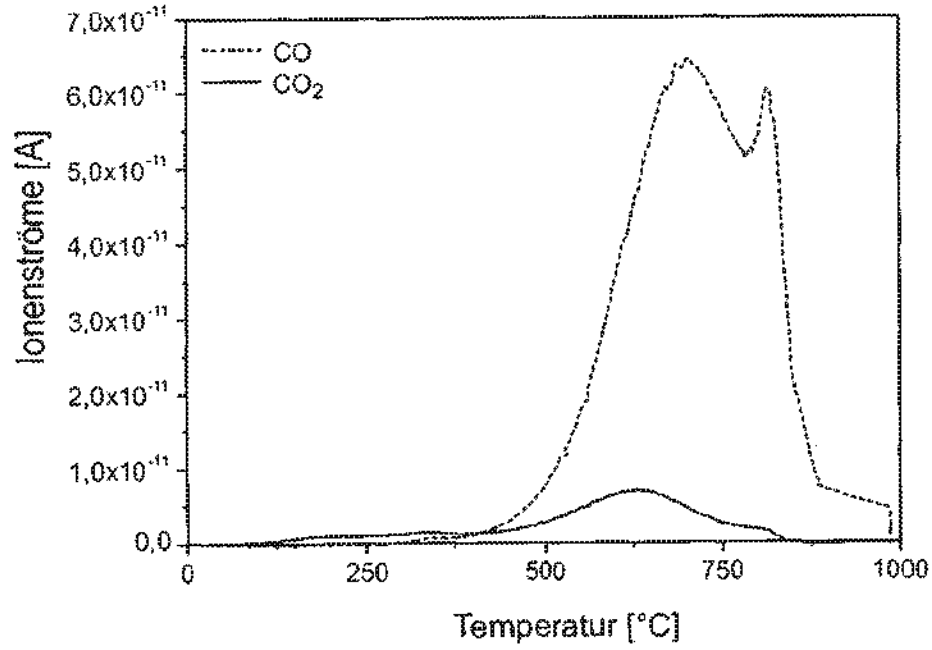

7,0x10⁻¹¹
6,0x10⁻¹¹
5,0x10⁻¹¹
4,0x10⁻¹¹
3,0x10⁻¹¹
2,0x10⁻¹¹
1,0x10⁻¹¹
0,0
Ionenströme [A]
CO
CO2
0
250
500
750
1000
Temperatur [°C]

