



Catalyst attrition in an ASTM fluidized bed



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ABSTRACT

Catalyst attrition is a major issue for the reliable and efficient performance of a fluidized-bed reactor. In this study, attrition behavior of a commercial FCC catalyst is examined in a laboratory-scale ASTM fluidized bed. An attrition rate model that combines the jet attrition and bubble-induced attrition is derived and confirmed to represent the measured total attrition rate versus the superficial gas velocity. The model parameters systematically vary with time, since the properties of the attrited particles change progressively. It is also shown that the minimum bubble attrition velocity is far larger than the minimum fluidizing velocity because energy is required to produce attrition. Furthermore, jet and bubble attritions are separated and their contributions are discussed. The contribution of jet attrition increases rapidly with increasing the gas velocity, and the fast increase in the jet attrition rate is the major cause of the increasingly serious catalyst attrition with increasing the gas velocity. It is also seen that regression analysis is an effective tool to the separation of attrition sources.

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1. Introduction

Fluidized beds are widely used in industrial processes such as drying, granulation, combustion, and fluid catalytic cracking (FCC), because materials mix and contact adequately with each other and heat transfers excellently [1,2]. However, particle attrition always exists in fluidized beds [3–6]. For example, in a fluidized-bed reactor, catalyst attrition will result in the generation of fines, loss of valuable material, degradation of catalyst efficiency, environmental pollution, etc. Furthermore, new-born small particles brought by attrition may damage fluidizing properties and process operating conditions [3,6]. Catalyst attrition is, therefore, a major issue for the reliable and efficient performance of an industrial fluidized-bed reactor.

Several methods have been developed to assess and study the attrition resistances of particle materials [7–9]. For powdered catalysts used in fluidized-bed reactors, two methods are often used in laboratory: ASTM air-jet attrition test [7,8] and jet-cup attrition test [9]. They are usually intended to rank different candidate catalysts with respect to their attrition resistances. Zhao et al. [10] reported that the jet-cup test needed less catalysts and was as adequate as the ASTM standard test for the prediction of the catalyst attrition resistance. However, attrition mechanism in an ASTM standard fluidized bed is much closer to that in an industrial fluidized bed.

There are two main types of attrition: particle fragmentation and surface abrasion [11–18]. Abrasion means the loss of edges and corners of particle surfaces, generation of son particles which are approximately the same size to the mother ones and a lot of fines, while fragmentation refers to a particle breaking into several son particles smaller than the mother one. Several sources of attrition can be identified in a fluidized bed system, e.g., grid jet attrition, bubble-induced attrition, and attrition in the cyclones [6,19]. For these attrition sources, several correlations have been developed to relate the measured attrition rate to the jet/superficial gas velocity, density, orifice diameter, etc. [4,12–15,20–24]. Attrition is also a time-dependent process and may change systematically with time [8,19,25,26]. Many materials show an early nonsteady-state attrition behavior, after which attrition decreases to a constant value [19,25,26]. Several models have been proposed to describe the time-dependence of attrition behavior, e.g., Gwyn equation [8] and exponential decay equation [26]. Moreover, in a fluidized bed, attrition is influenced by three categories of factors including particle properties (material properties, textural properties, mechanical strength, shape, size, and surface roughness, hardness, microcracks, etc.), fluidization conditions (gas velocity, pressure, temperature, density, humidity, etc.), and fluidized-bed structure parameters (most importantly, orifice number and diameter for multi-orifice distributor plates) [4,11–15,20–24,27].

Academic institutions active in catalysis research generally concentrate on the chemistry rather than the catalyst mechanical properties. Less effort has been made to investigate the attrition behavior and mechanism in fluidized beds, although a reasonable

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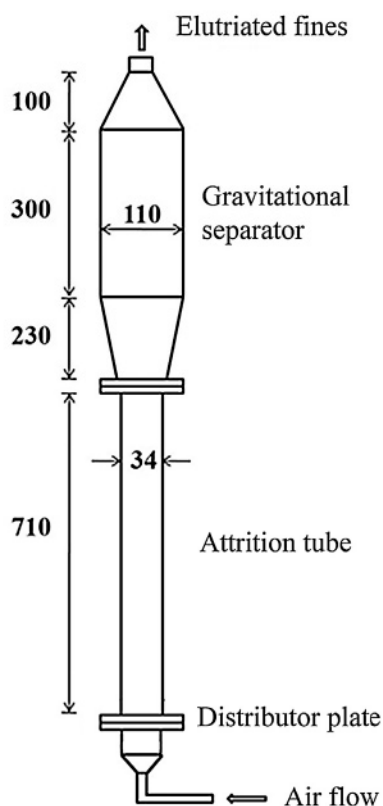


