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Tomkinson, Alexandra
Bailey Walsh & Co LLP
1 York Place
Leeds, LS1 2DR
ROYAUME UNI

Date
07.09.16

Reference	Application No./Patent No.
ATSAR-X11031-EP	11861339.7 - 1454 / 2688399
Applicant/Proprietor	
Enviro Tech Chemical Services Inc.	

Communication regarding the expiry of the time limit within which notice of opposition may be filed

You are hereby informed that on expiry of the nine-month time limit from the publication of the mention of the grant of European patent No. 2688399 no notice of opposition had reached the files.

The entry in the Register of European Patents will be automatically generated by the electronic data processing system.

For the Examining Division

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EPPU 02: 04.11.15 1454

CLEAN CHEMISTRY EXHIBIT 1027
IPR2025-01458 – Page 1

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Bailey Walsh & Co LLP
1 York Place
Leeds, LS1 2DR
ROYAUME UNI

Date
05.11.15

Reference	Application No./Patent No.
ATSAR-X11031-EP	11861339.7 - 1454 / 2688399
Applicant/Proprietor	
Enviro Tech Chemical Services Inc.	

Transmission of the certificate for a European patent pursuant to Rule 74 EPC

The certificate for a European patent is herewith transmitted.

The European patent specification can be downloaded from the EPO publication server **https://data.epo.org/publication-server/** (see OJ EPO 2005, 126).

Note:
A corrected title page of the European patent specification will be published, if the bibliographic data have been changed after completion of the technical preparations.

For the Examining Division

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Questions about this communication ?
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Tomkinson, Alexandra
Bailey Walsh & Co LLP
1 York Place
Leeds, LS1 2DR
ROYAUME UNI

Date
08.10.15

Reference	Application No./Patent No.
ATSAR-X11031-EP	11861339.7 - 1454 / 2688399
Applicant/Proprietor	
Enviro Tech Chemical Services Inc.	

Decision to grant a European patent pursuant to Article 97(1) EPC

Following examination of European patent application No. 11861339.7 a European patent with the title and the supporting documents indicated in the communication pursuant to Rule 71(3) EPC (EPO Form 2004C) or in the information (EPO Form 2004W, cf. Notice from the EPO dated 8 June 2015, OJ EPO 2015, A52) dated 27.07.15 is hereby granted in respect of the designated Contracting States.

Patent No. : 2688399
Date of filing : 24.03.11
Priority claimed : /

Designated Contracting States and Proprietor(s) : AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO PL PT RO RS SE SI SK SM TR
Enviro Tech Chemical Services Inc.
500 Winmoore Way
Modesto, CA 95358/US

This decision will take effect on the date on which the European Patent Bulletin mentions the grant (Art. 97(3) EPC).

The mention of the grant will be published in European Patent Bulletin 15/45 of 04.11.15.

Examining Division

Zanobini A Sawicki M Bertrand F

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Registered letter
EPO Form 2006A 07.15 (01/10/15)

to EPO postal service: 02.10.15

ANMERKUNG ZUR ENTSCHEIDUNG ÜBER DIE ERTEILUNG
EINES EUROPÄISCHEN PATENTS (EPA Form 2006)

1. **EPA Informationsbroschüre "Nationales Recht zum EPÜ"**
Diese Broschüre enthält nützliche Informationen zu den formalen Erfordernissen und den Handlungen, die vor den Patentbehörden der Vertragsstaaten vorzunehmen sind, um Rechte in diesen Staaten zu erlangen. Da diese Handlungen einem ständigen Wandel unterworfen sind, sollte immer nur die neueste Ausgabe der Broschüre benutzt werden. Nachträgliche Informationen werden im Amtsblatt veröffentlicht.

2. **Übersetzung der europäischen Patentschrift nach Artikel 65 (1) des Europäischen Patentübereinkommens**
Sie werden erneut darauf hingewiesen, dass bestimmte Vertragsstaaten nach Artikel 65 (1) EPÜ eine Übersetzung der europäischen Patentschrift verlangen; hierauf wird in der Mitteilung gemäß Regel 71 (5) EPÜ verwiesen. Die Nichteinreichung dieser Übersetzung kann zur Folge haben, dass das Patent in dem betreffenden Staat/in den betreffenden Staaten als von Anfang an nicht eingetreten gilt. Weitere Einzelheiten entnehmen Sie bitte der oben genannten Broschüre.

3. **Zahlung von Jahresgebühren für europäische Patente**
Nach Artikel 141 EPÜ können "nationale" Jahresgebühren für das europäische Patent für die Jahre erhoben werden, die an das Jahr anschließen, in dem der Hinweis auf die Erteilung des europäischen Patents im "Europäischen Patentblatt" bekanntgemacht wird. Weitere Einzelheiten entnehmen Sie bitte der oben genannten Broschüre.

NOTE RELATING TO THE DECISION TO GRANT A
EUROPEAN PATENT (EPO Form 2006)

1. **EPO Information Brochure "National law relating to the EPC"**
This brochure provides useful information regarding formal requirements and the steps to be taken before the patent authorities of the Contracting States in order to acquire rights in those states. Since the necessary steps are subject to change the latest edition of the brochure should always be used. Subsequent information is published in the Official Journal.

2. **Translation of the European patent application under Article 65(1) of the European Patent Convention**
Your attention is again drawn to the requirements regarding translation of the European patent specification laid down by a number of Contracting States under Article 65(1) EPC, to which reference is made in the communication under Rule 71(5) EPC. Failure to supply such translation(s) may result in the patent being deemed to be void "ab initio" in the State(s) in question. For further details you are recommended to consult the above-mentioned brochure.

3. **Payment of renewal fees for European patents**
Under Article 141 EPC "national" renewal fees in respect of a European patent may be imposed for the years which follow that in which the mention of the grant of the European patent is published in the "European Patent Bulletin". For further details you are recommended to consult the above-mentioned brochure.

REMARQUE RELATIVE A LA DECISION DE DELIVRANCE
D'UN BREVET EUROPEEN (OEB Form 2006)

1. **Brochure d'information de l'OEB "Droit national relatif à la CBE"**
Cette brochure fournit d'utiles renseignements sur les conditions de forme requises et sur les actes à accomplir auprès des offices de brevet des Etats contractants aux fins d'obtenir des droits dans les Etats contractants. Etant donné que les actes indispensables sont susceptibles de modifications, il serait bon de toujours consulter la dernière édition de la brochure. Toute information ultérieure est publiée au Journal Officiel.

2. **Traduction du fascicule du brevet européen en vertu de l'article 65(1) de la Convention sur le brevet européen**
Votre attention est de nouveau attirée sur l'obligation faite par certains Etats contractants, en vertu de l'article 65(1) CBE, de fournir une traduction du fascicule du brevet européen, à laquelle il est fait référence dans la notification établie conformément à la règle 71(5) CBE. Si la(les) traduction(s) n'est(ne sont) pas fournie(s), le brevet européen peut, dès l'origine, être réputé sans effet dans cet(ces) Etat(s). Pour plus de détails, nous vous renvoyons à la brochure susmentionnée.

3. **Paiement des taxes annuelles pour le brevet européen**
Conformément à l'article 141 CBE des taxes annuelles "nationales" dues au titre du brevet européen peuvent être perçues pour les années suivant celle au cours de laquelle la mention de la délivrance du brevet européen est publiée au "Bulletin européen des brevets. Pour plus de détails, nous vous renvoyons à la brochure susmentionnée.

CLEAN CHEMISTRY EXHIBIT 1027
IPR2025-01458 – Page 4

BaileyWalsh&Co

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A. Tomkinson, MA (Cantab) PhD, CPA, EPA
Associates: P. Stephenson, MSci, PhD, MRSC, CPA, EPA
M. Kellett, MITMA
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28. Sep. 2015

BY FACSIMILE
00 4989 2399 4465

Zur Kasse

Our ref: AT-SAR-X11031-EP
Your ref:
21 September 2015

Dear Sirs

Re: European Patent Application No. 11861339.7- 1454 in the name of Enviro Tech Chemical Services, Inc. --Methods and compositions for the generation of peracetic acid on site at the point- of- use

In connection with the above, and further to your letter dated 27th July 2015, I advise you that the text as specified to be granted, enclosed with the Communication under Rule 71(3) EPC, is acceptable.

I enclose a copy of the claims in each of the French and German languages, and would you please deduct a remittance of €915.00 in respect of payment of the grant and printing fees from Account No. 28050202 before the due date of 27th November 2015.

Yours faithfully
BAILEY WALSH & CO LLP

A. Tomkinson

Enc

Also at 20 Bootham, York, YO30 7BL, UK **Tel:** 0800 783 7623
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Revendications

1. Procédé destiné à générer une solution en non équilibre d'acide peracétique, comprenant les opérations consistant à :

a. mettre à disposition de l'eau ;

b. introduire une solution de peroxyde d'hydrogène / triacétine dans l'eau ;

c. mélanger la solution de peroxyde d'hydrogène / triacétine et l'eau afin de former un mélange ;

d. ajouter, au mélange, une source aqueuse d'un hydroxyde de métal alcalin sous la forme d'hydroxyde de sodium, cas dans lequel l'hydroxyde de sodium représente 1,82 % à 7,28 % en poids, en outre, cas dans lequel le rapport molaire de l'hydroxyde de sodium au peroxyde d'hydrogène et à la triacétine est de 4,2 : 3,8 : 1 ; et

e. former un milieu réactif comprenant une solution en non équilibre d'acide peracétique.

2. Procédé selon la revendication 1, comprenant en outre, avant l'étape (b), une étape de mise à disposition de la solution de peroxyde d'hydrogène / triacétine comprenant les opérations consistant à : introduire de la triacétine à une solution de peroxyde d'hydrogène aqueux ; et permettre à la triacétine et à la solution de peroxyde d'hydrogène aqueux de se mélanger afin de former la solution de peroxyde d'hydrogène / triacétine.

3. Procédé selon la revendication 1, l'eau fournie étant un flux aqueux.

4. Procédé selon la revendication 1, l'eau fournie étant contenue dans une cuve de mélangeage ou un autre récipient.

5. Procédé selon la revendication 1, l'étape (d) étant effectuée en simultanéité avec l'étape (b).

6. Procédé selon la revendication 1, comprenant en outre l'opération consistant à

laisser au milieu réactif une durée suffisante pour convertir le peroxyde d'hydrogène / triacétine en acide peracétique, ladite durée étant de 30 secondes à cinq minutes.

7. Procédé selon la revendication 1, comprenant en outre, après l'étape (e), une étape d'échantillonnage du milieu réactif afin de déterminer la durée requise pour optimiser la quantité de peroxyde d'hydrogène / triacétine qui est convertie en acide peracétique.

8. Procédé selon la revendication 1, la solution d'acide peracétique étant destinée à une utilisation immédiate.

9. Procédé selon la revendication 1, comprenant en outre, après l'étape (e), une étape d'adjonction d'une source d'acide pour stabiliser la solution en non équilibre d'acide peracétique en vue d'un stockage et d'une utilisation tout au long d'une journée de travail.

10. Procédé selon la revendication 1, la solution d'acide peracétique étant préparée sur un site équipé d'un point d'utilisation.

Patentansprüche

1. Verfahren zum Erzeugen einer Nichtgleichgewichtslösung von Peressigsäure, das Folgendes beinhaltet:

a. Bereitstellen von Wasser;

b. Einleiten einer Wasserstoffperoxid-Triacetin-Lösung in das Wasser;

c. Mischen von Wasserstoffperoxid-Triacetin-Lösung und Wasser, um ein Gemisch zu bilden;

d. Zugeben einer wässrigen Quelle eines Alkalimetallhydroxids in Form von Natriumhydroxid zu dem Gemisch, wobei das Natriumhydroxid 1,82 bis 7,28 Gew.-% ausmacht, wobei ferner das Molverhältnis zwischen Natriumhydroxid und Wasserstoffperoxid und Triacetin 4,2:3,8:1 beträgt, und

e. Bilden eines Reaktionsmediums, das eine Nichtgleichgewichtslösung von Peressigsäure beinhaltet.

2. Verfahren nach Anspruch 1, das ferner vor Schritt (b) einen Schritt des Bereitstellens der Wasserstoffperoxid-Triacetin-Lösung beinhaltet, der Folgendes beinhaltet: Einleiten von Triacetin in eine Lösung von wässrigem Wasserstoffperoxid; und Mischenlassen des Triacetins und der Lösung von wässrigem Wasserstoffperoxid, um die Wasserstoffperoxid-Triacetin-Lösung zu bilden.

3. Verfahren nach Anspruch 1, wobei das bereitgestellte Wasser ein wässriger Strom ist.

4. Verfahren nach Anspruch 1, wobei das bereitgestellte Wasser in einem Mischbehälter oder einem anderen Gefäß enthalten ist.

5. Verfahren nach Anspruch 1, wobei Schritt (d) gleichzeitig mit Schritt (b) ausgeführt wird.

6. Verfahren nach Anspruch 1, das ferner beinhaltet, dass dem Reaktionsmedium ausreichend Zeit zum Umwandeln des Wasserstoffperoxid-Triacetins in Peressigsäure gelassen wird, wobei die genannte Zeit 30 Sekunden bis fünf Minuten ist.

7. Verfahren nach Anspruch 1, das ferner nach Schritt (e) einen Schritt des Beprobens des Reaktionsmediums beinhaltet, um die Zeit zu ermitteln, die notwendig ist, um die Menge von Wasserstoffperoxid-Triacetin zu maximieren, das in Peressigsäure umgewandelt wird.

8. Verfahren nach Anspruch 1, wobei die Peressigsäurelösung für den sofortigen Gebrauch vorgesehen ist.

9. Verfahren nach Anspruch 1, das ferner nach Schritt (e) einen Schritt des Zugebens einer Säurequelle zum Stabilisieren der Nichtgleichgewichtslösung von Peressigsäure zum Lagern und Verwenden im Laufe eines Arbeitstages beinhaltet.

10. Verfahren nach Anspruch 1, wobei die Peressigsäurelösung an einem Ort mit einer Verwendungsstelle hergestellt wird.

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Patent application/patent No. (please use a separate form for each application)

EP 11861339.7-1454

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055	Additional copy of docs cited in search report		036	Renewal fee for the 6th year	
015	Claims fee(s) (Rules 45(1), 162(1)EPC)[5]			Validation for[8]	
005	Designaton fee(s)[4]				
006	Examination fee				
122	Fee for further processing (non-fee related cases)				
123	Fee for further processing (late payment of a fee)			Extension for[8]	
007	Fee for grant and printing or fee for grant incl. fee for publcation[6]	915.00			
008	Additional printing fee for 36th and each subsequent page[7]				
016	Claims fee according to Rule 71(6) EPC[5]				
121	Fee for further processing (late performance of acts R.71(3))				
022	Registering of transfer				

Notes 1 - 9 see overleaf.

Total 915.00

Signature (handwritten / no block letters)
Bailey Walsh & Co LLP

Place, Date
Leeds, England, 21st September 2015

EPO 1010 04.14

Bailey &

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M. Kellett, MITMA
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BY FACSIMILE
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Our ref: AT-SAR-X11031-EP
Your ref:
21 September 2015

Dear Sirs

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Yours faithfully
BAILEY WALSH & CO LLP

A. Tomkinson

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Duration: 21.09.2015 10:15:20 - 21.09.2015 10:16:03. This page 1 of 6 was completed at 21.09.2015 10:15:26
Received at the EPO on Sep 21, 2015 10:16:03. Page 1 of 6

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Patent application/patent No. (please use a separate form for each application)

EP 11861339.7-1454

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520	Additional fee (more than 35 pages) – entry EP phase		033	Renewal fee for the 3rd year	
	Additional fee for divisional applications (Rule 38(4)EPC)[3]		034	Renewal fee for the 4th year	
002	Fee for a European search		035	Renewal fee for the 5th year	
055	Additional copy of docs cited in search report		036	Renewal fee for the 6th year	
015	Claims fee(s) (Rules 45(1), 162(1)EPC)[3]			Validation for[8]	
005	Designaton fee(s)[4]				
006	Examination fee				
122	Fee for further processing (non-fee related cases)				
123	Fee for further processing (late payment of a fee)			Extension for[8]	
007	Fee for grant and printing or fee for grant incl. fee for publication[6]	915.00			
008	Additional printing fee for 36th and each subsequent page[7]				
016	Claims fee according to Rule 71(6) EPC[5]				
121	Fee for further processing (late performance of acts R.71(3))				
022	Registering of transfer				

Notes 1 - 9 see overleaf.

Total 915.00

Signature (handwritten/no block letters)
Bailey Walsh & Co LLP

Place, Date
Leeds, England, 21st September 2015

EPO 1010 04.14

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Payer's reference: AT-SAR-X11031-EP

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Patent application/patent No. (please use a separate form for each application)

EP 11861339.7-1454

PCT

Code		Amount/EUR	Code		Amount/EUR
001	Filing fee – EP direct[3]		010	Opposition fee	
501	Additional fee (more than 35 pages)		011	Fee for appeal	
020	Filing fee – entry EP phase[3] (Rule 159(1)(c) EPC)		029	Certified copy of application; priority document	
520	Additional fee (more than 35 pages) – entry EP phase		033	Renewal fee for the 3rd year	
	Additional fee for divisional applications (Rule 36(4)EPC)[3]		034	Renewal fee for the 4th year	
002	Fee for a European search		035	Renewal fee for the 5th year	
055	Additional copy of docs cited in search report		036	Renewal fee for the 6th year	
015	Claims fee(s) (Rules 45(1), 162(1)EPC)[3]			Validation for[5]	
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Notes 1 - 9 see overleaf.

Total: 915.00

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Annex to EPO Form 2004, Communication pursuant to Rule 71(3) EPC

Bibliographical data of European patent application No. 11 861 339.7

For the intended grant of the European patent, the bibliographical data are set out below, for information:

Title of invention:	- VERFAHREN UND ZUSAMMENSETZUNGEN ZUR ERZEUGUNG VON PERESSIGSÄURE DIREKT AM VERWENDUNGSORT - METHODS AND COMPOSITIONS FOR THE GENERATION OF PERACETIC ACID ON SITE AT THE POINT-OF-USE - PROCÉDÉS ET COMPOSITIONS POUR LA PRODUCTION D'ACIDE PERACÉTIQUE SUR UN SITE AU POINT D'UTILISATION
Classification:	INV. A01P1/00 A61K31/327 A61L2/18 A01N37/16
Date of filing:	24.03.2011
Priority claimed:	
Contracting States* for which fees have been paid:	AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO PL PT RO RS SE SI SK SM TR
Extension States* for which fees have been paid:	
Validation States* for which fees have been paid:	
Applicant(s):**	Enviro Tech Chemical Services Inc. 500 Winmoore Way Modesto, CA 95358 US
Inventor(s):	HARVEY, Michael, S. 712 Faust Road Modesto, CA 95358 US HOWARTH, Jonathan, N. 1848 Scenic Drive Modesto, CA 95355 US

*) If the time limit for the payment of designation fees according to Rule 39(1) EPC has not yet expired and the applicant has not withdrawn any designation, **all Contracting States/Extension States/Validation States** are currently still deemed to be designated.

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	See also Rule 71a(3) EPC and, if applicable, the above Note to users of the automatic debiting procedure.
**)	If two or more applicants have designated different Contracting States, this is indicated here.

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Application No.	Ref.	Date
11 861 339.7 - 1454	ATSAR-X11031-EP	27.07.2015
Applicant Enviro Tech Chemical Services Inc.		

Communication under Rule 71(3) EPC

1. Intention to grant

You are informed that the examining division intends to grant a European patent on the basis of the above application, with the text and drawings and the related bibliographic data as indicated below.

A copy of the relevant documents is enclosed.

1.1 In the text for the Contracting States:
AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO PL PT RO RS SE SI SK SM TR

Description, Pages

1-7, 9-15, 17-21, 23-41, 45-47	as published	
8, 8b	filed with telefax on 15-09-2014	
8a, 16, 22, 42-44, 48	filed in electronic form on	26-05-2015

Claims, Numbers

1-10	filed in electronic form on	26-05-2015

With the following amendments to the above-mentioned documents according to your request dated 26-05-2015: (2)

Description, Pages

Delete Previous: 49-59

With the following amendments to the above-mentioned documents proposed by the division

Description, Pages 1, 8, 10-12, 16, 18-21, 24, 25, 28, 29, 31, 43

Comments

DESCRIPTION
Page 1: Description adapted to amended claims (Art. 84 EPC)
Page 8: Description adapted to amended claims (Art. 84 EPC)
Page 10: "about"
Page 11: Description adapted to amended claims (Art. 84 EPC)
Page 12: Description adapted to amended claims (Art. 84 EPC)
Page 16: about
Page 24: about
Page 25: about
Page 28: about
Page 31: about

1.2 Bibliographic data

The title of the invention in the three official languages of the European Patent Office, the international patent classification, the designated contracting states, the registered name(s) of the applicant(s) and the other bibliographic data are shown on **EPO Form 2056** (enclosed).

2. Invitation

You are invited, **within a non-extendable period of four months** of notification of this communication,

2.1 to EITHER approve the text communicated above and verify the bibliographic data (Rule 71(5) EPC)

(1) by filing a translation of the claim(s) in the other two official languages of the EPO

	Fee code	EUR
(2a) by paying the fee for grant including the fee for publication:	**007**	915.00
minus any amount already paid (Rule 71a(5) EPC):		0.00
	Total amount:	915.00
(3) by paying additional claims fees under Rule 71(4) EPC; number of claims fees payable: 0	**016**	0.00
minus any amount already paid (Rule 71a(5) EPC):		0.00
	Total amount:	0.00

Important: If the translations of the claims and fees have already been filed and paid respectively in reply to a previous communication under Rule 71(3) EPC, e.g. in the case of resumption of examination after approval (see Guidelines C-V, 6), **agreement as to the text to be granted** (Rule 71a(1) EPC) must be expressed within the same time limit (e.g. by approving the text and verifying the bibliographic data, by confirming that grant proceedings can go ahead with the documents on file and/or by stating which translations of the claims already on file are to be used).

Note 1: See "Important notes concerning fee payments" below.

Note 2: Any overpaid "minus" amounts will be refunded when the decision to grant (EPO Form 2006A) has been issued.
Note 3: For the calculation of the grant fee under Article 2(2), No. 7, RFees (old fee structure), the number of pages is determined on the basis of a clean copy of the application documents, in which text deleted as a result of any amendments by the examining division is not shown. Such clean copy is made available via on-line file inspection only.

2.2 OR, in the case of disapproval, to request reasoned amendments or corrections to the text communicated above or keep to the latest text submitted by you (Rule 71(6) EPC).

In this case the translations of the claims and fee payments mentioned under point 2.1 above are NOT due.

The terms "amendment(s)" and "correction(s)" refer only to amendments or corrections of the application documents and not of other documents (e.g. bibliographic data, the designation of the inventor, etc.).

If filing amendments, you must identify them and indicate the basis for them in the application as filed. Failure to meet either requirement may lead to a communication from the examining division requesting that you correct this deficiency (Rule 137(4) EPC).

2.3 Bibliographic data

Where you request a change or correction of bibliographic data in response to the Rule 71(3) communication, this will **not** cause the sending of a further communication under Rule 71(3) EPC. You will still have to pay the fees and file translations in reply to the Rule 71(3) communication in the case of 2.1 above, unless you also file a reasoned request for amendments or corrections in response to the Rule 71(3) communication (see case 2.2 above).

3. Loss of rights

If neither of the two possible actions above (see points 2.1 or 2.2) is performed in due time, the European patent application will be deemed to be withdrawn (Rule 71(7) EPC).

4. Further procedure

4.1 In the case of point 2.1 above

4.1.1 The decision to grant the European patent will be issued, and the **mention of the grant** of the patent will be published in the European Patent Bulletin, if the requirements concerning the translation of the claims and the payment of all fees are fulfilled and there is agreement as to the text to be granted (Rule 71a(1) EPC).

Note on payment of the renewal fee:
If a renewal fee becomes due before the next possible date for publication of the mention of the grant of the European patent, publication will be effected only after the renewal fee and any additional fee have been paid (Rule 71a(4) EPC).

Under Article 86(2) EPC, the obligation to pay renewal fees to the European Patent Office terminates with the payment of the renewal fee due in respect of the year in which the mention of the grant of the European patent is published.

Note on payment of the designation fee(s):
If the designation fee(s) become(s) due after the communication under Rule 71(3) EPC, the mention of the grant of the European patent will not be published until these fees have been paid (Rule 71a(3) EPC).

4.1.2 After publication, the **European patent specification** can be downloaded free of charge from the EPO publication server https://data.epo.org/publication-server or ordered from the Vienna sub-office upon payment of a fee (OJ EPO 2005, 126).

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4.1.3 Filing of translations in the contracting states

As regards translation requirements prescribed by the contracting states under Article 65(1) EPC, please consult the website of the European Patent Office
www.epo.org ➞Law & practice ➞Legal texts, National law relating to the EPC
www.epo.org ➞Law & practice ➞All Legal texts ➞London Agreement

In the case of a valid extension or validation

As regards translation requirements prescribed by the extension or validation states, please consult the website of the European Patent Office
www.epo.org ➞Law & practice ➞Legal texts, National law relating to the EPC

Failure to supply a prescribed translation in a contracting state, or in an extension or validation state may result in the patent being deemed to be void *ab initio* in the state concerned (Art. 65(3) EPC).

4.2 In the case of 2.2 above

If the present communication under Rule 71(3) EPC is based on an auxiliary request and, within the time limit, you maintain the main request or a higher ranking request which is not allowable, the application will be refused (Art. 97(2) EPC).

If the examining division gives its consent to the requested amendments or corrections, it will issue a new communication under Rule 71(3) EPC; otherwise, it shall resume the examination proceedings (Rule 71(6) EPC).

5. Filing of a divisional application

Any divisional application relating to this European patent application must be filed directly with the European Patent Office in Munich, The Hague or Berlin and will be in the language of the proceedings for the present application, or if the latter was not in an official language of the EPO, the divisional application may be filed in the language of the present application as filed (see Article 76(1) and Rule 36(2) EPC). Any such divisional application must be filed while the present application is still pending (Rule 36(1) EPC; Guidelines A-IV, 1.1.1).

6. Important notes concerning fee payments

6.1 For all payments, please refer to the relevant **fee code(s).**

6.2 Automatic debiting procedure

The fee for grant, including the fee for publication, and any additional claims fees due under Rule 71(4) EPC will be debited automatically on the date of filing of the translations of the claims, or on the last day of the period of this communication. However, if the designation fee(s) become(s) due as set out in Rule 71a(3) EPC and/or a renewal fee becomes due as set out in Rule 71a(4) EPC, these should be paid separately by another permitted way of payment in order not to delay the publication of the mention of the grant. The same applies in these circumstances to the payment of extension and validation fees. For further details see the Arrangements for the automatic debiting procedure (AAD) and accompanying information from the EPO concerning the automatic debiting procedure (Annexes A.1 and A.2 to the Arrangements for deposit accounts (ADA) in Supplementary publication - OJ EPO 3/2015).

Note: If a waiver is expressed in response to a Rule 71(3) communication (see OJ EPO 2015, A52), the fee for grant, including the fee for publication/printing, and any additional claims fees will not be debited automatically. These fees must be paid separately by another means of payment allowed under the Rules relating to Fees.

6.3 Important information relating to fee amounts

Following any amendment to the Rules relating to Fees, the amount(s) mentioned in this communication may be different from the amount(s) **actually due on the date of payment.** The latest version of the Schedule of

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fees and expenses, published as a Supplement to the Official Journal of the EPO, is also available on the EPO website (www.epo.org) and can be found under www.epo.org/schedule-of-fees, which allows the viewing, downloading and searching for individual fee amounts, both current and previous.
Please note that procedural fees are usually adjusted every two years, on even years, with effect from 1 April.

Examining Division:

Chairman:	Bertrand, Franck
2nd Examiner:	Sawicki, Marcin
1st Examiner:	Zanobini, Alessandra

Heim, Patrick
For the Examining Division
Tel. No.: +49 89 2399 - 6070

Enclosures: Text intended for grant

EPO Form 2056

Europäisches Patentamt
European Patent Office
Office européen des brevets

European Patent Office
80298 MUNICH
GERMANY
Tel: +49 89 2399 0
Fax: +49 89 2399 4465

Application No.: 11 861 339.7

IV.2. Patent classification

The classification has been changed. It now is as follows:

INV. A01P1/00 A61K31/327 A61L2/18 A01N37/16

IV.3. Title of the invention

The title indicated on the published patent application remains unchanged. It reads as follows:

VERFAHREN UND ZUSAMMENSETZUNGEN ZUR ERZEUGUNG VON PERESSIGSÄURE DIREKT AM VERWENDUNGSORT

METHODS AND COMPOSITIONS FOR THE GENERATION OF PERACETIC ACID ON SITE AT THE POINT-OF-USE

PROCÉDÉS ET COMPOSITIONS POUR LA PRODUCTION D'ACIDE PERACÉTIQUE SUR UN SITE AU POINT D'UTILISATION

IV.4. Documentation

19/07/16
Date

Bertrand, Franck
Chairman

Zanobini, Alessandra
1st examiner

Sawicki, Marcin
2nd examiner

Enclosure(s):

METHODS AND COMPOSITIONS FOR THE GENERATION OF PERACETIC ACID ON SITE AT THE POINT-OF-USE

Michael S. Harvey and Jonathan N. Howarth

FIELD OF THE INVENITON

[0001] The invention relates to liquid compositions and methods of using the liquid compositions for the generation of a peroxyacetic acid sanitizer on site in proximity to the point-of-use.

BACKGROUND OF THE INVENTION

[0002] **A. Liquid PAA Disinfectants and Sanitizers**

[0003] Disinfectants and sanitizers based on peroxyacetic acid (PAA), also commonly known as peracetic acid, are used in the dairy, food and beverage processing industries for clean-in-place pipeline and equipment disinfecting and cleaning, for fruit and vegetable washing, and in the treatment of meat, poultry and seafood products. Peroxyacetic acid disinfectants are also used in the treatment of cooling water, process water, and municipal wastewater. Other uses include slime and biofilm removal in papermaking processes.

[0004] Peroxyacetic acid products are supplied as stable equilibrium ternary aqueous solutions of peroxyacetic acid, acetic acid, and hydrogen peroxide. They are prepared in advance of delivery, typically by reacting hydrogen peroxide with acetic acid in the presence of a mineral acid catalyst. Although some PAA is formed immediately, the PAA does not reach its maximum concentration until after several days. A metal chelating agent, such as hydroxyethylidene

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diphosphonic acid (HEDP) or dipicolinic acid, is also introduced to suppress the transition metal cation catalyzed decomposition of peroxygen compounds. The PAA product is then placed in containers, such as totes, drums and pails, in preparation for shipment to the end user. Typical commercial products have concentrations of PAA of about 1-15% w/w, although a concentration of up to 30% is possible. However, the latter concentration product only finds captive use because of its extremely hazardous and explosive properties.

[0005] There are several problems with the use of equilibrium solutions of PAA. First, the low concentration of PAA (1-15%) means that most of the weight (85-99%) of the products consist of substantial amounts of inert ingredients, such as water, acetic acid, and hydrogen peroxide. This results in the need for larger product storage areas and causes increased transportation and handling costs. Second, the products are inefficient in the use of both hydrogen peroxide (hydrogen peroxide) and acetic acid (AA). To maintain adequate storage stability of the PAA, either hydrogen peroxide or AA must be present at a level on a weight percentage basis that is greater than the level of PAA, which increases raw material costs. Third, the presence of the HEDP or dipicolinic acid stabilizer limits the amount of PAA that can be applied to certain foodstuffs because the amount of HEDP or dipicolinic acid is regulated by the Food and Drug Administration (FDA). Fourth, the reaction between hydrogen peroxide and AA is quite slow in some cases, and typically requires several days' time before the PAA product can be tested for quality confirmation. Therefore, manufacturers of these products must store large inventories of the PAA product before it can be shipped to the end user. Fifth, for transportation purposes, equilibrium solutions that are greater than 6% PAA are considered to be dangerous products and must be labeled with the DOT marking "Organic Peroxide", Hazard Class 5.2, 8 (oxidizer, corrosive). If producers and end-users exceed the yearly threshold amount of just 6,666 lbs. of 15% PAA, they must file Risk Management Plans with both federal EPA and state

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authorities. This is an arduous and time-consuming process. In addition, producers of equilibrium solutions of PAA that are over 6% PAA must obtain permitting by the local fire department and pay an extra hazardous material shipping fee when the product is shipped from their facility. On the other hand, there are much less stringent requirements for products that contain hydrogen peroxide, and all reporting, permitting and shipping restrictions are lifted for products that contain less than 27% hydrogen peroxide.

[0006] As a result of these problems, there have been various attempts to make non-equilibrium solutions of PAA on site, at the point-of-use. For example, U.S. patent no. 7,012,154 discloses a system in which AA, hydrogen peroxide, water, and sulfuric acid are fed to a jacketed reactor for the production of PAA. A wiped-film distillation column attached to the reactor condenses and isolates the pure PAA from the gas phase and immediately introduces it to the receiving water. This system suffers from a number of drawbacks. First, it is extremely capital intensive due to the high cost of the equipment, including the reactor, heater, pumps, distillation column, and computerized control system which ensures accurate metering of the reagents. Second, there are significant safety hazards associated with the production of pure PAA due to its explosive properties. Third, the equipment and synthesis process is very complex and requires knowledgeable and highly trained technicians to continuously operate and maintain the system in a safe and effective manner.

[0007] Other attempts to make non-equilibrium solutions of PAA at the point-of-use are based on electrolytic processes. U.S. patent nos. 6,171,551 and 6,387,236 disclose processes that employ a cell divided by an ion-exchange membrane in which PAA (and other oxidants including hydrogen peroxide and ozone) are produced in the anode compartment which consists of an aqueous solution of acetic acid or acetate salt. In addition to the high costs of electricity and electrolysis equipment, these processes also result in a very low yield of PAA from the

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acetyl precursor. For example, the '551 patent reports that less than 14 ppm of PAA was produced after 90 minutes of electrolysis of a 490,000 ppm anolyte of aqueous potassium acetate. In addition, these processes are difficult to perform intermittently.

[0008] Other methods of generating non-equilibrium solutions of PAA on site, using electrolysis, are described in WIPO International Publication Nos. WO 2004/0245116 and WO 2008/140988, and U.S. Patent Application Publication No. 2009/0314652. These references disclose cation membrane-divided electrolysis cells and the use of gas diffusion electrodes to effect the cathodic reduction of oxygen gas to hydrogen peroxide under alkaline conditions. The hydrogen peroxide was then allowed to react with acetic acid or an acetyl precursor to form PAA in the bulk solution, whereupon the catholyte was directed to the acidic anode compartment of the cell to stabilize the PAA. This system suffers from several disadvantages. Due to the low solubility of oxygen in water (about 8 ppm maximum), the concentration of electroactive species is very low, which forces the cell to operate at low current density (amperage per surface area of electrode). In order to produce a meaningful amount of hydrogen peroxide, the cells must have a very large surface area. This requires high capital equipment costs and a very large footprint for the electrolysis equipment. Another disadvantage of this system is that it is very difficult to maintain steady-state conditions and simultaneously balance the feed of acetic acid or acetyl precursor to the cathode compartment with the concurrent withdrawal of acidified PAA solution from the anode compartment. This is because cations carrying the cell current through the cation exchange membrane are always hydrated so as the cations move through the membrane, they are accompanied by water molecules. As a result, the volume of the anolyte decreases and the volume of the catholyte increases, making the steady-state condition difficult to maintain. It is difficult to perform this process intermittently. Finally, this system can only be of economic value if the source of oxygen is air, which comprises 23% oxygen. However, the carbon dioxide

contained in air causes carbonates to precipitate, which impedes the flow of electrical current, limiting or eliminating the production of hydrogen peroxide, and hence, PAA.

[0009] Another method of making a non-equilibrium solution of PAA at the point-of-use is disclosed in WIPO International Publication No. WO 2008/140988 and U.S. Patent Application Publication Nos. 2009/0005590 and 2007/0082832. These references disclose biosynthetic methods of producing peracids from carboxylic acids and carboxylic acid esters. These methods involve the use of perhydrolase enzymes to catalyze the perhydrolysis of the carboxylic acid or ester into the peracid using a solid or liquid source of hydrogen peroxide. Although these methods can produce compositions containing up to 20 parts of peracid to one part of hydrogen peroxide, they are limited by the amount of peracid that can be produced before the perhydrolase enzyme is oxidized by the reactant products and ceases to function as intended. The highest concentration of peracid disclosed by these references was 0.16% (1,600 mg/L). However, peracid or PAA solutions that are produced with enzymes have limited appeal because they are very expensive to produce, and for regulatory reasons, the enzyme must be removed from the solution before the solution can be applied to food or hard surfaces for disinfection purposes. Removing the enzyme from the solution is not an easy task; thus, it is typically not done. Therefore, PAA solutions prepared by enzymatic methods are not suitable for the more broad commercial uses in the food, dairy, beverage, meat, and poultry industries, which are regulated by the FDA and the Environmental Protection Agency (EPA).

[0010] Another method of generating non-equilibrium PAA at its point-of-use requires substituting the traditional mineral acid catalyst with sulfonic acid ion-exchange resins, as disclosed in U.S. patent no. 5,122,538. A solution containing a 1.5:1 mole ratio of AA to hydrogen peroxide was passed through a column packed with a sulfonic acid ion-exchange resin and produced a solution of 15% PAA within 30 minutes. The method described in the '538

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patent suffers from the limitation that it requires a large volume of expensive resin bed in order to be effective. Moreover, all existing ion exchange resin systems are limited by the fact the resin is subject to oxidative degradation by PAA and have a short limited lifespan.

[0011] Other attempts to produce non-equilibrium PAA solutions on site, at the point-of-use, for bleaching cellulosic materials have reacted hydrogen peroxide with acetic anhydride. For example, U.S. patent no. 3,432,546 discloses a process where hydrogen peroxide, acetic anhydride, and an ammonium hydroxide catalyst were metered to a tubular reactor to continuously produce a solution containing 3.25% PAA with a conversion of 78% hydrogen peroxide. However, the process generated measurable amounts of diacetyl peroxide (0.44%) which is an explosion hazard. Moreover, the reaction product would be unsuited for any application other than cellulosic bleaching purposes because there was no attempt to remove the ammonium hydroxide catalyst from the reaction medium. Ammonium hydroxide is an undesirable contaminant in PAA products that are used as disinfectants and sanitizers in the dairy, food, and beverage processing industries, and in PAA products used in fruit and vegetable washing and in the treatment of meat, poultry, and seafood.

[0012] Another process for generating non-equilibrium solutions of PAA on site, at the point-of-use was disclosed in U.S. Patent Application Publication No. 2009/0043132. This process utilized introduction of hydrogen peroxide into a sidestream of the water requiring treatment. This was followed by introducing acetic anhydride, whereupon PAA was generated *in-situ*. It was found that acetic anhydride preferentially reacted with hydrogen peroxide rather than undergo undesirable hydrolysis with water. Within 20 minutes, up to 3000 ppm of PAA was generated in the sidestream which was then reconstituted with the main body of water and diluted further. All processes that employ acetic anhydride suffer the limitation that acetic anhydride is expensive, very corrosive, an irritant, and highly flammable.

[0013] Yet another process for generating non-equilibrium solutions of PAA on site at the point-of-use is disclosed in WIPO International Publication No. WO 01/46519 A1. This process utilized the metering of an aqueous solution of hydrogen peroxide into an agitated tank and co-metering a solid dry source of tetraacetylethylenediamine (TAED) from a storage hopper using a screw feeder. The agitator kept the solid TAED suspended in the hydrogen peroxide solution which was then fed to an in-line static mixer where aqueous sodium hydroxide was introduced. The mixture was then directed through 200 meters of coiled tubing immersed in a cooling tank so that the temperature rise accompanying the exothermic reaction was contained. Upon exiting the coiled tubing, the mixture containing PAA could be directed to the water requiring treatment. Disadvantages of this approach include the difficulty of accurately metering a solid and a liquid simultaneously, and the high capital equipment cost of the metering system, electronic controllers, agitation tank, coiled tubular reactor, and the cooling system.

[0014] Thus, there is a need for a method to make non-equilibrium PAA on site at the point-of-use that addresses the above problems.

[0015] **B. Solid PAA Bleaches and Stain Removers**

[0016] PAA is also used in laundry bleaching applications where it is generated *in-situ* in the laundry wash water. The PAA is typically produced from a solid source of hydrogen peroxide, such as sodium percarbonate or sodium perborate. The hydrogen peroxide must be in the presence of a solid acetyl precursor, most typically, tetraacetylethylenediamine (TAED). When dissolved in laundry wash water, the TAED undergoes a perhydrolysis reaction with hydrogen peroxide to form PAA. TAED is the preferred acetyl precursor because it possesses low toxicity, is of low environmental impact, and is readily biodegradable. However, TAED-based laundry bleaches have several problems. First, of the four acetyl groups on TAED, only two are known to be available for perhydrolysis, making TAED an expensive acetyl precursor on

a weight basis. Second, TAED has low water solubility, especially at the cooler water temperature bleaching cycles that are less damaging to fabrics. This is a major drawback, as current consumer trends in energy conservation are towards bleaching laundry fabrics with cool temperature water, rather than heated water. Undissolved TAED in cool temperature water is less unavailable as an acetyl precursor for the dissolved source of hydrogen peroxide and can even deposit on fabrics, necessitating a separate rinse step to remove it. Third, over time, when exposed to high humidity, solid TAED can react with solid source of hydrogen peroxide and the free water to form PAA, as well as degrade the activity, making it less effective overtime. Because the PAA is volatile, it imparts an undesirable pungent odour to the product.

EP1618786 discloses a composition for bactericide production and a process for producing organic peracid. The sterilising agent is a water based solution obtained by combining triacetin and hydrogen peroxide. The ratio between these two components is 1/10 and 20/1. The formulation can comprise 50% by weight triacetin, 15% of hydrogen peroxide, 0.17% hydrogen peroxide and 57.3% of remaining peroxide.

JP2004230323 discloses a sterilising agent for hard surfaces. Triacetin is used as the agent capable of liberating acetic acid, hydrogen peroxide is added and a metal hydroxide has to be used to bring the pH to 8-12. One example discloses a formulation of 0.23mol of triacetin, 0.73mol of hydrogen peroxide and 0.5 mol of sodium hydroxide.

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US2010/0227000 discloses a multi-part kit system for the preparation of a disinfectant of the peracetic acid type comprising a solid part A comprising at least one acetyl donor, a liquid part B which can be an aqueous solution of hydrogen peroxide (5-10% by wt). A water solution of 0.05 to 5% sodium hydroxide is also added.

US6221341 discloses a teeth whitening composition. Peracetic acid is the whitening agent and is generated by glyceryl triacetate with the addition of hydrogen peroxide in the presence of a base the regulates the pH.

SUMMARY OF THE PRESENT INVENTION

According to the present invention there is provided a method of generating a non-equilibrium solution of peracetic acid, comprising:

a. providing water;

b. introducing a hydrogen peroxide-triacetin solution to the water;

c. mixing the hydrogen peroxide-triacetin solution and the water to form a mixture;

d. adding an aqueous source of an alkali metal hydroxide in the form of sodium hydroxide to the mixture, wherein the sodium hydroxide is 1.82% to 7.28% weight and the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is 4.2:3.8:1; and

e. forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

In one embodiment, liquid compositions for use in generating non-

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equilibrium solutions of PAA are provided. These hydrogen peroxide-acetyl precursor solutions comprise a solution of aqueous hydrogen peroxide, a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide, a trace of peracetic acid, and water. A preferable acetyl precursor is triacetin, which displays an unexpectedly high solubility in hydrogen peroxide. These hydrogen peroxide-acetyl precursor solutions may be used for generating a non-equilibrium solution of peracetic acid for use as a disinfectant or sanitizer on a site having a point-of-use of peracetic acid.

The hydrogen peroxide- acetyl precursor solutions are advantageous in that they are formulated such that sufficient acetyl precursor can be dissolved in an amount of the hydrogen peroxide that would fully perhydrolyze all of the acetyl groups of the acetyl precursor.

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Moverover, the hydrogen peroxide component of these compositions is remarkably stable. These compositions overcome one or more problems of the known prior art, and are free of the regulatory reporting, permitting, and shipping restrictions that govern equilibrium PAA products.

[0019] In another embodiment, a method of preparing a hydrogen peroxide-acetyl precursor solution is provided. The method comprises introducing a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide to a solution of aqueous hydrogen peroxide; and allowing the acetyl precursor and the aqueous hydrogen peroxide to mix to form a hydrogen peroxide-acetyl precursor solution.

[0020] A further embodiment is a method of continuously or intermittently generating a non-equilibrium solution of peracetic acid on a site having a point-of-use of peracetic acid for use as a disinfectant or sanitizer. Water is provided on the site having the point-of-use. The water may be a flowing aqueous stream or it may be contained in a mixing tank or other vessel. A hydrogen peroxide-acetyl precursor solution is provided on the site having the point-of-use. In one embodiment, the liquid acetyl precursor is triacetin and the solution that is formed is a hydrogen peroxide-triacetin solution. The hydrogen peroxide-acetyl precursor solution may be prepared off site and then transported to the site having the point-of-use, or the acetyl precursor and the aqueous hydrogen peroxide may be transported to the site having the point-of-use and then used to prepare the hydrogen peroxide-acetyl precursor solution on the site. The hydrogen peroxide-acetyl precursor solution is then introduced to the water. Alternatively, and in another embodiment, the acetyl precursor and the aqueous hydrogen peroxide may be introduced to the water separately, either sequentially, with either one first, or simultaneously. The hydrogen peroxide-acetyl precursor solution and the water are mixed to form a mixture. An aqueous source of an alkali metal or earth alkali metal hydroxide, such as sodium hydroxide, is provided on the site having the point-of-use. The aqueous source of an alkali metal or earth alkali metal

hydroxide is added to the mixture. Alternatively, and in another embodiment, the aqueous source of alkali metal or earth alkali metal hydroxide may be added simultaneously with the hydrogen peroxide-acetyl precursor solution or with the acetyl precursor and the aqueous hydrogen peroxide, to the water. A reaction medium comprising a non-equilibrium solution of peracetic acid is formed. In the reaction medium, the hydrogen peroxide reacts with the acetyl precursor to form peracetic acid. Almost instantaneously, a non-equilibrium solution of PAA is formed. Within 30 seconds to five minutes, the amount of hydrogen peroxide and acetyl precursor that are converted into peracetic acid is maximized. When triacetin is used as the acetyl precursor and assuming all three acetyl groups are reacted, 40.9% to 85.7% of the triacetin is converted into peracetic acid, and the percent of hydrogen peroxide remaining is 0.078% to 1.88%. The reaction medium may optionally be sampled at various times after the addition of the aqueous source of alkali metal or earth alkali metal hydroxide to determine the time required to maximize the amount of hydrogen peroxide and acetyl precursor that are converted into peracetic acid.

[0021] The non-equilibrium solutions of PAA that are formed comprise PAA, unreacted hydrogen peroxide, unreacted acetyl precursor, the product of the perhydrolysis reaction of the acetyl precursor, the aqueous source of alkali metal or earth alkali metal hydroxide, and water. The peracetic acid solutions are alkaline, having a pH of 11.2 to 13.37. The reaction is remarkably fast, proceeds with a high conversion of the acetyl precursor into PAA. This method produces solutions with a high level of PAA, from 1% to 7.1%.

[0022] The PAA solution is introduced to the receiving water at the point-of-use. It may be introduced immediately, or stabilized with the addition of a source of acid and used throughout a working day. The receiving water may be water used in the dairy, food, and beverage processing industries for clean-in-place pipeline and equipment cleaning; for fruit and

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vegetable washing; and in the treatment of meat, poultry, and seafood products. The receiving water may also be cooling water, oil and gas process water, or municipal wastewater. Other uses include slime and biofilm removal in papermaking processes.

[0023] This method addresses one or more problems of the known prior art. The method is easy to perform, inexpensive to operate, and requires only basic and common equipment. The method is safe, in that it does not isolate pure PAA, and there is no possibility of a catastrophic event due to an equipment failure, such as over-pressurization or explosions. When triacetin is used as the acetyl precursor, it is inexpensive, non-toxic, safe, non-corrosive, non-irritating, non-flammable, sanctioned as Generally Recognized as Safe (GRAS) by the FDA, and converted quickly and with a high conversion rate into PAA. The method does not require the use of perhydrolase enzymes, acetic acid, or acetic anhydride, all of which suffer from the aforementioned limitations and problems. The method generates a PAA product that does not contain ammonium hydroxide. The method can be performed to generate a PAA product in a continuous or intermittent fashion. Further, the method allows the end-user the choice of utilizing the on site generated PAA immediately or later over the course of a working day.

[0024]

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[0025]

BRIEF DESCRIPTION OF THE DRAWING

[0028] Figure 1 is a schematic representation of an example of a system that may be used to continuously or intermittently generate PAA for on a site having a point-of-use of peracetic acid.

DETAILED DESCRIPTION OF THE INVENTION

[0029] **1. Definitions**

[0030] The terms "PAA" and "peracetic acid" mean peroxyacetic acid or peracetic acid, and/or the conjugate base of peracetic acid (the peracetate ion).

[0031] The terms "percent" and "%" mean weight percent, except when referring to the percent converted.

[0032] The term "receiving water" means the water that is being treated.

[0033] The term "point-of-use" means the location where the peracetic acid enters the receiving water.

[0034] **2. Experimental Methods**

[0035] PAA and hydrogen peroxide in solution were determined using the ceric sulfate-sodium thiosulfate method. The method involves adding a known weight of sample containing the PAA and hydrogen peroxide (or dilution) to a beaker containing 50 ml of chilled 0.9 N sulfuric acid. Using a transfer pipette, 1-2 grams is accurately weighed to a sample cup and reverse osmosis (RO) or deionized water is added to provide a workable volume. Five drops of ferroin indicator is then added with stirring, and the sample is then titrated with 0.1 N ceric sulfate until the sample turns from purple to blue. The volume of ceric sulfate required in mL is recorded. Then, one or two small scoops of potassium iodide are added to the sample cup, which turns the solution dark brown. The sample is then titrated with 0.1 N sodium thiosulfate to discharge the brown coloration and form a pale straw coloration. Then, 10-15 drops of 0.5% starch indicator solution is introduced and the sample turns blue-black. The sodium thiosulfate is then added dropwise until the color transitions to a bright orange. The volume of sodium thiosulfate in mL was recorded. The percent hydrogen peroxide and the percent PAA are calculated as follows:

$$\% \text{ hydrogen peroxide} = \frac{\text{mL 0.1 N ceric sulfate} \times 0.17}{\text{wt. sample}}$$

$$\% \text{ PAA} = \frac{\text{mL 0.1 N sodium thiosulfate x 0.38}}{\text{wt. sample}}$$

[0036] For kinetic runs which involved measuring the PAA and hydrogen peroxide in samples at one, three, and five minute intervals after adding the reactants together, the generation reaction was quenched by adding a sample of the reaction medium to the sulfuric acid. This halted the generation reaction and stabilized the PAA and hydrogen peroxide to elevated pH degradation reactions. This permitted the samples to be taken at the appropriate time interval (one, three, and five minutes), and analyzed later.

[0037] In circumstances where low concentrations of PAA were used, the modified DPD method (U.S. patent no. 7,651,724) was employed. This analytical technique is based on a modified DPD (N,N-diethyl-*p*-phenylenediamine) colorimetric method accepted by the EPA for measuring total chlorine in drinking water and wastewater. It relies on the ability of PAA to behave like chlorine in that it rapidly and quantitatively oxidizes iodide ion (I^-) into iodine (I_2) that reacts with a color indicator (DPD), which turns the solution a shade of pink, the intensity of which is proportional to the concentration of the PAA. A colorimeter is used that is programmed to measure the intensity (absorbance) of the pink coloration and display the result in terms of ppm as Cl_2. A calculation converts this number into the ppm as PAA, based on the weight ratio of PAA to Cl_2 (76/71 = 1.07).

[0038] Hydrogen peroxide does not interfere with the measurement for PAA provided the analysis is completed within 30 seconds of introducing the DPD reagent. In order for the hydrogen peroxide to be measured, it must be activated by addition of a sodium molybdate catalyst and then given time (six minutes) to react with the I^- ion to liberate I_2. Upon addition of the DPD indicator, the intensity of the pink coloration measured by the colorimeter is now the

sum of the PAA and hydrogen peroxide concentrations expressed as ppm Cl_2. After subtracting the ppm reading obtained earlier for the contribution due to PAA, a calculation is then used to convert this number into ppm as hydrogen peroxide, and is based on the weight ratio of hydrogen peroxide to Cl_2 (34/71 = 0.479).

[0039] **3. Liquid Compositions for Generating Non-Equilibrium Solutions of PAA On a Site Having a Point-of-Use**

[0040] In an embodiment, liquid compositions for generating non-equilibrium solutions of PAA on a site having a point-of-use of peracetic acid are provided. These solutions are referred to as "hydrogen peroxide-acetyl precursor solutions." These solutions comprise aqueous hydrogen peroxide, a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide, a trace amount of peracetic acid, and water.

[0041] The hydrogen peroxide-acetyl precursor solutions are prepared by the following steps.

[0042] (a) Introducing a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide to a solution of aqueous hydrogen peroxide.

[0043] Triacetin is a preferable liquid acetyl precursor and 50% hydrogen peroxide is a preferable solution of aqueous hydrogen peroxide. The triacetin is preferably added to the 50% hydrogen peroxide at room temperature. Alternatively, 70% hydrogen peroxide can be used, but it is not widely available and requires special handling and transportation. Given that the solubility of triacetin in water is only 7% at 25°C, triacetin is unexpectedly highly soluble in 50% hydrogen peroxide. It solubilizes without an exotherm (temperature increase).

[0044] The mole ratio of hydrogen peroxide:triacetin is about 2.98:1 to about 12.84:1.

[0045] (b) Allowing the acetyl precursor and the aqueous hydrogen peroxide to mix to form a hydrogen peroxide-acetyl precursor solution.

[0046] The solution is mixed until a homogenous solution has been formed. The solution is allowed to mix by diffusion or by using a mixing device that is suitable for mixing liquids together. For example, 50% hydrogen peroxide can be introduced to a batch tank equipped with an overhead agitator blade. Triacetin can then be introduced to the 50% hydrogen peroxide when the agitator is in motion so that the components are thoroughly mixed.

[0047] The solution that is formed is a hydrogen peroxide-acetyl precursor solution, which also includes varying amounts of water, depending on the formulation used. When triacetin is used as the liquid acetyl precursor, the solution that is formed is a hydrogen peroxide-triacetin solution. One such hydrogen peroxide-triacetin solution comprises 23% to 40% hydrogen peroxide (from 50% aqueous hydrogen peroxide), 20% to 52% triacetin, water, and a trace amount of PAA that is formed within the first day of preparing the hydrogen peroxide-acetyl precursor solution. The pH is 1.46 to 2.2 and no deliberate attempt is made to adjust the pH further.

[0048] A preferable hydrogen peroxide-triacetin solution comprises 27.15% hydrogen peroxide (from 50% aqueous hydrogen peroxide), 45.67% triacetin, water, and a trace amount of PAA that is formed within the first day of preparing the hydrogen peroxide-acetyl precursor solution. The mole ratio of hydrogen peroxide:triacetin is 3.8:1.