Fig. 1f

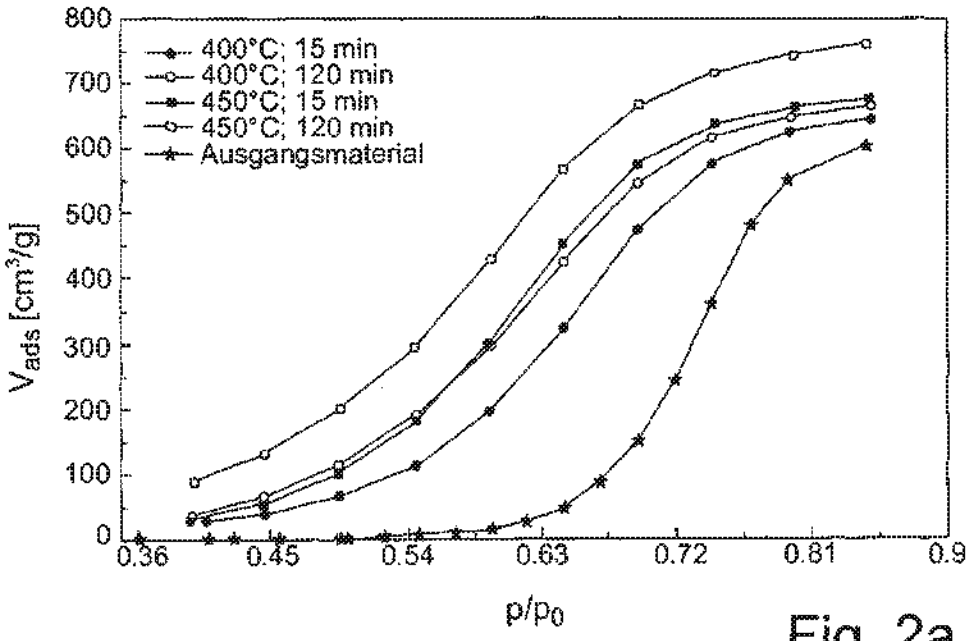


Fig. 2a

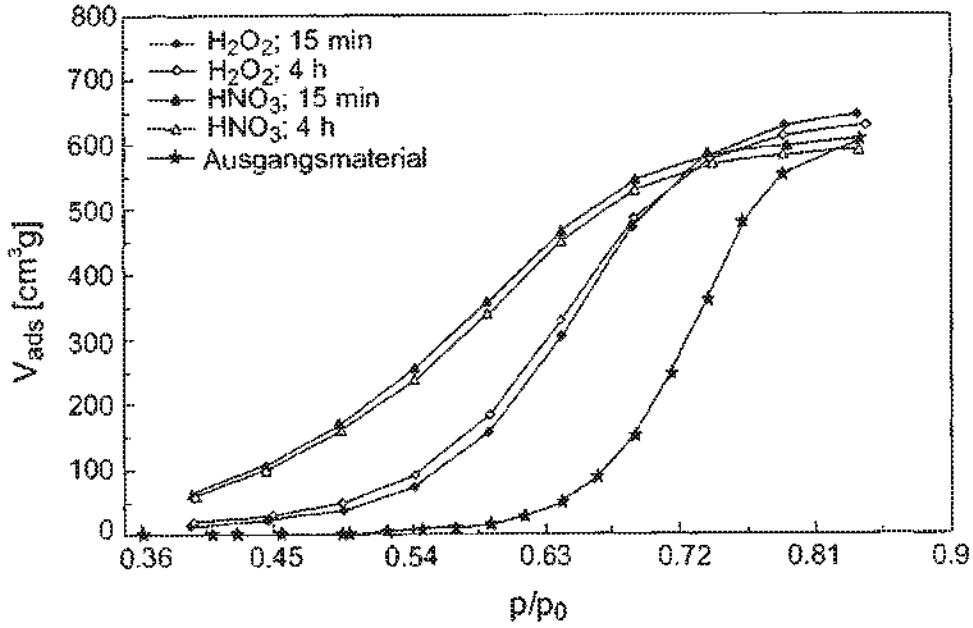


Fig. 2b

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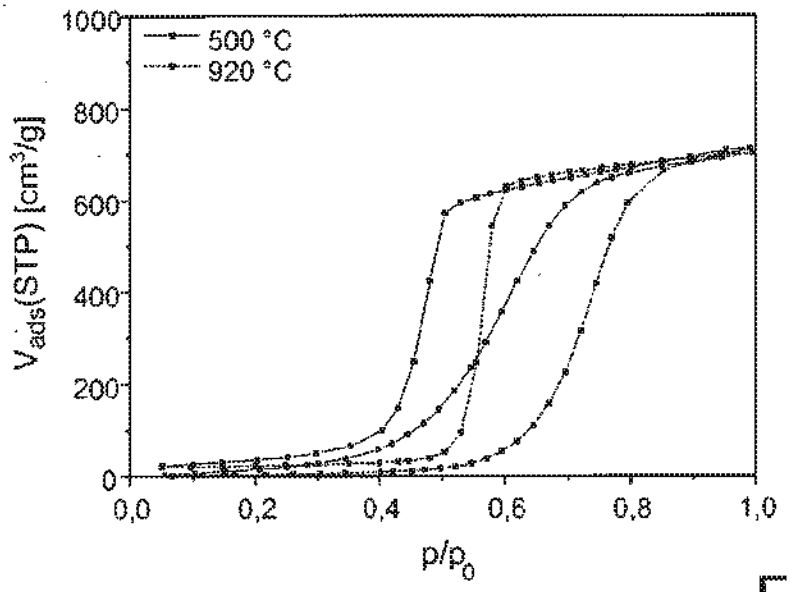

1000
800
600
400
200
0
500 °C
920 °C
V_{ads}(STP) [cm³/g]
0,0
0,2
0,4
0,6
0,8
1,0
p/p$_0$