Fig. 1. Schematic diagram of attrition apparatus (The physical unit of data is millimeter.).

amount of information is already available concerning the particle attrition. To our knowledge, no article is available so far in the open literature about the scientific basis for the ASTM air-jet attrition test; therefore, an interest in studying the attrition behavior in an ASTM fluidized bed arises. Moreover, sources of attrition in fluidized beds should be examined, and there is a lack of an efficient and quick method to separate the total attrition rate into contributions of different sources. Therefore, the issues of catalyst attrition call yet for further elucidation and advancement. In this work, dependence of attrition behavior of a commercial FCC catalyst on superficial gas velocity was examined in a laboratory-scale fluidized bed designed according to ASTM standard. The total attrition rate was correlated with a derived attrition rate equation. Sources of attrition were separated by a simple regression analysis method, and their contributions were discussed.

2. Experimental

2.1. Catalyst sample, apparatus, and attrition tests

A commercial FCC catalyst was used in this study. It consists mainly of Y-zeolite and kaolin, and is serving in a FCC unit in China petroleum industry. Its mean particle diameter, packing density, and surface area are about 109 μm , 0.885 g/cm^3 and 168.7 m^2/g , respectively. Other physical properties have been described elsewhere [26]. Prior to attrition tests, the catalyst was sieved by a 61-micron sieve to eliminate pre-existing fine particles, and then dried at 120 $^\circ\text{C}$ in air for 2 h to remove the effect of humidity.

A laboratory-scale fluidized bed, shown in Fig. 1, was employed as an attrition test apparatus. It was designed according to ASTM D 5757-11 standard [7], consisting of six parts: air supply system, gas distribution chamber, three-orifice distributor plate, attrition tube, gravitational separator, and fines collector. The distributor

plate contains three symmetrically arranged upward-facing holes with diameter 0.381 ± 0.005 mm, and these holes are 10 mm distant from the plate center. Other dimensions are marked in Fig. 1. For more information on the attrition apparatus, see ASTM standard [7] or one of our previous publications [26].

Attrition tests were carried out at room temperature. After 50 g of a dried catalyst was charged, an air flow was produced by air compressor and fed to the attrition apparatus to fluidize catalyst particles. Elutriated fines were collected, dried, and then weighed. Detailed operating steps were described in previous article [26]. It should be mentioned that after fines collector, air flow was directly emitted into the atmosphere; therefore, the temperature and pressure in the fluidized bed could be approximately regarded as normal temperature and pressure (NTP), i.e., 20 $^\circ\text{C}$ and 101.3 kPa. Under the operating condition, seven superficial gas velocities (0.0834, 0.0981, 0.1155, 0.1337, 0.1528, 0.1746, and 0.1951 m/s) were examined in this work. For each gas velocity, the mass of the elutriated fines was determined as a function of the attrition time, and then the total attrition rate was calculated.

2.2. Data analysis

In the literature [6,12,19,22,27,28], attrition rate is often defined as the mass of the elutriated fines per unit time:

$$R_a = \frac{dm_e}{dt} \quad (1)$$

where R_a is the attrition rate at attrition time t , and m_e the cumulative mass of the elutriated fines at time t . In a fluidized bed, two sources of attrition, i.e., grid jet attrition and bubble-induced attrition, can often be identified; therefore, the total attrition rate in the bed is the sum of the attrition rates of the two sources [6,19].

Grid jet attrition has been studied by several authors [9,10,23–25,27–31]. Considering the energy utilization of abrasion process, Werther and Xi [28] derived a relationship for grid jet attrition rate, which can be rewritten as

$$R_{a,j} = C_j \cdot d_{pb} \cdot \rho_f \cdot \frac{D_t^6}{d_{or}^4 \cdot n_{or}^2} \cdot u^3 \quad (2)$$

where $R_{a,j}$ is the jet attrition rate, C_j the jet attrition constant, d_{pb} the surface mean diameter of bed particles, n_{or} the number of orifices in the distribution plate, d_{or} the diameter of orifices, ρ_f the density of jet gas, D_t the diameter of fluidized-bed column, and u the superficial gas velocity. The mechanisms of bubble-induced attrition are not quite clear. There are various theoretical and empirical approaches [6,14,32] that can be summarized as