[0049] The hydrogen peroxide-acetyl precursor solutions prepared by this method may be used to generate non-equilibrium solutions of PAA on a site having a point-of-use of peracetic acid, in the methods described in section 4 below. The hydrogen peroxide-acetyl precursor solutions may be transported in any container suitable for transporting liquids, such as plastic tote bins of 1041-1249 L (275-330 gallons), drums of 57-208 L (15-55 gallons), 19 L (five-gallon) pails, 3.79 L (one-gallon) jugs, or other containers.

[0050] Example 1

[0051] Triacetin (83.05 g) was added to 50% hydrogen peroxide solution (75 g) to form 158.05 g of a mixture. There was no evolution of gas indicating the hydrogen peroxide had decomposed to oxygen, nor was there any exotherm indicating that a chemical reaction had occurred. The solution did not turn oily or cloudy but remained clear. The solution was calculated to comprise 23.73% hydrogen peroxide and 52.55% triacetin, with the balance being water, and had a measured pH of 2.2. The mole ratio of hydrogen peroxide to triacetin was calculated to be 2.98:1. Titration of the solution immediately upon preparation yielded 24.1% hydrogen peroxide, which is within experimental error of the calculated amount of hydrogen peroxide.

[0052] This experiment showed that triacetin is remarkably soluble in 50% hydrogen peroxide. The solubility of triacetin was found to be at least 52.55%. This is surprising, given that the solubility of triacetin in water is only 7% at 25°C.

[0053] In order to test the stability of the mixture, the amount of hydrogen peroxide remaining and the amount of PAA generated was tracked for several months. The mixture was held at ambient temperature away from sources of light. Table I summarizes the data.

[0054] Table I

Day #	% Hydrogen Peroxide Remaining	% PAA Generated
1	23.79	0.00
29	23.32	1.28
57	22.56	2.36
92	21.06	4.22
120	19.54	6.05
134	18.57	6.87

[0055] Table I shows that although there was a steady depletion in hydrogen peroxide, it was accompanied by an increase in PAA. By the end of the 134-day study, the composition had

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lost 5.22% hydrogen peroxide. Had all of the depleted hydrogen peroxide gone towards the generation of PAA, 11.67% PAA would have been generated. Because 6.87% PAA was generated, unaccounted losses of hydrogen peroxide amounted to 2.15%. This loss of hydrogen peroxide was surprisingly small given that hydrogen peroxide is often used to destroy organic compounds by oxidation and the composition originally contained 52.55% of triacetin, an organic compound.

[0056] Example 2

[0057] A formulation containing hydrogen peroxide and triacetin in a mole ratio of 12.84:1 was prepared by adding triacetin (100.0 g) to 50% hydrogen peroxide (400.0 g) so that the mixture was 40% hydrogen peroxide and 20% triacetin, with the balance being water. The pH of the solution was 1.46 and the pH of a 1:100 dilution was 3.69. To accelerate storage characteristics, the sample was placed in an incubator set for 30-31 °C (86-87 °F).

Table II shows the percent

of hydrogen peroxide remaining and the percent of PAA generated over a period of more than six months.

[0058] Table II

Days of Study	% Hydrogen Peroxide Remaining	% PAA Generated
1	40.21	0.004
33	38.02	3.499
54	35.5	5.992
96	29.27	8.36
127	25.05	6.66
173	18.49	4.43
201	15.67	3.51

[0059] Table II indicates that after 96 days, the generation of PAA maximized at 8.36% and corresponded to a loss of 10.94% hydrogen peroxide. Had all of the depleted hydrogen

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peroxide gone toward the generation of PAA, 24.45% PAA would have been generated. Because only 8.36% PAA was generated, unaccounted losses of hydrogen peroxide amounted to 7.2%.

[0060] Example 3

[0061] A formulation containing hydrogen peroxide and triacetin in a mole ratio of 5.8:1 was prepared by adding triacetin (37.5008 g) to 50% hydrogen peroxide (65.2504 g) so that the mixture was 31.75% hydrogen peroxide and 36.5% triacetin, with the balance being water. The pH of the solution was 1.57 and the pH of a 1:100 dilution was 5.17. To accelerate degradation, the sample was placed in an incubator set for 30-31 °C (86-87 °F).

Table III shows the percent of hydrogen peroxide remaining and the percent of PAA generated over a period of six days.

[0062] Table III

Days of Study	% Hydrogen Peroxide Remaining	% PAA Generated
1	31.72	0.33
6	31.63	0.716

[0063] In this study, after six days, the mixture had lost only 0.09% hydrogen peroxide, yet had generated an additional 0.386% PAA. Had all the loss of hydrogen peroxide been due to the formation of PAA, only an additional 0.201% would have been generated. It is clear that the analytical methods employed for solutions that are high in hydrogen peroxide but low in PAA are subject to detection limit errors.

[0064] Example 4

[0065] A formulation containing hydrogen peroxide and triacetin in a mole ratio of 3.2:1 was prepared by adding triacetin (35.0 g) to 50% hydrogen peroxide (35.0 g) so that the mixture was 25% hydrogen peroxide and 50% triacetin, with the balance being water. The pH of the solution was 1.66 and the pH of a 1:100 dilution was 4.09. To accelerate degradation, the sample

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was placed in an incubator set for 30-31 °C (86-87 °F).

Table IV shows the percent of hydrogen peroxide remaining and the percent of PAA generated over a period of 22 days.

[0066] Table IV

Days of Study	% Hydrogen Peroxide Remaining	% PAA Generated
1	25	0.62
8	24.51	1
15	24.17	1.17
22	23.68	1.14

[0067] Table IV indicates that after 15 days, the generation of PAA maximized at 1.17% and corresponded to a loss of 0.83% hydrogen peroxide. Had all of the depleted hydrogen peroxide gone toward the generation of PAA, 1.86% PAA would have been generated. Because only 1.17% PAA was generated, unaccounted losses of hydrogen peroxide amounted to 0.31%.

[0068] Example 5

[0069] A formulation containing hydrogen peroxide and triacetin in a mole ratio of 4.8:1 was prepared by adding triacetin (37.50 g) to 50% hydrogen peroxide (56.25 g) so that the mixture was 30% hydrogen peroxide and 40% triacetin, with the balance being water. The pH of the solution was 1.67 and the pH of a 1:100 dilution was 4.62. To accelerate degradation, the sample was placed in an incubator set for 86-87° F (30°-31° C).

Table V shows the percent of hydrogen peroxide remaining and the percent of PAA generated over a period of 43 days.

[0070] Table V

Days of Study	% Hydrogen Peroxide Remaining	% PAA Generated
1	30.08	0.38
8	29.85	0.784
15	29.63	1.22
22	29.37	1.71
29	28.76	2.26
36	28.67	2.78
43	28.31	3.41

[0071] Table V shows that although there was a steady depletion in hydrogen peroxide, it was accompanied by the associative increase of PAA. At the end of the 43-day study, the composition had lost 1.77% hydrogen peroxide. Had all of the depleted hydrogen peroxide gone toward the generation of PAA, 3.96% PAA would have been generated. Because only 3.41% PAA was generated, unaccounted losses of hydrogen peroxide amounted to 0.24%.

[0072] Example 6

[0073] A formulation containing hydrogen peroxide and triacetin in a mole ratio of 6.42:1 was prepared by adding triacetin (25.0007 g) to 50% hydrogen peroxide (50.0007 g) so that the mixture was 33.33% hydrogen peroxide and 33.33% triacetin, with the balance being water. The pH of the solution was 1.56 and the pH of a 1:100 dilution was 5.15. To accelerate degradation, the sample was placed in an incubator set for 30-31 °C (86-87 °F).

Table VI shows the percent

of hydrogen peroxide remaining and the percent of PAA generated over a period of 36 days.

[0074] Table VI

Days of Study	% Hydrogen Peroxide Remaining	% PAA Generated
1	33.13	0.81
8	32.88	1.35
15	32.61	1.88
22	31.69	2.58
29	31.75	3.03
36	31.29	3.28

[0075] Table VI shows that although there was a steady depletion in hydrogen peroxide, it was accompanied by an associative increase of PAA. At the end of the 36-day study, the composition had lost 1.84% hydrogen peroxide. Had all of the depleted hydrogen peroxide gone toward the generation of PAA, 4.11% PAA would have been generated. Because only 3.28% PAA was generated, unaccounted losses of hydrogen peroxide amounted to 0.37%.

[0076] Example 7

[0077] A 113.6 L (30-gallon) batch of 50% hydrogen peroxide and triacetin was prepared by blending 62 kg (137 lbs) triacetin with 74 kg (163 lbs) 50% hydrogen peroxide so that the mixture was 27.15% hydrogen peroxide and 45.67% triacetin, with the balance being water. Thus, the mole ratio of hydrogen peroxide:triacetin was 3.8:1. The mixture was stored in an opaque drum in a non-climate controlled environment. On storage, the mixture slowly formed PAA in the container. Since this reaction of hydrogen peroxide to PAA does not represent a destructive loss of total peroxygen, to determine the true stability of the hydrogen peroxide, both the hydrogen peroxide and the peracetic acid generated must be quantified and reported as total peroxygen recovered.

[0078] After 295 days of storage, the mixture was analyzed for total peroxygen recovered (expressed as hydrogen peroxide) to determine the percent loss of peroxygen. Using the eerie sulfate-iodometric titration method, the PAA generated and the hydrogen peroxide remaining were measured. The total peroxygen recovered after 295 days and the theoretical initial concentrations are shown in Table VII. All peroxygen is expressed as % hydrogen peroxide.

[0079] Table VII

	Initial Theoretical %	% (after 295 days)
Hydrogen Peroxide Recovered	27.17	15.20
PAA formed	0	15.57
Hydrogen Peroxide Reacted to form PAA	0	6.97
Total Recovered Hydrogen Peroxide	27.17	22.17
Total Hydrogen Peroxide Unproductive Loss		5.00

[0080] After 295 days, 22.17% of the total peroxygen was recovered as either titratable hydrogen peroxide or hydrogen peroxide reacted to form PAA. To determine the stability of the peroxygen in the mixture, the total recovered hydrogen peroxide after 295 days (22.17%) was

subtracted from the total recovered hydrogen peroxide of the initial theoretical value (27.17%). The result, 5%, is the loss of peroxygen (expressed as hydrogen peroxide) after 295 days of storage in a non-climate controlled environment.

4. Methods of Generating Non-Equilibrium PAA On Site at the Point-of-Use

[0081] Methods of continuously or intermittently generating non-equilibrium solutions of PAA on a site having a point-of-use of PAA for use as a disinfectant or sanitizer, using a source of water that is an aqueous stream or by a batch process using water in a container, are provided.

[0082] In one embodiment, a method of continuously or intermittently generating non-equilibrium solutions of PAA on a site having a point-of-use, using a source of water that is an aqueous stream, comprises the following steps.

[0083] (a) Providing water.

[0084] Flowing water is provided on a site having a point-of-use of peracetic acid.

[0085] The water may be flowing in, for example, a pipe, a flume, a canal, or other types of aqueous streams. The water pressure should be regulated and the flow rate should be monitored or measured. Any suitable flow meter may be used, such as a rotameter, a magnetic flow meter, an ultrasonic flow meter, a Doppler flow meter, a differential-pressure flow meter, a turbine flow meter, or a Coriolis flow meter.

[0086] The water should be softened, deionized, or of sufficient low hardness that it will not precipitate calcium salts when the alkali metal or earth alkali metal hydroxide is introduced in step (d).

[0087] (b) Introducing a hydrogen peroxide-acetyl precursor solution to the water.

[0088] A hydrogen peroxide-acetyl precursor solution is provided on the site having the point-of-use of the PAA. The hydrogen peroxide-acetyl precursor solution may be prepared by the steps set forth in section 3 above. The hydrogen peroxide-acetyl precursor solution may be

prepared off site and then transported to the site having the point-of-use, or the acetyl precursor and the aqueous hydrogen peroxide may be transported to the site having the point-of-use and used to prepare the hydrogen peroxide-acetyl precursor solution on the site.

[0089] A preferable liquid acetyl precursor is triacetin. When triacetin is used, a hydrogen peroxide-triacetin solution is introduced.

[0090] The hydrogen peroxide-acetyl precursor solution is introduced to the flowing water in an amount such that the hydrogen peroxide-acetyl precursor solution is 5.6% to 22.5% of the total.

[0091] The hydrogen peroxide-acetyl precursor solution is pumped from its container into the aqueous water stream, for example, through an injection quill mounted on the pipe. Any suitable pump capable of overcoming the pressure of the water flowing in the pipe may be used. Examples include a solenoid-driven or air-driven diaphragm pump or a peristaltic pump. The rate at which the hydrogen peroxide-acetyl precursor solution is pumped into the flowing water is governed by a controller that is interfaced to a flow meter that is measuring the flow of the water. In this way, the rate at which the hydrogen peroxide-acetyl precursor solution is pumped may be matched to the flow of the water in the aqueous stream. If the flow of the water slows, the rate at which the hydrogen peroxide-acetyl precursor solution is pumped should slow accordingly. If the flow of the water stops, the hydrogen peroxide-acetyl precursor pump should stop.

[0092] As an alternative to step (b), the liquid acetyl precursor and the solution of aqueous hydrogen peroxide may be introduced to the water separately, either simultaneously or sequentially. If they are introduced sequentially, either one may be added first. If the liquid acetyl precursor and the aqueous hydrogen peroxide are introduced separately, rather than as an hydrogen peroxide-acetyl precursor solution, then in step (c) the liquid acetyl precursor and the aqueous hydrogen peroxide are mixed with the water to form a mixture.

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[0093] (c) Mixing the hydrogen peroxide-acetyl precursor solution and the water to form a mixture.

[0094] Any mixing device suitable for mixing liquids may be used. An example is a static mixer located just after the point that the hydrogen peroxide-acetyl precursor solution is introduced to the flowing water. One type of static mixer utilizes a non-moving element such as a series of baffles. As the mixture flows through the static mixer under the motive force of the flowing water, the non-moving element divides the flow several times to provide radial mixing. Another type of static mixer utilizes a series of obstructions, such as column packing or glass beads, provided there is a low differential pressure drop across the mixer. The obstructions provide for turbulent mixing of the hydrogen peroxide-acetyl precursor solution and the flowing water. The mixing should yield a homogeneous solution with no concentration gradients before the next step is performed. The velocity of the water will determine the time it takes to complete the mixing and the efficiency of the mixing. For example, with ¾" pipe, a static mixer of ¾" diameter and 6" long, and a velocity of about 1 gal/min., mixing should be accomplished in less than about one second.

[0095] (d) Adding an aqueous source of an alkali metal or earth alkali metal hydroxide to the mixture.

[0096] An aqueous source of an alkali metal or earth alkali metal hydroxide is provided on the site having the point-of-use. A preferable aqueous source of an alkali metal hydroxide is sodium hydroxide, and 50% sodium hydroxide is most preferable. Other suitable alkali metal hydroxides include 45% potassium hydroxide.

[0097] Sufficient 50% NaOH is added to the flowing mixture such that the amount of sodium hydroxide is 1.82% to 7.28% of the total amount. When the acetyl precursor is triacetin, a preferred mole ratio of NaOH: hydrogen peroxide:triacetin is 4.2:3.8:1.

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[0098] The 50% NaOH solution is added to the mixture by pumping it from its container with a suitable pumping device and into the mixture through an injection quill mounted on the pipe. Any suitable pump capable of overcoming the hydraulic pressure of the pipe may be used. Examples include a solenoid-driven or air-driven diaphragm pump or a peristaltic pump.

[0099] The rate at which the 50% NaOH solution is pumped into the pipe is governed by the same controller that is interfaced to both the flow meter that is measuring the flow of the mixture and the pump controlling the rate of introduction of the hydrogen peroxide-acetyl precursor solution. In this way, the rate at which the 50% NaOH solution is introduced may be matched to both the flow of the mixture and the rate of introducing the hydrogen peroxide-acetyl precursor solution. If the flow of the mixture slows for any reason, the rate at which the 50% NaOH solution is pumped should slow accordingly. If the flow of the mixture stops, the 50% NaOH pump should stop as well.

[00100] Step (d) may be performed after step (c), or it may be performed simultaneously with step (b).

[00101] (e) Forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

[00102] The reaction medium that is formed in this step almost instantaneously forms a non-equilibrium solution of PAA. The hydrogen peroxide reacts with the acetyl precursor to form peracetic acid. Depending upon the temperature of the water, the efficiency of mixing, and the mole ratio of NaOH:hydrogen peroxide:acetyl precursor employed, the amount of hydrogen peroxide and acetyl precursor that are converted into PAA is maximized within about 30 seconds to about five minutes.

[00103] The non-equilibrium solutions of PAA prepared by this method comprise PAA, unreacted hydrogen peroxide, unreacted acetyl precursor, the product of the perhydrolysis

reaction of the acetyl precursor, the aqueous source of alkali metal or earth alkali metal hydroxide, and water. When the acetyl precursors is triacetin, the product of the perhydrolysis reaction is 1, 2, 3-propanetriol (glycerine).

[00104] This method may also include an optional step after step (e) of sampling the reaction medium at various times after the addition of the aqueous source of alkali metal or earth alkali metal hydroxide to determine the time required under the existing conditions to maximize the amount of hydrogen peroxide and acetyl precursor that are converted into PAA. This may be accomplished by directing the flow of the reaction medium through a series of residence chambers equipped with sampling ports. The residence chambers are designed to be of sufficient volume such that a certain amount of time has elapsed since the introduction of the 50% NaOH. Thus, at any given flow rate in the pipe, the first residence chamber is of a volume such that 30 seconds have elapsed since the introduction of 50% NaOH, one minute has elapsed by the time the flow reaches the sampling port immediately after the second residence chamber, and additional time elapses by the time the flow reaches the remaining sampling ports. Up to four residence chambers and sampling ports may be placed in series such as this to provide the user with about 30 seconds to five minutes of residence time after the introduction of the 50% NaOH. Once the flow of the reaction medium has been established, samples are drawn from all four sampling ports associated with the residence chambers and analyzed for peracetic acid. The sample which registers the highest amount of peracetic acid is deemed to be drawn from the sampling port associated with the residence chamber corresponding to the time of maximum conversion of the acetyl precursor into peracetic acid. At this point, the entire flow of reaction medium is directed through the sampling point of maximum conversion of acetyl precursor and to the point-of-use.

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[00105] In another embodiment, a method of continuously or intermittently generating a non-equilibrium solution of PAA on a site having a the point-of-use in a batch process comprises the following steps:

[00106] (a) Providing water.

[00107] A container of water, such as a mixing tank or other vessel, is provided on a site having a point-of-use of peracetic acid. The water should be softened, deionized, or of sufficient low hardness that it will not precipitate calcium salts when the alkali metal or earth alkali metal hydroxide is introduced in step (d).

[00108] (b) Introducing a hydrogen peroxide-acetyl precursor solution to the water.

[00109] A hydrogen peroxide-acetyl precursor solution is provided on the site having the point-of-use of the PAA. The hydrogen peroxide-acetyl precursor solution may be prepared by the steps set forth in section 3 above. The hydrogen peroxide-acetyl precursor solution may be prepared off site and then transported to the site having the point-of-use, or the acetyl precursor and the aqueous hydrogen peroxide may be transported to the site having the point-of-use and used to prepare the hydrogen peroxide-acetyl precursor solution on the site.

[00110] A preferable liquid acetyl precursor is triacetin. When triacetin is used, a hydrogen peroxide-triacetin solution is introduced.

[00111] The hydrogen peroxide-acetyl precursor solution is introduced to the water in an amount such that the hydrogen peroxide-acetyl precursor solution is 5.6% to 22.5% of the total.

[00112] The hydrogen peroxide-acetyl precursor solution is pumped from its container into the container of water through an injection quill mounted on the side of the container of water. Any suitable pump may be used. Examples include a solenoid-driven or air-driven diaphragm pump or a peristaltic pump.

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[00113] As an alternative to step (b), the liquid acetyl precursor and the solution of aqueous hydrogen peroxide may be introduced to the water separately, either simultaneously or sequentially. If they are introduced sequentially, either one may be added first. If the liquid acetyl precursor and the aqueous hydrogen peroxide are introduced separately, rather than as an hydrogen peroxide-acetyl precursor solution, then in step (c) the liquid acetyl precursor and the aqueous hydrogen peroxide are mixed with the water to form a mixture.

[00114] (c) Mixing the hydrogen peroxide-acetyl precursor solution and the water to form a mixture.

[00115] Any mixing device suitable for mixing liquids may be used. An example is an overhead stirrer, such as an agitator blade. The container of water is equipped with a baffle to assist the process of mixing. The mixing should yield a homogeneous solution with no concentration gradients before the next step is performed.

[00116] (d) Adding an aqueous source of an alkali metal or earth alkali metal hydroxide to the mixture.

[00117] An aqueous source of an alkali metal or earth alkali metal hydroxide is provided on the site having the point-of-use. A preferable aqueous source of an alkali metal hydroxide is sodium hydroxide, and 50% sodium hydroxide is most preferable. Other suitable alkali metal hydroxides include 45% potassium hydroxide.

[00118] Sufficient 50% NaOH is added to the mixture such that the amount of sodium hydroxide is 1.82% to 7.28% of the total amount. When the acetyl precursor is triacetin, a preferred mole ratio of NaOH: hydrogen peroxide:triacetin is 4.2:3.8:1.

[00119] The 50% NaOH solution is added to the mixture by pumping it from its container with a suitable pumping device and into the mixture through an injection quill mounted on the

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side of the container of water. Any suitable pump may be used. Examples include a solenoid-driven or air-driven diaphragm pump or a peristaltic pump.

[00120] Step (d) may be performed after step (c), or it may be performed simultaneously with step (b).

[00121] (e) Forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

[00122] The reaction medium that is formed in this step almost instantaneously forms a non-equilibrium solution of PAA. The hydrogen peroxide reacts with the acetyl precursor to form peracetic acid. Depending upon the temperature of the water, the efficiency of mixing, and the mole ratio of NaOH:hydrogen peroxide:acetyl precursor employed, the amount of hydrogen peroxide and acetyl precursor that are converted into PAA is maximized within about 30 seconds to about five minutes.

[00123] The non-equilibrium solutions of PAA prepared by this method comprise PAA, unreacted hydrogen peroxide, unreacted acetyl precursor, the product of the perhydrolysis reaction of the acetyl precursor, the aqueous source of alkali metal or earth alkali metal hydroxide, and water. When the acetyl precursors is triacetin, the product of the perhydrolysis reaction is 1, 2, 3-propanetriol (glycerine).

[00124] While not wishing to be bound by theory, it is believed that under elevated pH conditions created by the addition of a source of alkali metal hydroxide or earth alkali metal hydroxide, hydrogen peroxide dissociates according to the following equation:

$$H_2O_2 = H^+ + HO_2^-$$

[00125] The perhydroxyl anion (HO_2^-) then affects nucleophilic substitution reactions on the carbonyl groups of the acetyl precursor in a perhydrolysis reaction to form peracetic acid and the product of the perhydroloysis reaction of the acetyl precursor. When the acetyl precursor is

triacetin and all three acetyl groups are reacted, then the product of the perhydrolysis reaction is 1, 2, 3-propanetriol (glycerine).

[00126] In these methods, the perhydrolysis reaction is rapid and the maximum amount of PAA is generated within about 30 seconds to about five minutes at ambient temperature. The methods efficiently utilize the acetyl precursor and the source of hydrogen peroxide. When triacetin is used, assuming all three acetyl groups are reacted, about 40.9% to about 85.7% of the triacetin is converted into PAA. The percent of hydrogen peroxide remaining is about 0.078% to about 1.88%.

[00127] The non-equilibrium solutions of PAA prepared by these methods have several unique characteristics. The PAA solutions have high levels of PAA, from 1% to 7.1%. In addition, the PAA solutions are alkaline, having a pH of 11.2 to 13.37.

[00128] The non-equilibrium solutions of PAA may be immediately introduced to the receiving water at the point-of-use. Alternatively, the solutions may be stabilized by halting the decomposition of the PAA by adding a source of acid to lower the elevated pH of the reaction medium caused by the addition of an alkali metal or earth alkali metal hydroxide, and to provide neutral to mildly acid pH conditions to stabilize the PAA to elevated pH degradation. The acid-stabilized PAA solutions can then be stored and used at the point-of-use as required throughout a working day.

[00129] The non-equilibrium PAA solutions produced by these methods can be advantageously used wherever traditional equilibrium solutions of PAA are used. Thus, the PAA solutions may be used as a disinfectant and sanitizer in the dairy, food, and beverage processing industries for clean-in-place pipeline and equipment cleaning; for fruit and vegetable washing; and in the treatment of meat, poultry, and seafood products. These PAA solutions may also be

used in the treatment of cooling water, oil and gas process water, and municipal wastewater. Other uses include slime and biofilm removal in papermaking processes.

[00130] For both of the above methods, steps (a) through (e) may be performed on a continuous or intermittent basis. In a continuous basis, the steps are performed under conditions of steady state, and the PAA solution is produced at the same rate as a function of time. However, intermittent performing of steps (a) through (e) is possible if the demand for the PAA solution drops for any reason (e.g., change in working shift or the target level of PAA in the receiving water has been achieved), in which case, steps (a) through (e) may be discontinued and resumed when the demand increases.

[00131] Example 8

[00132] Tests were performed using about one liter of reverse osmosis (RO) or softened water which was buffered to an alkaline pH. The buffering media included 0.1 and 0.2 M Na_2CO_3 solutions, in addition to 0.1 and 0.2 M NaOH solutions. Sufficient buffering media was added to the water in order to achieve the desired pH. The amount of hydrogen peroxide (introduced as 50% hydrogen peroxide) and triacetin used in the tests is summarized in Table VIII. The reactants were introduced to the solution in one of three ways: simultaneously (referred to in Table VIII as "double"), sequentially (triacetin followed shortly by hydrogen peroxide), or by mixing the hydrogen peroxide and triacetin together and adding them in a single charge (referred to in Table VIII as "mixed"). All testing was performed at ambient temperature as the one-liter beaker was stirred with a magnetic stir bar. Using the ceric sulfate–iodometric titration method, the percent of PAA generated and the percent of hydrogen peroxide remaining were measured five minutes after introduction of the reactants to the buffer. The percent of the triacetin acetyl donor converted to PAA was calculated, assuming all three acetyl groups were available for perhydrolysis.

[00133] Table VIII

ıt #	Moles Hydrogen Peroxide/ Mole Triacetin	Type of Addition (Double, Sequential or Mixed)	pH of Buffer	Hydrogen Peroxide Used (Wt., g)	Triacetin Used (Wt., g)	% PAA Generated	% Hydrogen Peroxide Remainin g	% Triacetin Converted to PAA
	4.99	Double	10.50	8.60	5.53	0.170	0.329	29.6%
	5.47	Double	10.50	8.60	5.04	0.178	0.333	33.9%
	5.45	Double	10.80	8.61	5.07	0.192	0.329	36.4%
	5.68	Double	11.00	8.87	5.01	0.201	0.322	38.6%
	5.72	Double	11.00	9.13	5.12	0.068	0.416	12.8%
	5.49	Double	12.42	8.60	5.02	0.169	0.306	32.4%
	5.59	Double	12.91	8.60	4.93	0.320	0.265	62.4%
	5.53	Double	12.80	8.69	5.04	0.335	0.273	63.9%
	25.0	Double	12.65	9.03	1.16	0.104	0.413	85.9%
0	5.26	Double	12.60	8.70	5.30	0.298	0.247	54.1%
1	5.52	Sequential	12.83	8.68	5.04	0.276	0.248	52.4%
2	5.47	Sequential	12.81	17.11	10.03	0.120	0.371	11.4%
3	28.8	Mixed	11.12	8.74	0.973	0.039	0.398	38.9%
4	28.8	Mixed	11.99	8.75	0.974	0.038	0.374	37.9%
5	28.8	Mixed	13.02	8.69	0.967	0.055	0.367	55.2%
6	28.8	Mixed	7.91	8.70	0.968	0.024	0.435	23.7%
7	28.8	Mixed	11.22	8.76	0.975	0.029	0.309	28.4%
8	28.8	Mixed	12.07	8.68	0.966	0.028	0.311	27.7%
9	28.8	Mixed	13.22	8.72	0.971	0.026	0.321	25.6%
lup	25.2	Mixed	12.97	8.77	1.12	0.031	0.249	26.5%
0	25.2	Mixed	12.98	9.08	1.16	0.066	0.393	54.5%
lup	25.2	Mixed	13.00	9.10	1.16	0.074	0.409	61.0%
1	25.2	Mixed	13.02	9.10	1.16	0.02	0.261	16.5%
lup	25.2	Mixed	12.99	9.12	1.16	0.017	0.376	14.0%
2	16.0	Mixed	12.99	5.80	1.16	0.070	0.246	57.7%
3	16.0	Mixed	13.05	5.79	1.16	0.013	0.189	10.7%
4	16.0	Mixed	12.55	5.78	1.16	0.031	0.234	25.6%
5	16.0	Mixed	12.49	5.80	1.16	0.013	0.144	10.7%
6	16.0	Mixed	12.01	5.85	1.17	0.016	0.259	13.1%
7	16.0	Mixed	12.05	5.82	1.16	0.011	0.080	9.0%
8	16.0	Mixed	10.50	0.601	0.120	0.00077	0.028	6.1%
9	16.0	Mixed	10.97	0.595	0.119	0.00023	0.021	1.8%
0	16.0	Mixed	10.50	0.614	0.123	0.00012	0.019	0.9%
2	16.0	Mixed	10.97	0.595	0.119	0.00035	0.018	2.8%

[00134] Table VIII shows that the amount of PAA generated was independent of the method of introducing the triacetin and hydrogen peroxide to the buffer solutions. It can be seen that the pH of the buffer had a significant effect on the percent of the triacetin that was converted into PAA. Only when the pH was above 12 was a meaningful percent of the triacetin converted into PAA.

[00135] Example 9

[00136] A mixture of triacetin and hydrogen peroxide was prepared by blending 50% hydrogen peroxide (63.4%) and triacetin (36.6%). Thus, the mole ratio of hydrogen peroxide:triacetin was 5.57:1. The mixture (13.68 g) was added to soft water such that the final solution contained 5 g/L of triacetin and had a pH of 4.1. Then, in successive experiments, sufficient 50% NaOH was added to the beaker so that the mole ratio of NaOH:TA was varied from 3:1 (obtained by introducing 5.49 g of 50% NaOH) to 8:1 (obtained by introducing 14.68 g of 50% NaOH). Using the ceric sulfate–iodometric titration method, the percent of PAA generated and the percent of hydrogen peroxide remaining were measured over the next 20 minutes. The method was performed in duplicate for each sample point. The averages of the results for the 6:1 mole ratio of NaOH:TA are shown in Table IX.

[00137] Table IX

Time (min)	% PAA Generated	% Hydrogen Peroxide Remaining	% Triacetin Converted to PAA (3 acetyls)
0	0.015	0.437	2.9
1	0.275	0.298	53.1
3	0.312	0.274	60.2
5	0.326	0.258	62.9
11	0.302	0.233	58.3
21	0.246	0.199	47.5

[00138] It can be seen that even before the introduction of 50% NaOH, some PAA was present in solution as a result of being generated in the diluted mixture of the two components.

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Upon addition of the NaOH, PAA was immediately generated, with the maximum amount of 0.326% occurring at five minutes, corresponding to a yield of 62.9% assuming all three acetyl groups of triacetin were available for perhydrolysis.

[00139] This exercise was repeated for mole ratios of NaOH:triacetin ranging from 3:1 to 8:1. Table X summarizes the next set of data and shows the maximum percent of PAA generated, the time after the addition of NaOH that it took to reach the maximum percent of PAA generated, the pH after the addition of the NaOH, the percent of hydrogen peroxide remaining at the time of the maximum percent of PAA, and the calculated percent of triacetin converted to PAA assuming that all three acetyl groups were available for perhydrolysis.

[00140] Table X

Mole ratio NaOH:TA	Time at Max. PAA (min)	Max % PAA Generated	% Hydrogen Peroxide Remaining	% Triacetin Converted to PAA (3 acetyls)	pH
3:1	3	0.229	0.291	43.8	11.2
4:1	6	0.285	0.251	54.8	11.3
6:1	5	0.326	0.258	62.9	11.6
8:1	3	0.335	0.271	64.7	12.2

[00141] It can be seen that the amount of PAA generated, and hence the conversion of triacetin into PAA, increased with the increasing mole ratio of NaOH:TA. For all four mole ratios employed, the maximum amount of PAA was generated between three and six minutes. The optimum mole ratio of NaOH:TA was 6:1 as it resulted in an efficient utilization of triacetin converted into PAA, and because the pH of the resulting reaction medium would have less impact on the pH of the receiving water than the higher pH of the reaction medium resulting from the 8:1 mole ratio.

[00142] Example 10

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[00143] A series of experiments was then performed in which the mole ratio of NaOH:TA was fixed at 8:1 and the mole ratio of hydrogen peroxide:triacetin was varied from 2:1 to 8:1. Triacetin (5 g) was added to about 965 mL of softened water along with 50.7% hydrogen peroxide in successive tests (3.06 g for the 2:1 mole ratio; 6.19 g for the 4:1 mole ratio; 9.23 g for the 6:1 mole ratio; and 12.32 g for the 8:1 mole ratio) to yield a solution with a pH of about 4.8-5.5. The volume of the mixture was adjusted to 990 mL with softened water. With mixing, 50% NaOH (14.67 g) was introduced. Using the ceric sulfate–iodometric titration method, the percent of PAA generated and the percent of hydrogen peroxide remaining were measured over the next 20 minutes. The method was performed in duplicate for each sample point. The averages of the results for the kinetic run employing the mole ratio of 8:6:1 NaOH:hydrogen peroxide:TA are shown in Table XI.

[00144] Table XI

Time (min)	% PAA Generated	% Hydrogen Peroxide Remaining	% Triacetin Converted to PAA (3 acetyls)
1	0.331	0.317	63.8
3	0.330	0.303	63.6
5	0.319	0.303	61.5
10	0.300	0.295	57.8
20	0.256	0.274	49.3

[00145] Upon addition of the NaOH, PAA was immediately generated, with the maximum amount of 0.331% occurring after just one minute, corresponding to a calculated conversion of triacetin into PAA of 63.8%, assuming that all three acetyl groups of triacetin were available for perhydrolysis.

[00146] This exercise was repeated for mole ratios of hydrogen peroxide:triacetin ranging from 2:1 to 6:1. Table XII summarizes the data and shows the maximum percent of PAA generated, the time after the addition of NaOH that it took to reach the maximum percent of PAA

generated, the pH after the addition of the NaOH, the percent of hydrogen peroxide remaining at the time of the maximum percent of PAA, and the calculated percent of triacetin converted to PAA assuming that all three acetyl groups were available for perhydrolysis.

[00147] Table XII

Mole Ratio Hydrogen Peroxide:Triacetin	Time at Max. PAA (min)	Max % PAA Generated	% Hydrogen Peroxide Remaining	% Triacetin Converted to PAA (3 acetyls)	pH
2:1	3	0.214	0.079	40.9	12.6
4:1	1	0.272	0.195	51.9	12.4
6:1	1	0.331	0.317	63.8	12.3
8:1	3	0.374	0.445	72.0	11.9

[00148] It can be seen that the amount of PAA generated, and hence the conversion of triacetin into PAA, increased with the increasing mole ratio of hydrogen peroxide:triacetin. For all four mole ratios employed, the maximum amount of PAA was generated between one and three minutes. The optimum mole ratio of hydrogen peroxide:triacetin was 8:1 as it resulted in the most efficient utilization of triacetin converted into PAA.

[00149] Example 11

[00150] A further series of experiments was performed in which the mole ratio of hydrogen peroxide to triacetin was fixed at 4:1 and the molar ratio of NaOH to triacetin was varied from 3:1 to 8:1. Triacetin (5 g) was added to around 975 mL of softened water along with 50.7% hydrogen peroxide (6.18 g) to yield a solution with a pH of 4.8 to 5.3. With mixing, 50% NaOH was introduced in successive tests (5.51 g for the 3:1 mole ratio; 7.30 g for the 4:1 mole ratio; 11.03 g for the 6:1 mole ratio; and 14.67 g for the 8:1 mole ratio). The volume of the mixture was adjusted to 1000 mL with softened water. Using the ceric sulfate–iodometric titration method, the percent of PAA generated and the percent of hydrogen peroxide remaining were measured over the next 20 minutes. The method was performed in duplicate for each

sample point. The averages of the results for the kinetic run employing the mole ratio of NaOH:hydrogen peroxide:triacetin of 4:4:1 are shown in Table XIII.

[00151] Table XIII

Time (min)	% PAA Generated	% Hydrogen Peroxide Remaining	% Triacetin Converted to PAA (3 acetyls)
1	0.247	0.207	47.3
3	0.280	0.178	53.6
5	0.292	0.167	55.9
10	0.267	0.142	51.1
20	0.229	0.111	43.8

[00152] Upon addition of the NaOH, PAA was immediately generated, with the maximum amount of 0.292% occurring after five minutes, corresponding, to a calculated conversion of 55.9% triacetin into PAA assuming that all three acetyl groups of triacetin were available for perhydrolysis.

[00153] This exercise was repeated for mole ratios of NaOH:TA ranging from 3:1 to 8:1. Table XIV summarizes the data and records the maximum percent of PAA generated, the time after the addition of NaOH that it took to reach the maximum percent of PAA generated, the pH after the addition of the NaOH, the percent of hydrogen peroxide remaining at the time of the maximum percent of PAA, and the calculated percent of triacetin converted to PAA assuming that all three acetyl groups were available for perhydrolysis.

[00154] Table XIV

Mole Ratio NaOH:TA	Time at Max. PAA (min)	Max % PAA Generated	% Hydrogen Peroxide Remaining	% Triacetin Converted to PAA (3 acetyls)	pH
3:1	3	0.246	0.202	47.2	11.3
4:1	5	0.292	0.167	55.9	11.6
6:1	3	0.290	0.181	55.6	12.2
8:1	3	0.272	0.195	51.9	12.4

[00155] It can be seen that the amount of PAA generated maximized when the mole ratio of NaOH:hydrogen peroxide:triacetin was 4:4:1. The optimum mole ratio of NaOH:hydrogen peroxide:triacetin was 4:4:1 as it resulted in an efficient utilization of triacetin converted into PAA, and because the pH of the resulting reaction medium would have less impact on the pH of the receiving water than the higher pH of the reaction medium resulting from the 4:6:1 mole ratio of NaOH:hydrogen peroxide:triacetin. For all four mole ratios employed, the maximum amount of PAA was generated between three and five minutes.

[00156] Example 12

[00157] In the first test, a mixture of triacetin and hydrogen peroxide was prepared by blending 50% hydrogen peroxide (54.24%) and triacetin (45.76%). Thus, the mole ratio of hydrogen peroxide:triacetin was 3.65:1. The mixture (51.22 g) was dissolved in soft water (911.9 g) in a one-liter beaker to yield a solution with a pH of 6.47. Then, 50% NaOH (36.47 g) was introduced with stirring. Thus, the mole ratio of the combined mixture of NaOH:hydrogen peroxide:triacetin was 4.2:3.8:1. The pH of the resulting solution initially measured 12.1. Using the ceric sulfate–iodometric titration method, the PAA generated and the hydrogen peroxide remaining were measured over the next 10 minutes. It was noticed that the sample temperature rose immediately upon addition of the 50% NaOH.

[00158] In the second test, the first test was repeated, except that the water was chilled soft water.

[00159] In the third test, the first test was repeated using three times the original amount of hydrogen peroxide:triacetin mixture and three times the original amount of 50% NaOH. The mole ratio of the combined mixture of NaOH:hydrogen peroxide:triacetin remained the same as before, at 4.2:3.8:1. The hydrogen peroxide and triacetin mixture (153.96 g) was introduced to chilled soft water (735.6 g) in a one-liter beaker to yield a solution with an initial pH of 5.14.

Then, 50% NaOH (111.21 g) was introduced. Using the ceric sulfate–iodometric titration method, the PAA generated and the hydrogen peroxide remaining were measured over the next 10 minutes.

[00160] In the fourth test, the first test was repeated using four times the original amount of hydrogen peroxide:triacetin mixture and four times the original amount of 50% NaOH. The mole ratio of the combined mixture of NaOH:hydrogen peroxide:triacetin remained the same as before, at 4.2:3.8:1. The hydrogen peroxide and triacetin mixture (204.7 g) was introduced to chilled soft water (647.5 g) in a one-liter beaker to yield a solution with an initial pH of 6.32. Then, 50% NaOH (155.4 g) was introduced. Using the ceric sulfate–iodometric titration method, the PAA generated and the hydrogen peroxide remaining were measured over the next 10 minutes.

[00161] Table XV summarizes the results obtained for the four tests and shows the initial and final solution temperatures, the pH of the solution after the addition of the 50% NaOH, the maximum percent of PAA generated, the time of maximum PAA generation, and the percent of hydrogen peroxide remaining.

[00162] Table XV

Test #	Initial Temp. (°F)	Final Temp. (°F)	Final pH	Max. % PAA Generated (time, min)	% Hydrogen Peroxide Remaining
1	NM	NM	12.14	1.71 (1)	0.58
2	38	50	12.67	2.1 (4)	0.43
3	44	88	12.77	6.3 (1)	1.18
4	34	92	13.17	7.2 (1)	1.88

NM = not measured

[00163] Table XVI shows the percent of hydrogen peroxide converted overall, the percent of hydrogen peroxide converted to PAA, and the percent of triacetin converted to PAA

(assuming that all three acetyl groups on triacetin are available for conversion to PAA) for each of the tests in Table XV.

[00164] Table XVI

Test #	% Hydrogen Peroxide Converted Overall	% Hydrogen Peroxide Converted to PAA (at maximum conversion time)	% Triacetin Converted to PAA (3 acetyls)
1	67.8	88.1	69.7
2	68.8	94.4	85.8
3	71.6	94.3	85.7
4	66.1	88.1	73.8

[00165] The data in Tables XV and XVI indicate that far higher amounts of PAA are possible if the 50% sodium hydroxide solution is the last component of the mixture to be introduced. Further, up to 7.1% PAA was generated, although this solution got hot despite chilling the source water. Generation of the 6.3% PAA solution was very efficient because it represented a high 85.7% utilization of triacetin converted into PAA. There was clearly a linear relationship between Test 2, which generated 2.1% PAA, and Test 3, which utilized three times more of the reactants and generated 6.3% PAA. The data in Table XV indicate that the time of maximum conversion of triacetin was between one and four minutes. Thereafter, the amount of PAA in solution is reduced because it undergoes high pH degradation slowly over the following 10 minutes.

[00166] Example 13

[00167] Fig. 1 is a schematic representation of an example of a system 10 that was used to perform an embodiment of one of the methods described above to prepare a non-equilibrium solution of PAA on a site having a point-of-use. Triacetin was used as the acetyl precursor and 50% sodium hydroxide was used as the aqueous source of an alkali metal hydroxide.

[00168] A container 120 of a hydrogen peroxide-acetyl precursor solution, which was a hydrogen peroxide-triacetin solution, and a container 140 of 50% sodium hydroxide were each equipped with chemical delivery diaphragm pumps 115 and 135, respectively. Inlet water 90, which was softened, was provided from a water source on the site having the point-of-use. Inlet water 90 was directed through a pressure regulator 100 and flow meter 105, then into a section of pipe where the hydrogen peroxide-triacetin solution was introduced through injection quill 110. Mixing was accomplished using static mixer 125. Then, the sodium hydroxide solution was added through injection quill 130 to form a reaction medium.

[00169] The hydrogen peroxide-triacetin solution and the sodium hydroxide solution may be added in a sequential manner as described, where the hydrogen peroxide-triacetin solution was added first, or they may be added to the water simultaneously through a "T" fitting placed before static mixer 125. If a T fitting is used, the hydrogen peroxide- triacetin solution and the 50% sodium hydroxide solution are introduced to opposite ends of the T fitting and the mixture is injected into the pipe of water. In other embodiments, the hydrogen peroxide and triacetin may be added separately, or sequentially, with either one first, or simultaneously, with the sodium hydroxide added either simultaneously with, or after, the hydrogen peroxide and triacetin.

[00170] The reaction medium was introduced to a residence chamber 145 which provided reaction time and contained a packing material to promote mixing. Residence chamber 145 was designed to be of a volume such that at a total flow rate of 3.79 L (one gallon) per minute, by the time the reaction medium reached sampling port 160, 30 seconds had elapsed since the sodium hydroxide solution had been added. A pH probe 155 monitored the pH of the mixture.

[00171] A controller 150 was interfaced to the flow meter 105 of the inlet water 90, and also to the chemical delivery diaphragm pumps 115 and 135. The controller 150 monitored the

rate of the inlet water 90 and governed the rate at which the hydrogen peroxide-triacetin solution and the NaOH solution were introduced. If the flow of inlet water 90 decreased because of lower PAA requirements at the point-of-use 195, the rates at which the hydrogen peroxide-triacetin solution and the NaOH solution were pumped decreased accordingly. If the flow of inlet water 90 increased because of higher PAA requirements at the point-of-use 195, the rates at which the hydrogen peroxide-triacetin solution and the NaOH solution were pumped increased accordingly. If the flow of inlet water 90 stopped, pumps 115 and 135 stopped. Thus, the generation of PAA was both continuous and intermittent, and was tailored to the PAA requirements at the point-of-use.

[00172] The rate of the reaction between the hydrogen peroxide and the triacetin to form PAA is dependent upon the temperature of inlet water 90. If the temperature of inlet water 90 is high (for example, about 21 °C (70 °F)),

the maximum conversion of triacetin into PAA may occur after

30 seconds, in which case the entire reaction medium was directed to the point-of-use 195. However, if the temperature of inlet water 90 is low (for example, about 0 °C (32 °F)),

longer reaction

times may be necessary to maximize the conversion of triacetin into PAA. Then, the reaction medium was directed through one or more residence chambers 165, 175, and 185 which also contained a packing material to promote turbulence and cause thorough mixing. Residence chambers 165, 175, and 185 were designed to be of volumes such that at a total flow rate of 3.79 L (one gallon) per minute, the reaction medium reached sampling port 170 in one minute, sampling port 180 in two minutes, and sampling port 190 in five minutes, to permit sampling at different time intervals.

[00173] Thus, depending on the temperature of the inlet water, the time to achieve the maximum conversion of hydrogen peroxide and triacetin into PAA was determined by sampling and analyzing the solution at sample ports 160, 170, 180, and 190. The entire flow was then

directed from the sampling port with the highest amount of PAA to the point-of-use 195. In practice, the PAA generated from the reaction of hydrogen peroxide with triacetin is typically diluted at the point-of-use 195.

[00174] Point-of-use 195 may be recirculating cooling water; municipal wastewater; poultry chill tank water; water used to sanitize meat, poultry, or seafood products; fruit and vegetable rinse water; or water used to clean and sanitize equipment used in the dairy, food, or beverage processing industries.

[00175] Example 14

[00176] Referring again to Fig. 1, a 208 L (55-gallon) drum container 120 of a hydrogen peroxide-triacetin solution was prepared by mixing triacetin 114 kg (251.5 lbs) with 50% hydrogen peroxide 135 kg (298.4 lbs). The resulting 249.5 (550 lbs) of hydrogen peroxide-triacetin solution had a specific gravity of 1.19 g/mL (10 lbs/gal) and contained 54.3% hydrogen peroxide and 45.7% triacetin. Thus, the mole ratio of hydrogen peroxide:triacetin was 3.8:1. The apparatus depicted in Fig. 1 was used to continuously and intermittently prepare an approximately 1% PAA solution.

[00177] Softened local city water was directed through pressure regulator 100 at a flow rate of 3.79 L (one gallon) per minute as measured by flow meter 105. The container 120 of hydrogen peroxide-triacetin solution was equipped with a draw down tube so that the flow rate of the mixture could be measured. Diaphragm pump 115 injected the hydrogen peroxide-triacetin solution through injection quill 110. The hydrogen peroxide-triacetin solution was then mixed with the softened water using static mixer 125. A container 140 of 50% NaOH was also equipped with a draw down tube to measure the flow pumped by diaphragm pump 135 into injection quill 130.

[00178] The percent of PAA generated was measured by quenching samples drawn through the sampling ports in mineral acid, followed by use of the ceric sulfate-iodometric

titration method. Table XVII summarizes the results of several trials where it was sought to maximize the conversion of triacetin into PAA and minimize the amount of 50% NaOH used.

[00179] Table XVII

Trial #	Sample Port	% PAA Generated	% Triacetin Converted to PAA	Hydrogen Peroxide-Triacetin Mix Flow Rate (mL/min)	NaOH Flow Rate (mL/min)	pH (PAA solution)
1	160	1.083	65.42	115.73	112.11	~13
2	170	1.073	64.82	115.73	112.11	13.19
3	160	0.955	57.32	116.79	122.12	13.37
4	160	1.058	63.39	116.79	114.93	13.11
5	160	1.061	63.3	116.79	98.1	12.9
6	160	1.102	68.96	110.82	85.01	12.91
7	170	1.055	55.59	132.64	99.77	12.95
8	160	0.978	59.91	115.5	74	12.62
9	180	0.936	56.7	114.25	78	12.64

[00180] It can be seen that the amount of PAA generated, and hence the conversion of triacetin into PAA, increased with the increasing flow rate of NaOH. For this mole ratio of hydrogen peroxide:triacetin of 3.8:1, the maximum amount of PAA was generated between 30 seconds (sample port 160) and two minutes (sample port 180) from the time the NaOH was added at injection quill 130. The flow rate of 74-78 mL/min of 50% NaOH was the optimum flow rate as it resulted in an efficient utilization of triacetin converted into PAA, consumed only a moderate amount of 50% NaOH, and had a lower pH that would have less impact on the pH of the receiving water.

[00181] Example 15

[00182] *E. coli* O157:H7 bacteria (ATCC 35150) was cultured in nutrient broth (Sigma, St. Louis, MO) by incubation for two days at 35°C. The bacteria were separated from the nutrient broth by centrifugation and carefully resuspended in two liters of sterile phosphate buffer, which was then split into two one-liter test solutions.

[00183] *Salmonella* typhimurium bacteria (ATCC 14028) was cultured in nutrient broth (Sigma, St. Louis, MO) by incubation for two days at 35°C. The bacteria were separated from the nutrient broth by centrifugation and carefully resuspended in two liters of sterile phosphate buffer, which was then split into two one-liter test solutions.

[00184] The amount of *E. coli* O157:H7 and *Salmonella typhimurium* bacteria were measured by serial dilution and plating on 3M *E. coli* plates and 3M *Enterobacteriaceae* Petrifilms, respectively.

[00185] A 1000 ppm stock of PAA was made by weighing 0.6386 g of Perasan MP-2 (5.83% hydrogen peroxide and 15.66% PAA) and adding water up to 100 g. Perasan MP-2 is an equilibrium product of PAA (Enviro Tech Chemical Services, Inc., Modesto, California). Side-by-side, one of the *E. coli* and one of the *Salmonella* test solutions were treated with a nominal dose of 1.0 ppm PAA by adding one mL of the 1000 ppm PAA stock solution to each sample. After one minute, approximately 100 mL of each test solution was removed, and sodium thiosulfate (0.5 g) was added to neutralize the PAA and hydrogen peroxide oxidants. After five minutes, sodium thiosulfate (0.5 g) was added to the remaining test solution to neutralize the PAA and hydrogen peroxide in the rest of the test solutions. The amount of viable *E. coli* O157:H7 and *Salmonella typhimurium* bacteria remaining in the test solutions at the one-minute and five-minute contact times were measured by serial dilution and plating on 3M *E. coli* Petrifilms and 3M *Enterobacteriaceae* Petrifilms, respectively.

[00186] A 1% solution of PAA was prepared by combining 30.90 g of a mixture of 50% hydrogen peroxide and triacetin (54.22% hydrogen peroxide and 45.78% triacetin) with 929.4 g of water, and then adding 50% NaOH (40.53 g). The PAA solution had a pH of 12.79. The mole ratio of this mixture was 3.8:7.8:1 NaOH:hydrogen peroxide:triacetin. One minute after preparation, the second one-liter *E. coli* solution and the second one-liter *Salmonella* solution

were treated with a nominal dose of 1.0 ppm PAA by adding 0.10 g of the 1% PAA solution to each sample. The test solution contact times, neutralization, and plating procedures were replicated for those that employed PAA from Perasan MP-2.

[00187] All 3M *E. coli* Pertrifilms and 3M *Enterobacteriaceae* Petrifilms were incubated at 35°C for 24 hours. After incubation, the plates were enumerated.

[00188] Table XVIII shows the microbiological results of the *E. coli* O157:H7 test solutions before treatment with PAA, one minute after the samples were dosed with a nominal 1.0 ppm PAA, and five minutes after being dosed with PAA. The *E. coli* test solution started with $\log_{10}$ 6.99 CFU/mL prior to being treated with 1.0 ppm of PAA from Perasan MP-2 or with 1 ppm of PAA from the 1% PAA solution.

[00189] The *E. coli* test solution treated with 1.0 ppm PAA from Perasan MP-2 contained a $\log_{10}$ 6.74 CFU/mL after one minute. This corresponds to 43.48% reduction. After five minutes, $\log_{10}$ 6.34 CFU/mL remained (77.50% reduction). The *E. coli* solution treated with a nominal 1.0 ppm PAA from the 1% PAA solution contained a $\log_{10}$ 6.78 CFU/mL after one minute and 6.63 CFU/mL after five minutes, respectively, corresponding to 38.02% and 56.12% reductions, respectively.

[00190] Table XVIII

***E. coli* O157:H7**

Description	$\log_{10}$ CFU/ml remaining	$\log_{10}$ reduction	% reduction
Control	6.99	N/A	N/A
Perasan MP-2 (1 min)	6.74	0.25	43.48
Perasan MP-2 (5 min)	6.34	0.65	77.50
1% PAA solution (1 min)	6.78	0.21	38.02
1% PAA solution (5 min)	6.63	0.36	56.12

[00191] Table XIX shows the microbiological results of the *Salmonella typhimurium* test solutions before treatment with PAA, one minute after the samples were dosed with a nominal 1.0 ppm PAA, and five minutes after being dosed with PAA. The *Salmonella* test solution started with $\log_{10}$ 7.07 CFU/mL prior to being treated with PAA from Perasan MP-2 or with the 1% PAA solution.

[00192] The *Salmonella* solution treated with a nominal 1.0 ppm PAA from Perasan MP-2 contained a $\log_{10}$ 1.76 CFU/mL after one minute and 0.70 CFU/mL after five minutes, corresponding to >99.999% reduction at both contact times. The *Salmonella* test solution treated with 1.0 ppm PAA from the 1% PAA solution contained a $\log_{10}$ 6.69 CFU/mL after one minute. This corresponds to 58.31% reduction. After five minutes, $\log_{10}$ 4.95 CFU/mL remained (99.24% reduction).

[00193] Table XIX

Salmonella typhimurium

Description	$\log_{10}$ CFU/ml remaining	$\log_{10}$ reduction	% reduction
Control	7.07	N/A	N/A
Perasan MP-2 (1 min)	1.76	5.31	99.9995
Perasan MP-2 (5 min)	0.70	6.37	>99.9999
1% PAA solution (1 min)	6.69	0.38	58.31
1% PAA solution (5 min)	4.95	2.12	99.24

[00194] The data in tables XVIII and XIX demonstrate that the 1% PAA had antimicrobial efficacy. After five minutes, the efficacy of this PAA was 99.24%, about equal to that of the Perasan MP-2, which was > 99.9999%. The difference in efficacy at one minute was likely not of statistical significance, given that the comparison was only at the one-minute interval.