Fig. 3a

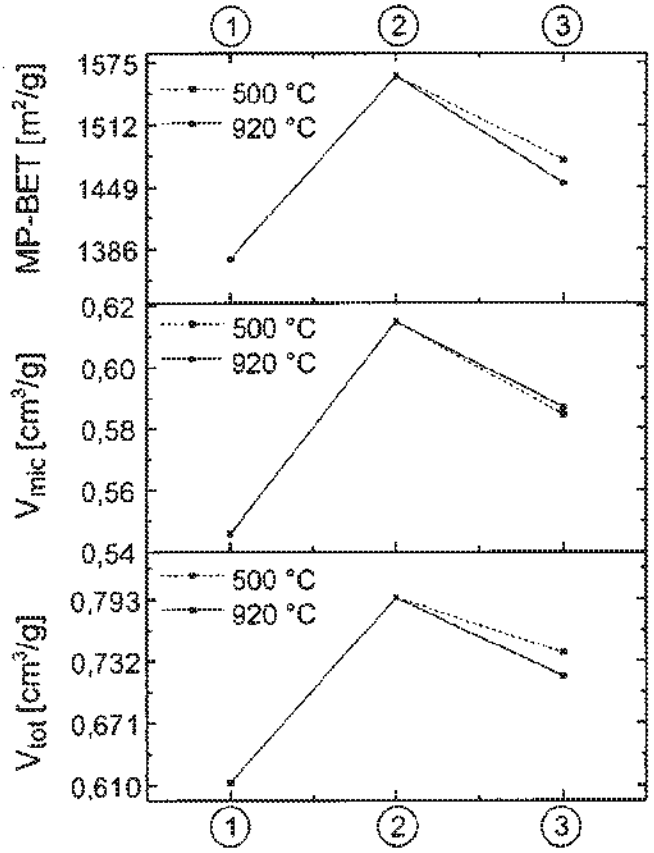

1
2
3
MP-BET [m²/g]
1575
1512
1449
1386
500 °C
920 °C
V_{mic} [cm³/g]
0,62
0,60
0,58
0,56
0,54
V_{tot} [cm³/g]
0,793
0,732
0,671
0,610