$$R_{a,b} = C_b \cdot m_b^n \cdot (u - u_{min})^m \quad (3)$$

where $R_{a,b}$ is the bubble attrition rate, C_b the bubble attrition constant, n and m the two power exponents, u_{min} a threshold velocity above which bubble-induced attrition occurs, and m_b the decreasing catalyst bed mass due to attrition ($m_b = m_0 - m_e$, where m_0 is the initial catalyst bed mass, i.e., the catalyst bed mass at time $t=0$. $m_0 = 50$ g in this study). Combining Eqs. (2) and (3), the total attrition rate in a fluidized bed can, therefore, be expressed as

$$R_{a,tot} = R_{a,j} + R_{a,b} = C_j \cdot d_{pb} \cdot \rho_f \cdot \frac{D_t^6}{d_{or}^4 \cdot n_{or}^2} \cdot u^3 + C_b \cdot m_b^n \cdot (u - u_{min})^m \quad (4)$$

It should be mentioned that the catalyst bed mass, m_b , decreases as the attrition time increases. Nevertheless, for a given attrition time, it will also decrease probably with increasing the superficial gas velocity. Suppose the decreasing catalyst bed mass (or the

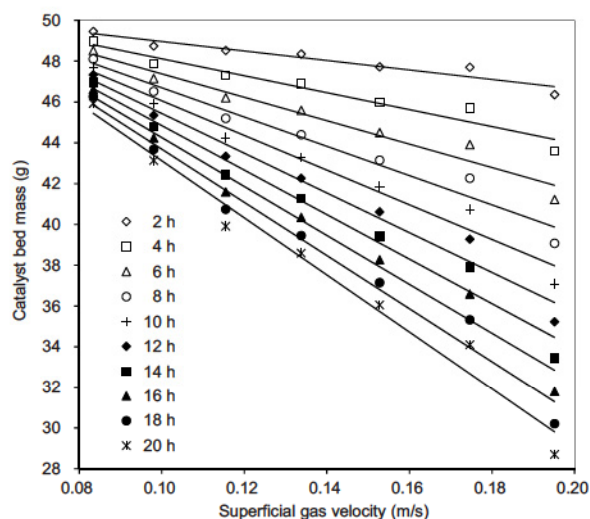


Fig. 2. Dependence of catalyst bed mass on air velocity and linear fittings of Eq. (5).

cumulative mass of the elutriated fines, m_e) has a linear relation with the gas velocity, i.e.,

$$m_b = -ku + b \quad (5)$$

Substituting Eq. (5) into Eq. (4), we get

$$R_{a,tot} = R_{a,j} + R_{a,b} = C_j \cdot C_0 \cdot u^3 + C_b \cdot (-ku + b)^n \cdot (u - u_{min})^m \quad (6)$$

where

$$C_0 = d_{pb} \cdot \rho_f \cdot \frac{D_t^6}{d_{or}^4 \cdot n_{or}^2} \quad (7)$$

For given bed structure, jet gas and catalyst particles, C_0 is a constant value; therefore, only seven parameters (k , b , C_j , C_b , u_{min} , n , and m) exist in Eq. (6), and they can be estimated with regression analysis. The mass of the elutriated fines is experimentally measured, and the parameters, k and b , are thus obtained directly by the least-squares linear fit of the bed mass data to Eq. (5). Then the total attrition rate is calculated according to the mass of the elutriated fines, and the other five parameters can be estimated by the least-squares nonlinear fit of the total attrition rate data to Eq. (6).

3. Results and discussion

3.1. Influence of gas velocity on the catalyst bed mass

Fig. 2 depicts the catalyst bed mass as a function of gas velocity at different attrition times, where the straight lines are plotted by linear fittings. The estimated parameters, k and b , and the goodness-of-fit results are listed in Table 1. Except for the fitting of the 2 h attrition data set, the other data set fittings all give high coefficients of determination, R^2 . Thus it can be seen that a good linear relationship exists between the catalyst bed mass and the gas velocity, which proves the accuracy of the above-made linear hypothesis.