Claims

1. A method of generating a non-equilibrium solution of peracetic acid, comprising:

a. providing water;

b. introducing a hydrogen peroxide-triacetin solution to the water;

c. mixing the hydrogen peroxide-triacetin solution and the water to form a mixture;

d. adding an aqueous source of an alkali metal hydroxide in the form of sodium hydroxide to the mixture, wherein the sodium hydroxide is 1.82% to 7.28% weight, further, wherein the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is 4.2:3.8:1; and

e. forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

2. The method of claim 1, further comprising, prior to step (b), a step of providing the hydrogen peroxide-triacetin solution comprising: introducing triacetin to a solution of aqueous hydrogen peroxide; and allowing the triacetin and the solution of aqueous hydrogen peroxide to mix to form the hydrogen peroxide-triacetin solution.

3. The method of claim 1, wherein the provided water is an aqueous stream.

4. The method of claim 1, wherein the provided water is contained in a mixing tank or other vessel.

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5. The method of claim 1, wherein step (d) is performed simultaneously with step (b).

6. The method of claim 1, further comprising allowing the reaction medium sufficient time to convert the hydrogen peroxide-triacetin into peracetic acid, said time being 30 seconds to five minutes.

7. The method of claim 1, further comprising, after step (e), a step of sampling the reaction medium to determine the time required to maximize the amount of hydrogen peroxide-triacetin that is converted into peracetic acid.

8. The method of claim 1, wherein the peracetic acid solution is for immediate use.

9. The method of claim 1, further comprising, after step (e), a step of adding a source of acid to stabilize the non-equilibrium solution of peracetic acid for storing and using throughout a working day.

10. The method of claim 1, wherein the peracetic acid solution is prepared on a site having a point-of-use.

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METHODS AND COMPOSITIONS FOR THE GENERATION OF PERACETIC ACID ON SITE AT THE POINT-OF-USE

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FIELD OF THE INVENITON

[0001] The invention relates to liquid compositions and methods of using the liquid compositions for the generation of a peroxyacetic acid sanitizer on site in proximity to the point-of-use. [redacted]

BACKGROUND OF THE INVENTION

[0002] **A. Liquid PAA Disinfectants and Sanitizers**

[0003] Disinfectants and sanitizers based on peroxyacetic acid (PAA), also commonly known as peracetic acid, are used in the dairy, food and beverage processing industries for clean-in-place pipeline and equipment disinfecting and cleaning, for fruit and vegetable washing, and in the treatment of meat, poultry and seafood products. Peroxyacetic acid disinfectants are also used in the treatment of cooling water, process water, and municipal wastewater. Other uses include slime and biofilm removal in papermaking processes.

[0004] Peroxyacetic acid products are supplied as stable equilibrium ternary aqueous solutions of peroxyacetic acid, acetic acid, and hydrogen peroxide. They are prepared in advance of delivery, typically by reacting hydrogen peroxide with acetic acid in the presence of a mineral acid catalyst. Although some PAA is formed immediately, the PAA does not reach its maximum concentration until after several days. A metal chelating agent, such as hydroxyethylidene

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diphosphonic acid (HEDP) or dipicolinic acid, is also introduced to suppress the transition metal cation catalyzed decomposition of peroxygen compounds. The PAA product is then placed in containers, such as totes, drums and pails, in preparation for shipment to the end user. Typical commercial products have concentrations of PAA of about 1-15% w/w, although a concentration of up to 30% is possible. However, the latter concentration product only finds captive use because of its extremely hazardous and explosive properties.

[0005] There are several problems with the use of equilibrium solutions of PAA. First, the low concentration of PAA (1-15%) means that most of the weight (85-99%) of the products consist of substantial amounts of inert ingredients, such as water, acetic acid, and hydrogen peroxide. This results in the need for larger product storage areas and causes increased transportation and handling costs. Second, the products are inefficient in the use of both hydrogen peroxide (hydrogen peroxide) and acetic acid (AA). To maintain adequate storage stability of the PAA, either hydrogen peroxide or AA must be present at a level on a weight percentage basis that is greater than the level of PAA, which increases raw material costs. Third, the presence of the HEDP or dipicolinic acid stabilizer limits the amount of PAA that can be applied to certain foodstuffs because the amount of HEDP or dipicolinic acid is regulated by the Food and Drug Administration (FDA). Fourth, the reaction between hydrogen peroxide and AA is quite slow in some cases, and typically requires several days' time before the PAA product can be tested for quality confirmation. Therefore, manufacturers of these products must store large inventories of the PAA product before it can be shipped to the end user. Fifth, for transportation purposes, equilibrium solutions that are greater than 6% PAA are considered to be dangerous products and must be labeled with the DOT marking "Organic Peroxide", Hazard Class 5.2, 8 (oxidizer, corrosive). If producers and end-users exceed the yearly threshold amount of just 6,666 lbs. of 15% PAA, they must file Risk Management Plans with both federal EPA and state

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authorities. This is an arduous and time-consuming process. In addition, producers of equilibrium solutions of PAA that are over 6% PAA must obtain permitting by the local fire department and pay an extra hazardous material shipping fee when the product is shipped from their facility. On the other hand, there are much less stringent requirements for products that contain hydrogen peroxide, and all reporting, permitting and shipping restrictions are lifted for products that contain less than 27% hydrogen peroxide.

[0006] As a result of these problems, there have been various attempts to make non-equilibrium solutions of PAA on site, at the point-of-use. For example, U.S. patent no. 7,012,154 discloses a system in which AA, hydrogen peroxide, water, and sulfuric acid are fed to a jacketed reactor for the production of PAA. A wiped-film distillation column attached to the reactor condenses and isolates the pure PAA from the gas phase and immediately introduces it to the receiving water. This system suffers from a number of drawbacks. First, it is extremely capital intensive due to the high cost of the equipment, including the reactor, heater, pumps, distillation column, and computerized control system which ensures accurate metering of the reagents. Second, there are significant safety hazards associated with the production of pure PAA due to its explosive properties. Third, the equipment and synthesis process is very complex and requires knowledgeable and highly trained technicians to continuously operate and maintain the system in a safe and effective manner.

[0007] Other attempts to make non-equilibrium solutions of PAA at the point-of-use are based on electrolytic processes. U.S. patent nos. 6,171,551 and 6,387,236 disclose processes that employ a cell divided by an ion-exchange membrane in which PAA (and other oxidants including hydrogen peroxide and ozone) are produced in the anode compartment which consists of an aqueous solution of acetic acid or acetate salt. In addition to the high costs of electricity and electrolysis equipment, these processes also result in a very low yield of PAA from the

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acetyl precursor. For example, the '551 patent reports that less than 14 ppm of PAA was produced after 90 minutes of electrolysis of a 490,000 ppm anolyte of aqueous potassium acetate. In addition, these processes are difficult to perform intermittently.

[0008] Other methods of generating non-equilibrium solutions of PAA on site, using electrolysis, are described in WIPO International Publication Nos. WO 2004/0245116 and WO 2008/140988, and U.S. Patent Application Publication No. 2009/0314652. These references disclose cation membrane-divided electrolysis cells and the use of gas diffusion electrodes to effect the cathodic reduction of oxygen gas to hydrogen peroxide under alkaline conditions. The hydrogen peroxide was then allowed to react with acetic acid or an acetyl precursor to form PAA in the bulk solution, whereupon the catholyte was directed to the acidic anode compartment of the cell to stabilize the PAA. This system suffers from several disadvantages. Due to the low solubility of oxygen in water (about 8 ppm maximum), the concentration of electroactive species is very low, which forces the cell to operate at low current density (amperage per surface area of electrode). In order to produce a meaningful amount of hydrogen peroxide, the cells must have a very large surface area. This requires high capital equipment costs and a very large footprint for the electrolysis equipment. Another disadvantage of this system is that it is very difficult to maintain steady-state conditions and simultaneously balance the feed of acetic acid or acetyl precursor to the cathode compartment with the concurrent withdrawal of acidified PAA solution from the anode compartment. This is because cations carrying the cell current through the cation exchange membrane are always hydrated so as the cations move through the membrane, they are accompanied by water molecules. As a result, the volume of the anolyte decreases and the volume of the catholyte increases, making the steady-state condition difficult to maintain. It is difficult to perform this process intermittently. Finally, this system can only be of economic value if the source of oxygen is air, which comprises 23% oxygen. However, the carbon dioxide

contained in air causes carbonates to precipitate, which impedes the flow of electrical current, limiting or eliminating the production of hydrogen peroxide, and hence, PAA.

[0009] Another method of making a non-equilibrium solution of PAA at the point-of-use is disclosed in WIPO International Publication No. WO 2008/140988 and U.S. Patent Application Publication Nos. 2009/0005590 and 2007/0082832. These references disclose biosynthetic methods of producing peracids from carboxylic acids and carboxylic acid esters. These methods involve the use of perhydrolase enzymes to catalyze the perhydrolysis of the carboxylic acid or ester into the peracid using a solid or liquid source of hydrogen peroxide. Although these methods can produce compositions containing up to 20 parts of peracid to one part of hydrogen peroxide, they are limited by the amount of peracid that can be produced before the perhydrolase enzyme is oxidized by the reactant products and ceases to function as intended. The highest concentration of peracid disclosed by these references was 0.16% (1,600 mg/L). However, peracid or PAA solutions that are produced with enzymes have limited appeal because they are very expensive to produce, and for regulatory reasons, the enzyme must be removed from the solution before the solution can be applied to food or hard surfaces for disinfection purposes. Removing the enzyme from the solution is not an easy task; thus, it is typically not done. Therefore, PAA solutions prepared by enzymatic methods are not suitable for the more broad commercial uses in the food, dairy, beverage, meat, and poultry industries, which are regulated by the FDA and the Environmental Protection Agency (EPA).

[0010] Another method of generating non-equilibrium PAA at its point-of-use requires substituting the traditional mineral acid catalyst with sulfonic acid ion-exchange resins, as disclosed in U.S. patent no. 5,122,538. A solution containing a 1.5:1 mole ratio of AA to hydrogen peroxide was passed through a column packed with a sulfonic acid ion-exchange resin and produced a solution of 15% PAA within 30 minutes. The method described in the '538

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patent suffers from the limitation that it requires a large volume of expensive resin bed in order to be effective. Moreover, all existing ion exchange resin systems are limited by the fact the resin is subject to oxidative degradation by PAA and have a short limited lifespan.

[0011] Other attempts to produce non-equilibrium PAA solutions on site, at the point-of-use, for bleaching cellulosic materials have reacted hydrogen peroxide with acetic anhydride. For example, U.S. patent no. 3,432,546 discloses a process where hydrogen peroxide, acetic anhydride, and an ammonium hydroxide catalyst were metered to a tubular reactor to continuously produce a solution containing 3.25% PAA with a conversion of 78% hydrogen peroxide. However, the process generated measurable amounts of diacetyl peroxide (0.44%) which is an explosion hazard. Moreover, the reaction product would be unsuited for any application other than cellulosic bleaching purposes because there was no attempt to remove the ammonium hydroxide catalyst from the reaction medium. Ammonium hydroxide is an undesirable contaminant in PAA products that are used as disinfectants and sanitizers in the dairy, food, and beverage processing industries, and in PAA products used in fruit and vegetable washing and in the treatment of meat, poultry, and seafood.

[0012] Another process for generating non-equilibrium solutions of PAA on site, at the point-of-use was disclosed in U.S. Patent Application Publication No. 2009/0043132. This process utilized introduction of hydrogen peroxide into a sidestream of the water requiring treatment. This was followed by introducing acetic anhydride, whereupon PAA was generated *in-situ*. It was found that acetic anhydride preferentially reacted with hydrogen peroxide rather than undergo undesirable hydrolysis with water. Within 20 minutes, up to 3000 ppm of PAA was generated in the sidestream which was then reconstituted with the main body of water and diluted further. All processes that employ acetic anhydride suffer the limitation that acetic anhydride is expensive, very corrosive, an irritant, and highly flammable.

[0013] Yet another process for generating non-equilibrium solutions of PAA on site at the point-of-use is disclosed in WIPO International Publication No. WO 01/46519 A1. This process utilized the metering of an aqueous solution of hydrogen peroxide into an agitated tank and co-metering a solid dry source of tetraacetylethylenediamine (TAED) from a storage hopper using a screw feeder. The agitator kept the solid TAED suspended in the hydrogen peroxide solution which was then fed to an in-line static mixer where aqueous sodium hydroxide was introduced. The mixture was then directed through 200 meters of coiled tubing immersed in a cooling tank so that the temperature rise accompanying the exothermic reaction was contained. Upon exiting the coiled tubing, the mixture containing PAA could be directed to the water requiring treatment. Disadvantages of this approach include the difficulty of accurately metering a solid and a liquid simultaneously, and the high capital equipment cost of the metering system, electronic controllers, agitation tank, coiled tubular reactor, and the cooling system.

[0014] Thus, there is a need for a method to make non-equilibrium PAA on site at the point-of-use that addresses the above problems.

[0015] **B. Solid PAA Bleaches and Stain Removers**

[0016] PAA is also used in laundry bleaching applications where it is generated *in-situ* in the laundry wash water. The PAA is typically produced from a solid source of hydrogen peroxide, such as sodium percarbonate or sodium perborate. The hydrogen peroxide must be in the presence of a solid acetyl precursor, most typically, tetraacetylethylenediamine (TAED). When dissolved in laundry wash water, the TAED undergoes a perhydrolysis reaction with hydrogen peroxide to form PAA. TAED is the preferred acetyl precursor because it possesses low toxicity, is of low environmental impact, and is readily biodegradable. However, TAED-based laundry bleaches have several problems. First, of the four acetyl groups on TAED, only two are known to be available for perhydrolysis, making TAED an expensive acetyl precursor on

a weight basis. Second, TAED has low water solubility, especially at the cooler water temperature bleaching cycles that are less damaging to fabrics. This is a major drawback, as current consumer trends in energy conservation are towards bleaching laundry fabrics with cool temperature water, rather than heated water. Undissolved TAED in cool temperature water is less unavailable as an acetyl precursor for the dissolved source of hydrogen peroxide and can even deposit on fabrics, necessitating a separate rinse step to remove it. Third, over time, when exposed to high humidity, solid TAED can react with solid source of hydrogen peroxide and the free water to form PAA, as well as degrade the activity, making it less effective overtime. Because the PAA is volatile, it imparts an undesirable pungent odour to the product. ~~Thus, there is a need for a solid peroxygen bleach that overcomes the deficiencies of the TAED containing bleaches~~.

EP1618786 discloses a composition for bactericide production and a process for producing organic peracid. The sterilising agent is a water based solution obtained by combining triacetin and hydrogen peroxide. The ratio between these two components is 1/10 and 20/1. The formulation can comprise 50% by weight triacetin, 15% of hydrogen peroxide, 0.17% hydrogen peroxide and 57.3% of remaining peroxide.

JP2004230323 discloses a sterilising agent for hard surfaces. Triacetin is used as the agent capable of liberating acetic acid, hydrogen peroxide is added and a metal hydroxide has to be used to bring the pH to 8-12. One example discloses a formulation of 0.23mol of triacetin, 0.73mol of hydrogen peroxide and 0.5 mol of sodium hydroxide.

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US2010/0227000 discloses a multi-part kit system for the preparation of a disinfectant of the peracetic acid type comprising a solid part A comprising at least one acetyl donor, a liquid part B which can be an aqueous solution of hydrogen peroxide (5-10% by wt). A water solution of 0.05 to 5% sodium hydroxide is also added.

US6221341 discloses a teeth whitening composition. Peracetic acid is the whitening agent and is generated by glyceryl triacetate with the addition of hydrogen peroxide in the presence of a base the regulates the pH.

SUMMARY OF THE PRESENT INVENTION

According to the present invention there is provided a method of generating a non-equilibrium solution of peracetic acid, comprising:

a. providing water;

b. introducing a hydrogen peroxide-triacetin solution to the water;

c. mixing the hydrogen peroxide-triacetin solution and the water to form a mixture;

d. adding an aqueous source of an alkali metal hydroxide in the form of sodium hydroxide to the mixture, wherein the sodium hydroxide is 1.82% to 7.28% weight and the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is 4.2:3.8:1; and

e. forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

In one embodiment, liquid compositions for use in generating non-

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equilibrium solutions of PAA are provided. These hydrogen peroxide-acetyl precursor solutions comprise a solution of aqueous hydrogen peroxide, a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide, a trace of peracetic acid, and water. A preferable acetyl precursor is triacetin, which displays an unexpectedly high solubility in hydrogen peroxide. These hydrogen peroxide-acetyl precursor solutions may be used for generating a non-equilibrium solution of peracetic acid for use as a disinfectant or sanitizer on a site having a point-of-use of peracetic acid.

The hydrogen peroxide- acetyl precursor solutions are advantageous in that they are formulated such that sufficient acetyl precursor can be dissolved in an amount of the hydrogen peroxide that would fully perhydrolyze all of the acetyl groups of the acetyl precursor.

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Moverover, the hydrogen peroxide component of these compositions is remarkably stable. These compositions overcome one or more problems of the known prior art, and are free of the regulatory reporting, permitting, and shipping restrictions that govern equilibrium PAA products.

[0019] In another embodiment, a method of preparing a hydrogen peroxide-acetyl precursor solution is provided. The method comprises introducing a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide to a solution of aqueous hydrogen peroxide; and allowing the acetyl precursor and the aqueous hydrogen peroxide to mix to form a hydrogen peroxide-acetyl precursor solution.

[0020] A further embodiment is a method of continuously or intermittently generating a non-equilibrium solution of peracetic acid on a site having a point-of-use of peracetic acid for use as a disinfectant or sanitizer. Water is provided on the site having the point-of-use. The water may be a flowing aqueous stream or it may be contained in a mixing tank or other vessel. A hydrogen peroxide-acetyl precursor solution is provided on the site having the point-of-use. In one embodiment, the liquid acetyl precursor is triacetin and the solution that is formed is a hydrogen peroxide-triacetin solution. The hydrogen peroxide-acetyl precursor solution may be prepared off site and then transported to the site having the point-of-use, or the acetyl precursor and the aqueous hydrogen peroxide may be transported to the site having the point-of-use and then used to prepare the hydrogen peroxide-acetyl precursor solution on the site. The hydrogen peroxide-acetyl precursor solution is then introduced to the water. Alternatively, and in another embodiment, the acetyl precursor and the aqueous hydrogen peroxide may be introduced to the water separately, either sequentially, with either one first, or simultaneously. The hydrogen peroxide-acetyl precursor solution and the water are mixed to form a mixture. An aqueous source of an alkali metal or earth alkali metal hydroxide, such as sodium hydroxide, is provided on the site having the point-of-use. The aqueous source of an alkali metal or earth alkali metal

hydroxide is added to the mixture. Alternatively, and in another embodiment, the aqueous source of alkali metal or earth alkali metal hydroxide may be added simultaneously with the hydrogen peroxide-acetyl precursor solution or with the acetyl precursor and the aqueous hydrogen peroxide, to the water. A reaction medium comprising a non-equilibrium solution of peracetic acid is formed. In the reaction medium, the hydrogen peroxide reacts with the acetyl precursor to form peracetic acid. Almost instantaneously, a non-equilibrium solution of PAA is formed. Within ~~about~~ 30 seconds to ~~about~~ five minutes, the amount of hydrogen peroxide and acetyl precursor that are converted into peracetic acid is maximized. When triacetin is used as the acetyl precursor and assuming all three acetyl groups are reacted, ~~about~~ 40.9% to ~~about~~ 85.7% of the triacetin is converted into peracetic acid, and the percent of hydrogen peroxide remaining is ~~about~~ 0.078% to ~~about~~ 1.88%. The reaction medium may optionally be sampled at various times after the addition of the aqueous source of alkali metal or earth alkali metal hydroxide to determine the time required to maximize the amount of hydrogen peroxide and acetyl precursor that are converted into peracetic acid.

[0021] The non-equilibrium solutions of PAA that are formed comprise PAA, unreacted hydrogen peroxide, unreacted acetyl precursor, the product of the perhydrolysis reaction of the acetyl precursor, the aqueous source of alkali metal or earth alkali metal hydroxide, and water. The peracetic acid solutions are alkaline, having a pH of ~~about~~ 11.2 to ~~about~~ 13.37. The reaction is remarkably fast, proceeds with a high conversion of the acetyl precursor into PAA. This method produces solutions with a high level of PAA, from ~~about~~ 1% to ~~about~~ 7.1%.

[0022] The PAA solution is introduced to the receiving water at the point-of-use. It may be introduced immediately, or stabilized with the addition of a source of acid and used throughout a working day. The receiving water may be water used in the dairy, food, and beverage processing industries for clean-in-place pipeline and equipment cleaning; for fruit and

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vegetable washing; and in the treatment of meat, poultry, and seafood products. The receiving water may also be cooling water, oil and gas process water, or municipal wastewater. Other uses include slime and biofilm removal in papermaking processes.

[0023] This method addresses one or more problems of the known prior art. The method is easy to perform, inexpensive to operate, and requires only basic and common equipment. The method is safe, in that it does not isolate pure PAA, and there is no possibility of a catastrophic event due to an equipment failure, such as over-pressurization or explosions. When triacetin is used as the acetyl precursor, it is inexpensive, non-toxic, safe, non-corrosive, non-irritating, non-flammable, sanctioned as Generally Recognized as Safe (GRAS) by the FDA, and converted quickly and with a high conversion rate into PAA. The method does not require the use of perhydrolase enzymes, acetic acid, or acetic anhydride, all of which suffer from the aforementioned limitations and problems. The method generates a PAA product that does not contain ammonium hydroxide. The method can be performed to generate a PAA product in a continuous or intermittent fashion. Further, the method allows the end-user the choice of utilizing the on site generated PAA immediately or later over the course of a working day.

[0024] ~~In another embodiment, a freely flowable, solid peroxygen composition for use in laundry bleaching and stain removal is provided. The solid composition comprises a liquid acetyl precursor, a water soluble solid source of hydrogen peroxide, and a water soluble solid source of alkalinity. The liquid acetyl precursor is preferably triacetin. The composition produces peracetic acid upon being introduced to water. Upon dissolution, the solid hydrogen peroxide source releases hydrogen peroxide and the solid source of alkalinity dissolves to increase the pH, resulting in the formation of peracetic acid from the reaction of the hydrogen peroxide and the acetyl precursor.~~

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[0025] The solid composition is used as a bleaching agent and a stain remover for the treatment of articles such as fabrics, dentures, textile garments, and equipment used in the food and beverage industry. The solid composition is advantageous in that it does not give rise to an undesirable PAA odor upon storage, contains an acetyl precursor that efficiently utilizes the acetyl groups it contains, and has a high solubility in cool temperature water which quickly and efficiently produces PAA without the need for elevated water temperatures. Moreover, the acetyl precursor used in making the solid composition may also be used in preparing the liquid compositions described in section 3 above, streamlining manufacturing practices by using the same acetyl precursor as an ingredient in liquid as well as solid compositions.

[0026] Surprisingly, this freely flowable, solid composition includes a liquid acetyl precursor, triacetin. In addition, it is surprising that this composition does not produce a strong odor of peracetic acid during storage.

[0027] In another embodiment, a method of preparing the freely flowable, solid peroxygen composition for use in laundry bleaching and stain removal is provided.

BRIEF DESCRIPTION OF THE DRAWING

[0028] Figure 1 is a schematic representation of an example of a system that may be used to continuously or intermittently generate PAA for on a site having a point-of-use of peracetic acid. acid In another embodiment, a freely flowable, solid peroxygen composition for use in laundry bleaching and stain removal is provided according to an embodiment.

DETAILED DESCRIPTION OF THE INVENTION

[0029] 1. **Definitions**

[0030] The terms "PAA" and "peracetic acid" mean peroxyacetic acid or peracetic acid, and/or the conjugate base of peracetic acid (the peracetate ion).

[0031] The terms "percent" and "%" mean weight percent, except when referring to the percent converted.

[0032] The term "receiving water" means the water that is being treated.

[0033] The term "point-of-use" means the location where the peracetic acid enters the receiving water.

[0034] **2. Experimental Methods**

[0035] PAA and hydrogen peroxide in solution were determined using the ceric sulfate-sodium thiosulfate method. The method involves adding a known weight of sample containing the PAA and hydrogen peroxide (or dilution) to a beaker containing 50 ml of chilled 0.9 N sulfuric acid. Using a transfer pipette, 1-2 grams is accurately weighed to a sample cup and reverse osmosis (RO) or deionized water is added to provide a workable volume. Five drops of ferroin indicator is then added with stirring, and the sample is then titrated with 0.1 N ceric sulfate until the sample turns from purple to blue. The volume of ceric sulfate required in mL is recorded. Then, one or two small scoops of potassium iodide are added to the sample cup, which turns the solution dark brown. The sample is then titrated with 0.1 N sodium thiosulfate to discharge the brown coloration and form a pale straw coloration. Then, 10-15 drops of 0.5% starch indicator solution is introduced and the sample turns blue-black. The sodium thiosulfate is then added dropwise until the color transitions to a bright orange. The volume of sodium thiosulfate in mL was recorded. The percent hydrogen peroxide and the percent PAA are calculated as follows:

$$\% \text{ hydrogen peroxide} = \frac{\text{mL 0.1 N ceric sulfate} \times 0.17}{\text{wt. sample}}$$

$$\% \text{ PAA} = \frac{\text{mL 0.1 N sodium thiosulfate x 0.38}}{\text{wt. sample}}$$

[0036] For kinetic runs which involved measuring the PAA and hydrogen peroxide in samples at one, three, and five minute intervals after adding the reactants together, the generation reaction was quenched by adding a sample of the reaction medium to the sulfuric acid. This halted the generation reaction and stabilized the PAA and hydrogen peroxide to elevated pH degradation reactions. This permitted the samples to be taken at the appropriate time interval (one, three, and five minutes), and analyzed later.

[0037] In circumstances where low concentrations of PAA were used, the modified DPD method (U.S. patent no. 7,651,724) was employed. This analytical technique is based on a modified DPD (N,N-diethyl-*p*-phenylenediamine) colorimetric method accepted by the EPA for measuring total chlorine in drinking water and wastewater. It relies on the ability of PAA to behave like chlorine in that it rapidly and quantitatively oxidizes iodide ion (I^-) into iodine (I_2) that reacts with a color indicator (DPD), which turns the solution a shade of pink, the intensity of which is proportional to the concentration of the PAA. A colorimeter is used that is programmed to measure the intensity (absorbance) of the pink coloration and display the result in terms of ppm as Cl_2. A calculation converts this number into the ppm as PAA, based on the weight ratio of PAA to Cl_2 (76/71 = 1.07).

[0038] Hydrogen peroxide does not interfere with the measurement for PAA provided the analysis is completed within 30 seconds of introducing the DPD reagent. In order for the hydrogen peroxide to be measured, it must be activated by addition of a sodium molybdate catalyst and then given time (six minutes) to react with the I^- ion to liberate I_2. Upon addition of the DPD indicator, the intensity of the pink coloration measured by the colorimeter is now the

sum of the PAA and hydrogen peroxide concentrations expressed as ppm Cl_2. After subtracting the ppm reading obtained earlier for the contribution due to PAA, a calculation is then used to convert this number into ppm as hydrogen peroxide, and is based on the weight ratio of hydrogen peroxide to Cl_2 (34/71 = 0.479).

[0039] **3. Liquid Compositions for Generating Non-Equilibrium Solutions of PAA On a Site Having a Point-of-Use**

[0040] In an embodiment, liquid compositions for generating non-equilibrium solutions of PAA on a site having a point-of-use of peracetic acid are provided. These solutions are referred to as "hydrogen peroxide-acetyl precursor solutions." These solutions comprise aqueous hydrogen peroxide, a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide, a trace amount of peracetic acid, and water.

[0041] The hydrogen peroxide-acetyl precursor solutions are prepared by the following steps.

[0042] (a) Introducing a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide to a solution of aqueous hydrogen peroxide.

[0043] Triacetin is a preferable liquid acetyl precursor and 50% hydrogen peroxide is a preferable solution of aqueous hydrogen peroxide. The triacetin is preferably added to the 50% hydrogen peroxide at room temperature. Alternatively, 70% hydrogen peroxide can be used, but it is not widely available and requires special handling and transportation. Given that the solubility of triacetin in water is only 7% at 25°C, triacetin is unexpectedly highly soluble in 50% hydrogen peroxide. It solubilizes without an exotherm (temperature increase).

[0044] The mole ratio of hydrogen peroxide:triacetin is about 2.98:1 to about 12.84:1.

[0045] (b) Allowing the acetyl precursor and the aqueous hydrogen peroxide to mix to form a hydrogen peroxide-acetyl precursor solution.

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[0046] The solution is mixed until a homogenous solution has been formed. The solution is allowed to mix by diffusion or by using a mixing device that is suitable for mixing liquids together. For example, 50% hydrogen peroxide can be introduced to a batch tank equipped with an overhead agitator blade. Triacetin can then be introduced to the 50% hydrogen peroxide when the agitator is in motion so that the components are thoroughly mixed.

[0047] The solution that is formed is a hydrogen peroxide-acetyl precursor solution, which also includes varying amounts of water, depending on the formulation used. When triacetin is used as the liquid acetyl precursor, the solution that is formed is a hydrogen peroxide-triacetin solution. One such hydrogen peroxide-triacetin solution comprises about 23% to about 40% hydrogen peroxide (from 50% aqueous hydrogen peroxide), about 20% to about 52% triacetin, water, and a trace amount of PAA that is formed within the first day of preparing the hydrogen peroxide-acetyl precursor solution. The pH is about 1.46 to about 2.2 and no deliberate attempt is made to adjust the pH further.

[0048] A preferable hydrogen peroxide-triacetin solution comprises about 27.15% hydrogen peroxide (from 50% aqueous hydrogen peroxide), about 45.67% triacetin, water, and a trace amount of PAA that is formed within the first day of preparing the hydrogen peroxide-acetyl precursor solution. The mole ratio of hydrogen peroxide:triacetin is about 3.8:1.

[0049] The hydrogen peroxide-acetyl precursor solutions prepared by this method may be used to generate non-equilibrium solutions of PAA on a site having a point-of-use of peracetic acid, in the methods described in section 4 below. The hydrogen peroxide-acetyl precursor solutions may be transported in any container suitable for transporting liquids, such as plastic tote bins of 1041-1249 L (275-330 gallons), drums of 57-208 L (15-55 gallons), 19 L (five-gallon) pails, 3.79 L (one-gallon) jugs, or other containers.

[0050] Example 1

[0051] Triacetin (83.05 g) was added to 50% hydrogen peroxide solution (75 g) to form 158.05 g of a mixture. There was no evolution of gas indicating the hydrogen peroxide had decomposed to oxygen, nor was there any exotherm indicating that a chemical reaction had occurred. The solution did not turn oily or cloudy but remained clear. The solution was calculated to comprise 23.73% hydrogen peroxide and 52.55% triacetin, with the balance being water, and had a measured pH of 2.2. The mole ratio of hydrogen peroxide to triacetin was calculated to be 2.98:1. Titration of the solution immediately upon preparation yielded 24.1% hydrogen peroxide, which is within experimental error of the calculated amount of hydrogen peroxide.

[0052] This experiment showed that triacetin is remarkably soluble in 50% hydrogen peroxide. The solubility of triacetin was found to be at least 52.55%. This is surprising, given that the solubility of triacetin in water is only 7% at 25°C.

[0053] In order to test the stability of the mixture, the amount of hydrogen peroxide remaining and the amount of PAA generated was tracked for several months. The mixture was held at ambient temperature away from sources of light. Table I summarizes the data.

[0054] Table I

Day #	% Hydrogen Peroxide Remaining	% PAA Generated
1	23.79	0.00
29	23.32	1.28
57	22.56	2.36
92	21.06	4.22
120	19.54	6.05
134	18.57	6.87

[0055] Table I shows that although there was a steady depletion in hydrogen peroxide, it was accompanied by an increase in PAA. By the end of the 134-day study, the composition had

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lost 5.22% hydrogen peroxide. Had all of the depleted hydrogen peroxide gone towards the generation of PAA, 11.67% PAA would have been generated. Because 6.87% PAA was generated, unaccounted losses of hydrogen peroxide amounted to 2.15%. This loss of hydrogen peroxide was surprisingly small given that hydrogen peroxide is often used to destroy organic compounds by oxidation and the composition originally contained 52.55% of triacetin, an organic compound.

[0056] Example 2

[0057] A formulation containing hydrogen peroxide and triacetin in a mole ratio of 12.84:1 was prepared by adding triacetin (100.0 g) to 50% hydrogen peroxide (400.0 g) so that the mixture was 40% hydrogen peroxide and 20% triacetin, with the balance being water. The pH of the solution was 1.46 and the pH of a 1:100 dilution was 3.69. To accelerate storage characteristics, the sample was placed in an incubator set for 2 ▛ 30-31 °C (86-87 °F). ▟ 2 ~~86-87° F.~~ Table II shows the percent of hydrogen peroxide remaining and the percent of PAA generated over a period of more than six months.

[0058] Table II

Days of Study	% Hydrogen Peroxide Remaining	% PAA Generated
1	40.21	0.004
33	38.02	3.499
54	35.5	5.992
96	29.27	8.36
127	25.05	6.66
173	18.49	4.43
201	15.67	3.51

[0059] Table II indicates that after 96 days, the generation of PAA maximized at 8.36% and corresponded to a loss of 10.94% hydrogen peroxide. Had all of the depleted hydrogen

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peroxide gone toward the generation of PAA, 24.45% PAA would have been generated. Because only 8.36% PAA was generated, unaccounted losses of hydrogen peroxide amounted to 7.2%.

[0060] Example 3

[0061] A formulation containing hydrogen peroxide and triacetin in a mole ratio of 5.8:1 was prepared by adding triacetin (37.5008 g) to 50% hydrogen peroxide (65.2504 g) so that the mixture was 31.75% hydrogen peroxide and 36.5% triacetin, with the balance being water. The pH of the solution was 1.57 and the pH of a 1:100 dilution was 5.17. To accelerate degradation, the sample was placed in an incubator set for 2 30-31 °C (86-87 °F). 2 ~~86-87 °F.~~ Table III shows the percent of hydrogen peroxide remaining and the percent of PAA generated over a period of six days.

[0062] Table III

Days of Study	% Hydrogen Peroxide Remaining	% PAA Generated
1	31.72	0.33
6	31.63	0.716

[0063] In this study, after six days, the mixture had lost only 0.09% hydrogen peroxide, yet had generated an additional 0.386% PAA. Had all the loss of hydrogen peroxide been due to the formation of PAA, only an additional 0.201% would have been generated. It is clear that the analytical methods employed for solutions that are high in hydrogen peroxide but low in PAA are subject to detection limit errors.

[0064] Example 4

[0065] A formulation containing hydrogen peroxide and triacetin in a mole ratio of 3.2:1 was prepared by adding triacetin (35.0 g) to 50% hydrogen peroxide (35.0 g) so that the mixture was 25% hydrogen peroxide and 50% triacetin, with the balance being water. The pH of the solution was 1.66 and the pH of a 1:100 dilution was 4.09. To accelerate degradation, the sample

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was placed in an incubator set for 2 ▛ 30-31 °C (86-87 °F). ▟ 2 ~~86-87° F.~~ Table IV shows the percent of hydrogen peroxide remaining and the percent of PAA generated over a period of 22 days.

[0066] Table IV

Days of Study	% Hydrogen Peroxide Remaining	% PAA Generated
1	25	0.62
8	24.51	1
15	24.17	1.17
22	23.68	1.14

[0067] Table IV indicates that after 15 days, the generation of PAA maximized at 1.17% and corresponded to a loss of 0.83% hydrogen peroxide. Had all of the depleted hydrogen peroxide gone toward the generation of PAA, 1.86% PAA would have been generated. Because only 1.17% PAA was generated, unaccounted losses of hydrogen peroxide amounted to 0.31%.

[0068] Example 5

[0069] A formulation containing hydrogen peroxide and triacetin in a mole ratio of 4.8:1 was prepared by adding triacetin (37.50 g) to 50% hydrogen peroxide (56.25 g) so that the mixture was 30% hydrogen peroxide and 40% triacetin, with the balance being water. The pH of the solution was 1.67 and the pH of a 1:100 dilution was 4.62. To accelerate degradation, the sample was placed in an incubator set for 86-87° 2 ▛ F (30°-31° C). ▟ 2 ~~F.~~ Table V shows the percent of hydrogen peroxide remaining and the percent of PAA generated over a period of 43 days.

[0070] Table V

Days of Study	% Hydrogen Peroxide Remaining	% PAA Generated
1	30.08	0.38
8	29.85	0.784
15	29.63	1.22
22	29.37	1.71
29	28.76	2.26
36	28.67	2.78
43	28.31	3.41

[0071] Table V shows that although there was a steady depletion in hydrogen peroxide, it was accompanied by the associative increase of PAA. At the end of the 43-day study, the composition had lost 1.77% hydrogen peroxide. Had all of the depleted hydrogen peroxide gone toward the generation of PAA, 3.96% PAA would have been generated. Because only 3.41% PAA was generated, unaccounted losses of hydrogen peroxide amounted to 0.24%.

[0072] Example 6

[0073] A formulation containing hydrogen peroxide and triacetin in a mole ratio of 6.42:1 was prepared by adding triacetin (25.0007 g) to 50% hydrogen peroxide (50.0007 g) so that the mixture was 33.33% hydrogen peroxide and 33.33% triacetin, with the balance being water. The pH of the solution was 1.56 and the pH of a 1:100 dilution was 5.15. To accelerate degradation, the sample was placed in an incubator set for 2 30-31 °C (86-87 °F). 2 ~~86-87° F.~~ Table VI shows the percent of hydrogen peroxide remaining and the percent of PAA generated over a period of 36 days.

[0074] Table VI

Days of Study	% Hydrogen Peroxide Remaining	% PAA Generated
1	33.13	0.81
8	32.88	1.35
15	32.61	1.88
22	31.69	2.58
29	31.75	3.03
36	31.29	3.28

[0075] Table VI shows that although there was a steady depletion in hydrogen peroxide, it was accompanied by an associative increase of PAA. At the end of the 36-day study, the composition had lost 1.84% hydrogen peroxide. Had all of the depleted hydrogen peroxide gone toward the generation of PAA, 4.11% PAA would have been generated. Because only 3.28% PAA was generated, unaccounted losses of hydrogen peroxide amounted to 0.37%.

[0076] Example 7

[0077] A 113.6 L (30-gallon) batch of 50% hydrogen peroxide and triacetin was prepared by blending 62 kg (137 lbs) triacetin with 74 kg (163 lbs) 50% hydrogen peroxide so that the mixture was 27.15% hydrogen peroxide and 45.67% triacetin, with the balance being water. Thus, the mole ratio of hydrogen peroxide:triacetin was 3.8:1. The mixture was stored in an opaque drum in a non-climate controlled environment. On storage, the mixture slowly formed PAA in the container. Since this reaction of hydrogen peroxide to PAA does not represent a destructive loss of total peroxygen, to determine the true stability of the hydrogen peroxide, both the hydrogen peroxide and the peracetic acid generated must be quantified and reported as total peroxygen recovered.

[0078] After 295 days of storage, the mixture was analyzed for total peroxygen recovered (expressed as hydrogen peroxide) to determine the percent loss of peroxygen. Using the eerie sulfate-iodometric titration method, the PAA generated and the hydrogen peroxide remaining were measured. The total peroxygen recovered after 295 days and the theoretical initial concentrations are shown in Table VII. All peroxygen is expressed as % hydrogen peroxide.

[0079] Table VII

	Initial Theoretical %	% (after 295 days)
Hydrogen Peroxide Recovered	27.17	15.20
PAA formed	0	15.57
Hydrogen Peroxide Reacted to form PAA	0	6.97
Total Recovered Hydrogen Peroxide	27.17	22.17
Total Hydrogen Peroxide Unproductive Loss		5.00

[0080] After 295 days, 22.17% of the total peroxygen was recovered as either titratable hydrogen peroxide or hydrogen peroxide reacted to form PAA. To determine the stability of the peroxygen in the mixture, the total recovered hydrogen peroxide after 295 days (22.17%) was

subtracted from the total recovered hydrogen peroxide of the initial theoretical value (27.17%). The result, 5%, is the loss of peroxygen (expressed as hydrogen peroxide) after 295 days of storage in a non-climate controlled environment.

4. Methods of Generating Non-Equilibrium PAA On Site at the Point-of-Use

[0081] Methods of continuously or intermittently generating non-equilibrium solutions of PAA on a site having a point-of-use of PAA for use as a disinfectant or sanitizer, using a source of water that is an aqueous stream or by a batch process using water in a container, are provided.

[0082] In one embodiment, a method of continuously or intermittently generating non-equilibrium solutions of PAA on a site having a point-of-use, using a source of water that is an aqueous stream, comprises the following steps.

[0083] (a) Providing water.

[0084] Flowing water is provided on a site having a point-of-use of peracetic acid.

[0085] The water may be flowing in, for example, a pipe, a flume, a canal, or other types of aqueous streams. The water pressure should be regulated and the flow rate should be monitored or measured. Any suitable flow meter may be used, such as a rotameter, a magnetic flow meter, an ultrasonic flow meter, a Doppler flow meter, a differential-pressure flow meter, a turbine flow meter, or a Coriolis flow meter.

[0086] The water should be softened, deionized, or of sufficient low hardness that it will not precipitate calcium salts when the alkali metal or earth alkali metal hydroxide is introduced in step (d).

[0087] (b) Introducing a hydrogen peroxide-acetyl precursor solution to the water.

[0088] A hydrogen peroxide-acetyl precursor solution is provided on the site having the point-of-use of the PAA. The hydrogen peroxide-acetyl precursor solution may be prepared by the steps set forth in section 3 above. The hydrogen peroxide-acetyl precursor solution may be

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prepared off site and then transported to the site having the point-of-use, or the acetyl precursor and the aqueous hydrogen peroxide may be transported to the site having the point-of-use and used to prepare the hydrogen peroxide-acetyl precursor solution on the site.

[0089] A preferable liquid acetyl precursor is triacetin. When triacetin is used, a hydrogen peroxide-triacetin solution is introduced.

[0090] The hydrogen peroxide-acetyl precursor solution is introduced to the flowing water in an amount such that the hydrogen peroxide-acetyl precursor solution is about 5.6% to about 22.5% of the total.

[0091] The hydrogen peroxide-acetyl precursor solution is pumped from its container into the aqueous water stream, for example, through an injection quill mounted on the pipe. Any suitable pump capable of overcoming the pressure of the water flowing in the pipe may be used. Examples include a solenoid-driven or air-driven diaphragm pump or a peristaltic pump. The rate at which the hydrogen peroxide-acetyl precursor solution is pumped into the flowing water is governed by a controller that is interfaced to a flow meter that is measuring the flow of the water. In this way, the rate at which the hydrogen peroxide-acetyl precursor solution is pumped may be matched to the flow of the water in the aqueous stream. If the flow of the water slows, the rate at which the hydrogen peroxide-acetyl precursor solution is pumped should slow accordingly. If the flow of the water stops, the hydrogen peroxide-acetyl precursor pump should stop.

[0092] As an alternative to step (b), the liquid acetyl precursor and the solution of aqueous hydrogen peroxide may be introduced to the water separately, either simultaneously or sequentially. If they are introduced sequentially, either one may be added first. If the liquid acetyl precursor and the aqueous hydrogen peroxide are introduced separately, rather than as an hydrogen peroxide-acetyl precursor solution, then in step (c) the liquid acetyl precursor and the aqueous hydrogen peroxide are mixed with the water to form a mixture.

[0093] (c) Mixing the hydrogen peroxide-acetyl precursor solution and the water to form a mixture.

[0094] Any mixing device suitable for mixing liquids may be used. An example is a static mixer located just after the point that the hydrogen peroxide-acetyl precursor solution is introduced to the flowing water. One type of static mixer utilizes a non-moving element such as a series of baffles. As the mixture flows through the static mixer under the motive force of the flowing water, the non-moving element divides the flow several times to provide radial mixing. Another type of static mixer utilizes a series of obstructions, such as column packing or glass beads, provided there is a low differential pressure drop across the mixer. The obstructions provide for turbulent mixing of the hydrogen peroxide-acetyl precursor solution and the flowing water. The mixing should yield a homogeneous solution with no concentration gradients before the next step is performed. The velocity of the water will determine the time it takes to complete the mixing and the efficiency of the mixing. For example, with ¾" pipe, a static mixer of ¾" diameter and 6" long, and a velocity of about 1 gal/min., mixing should be accomplished in less than about one second.

[0095] (d) Adding an aqueous source of an alkali metal or earth alkali metal hydroxide to the mixture.

[0096] An aqueous source of an alkali metal or earth alkali metal hydroxide is provided on the site having the point-of-use. A preferable aqueous source of an alkali metal hydroxide is sodium hydroxide, and 50% sodium hydroxide is most preferable. Other suitable alkali metal hydroxides include 45% potassium hydroxide.

[0097] Sufficient 50% NaOH is added to the flowing mixture such that the amount of sodium hydroxide is ~~about~~ 1.82% to ~~about~~ 7.28% of the total amount. When the acetyl precursor is triacetin, a preferred mole ratio of NaOH: hydrogen peroxide:triacetin is ~~about~~ 4.2:3.8:1.

[0098] The 50% NaOH solution is added to the mixture by pumping it from its container with a suitable pumping device and into the mixture through an injection quill mounted on the pipe. Any suitable pump capable of overcoming the hydraulic pressure of the pipe may be used. Examples include a solenoid-driven or air-driven diaphragm pump or a peristaltic pump.

[0099] The rate at which the 50% NaOH solution is pumped into the pipe is governed by the same controller that is interfaced to both the flow meter that is measuring the flow of the mixture and the pump controlling the rate of introduction of the hydrogen peroxide-acetyl precursor solution. In this way, the rate at which the 50% NaOH solution is introduced may be matched to both the flow of the mixture and the rate of introducing the hydrogen peroxide-acetyl precursor solution. If the flow of the mixture slows for any reason, the rate at which the 50% NaOH solution is pumped should slow accordingly. If the flow of the mixture stops, the 50% NaOH pump should stop as well.

[00100] Step (d) may be performed after step (c), or it may be performed simultaneously with step (b).

[00101] (e) Forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

[00102] The reaction medium that is formed in this step almost instantaneously forms a non-equilibrium solution of PAA. The hydrogen peroxide reacts with the acetyl precursor to form peracetic acid. Depending upon the temperature of the water, the efficiency of mixing, and the mole ratio of NaOH:hydrogen peroxide:acetyl precursor employed, the amount of hydrogen peroxide and acetyl precursor that are converted into PAA is maximized within about 30 seconds to about five minutes.

[00103] The non-equilibrium solutions of PAA prepared by this method comprise PAA, unreacted hydrogen peroxide, unreacted acetyl precursor, the product of the perhydrolysis

reaction of the acetyl precursor, the aqueous source of alkali metal or earth alkali metal hydroxide, and water. When the acetyl precursors is triacetin, the product of the perhydrolysis reaction is 1, 2, 3-propanetriol (glycerine).

[00104] This method may also include an optional step after step (e) of sampling the reaction medium at various times after the addition of the aqueous source of alkali metal or earth alkali metal hydroxide to determine the time required under the existing conditions to maximize the amount of hydrogen peroxide and acetyl precursor that are converted into PAA. This may be accomplished by directing the flow of the reaction medium through a series of residence chambers equipped with sampling ports. The residence chambers are designed to be of sufficient volume such that a certain amount of time has elapsed since the introduction of the 50% NaOH. Thus, at any given flow rate in the pipe, the first residence chamber is of a volume such that 30 seconds have elapsed since the introduction of 50% NaOH, one minute has elapsed by the time the flow reaches the sampling port immediately after the second residence chamber, and additional time elapses by the time the flow reaches the remaining sampling ports. Up to four residence chambers and sampling ports may be placed in series such as this to provide the user with about 30 seconds to five minutes of residence time after the introduction of the 50% NaOH. Once the flow of the reaction medium has been established, samples are drawn from all four sampling ports associated with the residence chambers and analyzed for peracetic acid. The sample which registers the highest amount of peracetic acid is deemed to be drawn from the sampling port associated with the residence chamber corresponding to the time of maximum conversion of the acetyl precursor into peracetic acid. At this point, the entire flow of reaction medium is directed through the sampling point of maximum conversion of acetyl precursor and to the point-of-use.

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[00105] In another embodiment, a method of continuously or intermittently generating a non-equilibrium solution of PAA on a site having a the point-of-use in a batch process comprises the following steps:

[00106] (a) Providing water.

[00107] A container of water, such as a mixing tank or other vessel, is provided on a site having a point-of-use of peracetic acid. The water should be softened, deionized, or of sufficient low hardness that it will not precipitate calcium salts when the alkali metal or earth alkali metal hydroxide is introduced in step (d).

[00108] (b) Introducing a hydrogen peroxide-acetyl precursor solution to the water.

[00109] A hydrogen peroxide-acetyl precursor solution is provided on the site having the point-of-use of the PAA. The hydrogen peroxide-acetyl precursor solution may be prepared by the steps set forth in section 3 above. The hydrogen peroxide-acetyl precursor solution may be prepared off site and then transported to the site having the point-of-use, or the acetyl precursor and the aqueous hydrogen peroxide may be transported to the site having the point-of-use and used to prepare the hydrogen peroxide-acetyl precursor solution on the site.

[00110] A preferable liquid acetyl precursor is triacetin. When triacetin is used, a hydrogen peroxide-triacetin solution is introduced.

[00111] The hydrogen peroxide-acetyl precursor solution is introduced to the water in an amount such that the hydrogen peroxide-acetyl precursor solution is [redacted] 5.6% to [redacted] 22.5% of the total.

[00112] The hydrogen peroxide-acetyl precursor solution is pumped from its container into the container of water through an injection quill mounted on the side of the container of water. Any suitable pump may be used. Examples include a solenoid-driven or air-driven diaphragm pump or a peristaltic pump.

[00113] As an alternative to step (b), the liquid acetyl precursor and the solution of aqueous hydrogen peroxide may be introduced to the water separately, either simultaneously or sequentially. If they are introduced sequentially, either one may be added first. If the liquid acetyl precursor and the aqueous hydrogen peroxide are introduced separately, rather than as an hydrogen peroxide-acetyl precursor solution, then in step (c) the liquid acetyl precursor and the aqueous hydrogen peroxide are mixed with the water to form a mixture.

[00114] (c) Mixing the hydrogen peroxide-acetyl precursor solution and the water to form a mixture.

[00115] Any mixing device suitable for mixing liquids may be used. An example is an overhead stirrer, such as an agitator blade. The container of water is equipped with a baffle to assist the process of mixing. The mixing should yield a homogeneous solution with no concentration gradients before the next step is performed.

[00116] (d) Adding an aqueous source of an alkali metal or earth alkali metal hydroxide to the mixture.

[00117] An aqueous source of an alkali metal or earth alkali metal hydroxide is provided on the site having the point-of-use. A preferable aqueous source of an alkali metal hydroxide is sodium hydroxide, and 50% sodium hydroxide is most preferable. Other suitable alkali metal hydroxides include 45% potassium hydroxide.

[00118] Sufficient 50% NaOH is added to the mixture such that the amount of sodium hydroxide is ~~about~~ 1.82% to ~~about~~ 7.28% of the total amount. When the acetyl precursor is triacetin, a preferred mole ratio of NaOH: hydrogen peroxide:triacetin is ~~about~~ 4.2:3.8:1.

[00119] The 50% NaOH solution is added to the mixture by pumping it from its container with a suitable pumping device and into the mixture through an injection quill mounted on the

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side of the container of water. Any suitable pump may be used. Examples include a solenoid-driven or air-driven diaphragm pump or a peristaltic pump.

[00120] Step (d) may be performed after step (c), or it may be performed simultaneously with step (b).

[00121] (e) Forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

[00122] The reaction medium that is formed in this step almost instantaneously forms a non-equilibrium solution of PAA. The hydrogen peroxide reacts with the acetyl precursor to form peracetic acid. Depending upon the temperature of the water, the efficiency of mixing, and the mole ratio of NaOH:hydrogen peroxide:acetyl precursor employed, the amount of hydrogen peroxide and acetyl precursor that are converted into PAA is maximized within about 30 seconds to about five minutes.

[00123] The non-equilibrium solutions of PAA prepared by this method comprise PAA, unreacted hydrogen peroxide, unreacted acetyl precursor, the product of the perhydrolysis reaction of the acetyl precursor, the aqueous source of alkali metal or earth alkali metal hydroxide, and water. When the acetyl precursors is triacetin, the product of the perhydrolysis reaction is 1, 2, 3-propanetriol (glycerine).

[00124] While not wishing to be bound by theory, it is believed that under elevated pH conditions created by the addition of a source of alkali metal hydroxide or earth alkali metal hydroxide, hydrogen peroxide dissociates according to the following equation:

$$H_2O_2 = H^+ + HO_2^-$$

[00125] The perhydroxyl anion (HO_2^-) then affects nucleophilic substitution reactions on the carbonyl groups of the acetyl precursor in a perhydrolysis reaction to form peracetic acid and the product of the perhydroloysis reaction of the acetyl precursor. When the acetyl precursor is

triacetin and all three acetyl groups are reacted, then the product of the perhydrolysis reaction is 1, 2, 3-propanetriol (glycerine).

[00126] In these methods, the perhydrolysis reaction is rapid and the maximum amount of PAA is generated within about 30 seconds to about five minutes at ambient temperature. The methods efficiently utilize the acetyl precursor and the source of hydrogen peroxide. When triacetin is used, assuming all three acetyl groups are reacted, about 40.9% to about 85.7% of the triacetin is converted into PAA. The percent of hydrogen peroxide remaining is about 0.078% to about 1.88%.

[00127] The non-equilibrium solutions of PAA prepared by these methods have several unique characteristics. The PAA solutions have high levels of PAA, from about 1% to about 7.1%. In addition, the PAA solutions are alkaline, having a pH of about 11.2 to about 13.37.

[00128] The non-equilibrium solutions of PAA may be immediately introduced to the receiving water at the point-of-use. Alternatively, the solutions may be stabilized by halting the decomposition of the PAA by adding a source of acid to lower the elevated pH of the reaction medium caused by the addition of an alkali metal or earth alkali metal hydroxide, and to provide neutral to mildly acid pH conditions to stabilize the PAA to elevated pH degradation. The acid-stabilized PAA solutions can then be stored and used at the point-of-use as required throughout a working day.

[00129] The non-equilibrium PAA solutions produced by these methods can be advantageously used wherever traditional equilibrium solutions of PAA are used. Thus, the PAA solutions may be used as a disinfectant and sanitizer in the dairy, food, and beverage processing industries for clean-in-place pipeline and equipment cleaning; for fruit and vegetable washing; and in the treatment of meat, poultry, and seafood products. These PAA solutions may also be

used in the treatment of cooling water, oil and gas process water, and municipal wastewater. Other uses include slime and biofilm removal in papermaking processes.

[00130] For both of the above methods, steps (a) through (e) may be performed on a continuous or intermittent basis. In a continuous basis, the steps are performed under conditions of steady state, and the PAA solution is produced at the same rate as a function of time. However, intermittent performing of steps (a) through (e) is possible if the demand for the PAA solution drops for any reason (e.g., change in working shift or the target level of PAA in the receiving water has been achieved), in which case, steps (a) through (e) may be discontinued and resumed when the demand increases.