Fig. 3b

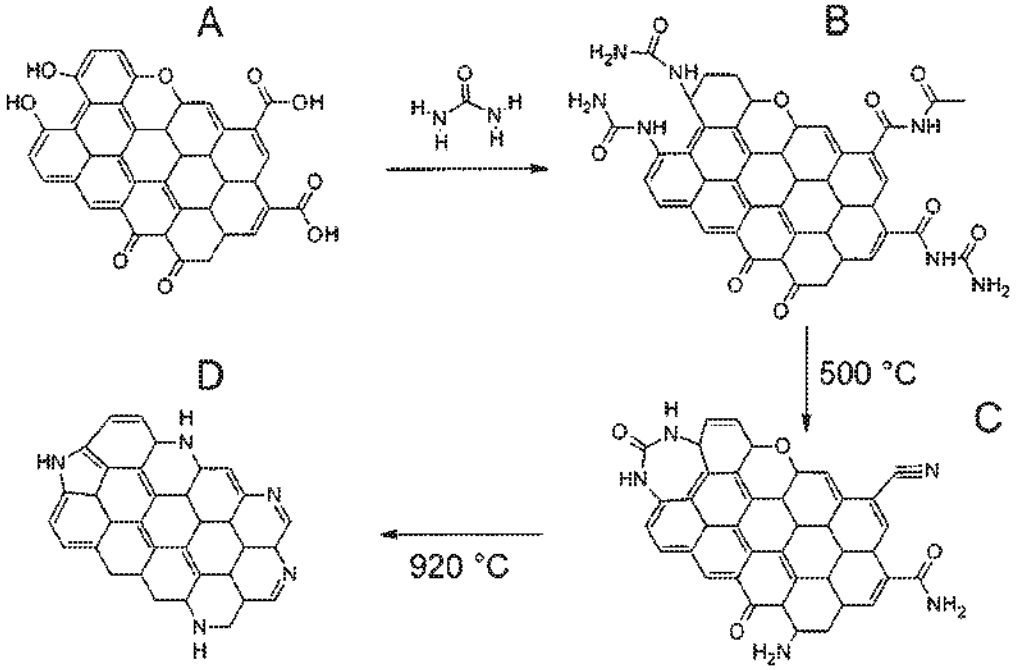

A
B
C
D
500 °C
920 °C

Fig. 4a

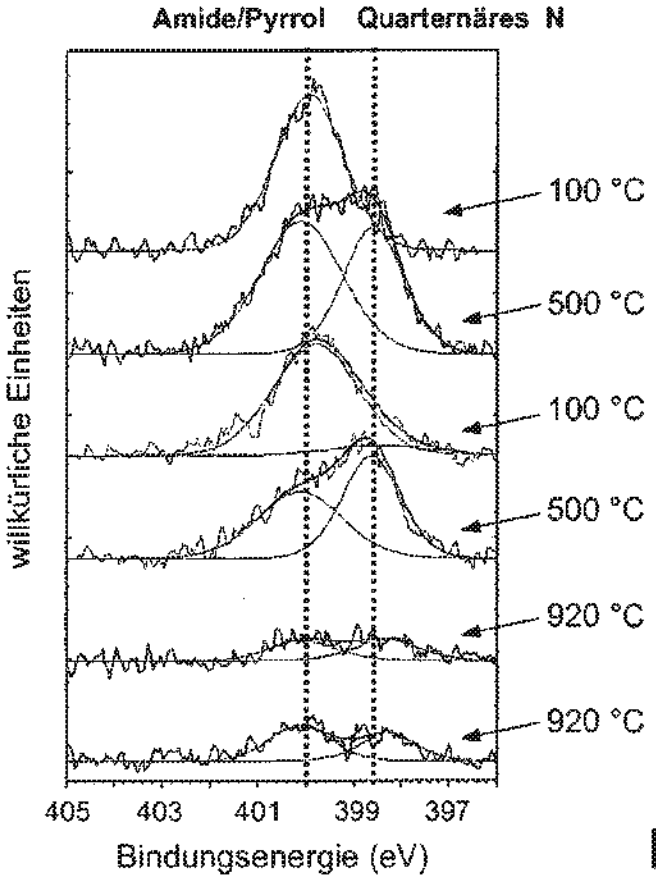

Amide/Pyrrol
Quarternäres N
100 °C
500 °C
100 °C
500 °C
920 °C
920 °C
willkürliche Einheiten
405
403
401
399
397
Bindungsenergie (eV)