As shown in Fig. 2, for any attrition time, the catalyst bed mass always, as expected, decreases as the superficial gas velocity increases, indicating that the larger the gas velocity, the more serious the catalyst attrition will be. It can be seen from Table 1 that the k value increases with increasing attrition time, revealing that a larger attrition time will lead to a more significant effect of the gas velocity on the catalyst bed mass. As mentioned above, attrition is a time-dependent process, and at the early nonsteady-state stage of attrition, fines are elutriated in small quantities, though the initial attrition rate is very large [19,25,26], which probably masks the effect of the gas velocity on the catalyst bed mass. It is also

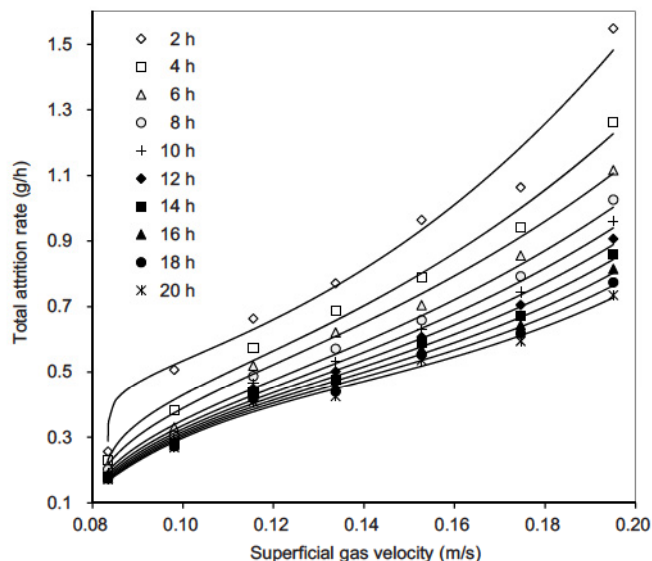


Fig. 3. Dependence of total attrition rate on air velocity and nonlinear fittings of Eq. (6).

clear from Fig. 1, which shows that the catalyst bed mass linearly decreases at a much faster speed for a larger attrition time.

3.2. Influence of gas velocity on the total attrition rate

The least-squares nonlinear fittings of Eq. (6) to the total attrition rate data are shown in Fig. 3, and the estimated parameters and goodness-of-fit results are given in Table 1. It can be seen that the coefficients of determination, R^2 , are all larger than 0.990, indicating that Eq. (6) gives fairly good fits to all the data sets. Fig. 3 also visualizes that the total attrition rate data follow Eq. (6) well for all the attrition times. These reveal that the derived total rate equation presents a suitable description of the total attrition rate versus the superficial gas velocity.

It can be seen from Fig. 3 that for any attrition time the total attrition rate increases with increasing the gas velocity, reaffirming that a large gas velocity intensifies the catalyst attrition. Furthermore, for a larger attrition time, the effect of the gas velocity on the total attrition rate is less significant, contrary to the effect of the gas velocity on the catalyst bed mass. It results from the fact that a larger attrition time leads to a smaller total attrition rate and thus weakens the effect of the gas velocity on the total attrition rate.

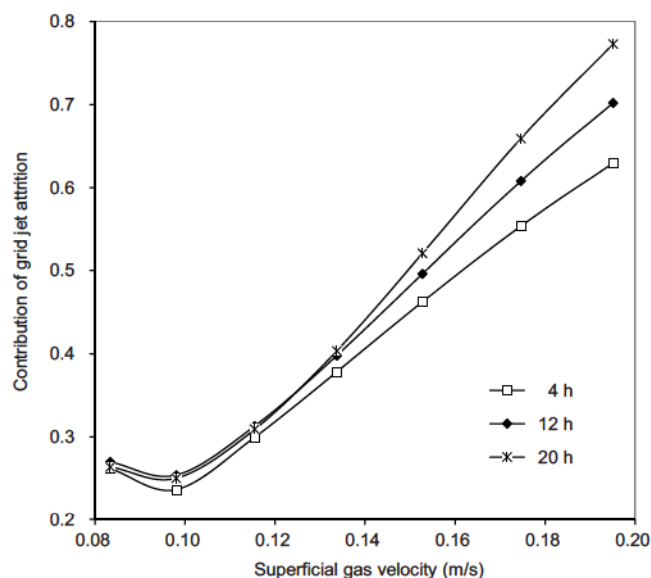
As depicted in Table 1, the estimated parameters in Eq. (6) vary with the attrition time. For instance, both the jet attrition constant, C_j , and the bubble attrition constant, C_b , generally decrease with increasing the attrition time. It is quite reasonable, since both attritions abate gradually with time, resulting in the decrease in the total attrition rate. Furthermore, as the attrition time increases, the two power exponents, n and m , both increase in the bubble-induced attrition item, showing that the time-dependence of the bubble attrition rate is much more complex than that of the jet attrition rate. It is also found from Table 1 that the minimum bubble attrition velocity, u_{min} , decreases slightly with time. The variations of all these parameters are essentially due to the fact that the properties of the attrited particles, particularly shape, size and surface characteristics, vary progressively with time.