[00131] Example 8

[00132] Tests were performed using about one liter of reverse osmosis (RO) or softened water which was buffered to an alkaline pH. The buffering media included 0.1 and 0.2 M Na_2CO_3 solutions, in addition to 0.1 and 0.2 M NaOH solutions. Sufficient buffering media was added to the water in order to achieve the desired pH. The amount of hydrogen peroxide (introduced as 50% hydrogen peroxide) and triacetin used in the tests is summarized in Table VIII. The reactants were introduced to the solution in one of three ways: simultaneously (referred to in Table VIII as "double"), sequentially (triacetin followed shortly by hydrogen peroxide), or by mixing the hydrogen peroxide and triacetin together and adding them in a single charge (referred to in Table VIII as "mixed"). All testing was performed at ambient temperature as the one-liter beaker was stirred with a magnetic stir bar. Using the ceric sulfate–iodometric titration method, the percent of PAA generated and the percent of hydrogen peroxide remaining were measured five minutes after introduction of the reactants to the buffer. The percent of the triacetin acetyl donor converted to PAA was calculated, assuming all three acetyl groups were available for perhydrolysis.

[00133] Table VIII

ıt #	Moles Hydrogen Peroxide/ Mole Triacetin	Type of Addition (Double, Sequential or Mixed)	pH of Buffer	Hydrogen Peroxide Used (Wt., g)	Triacetin Used (Wt., g)	% PAA Generated	% Hydrogen Peroxide Remainin g	% Triacetin Converted to PAA
	4.99	Double	10.50	8.60	5.53	0.170	0.329	29.6%
	5.47	Double	10.50	8.60	5.04	0.178	0.333	33.9%
	5.45	Double	10.80	8.61	5.07	0.192	0.329	36.4%
	5.68	Double	11.00	8.87	5.01	0.201	0.322	38.6%
	5.72	Double	11.00	9.13	5.12	0.068	0.416	12.8%
	5.49	Double	12.42	8.60	5.02	0.169	0.306	32.4%
	5.59	Double	12.91	8.60	4.93	0.320	0.265	62.4%
	5.53	Double	12.80	8.69	5.04	0.335	0.273	63.9%
	25.0	Double	12.65	9.03	1.16	0.104	0.413	85.9%
0	5.26	Double	12.60	8.70	5.30	0.298	0.247	54.1%
1	5.52	Sequential	12.83	8.68	5.04	0.276	0.248	52.4%
2	5.47	Sequential	12.81	17.11	10.03	0.120	0.371	11.4%
3	28.8	Mixed	11.12	8.74	0.973	0.039	0.398	38.9%
4	28.8	Mixed	11.99	8.75	0.974	0.038	0.374	37.9%
5	28.8	Mixed	13.02	8.69	0.967	0.055	0.367	55.2%
6	28.8	Mixed	7.91	8.70	0.968	0.024	0.435	23.7%
7	28.8	Mixed	11.22	8.76	0.975	0.029	0.309	28.4%
8	28.8	Mixed	12.07	8.68	0.966	0.028	0.311	27.7%
9	28.8	Mixed	13.22	8.72	0.971	0.026	0.321	25.6%
lup	25.2	Mixed	12.97	8.77	1.12	0.031	0.249	26.5%
0	25.2	Mixed	12.98	9.08	1.16	0.066	0.393	54.5%
lup	25.2	Mixed	13.00	9.10	1.16	0.074	0.409	61.0%
1	25.2	Mixed	13.02	9.10	1.16	0.02	0.261	16.5%
lup	25.2	Mixed	12.99	9.12	1.16	0.017	0.376	14.0%
2	16.0	Mixed	12.99	5.80	1.16	0.070	0.246	57.7%
3	16.0	Mixed	13.05	5.79	1.16	0.013	0.189	10.7%
4	16.0	Mixed	12.55	5.78	1.16	0.031	0.234	25.6%
5	16.0	Mixed	12.49	5.80	1.16	0.013	0.144	10.7%
6	16.0	Mixed	12.01	5.85	1.17	0.016	0.259	13.1%
7	16.0	Mixed	12.05	5.82	1.16	0.011	0.080	9.0%
8	16.0	Mixed	10.50	0.601	0.120	0.00077	0.028	6.1%
9	16.0	Mixed	10.97	0.595	0.119	0.00023	0.021	1.8%
0	16.0	Mixed	10.50	0.614	0.123	0.00012	0.019	0.9%
2	16.0	Mixed	10.97	0.595	0.119	0.00035	0.018	2.8%

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[00134] Table VIII shows that the amount of PAA generated was independent of the method of introducing the triacetin and hydrogen peroxide to the buffer solutions. It can be seen that the pH of the buffer had a significant effect on the percent of the triacetin that was converted into PAA. Only when the pH was above 12 was a meaningful percent of the triacetin converted into PAA.

[00135] Example 9

[00136] A mixture of triacetin and hydrogen peroxide was prepared by blending 50% hydrogen peroxide (63.4%) and triacetin (36.6%). Thus, the mole ratio of hydrogen peroxide:triacetin was 5.57:1. The mixture (13.68 g) was added to soft water such that the final solution contained 5 g/L of triacetin and had a pH of 4.1. Then, in successive experiments, sufficient 50% NaOH was added to the beaker so that the mole ratio of NaOH:TA was varied from 3:1 (obtained by introducing 5.49 g of 50% NaOH) to 8:1 (obtained by introducing 14.68 g of 50% NaOH). Using the ceric sulfate–iodometric titration method, the percent of PAA generated and the percent of hydrogen peroxide remaining were measured over the next 20 minutes. The method was performed in duplicate for each sample point. The averages of the results for the 6:1 mole ratio of NaOH:TA are shown in Table IX.

[00137] Table IX

Time (min)	% PAA Generated	% Hydrogen Peroxide Remaining	% Triacetin Converted to PAA (3 acetyls)
0	0.015	0.437	2.9
1	0.275	0.298	53.1
3	0.312	0.274	60.2
5	0.326	0.258	62.9
11	0.302	0.233	58.3
21	0.246	0.199	47.5

[00138] It can be seen that even before the introduction of 50% NaOH, some PAA was present in solution as a result of being generated in the diluted mixture of the two components.

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Upon addition of the NaOH, PAA was immediately generated, with the maximum amount of 0.326% occurring at five minutes, corresponding to a yield of 62.9% assuming all three acetyl groups of triacetin were available for perhydrolysis.

[00139] This exercise was repeated for mole ratios of NaOH:triacetin ranging from 3:1 to 8:1. Table X summarizes the next set of data and shows the maximum percent of PAA generated, the time after the addition of NaOH that it took to reach the maximum percent of PAA generated, the pH after the addition of the NaOH, the percent of hydrogen peroxide remaining at the time of the maximum percent of PAA, and the calculated percent of triacetin converted to PAA assuming that all three acetyl groups were available for perhydrolysis.

[00140] Table X

Mole ratio NaOH:TA	Time at Max. PAA (min)	Max % PAA Generated	% Hydrogen Peroxide Remaining	% Triacetin Converted to PAA (3 acetyls)	pH
3:1	3	0.229	0.291	43.8	11.2
4:1	6	0.285	0.251	54.8	11.3
6:1	5	0.326	0.258	62.9	11.6
8:1	3	0.335	0.271	64.7	12.2

[00141] It can be seen that the amount of PAA generated, and hence the conversion of triacetin into PAA, increased with the increasing mole ratio of NaOH:TA. For all four mole ratios employed, the maximum amount of PAA was generated between three and six minutes. The optimum mole ratio of NaOH:TA was 6:1 as it resulted in an efficient utilization of triacetin converted into PAA, and because the pH of the resulting reaction medium would have less impact on the pH of the receiving water than the higher pH of the reaction medium resulting from the 8:1 mole ratio.

[00142] Example 10

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[00143] A series of experiments was then performed in which the mole ratio of NaOH:TA was fixed at 8:1 and the mole ratio of hydrogen peroxide:triacetin was varied from 2:1 to 8:1. Triacetin (5 g) was added to about 965 mL of softened water along with 50.7% hydrogen peroxide in successive tests (3.06 g for the 2:1 mole ratio; 6.19 g for the 4:1 mole ratio; 9.23 g for the 6:1 mole ratio; and 12.32 g for the 8:1 mole ratio) to yield a solution with a pH of about 4.8-5.5. The volume of the mixture was adjusted to 990 mL with softened water. With mixing, 50% NaOH (14.67 g) was introduced. Using the ceric sulfate–iodometric titration method, the percent of PAA generated and the percent of hydrogen peroxide remaining were measured over the next 20 minutes. The method was performed in duplicate for each sample point. The averages of the results for the kinetic run employing the mole ratio of 8:6:1 NaOH:hydrogen peroxide:TA are shown in Table XI.

[00144] Table XI

Time (min)	% PAA Generated	% Hydrogen Peroxide Remaining	% Triacetin Converted to PAA (3 acetyls)
1	0.331	0.317	63.8
3	0.330	0.303	63.6
5	0.319	0.303	61.5
10	0.300	0.295	57.8
20	0.256	0.274	49.3

[00145] Upon addition of the NaOH, PAA was immediately generated, with the maximum amount of 0.331% occurring after just one minute, corresponding to a calculated conversion of triacetin into PAA of 63.8%, assuming that all three acetyl groups of triacetin were available for perhydrolysis.

[00146] This exercise was repeated for mole ratios of hydrogen peroxide:triacetin ranging from 2:1 to 6:1. Table XII summarizes the data and shows the maximum percent of PAA generated, the time after the addition of NaOH that it took to reach the maximum percent of PAA

generated, the pH after the addition of the NaOH, the percent of hydrogen peroxide remaining at the time of the maximum percent of PAA, and the calculated percent of triacetin converted to PAA assuming that all three acetyl groups were available for perhydrolysis.

[00147] Table XII

Mole Ratio Hydrogen Peroxide:Triacetin	Time at Max. PAA (min)	Max % PAA Generated	% Hydrogen Peroxide Remaining	% Triacetin Converted to PAA (3 acetyls)	pH
2:1	3	0.214	0.079	40.9	12.6
4:1	1	0.272	0.195	51.9	12.4
6:1	1	0.331	0.317	63.8	12.3
8:1	3	0.374	0.445	72.0	11.9

[00148] It can be seen that the amount of PAA generated, and hence the conversion of triacetin into PAA, increased with the increasing mole ratio of hydrogen peroxide:triacetin. For all four mole ratios employed, the maximum amount of PAA was generated between one and three minutes. The optimum mole ratio of hydrogen peroxide:triacetin was 8:1 as it resulted in the most efficient utilization of triacetin converted into PAA.

[00149] Example 11

[00150] A further series of experiments was performed in which the mole ratio of hydrogen peroxide to triacetin was fixed at 4:1 and the molar ratio of NaOH to triacetin was varied from 3:1 to 8:1. Triacetin (5 g) was added to around 975 mL of softened water along with 50.7% hydrogen peroxide (6.18 g) to yield a solution with a pH of 4.8 to 5.3. With mixing, 50% NaOH was introduced in successive tests (5.51 g for the 3:1 mole ratio; 7.30 g for the 4:1 mole ratio; 11.03 g for the 6:1 mole ratio; and 14.67 g for the 8:1 mole ratio). The volume of the mixture was adjusted to 1000 mL with softened water. Using the ceric sulfate–iodometric titration method, the percent of PAA generated and the percent of hydrogen peroxide remaining were measured over the next 20 minutes. The method was performed in duplicate for each

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sample point. The averages of the results for the kinetic run employing the mole ratio of NaOH:hydrogen peroxide:triacetin of 4:4:1 are shown in Table XIII.

[00151] Table XIII

Time (min)	% PAA Generated	% Hydrogen Peroxide Remaining	% Triacetin Converted to PAA (3 acetyls)
1	0.247	0.207	47.3
3	0.280	0.178	53.6
5	0.292	0.167	55.9
10	0.267	0.142	51.1
20	0.229	0.111	43.8

[00152] Upon addition of the NaOH, PAA was immediately generated, with the maximum amount of 0.292% occurring after five minutes, corresponding, to a calculated conversion of 55.9% triacetin into PAA assuming that all three acetyl groups of triacetin were available for perhydrolysis.

[00153] This exercise was repeated for mole ratios of NaOH:TA ranging from 3:1 to 8:1. Table XIV summarizes the data and records the maximum percent of PAA generated, the time after the addition of NaOH that it took to reach the maximum percent of PAA generated, the pH after the addition of the NaOH, the percent of hydrogen peroxide remaining at the time of the maximum percent of PAA, and the calculated percent of triacetin converted to PAA assuming that all three acetyl groups were available for perhydrolysis.

[00154] Table XIV

Mole Ratio NaOH:TA	Time at Max. PAA (min)	Max % PAA Generated	% Hydrogen Peroxide Remaining	% Triacetin Converted to PAA (3 acetyls)	pH
3:1	3	0.246	0.202	47.2	11.3
4:1	5	0.292	0.167	55.9	11.6
6:1	3	0.290	0.181	55.6	12.2
8:1	3	0.272	0.195	51.9	12.4

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[00155] It can be seen that the amount of PAA generated maximized when the mole ratio of NaOH:hydrogen peroxide:triacetin was 4:4:1. The optimum mole ratio of NaOH:hydrogen peroxide:triacetin was 4:4:1 as it resulted in an efficient utilization of triacetin converted into PAA, and because the pH of the resulting reaction medium would have less impact on the pH of the receiving water than the higher pH of the reaction medium resulting from the 4:6:1 mole ratio of NaOH:hydrogen peroxide:triacetin. For all four mole ratios employed, the maximum amount of PAA was generated between three and five minutes.

[00156] Example 12

[00157] In the first test, a mixture of triacetin and hydrogen peroxide was prepared by blending 50% hydrogen peroxide (54.24%) and triacetin (45.76%). Thus, the mole ratio of hydrogen peroxide:triacetin was 3.65:1. The mixture (51.22 g) was dissolved in soft water (911.9 g) in a one-liter beaker to yield a solution with a pH of 6.47. Then, 50% NaOH (36.47 g) was introduced with stirring. Thus, the mole ratio of the combined mixture of NaOH:hydrogen peroxide:triacetin was 4.2:3.8:1. The pH of the resulting solution initially measured 12.1. Using the ceric sulfate–iodometric titration method, the PAA generated and the hydrogen peroxide remaining were measured over the next 10 minutes. It was noticed that the sample temperature rose immediately upon addition of the 50% NaOH.

[00158] In the second test, the first test was repeated, except that the water was chilled soft water.

[00159] In the third test, the first test was repeated using three times the original amount of hydrogen peroxide:triacetin mixture and three times the original amount of 50% NaOH. The mole ratio of the combined mixture of NaOH:hydrogen peroxide:triacetin remained the same as before, at 4.2:3.8:1. The hydrogen peroxide and triacetin mixture (153.96 g) was introduced to chilled soft water (735.6 g) in a one-liter beaker to yield a solution with an initial pH of 5.14.

Then, 50% NaOH (111.21 g) was introduced. Using the ceric sulfate–iodometric titration method, the PAA generated and the hydrogen peroxide remaining were measured over the next 10 minutes.

[00160] In the fourth test, the first test was repeated using four times the original amount of hydrogen peroxide:triacetin mixture and four times the original amount of 50% NaOH. The mole ratio of the combined mixture of NaOH:hydrogen peroxide:triacetin remained the same as before, at 4.2:3.8:1. The hydrogen peroxide and triacetin mixture (204.7 g) was introduced to chilled soft water (647.5 g) in a one-liter beaker to yield a solution with an initial pH of 6.32. Then, 50% NaOH (155.4 g) was introduced. Using the ceric sulfate–iodometric titration method, the PAA generated and the hydrogen peroxide remaining were measured over the next 10 minutes.

[00161] Table XV summarizes the results obtained for the four tests and shows the initial and final solution temperatures, the pH of the solution after the addition of the 50% NaOH, the maximum percent of PAA generated, the time of maximum PAA generation, and the percent of hydrogen peroxide remaining.

[00162] Table XV

Test #	Initial Temp. (°F)	Final Temp. (°F)	Final pH	Max. % PAA Generated (time, min)	% Hydrogen Peroxide Remaining
1	NM	NM	12.14	1.71 (1)	0.58
2	38	50	12.67	2.1 (4)	0.43
3	44	88	12.77	6.3 (1)	1.18
4	34	92	13.17	7.2 (1)	1.88

NM = not measured

[00163] Table XVI shows the percent of hydrogen peroxide converted overall, the percent of hydrogen peroxide converted to PAA, and the percent of triacetin converted to PAA

(assuming that all three acetyl groups on triacetin are available for conversion to PAA) for each of the tests in Table XV.

[00164] Table XVI

Test #	% Hydrogen Peroxide Converted Overall	% Hydrogen Peroxide Converted to PAA (at maximum conversion time)	% Triacetin Converted to PAA (3 acetyls)
1	67.8	88.1	69.7
2	68.8	94.4	85.8
3	71.6	94.3	85.7
4	66.1	88.1	73.8

[00165] The data in Tables XV and XVI indicate that far higher amounts of PAA are possible if the 50% sodium hydroxide solution is the last component of the mixture to be introduced. Further, up to 7.1% PAA was generated, although this solution got hot despite chilling the source water. Generation of the 6.3% PAA solution was very efficient because it represented a high 85.7% utilization of triacetin converted into PAA. There was clearly a linear relationship between Test 2, which generated 2.1% PAA, and Test 3, which utilized three times more of the reactants and generated 6.3% PAA. The data in Table XV indicate that the time of maximum conversion of triacetin was between one and four minutes. Thereafter, the amount of PAA in solution is reduced because it undergoes high pH degradation slowly over the following 10 minutes.

[00166] Example 13

[00167] Fig. 1 is a schematic representation of an example of a system 10 that was used to perform an embodiment of one of the methods described above to prepare a non-equilibrium solution of PAA on a site having a point-of-use. Triacetin was used as the acetyl precursor and 50% sodium hydroxide was used as the aqueous source of an alkali metal hydroxide.

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[00168] A container 120 of a hydrogen peroxide-acetyl precursor solution, which was a hydrogen peroxide-triacetin solution, and a container 140 of 50% sodium hydroxide were each equipped with chemical delivery diaphragm pumps 115 and 135, respectively. Inlet water 90, which was softened, was provided from a water source on the site having the point-of-use. Inlet water 90 was directed through a pressure regulator 100 and flow meter 105, then into a section of pipe where the hydrogen peroxide-triacetin solution was introduced through injection quill 110. Mixing was accomplished using static mixer 125. Then, the sodium hydroxide solution was added through injection quill 130 to form a reaction medium.

[00169] The hydrogen peroxide-triacetin solution and the sodium hydroxide solution may be added in a sequential manner as described, where the hydrogen peroxide-triacetin solution was added first, or they may be added to the water simultaneously through a "T" fitting placed before static mixer 125. If a T fitting is used, the hydrogen peroxide- triacetin solution and the 50% sodium hydroxide solution are introduced to opposite ends of the T fitting and the mixture is injected into the pipe of water. In other embodiments, the hydrogen peroxide and triacetin may be added separately, or sequentially, with either one first, or simultaneously, with the sodium hydroxide added either simultaneously with, or after, the hydrogen peroxide and triacetin.

[00170] The reaction medium was introduced to a residence chamber 145 which provided reaction time and contained a packing material to promote mixing. Residence chamber 145 was designed to be of a volume such that at a total flow rate of 3.79 L (one gallon) per minute, by the time the reaction medium reached sampling port 160, 30 seconds had elapsed since the sodium hydroxide solution had been added. A pH probe 155 monitored the pH of the mixture.

[00171] A controller 150 was interfaced to the flow meter 105 of the inlet water 90, and also to the chemical delivery diaphragm pumps 115 and 135. The controller 150 monitored the

rate of the inlet water 90 and governed the rate at which the hydrogen peroxide-triacetin solution and the NaOH solution were introduced. If the flow of inlet water 90 decreased because of lower PAA requirements at the point-of-use 195, the rates at which the hydrogen peroxide-triacetin solution and the NaOH solution were pumped decreased accordingly. If the flow of inlet water 90 increased because of higher PAA requirements at the point-of-use 195, the rates at which the hydrogen peroxide-triacetin solution and the NaOH solution were pumped increased accordingly. If the flow of inlet water 90 stopped, pumps 115 and 135 stopped. Thus, the generation of PAA was both continuous and intermittent, and was tailored to the PAA requirements at the point-of-use.

[00172] The rate of the reaction between the hydrogen peroxide and the triacetin to form PAA is dependent upon the temperature of inlet water 90. If the temperature of inlet water 90 is high (for example, about ≥ 21 °C (70 °F)), the maximum conversion of triacetin into PAA may occur after 30 seconds, in which case the entire reaction medium was directed to the point-of-use 195. However, if the temperature of inlet water 90 is low (for example, about ≤ 0 °C (32 °F)), longer reaction times may be necessary to maximize the conversion of triacetin into PAA. Then, the reaction medium was directed through one or more residence chambers 165, 175, and 185 which also contained a packing material to promote turbulence and cause thorough mixing. Residence chambers 165, 175, and 185 were designed to be of volumes such that at a total flow rate of 3.79 L (one gallon) per minute, the reaction medium reached sampling port 170 in one minute, sampling port 180 in two minutes, and sampling port 190 in five minutes, to permit sampling at different time intervals.

[00173] Thus, depending on the temperature of the inlet water, the time to achieve the maximum conversion of hydrogen peroxide and triacetin into PAA was determined by sampling and analyzing the solution at sample ports 160, 170, 180, and 190. The entire flow was then

directed from the sampling port with the highest amount of PAA to the point-of-use 195. In practice, the PAA generated from the reaction of hydrogen peroxide with triacetin is typically diluted at the point-of-use 195.

[00174] Point-of-use 195 may be recirculating cooling water; municipal wastewater; poultry chill tank water; water used to sanitize meat, poultry, or seafood products; fruit and vegetable rinse water; or water used to clean and sanitize equipment used in the dairy, food, or beverage processing industries.

[00175] Example 14

[00176] Referring again to Fig. 1, a 208 L (55-gallon) drum container 120 of a hydrogen peroxide-triacetin solution was prepared by mixing triacetin 114 kg (251.5 lbs) with 50% hydrogen peroxide 135 kg (298.4 lbs). The resulting 249.5 (550 lbs) of hydrogen peroxide-triacetin solution had a specific gravity of 1.19 g/mL (10 lbs/gal) and contained 54.3% hydrogen peroxide and 45.7% triacetin. Thus, the mole ratio of hydrogen peroxide:triacetin was 3.8:1. The apparatus depicted in Fig. 1 was used to continuously and intermittently prepare an approximately 1% PAA solution.

[00177] Softened local city water was directed through pressure regulator 100 at a flow rate of 3.79 L (one gallon) per minute as measured by flow meter 105. The container 120 of hydrogen peroxide-triacetin solution was equipped with a draw down tube so that the flow rate of the mixture could be measured. Diaphragm pump 115 injected the hydrogen peroxide-triacetin solution through injection quill 110. The hydrogen peroxide-triacetin solution was then mixed with the softened water using static mixer 125. A container 140 of 50% NaOH was also equipped with a draw down tube to measure the flow pumped by diaphragm pump 135 into injection quill 130.

[00178] The percent of PAA generated was measured by quenching samples drawn through the sampling ports in mineral acid, followed by use of the ceric sulfate-iodometric

titration method. Table XVII summarizes the results of several trials where it was sought to maximize the conversion of triacetin into PAA and minimize the amount of 50% NaOH used.

[00179] Table XVII

Trial #	Sample Port	% PAA Generated	% Triacetin Converted to PAA	Hydrogen Peroxide-Triacetin Mix Flow Rate (mL/min)	NaOH Flow Rate (mL/min)	pH (PAA solution)
1	160	1.083	65.42	115.73	112.11	~13
2	170	1.073	64.82	115.73	112.11	13.19
3	160	0.955	57.32	116.79	122.12	13.37
4	160	1.058	63.39	116.79	114.93	13.11
5	160	1.061	63.3	116.79	98.1	12.9
6	160	1.102	68.96	110.82	85.01	12.91
7	170	1.055	55.59	132.64	99.77	12.95
8	160	0.978	59.91	115.5	74	12.62
9	180	0.936	56.7	114.25	78	12.64

[00180] It can be seen that the amount of PAA generated, and hence the conversion of triacetin into PAA, increased with the increasing flow rate of NaOH. For this mole ratio of hydrogen peroxide:triacetin of 3.8:1, the maximum amount of PAA was generated between 30 seconds (sample port 160) and two minutes (sample port 180) from the time the NaOH was added at injection quill 130. The flow rate of 74-78 mL/min of 50% NaOH was the optimum flow rate as it resulted in an efficient utilization of triacetin converted into PAA, consumed only a moderate amount of 50% NaOH, and had a lower pH that would have less impact on the pH of the receiving water.

[00181] Example 15

[00182] *E. coli* O157:H7 bacteria (ATCC 35150) was cultured in nutrient broth (Sigma, St. Louis, MO) by incubation for two days at 35°C. The bacteria were separated from the nutrient broth by centrifugation and carefully resuspended in two liters of sterile phosphate buffer, which was then split into two one-liter test solutions.

[00183] *Salmonella* typhimurium bacteria (ATCC 14028) was cultured in nutrient broth (Sigma, St. Louis, MO) by incubation for two days at 35°C. The bacteria were separated from the nutrient broth by centrifugation and carefully resuspended in two liters of sterile phosphate buffer, which was then split into two one-liter test solutions.

[00184] The amount of *E. coli* O157:H7 and *Salmonella typhimurium* bacteria were measured by serial dilution and plating on 3M *E. coli* plates and 3M *Enterobacteriaceae* Petrifilms, respectively.

[00185] A 1000 ppm stock of PAA was made by weighing 0.6386 g of Perasan MP-2 (5.83% hydrogen peroxide and 15.66% PAA) and adding water up to 100 g. Perasan MP-2 is an equilibrium product of PAA (Enviro Tech Chemical Services, Inc., Modesto, California). Side-by-side, one of the *E. coli* and one of the *Salmonella* test solutions were treated with a nominal dose of 1.0 ppm PAA by adding one mL of the 1000 ppm PAA stock solution to each sample. After one minute, approximately 100 mL of each test solution was removed, and sodium thiosulfate (0.5 g) was added to neutralize the PAA and hydrogen peroxide oxidants. After five minutes, sodium thiosulfate (0.5 g) was added to the remaining test solution to neutralize the PAA and hydrogen peroxide in the rest of the test solutions. The amount of viable *E. coli* O157:H7 and *Salmonella typhimurium* bacteria remaining in the test solutions at the one-minute and five-minute contact times were measured by serial dilution and plating on 3M *E. coli* Petrifilms and 3M *Enterobacteriaceae* Petrifilms, respectively.

[00186] A 1% solution of PAA was prepared by combining 30.90 g of a mixture of 50% hydrogen peroxide and triacetin (54.22% hydrogen peroxide and 45.78% triacetin) with 929.4 g of water, and then adding 50% NaOH (40.53 g). The PAA solution had a pH of 12.79. The mole ratio of this mixture was 3.8:7.8:1 NaOH:hydrogen peroxide:triacetin. One minute after preparation, the second one-liter *E. coli* solution and the second one-liter *Salmonella* solution

were treated with a nominal dose of 1.0 ppm PAA by adding 0.10 g of the 1% PAA solution to each sample. The test solution contact times, neutralization, and plating procedures were replicated for those that employed PAA from Perasan MP-2.

[00187] All 3M *E. coli* Pertrifilms and 3M *Enterobacteriaceae* Petrifilms were incubated at 35°C for 24 hours. After incubation, the plates were enumerated.

[00188] Table XVIII shows the microbiological results of the *E. coli* O157:H7 test solutions before treatment with PAA, one minute after the samples were dosed with a nominal 1.0 ppm PAA, and five minutes after being dosed with PAA. The *E. coli* test solution started with $\log_{10}$ 6.99 CFU/mL prior to being treated with 1.0 ppm of PAA from Perasan MP-2 or with 1 ppm of PAA from the 1% PAA solution.

[00189] The *E. coli* test solution treated with 1.0 ppm PAA from Perasan MP-2 contained a $\log_{10}$ 6.74 CFU/mL after one minute. This corresponds to 43.48% reduction. After five minutes, $\log_{10}$ 6.34 CFU/mL remained (77.50% reduction). The *E. coli* solution treated with a nominal 1.0 ppm PAA from the 1% PAA solution contained a $\log_{10}$ 6.78 CFU/mL after one minute and 6.63 CFU/mL after five minutes, respectively, corresponding to 38.02% and 56.12% reductions, respectively.

[00190] Table XVIII

***E. coli* O157:H7**

Description	$\log_{10}$ CFU/ml remaining	$\log_{10}$ reduction	% reduction
Control	6.99	N/A	N/A
Perasan MP-2 (1 min)	6.74	0.25	43.48
Perasan MP-2 (5 min)	6.34	0.65	77.50
1% PAA solution (1 min)	6.78	0.21	38.02
1% PAA solution (5 min)	6.63	0.36	56.12

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[00191] Table XIX shows the microbiological results of the *Salmonella typhimurium* test solutions before treatment with PAA, one minute after the samples were dosed with a nominal 1.0 ppm PAA, and five minutes after being dosed with PAA. The *Salmonella* test solution started with $\log_{10}$ 7.07 CFU/mL prior to being treated with PAA from Perasan MP-2 or with the 1% PAA solution.

[00192] The *Salmonella* solution treated with a nominal 1.0 ppm PAA from Perasan MP-2 contained a $\log_{10}$ 1.76 CFU/mL after one minute and 0.70 CFU/mL after five minutes, corresponding to >99.999% reduction at both contact times. The *Salmonella* test solution treated with 1.0 ppm PAA from the 1% PAA solution contained a $\log_{10}$ 6.69 CFU/mL after one minute. This corresponds to 58.31% reduction. After five minutes, $\log_{10}$ 4.95 CFU/mL remained (99.24% reduction).

[00193] Table XIX

Salmonella typhimurium

Description	$\log_{10}$ CFU/ml remaining	$\log_{10}$ reduction	% reduction
Control	7.07	N/A	N/A
Perasan MP-2 (1 min)	1.76	5.31	99.9995
Perasan MP-2 (5 min)	0.70	6.37	>99.9999
1% PAA solution (1 min)	6.69	0.38	58.31
1% PAA solution (5 min)	4.95	2.12	99.24

[00194] The data in tables XVIII and XIX demonstrate that the 1% PAA had antimicrobial efficacy. After five minutes, the efficacy of this PAA was 99.24%, about equal to that of the Perasan MP-2, which was > 99.9999%. The difference in efficacy at one minute was likely not of statistical significance, given that the comparison was only at the one-minute interval.

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Claims

1. A method of generating a non-equilibrium solution of peracetic acid, comprising:

a. providing water;

b. introducing a hydrogen peroxide-triacetin solution to the water;

c. mixing the hydrogen peroxide-triacetin solution and the water to form a mixture;

d. adding an aqueous source of an alkali metal hydroxide in the form of sodium hydroxide to the mixture, wherein the sodium hydroxide is 1.82% to 7.28% weight, further, wherein the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is 4.2:3.8:1; and

e. forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

2. The method of claim 1, further comprising, prior to step (b), a step of providing the hydrogen peroxide-triacetin solution comprising: introducing triacetin to a solution of aqueous hydrogen peroxide; and allowing the triacetin and the solution of aqueous hydrogen peroxide to mix to form the hydrogen peroxide-triacetin solution.

3. The method of claim 1, wherein the provided water is an aqueous stream.

4. The method of claim 1, wherein the provided water is contained in a mixing tank or other vessel.

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5. The method of claim 1, wherein step (d) is performed simultaneously with step (b).

6. The method of claim 1, further comprising allowing the reaction medium sufficient time to convert the hydrogen peroxide-triacetin into peracetic acid, said time being 30 seconds to five minutes.

7. The method of claim 1, further comprising, after step (e), a step of sampling the reaction medium to determine the time required to maximize the amount of hydrogen peroxide-triacetin that is converted into peracetic acid.

8. The method of claim 1, wherein the peracetic acid solution is for immediate use.

9. The method of claim 1, further comprising, after step (e), a step of adding a source of acid to stabilize the non-equilibrium solution of peracetic acid for storing and using throughout a working day.

10. The method of claim 1, wherein the peracetic acid solution is prepared on a site having a point-of-use.

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Submission number	3522313
Application number	EP11861339.7
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Receiving Office	European Patent Office, The Hague
Your reference	PS-KB-X11031-EP
Applicant	All applicants as on file
Documents submitted	package-data.xml epf1038.pdf (1 p.) CLMS-1.pdf\X11031-EP- Amended Claims Clean Copy.pdf (2 p.) DESC-1.pdf\X11031-EP- Clean Description.pdf (7 p.) ep-sfd-request.xml CLMS-HWA-1.pdf\X11031-EP-Amended Claims.pdf (2 p.) DESC-HWA-1.pdf\X11031-EP-Amended Description.pdf (8 p.) EPLETT-1.pdf\X11031-EP-Letter.pdf (1 p.)
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Correction by the EPO of errors in debit instructions filed by eOLF
Errors in debit instructions filed by eOLF that are caused by the editing of Form 1038E entries or the continued use of outdated software (all forms) may be corrected automatically by the EPO, leaving the payment date unchanged (see decision T 152/82, OJ EPO 1984, 301 and point 6.3 ff ADA, Supplement to OJ EPO 10/2007).

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Claims

1. A method of generating a non-equilibrium solution of peracetic acid, comprising:

a. providing water;

b. introducing a hydrogen peroxide-triacetin solution to the water;

c. mixing the hydrogen peroxide-triacetin solution and the water to form a mixture;

d. adding an aqueous source of an alkali metal hydroxide in the form of sodium hydroxide to the mixture, wherein the sodium hydroxide is ~~about~~ 1.82% to 7.28% weight, further, wherein the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is ~~about~~ 4.2:3.8:1; and

e. forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

2. The method of claim 1, further comprising, prior to step (b), a step of providing the hydrogen peroxide-triacetin solution comprising: introducing triacetin to a solution of aqueous hydrogen peroxide; and allowing the triacetin and the solution of aqueous hydrogen peroxide to mix to form the hydrogen peroxide-triacetin solution.

3. The method of claim 1, wherein the provided water is an aqueous stream.

4. The method of claim 1, wherein the provided water is contained in a mixing tank or other vessel.

5. The method of claim 1, wherein step (d) is performed simultaneously with step (b).

6. The method of claim 1, further comprising allowing the reaction medium sufficient time to convert the hydrogen peroxide-triacetin into peracetic acid, said time being ~~about~~ 30 seconds to ~~about~~ five minutes.

7. The method of claim 1, further comprising, after step (e), a step of sampling the reaction medium to determine the time required to maximize the amount of hydrogen peroxide-triacetin that is converted into peracetic acid.

8. The method of claim 1, wherein the peracetic acid solution is for immediate use.

9. The method of claim 1, further comprising, after step (e), a step of adding a source of acid to stabilize the non-equilibrium solution of peracetic acid for storing and using throughout a working day.

10. The method of claim 1, wherein the peracetic acid solution is prepared on a site having a point-of-use.

US2010/0227000 discloses a multi-part kit system for the preparation of a disinfectant of the peracetic acid type comprising a solid part A comprising at least one acetyl donor, a liquid part B which can be an aqueous solution of hydrogen peroxide (5-10% by wt). A water solution of 0.05 to 5% sodium hydroxide is also added.

US6221341 discloses a teeth whitening composition. Peracetic acid is the whitening agent and is generated by glyceryl triacetate with the addition of hydrogen peroxide in the presence of a base the regulates the pH.

SUMMARY OF THE PRESENT INVENTION

According to the present invention there is provided a method of generating a non-equilibrium solution of peracetic acid, comprising:

a. providing water;

b. introducing a hydrogen peroxide-triacetin solution to the water;

c. mixing the hydrogen peroxide-triacetin solution and the water to form a mixture;

d. adding an aqueous source of an alkali metal hydroxide in the form of sodium hydroxide to the mixture, wherein the sodium hydroxide is ~~about~~ 1.82% to 7.28% weight and the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is ~~about~~ 4.2:3.8:1; and

e. forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

In one embodiment, liquid compositions for use in generating non-

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[0046] The solution is mixed until a homogenous solution has been formed. The solution is allowed to mix by diffusion or by using a mixing device that is suitable for mixing liquids together. For example, 50% hydrogen peroxide can be introduced to a batch tank equipped with an overhead agitator blade. Triacetin can then be introduced to the 50% hydrogen peroxide when the agitator is in motion so that the components are thoroughly mixed.

[0047] The solution that is formed is a hydrogen peroxide-acetyl precursor solution, which also includes varying amounts of water, depending on the formulation used. When triacetin is used as the liquid acetyl precursor, the solution that is formed is a hydrogen peroxide-triacetin solution. One such hydrogen peroxide-triacetin solution comprises about 23% to about 40% hydrogen peroxide (from 50% aqueous hydrogen peroxide), about 20% to about 52% triacetin, water, and a trace amount of PAA that is formed within the first day of preparing the hydrogen peroxide-acetyl precursor solution. The pH is about 1.46 to about 2.2 and no deliberate attempt is made to adjust the pH further.

[0048] A preferable hydrogen peroxide-triacetin solution comprises about 27.15% hydrogen peroxide (from 50% aqueous hydrogen peroxide), about 45.67% triacetin, water, and a trace amount of PAA that is formed within the first day of preparing the hydrogen peroxide-acetyl precursor solution. The mole ratio of hydrogen peroxide:triacetin is about 3.8:1.

[0049] The hydrogen peroxide-acetyl precursor solutions prepared by this method may be used to generate non-equilibrium solutions of PAA on a site having a point-of-use of peracetic acid, in the methods described in section 4 below. The hydrogen peroxide-acetyl precursor solutions may be transported in any container suitable for transporting liquids, such as plastic tote bins of 1041-1249 L (275-330 gallons), drums of 57-208 L (15-55 gallons), 19 L (five-gallon) pails, 3.79 L (one-gallon) jugs, or other containers.

[0050] Example 1

[0076] Example 7

[0077] A 113.6 L (30-gallon) batch of 50% hydrogen peroxide and triacetin was prepared by blending 62 kg (137 lbs) triacetin with 74 kg (163 lbs) 50% hydrogen peroxide so that the mixture was 27.15% hydrogen peroxide and 45.67% triacetin, with the balance being water. Thus, the mole ratio of hydrogen peroxide:triacetin was 3.8:1. The mixture was stored in an opaque drum in a non-climate controlled environment. On storage, the mixture slowly formed PAA in the container. Since this reaction of hydrogen peroxide to PAA does not represent a destructive loss of total peroxygen, to determine the true stability of the hydrogen peroxide, both the hydrogen peroxide and the peracetic acid generated must be quantified and reported as total peroxygen recovered.

[0078] After 295 days of storage, the mixture was analyzed for total peroxygen recovered (expressed as hydrogen peroxide) to determine the percent loss of peroxygen. Using the eerie sulfate-iodometric titration method, the PAA generated and the hydrogen peroxide remaining were measured. The total peroxygen recovered after 295 days and the theoretical initial concentrations are shown in Table VII. All peroxygen is expressed as % hydrogen peroxide.

[0079] Table VII

	Initial Theoretical %	% (after 295 days)
Hydrogen Peroxide Recovered	27.17	15.20
PAA formed	0	15.57
Hydrogen Peroxide Reacted to form PAA	0	6.97
Total Recovered Hydrogen Peroxide	27.17	22.17
Total Hydrogen Peroxide Unproductive Loss		5.00

[0080] After 295 days, 22.17% of the total peroxygen was recovered as either titratable hydrogen peroxide or hydrogen peroxide reacted to form PAA. To determine the stability of the peroxygen in the mixture, the total recovered hydrogen peroxide after 295 days (22.17%) was

[00168] A container 120 of a hydrogen peroxide-acetyl precursor solution, which was a hydrogen peroxide-triacetin solution, and a container 140 of 50% sodium hydroxide were each equipped with chemical delivery diaphragm pumps 115 and 135, respectively. Inlet water 90, which was softened, was provided from a water source on the site having the point-of-use. Inlet water 90 was directed through a pressure regulator 100 and flow meter 105, then into a section of pipe where the hydrogen peroxide-triacetin solution was introduced through injection quill 110. Mixing was accomplished using static mixer 125. Then, the sodium hydroxide solution was added through injection quill 130 to form a reaction medium.

[00169] The hydrogen peroxide-triacetin solution and the sodium hydroxide solution may be added in a sequential manner as described, where the hydrogen peroxide-triacetin solution was added first, or they may be added to the water simultaneously through a "T" fitting placed before static mixer 125. If a T fitting is used, the hydrogen peroxide- triacetin solution and the 50% sodium hydroxide solution are introduced to opposite ends of the T fitting and the mixture is injected into the pipe of water. In other embodiments, the hydrogen peroxide and triacetin may be added separately, or sequentially, with either one first, or simultaneously, with the sodium hydroxide added either simultaneously with, or after, the hydrogen peroxide and triacetin.

[00170] The reaction medium was introduced to a residence chamber 145 which provided reaction time and contained a packing material to promote mixing. Residence chamber 145 was designed to be of a volume such that at a total flow rate of 3.79 L (one gallon) per minute, by the time the reaction medium reached sampling port 160, 30 seconds had elapsed since the sodium hydroxide solution had been added. A pH probe 155 monitored the pH of the mixture.

[00171] A controller 150 was interfaced to the flow meter 105 of the inlet water 90, and also to the chemical delivery diaphragm pumps 115 and 135. The controller 150 monitored the

rate of the inlet water 90 and governed the rate at which the hydrogen peroxide-triacetin solution and the NaOH solution were introduced. If the flow of inlet water 90 decreased because of lower PAA requirements at the point-of-use 195, the rates at which the hydrogen peroxide-triacetin solution and the NaOH solution were pumped decreased accordingly. If the flow of inlet water 90 increased because of higher PAA requirements at the point-of-use 195, the rates at which the hydrogen peroxide-triacetin solution and the NaOH solution were pumped increased accordingly. If the flow of inlet water 90 stopped, pumps 115 and 135 stopped. Thus, the generation of PAA was both continuous and intermittent, and was tailored to the PAA requirements at the point-of-use.

[00172] The rate of the reaction between the hydrogen peroxide and the triacetin to form PAA is dependent upon the temperature of inlet water 90. If the temperature of inlet water 90 is high (for example, about 70°F), the maximum conversion of triacetin into PAA may occur after 30 seconds, in which case the entire reaction medium was directed to the point-of-use 195. However, if the temperature of inlet water 90 is low (for example, about 32°F), longer reaction times may be necessary to maximize the conversion of triacetin into PAA. Then, the reaction medium was directed through one or more residence chambers 165, 175, and 185 which also contained a packing material to promote turbulence and cause thorough mixing. Residence chambers 165, 175, and 185 were designed to be of volumes such that at a total flow rate of 3.79 L (one gallon) per minute, the reaction medium reached sampling port 170 in one minute, sampling port 180 in two minutes, and sampling port 190 in five minutes, to permit sampling at different time intervals.

[00173] Thus, depending on the temperature of the inlet water, the time to achieve the maximum conversion of hydrogen peroxide and triacetin into PAA was determined by sampling and analyzing the solution at sample ports 160, 170, 180, and 190. The entire flow was then

directed from the sampling port with the highest amount of PAA to the point-of-use 195. In practice, the PAA generated from the reaction of hydrogen peroxide with triacetin is typically diluted at the point-of-use 195.

[00174] Point-of-use 195 may be recirculating cooling water; municipal wastewater; poultry chill tank water; water used to sanitize meat, poultry, or seafood products; fruit and vegetable rinse water; or water used to clean and sanitize equipment used in the dairy, food, or beverage processing industries.

[00175] Example 14

[00176] Referring again to Fig. 1, a 208 L (55-gallon) drum container 120 of a hydrogen peroxide-triacetin solution was prepared by mixing triacetin 114 kg (251.5 lbs) with 50% hydrogen peroxide 135 kg (298.4 lbs). The resulting 249.5 (550 lbs) of hydrogen peroxide-triacetin solution had a specific gravity of 1.19 g/mL (10 lbs/gal) and contained 54.3% hydrogen peroxide and 45.7% triacetin. Thus, the mole ratio of hydrogen peroxide:triacetin was 3.8:1. The apparatus depicted in Fig. 1 was used to continuously and intermittently prepare an approximately 1% PAA solution.

[00177] Softened local city water was directed through pressure regulator 100 at a flow rate of 3.79 L (one gallon) per minute as measured by flow meter 105. The container 120 of hydrogen peroxide-triacetin solution was equipped with a draw down tube so that the flow rate of the mixture could be measured. Diaphragm pump 115 injected the hydrogen peroxide-triacetin solution through injection quill 110. The hydrogen peroxide-triacetin solution was then mixed with the softened water using static mixer 125. A container 140 of 50% NaOH was also equipped with a draw down tube to measure the flow pumped by diaphragm pump 135 into injection quill 130.

[00178] The percent of PAA generated was measured by quenching samples drawn through the sampling ports in mineral acid, followed by use of the ceric sulfate-iodometric

[00191] Table XIX shows the microbiological results of the *Salmonella typhimurium* test solutions before treatment with PAA, one minute after the samples were dosed with a nominal 1.0 ppm PAA, and five minutes after being dosed with PAA. The *Salmonella* test solution started with $\log_{10}$ 7.07 CFU/mL prior to being treated with PAA from Perasan MP-2 or with the 1% PAA solution.

[00192] The *Salmonella* solution treated with a nominal 1.0 ppm PAA from Perasan MP-2 contained a $\log_{10}$ 1.76 CFU/mL after one minute and 0.70 CFU/mL after five minutes, corresponding to >99.999% reduction at both contact times. The *Salmonella* test solution treated with 1.0 ppm PAA from the 1% PAA solution contained a $\log_{10}$ 6.69 CFU/mL after one minute. This corresponds to 58.31% reduction. After five minutes, $\log_{10}$ 4.95 CFU/mL remained (99.24% reduction).

[00193] Table XIX

Salmonella typhimurium

Description	$\log_{10}$ CFU/ml remaining	$\log_{10}$-reduction	% reduction
Control	7.07	N/A	N/A
Perasan MP-2 (1 min)	1.76	5.31	99.9995
Perasan MP-2 (5 min)	0.70	6.37	>99.9999
1% PAA solution (1 min)	6.69	0.38	58.31
1% PAA solution (5 min)	4.95	2.12	99.24

[00194] The data in tables XVIII and XIX demonstrate that the 1% PAA had antimicrobial efficacy. After five minutes, the efficacy of this PAA was 99.24%, about equal to that of the Perasan MP-2, which was > 99.9999%. The difference in efficacy at one minute was likely not of statistical significance, given that the comparison was only at the one-minute interval.

~~[00195] 5. Solid Compositions for Bleaching and Stain Removal~~

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~~[00241] It can be seen that, in general, the composition displayed enhanced stain removal~~ properties at elevated temperature. Excellent stain removal properties were obtained for black tea and black coffee, and more moderate performance with red wine and blood. The composition had virtually no effect on grass stains or the chlorophyll pigment that was acetone-extracted from green grass.

[00242] The invention has been described above with the reference to the preferred embodiments. Those skilled in the art may envision other embodiments and variations of the ~~invention that fall within the scope of the claims.~~

pages 49-59 deleted.

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Claims

1. A method of generating a non-equilibrium solution of peracetic acid, comprising:

a. providing water;

b. introducing a hydrogen peroxide-triacetin solution to the water;

c. mixing the hydrogen peroxide-triacetin solution and the water to form a mixture;

d. adding an aqueous source of an alkali metal hydroxide in the form of sodium hydroxide to the mixture, wherein the sodium hydroxide is 1.82% to 7.28% weight, further, wherein the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is 4.2:3.8:1; and

e. forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

2. The method of claim 1, further comprising, prior to step (b), a step of providing the hydrogen peroxide-triacetin solution comprising: introducing triacetin to a solution of aqueous hydrogen peroxide; and allowing the triacetin and the solution of aqueous hydrogen peroxide to mix to form the hydrogen peroxide-triacetin solution.

3. The method of claim 1, wherein the provided water is an aqueous stream.

4. The method of claim 1, wherein the provided water is contained in a mixing tank or other vessel.

5. The method of claim 1, wherein step (d) is performed simultaneously with step (b).

6. The method of claim 1, further comprising allowing the reaction medium sufficient time to convert the hydrogen peroxide-triacetin into peracetic acid, said time being 30 seconds to five minutes.

7. The method of claim 1, further comprising, after step (e), a step of sampling the reaction medium to determine the time required to maximize the amount of hydrogen peroxide-triacetin that is converted into peracetic acid.

8. The method of claim 1, wherein the peracetic acid solution is for immediate use.

9. The method of claim 1, further comprising, after step (e), a step of adding a source of acid to stabilize the non-equilibrium solution of peracetic acid for storing and using throughout a working day.

10. The method of claim 1, wherein the peracetic acid solution is prepared on a site having a point-of-use.

US2010/0227000 discloses a multi-part kit system for the preparation of a disinfectant of the peracetic acid type comprising a solid part A comprising at least one acetyl donor, a liquid part B which can be an aqueous solution of hydrogen peroxide (5-10% by wt). A water solution of 0.05 to 5% sodium hydroxide is also added.

US6221341 discloses a teeth whitening composition. Peracetic acid is the whitening agent and is generated by glyceryl triacetate with the addition of hydrogen peroxide in the presence of a base the regulates the pH.

SUMMARY OF THE PRESENT INVENTION

According to the present invention there is provided a method of generating a non-equilibrium solution of peracetic acid, comprising:

a. providing water;

b. introducing a hydrogen peroxide-triacetin solution to the water;

c. mixing the hydrogen peroxide-triacetin solution and the water to form a mixture;

d. adding an aqueous source of an alkali metal hydroxide in the form of sodium hydroxide to the mixture, wherein the sodium hydroxide is 1.82% to 7.28% weight and the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is 4.2:3.8:1; and

e. forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

In one embodiment, liquid compositions for use in generating non-

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[0046] The solution is mixed until a homogenous solution has been formed. The solution is allowed to mix by diffusion or by using a mixing device that is suitable for mixing liquids together. For example, 50% hydrogen peroxide can be introduced to a batch tank equipped with an overhead agitator blade. Triacetin can then be introduced to the 50% hydrogen peroxide when the agitator is in motion so that the components are thoroughly mixed.

[0047] The solution that is formed is a hydrogen peroxide-acetyl precursor solution, which also includes varying amounts of water, depending on the formulation used. When triacetin is used as the liquid acetyl precursor, the solution that is formed is a hydrogen peroxide-triacetin solution. One such hydrogen peroxide-triacetin solution comprises about 23% to about 40% hydrogen peroxide (from 50% aqueous hydrogen peroxide), about 20% to about 52% triacetin, water, and a trace amount of PAA that is formed within the first day of preparing the hydrogen peroxide-acetyl precursor solution. The pH is about 1.46 to about 2.2 and no deliberate attempt is made to adjust the pH further.

[0048] A preferable hydrogen peroxide-triacetin solution comprises about 27.15% hydrogen peroxide (from 50% aqueous hydrogen peroxide), about 45.67% triacetin, water, and a trace amount of PAA that is formed within the first day of preparing the hydrogen peroxide-acetyl precursor solution. The mole ratio of hydrogen peroxide:triacetin is about 3.8:1.

[0049] The hydrogen peroxide-acetyl precursor solutions prepared by this method may be used to generate non-equilibrium solutions of PAA on a site having a point-of-use of peracetic acid, in the methods described in section 4 below. The hydrogen peroxide-acetyl precursor solutions may be transported in any container suitable for transporting liquids, such as plastic tote bins of 1041-1249 L (275-330 gallons), drums of 57-208 L (15-55 gallons), 19 L (five-gallon) pails, 3.79 L (one-gallon) jugs, or other containers.

[0050] Example 1

[0076] Example 7

[0077] A 113.6 L (30-gallon) batch of 50% hydrogen peroxide and triacetin was prepared by blending 62 kg (137 lbs) triacetin with 74 kg (163 lbs) 50% hydrogen peroxide so that the mixture was 27.15% hydrogen peroxide and 45.67% triacetin, with the balance being water. Thus, the mole ratio of hydrogen peroxide:triacetin was 3.8:1. The mixture was stored in an opaque drum in a non-climate controlled environment. On storage, the mixture slowly formed PAA in the container. Since this reaction of hydrogen peroxide to PAA does not represent a destructive loss of total peroxygen, to determine the true stability of the hydrogen peroxide, both the hydrogen peroxide and the peracetic acid generated must be quantified and reported as total peroxygen recovered.

[0078] After 295 days of storage, the mixture was analyzed for total peroxygen recovered (expressed as hydrogen peroxide) to determine the percent loss of peroxygen. Using the eerie sulfate-iodometric titration method, the PAA generated and the hydrogen peroxide remaining were measured. The total peroxygen recovered after 295 days and the theoretical initial concentrations are shown in Table VII. All peroxygen is expressed as % hydrogen peroxide.

[0079] Table VII

	Initial Theoretical %	% (after 295 days)
Hydrogen Peroxide Recovered	27.17	15.20
PAA formed	0	15.57
Hydrogen Peroxide Reacted to form PAA	0	6.97
Total Recovered Hydrogen Peroxide	27.17	22.17
Total Hydrogen Peroxide Unproductive Loss		5.00

[0080] After 295 days, 22.17% of the total peroxygen was recovered as either titratable hydrogen peroxide or hydrogen peroxide reacted to form PAA. To determine the stability of the peroxygen in the mixture, the total recovered hydrogen peroxide after 295 days (22.17%) was

[00168] A container 120 of a hydrogen peroxide-acetyl precursor solution, which was a hydrogen peroxide-triacetin solution, and a container 140 of 50% sodium hydroxide were each equipped with chemical delivery diaphragm pumps 115 and 135, respectively. Inlet water 90, which was softened, was provided from a water source on the site having the point-of-use. Inlet water 90 was directed through a pressure regulator 100 and flow meter 105, then into a section of pipe where the hydrogen peroxide-triacetin solution was introduced through injection quill 110. Mixing was accomplished using static mixer 125. Then, the sodium hydroxide solution was added through injection quill 130 to form a reaction medium.

[00169] The hydrogen peroxide-triacetin solution and the sodium hydroxide solution may be added in a sequential manner as described, where the hydrogen peroxide-triacetin solution was added first, or they may be added to the water simultaneously through a "T" fitting placed before static mixer 125. If a T fitting is used, the hydrogen peroxide- triacetin solution and the 50% sodium hydroxide solution are introduced to opposite ends of the T fitting and the mixture is injected into the pipe of water. In other embodiments, the hydrogen peroxide and triacetin may be added separately, or sequentially, with either one first, or simultaneously, with the sodium hydroxide added either simultaneously with, or after, the hydrogen peroxide and triacetin.

[00170] The reaction medium was introduced to a residence chamber 145 which provided reaction time and contained a packing material to promote mixing. Residence chamber 145 was designed to be of a volume such that at a total flow rate of 3.79 L (one gallon) per minute, by the time the reaction medium reached sampling port 160, 30 seconds had elapsed since the sodium hydroxide solution had been added. A pH probe 155 monitored the pH of the mixture.

[00171] A controller 150 was interfaced to the flow meter 105 of the inlet water 90, and also to the chemical delivery diaphragm pumps 115 and 135. The controller 150 monitored the

rate of the inlet water 90 and governed the rate at which the hydrogen peroxide-triacetin solution and the NaOH solution were introduced. If the flow of inlet water 90 decreased because of lower PAA requirements at the point-of-use 195, the rates at which the hydrogen peroxide-triacetin solution and the NaOH solution were pumped decreased accordingly. If the flow of inlet water 90 increased because of higher PAA requirements at the point-of-use 195, the rates at which the hydrogen peroxide-triacetin solution and the NaOH solution were pumped increased accordingly. If the flow of inlet water 90 stopped, pumps 115 and 135 stopped. Thus, the generation of PAA was both continuous and intermittent, and was tailored to the PAA requirements at the point-of-use.