Fig. 4b

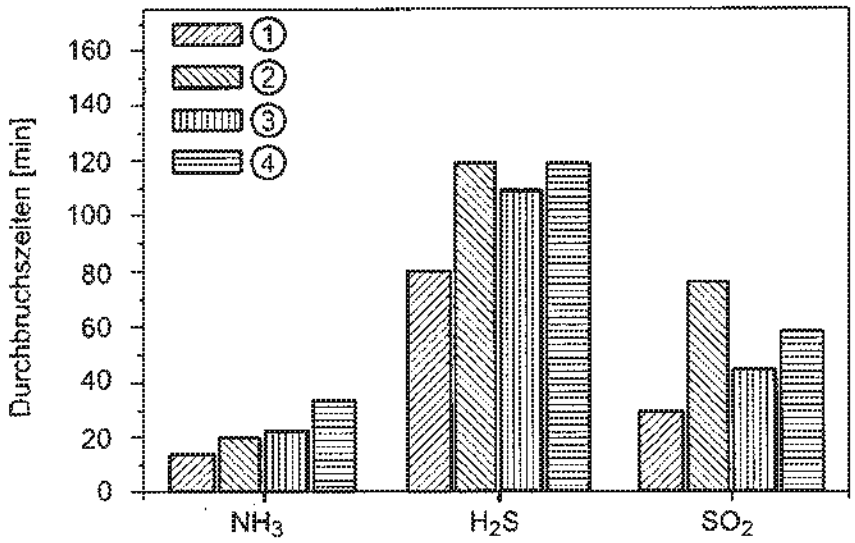


Fig. 5

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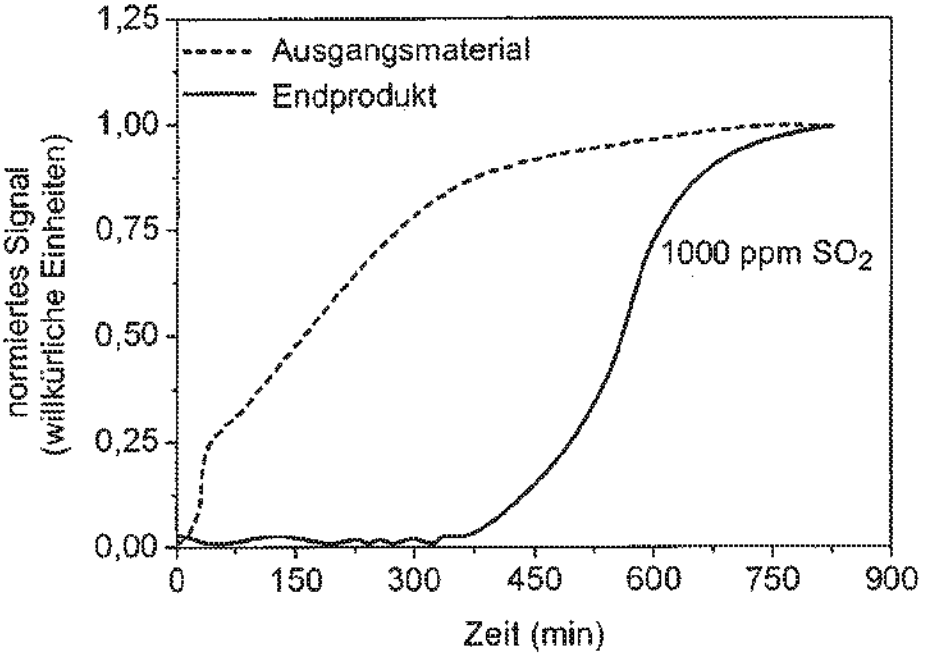

1,25
1,00
0,75
0,50
0,25
0,00
normiertes Signal
(willkürliche Einheiten)
Ausgangsmaterial
Endprodukt
1000 ppm SO_2
0
150
300
450
600
750
900
Zeit (min)

Fig. 6a

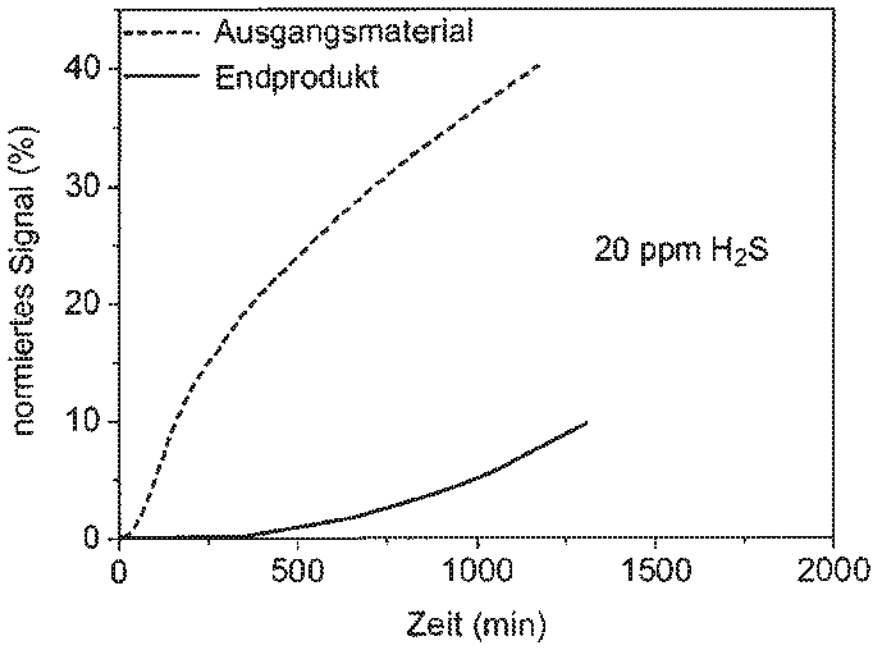

40
30
20
10
0
normiertes Signal (%)
Ausgangsmaterial
Endprodukt
20 ppm H_2S
0
500
1000
1500
2000
Zeit (min)