As is well known, catalyst particles in a bed will not be fluidized unless the pressure drop of the bed is greater than the catalyst bed weight per unit cross-sectional area of bed [14,32]. Before the beginning of fluidization, the pressure drop will increase with increasing the gas velocity. As the pressure drop increases to be equal to the catalyst bed weight per unit area, catalyst particles

Table 1

Fitting analyses of the catalyst bed mass and total attrition rate data.

t (h)	Linear fitting of Eq. (5)			Nonlinear fitting of Eq. (6)					
	k	b	R^2	$C_j \cdot C_0$	C_b	n	m	$u_{\min}$ (m/s)	R^2
2	23.18	51.28	0.914	138.95	2.47×10^{-2}	0.805	0.088	0.0834	0.990
4	41.50	52.27	0.944	104.07	7.20×10^{-4}	1.857	0.269	0.0820	0.996
6	57.36	53.11	0.958	89.41	2.65×10^{-4}	2.192	0.375	0.0798	0.997
8	71.72	53.87	0.965	84.57	9.26×10^{-5}	2.506	0.432	0.0788	0.998
10	85.01	54.56	0.969	83.95	1.64×10^{-5}	2.998	0.482	0.0782	0.998
12	97.43	55.19	0.972	84.04	2.77×10^{-6}	3.519	0.543	0.0775	0.998
14	109.09	55.75	0.973	83.54	6.74×10^{-7}	3.962	0.617	0.0766	0.998
16	120.04	56.27	0.974	81.88	3.39×10^{-7}	4.219	0.693	0.0757	0.999
18	130.34	56.72	0.975	79.23	3.46×10^{-7}	4.284	0.762	0.0746	0.999
20	140.02	57.13	0.976	75.74	6.68×10^{-7}	4.162	0.811	0.0738	0.999

**Fig. 4.** Contribution of grid jet attrition as a function of air velocity.

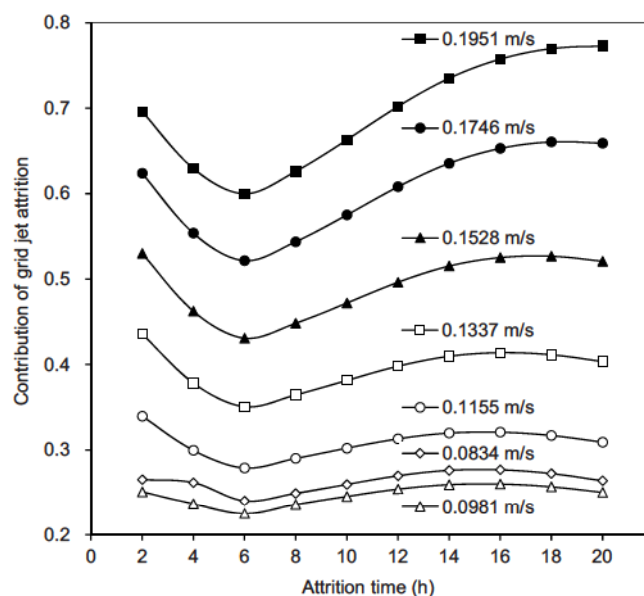
reach a critical state between static and suspended states. Here, the superficial gas velocity is often called minimum (or critical) fluidizing velocity, u_{mf} [33]. Minimum fluidizing velocity can be accurately measured by experiments, and it can be also determined by the method of calculation. In this study, u_{mf} was estimated by an empirical formula reported in a fluidization textbook [33]. The obtained u_{mf} is about 0.00347 m/s, far less than all the $u_{\min}$ (minimum bubble attrition velocity) values listed in Table 1. This reveals that the minimum fluidizing velocity cannot generate bubble attrition, because energy is required to produce attrition after the beginning of particle fluidization.