[00172] The rate of the reaction between the hydrogen peroxide and the triacetin to form PAA is dependent upon the temperature of inlet water 90. If the temperature of inlet water 90 is high (for example, about 70°F), the maximum conversion of triacetin into PAA may occur after 30 seconds, in which case the entire reaction medium was directed to the point-of-use 195. However, if the temperature of inlet water 90 is low (for example, about 32°F), longer reaction times may be necessary to maximize the conversion of triacetin into PAA. Then, the reaction medium was directed through one or more residence chambers 165, 175, and 185 which also contained a packing material to promote turbulence and cause thorough mixing. Residence chambers 165, 175, and 185 were designed to be of volumes such that at a total flow rate of 3.79 L (one gallon) per minute, the reaction medium reached sampling port 170 in one minute, sampling port 180 in two minutes, and sampling port 190 in five minutes, to permit sampling at different time intervals.

[00173] Thus, depending on the temperature of the inlet water, the time to achieve the maximum conversion of hydrogen peroxide and triacetin into PAA was determined by sampling and analyzing the solution at sample ports 160, 170, 180, and 190. The entire flow was then

directed from the sampling port with the highest amount of PAA to the point-of-use 195. In practice, the PAA generated from the reaction of hydrogen peroxide with triacetin is typically diluted at the point-of-use 195.

[00174] Point-of-use 195 may be recirculating cooling water; municipal wastewater; poultry chill tank water; water used to sanitize meat, poultry, or seafood products; fruit and vegetable rinse water; or water used to clean and sanitize equipment used in the dairy, food, or beverage processing industries.

[00175] Example 14

[00176] Referring again to Fig. 1, a 208 L (55-gallon) drum container 120 of a hydrogen peroxide-triacetin solution was prepared by mixing triacetin 114 kg (251.5 lbs) with 50% hydrogen peroxide 135 kg (298.4 lbs). The resulting 249.5 (550 lbs) of hydrogen peroxide-triacetin solution had a specific gravity of 1.19 g/mL (10 lbs/gal) and contained 54.3% hydrogen peroxide and 45.7% triacetin. Thus, the mole ratio of hydrogen peroxide:triacetin was 3.8:1. The apparatus depicted in Fig. 1 was used to continuously and intermittently prepare an approximately 1% PAA solution.

[00177] Softened local city water was directed through pressure regulator 100 at a flow rate of 3.79 L (one gallon) per minute as measured by flow meter 105. The container 120 of hydrogen peroxide-triacetin solution was equipped with a draw down tube so that the flow rate of the mixture could be measured. Diaphragm pump 115 injected the hydrogen peroxide-triacetin solution through injection quill 110. The hydrogen peroxide-triacetin solution was then mixed with the softened water using static mixer 125. A container 140 of 50% NaOH was also equipped with a draw down tube to measure the flow pumped by diaphragm pump 135 into injection quill 130.

[00178] The percent of PAA generated was measured by quenching samples drawn through the sampling ports in mineral acid, followed by use of the ceric sulfate-iodometric

[00191] Table XIX shows the microbiological results of the *Salmonella typhimurium* test solutions before treatment with PAA, one minute after the samples were dosed with a nominal 1.0 ppm PAA, and five minutes after being dosed with PAA. The *Salmonella* test solution started with $\log_{10}$ 7.07 CFU/mL prior to being treated with PAA from Perasan MP-2 or with the 1% PAA solution.

[00192] The *Salmonella* solution treated with a nominal 1.0 ppm PAA from Perasan MP-2 contained a $\log_{10}$ 1.76 CFU/mL after one minute and 0.70 CFU/mL after five minutes, corresponding to >99.999% reduction at both contact times. The *Salmonella* test solution treated with 1.0 ppm PAA from the 1% PAA solution contained a $\log_{10}$ 6.69 CFU/mL after one minute. This corresponds to 58.31% reduction. After five minutes, $\log_{10}$ 4.95 CFU/mL remained (99.24% reduction).

[00193] Table XIX

Salmonella typhimurium

Description	$\log_{10}$ CFU/ml remaining	$\log_{10}$ reduction	% reduction
Control	7.07	N/A	N/A
Perasan MP-2 (1 min)	1.76	5.31	99.9995
Perasan MP-2 (5 min)	0.70	6.37	>99.9999
1% PAA solution (1 min)	6.69	0.38	58.31
1% PAA solution (5 min)	4.95	2.12	99.24

[00194] The data in tables XVIII and XIX demonstrate that the 1% PAA had antimicrobial efficacy. After five minutes, the efficacy of this PAA was 99.24%, about equal to that of the Perasan MP-2, which was > 99.9999%. The difference in efficacy at one minute was likely not of statistical significance, given that the comparison was only at the one-minute interval.

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IPR2025-01458 – Page 150

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Letter accompanying subsequently filed items

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The document(s) listed below is (are) subsequently filed documents pertaining to the following application:

Application number	11861339.7
Applicant's or representative's reference	PS-KB-X11031-EP

	Description of document	Original file name	Assigned file name
1	Amended claims with annotations	X11031-EP- Amended Claims.pdf	CLMS-HWA-1.pdf
2	Amended claims (clean copy)	X11031-EP- Amended Claims Clean Copy.pdf	CLMS-1.pdf
3	Amended description with annotations	X11031-EP- Amended Description.pdf	DESC-HWA-1.pdf
4	Amended description (clean copy)	X11031-EP- Clean Description.pdf	DESC-1.pdf
5	Letter relating to the search and examination procedure	X11031-EP-Letter.pdf	EPLETT-1.pdf

Signatures

Place:	**Leeds, UK, LS1 2DR**
Date:	**26 May 2015**
Signed by:	**Philip Stephenson 22528**
Association:	**Bailey Walsh & Co LLP**
Representative name:	**Philip Stephenson**
Capacity:	**(Representative)**

European Patent Office
Erhardstrasse 27
D-80298 Munich
Germany

Our ref.: PS-KM-X11031-EP
Your ref.: 8658/17669P
26 May 2015

Dear Sirs,

Re.: European Patent Application No. 11861339.7 in the name of Enviro Tech Chemical Services, Inc. --Methods and compositions for the generation of peracetic acid on site at the point- of- use

Further to our previous correspondence in relation to the abovementioned application and in particular the communication pursuant to Article 94(3) EPC dated 29th January 2015, we provide an amended set of claims and description which deal with the objections raised in the Examination Report. We also enclose clean copies of the same.

Specifically, claims 1 and 6 have the term "about" removed. In addition, the description has been amended to bring it into conformity with these amended claims and replacement pages are provided indicating the appropriate SI units.

We believe these amendments place the application in line for grant and we look forward to receiving the communication pursue to Rule 71(3) EPC in due course.

Yours faithfully,
BAILEY WALSH & CO LLP

P. Stephenson

Enc

The attorney A. Tomkinson called to obtain some clarification concerning the last communication of the examiner (21.01.2015).

The communication contained two opposite statements concerning inventive step of claims 1 to 10.

The inventive step was acknowledged for claims 1 to 10, in agreement with what the communication was then explaining in the paragraph on Article 56 EPC (paragraph 4), where it is said that deriving the claims from the prior-art was not obvious.

CLEAN CHEMISTRY EXHIBIT 1027
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Application No.	Ref.	Date
11 861 339.7 - 1454	ATSAR-X11031-EP	26.03.2015
Applicant Enviro Tech Chemical Services Inc.		

Result of consultation

A copy of the result of consultation of 25.03.2015 is enclosed for your information.

Zanobini, Alessandra
For the Examining Division

Enclosure(s): Copy of result of consultation (Form 2036)

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Application No. : 11 861 339.7

Consultation by telephone with the applicant / representative

Despatch for information

Participants

Applicant: Enviro Tech Chemical Services Inc.

Representative: Tomkinson, Alexandra

Member(s) of the Examining Division: Zanobini, Alessandra

Result of consultation

See Separate Sheet

25.03.2015
. . . . . .
Date

Zanobini, Alessandra
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BY FACSIMILE
00498923994465

EPO - Munich
27
12. Feb. 2015

Our ref.: AT-KM-X11031-EP
Your ref.: 8658/17669P
9 February 2015

Confirmation only already sent by fax

Dear Sirs,

Re.: European Patent Application No. 11861339.7 in the name of Enviro Tech Chemical Services, Inc. --Methods and compositions for the generation of peracetic acid on site at the point- of- use

We have received the Examination Report of 29th January 2015.

We note that there appears to be some inconsistencies with section 4 of the Report.

At the start of point 4 of the inventive step section, the examiner indicates that the subject matter of claims 1-10 involves an inventive step. However, the sixth paragraph on page two of the Report indicates that the claims of the application do not contain an inventive step. Further still, the examiner states at the end of section 4 that the claims do indeed appear to have an inventive step. Please can the examiner confirm whether there are any objections in respect of inventive step which the applicant has to overcome when responding to the latest Examination Report.

We look forward to hearing from you as soon as possible so that we can report the Examination Report to the client.

Yours faithfully,
BAILEY WALSH & CO LLP

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9 February 2015

Dear Sirs,

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We look forward to hearing from you as soon as possible so that we can report the Examination Report to the client.

Yours faithfully,
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Received at the EPO on Feb 09, 2015 12:30:40. Page 1 of 2

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Received at the EPO on Feb 09, 2015 12:30:40. Page 2 of 2

The examination is being carried out on the **following application documents**

Description, Pages

1-7, 9-59	as published
8, 8a, 8b	filed with telefax on 15-09-2014

Claims, Numbers

1-10	filed with telefax on 15-09-2014

Drawings, Sheets

1	as published

1 Article 123(2) EPC

The amendments filed with the letter dated 15.09.2014 do not introduce subject-matter which extends beyond the content of the application as filed, according to Article 123(2) EPC.

2 Clarity (Article 84 EPC)

The application does not meet the requirements of Article 84 EPC, because claims 1 and 6 are not clear.

The term "about" used in claims 1 and 6 are vague and unclear and leave the reader in doubt as to the meaning of the technical features to which they refer, thereby rendering the definition of the subject-matter of said claims unclear (Article 84 EPC).

3 Novelty (Article 54(1) and 54(2) EPC)

The present application meets the requirements of Article 52(1) EPC because the subject-matter of claims 1 to 10 is new within the meaning of Article 54(1) and (2) EPC.

No document disclosing a method of generating a non-equilibrium solution of peracetic acid was found, wherein the mole ratio sodium hydroxide to hydrogen peroxide to triacetin that is used is 4.2 to 3.8 to 1 and wherein the amount of sodium hydroxide is 1.82 to 7.28 by weight.

4 Inventive Step (Article 56 EPC)

The present application meets the requirements of Article 52(1) EPC because the subject-matter of claims 1 to 10 involves an inventive step within the meaning of Article 56 EPC.

D5 is considered to be the closest prior-art to the subject-matter of claims 1 to 10 and discloses a process of continuous generation of peroxyacid from hydrogen peroxide and an activator that are mixed in a tank. The tank is injected with a sodium hydroxide solution so that a desired pH is obtained (8 to 8.5) in order to help the conversion of the acetyl source to peracetate. The mole ratio of peroxide to acetyl is normally between 3.35:1 to 5:1.

The difference with claims 1 to 10 is in the acetyl source used that is TAED and in the mole ratio of used ingredients.

According to the present application, the pH is relevant for the conversion to PAA in the ideal time of 3 to 5 minutes. High pH values are obtained that drive the conversion of triacetin to peracetic acid rapidly, by contacting the triacetin-peroxide solutions with aqueous sodium hydroxide. At different mole ratios between sodium hydroxide and triacetin the conversion to PAA and the time needed are monitored and the best mole ratios and time of conversion are determined. The disinfecting effect of the obtained solution is tested against *E. coli* and *Salmonella* and compared to the effect of a commercial equilibrium solution of PAA with the same concentration (1 ppm) of PAA. The effect is comparable.

Therefore the problem solved by the present invention appears to provide a non-equilibrium solution of peracetic acid, thus a sanitizer to be used on site.

The solution proposed in claims 1 to 10 of the present application cannot be considered to involve an inventive step (Articles 52(1) and 56 EPC).

The problem of generating in situ sanitizing agents based on peroxicarboxylic acids is known in the art, because of the danger of disposing of highly concentrated peroxide solutions, due to their hazard.The use of multi-part kits like the ones disclosed in **D3** is known in the art and also is common the praxis of using an excess of sodium hydroxide. This excess forces the equilibrium in the sense of the development of PAA in situ, as disclosed in **D5**. Nevertheless, starting from **D5** nothing would suggest to replace TAED with triacetin, nor the exact mole ratio between the components that is suggested in claim 1 and that gives rise to an efficient disinfecting solution.

5 Other Remarks

When filing amended claims to avoid the clarity issues the applicant should at the same time bring the description into conformity with the amended claims. From paragraph [00195] on, for example, examples and compositions listed in the

description are not covered by the claims and should be removed. Care should be taken during revision, especially of the introductory portion and of any statements of problem or advantage, not to add subject-matter which extends beyond the content of the application as originally filed (Article 123(2) EPC).

The units (for ex. on par. [0049]) should meet the requirements of Rule 49(10) EPC and should be replaced by the appropriate SI units (see Guidelines F-II, Annex, 2). The present expressions should, however, be retained in parentheses after the replacement expressions. The applicant should take care of meeting the requirements of Rule 49 anywhere else in the description, while bringing the description into conformity with the claims.

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Application No.	Ref.	Date
11 861 339.7 - 1454	ATSAR-X11031-EP	29.01.2015
Applicant: Enviro Tech Chemical Services Inc.		

Communication pursuant to Article 94(3) EPC

The examination of the above-identified application has revealed that it does not meet the requirements of the European Patent Convention for the reasons enclosed herewith. If the deficiencies indicated are not rectified the application may be refused pursuant to Article 97(2) EPC.

You are invited to file your observations and insofar as the deficiencies are such as to be rectifiable, to correct the indicated deficiencies within a period

of 4 months

from the notification of this communication, this period being computed in accordance with Rules 126(2) and 131(2) and (4) EPC. One set of amendments to the description, claims and drawings is to be filed within the said period on separate sheets (R. 50(1) EPC).

If filing amendments, you must identify them and indicate the basis for them in the application as filed. Failure to meet either requirement may lead to a communication from the Examining Division requesting that you correct this deficiency (R. 137(4) EPC).

Failure to comply with this invitation in due time will result in the application being deemed to be withdrawn (Art. 94(4) EPC).

Registered letter
EPO Form 2001 12.10CSX

Zanobini, Alessandra
Primary Examiner
For the Examining Division

Enclosure(s): 3 page/s reasons (Form 2906)

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IPR2025-01458 – Page 164

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A. Tomkinson, MA (Cantab) PhD, CPA, EPA

Associates: P. Stephenson, MSci, PhD, MRSC, CPA, EPA
M. Kellett, MITMA
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Our ref: X11031-EP
14 January 2015

Zur Kasse

Dear Sirs

Re: European Patent Application No. 11861339.7 in the name of Enviro Tech Chemical Services, Inc. --Methods and compositions for the generation of peracetic acid on site at the point- of- use

Would you please deduct **€810** from our Account No. 28050202 in payment of the 5th annuity due on the above application before the end of March 2015.

Yours faithfully
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Notes 1 - 9 see overleaf.

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Please find enclosed the documents detailed below with the couriered copy of this faxed cover letter.

Our Ref	Application No.	Document Type	No. Pages	Fee Codes	Total Fee
X11031DIV2-EP	14185786.2	5th Annuity Fee	2	035	€810.00
X11031DIV1-EP	14185791.2	5th Annuity Fee	2	035	€810.00
X11031-EP	11861339.7	5th Annuity Fee	2	035	€810.00
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Our ref: X11031-EP
14 January 2015

Dear Sirs

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Would you please deduct **€810** from our Account No. 28050202 in payment of the 5th annuity due on the above application before the end of March 2015.

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Notes 1 - 9 see overleaf.

Signature (handwritten/no block letters): Bailey Walsh + Co

Place, Date: Leeds, UK; 14 January 2015

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-Claims

1. A method of generating a non-equilibrium solution of peracetic acid, comprising:

a. providing water;

b. introducing a hydrogen peroxide-triacetin solution to the water;

c. mixing the hydrogen peroxide-triacetin solution and the water to form a mixture;

d. adding an aqueous source of an alkali metal ~~or earth alkali metal~~ hydroxide in the form of sodium hydroxide to the mixture, wherein the sodium hydroxide is about 1.82% to 7.28% weight, further, wherein the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is about 4.2:3.8:1; and

e. forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

2. The method of claim 1, further comprising, prior to step (b), a step of providing the hydrogen peroxide-triacetin solution comprising: introducing triacetin to a solution of aqueous hydrogen peroxide; and allowing the triacetin and the solution of aqueous hydrogen peroxide to mix to form the hydrogen peroxide-triacetin solution.

~~3. The method of claim 1, wherein the hydrogen peroxide-triacetin solution has a mole ratio of hydrogen peroxide to triacetin of about 2.98:1 to about 12.84:1.~~

~~4.~~ 3. The method of claim 1, wherein the provided water is an aqueous stream.

~~5.~~ 4. The method of claim 1, wherein the provided water is contained in a mixing tank or other vessel.

~~6. The method of claim 1, wherein the aqueous source of the alkali metal or earth alkali metal hydroxide is sodium hydroxide.~~

~~7. The method of claim 6, wherein the sodium hydroxide is about 1.82% to about 7.28%.~~

~~8.~~ 5. The method of claim 1, wherein step (d) is performed simultaneously with step (b).

~~9.~~ 6. The method of claim 1, further comprising allowing the reaction medium sufficient time to ~~maximize the conversion~~convert ~~of~~ the hydrogen peroxide-triacetin into peracetic acid, said time being .

~~10. The method of claim 9, wherein the time to maximize the conversion of the hydrogen peroxide-triacetin into peracetic acid is~~ about 30 seconds to about five minutes.

~~11.~~ 7. The method of claim 1, further comprising, after step (e), a step of sampling the reaction medium to determine the time required to maximize the amount of hydrogen peroxide-triacetin that is converted into peracetic acid.

~~12. The method of claim 9, wherein the percent of triacetin that is converted into peracetic acid is about 40.9% to about 85.7%.~~

~~13. The method of claim 7, wherein the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is about 4.2:3.8:1.~~

~~14. The method of claim 1, wherein steps (a) through (e) are performed on a continuous basis.~~

~~15. The method of claim 1, wherein steps (a) through (e) are performed on an intermittent basis.~~

~~16.~~ 8. The method of claim 1, wherein the peracetic acid solution is for immediate use.

~~17.~~ 9. The method of claim 1, further comprising, after step (e), a step of adding a source of acid to stabilize the non-equilibrium solution of peracetic acid for storing and using throughout a working day.

~~18.~~ 10. The method of claim 1, wherein the peracetic acid solution is prepared on a site having a point-of-use.

comprising a solid part A comprising at least one acetyl donor, a liquid part B which can be an aqueous solution of hydrogen peroxide (5-10% by wt). A water solution of 0.05 to 5% sodium hydroxide is also added.

US6221341 discloses a teeth whitening composition. Peracetic acid is the whitening agent and is generated by glyceryl triacetate with the addition of hydrogen peroxide in the presence of a base the regulates the pH.

SUMMARY OF THE PRESENT INVENTION

According to the present invention there is provided a method of generating a non-equilibrium solution of peracetic acid, comprising:

a. providing water;

b. introducing a hydrogen peroxide-triacetin solution to the water;

c. mixing the hydrogen peroxide-triacetin solution and the water to form a mixture;

d. adding an aqueous source of an alkali metal hydroxide in the form of sodium hydroxide to the mixture; wherein the sodium hydroxide is about 1.82% to 7.28% weight and the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is about 4.2:3.8:1, and

e. forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

In one embodiment, liquid compositions for use in generating non-equilibrium solutions of PAA are provided. These hydrogen peroxide-acetyl precursor solutions comprise a solution of aqueous hydrogen peroxide, a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide, a trace

of peracetic acid, and water. A preferable acetyl precursor is triacetin, which displays an unexpectedly high solubility in hydrogen peroxide. These hydrogen peroxide-acetyl precursor solutions may be used for generating a non-equilibrium solution of peracetic acid for use as a disinfectant or sanitizer on a site having a point-of-use of peracetic acid.

The hydrogen peroxide- acetyl precursor solutions are advantageous in that they are formulated such that sufficient acetyl precursor can be dissolved in an amount of the hydrogen peroxide that would fully perhydrolyze all of the acetyl groups of the acetyl precursor.

a weight basis. Second, TAED has low water solubility, especially at the cooler water temperature bleaching cycles that are less damaging to fabrics. This is a major drawback, as current consumer trends in energy conservation are towards bleaching laundry fabrics with cool temperature water, rather than heated water. Undissolved TAED in cool temperature water is less unavailable as an acetyl precursor for the dissolved source of hydrogen peroxide and can even deposit on fabrics, necessitating a separate rinse step to remove it. Third, over time, when exposed to high humidity, solid TAED can react with solid source of hydrogen peroxide and the free water to form PAA, as well as degrade the activity, making it less effective overtime. Because the PAA is volatile, it imparts an undesirable pungent odour to the product. Thus, there is a need for a solid peroxygen bleach that overcomes the deficiencies of the TAED-containing bleaches.

EP1618786 discloses a composition for bactericide production and a process for producing organic peracid. The sterilising agent is a water based solution obtained by combining triacetin and hydrogen peroxide. The ratio between these two components is 1/10 and 20/1. The formulation can comprise 50% by weight triacetin, 15% of hydrogen peroxide, 0.17% hydrogen peroxide and 57.3% of remaining peroxide.

JP2004230323 discloses a sterilising agent for hard surfaces. Triacetin is used as the agent capable of liberating acetic acid, hydrogen peroxide is added and a metal hydroxide has to be used to bring the pH to 8-12. One example discloses a formulation of 0.23mol of triacetin, 0.73mol of hydrogen peroxide and 0.5 mol of sodium hydroxide.

US2010/0227000 discloses a multi-part kit system for the preparation of a disinfectant of the peracetic acid type

Claims

1. A method of generating a non-equilibrium solution of peracetic acid, comprising:

a. providing water;

b. introducing a hydrogen peroxide-triacetin solution to the water;

c. mixing the hydrogen peroxide-triacetin solution and the water to form a mixture;

d. adding an aqueous source of an alkali metal hydroxide in the form of sodium hydroxide to the mixture, wherein the sodium hydroxide is about 1.82% to 7.28% weight, further, wherein the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is about 4.2:3.8:1; and

e. forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

2. The method of claim 1, further comprising, prior to step (b), a step of providing the hydrogen peroxide-triacetin solution comprising: introducing triacetin to a solution of aqueous hydrogen peroxide; and allowing the triacetin and the solution of aqueous hydrogen peroxide to mix to form the hydrogen peroxide-triacetin solution.

3. The method of claim 1, wherein the provided water is an aqueous stream.

4. The method of claim 1, wherein the provided water is contained in a mixing tank or other vessel.

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5. The method of claim 1, wherein step (d) is performed simultaneously with step (b).

6. The method of claim 1, further comprising allowing the reaction medium sufficient time to convert the hydrogen peroxide-triacetin into peracetic acid, said time being about 30 seconds to about five minutes.

7. The method of claim 1, further comprising, after step (e), a step of sampling the reaction medium to determine the time required to maximize the amount of hydrogen peroxide-triacetin that is converted into peracetic acid.

8. The method of claim 1, wherein the peracetic acid solution is for immediate use.

9. The method of claim 1, further comprising, after step (e), a step of adding a source of acid to stabilize the non-equilibrium solution of peracetic acid for storing and using throughout a working day.

10. The method of claim 1, wherein the peracetic acid solution is prepared on a site having a point-of-use.

a weight basis. Second, TAED has low water solubility, especially at the cooler water temperature bleaching cycles that are less damaging to fabrics. This is a major drawback, as current consumer trends in energy conservation are towards bleaching laundry fabrics with cool temperature water, rather than heated water. Undissolved TAED in cool temperature water is less unavailable as an acetyl precursor for the dissolved source of hydrogen peroxide and can even deposit on fabrics, necessitating a separate rinse step to remove it. Third, over time, when exposed to high humidity, solid TAED can react with solid source of hydrogen peroxide and the free water to form PAA, as well as degrade the activity, making it less effective overtime. Because the PAA is volatile, it imparts an undesirable pungent odour to the product. Thus, there is a need for a solid peroxygen bleach that overcomes the deficiencies of the TAED-containing bleaches.

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US2010/0227000 discloses a multi-part kit system for the preparation of a disinfectant of the peracetic acid type comprising a solid part A comprising at least one acetyl donor, a liquid part B which can be an aqueous solution of hydrogen peroxide (5-10% by wt). A water solution of 0.05 to 5% sodium hydroxide is also added.

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c. mixing the hydrogen peroxide-triacetin solution and the water to form a mixture;

d. adding an aqueous source of an alkali metal hydroxide in the form of sodium hydroxide to the mixture, wherein the sodium hydroxide is about 1.82% to 7.28% weight and the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is about 4.2:3.8:1; and

e. forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

In one embodiment, liquid compositions for use in generating non-

CLEAN CHEMISTRY EXHIBIT 1027
IPR2025-01458 – Page 180

equilibrium solutions of PAA are provided. These hydrogen peroxide-acetyl precursor solutions comprise a solution of aqueous hydrogen peroxide, a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide, a trace of peracetic acid, and water. A preferable acetyl precursor is triacetin, which displays an unexpectedly high solubility in hydrogen peroxide. These hydrogen peroxide-acetyl precursor solutions may be used for generating a non-equilibrium solution of peracetic acid for use as a disinfectant or sanitizer on a site having a point-of-use of peracetic acid.

The hydrogen peroxide- acetyl precursor solutions are advantageous in that they are formulated such that sufficient acetyl precursor can be dissolved in an amount of the hydrogen peroxide that would fully perhydrolyze all of the acetyl groups of the acetyl precursor.

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Our ref: X11031-EP
12 September 2014

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Patent application/patent No. (please use a separate form for each application)

EP 11861339.7

PCT

Code		Amount/EUR	Code		Amount/EUR
001	Filing fee – EP direct[3]		010	Opposition fee	
501	Additional fee (more than 35 pages)		011	Fee for appeal	
020	Filing fee – entry EP phase[3] (Rule 159(1)(c) EPC)		029	Certified copy of application; priority document	
520	Additional fee (more than 35 pages) – entry EP phase		033	Renewal fee for the 3rd year	
	Additional fee for divisional applications (Rule 38(4)EPC)[9]		034	Renewal fee for the 4th year	580.00
002	Fee for a European search		035	Renewal fee for the 5th year	
055	Additional copy of docs cited in search report		036	Renewal fee for the 6th year	
015	Claims fee(s) (Rules 45(1), 162(1)EPC)[5]			Validation for[8]	
005	Designaton fee(s)[4]		094	Additional fee for 4th year	290.00
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122	Fee for further processing (non-fee related cases)				
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008	Additional printing fee for 36th and each subsequent page[7]				
016	Claims fee according to Rule 71(6) EPC[5]				
121	Fee for further processing (late performance of acts R. 71(3))				
022	Registering of transfer				

Notes 1 - 9 see overleaf.

Total 870.00

Signature (handwritten / no block letters)

Place, Date
Leeds, UK; 12th September 2014

EPO 1010 04.14

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BY FACSIMILE

Our ref.: AT-X11031-EP
Your ref.: 8658/17669P
15 September 2014

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Dear Sirs,

Re.: European Patent Application No. 11861339.7 in the name of Enviro Tech Chemical Services, Inc. --Methods and compositions for the generation of peracetic acid on site at the point- of- use

Further to issuance of the examination report dated 19th March 2014 and the two month extension of time, please find attached amended pages of the application, together with a marked up version of the pages as amended and an indication below as to the amendments that have been made.

Claim Amendments

Claim 1 has been amended to include the subject matter of previous claims 6, 7 and 13.

Previous claims 3, 6, 7, 12-15 have been deleted and the remaining claims have been renumbered accordingly.

Objections in the Examination Report

With regard to the clarity objection under Art. 84 EPC, the system units in previous claims 7 and 12 are % weight as evidenced by paragraph 0031 of the application and have

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been included in the amended claims.

The term "maximise the conversion of the hydrogen peroxide-triacetin into peracetic acid" used in previous claim 9 has been amended to read "convert the hydrogen peroxide-triacetin into peracetic acid" and the subject matter of previous claim 10 has been combined with previous claim 9 (now claim 6). This now defines the method step in amended claim 6 in concrete terms.

Previous claims 12, 14 and 15 of the application have been deleted in order to overcome the clarity objections under Art. 84 EPC.

Reference to prior art documents D1-D4 has been made on page 8 of the present application. Reference to D5 (WO01/46519) is already present on page 7 of the application. A statement of invention on page 8 of the application has been added to conform to the amended claim 1. Pages 8, 8a and 8b are enclosed herewith to replace previous page 8.

We trust the above amendments are sufficient to overcome the examiner's objections and we look forward to receiving confirmation to this effect in due course.

Yours faithfully,
BAILEY WALSH & CO LLP

A. Tomkinson

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-Claims

1. A method of generating a non-equilibrium solution of peracetic acid, comprising:

a. providing water;

b. introducing a hydrogen peroxide-triacetin solution to the water;

c. mixing the hydrogen peroxide-triacetin solution and the water to form a mixture;

d. adding an aqueous source of an alkali metal ~~or earth alkali metal~~ hydroxide in the form of sodium hydroxide to the mixture, wherein the sodium hydroxide is about 1.82% to 7.28% weight, further, wherein the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is about 4.2:3.8:1; and

e. forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

2. The method of claim 1, further comprising, prior to step (b), a step of providing the hydrogen peroxide-triacetin solution comprising: introducing triacetin to a solution of aqueous hydrogen peroxide; and allowing the triacetin and the solution of aqueous hydrogen peroxide to mix to form the hydrogen peroxide-triacetin solution.

~~3. The method of claim 1, wherein the hydrogen peroxide triacetin solution has a mole ratio of hydrogen peroxide to triacetin of about 2.98:1 to about 12.84:1.~~

~~4.~~ 3. The method of claim 1, wherein the provided water is an aqueous stream.

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~~5.~~ 4. The method of claim 1, wherein the provided water is contained in a mixing tank or other vessel.

~~6. The method of claim 1, wherein the aqueous source of the alkali metal or earth alkali metal hydroxide is sodium hydroxide.~~

~~7. The method of claim 6, wherein the sodium hydroxide is about 1.82% to about 7.28%.~~

~~8.~~ 5. The method of claim 1, wherein step (d) is performed simultaneously with step (b).

~~9.~~ 6. The method of claim 1, further comprising allowing the reaction medium sufficient time to ~~maximize the conversion~~convert ~~of~~ the hydrogen peroxide-triacetin into peracetic acid, said time being .

~~10. The method of claim 9, wherein the time to maximize the conversion of the hydrogen peroxide triacetin into peracetic acid is~~ about 30 seconds to about five minutes.

~~11.~~ 7. The method of claim 1, further comprising, after step (e), a step of sampling the reaction medium to determine the time required to maximize the amount of hydrogen peroxide-triacetin that is converted into peracetic acid.

~~12. The method of claim 9, wherein the percent of triacetin that is converted into peracetic acid is about 40.9% to about 85.7%.~~

Duration: 15.09.2014 14:56:56 - 15.09.2014 14:58:17. This page 7 of 13 was completed at 15.09.2014 14:57:42
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~~13. The method of claim 7, wherein the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is about 4.2:3.8:1.~~

~~14. The method of claim 1, wherein steps (a) through (e) are performed on a continuous basis.~~

~~15. The method of claim 1, wherein steps (a) through (e) are performed on an intermittent basis.~~

~~16.~~ 8. The method of claim 1, wherein the peracetic acid solution is for immediate use.

~~17.~~ 9. The method of claim 1, further comprising, after step (e), a step of adding a source of acid to stabilize the non-equilibrium solution of peracetic acid for storing and using throughout a working day.

~~18.~~ 10. The method of claim 1, wherein the peracetic acid solution is prepared on a site having a point-of-use.

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comprising a solid part A comprising at least one acetyl donor, a liquid part B which can be an aqueous solution of hydrogen peroxide (5-10% by wt). A water solution of 0.05 to 5% sodium hydroxide is also added.

US6221341 discloses a teeth whitening composition. Peracetic acid is the whitening agent and is generated by glyceryl triacetate with the addition of hydrogen peroxide in the presence of a base the regulates the pH.

SUMMARY OF THE PRESENT INVENTION

According to the present invention there is provided a method of generating a non-equilibrium solution of peracetic acid, comprising:

a. providing water;

b. introducing a hydrogen peroxide-triacetin solution to the water;

c. mixing the hydrogen peroxide-triacetin solution and the water to form a mixture;

d. adding an aqueous source of an alkali metal hydroxide in the form of sodium hydroxide to the mixture; wherein the sodium hydroxide is about 1.82% to 7.28% weight and the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is about 4.2:3.8:1, and

e. forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

In one embodiment, liquid compositions for use in generating non-equilibrium solutions of PAA are provided. These hydrogen peroxide-acetyl precursor solutions comprise a solution of aqueous hydrogen peroxide, a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide, a trace

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of peracetic acid, and water. A preferable acetyl precursor is triacetin, which displays an unexpectedly high solubility in hydrogen peroxide. These hydrogen peroxide-acetyl precursor solutions may be used for generating a non-equilibrium solution of peracetic acid for use as a disinfectant or sanitizer on a site having a point-of-use of peracetic acid.

The hydrogen peroxide- acetyl precursor solutions are advantageous in that they are formulated such that sufficient acetyl precursor can be dissolved in an amount of the hydrogen peroxide that would fully perhydrolyze all of the acetyl groups of the acetyl precursor.

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a weight basis. Second, TAED has low water solubility, especially at the cooler water temperature bleaching cycles that are less damaging to fabrics. This is a major drawback, as current consumer trends in energy conservation are towards bleaching laundry fabrics with cool temperature water, rather than heated water. Undissolved TAED in cool temperature water is less unavailable as an acetyl precursor for the dissolved source of hydrogen peroxide and can even deposit on fabrics, necessitating a separate rinse step to remove it. Third, over time, when exposed to high humidity, solid TAED can react with solid source of hydrogen peroxide and the free water to form PAA, as well as degrade the activity, making it less effective overtime. Because the PAA is volatile, it imparts an undesirable pungent odour to the product. Thus, there is a need for a solid peroxygen bleach that overcomes the deficiencies of the TAED-containing bleaches.

EP1618786 discloses a composition for bactericide production and a process for producing organic peracid. The sterilising agent is a water based solution obtained by combining triacetin and hydrogen peroxide. The ratio between these two components is 1/10 and 20/1. The formulation can comprise 50% by weight triacetin, 15% of hydrogen peroxide, 0.17% hydrogen peroxide and 57.3% of remaining peroxide.

JP2004230323 discloses a sterilising agent for hard surfaces. Triacetin is used as the agent capable of liberating acetic acid, hydrogen peroxide is added and a metal hydroxide has to be used to bring the pH to 8-12. One example discloses a formulation of 0.23mol of triacetin, 0.73mol of hydrogen peroxide and 0.5 mol of sodium hydroxide.

US2010/0227000 discloses a multi-part kit system for the preparation of a disinfectant of the peracetic acid type

CLEAN CHEMISTRY EXHIBIT 1027
IPR2025-01458 – Page 191

Claims

1. A method of generating a non-equilibrium solution of peracetic acid, comprising:

a. providing water;

b. introducing a hydrogen peroxide-triacetin solution to the water;

c. mixing the hydrogen peroxide-triacetin solution and the water to form a mixture;

d. adding an aqueous source of an alkali metal hydroxide in the form of sodium hydroxide to the mixture, wherein the sodium hydroxide is about 1.82% to 7.28% weight, further, wherein the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is about 4.2:3.8:1; and

e. forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

2. The method of claim 1, further comprising, prior to step (b), a step of providing the hydrogen peroxide-triacetin solution comprising: introducing triacetin to a solution of aqueous hydrogen peroxide; and allowing the triacetin and the solution of aqueous hydrogen peroxide to mix to form the hydrogen peroxide-triacetin solution.

3. The method of claim 1, wherein the provided water is an aqueous stream.

4. The method of claim 1, wherein the provided water is contained in a mixing tank or other vessel.

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5. The method of claim 1, wherein step (d) is performed simultaneously with step (b).

6. The method of claim 1, further comprising allowing the reaction medium sufficient time to convert the hydrogen peroxide-triacetin into peracetic acid, said time being about 30 seconds to about five minutes.

7. The method of claim 1, further comprising, after step (e), a step of sampling the reaction medium to determine the time required to maximize the amount of hydrogen peroxide-triacetin that is converted into peracetic acid.

8. The method of claim 1, wherein the peracetic acid solution is for immediate use.

9. The method of claim 1, further comprising, after step (e), a step of adding a source of acid to stabilize the non-equilibrium solution of peracetic acid for storing and using throughout a working day.

10. The method of claim 1, wherein the peracetic acid solution is prepared on a site having a point-of-use.

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CLEAN CHEMISTRY EXHIBIT 1027
IPR2025-01458 – Page 193

a weight basis. Second, TAED has low water solubility, especially at the cooler water temperature bleaching cycles that are less damaging to fabrics. This is a major drawback, as current consumer trends in energy conservation are towards bleaching laundry fabrics with cool temperature water, rather than heated water. Undissolved TAED in cool temperature water is less unavailable as an acetyl precursor for the dissolved source of hydrogen peroxide and can even deposit on fabrics, necessitating a separate rinse step to remove it. Third, over time, when exposed to high humidity, solid TAED can react with solid source of hydrogen peroxide and the free water to form PAA, as well as degrade the activity, making it less effective overtime. Because the PAA is volatile, it imparts an undesirable pungent odour to the product. Thus, there is a need for a solid peroxygen bleach that overcomes the deficiencies of the TAED-containing bleaches.

EP1618786 discloses a composition for bactericide production and a process for producing organic peracid. The sterilising agent is a water based solution obtained by combining triacetin and hydrogen peroxide. The ratio between these two components is 1/10 and 20/1. The formulation can comprise 50% by weight triacetin, 15% of hydrogen peroxide, 0.17% hydrogen peroxide and 57.3% of remaining peroxide.

JP2004230323 discloses a sterilising agent for hard surfaces. Triacetin is used as the agent capable of liberating acetic acid, hydrogen peroxide is added and a metal hydroxide has to be used to bring the pH to 8-12. One example discloses a formulation of 0.23mol of triacetin, 0.73mol of hydrogen peroxide and 0.5 mol of sodium hydroxide.

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IPR2025-01458 – Page 194

US2010/0227000 discloses a multi-part kit system for the preparation of a disinfectant of the peracetic acid type comprising a solid part A comprising at least one acetyl donor, a liquid part B which can be an aqueous solution of hydrogen peroxide (5-10% by wt). A water solution of 0.05 to 5% sodium hydroxide is also added.

US6221341 discloses a teeth whitening composition. Peracetic acid is the whitening agent and is generated by glyceryl triacetate with the addition of hydrogen peroxide in the presence of a base the regulates the pH.

SUMMARY OF THE PRESENT INVENTION

According to the present invention there is provided a method of generating a non-equilibrium solution of peracetic acid, comprising:

a. providing water;

b. introducing a hydrogen peroxide-triacetin solution to the water;

c. mixing the hydrogen peroxide-triacetin solution and the water to form a mixture;

d. adding an aqueous source of an alkali metal hydroxide in the form of sodium hydroxide to the mixture, wherein the sodium hydroxide is about 1.82% to 7.28% weight and the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is about 4.2:3.8:1; and

e. forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

In one embodiment, liquid compositions for use in generating non-

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equilibrium solutions of PAA are provided. These hydrogen peroxide-acetyl precursor solutions comprise a solution of aqueous hydrogen peroxide, a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide, a trace of peracetic acid, and water. A preferable acetyl precursor is triacetin, which displays an unexpectedly high solubility in hydrogen peroxide. These hydrogen peroxide-acetyl precursor solutions may be used for generating a non-equilibrium solution of peracetic acid for use as a disinfectant or sanitizer on a site having a point-of-use of peracetic acid.

The hydrogen peroxide- acetyl precursor solutions are advantageous in that they are formulated such that sufficient acetyl precursor can be dissolved in an amount of the hydrogen peroxide that would fully perhydrolyze all of the acetyl groups of the acetyl precursor.

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D. Corbyn, MChem, EPA

European Patent Office
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D-80298 Munich
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BY FACSIMILE

Our ref.: AT-X11031-EP
Your ref.: 8658/17669P
15 September 2014

Dear Sirs,

Re.: European Patent Application No. 11861339.7 in the name of Enviro Tech Chemical Services, Inc. --Methods and compositions for the generation of peracetic acid on site at the point- of- use

Further to issuance of the examination report dated 19th March 2014 and the two month extension of time, please find attached amended pages of the application, together with a marked up version of the pages as amended and an indication below as to the amendments that have been made.

Claim Amendments

Claim 1 has been amended to include the subject matter of previous claims 6, 7 and 13.

Previous claims 3, 6, 7, 12-15 have been deleted and the remaining claims have been renumbered accordingly.

Objections in the Examination Report

With regard to the clarity objection under Art. 84 EPC, the system units in previous claims 7 and 12 are % weight as evidenced by paragraph 0031 of the application and have

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been included in the amended claims.

The term "maximise the conversion of the hydrogen peroxide-triacetin into peracetic acid" used in previous claim 9 has been amended to read "convert the hydrogen peroxide-triacetin into peracetic acid" and the subject matter of previous claim 10 has been combined with previous claim 9 (now claim 6). This now defines the method step in amended claim 6 in concrete terms.

Previous claims 12, 14 and 15 of the application have been deleted in order to overcome the clarity objections under Art. 84 EPC.

Reference to prior art documents D1-D4 has been made on page 8 of the present application. Reference to D5 (WO01/46519) is already present on page 7 of the application. A statement of invention on page 8 of the application has been added to conform to the amended claim 1. Pages 8, 8a and 8b are enclosed herewith to replace previous page 8.

We trust the above amendments are sufficient to overcome the examiner's objections and we look forward to receiving confirmation to this effect in due course.

Yours faithfully,
BAILEY WALSH & CO LLP

A. Tomkinson

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D. Corbyn, MChem, EPA

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BY FACSIMILE
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Our ref: X11031-EP
12 September 2014

Dear Sirs

Re: European Patent Application No. 11861339.7 in the name of Enviro Tech Chemical Services, Inc. --Methods and compositions for the generation of peracetic acid on site at the point- of- use

Would you please deduct **€870** from our Account No. 28050202 in payment of the 4th annuity, under extension, due on the above application before the end of September 2014.

Yours faithfully
BAILEY WALSH & CO. LLP

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035	Renewal fee for the 5th year	
036	Renewal fee for the 6th year	
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	Extension for[9]	

Total 870.00

Notes 1 - 9 see overleaf.

Signature (handwritten/no block letters)

Place, Date
Leeds, UK; 12th September 2014

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094	Additional fee for 4th year	290.00
	Extension for[9]	
	Total	870.00

Notes 1 - 9 see overleaf.

Signature (handwritten/no block letters)

Place, Date: Leeds, UK; 12th September 2014

EPO 1010 04.14

The attorney A. Tomkinson called to obtain some clarification concerning the last communication of the examiner (ESOP).

The communication contained two opposite statements concerning inventive step of claim 4.

The inventive step was acknowledged only for claims 7 and 13. Claim 4 was confirmed as not inventive, in agreement with what the communication is then explaining in the paragraph on Article 56 EPC.

CLEAN CHEMISTRY EXHIBIT 1027
IPR2025-01458 – Page 202

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Fax: +49 89 2399 4465

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Tel: +49 89 2399 - 8139
or call
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Substantive Examiner
Name: Zanobini, Alessandra
Tel: +49 89 2399 - 7850

Application No.	Ref.	Date
11 861 339.7 - 1454	ATSAR-X11031-EP	16.07.2014
Applicant Enviro Tech Chemical Services Inc.		

Result of consultation

A copy of the result of consultation of 10.07.2014 is enclosed for your information.

Zanobini, Alessandra
For the Examining Division

Enclosure(s): Copy of result of consultation (Form 2036)

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European Patent Office
Office européen des brevets

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80298 MUNICH
GERMANY
Tel: +49 89 2399 0
Fax: +49 89 2399 4465

Application No. : 11 861 339.7

Consultation by telephone with the applicant / representative

Despatch for information

Participants

Applicant: Enviro Tech Chemical Services Inc.

Representative: Tomkinson, Alexandra

Member(s) of the Examining Division: Zanobini, Alessandra

Result of consultation

See Separate Sheet

10.07.2014
. . . . . .
Date

Zanobini, Alessandra
. . . . . .
Examiner

Enclosure(s):

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European Patent Office
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GERMANY

Questions about this communication ?
Contact Customer Services at www.epo.org/contact

Tomkinson, Alexandra
Bailey Walsh & Co LLP
1 York Place
Leeds, LS1 2DR
ROYAUME UNI

Date: 08.07.14

Reference	Application No./Patent No.
ATSAR-X11031-EP	11861339.7 - 1454 / 2688399

Applicant/Proprietor
Enviro Tech Chemical Services Inc.

Extension of time limit pursuant to Rule 132(2) EPC

Examination procedure

With reference to your request, the time limit for replying to the communication pursuant to Article 94(3) EPC dated 19.03.14 has been extended

by 2 months

to a total of 6 months

from the date of notification of the above-mentioned communication.

Please note: To the extent that your request exceeded the above extension, your request has been refused.

Note
The granting of extensions to time limits is governed by the Implementing Regulations to the EPC and the Guidelines for Examination in the EPO, part E-VII, 1.6. A request for extension, which would result in the total period set exceeding six months, must be reasoned and supported by evidence.

If no reply to the communication is received in due time, the European patent application will be deemed to be withdrawn (Art. 94(4) EPC).

For the Examining Division

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BaileyWalsh&Co

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Partners: G.Wood, MEng, CPA, EPA
P.B. Dyson, LLB (Hons), ACIS, MITMA
A. Tomkinson, MA (Cantab) PhD, CPA, EPA
Associates: P. Stephenson, MSci, PhD, MRSC, CPA, EPA
M. Kellett, MITMA
D. Corbyn, MChem, EPA

EPO - Munich
75
07 Juli 2014

European Patent Office
Erhardstrasse 27
D-80298 Munich
Germany

BY FACSIMILE

Our ref.: AT-X11031-EP
Your ref.: 8658/17669P
3 July 2014

Confirmation only already sent by fax

Dear Sirs,

Re.: European Patent Application No. 11861339.7 in the name of Enviro Tech Chemical Services, Inc. --Methods and compositions for the generation of peracetic acid on site at the point- of- use

Further ot issuance of the examination report dated 19th March 2014, we hereby request a two month extension of time in which to respond to the objections in the report.

We look forward to receiving confirmation that the extension of time has been allowed in due course.

Yours faithfully,
BAILEY WALSH & CO LLP

A-T

A. Tomkinson

Also at 20 Bootham, York, YO30 7BL, UK **Tel:** 0800 783 7623
3M Buckley Innovation Centre, Firth Street, Huddersfield, HD1 3BD, UK **Tel:** 0800 783 7623
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A. Tomkinson, MA (Cantab) PhD, CPA, EPA
Associates: P. Stephenson, MSci, PhD, MRSC, CPA, EPA
M. Kellett, MITMA
D. Corbyn, MChem, EPA

European Patent Office
Erhardstrasse 27
D-80298 Munich
Germany

BY FACSIMILE

Our ref.: AT-X11031-EP
Your ref.: 8658/17669P
3 July 2014

Dear Sirs,

Re.: European Patent Application No. 11861339.7 in the name of Enviro Tech Chemical Services, Inc. --Methods and compositions for the generation of peracetic acid on site at the point- of- use

Further ot issuance of the examination report dated 19th March 2014, we hereby request a two month extension of time in which to respond to the objections in the report.

We look forward to receiving confirmation that the extension of time has been allowed in due course.

Yours faithfully,
BAILEY WALSH & CO LLP

A. Tomkinson

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Duration: 03.07.2014 14:50:32 - 03.07.2014 14:50:38. This page 1 of 1 was completed at 03.07.2014 14:50:38
Received at the EPO on Jul 03, 2014 14:50:38. Page 1 of 1

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11861339.7 - 1454 / 2688399 27.05.14

Client Database System (CDS) - clean up.

Application Nr.: 11861339.7

Following clean up action in CDS the entries concerning the **Representative for the applicant** have been amended and are now as follows:

Tomkinson, Alexandra
Bailey Walsh & Co LLP
1 York Place
Leeds, LS1 2SD
GB

Where appropriate, the Register of European Patents will be updated to show the amended details.

For questions please contact the Client Data Registration department of the European Patent Office in Munich, telephone +49 (0)89 2399 2780.

EB21171

EPO Form 2596C 04.08

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P. B. Dyson, LLB (Hons), ACIS, MITMA
A. Tomkinson, MA (Cantab) PhD, CPA, EPA
Associates: P. Stephenson, MSci, PhD, MRSC, CPA, EPA
M. Kellett, MITMA
D. Corbyn, MChem (Hons), EPA

European Patent Office
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Germany

EPO - Munich
27
24. Mai 2014

BY FACSIMILE

Our ref.: AT-G48759
Your ref.:
22 May 2014

Confirmation only already sent by fax

Dear Sirs,

Re.: Notification of Change of Address of Bailey Walsh & Co. LLP - Deposit Account 28050202

We are the representatives and address for service for a large number of patent cases at the EPO and also have a deposit account with you (28050202). We have moved offices from:

5 York Place,
Leeds
LS1 2SD
UK

to

1 York Place,
Leeds,
LS1 2DR
UK

Please can you record the change of address for all patent cases so that all correspondence relating to the cases we are recorded as the address for service/representatives for is forwarded to our new offices. If you have any queries, please contact myself, Alexandra Tomkinson (Partner) at Bailey Walsh & Co. LLP. Our e-mail address and contact phone numbers and fax numbers will remain the same.

Yours faithfully,
BAILEY WALSH & CO LLP

A. Tomkinson

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P. B. Dyson, LLB (Hons), ACIS, MITMA
A. Tomkinson, MA (Cantab) PhD, CPA, EPA

Associates: P. Stephenson, MSci, PhD, MRSC, CPA, EPA
M. Kellett, MITMA
D. Corbyn, MChem (Hons), EPA

European Patent Office
Erhardstrasse 27
D-80298 Munich
Germany

BY FACSIMILE

Our ref.: AT-G48759
Your ref.:
22 May 2014

Dear Sirs,

Re.: Notification of Change of Address of Bailey Walsh & Co. LLP - Deposit Account 28050202

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to

1 York Place,
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UK

Please can you record the change of address for all patent cases so that all correspondence relating to the cases we are recorded as the address for service/representatives for is forwarded to our new offices. If you have any queries, please contact myself, Alexandra Tomkinson (Partner) at Bailey Walsh & Co. LLP. Our e-mail address and contact phone numbers and fax numbers will remain the same.

Duration: 22.05.2014 14:24:38 - 22.05.2014 14:24:50. This page 1 of 2 was completed at 22.05.2014 14:24:46
Received at the EPO on May 22, 2014 14:24:50. Page 1 of 2

Yours faithfully,
BAILEY WALSH & CO LLP

A. Tomkinson

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Duration: 22.05.2014 14:24:38 - 22.05.2014 14:24:50. This page 2 of 2 was completed at 22.05.2014 14:24:50
Received at the EPO on May 22, 2014 14:24:50. Page 2 of 2

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Tomkinson, Alexandra
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Leeds LS1 2SD
ROYAUME UNI

Date
07.05.14

Reference	Application No./Patent No.
ATSAR-X11031-EP	11861339.7 - 1454 / 2688399
Applicant/Proprietor	
Enviro Tech Chemical Services Inc.	

Notice drawing attention to Rule 51(2) EPC, Article 2 No. 5 of the Rules relating to Fees,
- Payment of the renewal fee plus additional fee -

The renewal fee for the 04. year fell due on 31.03.14 unless this date falls within the period covered by an interruption of the proceedings in accordance with Rule 142(1) EPC, or a request for re-establishment of rights is pending (Art. 122, R. 51(4) EPC).

The current rate of the renewal fee amounts to **EUR 580,00** (see current Schedule of fees and costs).

The renewal fee was not paid by the due date.
The renewal fee may still be validly paid **up to the last day of the sixth calendar month** following the due date, provided that the additional fee (**50%** of the renewal fee) is paid at the same time, see OJ EPO 2008, 5.

Within the above period, which cannot be extended, the following fees are to be paid:

Renewal fee for the 04. year:	EUR	580,00
Additional fee:	EUR	290,00
TOTAL AMOUNT	EUR	870,00

If the renewal fee and the additional fee are not paid in due time, the European patent application shall be deemed to be withdrawn (Art. 86(1) EPC).

Note to users of the automatic debiting procedure
The normal time limit for payment of the above renewal fee had already expired when the automatic debit order was received. The renewal fee and the surcharge will be debited automatically on the last day of the six-month period (Supplementary publication 4 - OJ EPO 2014).

Important information concerning fee amounts
Following any amendment to the Rules relating to Fees, the amount(s) mentioned in this communication may be different from the amount(s) **actually due on the date of payment.** The latest version of the Schedule of fees and expenses, published as a Supplement to the Official Journal of the EPO, is also available on the EPO website (**www.epo.org**) and can be found under **www.epo.org/schedule-of-fees**, which allows the viewing, downloading and searching for individual fee amounts, both current and previous.
Please note that procedural fees are usually adjusted every two years, on even years, with effect from 1 April.

Payments by cheque delivered or sent direct to the EPO are no longer accepted as from 1 April 2008 (see OJ EPO 2007, 626).

For the Examining Division



CLEAN CHEMISTRY EXHIBIT 1027
IPR2025-01458 – Page 214

The examination is being carried out on the **following application documents**

Description, Pages

1-59 as published

Claims, Numbers

1-18 filed with entry into the regional phase before the EPO

Drawings, Sheets

1 as published

Reference is made to the following documents; the numbering will be adhered to in the rest of the procedure.

D1 EP 1 618 786 A1 (KAO CORP [JP]) 25 January 2006 (2006-01-25)

D2 JP 2004 230323 A (MATSUSHITA ELECTRIC IND CO LTD) 19 August 2004 (2004-08-19)

D3 US 2010/227000 A1 (AMES KELLY ANN [GB] ET AL BOARD KELLY ANN [GB] ET AL) 9 September 2010 (2010-09-09)

D4 US 6 221 341 B1 (MONTGOMERY R ERIC [US]) 24 April 2001 (2001-04-24)

D5 WO 01/46519 A1 (WARWICK INTERNAT GROUP LTD [GB]; WITHENSHAW JOHN DAVID [GB]; SKETT MAT) 28 June 2001 (2001-06-28)

The present application concerns a method for the generation of peracetic acid in a water solution. A solution of hydrogen peroxide and triacetin is added to water and a solution of a metal hydroxide is added to the mixture and stirred. A certain molar ratio within the components ensures a quick formation of peroxyacetic acid: the ratio hydrogen peroxide to acetine is from 2:98:1 to 12:84:1. The method is used in mixing tanks or under aqueous stream. A source of acid can be added to stabilize the non-equilibrium solution of peracetic acid.

Clarity (Article 84 EPC)

The application does not meet the requirements of Article 84 EPC, because claims 7, 9-12, 14-15 are not clear.

Claims 7 and 12 are not clear because the amount of sodium hydroxide and triacetin are not expressed precisely, in both cases the system units are missing.