Fig. 6b

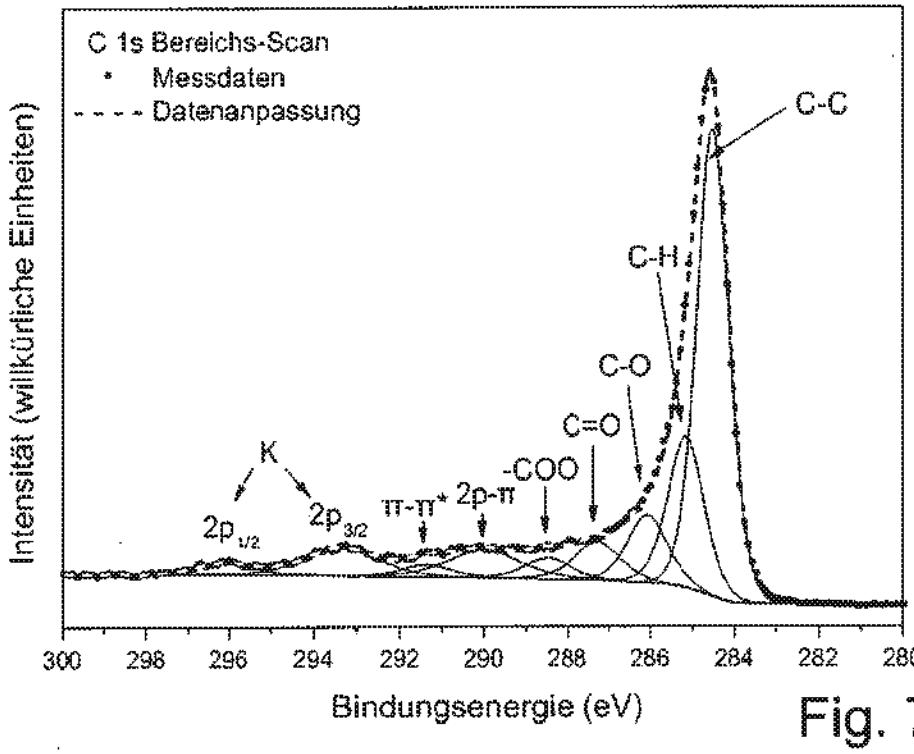

C 1s Bereichs-Scan
Messdaten
Datenanpassung
C-C
C-H
C-O
C=O
-COO
2p-π
π-π*
K
2p1/2
2p3/2
Intensität (willkürliche Einheiten)
300 298 296 294 292 290 288 286 284 282 280
Bindungsenergie (eV)

Fig. 7a

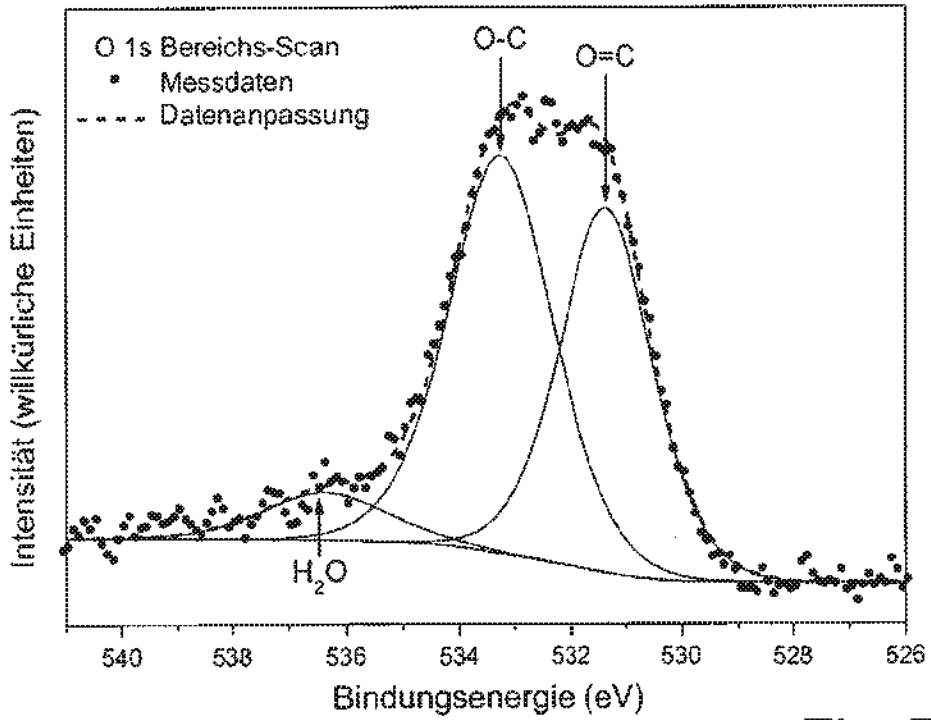

O 1s Bereichs-Scan
Messdaten
Datenanpassung
O-C
O=C
H2O
Intensität (willkürliche Einheiten)
540 538 536 534 532 530 528 526
Bindungsenergie (eV)

Fig. 7b