3.3. Contribution of grid jet attrition

As shown above, the total attrition rate in a fluidized bed is the sum of the grid jet attrition rate and bubble-induced attrition rate. The parameters in Eq. (6) are estimated by regression analysis, and then the total attrition rate can be separated into contributions of the two attrition sources. For instance, the contribution of grid jet attrition, η , can be expressed as

$$\eta = \frac{R_{a,j}}{R_{a,tot}} \quad (8)$$

In Fig. 4, the plots of the jet attrition contribution versus the gas velocity are illustrated. For the sake of clarity, only three selected attrition times are shown in the figure. Other attrition times give a similar curve behavior. It can be found that in the examined range of the gas velocity, the contribution of jet attrition decreases slightly

**Fig. 5.** Contribution of grid jet attrition as a function of attrition time.

at first and then increases rapidly with the increase of the gas velocity. The slight reduction in the 0.08–0.10 m/s range is probably attributed to the somewhat bad randomness of a small-scale fluidized bed used in this study, leading to the uncontrollable variation of the properties of the catalyst beds used by different experiments, and thus to the attrition behavior changes, especially at low gas velocities.

It can be seen from Eq. (3) that the bubble attrition rate is closely related to the decreasing catalyst bed mass, and thus a complex relationship exists between the bubble attrition rate and gas velocity (see Eq. (6)). However, the jet attrition rate has no connection with the catalyst bed mass, and it always increases as the gas velocity increases (see Eqs. (2) or (6)). These provide strong support for the rapidly increasing contribution of the jet attrition with increasing the gas velocity. As shown in Fig. 4, for any attrition time, at low gas velocities, the bubble-induced attrition is more dominant than the jet attrition; however, at high gas velocities, the jet attrition plays a dominant role. Especially, as the gas velocity approaches 0.2 m/s, the jet attrition contribution reaches about 70%. These results reveal that the fast increase in the jet attrition rate is the major cause of the more and more serious catalyst attrition occurring as the gas velocity increases.

Furthermore, Fig. 4 indicates that at higher gas velocities, the attrition time has a larger effect on the jet attrition contribution. To further clarify the effect of the attrition time, the contribution of grid jet attrition versus the attrition time is plotted in Fig. 5. A striking feature can be seen that the jet attrition contribution decreases

at the early nonsteady-state stage of attrition and then increases with increasing the attrition time. Nevertheless, compared to the effect of the gas velocity, the attrition time has less influence on the jet attrition contribution.

It should be mentioned that the jet attrition, as its name implies, takes place in regions near the orifices in the distributor plate, and thereby its rate has no distinct connection with the catalyst bed mass. In contrast, the bubble-induced attrition happens for all the fluidized particles, and its rate is clearly related to the catalyst bed mass. Previous authors [6,14,32] usually neglected the variation of the catalyst bed mass, and the initial catalyst bed mass was thus used in their bubble attrition rate equations. Nevertheless, the catalyst bed mass always decreases with increasing the superficial gas velocity or attrition time, as shown in Fig. 2. In this study, a decreasing catalyst bed mass was, therefore, introduced into the bubble attrition rate, and then a more adequate total attrition rate model, i.e. Eq. (6), was derived, as shown in Table 1 and Fig. 3. Moreover, in the literature [6,19], a series of preliminary experiments must be used to separate the total attrition rate into contributions of different attrition sources. However, in this study, jet and bubble attritions were directly separated by regression analysis. Results reveal that it gives a simple, efficient and quick method for the separation of attrition sources.

4. Conclusions

The derived attrition rate equation presents a suitable description of the measured total attrition rate versus the superficial gas velocity in a small-scale fluidized bed. The estimated parameters in the equation systematically vary with the attrition time, due to the fact that the properties of the attrited particles change progressively. It is seen that the minimum fluidizing velocity cannot generate bubble attrition and that it is much less than the minimum bubble attrition velocity because energy is required to produce attrition. Experimental results also indicate that as the gas velocity increases, the catalyst bed mass decreases linearly and the total attrition rate increases. For a larger attrition time, the effect of the gas velocity on the total attrition rate is less significant, while its effect on the catalyst bed mass is more significant. Furthermore, it is found that the contribution of jet attrition generally increases rapidly with the increase of the gas velocity. At high gas velocities, the jet attrition plays a dominant role for any attrition time. Thus the fast increase in the jet attrition rate is the major cause of the increasingly serious catalyst attrition with increasing the gas velocity. Finally, regression analysis is shown to be an effective tool to the separation of attrition sources.

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