The relative term "maximize the conversion of the hydrogen peroxide-triacetin into peracetic acid" used in claims 9-11 has no well-recognized meaning and leaves the reader in doubt as to the meaning of the technical features to which it refers, thereby rendering the definition of the subject-matter of said claims unclear (Article 84 EPC). Moreover, such a formulation is not allowable because it appears possible to define the subject-matter in more concrete terms, viz. in terms of how the effect is to be achieved.

Claims 12 do not meet the requirements of Article 84 EPC in that the matter for which protection is sought is not defined. The claims attempt to define the subject-matter in terms of the result to be achieved. Such a definition is only allowable under the conditions elaborated in the Guidelines F-IV, 4.10. In this instance, however, such a formulation is not allowable because it appears possible to define the subject-matter in more concrete terms, viz. in terms of how the effect is to be achieved.

The terms "continuous" and "intermittent" used in claims 14 and 15 are vague and unclear and leave the reader in doubt as to the meaning of the technical features to which they refer, thereby rendering the definition of the subject-matter of said claims unclear (Article 84 EPC). The process of sampling is believed to take place with a certain frequence and it is not clear when a certain frequence is to be interpreted as continuous sampling and when the sampling is instead processed after intervals such as to require the definition of "intermittent".

Claim 12 has been considered as not novel since the amount have been considered as expressed in wt. %. Claims 14 and 15 have been considered as not novel because the repetition of the steps makes sense only if the method is applied when water is used in an aqueous stream. They therefore appear not to make sense if the method concerns a mixing tank application.

Novelty (Article 54(1) and 54(2) EPC)

The present application does not meet the requirements of Article 52(1) EPC because the subject-matter of claims 1-3, 5-6, 8-12, 14-18 is not new within the meaning of Article 54(1) and (2) EPC.

D1 describes a process for the production of a bactericide based on an organic peracid. The sterilizing agent is a water based solution obtained by combining triacetin and hydrogen peroxide. The ratio between these two component has to be comprised between 1/10 and 20/1. The variation in the peroxyacid concentration is controlled (par. [0082-[0084], example 1, tables 1 to 5). Table 10, example 3 displays the control of *Bacillus cereus* and *B. subtilis* by triacetin solutions comprising hydrogen peroxide to wich a base is added to regulate the pH. Table 11 displays a formulation which

CLEAN CHEMISTRY EXHIBIT 1027
IPR2025-01458 – Page 216

comprises 50% by weight of triacetin, 15% of hydrogen peroxide, 0.17% of hydrogen peroxide and 57.3% of remaining peroxide. **D1** is considered as novelty destroying for claims 1-2, 5-6, 8, 11-12, 16-18.

D2 is considered as novelty destroying for claims 1-3, 5-6, 16, 18. It describes a sterilising agent for hard surfaces. Triacetin is used as the agent capable of liberating acetic acid, hydrogen peroxide is to be added and a metal hydroxide has to be used to bring the pH to 8-12. Paragraph [0057] describes an example where 0.23 mol of triacetin, 0,73 of hydrogen peroxide and 0,5 of sodium hydroxide are used.

D3 describes a solid constituent A that is an acetyl source (TAED preferably) and that is combined with a liquid part B that is an aqueous solution of hydrogen peroxide (5-10% by wt.). A water solution of 0.05 to 5% alkali is also added by adding sodium hydroxide. The pH is maintained between 5 and 8 and the PAA generation takes 5 to 15 minutes. It is a method to generate PAA using a kit of parts where one part is the solid acetyl donor and another part is the liquid composition of hydrogen peroxide.

D4 discloses a teeth whitening composition. Peracetic acid is the whitening agent and is generated by glyceryl triacetate with the addition of hydrogen peroxide and in the presence of a base that regulates the pH. The most favourable conditions are at higher pH where the whitening agent works faster. The human use determines the reagent and the conditions used (col. 3, l. 55-col. 4, l. 57, col. 5, l. 58-col. 7, l. 16 and col. 7, l. 60-col. 8, l. 23, examples I-III and V).

Inventive Step (Article 56 EPC)

The present application does not meet the requirements of Article 52(1) EPC because the subject-matter of claims 4, 7, 13 does not involve an inventive step within the meaning of Article 56 EPC.

D1 is considered to be the prior art closest to the subject-matter of claims 4, 7, 13 and discloses the production of a bactericide based on peracetic acid.

The subject-matter of claim 4 therefore differs from this known **D1** in that the bactericide is prepared in an aqueous stream and not in a tank.

According to the present application, the point of use can be at the end of a pipe where water is injected and subsequently the required components (triacetin-hydrogen peroxide solution and hydroxide solution respectively) are added simultaneously or one after the other.

The problem to be solved by the present invention may therefore be regarded as how to provide a method that generates a non-equilibrium solution of peracetic acid in an aqueous stream.

CLEAN CHEMISTRY EXHIBIT 1027
IPR2025-01458 – Page 217

The solution proposed in claim 4 of the present application cannot be considered to involve an inventive step (Articles 52(1) and 56 EPC). Continuous peroxiacid generating systems are known in the art as in the case of **D5**, where a different source of acetyl groups is used rather than triacetin. **D5** discloses a pipe system (see Fig. 1) where hydrogen peroxide and TAED are introduced in a mixer and subsequently hydroxide anions are added as a solution of sodium hydroxide (p. 3, l. 6-10, p. 4, l. 14-28). A final container allows further mixing with the last added ingredient (p. 8, l. 14-26). The person skilled in the art would not need to use any inventive skill to adapt the steps of the present application to a pipe system like the one of **D5**. An inventive step can not be acknowledged for claim 4.

The subject-matter of claims 7 and 13 therefore differs from this known **D1** in that the ratio between the components does not correspond to the ratio disclosed in claim 13. A higher amount of metal hydroxide is used in the present application.

According to the present application, when the hydroxide solution is added and the pH is highly alkaline. The reaction is remarkably fast, proceed with a high conversion of the acetyl precursor into PAA.

The problem to be solved by the present invention may therefore be regarded as how to provide an alternative method of preparation of a solution of peracetic acid (PAA) that is not at the equilibrium.

The solution proposed in claim 7 and 13 of the present application can be considered to involve an inventive step (Articles 52(1) and 56 EPC). The displayed examples show the rapid generation of PAA and a sanitizing effect on *E. coli* and *Salmonella* strains. The man skilled in the art, although aware of the possibility of using the same ingredients (triacetin, hydrogen peroxide and sodium hydroxide), would not have been able, starting from **D1** to solve the problem posed in the same way without needing an inventive step. The solution proposed in claims 7 and 13 is therefore considered to involve an inventive step.

Acknowledgement of the Prior-Art

To meet the requirements of Rule 42(1)(b) EPC, **D1** to **D5** should be identified in the description and the relevant background art disclosed therein should be briefly discussed.

CLEAN CHEMISTRY EXHIBIT 1027
IPR2025-01458 – Page 218

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Formalities Officer
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or call
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Substantive Examiner
Name: Zanobini, Alessandra
Tel: +49 89 2399 - 7850

Application No.	Ref.	Date
11 861 339.7 - 1454	ATSAR-X11031-EP	19.03.2014
Applicant Enviro Tech Chemical Services Inc.		

Communication pursuant to Article 94(3) EPC

The examination of the above-identified application has revealed that it does not meet the requirements of the European Patent Convention for the reasons enclosed herewith. If the deficiencies indicated are not rectified the application may be refused pursuant to Article 97(2) EPC.

You are invited to file your observations and insofar as the deficiencies are such as to be rectifiable, to correct the indicated deficiencies within a period

of 4 months

from the notification of this communication, this period being computed in accordance with Rules 126(2) and 131(2) and (4) EPC. One set of amendments to the description, claims and drawings is to be filed within the said period on separate sheets (R. 50(1) EPC).

If filing amendments, you must identify them and indicate the basis for them in the application as filed. Failure to meet either requirement may lead to a communication from the Examining Division requesting that you correct this deficiency (R. 137(4) EPC).

Failure to comply with this invitation in due time will result in the application being deemed to be withdrawn (Art. 94(4) EPC).

The notification of this communication starts the 24-month period according to Rule 36(1)(a) EPC for filing a voluntary divisional application divided from this application or from any sequential application. For further information see Guidelines for Examination, A-IV, 1.1.1.2 and 1.1.1.4.

Important note

As from **01.04.2014** the 24-month time period under Rule 36(1)(a) EPC for filing a voluntary divisional application shall cease to apply. Thus, a divisional application may be filed while the earlier European patent application is still **pending**.

For further details, see the information published on the EPO website under www.epo.org.

Registered letter
EPO Form 2001 12.10CSX

Zanobini, Alessandra
Primary Examiner
For the Examining Division

Enclosure(s): 4 page/s reasons (Form 2906)
A copy of a machine translation of JP2004230323 has to be provided for the applicant.

CLEAN CHEMISTRY EXHIBIT 1027
IPR2025-01458 – Page 220

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Tomkinson, Alexandra
Bailey Walsh & Co
5 York Place
Leeds LS1 2SD
ROYAUME UNI

For any questions about this communication:
Tel.:+31 (0)70 340 45 00

Date
04.03.14

Reference	Application No./Patent No.
ATSAR-X11031-EP	11861339.7 - 1454 / 2688399
Applicant/Proprietor	
Enviro Tech Chemical Services Inc.	

Communication

The European search report (under R. 61 or R. 63 EPC) or the supplementary european search report (Art. 153(7) EPC) is enclosed. An official communication under Rule 71(1) or Rule 71(3) EPC will be dispatched in due course (R. 62 EPC).

Copies of documents cited in the European search report are attached.

☑ 0 additional set(s) of copies of such documents is (are) enclosed as well.

The following have been approved:

☑ Abstract ☑ Title

☐ The Abstract was modified and the definitive text is attached to this communication.

The following figure(s) will be published together with the abstract:

Refund of search fee
If applicable under Article 9 Rules relating to fees, a separate communication from the Receiving Section on the refund of the search fee will be sent later.

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Europäisches Patentamt
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SUPPLEMENTARY EUROPEAN SEARCH REPORT

Application Number
EP 11 86 1339

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X Y	EP 1 618 786 A1 (KAO CORP [JP]) 25 January 2006 (2006-01-25) * paragraphs [0001] - [0005], [0023] - [0025], [0046] - [0047] * * example 1; tables 1-5 * * example 3; table 10 * * example 4; table 11 * * example 6; tables 23, 24 *	1,2,5,6, 8,11,16, 17 4	INV. A01N37/16 A01N25/00 A61K31/075 A61L9/00 A61L2/18 A61K31/327
X	JP 2004 230323 A (MATSUSHITA ELECTRIC IND CO LTD) 19 August 2004 (2004-08-19) * abstract * * paragraphs [0001], [0008], [0011] - [0014], [0057] *	1-3,5,6, 16-18	
A	US 2010/227000 A1 (AMES KELLY ANN [GB] ET AL BOARD KELLY ANN [GB] ET AL) 9 September 2010 (2010-09-09) * paragraphs [0002] - [0006], [0012], [0016] - [0017], [0051] - [0061], [0064], [0083] - [0097] *	1-18	TECHNICAL FIELDS SEARCHED (IPC) A01N
A	US 6 221 341 B1 (MONTGOMERY R ERIC [US]) 24 April 2001 (2001-04-24) * column 3, line 55 - column 4, line 57 * * column 5, line 58 - column 7, line 16 * * column 7, line 60 - column 8, line 23 * * examples I-III, V *	1-18	

-/--

The supplementary search report has been based on the last set of claims valid and available at the start of the search.

Place of search	Date of completion of the search	Examiner
Munich	25 February 2014	Zanobini, Alessandra

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
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EPO FORM 1503 03.82 (P04C04)

CLEAN CHEMISTRY EXHIBIT 1027
IPR2025-01458 – Page 222

Europäisches Patentamt
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SUPPLEMENTARY EUROPEAN SEARCH REPORT

Application Number
EP 11 86 1339

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
Y	WO 01/46519 A1 (WARWICK INTERNAT GROUP LTD [GB]; WITHENSHAW JOHN DAVID [GB]; SKETT MAT) 28 June 2001 (2001-06-28) * page 1, line 1 - line 23 * * page 2, line 3 - line 12 * * page 3, line 6 - line 10 * * page 4, line 14 - line 28 * * page 6, line 9 - line 24 * * page 8, line 14 - line 26 * * figure 1 * -----	4	

TECHNICAL FIELDS SEARCHED (IPC)

The supplementary search report has been based on the last set of claims valid and available at the start of the search.

Place of search	Date of completion of the search	Examiner
Munich	25 February 2014	Zanobini, Alessandra

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
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1

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CLEAN CHEMISTRY EXHIBIT 1027
IPR2025-01458 – Page 223

ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO. EP 11 86 1339

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on
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25-02-2014

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 1618786 A1	25-01-2006	EP 1618786 A1	25-01-2006
		KR 20050114707 A	06-12-2005
		TW I379641 B	21-12-2012
		US 2007274857 A1	29-11-2007
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WO 0146519 A1	28-06-2001	AU 5441701 A	03-07-2001
		WO 0146519 A1	28-06-2001

EPO FORM P0459

For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

Europäisches Patentamt
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Application No.: 11861339.7

The search has started on 24.02.14 (see Notice from the European Patent Office dated 03/2013, OJ EPO 2013, 153).

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Date
07.01.14

Reference	Application No./Patent No.
ATSAR-X11031-EP	11861339.7 - 1454 / 2688399 PCT/US2011000539
Applicant/Proprietor	
Enviro Tech Chemical Services Inc.	

Communication of European publication number and information on the application of Article 67(3) EPC

The provisional protection under Article 67(1) and (2) EPC in the individual Contracting States becomes effective only when the conditions referred to in Article 67(3) EPC have been fulfilled (for further details, see information brochure of the European Patent Office "National Law relating to the EPC" and additional information in the Official Journal of the European Patent Office).

Pursuant to Article 153(3) EPC the publication under Article 21 PCT of an international application for which the European Patent Office is a designated or elected Office takes the place of the publication of a European patent application.

The bibliographic data of the above-mentioned Euro-PCT application will be published on 29.01.14 in Section I.1 of the European Patent Bulletin. The European publication number is 2688399.

In all future communications to the European Patent Office, please quote the application number plus Directorate number.

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Date
29.10.13

Reference	Application No./Patent No.
	11861339.7 - 1454 PCT/US2011000539
Applicant/Proprietor	
Enviro Tech Chemical Services Inc.	

Notification of the data mentioned in Rule 19(3) EPC

In the above-identified patent application you are designated as inventor/co-inventor.
Pursuant to Rule 19(3) EPC the following data are notified herewith:

DATE OF FILING	: 24.03.11
PRIORITY	: / /
TITLE	: METHODS AND COMPOSITIONS FOR THE GENERATION OF PERACETIC ACID ON SITE AT THE POINT-OF-USE
DESIGNATED STATES	: AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO PL PT RO RS SE SI SK SM TR

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40 EUROPEAN PATENT CONVENTION
YEARS 1973-2013
Innovation matters
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Date
29.10.13

Reference	Application No./Patent No.
	11861339.7 - 1454 PCT/US2011000539
Applicant/Proprietor	
Enviro Tech Chemical Services Inc.	

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Date
29.10.13

Reference	Application No./Patent No.
	11861339.7 - 1454 PCT/US2011000539
Applicant/Proprietor	
Enviro Tech Chemical Services Inc.	

Notification of the data mentioned in Rule 19(3) EPC

In the above-identified patent application you are designated as inventor/co-inventor.
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DATE OF FILING	: 24.03.11
PRIORITY	: / /
TITLE	: METHODS AND COMPOSITIONS FOR THE GENERATION OF PERACETIC ACID ON SITE AT THE POINT-OF-USE
DESIGNATED STATES	: AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO PL PT RO RS SE SI SK SM TR

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Date: 14.10.13

Reference	Application No./Patent No.
ATSAR-X11031-EP	11861339.7 - 1454 PCT/US2011000539
Applicant/Proprietor: Enviro Tech Chemical Services Inc.	

Refund of fees

The following fee(s) was/were paid in respect of the application 11861339.7:

Fee	Reference	Voucher No	Date	Currency	Amount
Claims fee	015	00976921	06.09.13	EUR	1 800,00

According to the present state of the file the refund will be made by:

CREDITING THE DEPOSIT ACCOUNT 28050202 Bailey Walsh & Co..

Amount refundable:	Reference	Currency	Amount	Voucher No
	015	EUR	1.125,00	01033644

Reason for refund: Overpayment

The Authorising Officer
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PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY
(Chapter I of the Patent Cooperation Treaty)

(PCT Rule 44*bis*)

Applicant's or agent's file reference 8568/17669P	**FOR FURTHER ACTION** See item 4 below	
International application No. PCT/US2011/000539	International filing date *(day/month/year)* 24 March 2011 (24.03.2011)	Priority date *(day/month/year)*
International Patent Classification (8th edition unless older edition indicated) See relevant information in Form PCT/ISA/237		
Applicant ENVIRO TECH CHEMICAL SERVICES, INC.		

1. This international preliminary report on patentability (Chapter I) is issued by the International Bureau on behalf of the International Searching Authority under Rule 44 *bis*.1(a).

2. This REPORT consists of a total of 12 sheets, including this cover sheet.

   In the attached sheets, any reference to the written opinion of the International Searching Authority should be read as a reference to the international preliminary report on patentability (Chapter I) instead.

3. This report contains indications relating to the following items:

☒	Box No. I	Basis of the report
☐	Box No. II	Priority
☐	Box No. III	Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
☐	Box No. IV	Lack of unity of invention
☒	Box No. V	Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
☐	Box No. VI	Certain documents cited
☐	Box No. VII	Certain defects in the international application
☐	Box No. VIII	Certain observations on the international application

4. The International Bureau will communicate this report to designated Offices in accordance with Rules 44*bis*.3(c) and 93*bis*.1 but not, except where the applicant makes an express request under Article 23(2), before the expiration of 30 months from the priority date (Rule 44*bis* .2).

	Date of issuance of this report 24 September 2013 (24.09.2013)
The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No. +41 22 338 82 70	Authorized officer Athina Nickitas-Etienne e-mail: pt04.pct@wipo.int

Form PCT/IB/373 (January 2004)

PATENT COOPERATION TREATY

From the
INTERNATIONAL SEARCHING AUTHORITY

To: AUDREY A. MILLEMANN
WEINTRAUB GENSHLEA CHEDIAK LAW CORPORATION
400 CAPITOL MALL, 11TH FLOOR
SACRAMENTO, CA 95814

PCT

WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY

(PCT Rule 43*bis*.1)

Date of mailing *(day/month/year)*	03 JUN 2011
Applicant's or agent's file reference 8568/17669P	**FOR FURTHER ACTION** See paragraph 2 below

International application No.	International filing date *(day/month/year)*	Priority date *(day/month/year)*
PCT/US 11/00539	24 March 2011 (24.03.2011)	24 March 2011 (24.03.2011)

International Patent Classification (IPC) or both national classification and IPC
IPC(8) - A01N 37/16; A01N 25/00; A61K 31/075; A61L 9/00 (2011.01)
USPC - 424/405; 422/28; 422/29; 514/714

Applicant ENVIRO TECH CHEMICAL SERVICES, INC.

1. This opinion contains indications relating to the following items:

- ☒ Box No. I Basis of the opinion
- ☐ Box No. II Priority
- ☐ Box No. III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- ☐ Box No. IV Lack of unity of invention
- ☒ Box No. V Reasoned statement under Rule 43*bis*.1(a)(i) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- ☐ Box No. VI Certain documents cited
- ☐ Box No. VII Certain defects in the international application
- ☐ Box No. VIII Certain observations on the international application

2. **FURTHER ACTION**

If a demand for international preliminary examination is made, this opinion will be considered to be a written opinion of the International Preliminary Examining Authority ("IPEA") except that this does not apply where the applicant chooses an Authority other than this one to be the IPEA and the chosen IPEA has notified the International Bureau under Rule 66.1*bis*(b) that written opinions of this International Searching Authority will not be so considered.

If this opinion is, as provided above, considered to be a written opinion of the IPEA, the applicant is invited to submit to the IPEA a written reply together, where appropriate, with amendments, before the expiration of 3 months from the date of mailing of Form PCT/ISA/220 or before the expiration of 22 months from the priority date, whichever expires later.

For further options, see Form PCT/ISA/220.

3. For further details, see notes to Form PCT/ISA/220.

Name and mailing address of the ISA/US	Date of completion of this opinion	Authorized officer:
Mail Stop PCT, Attn: ISA/US Commissioner for Patents P.O. Box 1450, Alexandria, Virginia 22313-1450 Facsimile No. 571-273-3201	16 May 2011 (16.05.2011)	Lee W. Young PCT Helpdesk: 571-272-4300 PCT OSP: 571-272-7774

Form PCT/ISA/237 (cover sheet) (July 2009)

WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY

International application No.
PCT/US 11/00539

Box No. I Basis of this opinion

1. With regard to the **language**, this opinion has been established on the basis of:

   ☒ the international application in the language in which it was filed.

   ☐ a translation of the international application into _____ which is the language of a translation furnished for the purposes of international search (Rules 12.3(a) and 23.1(b)).

2. ☐ This opinion has been established taking into account the **rectification of an obvious mistake** authorized by or notified to this Authority under Rule 91 (Rule 43*bis*.1(a))

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, this opinion has been established on the basis of a sequence listing filed or furnished:

   a. (means)

      ☐ on paper

      ☐ in electronic form

   b. (time)

      ☐ in the international application as filed

      ☐ together with the international application in electronic form

      ☐ subsequently to this Authority for the purposes of search

4. ☐ In addition, in the case that more than one version or copy of a sequence listing has been filed or furnished, the required statements that the information in the subsequent or additional copies is identical to that in the application as filed or does not go beyond the application as filed, as appropriate, were furnished.

5. Additional comments:

WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY

International application No.
PCT/US 11/00539

Box No. V **Reasoned statement under Rule 43*bis*.1(a)(i) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

1. Statement

Novelty (N)	Claims	7-77, 81	YES
	Claims	1-6, 78-80, 82-91	NO
Inventive step (IS)	Claims	NONE	YES
	Claims	1-91	NO
Industrial applicability (IA)	Claims	1-91	YES
	Claims	NONE	NO

2. Citations and explanations:

Claims 1-6, 78-80, and 82-91 lack novelty under PCT Article 33(2) as being anticipated by US 2010/0227000 A1 to Ames et al. (hereinafter "Ames").

Regarding claim 1; Ames discloses a hydrogen peroxide-acetyl precursor solution (para [0007] and [0067]), comprising: (a) aqueous hydrogen peroxide (para [0007] and [0067], with para [0051]); (b) a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide (para [0007] and [0067], with para [0015]); (c) a trace amount of peracetic acid (para [0067]); and (d) water (para [0067]).

Regarding claim 2; claim 1 is anticipated as above. Furthermore, Ames discloses wherein said liquid acetyl precursor is triacetin (para [0007], with para [0015]).

Regarding claim 3; claim 2 is anticipated as above. Furthermore, Ames discloses wherein said aqueous hydrogen peroxide is about 23% to about 40% (para [0013]) and said triacetin is about 20% to about 53% (para [0013]), and further wherein the mole ratio of said hydrogen peroxide to said triacetin is about 2.98: 1 to about 12.84: 1 (para [0013]).

Regarding claim 4; claim 3 is anticipated as above. Furthermore, Ames discloses wherein said aqueous hydrogen peroxide is about 27% (para [0013]) and said triacetin is about 46% (para [0013]), and further wherein said mole ratio of said hydrogen peroxide to said triacetin is about 3.8: 1 (para [0013]).

Regarding claim 5; Ames discloses a method of preparing a hydrogen peroxide-acetyl precursor solution (para [0007] and [0067]), comprising:
(a) introducing a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide (para [0007] and [0067], with para [0015]) to a solution of aqueous hydrogen peroxide (para [0007] and [0067], with para [0051]); and (b) allowing the liquid acetyl precursor and the solution of aqueous hydrogen peroxide to mix to form a hydrogen peroxide-acetyl precursor solution (para [0067]).

Regarding claim 6; claim 5 is anticipated as above. Furthermore, Ames discloses wherein the liquid acetyl precursor is triacetin (para [0007], with para [0015]).

Regarding claim 78; Ames discloses a freely-flowable solid composition, comprising: (a) a liquid acetyl precursor (para [0007], "part A which comprises at least one acetyl donor", with para [0015], "part A... may comprise liquid constituents"); (b) a water-soluble solid source of hydrogen peroxide (para [0007], "a liquid part B in the form of an aqueous composition which comprises... hydrogen peroxide"); and (c) a water-soluble solid source of alkalinity (para [0019], "part A may comprise... inorganic bases," with para [0022], "sodium hydroxide").

Regarding claim 79; claim 78 is anticipated as above. Furthermore, Ames discloses wherein said liquid acetyl precursor is triacetin (para [0007], with para [0015]).

Regarding claim 80; claim 79 is anticipated as above. Furthermore, Ames discloses wherein said triacetin is about 0.99% to about 8.45% (para [0013]).

Regarding claim 82; claim 78 is anticipated as above. Furthermore, Ames discloses wherein said solid source of alkalinity is about 24% to about 44% of the total (para [0013] and [0022]).

Regarding claim 83; Ames discloses a method of making a solid composition for bleaching and stain removal (para [0063]), comprising: forming a homogenous freely-flowable solid (para [0063]) by blending a liquid acetyl precursor (para [0007], "part A which comprises at least one acetyl donor", with para [0015], "part A... may comprise liquid constituents"), a water-soluble solid source of hydrogen peroxide (para [0007], "a liquid part B in the form of an aqueous composition which comprises... hydrogen peroxide"), and a water-soluble solid source of alkalinity (para [0019], "part A may comprise... inorganic bases," with para [0022], "sodium hydroxide").

Regarding claim 84; claim 83 is anticipated as above. Furthermore, Ames discloses wherein said liquid acetyl precursor is triacetin (para [0007], with para [0015]).

---------- continued in Supplemental Box ----------

WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY

International application No.
PCT/US 11/00539

Supplemental Box

In case **the space in any of the preceding boxes is not sufficient.**
Continuation of:
Box V.2. Citations and explanations:

Regarding claim 85; claim 84 is anticipated as above. Furthermore, Ames discloses wherein said triacetin is about 0.99% to about 8.45% (para [0013]).

Regarding claim 86; claim 83 is anticipated as above. Furthermore, Ames discloses wherein the solid source of hydrogen peroxide is about 13% to about 54% (para [0013]).

Regarding claim 87; claim 83 is anticipated as above. Furthermore, Ames discloses wherein the solid source of alkalinity is about 24% to about 44% of the total (para [0013] and [0022]).

Regarding claim 88; claim 83 is anticipated as above. Furthermore, Ames discloses wherein the method further comprises adding a metal chelating agent (para [0024]).

Regarding claim 89; claim 83 is anticipated as above. Furthermore, Ames discloses wherein the method further comprises adding a surfactant (para [0007]).

Regarding claim 90; claim 83 is anticipated as above. Furthermore, Ames discloses wherein the method further comprises adding a compound to sequester water hardness (para [0024], disclosing inclusion of sequestering agents such as EDTA and DTPA).

Regarding claim 91; claim 83 is anticipated as above. Furthermore, Ames discloses wherein the method further comprises adding an inert solid filler (para [0021]).

Claim 81 lacks an inventive step under PCT Article 33(3) as being obvious over Ames.

Regarding claim 81; claim 78 is anticipated as above. Furthermore, Ames teaches wherein said solid source of hydrogen peroxide is about 13% to about 54% of the total (para [0013]). Ames fails to expressly teach wherein said solid source of hydrogen peroxide is sodium percarbonate. However, Ames does teach inorganic percarbonate salts as being a known and routine source of hydrogen peroxide for in-situ generation of peracetic acid for use by those with ordinary skill in the art; such that one with ordinary skill in the art could confirm without undue experimentation the effectiveness of using inorganic percarbonates, such as sodium percarbonate, as the hydrogen peroxide component taught in Ames.

Claims 7-10, 13-20, 23-29, 32-39, 42-56, 59-65, and 68-77 lack an inventive step under PCT Article 33(3) as being obvious over Ames in view of US 2009/0175956 A1 to Buschmann et al. (hereinafter "Buschmann").

Regarding claim 7;
- Ames teaches a method of generating a solution of peracetic acid (para [0063] and [0067]), comprising: (a) providing water (para [0063] and [0067]); (b) introducing a hydrogen peroxide-acetyl precursor solution to the water (para [0063] and [0067]); and (c) mixing the hydrogen peroxide-acetyl precursor solution and the water to form a mixture (para [0063] and [0067]).
- Ames fails to expressly teach wherein the method further comprises: (d) adding an aqueous source of an alkali metal or earth alkali metal hydroxide to the mixture. However, Ames and Buschmann teach this claim limitation as being obvious to one with ordinary skill in the art. Specifically, Ames teaches the inclusion alkali metal hydroxides, such as sodium hydroxide, within the initial mixture composition (para [0019] and [0051], with para [0022] and [0053]). Ames also specifically teaches the use of the alkali metal hydroxide component to raise the pH level of the composition (para [0053]). Ames fails to teach wherein the alkali metal hydroxide is added separately and after the other components. Buschmann teaches a similar method and composition for generating a non-equilibrium peracetic acid composition (para [0027] and [0033]) which comprises combining hydrogen peroxide and an acetyl precursor, such as triacetin, with water (para [0027] and [0033]), along with a pH-adjusting alkali earth metal hydroxide component, such as sodium hydroxide (para [0033]-[0034]). Buschmann also teaches that peractetic acid solutions are much more stable for "stabilization and storage" at acidic or neutral pH levels of 7.0 or below (para [0037] and [0042]), but also teaches that a "caustic pH" above 10 will accelerate the production of peractetic acid because "the hydrogen peroxide anion is a much better nucleophile than hydrogen peroxide" (para [0033]). The teachings in Buschmann indicate that a peracetic acid composition, such as one comprising hydrogen peroxide reacted with an acetyl donor, can be stabilized in a slow-reacting and non-decomposing state by maintaining a low pH in the composition, wherein the peracetic acid production in the solution can then be significantly accelerated when desired by a drastic increase in pH, such as by the addition of strong bases such as sodium hydroxide. In light of Buschmann, one with ordinary skill in the art could confirm without undue experimentation the effectiveness of adding the aqueous source of an alkali metal hydroxide in a separate and later step than the hydrogen peroxide-acetyl precursor solution.
- Buschmann also teaches wherein the method further comprises: (e) forming a reaction medium comprising a non-equilibrium solution of peracetic acid (para [0033]).
- It would have been obvious to one with ordinary skill in the art to combine the components taught in Buschmann with the method and composition taught in Ames because both references relate to methods and compositions for generating peracetic acid which comprise combining hydrogen peroxide and an acetyl precursor, such as triacetin, with water, along with a pH-adjusting alkali earth metal hydroxide component, such as sodium hydroxide; wherein Buschmann adds specificity to the method and composition by teaching additional components and specific concentrations which can be used to optimize the method and composition taught in Ames. The additional specificity taught in Buschmann improves the applicability, effectiveness, efficiency and marketability of the method and composition taught in Ames.

---------- continued in next Supplemental Box ----------

WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY

International application No. PCT/US 11/00539

Supplemental Box

In case **the space in any of the preceding boxes is not sufficient.**
Continuation of:
Previous Supplemental Box

Regarding claim 8; claim 7 is obvious as above. Furthermore, Ames teaches wherein the method further comprises, prior to step (b), a step of providing the hydrogen peroxide-acetyl precursor solution (para [0007] and [0067]) comprising: (a) introducing a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide (para [0007] and [0067], with para [0015]) to a solution of aqueous hydrogen peroxide (para [0007] and [0067], with para [0051]); and (b) allowing the liquid acetyl precursor and the solution of aqueous hydrogen peroxide to mix to form a hydrogen peroxide-acetyl precursor solution (para [0067]).

Regarding claim 9; claim 8 is obvious as above. Furthermore, Ames teaches wherein the liquid acetyl precursor is triacetin defining a hydrogen peroxide-triacetin solution (para [0007], with para [0015]).

Regarding claim 10; claim 9 is obvious as above. Furthermore, Ames teaches wherein the hydrogen peroxide-triacetin solution has a mole ratio of hydrogen peroxide to triacetin of about 2.98:1 to about 12.84:1 (para [0013]).

Regarding claim 13; claim 9 is obvious as above. Furthermore, Ames teaches wherein the aqueous source of the alkali metal or earth alkali metal hydroxide is sodium hydroxide (para [0053]).

Regarding claim 14; claim 13 is obvious as above. Furthermore, Ames teaches wherein the sodium hydroxide is about 1.82% to about 7.28% (para [0013]).

Regarding claim 15; claim 7 is obvious as above. Furthermore, Ames teaches wherein step (d) is performed simultaneously with step (b) (para [0022], teaching wherein the alkali metal hydroxide is included in the same mixture component as the acetyl donor).

Regarding claims 16 and 17; claim 7 is obvious as above. Furthermore, Ames teaches wherein the method further comprises allowing the reaction medium sufficient time to maximize the conversion of the hydrogen peroxide and the acetyl precursor into peracetic acid; more specifically wherein about 30 seconds to about five minutes (para [0064]).

Regarding claim 18; claim 17 is obvious as above. Ames and Buschmann fail to expressly teach wherein the method further comprises, after step (e), a step of sampling the reaction medium to determine the time required to maximize the amount of hydrogen peroxide and acetyl precursor that are converted into peracetic acid. However, Ames teaches that activation time of the peracetic acid solution can depend on the temperature of the solution (para [0064]). Buschmann further teaches the the rate of peracetic acid production is dependent on the pH of the solution (para [0039] and [0042]). Combining the teachings in Ames and Buschmann would indicate that optimal reaction times could be determined by measuring various rate-controlling solution properties, such as temperature and pH; such that one with ordinary skill in the art could confirm without undue experimentation the effectiveness of including an additional sampling step measure solution properties and determine the optimal time required to maximize the amount of hydrogen peroxide and acetyl precursor that are converted into peracetic acid.

Regarding claim 19; claim 16 is obvious as above. Furthermore, Buschmann teaches wherein the acetyl precursor is triacetin (para [0027] and [0030]), and the percent of triacetin that is converted into peracetic acid is about 40.9% to about 85.7% (para [0035]).

Regarding claim 20; claim 14 is obvious as above. Furthermore, Ames teaches wherein the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is about 4.2:3.8:1 (para [0013]).

Regarding claim 23; claim 7 is obvious as above. Furthermore, Ames teaches wherein the peracetic acid solution is for immediate use (para [0064]).

Regarding claim 24; claim 7 is obvious as above. Furthermore, Buschmann teaches wherein the method further comprises, after step (e), a step of adding a source of acid to stabilize the non-equilibrium solution of peracetic acid for storing and using throughout a working day (para [0042]).

Regarding claim 25; claim 7 is obvious as above. Furthermore, Ames teaches wherein the peracetic acid solution is prepared on a site having a point-of-use (para [0064] and [0067]).

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Regarding claim 26;
- Ames teaches a method of generating a solution of peracetic acid for use on site having a point-of-use of peracetic acid (para [0063]-[0064] and [0067]), comprising: (a) providing water on a site (para [0063]-[0064] and [0067]); (b) providing a hydrogen peroxide-acetyl precursor solution on the site having a point-of-use (para [0064] and [0067]);
(c) introducing the hydrogen peroxide-acetyl precursor solution to the water (para [0063] and [0067]); and (d) mixing the hydrogen peroxide -acetyl precursor solution and the water to form a mixture (para [0063] and [0067]).
- Ames fails to expressly teach wherein the method further comprises: (e) providing an aqueous source of an alkali metal or earth alkali metal hydroxide on the site having the point-of-use; and (f) adding the aqueous source of an alkali metal or earth alkali metal hydroxide to the mixture. However, Ames and Buschmann teach this claim limitation as being obvious to one with ordinary skill in the art. Specifically, Ames teaches the inclusion alkali metal hydroxides, such as sodium hydroxide, within the initial mixture composition (para [0019] and [0051], with para [0022] and [0053]). Ames also specifically teaches the use of the alkali metal hydroxide component to raise the pH level of the composition (para [0053]). Ames fails to teach wherein the alkali metal hydroxide is added separately and after the other components. Buschmann teaches a similar method and composition for generating a non-equilibrium peracetic acid composition (para [0027] and [0033]) which comprises combining hydrogen peroxide and an acetyl precursor, such as triacetin, with water (para [0027] and [0033]), along with a pH-adjusting alkali earth metal hydroxide component, such as sodium hydroxide (para [0033]-[0034]). Buschmann also teaches that peractetic acid solutions are much more stable for "stabilization and storage" at acidic or neutral pH levels of 7.0 or below (para [0037] and [0042]), but also teaches that a "caustic pH" above 10 will accelerate the production of peractetic acid because "the hydrogen peroxide anion is a much better nucleophile than hydrogen peroxide" (para [0033]). The teachings in Buschmann indicate that a peracetic acid composition, such as one comprising hydrogen peroxide reacted with an acetyl donor, can be stabilized in a slow-reacting and non-decomposing state by maintaining a low pH in the composition, wherein the peracetic acid production in the solution can then be significantly accelerated when desired by a drastic increase in pH, such as by the addition of strong bases such as sodium hydroxide. In light of Buschmann, one with ordinary skill in the art could confirm without undue experimentation the effectiveness of adding the aqueous source of an alkali metal hydroxide in a separate and later step than the hydrogen peroxide-acetyl precursor solution.
- Buschmann also teaches wherein the method further comprises: (g) forming a reaction medium comprising a non-equilibrium solution of peracetic acid for use at the point-of-use of peracetic acid (para [0033]).
- It would have been obvious to one with ordinary skill in the art to combine the components taught in Buschmann with the method and composition taught in Ames because both references relate to methods and compositions for generating peracetic acid which comprise combining hydrogen peroxide and an acetyl precursor, such as triacetin, with water, along with a pH-adjusting alkali earth metal hydroxide component, such as sodium hydroxide; wherein Buschmann adds specificity to the method and composition by teaching additional components and specific concentrations which can be used to optimize the method and composition taught in Ames. The additional specificity taught in Buschmann improves the applicability, effectiveness, efficiency and marketability of the method and composition taught in Ames.

Regarding claim 27; claim 26 is obvious as above. Furthermore, Ames teaches wherein the method further comprises, prior to step (b), a step of providing the hydrogen peroxide-acetyl precursor solution (para [0007] and [0067]) comprising: (a) introducing a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide (para [0007] and [0067], with para [0015]) to a solution of aqueous hydrogen peroxide (para [0007] and [0067], with para [0051]); and (b) allowing the liquid acetyl precursor and the solution of aqueous hydrogen peroxide to mix to form a hydrogen peroxide-acetyl precursor solution (para [0067]).

Regarding claim 28; claim 8 is obvious as above. Furthermore, Ames teaches wherein the liquid acetyl precursor is triacetin defining a hydrogen peroxide-triacetin solution (para [0007], with para [0015]).

Regarding claim 29; claim 28 is obvious as above. Furthermore, Ames teaches wherein the hydrogen peroxide-triacetin solution has a mole ratio of hydrogen peroxide to triacetin of about 2.98:1 to about 12.84:1 (para [0013]).

Regarding claim 32; claim 28 is obvious as above. Furthermore, Ames teaches wherein the aqueous source of the alkali metal or earth alkali metal hydroxide is sodium hydroxide (para [0053]).

Regarding claim 33; claim 32 is obvious as above. Furthermore, Ames teaches wherein the sodium hydroxide is about 1.82% to about 7.28% (para [0013]).

Regarding claim 34; claim 26 is obvious as above. Furthermore, Ames teaches wherein step (d) is performed simultaneously with step (b) (para [0066]).

Regarding claims 35 and 36; claim 26 is obvious as above. Furthermore, Ames teaches wherein the method further comprises allowing the reaction medium sufficient time to maximize the conversion of the hydrogen peroxide and the acetyl precursor into peracetic acid; more specifically wherein about 30 seconds to about five minutes (para [0064]).

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Regarding claim 37; claim 36 is obvious as above. Ames and Buschmann fail to expressly teach wherein the method further comprises, after step (e), a step of sampling the reaction medium to determine the time required to maximize the amount of hydrogen peroxide and acetyl precursor that are converted into peracetic acid. However, Ames teaches that activation time of the peracetic acid solution can depend on the temperature of the solution (para [0064]). Buschmann further teaches the the rate of peracetic acid production is dependent on the pH of the solution (para [0039] and [0042]). Combining the teachings in Ames and Buschmann would indicate that optimal reaction times could be determined by measuring various rate-controlling solution properties, such as temperature and pH; such that one with ordinary skill in the art could confirm without undue experimentation the effectiveness of including an additional sampling step measure solution properties and determine the optimal time required to maximize the amount of hydrogen peroxide and acetyl precursor that are converted into peracetic acid.

Regarding claim 38; claim 35 is obvious as above. Furthermore, Buschmann teaches wherein the acetyl precursor is triacetin (para [0027] and [0030]), and the percent of triacetin that is converted into peracetic acid is about 40.9% to about 85.7% (para [0035]).

Regarding claim 39; claim 33 is obvious as above. Furthermore, Ames teaches wherein the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is about 4.2:3.8:1 (para [0013]).

Regarding claim 42; claim 26 is obvious as above. Furthermore, Ames teaches wherein the peracetic acid solution is for immediate use (para [0064]).

Regarding claim 43; claim 26 is obvious as above. Furthermore, Buschmann teaches wherein the method further comprises, after step (e), a step of adding a source of acid to stabilize the non-equilibrium solution of peracetic acid for storing and using throughout a working day (para [0042]).

Regarding claim 44; claim 26 is obvious as above. Furthermore, Ames teaches wherein the peracetic acid solution is prepared on a site having a point-of-use (para [0064] and [0067])

Regarding claim 45; claim 7 is obvious as above. Furthermore, Buschmann teaches wherein the peracetic acid made by the method of claim 7 is in a non-equilibrium solution for use as a disinfectant or sanitizer (para [0023]).

Regarding claim 46; claim 45 is obvious as above. Furthermore, Ames teaches wherein the solution is for clean-in-place pipeline and equipment cleaning (para [0069]).

Regarding claim 47; claim 45 is obvious as above. Furthermore, Buschmann teaches wherein the solution is for use in washing fruits and vegetables (para [0031]).

Regarding claim 48; claim 45 is obvious as above. Furthermore, Buschmann teaches wherein the solution is for use in the treatment of meat, poultry, and seafood (para [0031]).

Regarding claim 49;
- Ames teaches a method for sanitizing a surface (para [0069]), which comprises generating a solution of peracetic acid (para [0007] and [0067]) comprising: (a) peracetic acid (para [0007] and [0067]), (b) hydrogen peroxide (para [0007] and [0067]), (c) triacetin (para [0007] and [0067], with para [0017]), (e) a source of an alkali metal or earth alkali metal hydroxide (para [0007], [0051] and [0053]), and (f) water (para [0007] and [0067]).
- Ames fails to expressly teach further claim limitations which are taught by Buschmann, including wherein the solution further comprises (d) 1,2,3-propanetriol (para [0023] and [0030], "glycerol"); and wherien the solution is a non-equilibrium solution (para [0023]).
- It would have been obvious to one with ordinary skill in the art to combine the components taught in Buschmann with the method and composition taught in Ames because both references relate to methods and compositions for generating peracetic acid which comprise combining hydrogen peroxide and an acetyl precursor, such as triacetin, with water, along with a pH-adjusting alkali earth metal hydroxide component, such as sodium hydroxide; wherein Buschmann adds specificity to the method and composition by teaching additional components and specific concentrations which can be used to optimize the method and composition taught in Ames. The additional specificity taught in Buschmann improves the applicability, effectiveness, efficiency and marketability of the method and composition taught in Ames.

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Regarding claim 50;
- Ames teaches a solution of peracetic acid (para [0007] and [0067]) comprising: (a) peracetic acid (para [0007] and [0067]), (b) hydrogen peroxide (para [0007] and [0067]), (c) triacetin (para [0007] and [0067], with para [0017]), (e) an aqueous source of an alkali metal or earth alkali metal hydroxide (para [0007], [0051] and [0053]), and (f) water (para [0007] and [0067]).
- Ames fails to expressly teach further claim limitations which are taught by Buschmann, including wherein the solution further comprises (d) 1,2,3-propanetriol (para [0023] and [0030], "glycerol"); and wherien the solution is a non-equilibrium solution (para [0023]).
- It would have been obvious to one with ordinary skill in the art to combine the components taught in Buschmann with the method and composition taught in Ames because both references relate to methods and compositions for generating peracetic acid which comprise combining hydrogen peroxide and an acetyl precursor, such as triacetin, with water, along with a pH-adjusting alkali earth metal hydroxide component, such as sodium hydroxide; wherein Buschmann adds specificity to the method and composition by teaching additional components and specific concentrations which can be used to optimize the method and composition taught in Ames. The additional specificity taught in Buschmann improves the applicability, effectiveness, efficiency and marketability of the method and composition taught in Ames.

Regarding claim 51; claim 50 is obvious as above. Furthermore, Buschmann teaches wherein said solution has a pH of about 11.2 to about 13.37 (para [0034]).

Regarding claim 52; claim 50 is obvious as above. Furthermore, Ames teaches wherein said peracetic acid is about 1 % to about 7.1 % (para [0067]).

Regarding claim 53;
- Ames teaches a method of generating a solution of peracetic acid (para [0063] and [0067]), comprising: (a) providing water (para [0063] and [0067]); (b) introducing a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide (para [0007] and [0067], with para [0017]) and aqueous hydrogen peroxide (para [0007] and [0067]) to the water (para [0063] and [0067]); and (c) mixing the liquid acetyl precursor and the aqueous hydrogen peroxide with the water to form a mixture (para [0063] and [0067]).
- Ames fails to expressly teach wherein the method further comprises: (d) adding an aqueous source of an alkali metal or earth alkali metal hydroxide to the mixture. However, Ames and Buschmann teach this claim limitation as being obvious to one with ordinary skill in the art. Specifically, Ames teaches the inclusion alkali metal hydroxides, such as sodium hydroxide, within the initial mixture composition (para [0019] and [0051], with para [0022] and [0053]). Ames also specifically teaches the use of the alkali metal hydroxide component to raise the pH level of the composition (para [0053]). Ames fails to teach wherein the alkali metal hydroxide is added separately and after the other components. Buschmann teaches a similar method and composition for generating a non-equilibrium peracetic acid composition (para [0027] and [0033]) which comprises combining hydrogen peroxide and an acetyl precursor, such as triacetin, with water (para [0027] and [0033]), along with a pH-adjusting alkali earth metal hydroxide component, such as sodium hydroxide (para [0033]-[0034]). Buschmann also teaches that peractetic acid solutions are much more stable for "stabilization and storage" at acidic or neutral pH levels of 7.0 or below (para [0037] and [0042]), but also teaches that a "caustic pH" above 10 will accelerate the production of peractetic acid because "the hydrogen peroxide anion is a much better nucleophile than hydrogen peroxide" (para [0033]). The teachings in Buschmann indicate that a peracetic acid composition, such as one comprising hydrogen peroxide reacted with an acetyl donor, can be stabilized in a slow-reacting and non-decomposing state by maintaining a low pH in the composition, wherein the peracetic acid production in the solution can then be significantly accelerated when desired by a drastic increase in pH, such as by the addition of strong bases such as sodium hydroxide. In light of Buschmann, one with ordinary skill in the art could confirm without undue experimentation the effectiveness of adding the aqueous source of an alkali metal hydroxide in a separate and later step than the hydrogen peroxide-acetyl precursor solution.
- Buschmann also teaches wherein the method further comprises: (e) forming a reaction medium comprising a non-equilibrium solution of peracetic acid (para [0033]).
- It would have been obvious to one with ordinary skill in the art to combine the components taught in Buschmann with the method and composition taught in Ames because both references relate to methods and compositions for generating peracetic acid which comprise combining hydrogen peroxide and an acetyl precursor, such as triacetin, with water, along with a pH-adjusting alkali earth metal hydroxide component, such as sodium hydroxide; wherein Buschmann adds specificity to the method and composition by teaching additional components and specific concentrations which can be used to optimize the method and composition taught in Ames. The additional specificity taught in Buschmann improves the applicability, effectiveness, efficiency and marketability of the method and composition taught in Ames.

Regarding claim 54; claim 53 is obvious as above. Furthermore, Ames teaches wherein the liquid acetyl precursor is triacetin defining a hydrogen peroxide-triacetin solution (para [0007], with para [0015]).

Regarding claims 55 and 56; claim 53 is obvious as above. Ames and Buschmann fail to expressly teach wherein the liquid acetyl precursor and the aqueous hydrogen peroxide are introduced simultaneously, or introduced sequentially with either one first. However, combining solution components simultaneously or sequentially are both known and routine methods of forming a mixture of components to those with ordinary skill in the art; wherein one with ordinary skill in the art could confirm without undue experimentation the effectiveness of introducing the components taught in Ames and Buschmann either simultaneously or sequentially.

Regarding claim 59; claim 54 is obvious as above. Furthermore, Ames teaches wherein the aqueous source of the alkali metal or earth alkali metal hydroxide is sodium hydroxide (para [0053]).

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Regarding claim 60; claim 59 is obvious as above. Furthermore, Ames teaches wherein the sodium hydroxide is about 1.82% to about 7.28% (para [0013]).

Regarding claim 61; claim 53 is obvious as above. Furthermore, Ames teaches wherein step (d) is performed simultaneously with step (b) (para [0022], teaching wherein the alkali metal hydroxide is included in the same mixture component as the acetyl donor).

Regarding claims 62 and 63; claim 53 is obvious as above. Furthermore, Ames teaches wherein the method further comprises allowing the reaction medium sufficient time to maximize the conversion of the hydrogen peroxide and the acetyl precursor into peracetic acid; more specifically wherein about 30 seconds to about five minutes (para [0064]).

Regarding claim 64; claim 63 is obvious as above. Furthermore, Buschmann teaches wherein the acetyl precursor is triacetin (para [0027] and [0030]), and the percent of triacetin that is converted into peracetic acid is about 40.9% to about 85.7% (para [0035]).

Regarding claim 65; claim 60 is obvious as above. Furthermore, Ames teaches wherein the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is about 4.2:3.8:1 (para [0013]).

Regarding claim 68;
- Ames teaches a method of generating a solution of peracetic acid on a site having a point-of-use (para [0063]-[0064] and [0067]), comprising: (a) providing water on a site (para [0063]-[0064] and [0067]); (b) providing a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide (para [0007] and [0067], with para [0017]) and aqueous hydrogen peroxide (para [0007] and [0067]) on the site having a point-of-use (para [0064] and [0067]); (c) introducing the liquid acetyl precursor and the aqueous hydrogen peroxide to the water (para [0063] and [0067]); and (d) mixing the aqueous hydrogen peroxide and the liquid acetyl precursor solution with the water to form a mixture (para [0063] and [0067]).
- Ames fails to expressly teach wherein the method further comprises: (e) providing an aqueous source of an alkali metal or earth alkali metal hydroxide on the site having the point-of-use; and (f) adding the aqueous source of an alkali metal or earth alkali metal hydroxide to the mixture. However, Ames and Buschmann teach this claim limitation as being obvious to one with ordinary skill in the art. Specifically, Ames teaches the inclusion alkali metal hydroxides, such as sodium hydroxide, within the initial mixture composition (para [0019] and [0051], with para [0022] and [0053]). Ames also specifically teaches the use of the alkali metal hydroxide component to raise the pH level of the composition (para [0053]). Ames fails to teach wherein the alkali metal hydroxide is added separately and after the other components. Buschmann teaches a similar method and composition for generating a non-equilibrium peracetic acid composition (para [0027] and [0033]) which comprises combining hydrogen peroxide and an acetyl precursor, such as triacetin, with water (para [0027] and [0033]), along with a pH-adjusting alkali earth metal hydroxide component, such as sodium hydroxide (para [0033]-[0034]). Buschmann also teaches that peractetic acid solutions are much more stable for "stabilization and storage" at acidic or neutral pH levels of 7.0 or below (para [0037] and [0042]), but also teaches that a "caustic pH" above 10 will accelerate the production of peractetic acid because "the hydrogen peroxide anion is a much better nucleophile than hydrogen peroxide" (para [0033]). The teachings in Buschmann indicate that a peracetic acid composition, such as one comprising hydrogen peroxide reacted with an acetyl donor, can be stabilized in a slow-reacting and non-decomposing state by maintaining a low pH in the composition, wherein the peracetic acid production in the solution can then be significantly accelerated when desired by a drastic increase in pH, such as by the addition of strong bases such as sodium hydroxide. In light of Buschmann, one with ordinary skill in the art could confirm without undue experimentation the effectiveness of adding the aqueous source of an alkali metal hydroxide in a separate and later step than the hydrogen peroxide-acetyl precursor solution.
- Buschmann also teaches wherein the method further comprises: (g) forming a reaction medium comprising a non-equilibrium solution of peracetic acid for use at the point-of-use (para [0033]).
- It would have been obvious to one with ordinary skill in the art to combine the components taught in Buschmann with the method and composition taught in Ames because both references relate to methods and compositions for generating peracetic acid which comprise combining hydrogen peroxide and an acetyl precursor, such as triacetin, with water, along with a pH-adjusting alkali earth metal hydroxide component, such as sodium hydroxide; wherein Buschmann adds specificity to the method and composition by teaching additional components and specific concentrations which can be used to optimize the method and composition taught in Ames. The additional specificity taught in Buschmann improves the applicability, effectiveness, efficiency and marketability of the method and composition taught in Ames.

Regarding claim 69; claim 68 is obvious as above. Furthermore, Ames teaches wherein the liquid acetyl precursor is triacetin (para [0007], with para [0015]).

Regarding claim 70; claim 68 is obvious as above. Furthermore, Ames teaches wherein the aqueous source of the alkali metal or earth alkali metal hydroxide is sodium hydroxide (para [0053]).

Regarding claim 71; claim 70 is obvious as above. Furthermore, Ames teaches wherein the sodium hydroxide is about 1.82% to about 7.28% (para [0013]).

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Regarding claims 72 and 73; claim 68 is obvious as above. Furthermore, Ames teaches wherein the method further comprises allowing the reaction medium sufficient time to maximize the conversion of the hydrogen peroxide and the acetyl precursor into peracetic acid; more specifically wherein about 30 seconds to about five minutes (para [0064]).

Regarding claim 74; claim 68 is obvious as above. Ames and Buschmann fail to expressly teach wherein the method further comprises, after step (g), a step of sampling the reaction medium to determine the time required to maximize the amount of hydrogen peroxide and acetyl precursor that are converted into peracetic acid. However, Ames teaches that activation time of the peracetic acid solution can depend on the temperature of the solution (para [0064]). Buschmann further teaches the the rate of peracetic acid production is dependent on the pH of the solution (para [0039] and [0042]). Combining the teachings in Ames and Buschmann would indicate that optimal reaction times could be determined by measuring various rate-controlling solution properties, such as temperature and pH; such that one with ordinary skill in the art could confirm without undue experimentation the effectiveness of including an additional sampling step measure solution properties and determine the optimal time required to maximize the amount of hydrogen peroxide and acetyl precursor that are converted into peracetic acid.

Regarding claim 75; claim 72 is obvious as above. Furthermore, Buschmann teaches wherein the acetyl precursor is triacetin (para [0027] and [0030]), and the percent of triacetin that is converted into peracetic acid is about 40.9% to about 85.7% (para [0035]).

Regarding claim 76; claim 71 is obvious as above. Furthermore, Ames teaches wherein the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is about 4.2:3.8:1 (para [0013]).

Regarding claim 77;
- Ames teaches an on site peracetic acid generation system (para [0013], [0064] and [0067]), comprising: (a) a first container comprising a first solution (para [0013] and [0067]) wherein said first solution comprises: (i) aqueous hydrogen peroxide (para [0007] and [0067]); (ii) a liquid acetyl precursor that is soluble in said aqueous hydrogen peroxide (para [0007] and [0067], with para [0017]); (iii) a trace amount of peracetic acid (para [0067]); and (iv) water (para [0007] and [0067]); and (b) a second container comprising a second solution (para [0013]).
- Ames fails to expressly teach wherein the second solution comprises: (i) an aqueous source of an alkali metal or earth alkali metal hydroxide. However, Ames and Buschmann teach this claim limitation as being obvious to one with ordinary skill in the art. Specifically, Ames teaches the inclusion alkali metal hydroxides, such as sodium hydroxide, within the initial mixture composition (para [0019] and [0051], with para [0022] and [0053]). Ames also specifically teaches the use of the alkali metal hydroxide component to raise the pH level of the composition (para [0053]). Ames fails to teach wherein the alkali metal hydroxide is added separately from other components. Buschmann teaches a similar method and composition for generating a non-equilibrium peracetic acid composition (para [0027] and [0033]) which comprises combining hydrogen peroxide and an acetyl precursor, such as triacetin, with water (para [0027] and [0033]), along with a pH-adjusting alkali earth metal hydroxide component, such as sodium hydroxide (para [0033]-[0034]). Buschmann also teaches that peractetic acid solutions are much more stable for "stabilization and storage" at acidic or neutral pH levels of 7.0 or below (para [0037] and [0042]), but also teaches that a "caustic pH" above 10 will accelerate the production of peractetic acid because "the hydrogen peroxide anion is a much better nucleophile than hydrogen peroxide" (para [0033]). The teachings in Buschmann indicate that a peracetic acid composition, such as one comprising hydrogen peroxide reacted with an acetyl donor, can be stabilized in a slow-reacting and non-decomposing state by maintaining a low pH in the composition, wherein the peracetic acid production in the solution can then be significantly accelerated when desired by a drastic increase in pH, such as by the addition of strong bases such as sodium hydroxide. In light of Buschmann, one with ordinary skill in the art could confirm without undue experimentation the effectiveness of including the aqueous source of an alkali metal hydroxide in a separate container from the hydrogen peroxide - acetyl precursor mixture, specifically when the system is used for on site generation of point-of-use peracetic acid solutions.
- Ames further teaches wherein the second container can comprise: (ii) means for mixing the first solution, water, and the second solution to generate a non-equilibrium peracetic acid solution (para [0063]).
- Buschmann also teaches wherein the system forms a non-equilibrium solution of peracetic acid (para [0033]).
- It would have been obvious to one with ordinary skill in the art to combine the components taught in Buschmann with the system and composition taught in Ames because both references relate to systems and compositions for generating peracetic acid which comprise combining hydrogen peroxide and an acetyl precursor, such as triacetin, with water, along with a pH-adjusting alkali earth metal hydroxide component, such as sodium hydroxide; wherein Buschmann adds specificity to the system and composition by teaching additional components and specific concentrations which can be used to optimize the system and composition taught in Ames. The additional specificity taught in Buschmann improves the applicability, effectiveness, efficiency and marketability of the system and composition taught in Ames.

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Claims 11, 12, 21, 22, 30, 31, 40, 41, 57, 58, 66, and 67 lack an inventive step under PCT Article 33(3) as being obvious over Ames in view of Buschmann, and in further view of WO 2001/046519 A1 to Withenshaw et al. (hereinafter "Withenshaw").

Regarding claims 11, 30 and 57; claims 7, 26 and 53 are obvious as above.
- Ames and Buschmann fail to expressly teach further claim limitations which are taught by Withenshaw, including wherein the provided water is an aqueous stream (pg 4, ln 27-28 and pg 8, ln 27-28).
- It would have been obvious to one with ordinary skill in the art to combine the components taught in Withenshaw with the method and composition taught in Ames and Buschmann because all three references relate to methods and compositions for generating peracetic acid which comprise combining hydrogen peroxide and an acetyl precursor, such as triacetin, with water, along with a pH-adjusting alkali earth metal hydroxide component, such as sodium hydroxide; wherein Withenshaw adds specificity to the method and composition by teaching additional components, processing steps, and specific concentrations which can be used to optimize the system and composition taught in Ames and Buschmann. The additional specificity taught in Withenshaw improves the applicability, effectiveness, efficiency and marketability of the method and composition taught in Ames and Buschmann.

Regarding claims 12, 31 and 58; claims 7, 26 and 53 are obvious as above.
- Ames and Buschmann fail to expressly teach further claim limitations which are taught by Withenshaw, including wherein the provided water is contained in a mixing tank or other vessel (pg 8, ln 14-15).
- It would have been obvious to one with ordinary skill in the art to combine the components taught in Withenshaw with the method and composition taught in Ames and Buschmann because all three references relate to methods and compositions for generating peracetic acid which comprise combining hydrogen peroxide and an acetyl precursor, such as triacetin, with water, along with a pH-adjusting alkali earth metal hydroxide component, such as sodium hydroxide; wherein Withenshaw adds specificity to the method and composition by teaching additional components, processing steps, and specific concentrations which can be used to optimize the system and composition taught in Ames and Buschmann. The additional specificity taught in Withenshaw improves the applicability, effectiveness, efficiency and marketability of the method and composition taught in Ames and Buschmann.

Regarding claims 21, 40 and 66; claims 7, 26 and 53 are obvious as above.
- Ames and Buschmann fail to expressly teach further claim limitations which are taught by Withenshaw, including wherein steps (a) through (e) are performed on a continuous basis (pg 3, ln 6-7).
- It would have been obvious to one with ordinary skill in the art to combine the components taught in Withenshaw with the method and composition taught in Ames and Buschmann because all three references relate to methods and compositions for generating peracetic acid which comprise combining hydrogen peroxide and an acetyl precursor, such as triacetin, with water, along with a pH-adjusting alkali earth metal hydroxide component, such as sodium hydroxide; wherein Withenshaw adds specificity to the method and composition by teaching additional components, processing steps, and specific concentrations which can be used to optimize the system and composition taught in Ames and Buschmann. The additional specificity taught in Withenshaw improves the applicability, effectiveness, efficiency and marketability of the method and composition taught in Ames and Buschmann.

Regarding claims 22, 41 and 67; claims 7, 26 and 53 are obvious as above.
- Ames and Buschmann fail to expressly teach further claim limitations which are taught by Withenshaw, including wherein steps (a) through (e) are performed on an intermittent basis (pg 5, ln 2, "semi-batch operation", with pg 8, ln 14).
- It would have been obvious to one with ordinary skill in the art to combine the components taught in Withenshaw with the method and composition taught in Ames and Buschmann because all three references relate to methods and compositions for generating peracetic acid which comprise combining hydrogen peroxide and an acetyl precursor, such as triacetin, with water, along with a pH-adjusting alkali earth metal hydroxide component, such as sodium hydroxide; wherein Withenshaw adds specificity to the method and composition by teaching additional components, processing steps, and specific concentrations which can be used to optimize the system and composition taught in Ames and Buschmann. The additional specificity taught in Withenshaw improves the applicability, effectiveness, efficiency and marketability of the method and composition taught in Ames and Buschmann.

Claims 1-91 have industrial applicability as defined by PCT Article 33(4) because the subject matter can be made or used by industry.

Clean Claims

1. A method of generating a non-equilibrium solution of peracetic acid, comprising:

a. providing water;

b. introducing a hydrogen peroxide-triacetin solution to the water;

c. mixing the hydrogen peroxide-triacetin solution and the water to form a mixture;

d. adding an aqueous source of an alkali metal or earth alkali metal hydroxide to the mixture; and

e. forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

2. The method of claim 1, further comprising, prior to step (b), a step of providing the hydrogen peroxide-triacetin solution comprising: introducing triacetin to a solution of aqueous hydrogen peroxide; and allowing the triacetin and the solution of aqueous hydrogen peroxide to mix to form the hydrogen peroxide-triacetin solution.

3. The method of claim 1, wherein the hydrogen peroxide-triacetin solution has a mole ratio of hydrogen peroxide to triacetin of about 2.98:1 to about 12.84:1.

4. The method of claim 1, wherein the provided water is an aqueous stream.

5. The method of claim 1, wherein the provided water is contained in a mixing tank or other vessel.

6. The method of claim 1, wherein the aqueous source of the alkali metal or earth alkali metal hydroxide is sodium hydroxide.

7. The method of claim 6, wherein the sodium hydroxide is about 1.82% to about 7.28%.

8. The method of claim 1, wherein step (d) is performed simultaneously with step (b).

9. The method of claim 1, further comprising allowing the reaction medium sufficient time to maximize the conversion of the hydrogen peroxide-triacetin into peracetic acid.

10. The method of claim 9, wherein the time to maximize the conversion of the hydrogen peroxide-triacetin into peracetic acid is about 30 seconds to about five minutes.

11. The method of claim 1, further comprising, after step (e), a step of sampling the reaction medium to determine the time required to maximize the amount of hydrogen peroxide-triacetin that is converted into peracetic acid.

12. The method of claim 9, wherein the percent of triacetin that is converted into peracetic acid is about 40.9% to about 85.7%.

13. The method of claim 7, wherein the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is about 4.2:3.8:1.

14. The method of claim 1, wherein steps (a) through (e) are performed on a continuous basis.

15. The method of claim 1, wherein steps (a) through (e) are performed on an intermittent basis.

16. The method of claim 1, wherein the peracetic acid solution is for immediate use.

17. The method of claim 1, further comprising, after step (e), a step of adding a source of acid to stabilize the non-equilibrium solution of peracetic acid for storing and using throughout a working day.

18. The method of claim 1, wherein the peracetic acid solution is prepared on a site having a point-of-use.

CLEAN CHEMISTRY EXHIBIT 1027
IPR2025-01458 – Page 246

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Web site: www.bailey-walsh.com



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A. Tomkinson, MA (Cantab) PhD, CPA, EPA
Associates: P. Stephenson, MSci, PhD, MRSC, CPA, EPA
M. Kellett, MITMA
D. Corbyn, MChem (Hons)

EPO - Munich
81
04. Okt. 2013

European Patent Office
Erhardstrasse 27
D-80298 Munich
Germany

BY FACSIMILE

Our ref.: AT-X11031-EP
Your ref.: 8658/17669P
30 Sep 2013

Confirmation only
already sent by fax

Dear Sirs,

Re.: European Patent Application No. 11861339.7 in the name of Enviro Tech Chemical Services, Inc. --Methods and compositions for the generation of peracetic acid on site at the point- of- use

Further to a telephone conversation between myself and a member of staff at the EPO on Friday 27th September 2013, we attach a clean copy of the claims for prosecution at the EPO.

We have been informed by the US attorney who prosecuted the corresponding US Patent Application that some errors were identified in the claims after acceptance of the claims by the USPTO. The US Patent is due to issue on 1st October and is likely to contain the errors.

The errors are as follows:

1. Previous claims 10 and 13 (now claims 3 and 6) depended from claim 9 but claim 9 had been cancelled. Therefore they should have depended from claim 7 (now claim 1). this amendment has been made in the clean copy.

2. Previous claim 28 should have been cancelled as it was broader than the amendment



made to previous claim 7. This results in rendering previous claims 29, 32, 33 and 39 duplicative. These claims have all been deleted in the clean copy of the amended claims.

We trust the above amendments are allowable as they are to correct obvious errors but please let me know if you have any queries.

Yours faithfully,
BAILEY WALSH & CO LLP

A. Tomkinson

Clean Claims

1. A method of generating a non-equilibrium solution of peracetic acid, comprising:

a. providing water;

b. introducing a hydrogen peroxide-triacetin solution to the water;

c. mixing the hydrogen peroxide-triacetin solution and the water to form a mixture;

d. adding an aqueous source of an alkali metal or earth alkali metal hydroxide to the mixture; and

e. forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

2. The method of claim 1, further comprising, prior to step (b), a step of providing the hydrogen peroxide-triacetin solution comprising: introducing triacetin to a solution of aqueous hydrogen peroxide; and allowing the triacetin and the solution of aqueous hydrogen peroxide to mix to form the hydrogen peroxide-triacetin solution.

3. The method of claim 1, wherein the hydrogen peroxide-triacetin solution has a mole ratio of hydrogen peroxide to triacetin of about 2.98:1 to about 12.84:1.

4. The method of claim 1, wherein the provided water is an aqueous stream.

5. The method of claim 1, wherein the provided water is contained in a mixing tank or other vessel.

Duration: 30.09.2013 16:46:48 - 30.09.2013 16:47:17. This page 3 of 5 was completed at 30.09.2013 16:47:07
Received at the EPO on Sep 30, 2013 16:47:17. Page 3 of 5

CLEAN CHEMISTRY EXHIBIT 1027
IPR2025-01458 – Page 249

6. The method of claim 1, wherein the aqueous source of the alkali metal or earth alkali metal hydroxide is sodium hydroxide.

7. The method of claim 6, wherein the sodium hydroxide is about 1.82% to about 7.28%.

8. The method of claim 1, wherein step (d) is performed simultaneously with step (b).

9. The method of claim 1, further comprising allowing the reaction medium sufficient time to maximize the conversion of the hydrogen peroxide-triacetin into peracetic acid.

10. The method of claim 9, wherein the time to maximize the conversion of the hydrogen peroxide-triacetin into peracetic acid is about 30 seconds to about five minutes.

11. The method of claim 1, further comprising, after step (e), a step of sampling the reaction medium to determine the time required to maximize the amount of hydrogen peroxide-triacetin that is converted into peracetic acid.

12. The method of claim 9, wherein the percent of triacetin that is converted into peracetic acid is about 40.9% to about 85.7%.

13. The method of claim 7, wherein the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is about 4.2:3.8:1.

Duration: 30.09.2013 16:46:48 - 30.09.2013 16:47:17. This page 4 of 5 was completed at 30.09.2013 16:47:13
Received at the EPO on Sep 30, 2013 16:47:17. Page 4 of 5

CLEAN CHEMISTRY EXHIBIT 1027
IPR2025-01458 – Page 250

14. The method of claim 1, wherein steps (a) through (e) are performed on a continuous basis.

15. The method of claim 1, wherein steps (a) through (e) are performed on an intermittent basis.

16. The method of claim 1, wherein the peracetic acid solution is for immediate use.

17. The method of claim 1, further comprising, after step (e), a step of adding a source of acid to stabilize the non-equilibrium solution of peracetic acid for storing and using throughout a working day.

18. The method of claim 1, wherein the peracetic acid solution is prepared on a site having a point-of-use.

Duration: 30.09.2013 16:46:48 - 30.09.2013 16:47:17. This page 5 of 5 was completed at 30.09.2013 16:47:17
Received at the EPO on Sep 30, 2013 16:47:17. Page 5 of 5

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Associates: P. Stephenson, MSci, PhD, MRSC, CPA, EPA
M. Kellett, MITMA
D. Corbyn, MChem (Hons)

European Patent Office
Erhardstrasse 27
D-80298 Munich
Germany

BY FACSIMILE

Our ref.: AT-X11031-EP
Your ref.: 8658/17669P
30 Sep 2013

Dear Sirs,

Re.: European Patent Application No. 11861339.7 in the name of Enviro Tech Chemical Services, Inc. --Methods and compositions for the generation of peracetic acid on site at the point- of- use

Further to a telephone conversation between myself and a member of staff at the EPO on Friday 27th September 2013, we attach a clean copy of the claims for prosecution at the EPO.

We have been informed by the US attorney who prosecuted the corresponding US Patent Application that some errors were identified in the claims after acceptance of the claims by the USPTO. The US Patent is due to issue on 1st October and is likely to contain the errors.

The errors are as follows:

1. Previous claims 10 and 13 (now claims 3 and 6) depended from claim 9 but claim 9 had been cancelled. Therefore they should have depended from claim 7 (now claim 1). this amendment has been made in the clean copy.

2. Previous claim 28 should have been cancelled as it was broader than the amendment

Duration: 30.09.2013 16:46:48 - 30.09.2013 16:47:17. This page 1 of 5 was completed at 30.09.2013 16:46:56
Received at the EPO on Sep 30, 2013 16:47:17. Page 1 of 5

made to previous claim 7. This results in rendering previous claims 29, 32, 33 and 39 duplicative. These claims have all been deleted in the clean copy of the amended claims.

We trust the above amendments are allowable as they are to correct obvious errors but please let me know if you have any queries.

Yours faithfully,
BAILEY WALSH & CO LLP

A. Tomkinson

Duration: 30.09.2013 16:46:48 - 30.09.2013 16:47:17. This page 2 of 5 was completed at 30.09.2013 16:47:01
Received at the EPO on Sep 30, 2013 16:47:17. Page 2 of 5

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European Patent Office
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Telefonische Rücksprache / Consultation by telephone / Entretien téléphonique

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Mit dem ☐ Anmelder ☐ Vertreter
With the ☐ applicant ☑ representative
Avec le ☐ demandeur ☐ mandataire

und zwar / i.e. / c'est-à-dire

mit / with / avec : Ms. Alexandra Tomkinson

Anmeldung Nr. / Application No. / Demande N° 11861339.7

Gegenstand / Subject / Objet :

I informed the representative, that 23 amended claims were mentioned in the Entry into the European Phase filed online on 06.09.2013, however the document called "clean copy of 23 allowed claims" contains claims Nr. 7 to 39 which are 32 claims in total.
Ms. Tomkinson will send us the correct copy of the amended claims properly numbered.

Rijswijk, 27-09-2013 / 11:00 Vencourova, Lenka
Datum/date Uhrzeit/time/heure Unterschrift/signature

LV50776

EPO Form 1326 12.07

Europäisches Patentamt
European Patent Office
Office européen des brevets

Acknowledgement of receipt

We hereby acknowledge receipt of the form for entry into the European phase (EPO as designated or elected Office) as follows:

Submission number	2288430
PCT application number	PCT/US2011/000539
EP application number	11861339.7
Date of receipt	06 September 2013
Receiving Office	European Patent Office, The Hague
Your reference	ATSAR-X11031-EP
Applicant	
Country	

Documents submitted	package-data.xml application-body.xml AMSPECEPO-1.pdf\X11031-EP - US Allowed Claims.DOC.pdf (4 p.) OTHER-2.pdf\X11031-EP - USPTO Office Action dated 8 November 2012.pdf (10 p.) OTHER-4.pdf\X11031-EP - USPTO Office Action dated 15 July 2013.pdf (7 p.)	ep-euro-pct.xml epf1200.pdf (5 p.) OTHER-1.pdf\X11031-EP - EPO Form 1009 US.pdf (1 p.) OTHER-3.pdf\X11031-EP - USPTO Office Action dated 1 April 2013.pdf (9 p.) OTHER-5.pdf\X11031-EP - EPO Form 1005.pdf (1 p.)

Submitted by	CN=Alexandra Tomkinson 4603
Method of submission	Online
Date and time receipt generated	06 September 2013, 14:42 (CEST)

CLEAN CHEMISTRY EXHIBIT 1027
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Message Digest	31:BD:33:BF:F1:44:82:4D:61:A0:C8:2C:6C:23:FA:6B:08:96:E7:A2

Correction by the EPO of errors in debit instructions filed by eOLF
Errors in debit instructions filed by eOLF that are caused by the editing of Form 1038E entries or the continued use of outdated software (all forms) may be corrected automatically by the EPO, leaving the payment date unchanged (see decision T 152/82, OJ EPO 1984, 301 and point 6.3 ff ADA, Supplement to OJ EPO 10/2007).

/European Patent Office/

7. A method of generating a non-equilibrium solution of peracetic acid, comprising:

a. providing water;

b. introducing a hydrogen peroxide-triacetin solution to the water;

c. mixing the hydrogen peroxide-triacetin solution and the water to form a mixture;

d. adding an aqueous source of an alkali metal or earth alkali metal hydroxide to the mixture; and

e. forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

8. The method of claim 7, further comprising, prior to step (b), a step of providing the hydrogen peroxide-triacetin solution comprising: introducing triacetin to a solution of aqueous hydrogen peroxide; and allowing the triacetin and the solution of aqueous hydrogen peroxide to mix to form the hydrogen peroxide-triacetin solution.

10. The method of claim 9, wherein the hydrogen peroxide-triacetin solution has a mole ratio of hydrogen peroxide to triacetin of about 2.98:1 to about 12.84:1.

11. The method of claim 7, wherein the provided water is an aqueous stream.

12. The method of claim 7, wherein the provided water is contained in a mixing tank or other vessel.

13. The method of claim 9, wherein the aqueous source of the alkali metal or earth alkali metal hydroxide is sodium hydroxide.

14. The method of claim 13, wherein the sodium hydroxide is about 1.82% to about 7.28%.

15. The method of claim 7, wherein step (d) is performed simultaneously with step (b).

16. The method of claim 7, further comprising allowing the reaction medium sufficient time to maximize the conversion of the hydrogen peroxide-triacetin into peracetic acid.

17. The method of claim 16, wherein the time to maximize the conversion of the hydrogen peroxide-triacetin into peracetic acid is about 30 seconds to about five minutes.

18. The method of claim 17, further comprising, after step (e), a step of sampling the reaction medium to determine the time required to maximize the amount of hydrogen peroxide-triacetin that is converted into peracetic acid.

19. The method of claim 16, wherein the percent of triacetin that is converted into peracetic acid is about 40.9% to about 85.7%.

20. The method of claim 14, wherein the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is about 4.2:3.8:1.

21. The method of claim 7, wherein steps (a) through (e) are performed on a continuous basis.

22. The method of claim 7, wherein steps (a) through (e) are performed on an intermittent basis.

23. The method of claim 7, wherein the peracetic acid solution is for immediate use.

24. The method of claim 7, further comprising, after step (e), a step of adding a source of acid to stabilize the non-equilibrium solution of peracetic acid for storing and using throughout a working day.

25. The method of claim 7, wherein the peracetic acid solution is prepared on a site having a point-of-use.

28. The method of claim 8, wherein the liquid acetyl precursor is triacetin defining a hydrogen peroxide-triacetin solution.

29. The method of claim 28, wherein the hydrogen peroxide-triacetin solution has a mole ratio of hydrogen peroxide to triacetin of about 2.98:1 to about 12.84:1.

32. The method of claim 28, wherein the aqueous source of the alkali metal or earth alkali metal hydroxide is sodium hydroxide.

33. The method of claim 32, wherein the sodium hydroxide is about 1.82% to about 7.28%.

39. The method of claim 33, wherein the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is about 4.2:3.8:1.

CLEAN CHEMISTRY EXHIBIT 1027
IPR2025-01458 – Page 260

Europäisches Patentamt
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Office européen des brevets

Entry into the European phase (EPO as designated or elected Office)

To the European Patent Office

European application number	EP11861339.7
PCT application number	PCT/US2011/000539
PCT publication number	WO2012128734
Applicant's or representative's reference	ATSAR-X11031-EP
International Filing Date	24.03.2011
International Searching Authority (ISA)	US
International Preliminary Examining Authority (IPEA)	not applicable

1. Applicant

Indications concerning the applicant(s) are contained in the international publication or were recorded by the International Bureau after the international publication. ☒

Changes which have not yet been recorded by the International Bureau are set out here: ☐

2. Representative

This is the representative who will be listed in the Register of European Patents and to whom notifications will be made

Representative 1

Name:	Tomkinson, Ms Alexandra
Company:	Bailey Walsh & Co LLP
Address of place of business:	5 York Place
	Leeds, Yorkshire, LS1 SD
	United Kingdom
Telephone:	00 44 113 243 3824
Fax:	00 44 113 244 5699
e-mail:	mail@bailey-walsh.com

3. Authorisation

An individual authorisation is attached. ☐

A general authorisation has been registered under No: ☐

A general authorisation has been filed, but not yet registered. ☐

The authorisation filed with the EPO as PCT receiving Office expressly includes the European phase. ☐

4. Request for examination

Examination of the application under Art. 94 EPC is hereby requested. The examination fee is being (has been, will be) paid. ☒

Request for examination in an admissible non-EPO language: ☐

The applicant waives his right to be asked under Rule 70(2) EPC whether he wishes to proceed further with the application. ☒

5. Copies

Additional copies of the documents cited in the supplementary European search report are requested. ☐

Number of additional sets of copies

6. Documents intended for proceedings before the EPO

Number of claims on entry into the European phase: 23

6.1 Proceedings before the EPO as designated Office (PCT I) are to be based on the following documents:

the application documents published by the International Bureau (with all claims, description and drawings), where applicable with amended claims under Art. 19 PCT ☐

unless replaced by the amendments attached. ☒

CLEAN CHEMISTRY EXHIBIT 1027
IPR2025-01458 – Page 261

Where necessary, clarifications should be attached as `Other documents`	
6.2 Proceedings before the EPO as elected Office (PCT II) are to be based on the following documents:	
the documents on which the international preliminary examination report is based, including any annexes	☐
unless replaced by the amendments attached.	☐
Where necessary, clarifications should be attached as `Other documents`	
If the EPO as International Preliminary Examining Authority has been supplied with test reports, these may be used as the basis of proceedings before the EPO.	☐
6.3 A copy of the results of the search carried out by the authority with which the previous application(s) whose priority is claimed was (were) filed is attached (R. 141(1) EPC).	☐
6.4 The applicant waives his right to the communication under Rules 161(1) or (2) and 162 EPC.	☒
7. Translations	
Translations in one of the official languages of the EPO (English, French, German) are attached as crossed below:	☐
** In proceedings before the EPO as designated or elected Office (PCT I + II):*	
Translation of the international application (description, claims, any text in the drawings) as originally filed, of the abstract as published and of any indication under Rule 13bis.3 and 13bis.4 PCT regarding biological material	☐
Translation of the priority application(s) (to be filed only at the EPO's request, Rule 53(3) EPC)	☐
It is hereby declared that the international application as originally filed is a complete translation of the previous application (Rule 53(3) EPC)	☐
** In addition, in proceedings before the EPO as designated Office (PCT I):*	
Translation of amended claims and any statement under Art. 19 PCT, if the claims as amended are to form the basis for the proceedings before the EPO (see Section 6).	☐
** In addition, in proceedings before the EPO as elected Office (PCT II):*	
Translation of annexes to the international preliminary examination report	☐
8. Biological material	
The invention uses and/or relates to biological material deposited under Rule 31 EPC.	☐
The particulars referred to in Rule 31(1)(c) EPC (if not yet known, the depositary institution and the identification reference(s)) [number, symbols, etc.] of the depositor) are given in the international publication or in the translation submitted in Section 7 on:	☐
page(s) / line(s)	
The receipt(s) of deposit issued by the depositary institution	
is (are) enclosed.	☐
will be filed later.	☐
Waiver of the right to an undertaking from the requester pursuant to Rule 33(2) EPC attached.	☐
9. Nucleotide and amino acid sequences	
The international application discloses nucleotide and/or amino acid sequences.	☐
9.1 The sequence listing was filed under Rule 5.2(a) PCT, or furnished to the EPO as ISA under Rule 13ter.1(a) PCT, or is otherwise available to the EPO, in computer-readable format in accordance with WIPO ST.25.	☐
9.2	
The sequence listing is attached in computer-readable format in accordance with WIPO Standard ST.25	☐
The sequence listing is attached in PDF format.	☐
The sequence listing does not include matter which goes beyond the content of the application as filed.	☐
10. Designation fees	
All the contracting states party to the EPC at the time of filing of the international patent application and designated in the international application are deemed to	☒

be designated (see Article 79(1) EPC).

The following states, which were contracting states to the EPC at the time of filing of the international application, are designated:

AL AT BE BG CH&LI CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LT LU LV MC MK MT NL NO PL PT RO RS SE SI SK SM TR

11. Extension of the European patent

☐

This application is deemed to be a request to extend the European patent application and the European patent granted in respect of it to all the non-contracting states to the EPC designated in the international application and with which extension agreements are in force on the date on which the international application is filed. However, the extension only takes effect if the prescribed extension fee is paid.

It is currently intended to pay the extension fee for the following states:

Note: Under the automatic debiting procedure, extension fees will only be debited for states indicated here, unless the EPO is instructed otherwise before expiry of the period for payment.

12. List of enclosed documents

	Description of document	Original file name	Assigned file name
1	Combined Amendments	X11031-EP - US Allowed Claims.DOC.pdf	AMSPECEPO-1.pdf
2	EPO Form 1009 US	X11031-EP - EPO Form 1009 US.pdf	OTHER-1.pdf
3	USPTO Office Action 8 November 2012	X11031-EP - USPTO Office Action dated 8 November 2012.pdf	OTHER-2.pdf
4	USPTO Office Action dated 1 April 2013	X11031-EP - USPTO Office Action dated 1 April 2013.pdf	OTHER-3.pdf
5	USPTO Office Action dated 15 July 2013	X11031-EP - USPTO Office Action dated 15 July 2013.pdf	OTHER-4.pdf
6	EPO Form 1005	X11031-EP - EPO Form 1005.pdf	OTHER-5.pdf

13. Mode of payment: Debit from deposit account

☒

Currency: EUR

The European Patent Office is hereby authorised, to debit from the deposit account with the EPO any fees and costs indicated on the fees page.

Deposit account number: 28050202

Account holder: Bailey Walsh & Co LLP

14. Any refunds should be made to the following EPO deposit account:

☒

Number and account holder: Bailey Walsh & Co LLP, 28050202

15. Fees

		Factor/reduction applied	Fee schedule	Amount to be paid
15-1	002 Fee for a European search - Applications filed on/after 01.07.2005	-190	1 165.00	975.00
15-2	005e Designation fee - For all contracting States designated for applications filed on/after 01.04.2009	1	555.00	555.00
15-3	006 Examination fee - For applications filed on/after 01.07.2005	1	1 555.00	1 555.00
15-4	015 Claims fee - For the 16th to the 50th claim	8	225.00	1 800.00
15-5	020 Filing fee - entry EP phase - online	1	115.00	115.00
15-6	033 Renewal fee for the 3rd year	1	445.00	445.00
15-7	520 Additional filing fee for the 36th and each subsequent page - entry into EP phase	30	14.00	420.00
	Total:		**EUR**	**5 865.00**

16. Annotations

16-1. Note (for EPO) (EP Phase)

European Patent Application No. 11861339.7 in the name of Envirotech Chemical Services, Inc (A Tomkinson)

	In connection with the abovementioned patent application please find enclosed the following documents to enable the application to proceed under the "Patent Prosecution Highway" (PPH)" :- Form EPA/EPO/OEB 1009 US USPTO Office Action dated 8 November 2012 USPTO Office Action dated 1 April 2013 USPTO Office Action dated 15 July 2013 Copy of Claims allowed by USPTO We hereby request accelerated search and examination of the application under "PACE" and enclose the Form EPA/EPO/OEB 1005 in respect of this matter.

17. Signature(s) of applicant(s) or representative

Place:	**Leeds, UK**
Date:	**06 September 2013**
Signed by:	**, Alexandra Tomkinson 4603**
Capacity:	**(Representative)**

Table for section 6 of Form 1200.3

In accordance with the Notice from the European Patent Office dated 26 January 2009 concerning the 2009 fee structure (OJ EPO 2009, 118, and Guidelines for Examination in the EPO, April 2009, A-III, 13.2), the amount of the additional fee (Art. 2, item 1a, Rules relating to Fees) for the pages of this European patent application is calculated as follows:

Documents intended for proceedings before the EPO (R. 159 (1) (b) EPC) and for calculating the additional fee (Art. 2, item 1a, RFees):

		Page(s) from ... to ...	Number of pages
Description:	International application as published	1-59	59
Claims:	Amendments filed on entry into European phase	1-4	4
Drawings:	International application as published	1-1	1
Abstract:	Default count: one page		1
Total number of pages			65
Fee-exempt pages (Art. 2, item 1a, RFees)			-35
Number of pages to be paid for			30
			(x 14 EUR per page)
Total amount payable		EUR	420

ATSAR-X11031-EP

Europäisches Patentamt
European Patent Office
Office européen des brevets

European Patent Office
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MILLEMANN, Audrey, A.
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400 Capital Mall, 11th Floor
Sacramento, CA 95814
ETATS-UNIS D'AMERIQUE

Date
31.07.13

Reference	Application No./Patent No.
	11861339.7 - 1454 PCT/US2011000539
Applicant/Proprietor	
Enviro Tech Chemical Services Inc.	

Entry into the European phase before the European Patent Office

The following information describes the procedural steps required for entry into the European phase before the European Patent Office (EPO). You are advised to read it carefully because failure to take the necessary action in due time can lead to a loss of rights.

1. The above mentioned international patent application has been given the **European application No. 11861339.7**.

2. Applicants **without a residence or their principal place of business** in an EPC Contracting State may themselves initiate European processing of their international applications, provided they do so before expiry of the 31st month from the priority date.

   During the European phase before the EPO as designated or elected Office, however, such applicants **must** be represented by a professional representative (Art. 133(2) and Art. 134(1) and (8) EPC).

   Where, at the expiry of the time period laid down in Rule 163(5) EPC, the requirements of Article 133(2) EPC have not been complied with, the European patent application will be **refused**, pursuant to Rule 163(6) EPC.

   Please note that a professional representative authorised to act before the EPO and who acted for the applicant during the international phase does not automatically become the representative for the European phase. Applicants are therefore strongly advised to appoint in good time any representative they wish to initiate the European phase for them; otherwise the EPO has to send all communications directly to the applicant.

3. Applicants **with a residence or their principal place of business** in an EPC Contracting State are not obliged to appoint for the European phase a professional representative authorised to act before the EPO. However, in view of the complexity of the procedure it is recommended that they do so.

4. Applicants and professional representatives are also strongly advised to initiate the European phase using **EPO Form 1200**. It is available free of charge from the EPO or via the EPO website at **www.epo.org**. Similarly, it can be or generated with the Online Filing software, obtainable free of charge from the EPO (**www.epoline.org**) The use of the form is not compulsory.

5. Where the EPO acts as designated or elected Office (Art. 22(1) and (3) and 39(1) PCT), to enter the European phase before the EPO, the **following acts** must be performed by the applicant within **31 months** from the date of filing of the international application or (where applicable) the earliest priority date:

   a) Supply a translation of the international application into an EPO official language, if the International Bureau did not publish the application in such language (Art. 22(1) PCT and R. 159(1)(a) EPC);

   b) Specify the application documents, as originally filed or as amended, on which the European grant procedure is to be based (R. 159(1)(b) EPC);

   c) Pay the filing fee and, where applicable, the additional fee for a European patent application comprising more than 35 pages (R. 159(1)(c) EPC, Art. 2, items 1, 1a Rules relating to Fees);

   d) Pay the search fee where a supplementary European search report has to be drawn up (R. 159(1)(e) EPC);

   e) Pay the designation fee if the time limit laid down in Rule 39(1) EPC (i.e. six months after publication of the international search report) has expired before the 31-month period pursuant to Rule 159(1) EPC (R. 159(1)(d) EPC);

   f) File the written request for examination and pay the examination fee if the time limit laid down in Rule 70(1) EPC has expired before the 31-month period pursuant to Rule 159(1) EPC (R. 159(1)(f) EPC);

   g) Pay the renewal fee in respect of the third year, if the fee has fallen due (see Rule 51(1) EPC) before expiry of the 31-month period pursuant to Rule 159(1) EPC (R. 159(1)(g) EPC);

   h) File, where applicable, the certificate of exhibition referred to in Article 55(2) and Rule 25 EPC (R. 159(1)(h) EPC);

   i) Pay the claims fees for the sixteenth and each subsequent claim when the application documents on which the European grant procedure is to be based comprise more than fifteen claims (R. 162(1) EPC). For applications entering the European phase on or after 1 April 2009, a higher amount is payable for the 51st and each subsequent claim (Decision of the Administrative Council of 14 December 2007 amending the Rules relating to Fees, OJ EPO 2008, 10).

   If either the translation of the international application or the request for examination is not filed in time, or if the filing fee, the additional fee, the search fee, the designation fee or the examination fee is not paid in due time, the application shall be deemed to be withdrawn (R. 160(1) EPC).

6. Payment of fees

   An up-to-date guidance for the payment of fees, expenses and prices and a list of the euro accounts of the European Patent Organisation are published in each issue of the Official Journal of the EPO. The guidance includes inter alia a reference to the latest version of the Schedule of fees and expenses where the amounts of fees are set out.

   The Schedule of fees and expenses, published as a Supplement to the Official Journal of the EPO, is also available on the EPO website (**www.epo.org**) and can be found under **www.epo.org/schedule-of-fees**, which allows the viewing, downloading and searching for individual fee amounts, both current and previous.

   Applicants should always check the fee amounts applying at the time of payment.

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Payments can be validly made by any person. Permissible methods of payment are laid down in Article 5 Rules relating to Fees. Please note that payment cannot be made by cheque sent to the EPO.

For information on the calculation of the additional fee for applications comprising more than 35 pages, see Notice from the European Patent Office dated 26 January 2009 concerning the 2009 fee structure (OJ EPO 2009, 118), Notice supplementing the notice from the European Patent Office dated 26 January 2009 concerning the 2009 fee structure (OJ EPO 2009, 338) and Guidelines for Examination in the EPO, April 2010, A-III, 13.2.

For an overview of search and examination fees, see Notice from the European Patent Office dated 6 February 2012 (OJ EPO 2012, 212). Fee information is also published on the EPO website under **www.epo.org/fees.**

7. Restoration of priority right

   Where the international application contains a priority claim to an earlier application and it has been filed within two months from the expiration of the 12-month priority period, a request for restoration before the EPO as designated Office (R. 49*ter*.2 PCT) applies under the following circumstances:

   a) No request for restoration filed before the receiving Office (RO) during the international phase (R. 26*bis*.3 PCT)
   b) Negative decision by the RO irrespective of the criterion applied (due care/ unintentionality)
   c) Positive decision by the RO based on the unintentionality criterion.

   For a request to be admissible, it must be filed and the requisite fee must be paid (R. 49*ter*.2(b)(iii) and R. 49*ter*.2(d) PCT) within one month from the applicable time limit under Article 22 PCT for entering the regional phase (R. 49*ter*.2(b)(i) PCT). The request for restoration also needs to state the reasons for the failure to file the international application within the priority period (Rule 49*ter*.2(b)(ii) PCT).

8. If the applicant had appointed a representative during the application's international phase, the present Form will be sent to the representative, asking him to inform the applicant accordingly.
   All subsequent communications will be sent to the applicant, or - if the EPO is informed of his appointment in time - to the applicant's European representative.

9. For more details about time limits and procedural acts before the EPO as designated or elected Office, see the EPO brochure "How to get an European patent", Guide for applicants - Part 2, PCT procedure before the EPO - "Euro-PCT" (EPO PCT Guide). This brochure, the list of professional representatives before the EPO, Form 1200 and details of the latest fees are available on the Internet under **http://www.epo.org**.

10. **Programme for accelerated prosecution of European patent applications - "PACE"**

    Applicants wishing to accelerate proceedings before the EPO as designated or elected Office may request accelerated search and/or examination under the PACE programme (Notice from the European Patent Office dated 4 May 2010 concerning the programme for accelerated prosecution of European patent applications - "PACE", OJ EPO 2010, 352). To be as effective as possible, this should preferably be requested immediately on entry into the European phase.

Receiving Section



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INTERNATIONAL SEARCH REPORT

International application No.
PCT/US 11/00539

A. CLASSIFICATION OF SUBJECT MATTER
IPC(8) - A01N 37/16; A01N 25/00; A61K 31/075; A61L 9/00 (2011.01)
USPC - 424/405 ; 422/28; 422/29; 514/714
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)
USPC - 424/405 ; 422/28; 422/29; 514/714

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
USPC - 424/405, 424/616, 424/719; 422/28; 422/29; 510/110; 514/714 (words only)

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
*** Databases: WEST (PGPB, USPT, USOC, EPAB, JPAB); Google
*** Search Terms Used: Enviro Tech, Harvey, Howarth, paracetic acid, peroxyacetic acid, hydrogen peroxide, acetyl, donor, precursor, aqueous, non-equilibrium, triacetin, sodium hydroxide, point-of-use, premixed, premixing,

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X -- Y	US 2010/0227000 A1 (Ames et al.) 09 September 2010 (09.09.2010), especially para [0004], [0007], [0013], [0015], [0019], [0021]-[0022], [0024], [0051], [0053], [0063]-[0064], [0066]-[0067], [0069]	1-6, 78-91 ---------- 7-77
Y	US 2009/0175956 A1 (Buschmann et al.) 09 July 2009 (09.07.2009), especially para [0023], [0027], [0030]-[0031], [0033]-[0035], [0037], [0039], [0042]	7-77
Y	WO 2001/046519 A1 (Withenshaw et al.) 28 June 2001 (28.06.2001), especially pg 3, ln 6-7; pg 4, ln 27-28; pg 5, ln 2; pg 8, ln 14-15, 27-28	11, 12, 21, 22, 30, 31, 40, 41, 57, 58, 66, 67
A	US 2010/0196505 A1 (Kaiser et al.) 05 August 2010 (05.08.2010), claim 1, para [0052]-[0058], [0083]	1-91

☐ Further documents are listed in the continuation of Box C. ☐

* 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 application or patent 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
16 May 2011 (16.05.2011)	03 JUN 2011
Name and mailing address of the ISA/US Mail Stop PCT, Attn: ISA/US, Commissioner for Patents P.O. Box 1450, Alexandria, Virginia 22313-1450 Facsimile No. 571-273-3201	Authorized officer: Lee W. Young PCT Helpdesk: 571-272-4300 PCT OSP: 571-272-7774

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(54) **Title**: METHODS AND COMPOSITIONS FOR THE GENERATION OF PERACETIC ACID ON SITE AT THE POINT-OF-USE

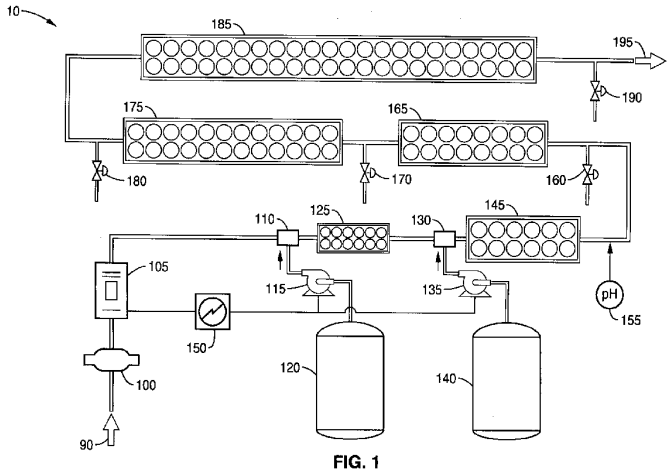


FIG. 1

(57) **Abstract**: Methods and compositions for the generation of a peroxyacetic acid sanitizer in proximity to the point-of-use are disclosed. These methods comprise introducing a hydrogen peroxide-acetyl precursor solution to water, mixing, and then adding an aqueous source of a alkali metal or earth alkali metal hydroxide. Triacetin is a preferred acetyl precursor and is converted rapidly and with a high conversion rate into peracetic acid. These methods produce solutions with a high level of peracetic acid. Methods for preparing the hydrogen peroxide- acetyl precursor solution are also provided. Also disclosed are solid compositions comprising a liquid acetyl precursor, a water-soluble source of hydrogen peroxide, and a water-soluble source of alkalinity. The solid composition is a freely-flowable solid that is used as a bleaching agent and a stain remover for the treatment of articles such as fabrics, dentures, textile garments, and equipment used in the food and beverage industry.

WO 2012/128734 A1

METHODS AND COMPOSITIONS FOR THE GENERATION OF PERACETIC ACID ON SITE AT THE POINT-OF-USE

Michael S. Harvey and Jonathan N. Howarth

FIELD OF THE INVENITON

[0001] The invention relates to liquid compositions and methods of using the liquid compositions for the generation of a peroxyacetic acid sanitizer on site in proximity to the point-of-use. The invention also relates to a solid bleaching and stain removal composition.

BACKGROUND OF THE INVENTION

[0002] **A. Liquid PAA Disinfectants and Sanitizers**

[0003] Disinfectants and sanitizers based on peroxyacetic acid (PAA), also commonly known as peracetic acid, are used in the dairy, food and beverage processing industries for clean-in-place pipeline and equipment disinfecting and cleaning, for fruit and vegetable washing, and in the treatment of meat, poultry and seafood products. Peroxyacetic acid disinfectants are also used in the treatment of cooling water, process water, and municipal wastewater. Other uses include slime and biofilm removal in papermaking processes.

[0004] Peroxyacetic acid products are supplied as stable equilibrium ternary aqueous solutions of peroxyacetic acid, acetic acid, and hydrogen peroxide. They are prepared in advance of delivery, typically by reacting hydrogen peroxide with acetic acid in the presence of a mineral acid catalyst. Although some PAA is formed immediately, the PAA does not reach its maximum concentration until after several days. A metal chelating agent, such as hydroxyethylidene

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diphosphonic acid (HEDP) or dipicolinic acid, is also introduced to suppress the transition metal cation catalyzed decomposition of peroxygen compounds. The PAA product is then placed in containers, such as totes, drums and pails, in preparation for shipment to the end user. Typical commercial products have concentrations of PAA of about 1-15% w/w, although a concentration of up to 30% is possible. However, the latter concentration product only finds captive use because of its extremely hazardous and explosive properties.

[0005] There are several problems with the use of equilibrium solutions of PAA. First, the low concentration of PAA (1-15%) means that most of the weight (85-99%) of the products consist of substantial amounts of inert ingredients, such as water, acetic acid, and hydrogen peroxide. This results in the need for larger product storage areas and causes increased transportation and handling costs. Second, the products are inefficient in the use of both hydrogen peroxide (hydrogen peroxide) and acetic acid (AA). To maintain adequate storage stability of the PAA, either hydrogen peroxide or AA must be present at a level on a weight percentage basis that is greater than the level of PAA, which increases raw material costs. Third, the presence of the HEDP or dipicolinic acid stabilizer limits the amount of PAA that can be applied to certain foodstuffs because the amount of HEDP or dipicolinic acid is regulated by the Food and Drug Administration (FDA). Fourth, the reaction between hydrogen peroxide and AA is quite slow in some cases, and typically requires several days' time before the PAA product can be tested for quality confirmation. Therefore, manufacturers of these products must store large inventories of the PAA product before it can be shipped to the end user. Fifth, for transportation purposes, equilibrium solutions that are greater than 6% PAA are considered to be dangerous products and must be labeled with the DOT marking "Organic Peroxide", Hazard Class 5.2, 8 (oxidizer, corrosive). If producers and end-users exceed the yearly threshold amount of just 6,666 lbs. of 15% PAA, they must file Risk Management Plans with both federal EPA and state

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authorities. This is an arduous and time-consuming process. In addition, producers of equilibrium solutions of PAA that are over 6% PAA must obtain permitting by the local fire department and pay an extra hazardous material shipping fee when the product is shipped from their facility. On the other hand, there are much less stringent requirements for products that contain hydrogen peroxide, and all reporting, permitting and shipping restrictions are lifted for products that contain less than 27% hydrogen peroxide.

[0006] As a result of these problems, there have been various attempts to make non-equilibrium solutions of PAA on site, at the point-of-use. For example, U.S. patent no. 7,012,154 discloses a system in which AA, hydrogen peroxide, water, and sulfuric acid are fed to a jacketed reactor for the production of PAA. A wiped-film distillation column attached to the reactor condenses and isolates the pure PAA from the gas phase and immediately introduces it to the receiving water. This system suffers from a number of drawbacks. First, it is extremely capital intensive due to the high cost of the equipment, including the reactor, heater, pumps, distillation column, and computerized control system which ensures accurate metering of the reagents. Second, there are significant safety hazards associated with the production of pure PAA due to its explosive properties. Third, the equipment and synthesis process is very complex and requires knowledgeable and highly trained technicians to continuously operate and maintain the system in a safe and effective manner.

[0007] Other attempts to make non-equilibrium solutions of PAA at the point-of-use are based on electrolytic processes. U.S. patent nos. 6,171,551 and 6,387,236 disclose processes that employ a cell divided by an ion-exchange membrane in which PAA (and other oxidants including hydrogen peroxide and ozone) are produced in the anode compartment which consists of an aqueous solution of acetic acid or acetate salt. In addition to the high costs of electricity and electrolysis equipment, these processes also result in a very low yield of PAA from the

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acetyl precursor. For example, the '551 patent reports that less than 14 ppm of PAA was produced after 90 minutes of electrolysis of a 490,000 ppm anolyte of aqueous potassium acetate. In addition, these processes are difficult to perform intermittently.

[0008] Other methods of generating non-equilibrium solutions of PAA on site, using electrolysis, are described in WIPO International Publication Nos. WO 2004/0245116 and WO 2008/140988, and U.S. Patent Application Publication No. 2009/0314652. These references disclose cation membrane-divided electrolysis cells and the use of gas diffusion electrodes to effect the cathodic reduction of oxygen gas to hydrogen peroxide under alkaline conditions. The hydrogen peroxide was then allowed to react with acetic acid or an acetyl precursor to form PAA in the bulk solution, whereupon the catholyte was directed to the acidic anode compartment of the cell to stabilize the PAA. This system suffers from several disadvantages. Due to the low solubility of oxygen in water (about 8 ppm maximum), the concentration of electroactive species is very low, which forces the cell to operate at low current density (amperage per surface area of electrode). In order to produce a meaningful amount of hydrogen peroxide, the cells must have a very large surface area. This requires high capital equipment costs and a very large footprint for the electrolysis equipment. Another disadvantage of this system is that it is very difficult to maintain steady-state conditions and simultaneously balance the feed of acetic acid or acetyl precursor to the cathode compartment with the concurrent withdrawal of acidified PAA solution from the anode compartment. This is because cations carrying the cell current through the cation exchange membrane are always hydrated so as the cations move through the membrane, they are accompanied by water molecules. As a result, the volume of the anolyte decreases and the volume of the catholyte increases, making the steady-state condition difficult to maintain. It is difficult to perform this process intermittently. Finally, this system can only be of economic value if the source of oxygen is air, which comprises 23% oxygen. However, the carbon dioxide

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contained in air causes carbonates to precipitate, which impedes the flow of electrical current, limiting or eliminating the production of hydrogen peroxide, and hence, PAA.

[0009] Another method of making a non-equilibrium solution of PAA at the point-of-use is disclosed in WIPO International Publication No. WO 2008/140988 and U.S. Patent Application Publication Nos. 2009/0005590 and 2007/0082832. These references disclose biosynthetic methods of producing peracids from carboxylic acids and carboxylic acid esters. These methods involve the use of perhydrolase enzymes to catalyze the perhydrolysis of the carboxylic acid or ester into the peracid using a solid or liquid source of hydrogen peroxide. Although these methods can produce compositions containing up to 20 parts of peracid to one part of hydrogen peroxide, they are limited by the amount of peracid that can be produced before the perhydrolase enzyme is oxidized by the reactant products and ceases to function as intended. The highest concentration of peracid disclosed by these references was 0.16% (1,600 mg/L). However, peracid or PAA solutions that are produced with enzymes have limited appeal because they are very expensive to produce, and for regulatory reasons, the enzyme must be removed from the solution before the solution can be applied to food or hard surfaces for disinfection purposes. Removing the enzyme from the solution is not an easy task; thus, it is typically not done. Therefore, PAA solutions prepared by enzymatic methods are not suitable for the more broad commercial uses in the food, dairy, beverage, meat, and poultry industries, which are regulated by the FDA and the Environmental Protection Agency (EPA).

[0010] Another method of generating non-equilibrium PAA at its point-of-use requires substituting the traditional mineral acid catalyst with sulfonic acid ion-exchange resins, as disclosed in U.S. patent no. 5,122,538. A solution containing a 1.5:1 mole ratio of AA to hydrogen peroxide was passed through a column packed with a sulfonic acid ion-exchange resin and produced a solution of 15% PAA within 30 minutes. The method described in the '538

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patent suffers from the limitation that it requires a large volume of expensive resin bed in order to be effective. Moreover, all existing ion exchange resin systems are limited by the fact the resin is subject to oxidative degradation by PAA and have a short limited lifespan.

[0011] Other attempts to produce non-equilibrium PAA solutions on site, at the point-of-use, for bleaching cellulosic materials have reacted hydrogen peroxide with acetic anhydride. For example, U.S. patent no. 3,432,546 discloses a process where hydrogen peroxide, acetic anhydride, and an ammonium hydroxide catalyst were metered to a tubular reactor to continuously produce a solution containing 3.25% PAA with a conversion of 78% hydrogen peroxide. However, the process generated measurable amounts of diacetyl peroxide (0.44%) which is an explosion hazard. Moreover, the reaction product would be unsuited for any application other than cellulosic bleaching purposes because there was no attempt to remove the ammonium hydroxide catalyst from the reaction medium. Ammonium hydroxide is an undesirable contaminant in PAA products that are used as disinfectants and sanitizers in the dairy, food, and beverage processing industries, and in PAA products used in fruit and vegetable washing and in the treatment of meat, poultry, and seafood.

[0012] Another process for generating non-equilibrium solutions of PAA on site, at the point-of-use was disclosed in U.S. Patent Application Publication No. 2009/0043132. This process utilized introduction of hydrogen peroxide into a sidestream of the water requiring treatment. This was followed by introducing acetic anhydride, whereupon PAA was generated *in-situ*. It was found that acetic anhydride preferentially reacted with hydrogen peroxide rather than undergo undesirable hydrolysis with water. Within 20 minutes, up to 3000 ppm of PAA was generated in the sidestream which was then reconstituted with the main body of water and diluted further. All processes that employ acetic anhydride suffer the limitation that acetic anhydride is expensive, very corrosive, an irritant, and highly flammable.

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[0013] Yet another process for generating non-equilibrium solutions of PAA on site at the point-of-use is disclosed in WIPO International Publication No. WO 01/46519 A1. This process utilized the metering of an aqueous solution of hydrogen peroxide into an agitated tank and co-metering a solid dry source of tetraacetylethylenediamine (TAED) from a storage hopper using a screw feeder. The agitator kept the solid TAED suspended in the hydrogen peroxide solution which was then fed to an in-line static mixer where aqueous sodium hydroxide was introduced. The mixture was then directed through 200 meters of coiled tubing immersed in a cooling tank so that the temperature rise accompanying the exothermic reaction was contained. Upon exiting the coiled tubing, the mixture containing PAA could be directed to the water requiring treatment. Disadvantages of this approach include the difficulty of accurately metering a solid and a liquid simultaneously, and the high capital equipment cost of the metering system, electronic controllers, agitation tank, coiled tubular reactor, and the cooling system.

[0014] Thus, there is a need for a method to make non-equilibrium PAA on site at the point-of-use that addresses the above problems.

[0015] **B. Solid PAA Bleaches and Stain Removers**

[0016] PAA is also used in laundry bleaching applications where it is generated *in-situ* in the laundry wash water. The PAA is typically produced from a solid source of hydrogen peroxide, such as sodium percarbonate or sodium perborate. The hydrogen peroxide must be in the presence of a solid acetyl precursor, most typically, tetraacetylethylenediamine (TAED). When dissolved in laundry wash water, the TAED undergoes a perhydrolysis reaction with hydrogen peroxide to form PAA. TAED is the preferred acetyl precursor because it possesses low toxicity, is of low environmental impact, and is readily biodegradable. However, TAED-based laundry bleaches have several problems. First, of the four acetyl groups on TAED, only two are known to be available for perhydrolysis, making TAED an expensive acetyl precursor on

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a weight basis. Second, TAED has low water solubility, especially at the cooler water temperature bleaching cycles that are less damaging to fabrics. This is a major drawback, as current consumer trends in energy conservation are towards bleaching laundry fabrics with cool temperature water, rather than heated water. Undissolved TAED in cool temperature water is less unavailable as an acetyl precursor for the dissolved source of hydrogen peroxide and can even deposit on fabrics, necessitating a separate rinse step to remove it. Third, over time, when exposed to high humidity, solid TAED can react with the solid source of hydrogen peroxide and the free water to form PAA, as well as degrade the activity, making it less effective over time. Because the PAA is volatile, it imparts an undesirable pungent odor to the product. Thus, there is a need for a solid, peroxygen bleach that overcomes the deficiencies of the TAED-containing bleaches.

SUMMARY OF THE INVENTION

[0017] In an embodiment, liquid compositions for use in generating non-equilibrium solutions of PAA are provided. These hydrogen peroxide-acetyl precursor solutions comprise a solution of aqueous hydrogen peroxide, a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide, a trace amount of peracetic acid, and water. A preferable acetyl precursor is triacetin, which displays an unexpectedly high solubility in hydrogen peroxide. These hydrogen peroxide-acetyl precursor solutions may be used for generating a non-equilibrium solution of peracetic acid for use as a disinfectant or sanitizer on a site having a point-of-use of peracetic acid.

[0018] The hydrogen peroxide-acetyl precursor solutions are advantageous in that they are formulated such that sufficient acetyl precursor can be dissolved in an amount of hydrogen peroxide that would fully perhydrolyze all of the acetyl groups of the acetyl precursor.

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Moverover, the hydrogen peroxide component of these compositions is remarkably stable. These compositions overcome one or more problems of the known prior art, and are free of the regulatory reporting, permitting, and shipping restrictions that govern equilibrium PAA products.

[0019] In another embodiment, a method of preparing a hydrogen peroxide-acetyl precursor solution is provided. The method comprises introducing a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide to a solution of aqueous hydrogen peroxide; and allowing the acetyl precursor and the aqueous hydrogen peroxide to mix to form a hydrogen peroxide-acetyl precursor solution.

[0020] A further embodiment is a method of continuously or intermittently generating a non-equilibrium solution of peracetic acid on a site having a point-of-use of peracetic acid for use as a disinfectant or sanitizer. Water is provided on the site having the point-of-use. The water may be a flowing aqueous stream or it may be contained in a mixing tank or other vessel. A hydrogen peroxide-acetyl precursor solution is provided on the site having the point-of-use. In one embodiment, the liquid acetyl precursor is triacetin and the solution that is formed is a hydrogen peroxide-triacetin solution. The hydrogen peroxide-acetyl precursor solution may be prepared off site and then transported to the site having the point-of-use, or the acetyl precursor and the aqueous hydrogen peroxide may be transported to the site having the point-of-use and then used to prepare the hydrogen peroxide-acetyl precursor solution on the site. The hydrogen peroxide-acetyl precursor solution is then introduced to the water. Alternatively, and in another embodiment, the acetyl precursor and the aqueous hydrogen peroxide may be introduced to the water separately, either sequentially, with either one first, or simultaneously. The hydrogen peroxide-acetyl precursor solution and the water are mixed to form a mixture. An aqueous source of an alkali metal or earth alkali metal hydroxide, such as sodium hydroxide, is provided on the site having the point-of-use. The aqueous source of an alkali metal or earth alkali metal

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hydroxide is added to the mixture. Alternatively, and in another embodiment, the aqueous source of alkali metal or earth alkali metal hydroxide may be added simultaneously with the hydrogen peroxide-acetyl precursor solution or with the acetyl precursor and the aqueous hydrogen peroxide, to the water. A reaction medium comprising a non-equilibrium solution of peracetic acid is formed. In the reaction medium, the hydrogen peroxide reacts with the acetyl precursor to form peracetic acid. Almost instantaneously, a non-equilibrium solution of PAA is formed. Within about 30 seconds to about five minutes, the amount of hydrogen peroxide and acetyl precursor that are converted into peracetic acid is maximized. When triacetin is used as the acetyl precursor and assuming all three acetyl groups are reacted, about 40.9% to about 85.7% of the triacetin is converted into peracetic acid, and the percent of hydrogen peroxide remaining is about 0.078% to about 1.88%. The reaction medium may optionally be sampled at various times after the addition of the aqueous source of alkali metal or earth alkali metal hydroxide to determine the time required to maximize the amount of hydrogen peroxide and acetyl precursor that are converted into peracetic acid.

[0021] The non-equilibrium solutions of PAA that are formed comprise PAA, unreacted hydrogen peroxide, unreacted acetyl precursor, the product of the perhydrolysis reaction of the acetyl precursor, the aqueous source of alkali metal or earth alkali metal hydroxide, and water. The peracetic acid solutions are alkaline, having a pH of about 11.2 to about 13.37. The reaction is remarkably fast, proceeds with a high conversion of the acetyl precursor into PAA. This method produces solutions with a high level of PAA, from about 1% to about 7.1%.

[0022] The PAA solution is introduced to the receiving water at the point-of-use. It may be introduced immediately, or stabilized with the addition of a source of acid and used throughout a working day. The receiving water may be water used in the dairy, food, and beverage processing industries for clean-in-place pipeline and equipment cleaning; for fruit and

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vegetable washing; and in the treatment of meat, poultry, and seafood products. The receiving water may also be cooling water, oil and gas process water, or municipal wastewater. Other uses include slime and biofilm removal in papermaking processes.

[0023] This method addresses one or more problems of the known prior art. The method is easy to perform, inexpensive to operate, and requires only basic and common equipment. The method is safe, in that it does not isolate pure PAA, and there is no possibility of a catastrophic event due to an equipment failure, such as over-pressurization or explosions. When triacetin is used as the acetyl precursor, it is inexpensive, non-toxic, safe, non-corrosive, non-irritating, non-flammable, sanctioned as Generally Recognized as Safe (GRAS) by the FDA, and converted quickly and with a high conversion rate into PAA. The method does not require the use of perhydrolase enzymes, acetic acid, or acetic anhydride, all of which suffer from the aforementioned limitations and problems. The method generates a PAA product that does not contain ammonium hydroxide. The method can be performed to generate a PAA product in a continuous or intermittent fashion. Further, the method allows the end-user the choice of utilizing the on site generated PAA immediately or later over the course of a working day.

[0024] In another embodiment, a freely-flowable, solid peroxygen composition for use in laundry bleaching and stain removal is provided. The solid composition comprises a liquid acetyl precursor, a water-soluble solid source of hydrogen peroxide, and a water-soluble solid source of alkalinity. The liquid acetyl precursor is preferably triacetin The composition produces peracetic acid upon being introduced to water. Upon dissolution, the solid hydrogen peroxide source releases hydrogen peroxide and the solid source of alkalinity dissolves to increase the pH, resulting in the formation of peracetic acid from the reaction of the hydrogen peroxide and the acetyl precursor.

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[0025] The solid composition is used as a bleaching agent and a stain remover for the treatment of articles such as fabrics, dentures, textile garments, and equipment used in the food and beverage industry. The solid composition is advantageous in that it does not give rise to an undesirable PAA odor upon storage, contains an acetyl precursor that efficiently utilizes the acetyl groups it contains, and has a high solubility in cool temperature water which quickly and efficiently produces PAA without the need for elevated water temperatures. Moreover, the acetyl precursor used in making the solid composition may also be used in preparing the liquid compositions described in section 3 above, streamlining manufacturing practices by using the same acetyl precursor as an ingredient in liquid as well as solid compositions.

[0026] Surprisingly, this freely-flowable, solid composition includes a liquid acetyl precursor, triacetin. In addition, it is surprising that this composition does not produce a strong odor of peracetic acid during storage.

[0027] In another embodiment, a method of preparing the freely-flowable, solid peroxygen composition for use in laundry bleaching and stain removal is provided.

BRIEF DESCRIPTION OF THE DRAWING

[0028] Figure 1 is a schematic representation of an example of a system that may be used to continuously or intermittently generate PAA for on a site having a point-of-use of peracetic acid In another embodiment, a freely-flowable, solid peroxygen composition for use in laundry bleaching and stain removal is provided according to an embodiment.

DETAILED DESCRIPTION OF THE INVENTION

[0029] 1. **Definitions**

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[0030] The terms "PAA" and "peracetic acid" mean peroxyacetic acid or peracetic acid, and/or the conjugate base of peracetic acid (the peracetate ion).

[0031] The terms "percent" and "%" mean weight percent, except when referring to the percent converted.

[0032] The term "receiving water" means the water that is being treated.

[0033] The term "point-of-use" means the location where the peracetic acid enters the receiving water.

[0034] **2. Experimental Methods**

[0035] PAA and hydrogen peroxide in solution were determined using the ceric sulfate-sodium thiosulfate method. The method involves adding a known weight of sample containing the PAA and hydrogen peroxide (or dilution) to a beaker containing 50 ml of chilled 0.9 N sulfuric acid. Using a transfer pipette, 1-2 grams is accurately weighed to a sample cup and reverse osmosis (RO) or deionized water is added to provide a workable volume. Five drops of ferroin indicator is then added with stirring, and the sample is then titrated with 0.1 N ceric sulfate until the sample turns from purple to blue. The volume of ceric sulfate required in mL is recorded. Then, one or two small scoops of potassium iodide are added to the sample cup, which turns the solution dark brown. The sample is then titrated with 0.1 N sodium thiosulfate to discharge the brown coloration and form a pale straw coloration. Then, 10-15 drops of 0.5% starch indicator solution is introduced and the sample turns blue-black. The sodium thiosulfate is then added dropwise until the color transitions to a bright orange. The volume of sodium thiosulfate in mL was recorded. The percent hydrogen peroxide and the percent PAA are calculated as follows:

$$\% \text{ hydrogen peroxide} = \frac{\text{mL 0.1 N ceric sulfate} \times 0.17}{\text{wt. sample}}$$

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$$\% \text{ PAA} = \frac{\text{mL 0.1 N sodium thiosulfate x 0.38}}{\text{wt. sample}}$$

[0036] For kinetic runs which involved measuring the PAA and hydrogen peroxide in samples at one, three, and five minute intervals after adding the reactants together, the generation reaction was quenched by adding a sample of the reaction medium to the sulfuric acid. This halted the generation reaction and stabilized the PAA and hydrogen peroxide to elevated pH degradation reactions. This permitted the samples to be taken at the appropriate time interval (one, three, and five minutes), and analyzed later.

[0037] In circumstances where low concentrations of PAA were used, the modified DPD method (U.S. patent no. 7,651,724) was employed. This analytical technique is based on a modified DPD (N,N-diethyl-*p*-phenylenediamine) colorimetric method accepted by the EPA for measuring total chlorine in drinking water and wastewater. It relies on the ability of PAA to behave like chlorine in that it rapidly and quantitatively oxidizes iodide ion (I^-) into iodine (I_2) that reacts with a color indicator (DPD), which turns the solution a shade of pink, the intensity of which is proportional to the concentration of the PAA. A colorimeter is used that is programmed to measure the intensity (absorbance) of the pink coloration and display the result in terms of ppm as Cl_2. A calculation converts this number into the ppm as PAA, based on the weight ratio of PAA to Cl_2 (76/71 = 1.07).

[0038] Hydrogen peroxide does not interfere with the measurement for PAA provided the analysis is completed within 30 seconds of introducing the DPD reagent. In order for the hydrogen peroxide to be measured, it must be activated by addition of a sodium molybdate catalyst and then given time (six minutes) to react with the I^- ion to liberate I_2. Upon addition of the DPD indicator, the intensity of the pink coloration measured by the colorimeter is now the

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sum of the PAA and hydrogen peroxide concentrations expressed as ppm Cl_2. After subtracting the ppm reading obtained earlier for the contribution due to PAA, a calculation is then used to convert this number into ppm as hydrogen peroxide, and is based on the weight ratio of hydrogen peroxide to Cl_2 (34/71 = 0.479).

[0039] 3. **Liquid Compositions for Generating Non-Equilibrium Solutions of PAA On a Site Having a Point-of-Use**

[0040] In an embodiment, liquid compositions for generating non-equilibrium solutions of PAA on a site having a point-of-use of peracetic acid are provided. These solutions are referred to as "hydrogen peroxide-acetyl precursor solutions." These solutions comprise aqueous hydrogen peroxide, a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide, a trace amount of peracetic acid, and water.

[0041] The hydrogen peroxide-acetyl precursor solutions are prepared by the following steps.

[0042] (a) Introducing a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide to a solution of aqueous hydrogen peroxide.

[0043] Triacetin is a preferable liquid acetyl precursor and 50% hydrogen peroxide is a preferable solution of aqueous hydrogen peroxide. The triacetin is preferably added to the 50% hydrogen peroxide at room temperature. Alternatively, 70% hydrogen peroxide can be used, but it is not widely available and requires special handling and transportation. Given that the solubility of triacetin in water is only 7% at 25°C, triacetin is unexpectedly highly soluble in 50% hydrogen peroxide. It solubilizes without an exotherm (temperature increase).

[0044] The mole ratio of hydrogen peroxide:triacetin is about 2.98:1 to about 12.84:1.

[0045] (b) Allowing the acetyl precursor and the aqueous hydrogen peroxide to mix to form a hydrogen peroxide-acetyl precursor solution.

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[0046] The solution is mixed until a homogenous solution has been formed. The solution is allowed to mix by diffusion or by using a mixing device that is suitable for mixing liquids together. For example, 50% hydrogen peroxide can be introduced to a batch tank equipped with an overhead agitator blade. Triacetin can then be introduced to the 50% hydrogen peroxide when the agitator is in motion so that the components are thoroughly mixed.

[0047] The solution that is formed is a hydrogen peroxide-acetyl precursor solution, which also includes varying amounts of water, depending on the formulation used. When triacetin is used as the liquid acetyl precursor, the solution that is formed is a hydrogen peroxide-triacetin solution. One such hydrogen peroxide-triacetin solution comprises about 23% to about 40% hydrogen peroxide (from 50% aqueous hydrogen peroxide), about 20% to about 52% triacetin, water, and a trace amount of PAA that is formed within the first day of preparing the hydrogen peroxide-acetyl precursor solution. The pH is about 1.46 to about 2.2 and no deliberate attempt is made to adjust the pH further.

[0048] A preferable hydrogen peroxide-triacetin solution comprises about 27.15% hydrogen peroxide (from 50% aqueous hydrogen peroxide), about 45.67% triacetin, water, and a trace amount of PAA that is formed within the first day of preparing the hydrogen peroxide-acetyl precursor solution. The mole ratio of hydrogen peroxide:triacetin is about 3.8:1.

[0049] The hydrogen peroxide-acetyl precursor solutions prepared by this method may be used to generate non-equilibrium solutions of PAA on a site having a point-of-use of peracetic acid, in the methods described in section 4 below. The hydrogen peroxide-acetyl precursor solutions may be transported in any container suitable for transporting liquids, such as plastic tote bins of 275-330 gallons, drums of 15-55 gallons, five-gallon pails, one-gallon jugs, or other containers.

[0050] Example 1

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[0051] Triacetin (83.05 g) was added to 50% hydrogen peroxide solution (75 g) to form 158.05 g of a mixture. There was no evolution of gas indicating the hydrogen peroxide had decomposed to oxygen, nor was there any exotherm indicating that a chemical reaction had occurred. The solution did not turn oily or cloudy but remained clear. The solution was calculated to comprise 23.73% hydrogen peroxide and 52.55% triacetin, with the balance being water, and had a measured pH of 2.2. The mole ratio of hydrogen peroxide to triacetin was calculated to be 2.98:1. Titration of the solution immediately upon preparation yielded 24.1% hydrogen peroxide, which is within experimental error of the calculated amount of hydrogen peroxide.

[0052] This experiment showed that triacetin is remarkably soluble in 50% hydrogen peroxide. The solubility of triacetin was found to be at least 52.55%. This is surprising, given that the solubility of triacetin in water is only 7% at 25°C.

[0053] In order to test the stability of the mixture, the amount of hydrogen peroxide remaining and the amount of PAA generated was tracked for several months. The mixture was held at ambient temperature away from sources of light. Table I summarizes the data.

[0054] Table I

Day #	% Hydrogen Peroxide Remaining	% PAA Generated
1	23.79	0.00
29	23.32	1.28
57	22.56	2.36
92	21.06	4.22
120	19.54	6.05
134	18.57	6.87

[0055] Table I shows that although there was a steady depletion in hydrogen peroxide, it was accompanied by an increase in PAA. By the end of the 134-day study, the composition had

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lost 5.22% hydrogen peroxide. Had all of the depleted hydrogen peroxide gone towards the generation of PAA, 11.67% PAA would have been generated. Because 6.87% PAA was generated, unaccounted losses of hydrogen peroxide amounted to 2.15%. This loss of hydrogen peroxide was surprisingly small given that hydrogen peroxide is often used to destroy organic compounds by oxidation and the composition originally contained 52.55% of triacetin, an organic compound.

[0056] Example 2

[0057] A formulation containing hydrogen peroxide and triacetin in a mole ratio of 12.84:1 was prepared by adding triacetin (100.0 g) to 50% hydrogen peroxide (400.0 g) so that the mixture was 40% hydrogen peroxide and 20% triacetin, with the balance being water. The pH of the solution was 1.46 and the pH of a 1:100 dilution was 3.69. To accelerate storage characteristics, the sample was placed in an incubator set for 86-87° F. Table II shows the percent of hydrogen peroxide remaining and the percent of PAA generated over a period of more than six months.

[0058] Table II

Days of Study	% Hydrogen Peroxide Remaining	% PAA Generated
1	40.21	0.004
33	38.02	3.499
54	35.5	5.992
96	29.27	8.36
127	25.05	6.66
173	18.49	4.43
201	15.67	3.51

[0059] Table II indicates that after 96 days, the generation of PAA maximized at 8.36% and corresponded to a loss of 10.94% hydrogen peroxide. Had all of the depleted hydrogen

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peroxide gone toward the generation of PAA, 24.45% PAA would have been generated. Because only 8.36% PAA was generated, unaccounted losses of hydrogen peroxide amounted to 7.2%.

[0060] Example 3

[0061] A formulation containing hydrogen peroxide and triacetin in a mole ratio of 5.8:1 was prepared by adding triacetin (37.5008 g) to 50% hydrogen peroxide (65.2504 g) so that the mixture was 31.75% hydrogen peroxide and 36.5% triacetin, with the balance being water. The pH of the solution was 1.57 and the pH of a 1:100 dilution was 5.17. To accelerate degradation, the sample was placed in an incubator set for 86-87° F. Table III shows the percent of hydrogen peroxide remaining and the percent of PAA generated over a period of six days.

[0062] Table III

Days of Study	% Hydrogen Peroxide Remaining	% PAA Generated
1	31.72	0.33
6	31.63	0.716

[0063] In this study, after six days, the mixture had lost only 0.09% hydrogen peroxide, yet had generated an additional 0.386% PAA. Had all the loss of hydrogen peroxide been due to the formation of PAA, only an additional 0.201% would have been generated. It is clear that the analytical methods employed for solutions that are high in hydrogen peroxide but low in PAA are subject to detection limit errors.

[0064] Example 4

[0065] A formulation containing hydrogen peroxide and triacetin in a mole ratio of 3.2:1 was prepared by adding triacetin (35.0 g) to 50% hydrogen peroxide (35.0 g) so that the mixture was 25% hydrogen peroxide and 50% triacetin, with the balance being water. The pH of the solution was 1.66 and the pH of a 1:100 dilution was 4.09. To accelerate degradation, the sample

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was placed in an incubator set for 86-87° F. Table IV shows the percent of hydrogen peroxide remaining and the percent of PAA generated over a period of 22 days.

[0066] Table IV

Days of Study	% Hydrogen Peroxide Remaining	% PAA Generated
1	25	0.62
8	24.51	1
15	24.17	1.17
22	23.68	1.14

[0067] Table IV indicates that after 15 days, the generation of PAA maximized at 1.17% and corresponded to a loss of 0.83% hydrogen peroxide. Had all of the depleted hydrogen peroxide gone toward the generation of PAA, 1.86% PAA would have been generated. Because only 1.17% PAA was generated, unaccounted losses of hydrogen peroxide amounted to 0.31%.

[0068] Example 5

[0069] A formulation containing hydrogen peroxide and triacetin in a mole ratio of 4.8:1 was prepared by adding triacetin (37.50 g) to 50% hydrogen peroxide (56.25 g) so that the mixture was 30% hydrogen peroxide and 40% triacetin, with the balance being water. The pH of the solution was 1.67 and the pH of a 1:100 dilution was 4.62. To accelerate degradation, the sample was placed in an incubator set for 86-87° F. Table V shows the percent of hydrogen peroxide remaining and the percent of PAA generated over a period of 43 days.

[0070] Table V

Days of Study	% Hydrogen Peroxide Remaining	% PAA Generated
1	30.08	0.38
8	29.85	0.784
15	29.63	1.22
22	29.37	1.71
29	28.76	2.26
36	28.67	2.78
43	28.31	3.41

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[0071] Table V shows that although there was a steady depletion in hydrogen peroxide, it was accompanied by the associative increase of PAA. At the end of the 43-day study, the composition had lost 1.77% hydrogen peroxide. Had all of the depleted hydrogen peroxide gone toward the generation of PAA, 3.96% PAA would have been generated. Because only 3.41% PAA was generated, unaccounted losses of hydrogen peroxide amounted to 0.24%.

[0072] Example 6

[0073] A formulation containing hydrogen peroxide and triacetin in a mole ratio of 6.42:1 was prepared by adding triacetin (25.0007 g) to 50% hydrogen peroxide (50.0007 g) so that the mixture was 33.33% hydrogen peroxide and 33.33% triacetin, with the balance being water. The pH of the solution was 1.56 and the pH of a 1:100 dilution was 5.15. To accelerate degradation, the sample was placed in an incubator set for 86-87° F. Table VI shows the percent of hydrogen peroxide remaining and the percent of PAA generated over a period of 36 days.

[0074] Table VI

Days of Study	% Hydrogen Peroxide Remaining	% PAA Generated
1	33.13	0.81
8	32.88	1.35
15	32.61	1.88
22	31.69	2.58
29	31.75	3.03
36	31.29	3.28

[0075] Table VI shows that although there was a steady depletion in hydrogen peroxide, it was accompanied by an associative increase of PAA. At the end of the 36-day study, the composition had lost 1.84% hydrogen peroxide. Had all of the depleted hydrogen peroxide gone toward the generation of PAA, 4.11% PAA would have been generated. Because only 3.28% PAA was generated, unaccounted losses of hydrogen peroxide amounted to 0.37%.

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[0076] Example 7

[0077] A 30-gallon batch of 50% hydrogen peroxide and triacetin was prepared by blending 137 lbs triacetin with 163 lbs 50% hydrogen peroxide so that the mixture was 27.15% hydrogen peroxide and 45.67% triacetin, with the balance being water. Thus, the mole ratio of hydrogen peroxide:triacetin was 3.8:1. The mixture was stored in an opaque drum in a non-climate controlled environment. On storage, the mixture slowly formed PAA in the container. Since this reaction of hydrogen peroxide to PAA does not represent a destructive loss of total peroxygen, to determine the true stability of the hydrogen peroxide, both the hydrogen peroxide and the peracetic acid generated must be quantified and reported as total peroxygen recovered.

[0078] After 295 days of storage, the mixture was analyzed for total peroxygen recovered (expressed as hydrogen peroxide) to determine the percent loss of peroxygen. Using the ceric sulfate–iodometric titration method, the PAA generated and the hydrogen peroxide remaining were measured. The total peroxygen recovered after 295 days and the theoretical initial concentrations are shown in Table VII. All peroxygen is expressed as % hydrogen peroxide.

[0079] Table VII

	Initial Theoretical %	% (after 295 days)
Hydrogen Peroxide Recovered	27.17	15.20
PAA formed	0	15.57
Hydrogen Peroxide Reacted to form PAA	0	6.97
Total Recovered Hydrogen Peroxide	27.17	22.17
Total Hydrogen Peroxide Unproductive Loss		5.00

[0080] After 295 days, 22.17% of the total peroxygen was recovered as either titratable hydrogen peroxide or hydrogen peroxide reacted to form PAA. To determine the stability of the peroxygen in the mixture, the total recovered hydrogen peroxide after 295 days (22.17%) was

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subtracted from the total recovered hydrogen peroxide of the initial theoretical value (27.17%). The result, 5%, is the loss of peroxygen (expressed as hydrogen peroxide) after 295 days of storage in a non-climate controlled environment.

4. Methods of Generating Non-Equilibrium PAA On Site at the Point-of-Use

[0081] Methods of continuously or intermittently generating non-equilibrium solutions of PAA on a site having a point-of-use of PAA for use as a disinfectant or sanitizer, using a source of water that is an aqueous stream or by a batch process using water in a container, are provided.

[0082] In one embodiment, a method of continuously or intermittently generating non-equilibrium solutions of PAA on a site having a point-of-use, using a source of water that is an aqueous stream, comprises the following steps.

[0083] (a) Providing water.

[0084] Flowing water is provided on a site having a point-of-use of peracetic acid.

[0085] The water may be flowing in, for example, a pipe, a flume, a canal, or other types of aqueous streams. The water pressure should be regulated and the flow rate should be monitored or measured. Any suitable flow meter may be used, such as a rotameter, a magnetic flow meter, an ultrasonic flow meter, a Doppler flow meter, a differential-pressure flow meter, a turbine flow meter, or a Coriolis flow meter.

[0086] The water should be softened, deionized, or of sufficient low hardness that it will not precipitate calcium salts when the alkali metal or earth alkali metal hydroxide is introduced in step (d).

[0087] (b) Introducing a hydrogen peroxide-acetyl precursor solution to the water.

[0088] A hydrogen peroxide-acetyl precursor solution is provided on the site having the point-of-use of the PAA. The hydrogen peroxide-acetyl precursor solution may be prepared by the steps set forth in section 3 above. The hydrogen peroxide-acetyl precursor solution may be

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prepared off site and then transported to the site having the point-of-use, or the acetyl precursor and the aqueous hydrogen peroxide may be transported to the site having the point-of-use and used to prepare the hydrogen peroxide-acetyl precursor solution on the site.

[0089] A preferable liquid acetyl precursor is triacetin. When triacetin is used, a hydrogen peroxide-triacetin solution is introduced.

[0090] The hydrogen peroxide-acetyl precursor solution is introduced to the flowing water in an amount such that the hydrogen peroxide-acetyl precursor solution is about 5.6% to about 22.5% of the total.

[0091] The hydrogen peroxide-acetyl precursor solution is pumped from its container into the aqueous water stream, for example, through an injection quill mounted on the pipe. Any suitable pump capable of overcoming the pressure of the water flowing in the pipe may be used. Examples include a solenoid-driven or air-driven diaphragm pump or a peristaltic pump. The rate at which the hydrogen peroxide-acetyl precursor solution is pumped into the flowing water is governed by a controller that is interfaced to a flow meter that is measuring the flow of the water. In this way, the rate at which the hydrogen peroxide-acetyl precursor solution is pumped may be matched to the flow of the water in the aqueous stream. If the flow of the water slows, the rate at which the hydrogen peroxide-acetyl precursor solution is pumped should slow accordingly. If the flow of the water stops, the hydrogen peroxide-acetyl precursor pump should stop.

[0092] As an alternative to step (b), the liquid acetyl precursor and the solution of aqueous hydrogen peroxide may be introduced to the water separately, either simultaneously or sequentially. If they are introduced sequentially, either one may be added first. If the liquid acetyl precursor and the aqueous hydrogen peroxide are introduced separately, rather than as an hydrogen peroxide-acetyl precursor solution, then in step (c) the liquid acetyl precursor and the aqueous hydrogen peroxide are mixed with the water to form a mixture.

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[0093] (c) Mixing the hydrogen peroxide-acetyl precursor solution and the water to form a mixture.

[0094] Any mixing device suitable for mixing liquids may be used. An example is a static mixer located just after the point that the hydrogen peroxide-acetyl precursor solution is introduced to the flowing water. One type of static mixer utilizes a non-moving element such as a series of baffles. As the mixture flows through the static mixer under the motive force of the flowing water, the non-moving element divides the flow several times to provide radial mixing. Another type of static mixer utilizes a series of obstructions, such as column packing or glass beads, provided there is a low differential pressure drop across the mixer. The obstructions provide for turbulent mixing of the hydrogen peroxide-acetyl precursor solution and the flowing water. The mixing should yield a homogeneous solution with no concentration gradients before the next step is performed. The velocity of the water will determine the time it takes to complete the mixing and the efficiency of the mixing. For example, with ¾" pipe, a static mixer of ¾" diameter and 6" long, and a velocity of about 1 gal/min., mixing should be accomplished in less than about one second.

[0095] (d) Adding an aqueous source of an alkali metal or earth alkali metal hydroxide to the mixture.

[0096] An aqueous source of an alkali metal or earth alkali metal hydroxide is provided on the site having the point-of-use. A preferable aqueous source of an alkali metal hydroxide is sodium hydroxide, and 50% sodium hydroxide is most preferable. Other suitable alkali metal hydroxides include 45% potassium hydroxide.

[0097] Sufficient 50% NaOH is added to the flowing mixture such that the amount of sodium hydroxide is about 1.82% to about 7.28% of the total amount. When the acetyl precursor is triacetin, a preferred mole ratio of NaOH: hydrogen peroxide:triacetin is about 4.2:3.8:1.

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[0098] The 50% NaOH solution is added to the mixture by pumping it from its container with a suitable pumping device and into the mixture through an injection quill mounted on the pipe. Any suitable pump capable of overcoming the hydraulic pressure of the pipe may be used. Examples include a solenoid-driven or air-driven diaphragm pump or a peristaltic pump.

[0099] The rate at which the 50% NaOH solution is pumped into the pipe is governed by the same controller that is interfaced to both the flow meter that is measuring the flow of the mixture and the pump controlling the rate of introduction of the hydrogen peroxide-acetyl precursor solution. In this way, the rate at which the 50% NaOH solution is introduced may be matched to both the flow of the mixture and the rate of introducing the hydrogen peroxide-acetyl precursor solution. If the flow of the mixture slows for any reason, the rate at which the 50% NaOH solution is pumped should slow accordingly. If the flow of the mixture stops, the 50% NaOH pump should stop as well.

[00100] Step (d) may be performed after step (c), or it may be performed simultaneously with step (b).

[00101] (e) Forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

[00102] The reaction medium that is formed in this step almost instantaneously forms a non-equilibrium solution of PAA. The hydrogen peroxide reacts with the acetyl precursor to form peracetic acid. Depending upon the temperature of the water, the efficiency of mixing, and the mole ratio of NaOH:hydrogen peroxide:acetyl precursor employed, the amount of hydrogen peroxide and acetyl precursor that are converted into PAA is maximized within about 30 seconds to about five minutes.

[00103] The non-equilibrium solutions of PAA prepared by this method comprise PAA, unreacted hydrogen peroxide, unreacted acetyl precursor, the product of the perhydrolysis

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reaction of the acetyl precursor, the aqueous source of alkali metal or earth alkali metal hydroxide, and water. When the acetyl precursors is triacetin, the product of the perhydrolysis reaction is 1, 2, 3-propanetriol (glycerine).

[00104] This method may also include an optional step after step (e) of sampling the reaction medium at various times after the addition of the aqueous source of alkali metal or earth alkali metal hydroxide to determine the time required under the existing conditions to maximize the amount of hydrogen peroxide and acetyl precursor that are converted into PAA. This may be accomplished by directing the flow of the reaction medium through a series of residence chambers equipped with sampling ports. The residence chambers are designed to be of sufficient volume such that a certain amount of time has elapsed since the introduction of the 50% NaOH. Thus, at any given flow rate in the pipe, the first residence chamber is of a volume such that 30 seconds have elapsed since the introduction of 50% NaOH, one minute has elapsed by the time the flow reaches the sampling port immediately after the second residence chamber, and additional time elapses by the time the flow reaches the remaining sampling ports. Up to four residence chambers and sampling ports may be placed in series such as this to provide the user with about 30 seconds to five minutes of residence time after the introduction of the 50% NaOH. Once the flow of the reaction medium has been established, samples are drawn from all four sampling ports associated with the residence chambers and analyzed for peracetic acid. The sample which registers the highest amount of peracetic acid is deemed to be drawn from the sampling port associated with the residence chamber corresponding to the time of maximum conversion of the acetyl precursor into peracetic acid. At this point, the entire flow of reaction medium is directed through the sampling point of maximum conversion of acetyl precursor and to the point-of-use.

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[00105] In another embodiment, a method of continuously or intermittently generating a non-equilibrium solution of PAA on a site having a the point-of-use in a batch process comprises the following steps:

[00106] (a) Providing water.

[00107] A container of water, such as a mixing tank or other vessel, is provided on a site having a point-of-use of peracetic acid. The water should be softened, deionized, or of sufficient low hardness that it will not precipitate calcium salts when the alkali metal or earth alkali metal hydroxide is introduced in step (d).

[00108] (b) Introducing a hydrogen peroxide-acetyl precursor solution to the water.

[00109] A hydrogen peroxide-acetyl precursor solution is provided on the site having the point-of-use of the PAA. The hydrogen peroxide-acetyl precursor solution may be prepared by the steps set forth in section 3 above. The hydrogen peroxide-acetyl precursor solution may be prepared off site and then transported to the site having the point-of-use, or the acetyl precursor and the aqueous hydrogen peroxide may be transported to the site having the point-of-use and used to prepare the hydrogen peroxide-acetyl precursor solution on the site.

[00110] A preferable liquid acetyl precursor is triacetin. When triacetin is used, a hydrogen peroxide-triacetin solution is introduced.

[00111] The hydrogen peroxide-acetyl precursor solution is introduced to the water in an amount such that the hydrogen peroxide-acetyl precursor solution is about 5.6% to about 22.5% of the total.

[00112] The hydrogen peroxide-acetyl precursor solution is pumped from its container into the container of water through an injection quill mounted on the side of the container of water. Any suitable pump may be used. Examples include a solenoid-driven or air-driven diaphragm pump or a peristaltic pump.

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[00113] As an alternative to step (b), the liquid acetyl precursor and the solution of aqueous hydrogen peroxide may be introduced to the water separately, either simultaneously or sequentially. If they are introduced sequentially, either one may be added first. If the liquid acetyl precursor and the aqueous hydrogen peroxide are introduced separately, rather than as an hydrogen peroxide-acetyl precursor solution, then in step (c) the liquid acetyl precursor and the aqueous hydrogen peroxide are mixed with the water to form a mixture.

[00114] (c) Mixing the hydrogen peroxide-acetyl precursor solution and the water to form a mixture.

[00115] Any mixing device suitable for mixing liquids may be used. An example is an overhead stirrer, such as an agitator blade. The container of water is equipped with a baffle to assist the process of mixing. The mixing should yield a homogeneous solution with no concentration gradients before the next step is performed.

[00116] (d) Adding an aqueous source of an alkali metal or earth alkali metal hydroxide to the mixture.

[00117] An aqueous source of an alkali metal or earth alkali metal hydroxide is provided on the site having the point-of-use. A preferable aqueous source of an alkali metal hydroxide is sodium hydroxide, and 50% sodium hydroxide is most preferable. Other suitable alkali metal hydroxides include 45% potassium hydroxide.

[00118] Sufficient 50% NaOH is added to the mixture such that the amount of sodium hydroxide is about 1.82% to about 7.28% of the total amount. When the acetyl precursor is triacetin, a preferred mole ratio of NaOH: hydrogen peroxide:triacetin is about 4.2:3.8:1.

[00119] The 50% NaOH solution is added to the mixture by pumping it from its container with a suitable pumping device and into the mixture through an injection quill mounted on the

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side of the container of water. Any suitable pump may be used. Examples include a solenoid-driven or air-driven diaphragm pump or a peristaltic pump.

[00120] Step (d) may be performed after step (c), or it may be performed simultaneously with step (b).

[00121] (e) Forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

[00122] The reaction medium that is formed in this step almost instantaneously forms a non-equilibrium solution of PAA. The hydrogen peroxide reacts with the acetyl precursor to form peracetic acid. Depending upon the temperature of the water, the efficiency of mixing, and the mole ratio of NaOH:hydrogen peroxide:acetyl precursor employed, the amount of hydrogen peroxide and acetyl precursor that are converted into PAA is maximized within about 30 seconds to about five minutes.

[00123] The non-equilibrium solutions of PAA prepared by this method comprise PAA, unreacted hydrogen peroxide, unreacted acetyl precursor, the product of the perhydrolysis reaction of the acetyl precursor, the aqueous source of alkali metal or earth alkali metal hydroxide, and water. When the acetyl precursors is triacetin, the product of the perhydrolysis reaction is 1, 2, 3-propanetriol (glycerine).

[00124] While not wishing to be bound by theory, it is believed that under elevated pH conditions created by the addition of a source of alkali metal hydroxide or earth alkali metal hydroxide, hydrogen peroxide dissociates according to the following equation:

$$H_2O_2 = H^+ + HO_2^-$$

[00125] The perhydroxyl anion (HO_2^-) then affects nucleophilic substitution reactions on the carbonyl groups of the acetyl precursor in a perhydrolysis reaction to form peracetic acid and the product of the perhydroloysis reaction of the acetyl precursor. When the acetyl precursor is

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triacetin and all three acetyl groups are reacted, then the product of the perhydrolysis reaction is 1, 2, 3-propanetriol (glycerine).

[00126] In these methods, the perhydrolysis reaction is rapid and the maximum amount of PAA is generated within about 30 seconds to about five minutes at ambient temperature. The methods efficiently utilize the acetyl precursor and the source of hydrogen peroxide. When triacetin is used, assuming all three acetyl groups are reacted, about 40.9% to about 85.7% of the triacetin is converted into PAA. The percent of hydrogen peroxide remaining is about 0.078% to about 1.88%.

[00127] The non-equilibrium solutions of PAA prepared by these methods have several unique characteristics. The PAA solutions have high levels of PAA, from about 1% to about 7.1%. In addition, the PAA solutions are alkaline, having a pH of about 11.2 to about 13.37.

[00128] The non-equilibrium solutions of PAA may be immediately introduced to the receiving water at the point-of-use. Alternatively, the solutions may be stabilized by halting the decomposition of the PAA by adding a source of acid to lower the elevated pH of the reaction medium caused by the addition of an alkali metal or earth alkali metal hydroxide, and to provide neutral to mildly acid pH conditions to stabilize the PAA to elevated pH degradation. The acid-stabilized PAA solutions can then be stored and used at the point-of-use as required throughout a working day.

[00129] The non-equilibrium PAA solutions produced by these methods can be advantageously used wherever traditional equilibrium solutions of PAA are used. Thus, the PAA solutions may be used as a disinfectant and sanitizer in the dairy, food, and beverage processing industries for clean-in-place pipeline and equipment cleaning; for fruit and vegetable washing; and in the treatment of meat, poultry, and seafood products. These PAA solutions may also be

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used in the treatment of cooling water, oil and gas process water, and municipal wastewater. Other uses include slime and biofilm removal in papermaking processes.

[00130] For both of the above methods, steps (a) through (e) may be performed on a continuous or intermittent basis. In a continuous basis, the steps are performed under conditions of steady state, and the PAA solution is produced at the same rate as a function of time. However, intermittent performing of steps (a) through (e) is possible if the demand for the PAA solution drops for any reason (e.g., change in working shift or the target level of PAA in the receiving water has been achieved), in which case, steps (a) through (e) may be discontinued and resumed when the demand increases.

[00131] Example 8

[00132] Tests were performed using about one liter of reverse osmosis (RO) or softened water which was buffered to an alkaline pH. The buffering media included 0.1 and 0.2 M Na_2CO_3 solutions, in addition to 0.1 and 0.2 M NaOH solutions. Sufficient buffering media was added to the water in order to achieve the desired pH. The amount of hydrogen peroxide (introduced as 50% hydrogen peroxide) and triacetin used in the tests is summarized in Table VIII. The reactants were introduced to the solution in one of three ways: simultaneously (referred to in Table VIII as "double"), sequentially (triacetin followed shortly by hydrogen peroxide), or by mixing the hydrogen peroxide and triacetin together and adding them in a single charge (referred to in Table VIII as "mixed"). All testing was performed at ambient temperature as the one-liter beaker was stirred with a magnetic stir bar. Using the ceric sulfate–iodometric titration method, the percent of PAA generated and the percent of hydrogen peroxide remaining were measured five minutes after introduction of the reactants to the buffer. The percent of the triacetin acetyl donor converted to PAA was calculated, assuming all three acetyl groups were available for perhydrolysis.

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[00133] Table VIII

t #	Moles Hydrogen Peroxide/ Mole Triacetin	Type of Addition (Double, Sequential or Mixed)	pH of Buffer	Hydrogen Peroxide Used (Wt., g)	Triacetin Used (Wt., g)	% PAA Generated	% Hydrogen Peroxide Remainin g	% Triacetin Converted to PAA
	4.99	Double	10.50	8.60	5.53	0.170	0.329	29.6%
	5.47	Double	10.50	8.60	5.04	0.178	0.333	33.9%
	5.45	Double	10.80	8.61	5.07	0.192	0.329	36.4%
	5.68	Double	11.00	8.87	5.01	0.201	0.322	38.6%
	5.72	Double	11.00	9.13	5.12	0.068	0.416	12.8%
	5.49	Double	12.42	8.60	5.02	0.169	0.306	32.4%
	5.59	Double	12.91	8.60	4.93	0.320	0.265	62.4%
	5.53	Double	12.80	8.69	5.04	0.335	0.273	63.9%
	25.0	Double	12.65	9.03	1.16	0.104	0.413	85.9%
0	5.26	Double	12.60	8.70	5.30	0.298	0.247	54.1%
1	5.52	Sequential	12.83	8.68	5.04	0.276	0.248	52.4%
2	5.47	Sequential	12.81	17.11	10.03	0.120	0.371	11.4%
3	28.8	Mixed	11.12	8.74	0.973	0.039	0.398	38.9%
4	28.8	Mixed	11.99	8.75	0.974	0.038	0.374	37.9%
5	28.8	Mixed	13.02	8.69	0.967	0.055	0.367	55.2%
6	28.8	Mixed	7.91	8.70	0.968	0.024	0.435	23.7%
7	28.8	Mixed	11.22	8.76	0.975	0.029	0.309	28.4%
8	28.8	Mixed	12.07	8.68	0.966	0.028	0.311	27.7%
9	28.8	Mixed	13.22	8.72	0.971	0.026	0.321	25.6%
lup	25.2	Mixed	12.97	8.77	1.12	0.031	0.249	26.5%
0	25.2	Mixed	12.98	9.08	1.16	0.066	0.393	54.5%
lup	25.2	Mixed	13.00	9.10	1.16	0.074	0.409	61.0%
1	25.2	Mixed	13.02	9.10	1.16	0.02	0.261	16.5%
lup	25.2	Mixed	12.99	9.12	1.16	0.017	0.376	14.0%
2	16.0	Mixed	12.99	5.80	1.16	0.070	0.246	57.7%
3	16.0	Mixed	13.05	5.79	1.16	0.013	0.189	10.7%
4	16.0	Mixed	12.55	5.78	1.16	0.031	0.234	25.6%
5	16.0	Mixed	12.49	5.80	1.16	0.013	0.144	10.7%
6	16.0	Mixed	12.01	5.85	1.17	0.016	0.259	13.1%
7	16.0	Mixed	12.05	5.82	1.16	0.011	0.080	9.0%
8	16.0	Mixed	10.50	0.601	0.120	0.00077	0.028	6.1%
9	16.0	Mixed	10.97	0.595	0.119	0.00023	0.021	1.8%
0	16.0	Mixed	10.50	0.614	0.123	0.00012	0.019	0.9%
2	16.0	Mixed	10.97	0.595	0.119	0.00035	0.018	2.8%

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[00134] Table VIII shows that the amount of PAA generated was independent of the method of introducing the triacetin and hydrogen peroxide to the buffer solutions. It can be seen that the pH of the buffer had a significant effect on the percent of the triacetin that was converted into PAA. Only when the pH was above 12 was a meaningful percent of the triacetin converted into PAA.

[00135] Example 9

[00136] A mixture of triacetin and hydrogen peroxide was prepared by blending 50% hydrogen peroxide (63.4%) and triacetin (36.6%). Thus, the mole ratio of hydrogen peroxide:triacetin was 5.57:1. The mixture (13.68 g) was added to soft water such that the final solution contained 5 g/L of triacetin and had a pH of 4.1. Then, in successive experiments, sufficient 50% NaOH was added to the beaker so that the mole ratio of NaOH:TA was varied from 3:1 (obtained by introducing 5.49 g of 50% NaOH) to 8:1 (obtained by introducing 14.68 g of 50% NaOH). Using the ceric sulfate–iodometric titration method, the percent of PAA generated and the percent of hydrogen peroxide remaining were measured over the next 20 minutes. The method was performed in duplicate for each sample point. The averages of the results for the 6:1 mole ratio of NaOH:TA are shown in Table IX.

[00137] Table IX

Time (min)	% PAA Generated	% Hydrogen Peroxide Remaining	% Triacetin Converted to PAA (3 acetyls)
0	0.015	0.437	2.9
1	0.275	0.298	53.1
3	0.312	0.274	60.2
5	0.326	0.258	62.9
11	0.302	0.233	58.3
21	0.246	0.199	47.5

[00138] It can be seen that even before the introduction of 50% NaOH, some PAA was present in solution as a result of being generated in the diluted mixture of the two components.

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Upon addition of the NaOH, PAA was immediately generated, with the maximum amount of 0.326% occurring at five minutes, corresponding to a yield of 62.9% assuming all three acetyl groups of triacetin were available for perhydrolysis.

[00139] This exercise was repeated for mole ratios of NaOH:triacetin ranging from 3:1 to 8:1. Table X summarizes the next set of data and shows the maximum percent of PAA generated, the time after the addition of NaOH that it took to reach the maximum percent of PAA generated, the pH after the addition of the NaOH, the percent of hydrogen peroxide remaining at the time of the maximum percent of PAA, and the calculated percent of triacetin converted to PAA assuming that all three acetyl groups were available for perhydrolysis.

[00140] Table X

Mole ratio NaOH:TA	Time at Max. PAA (min)	Max % PAA Generated	% Hydrogen Peroxide Remaining	% Triacetin Converted to PAA (3 acetyls)	pH
3:1	3	0.229	0.291	43.8	11.2
4:1	6	0.285	0.251	54.8	11.3
6:1	5	0.326	0.258	62.9	11.6
8:1	3	0.335	0.271	64.7	12.2

[00141] It can be seen that the amount of PAA generated, and hence the conversion of triacetin into PAA, increased with the increasing mole ratio of NaOH:TA. For all four mole ratios employed, the maximum amount of PAA was generated between three and six minutes. The optimum mole ratio of NaOH:TA was 6:1 as it resulted in an efficient utilization of triacetin converted into PAA, and because the pH of the resulting reaction medium would have less impact on the pH of the receiving water than the higher pH of the reaction medium resulting from the 8:1 mole ratio.

[00142] Example 10

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[00143] A series of experiments was then performed in which the mole ratio of NaOH:TA was fixed at 8:1 and the mole ratio of hydrogen peroxide:triacetin was varied from 2:1 to 8:1. Triacetin (5 g) was added to about 965 mL of softened water along with 50.7% hydrogen peroxide in successive tests (3.06 g for the 2:1 mole ratio; 6.19 g for the 4:1 mole ratio; 9.23 g for the 6:1 mole ratio; and 12.32 g for the 8:1 mole ratio) to yield a solution with a pH of about 4.8-5.5. The volume of the mixture was adjusted to 990 mL with softened water. With mixing, 50% NaOH (14.67 g) was introduced. Using the ceric sulfate–iodometric titration method, the percent of PAA generated and the percent of hydrogen peroxide remaining were measured over the next 20 minutes. The method was performed in duplicate for each sample point. The averages of the results for the kinetic run employing the mole ratio of 8:6:1 NaOH:hydrogen peroxide:TA are shown in Table XI.

[00144] Table XI

Time (min)	% PAA Generated	% Hydrogen Peroxide Remaining	% Triacetin Converted to PAA (3 acetyls)
1	0.331	0.317	63.8
3	0.330	0.303	63.6
5	0.319	0.303	61.5
10	0.300	0.295	57.8
20	0.256	0.274	49.3

[00145] Upon addition of the NaOH, PAA was immediately generated, with the maximum amount of 0.331% occurring after just one minute, corresponding to a calculated conversion of triacetin into PAA of 63.8%, assuming that all three acetyl groups of triacetin were available for perhydrolysis.

[00146] This exercise was repeated for mole ratios of hydrogen peroxide:triacetin ranging from 2:1 to 6:1. Table XII summarizes the data and shows the maximum percent of PAA generated, the time after the addition of NaOH that it took to reach the maximum percent of PAA

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generated, the pH after the addition of the NaOH, the percent of hydrogen peroxide remaining at the time of the maximum percent of PAA, and the calculated percent of triacetin converted to PAA assuming that all three acetyl groups were available for perhydrolysis.

[00147] Table XII

Mole Ratio Hydrogen Peroxide:Triacetin	Time at Max. PAA (min)	Max % PAA Generated	% Hydrogen Peroxide Remaining	% Triacetin Converted to PAA (3 acetyls)	pH
2:1	3	0.214	0.079	40.9	12.6
4:1	1	0.272	0.195	51.9	12.4
6:1	1	0.331	0.317	63.8	12.3
8:1	3	0.374	0.445	72.0	11.9

[00148] It can be seen that the amount of PAA generated, and hence the conversion of triacetin into PAA, increased with the increasing mole ratio of hydrogen peroxide:triacetin. For all four mole ratios employed, the maximum amount of PAA was generated between one and three minutes. The optimum mole ratio of hydrogen peroxide:triacetin was 8:1 as it resulted in the most efficient utilization of triacetin converted into PAA.

[00149] Example 11

[00150] A further series of experiments was performed in which the mole ratio of hydrogen peroxide to triacetin was fixed at 4:1 and the molar ratio of NaOH to triacetin was varied from 3:1 to 8:1. Triacetin (5 g) was added to around 975 mL of softened water along with 50.7% hydrogen peroxide (6.18 g) to yield a solution with a pH of 4.8 to 5.3. With mixing, 50% NaOH was introduced in successive tests (5.51 g for the 3:1 mole ratio; 7.30 g for the 4:1 mole ratio; 11.03 g for the 6:1 mole ratio; and 14.67 g for the 8:1 mole ratio). The volume of the mixture was adjusted to 1000 mL with softened water. Using the ceric sulfate–iodometric titration method, the percent of PAA generated and the percent of hydrogen peroxide remaining were measured over the next 20 minutes. The method was performed in duplicate for each

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sample point. The averages of the results for the kinetic run employing the mole ratio of NaOH:hydrogen peroxide:triacetin of 4:4:1 are shown in Table XIII.

[00151] Table XIII

Time (min)	% PAA Generated	% Hydrogen Peroxide Remaining	% Triacetin Converted to PAA (3 acetyls)
1	0.247	0.207	47.3
3	0.280	0.178	53.6
5	0.292	0.167	55.9
10	0.267	0.142	51.1
20	0.229	0.111	43.8

[00152] Upon addition of the NaOH, PAA was immediately generated, with the maximum amount of 0.292% occurring after five minutes, corresponding, to a calculated conversion of 55.9% triacetin into PAA assuming that all three acetyl groups of triacetin were available for perhydrolysis.

[00153] This exercise was repeated for mole ratios of NaOH:TA ranging from 3:1 to 8:1. Table XIV summarizes the data and records the maximum percent of PAA generated, the time after the addition of NaOH that it took to reach the maximum percent of PAA generated, the pH after the addition of the NaOH, the percent of hydrogen peroxide remaining at the time of the maximum percent of PAA, and the calculated percent of triacetin converted to PAA assuming that all three acetyl groups were available for perhydrolysis.

[00154] Table XIV

Mole Ratio NaOH:TA	Time at Max. PAA (min)	Max % PAA Generated	% Hydrogen Peroxide Remaining	% Triacetin Converted to PAA (3 acetyls)	pH
3:1	3	0.246	0.202	47.2	11.3
4:1	5	0.292	0.167	55.9	11.6
6:1	3	0.290	0.181	55.6	12.2
8:1	3	0.272	0.195	51.9	12.4

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[00155] It can be seen that the amount of PAA generated maximized when the mole ratio of NaOH:hydrogen peroxide:triacetin was 4:4:1. The optimum mole ratio of NaOH:hydrogen peroxide:triacetin was 4:4:1 as it resulted in an efficient utilization of triacetin converted into PAA, and because the pH of the resulting reaction medium would have less impact on the pH of the receiving water than the higher pH of the reaction medium resulting from the 4:6:1 mole ratio of NaOH:hydrogen peroxide:triacetin. For all four mole ratios employed, the maximum amount of PAA was generated between three and five minutes.

[00156] Example 12

[00157] In the first test, a mixture of triacetin and hydrogen peroxide was prepared by blending 50% hydrogen peroxide (54.24%) and triacetin (45.76%). Thus, the mole ratio of hydrogen peroxide:triacetin was 3.65:1. The mixture (51.22 g) was dissolved in soft water (911.9 g) in a one-liter beaker to yield a solution with a pH of 6.47. Then, 50% NaOH (36.47 g) was introduced with stirring. Thus, the mole ratio of the combined mixture of NaOH:hydrogen peroxide:triacetin was 4.2:3.8:1. The pH of the resulting solution initially measured 12.1. Using the ceric sulfate–iodometric titration method, the PAA generated and the hydrogen peroxide remaining were measured over the next 10 minutes. It was noticed that the sample temperature rose immediately upon addition of the 50% NaOH.

[00158] In the second test, the first test was repeated, except that the water was chilled soft water.

[00159] In the third test, the first test was repeated using three times the original amount of hydrogen peroxide:triacetin mixture and three times the original amount of 50% NaOH. The mole ratio of the combined mixture of NaOH:hydrogen peroxide:triacetin remained the same as before, at 4.2:3.8:1. The hydrogen peroxide and triacetin mixture (153.96 g) was introduced to chilled soft water (735.6 g) in a one-liter beaker to yield a solution with an initial pH of 5.14.

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Then, 50% NaOH (111.21 g) was introduced. Using the ceric sulfate–iodometric titration method, the PAA generated and the hydrogen peroxide remaining were measured over the next 10 minutes.

[00160] In the fourth test, the first test was repeated using four times the original amount of hydrogen peroxide:triacetin mixture and four times the original amount of 50% NaOH. The mole ratio of the combined mixture of NaOH:hydrogen peroxide:triacetin remained the same as before, at 4.2:3.8:1. The hydrogen peroxide and triacetin mixture (204.7 g) was introduced to chilled soft water (647.5 g) in a one-liter beaker to yield a solution with an initial pH of 6.32. Then, 50% NaOH (155.4 g) was introduced. Using the ceric sulfate–iodometric titration method, the PAA generated and the hydrogen peroxide remaining were measured over the next 10 minutes.

[00161] Table XV summarizes the results obtained for the four tests and shows the initial and final solution temperatures, the pH of the solution after the addition of the 50% NaOH, the maximum percent of PAA generated, the time of maximum PAA generation, and the percent of hydrogen peroxide remaining.

[00162] Table XV

Test #	Initial Temp. (°F)	Final Temp. (°F)	Final pH	Max. % PAA Generated (time, min)	% Hydrogen Peroxide Remaining
1	NM	NM	12.14	1.71 (1)	0.58
2	38	50	12.67	2.1 (4)	0.43
3	44	88	12.77	6.3 (1)	1.18
4	34	92	13.17	7.2 (1)	1.88

NM = not measured

[00163] Table XVI shows the percent of hydrogen peroxide converted overall, the percent of hydrogen peroxide converted to PAA, and the percent of triacetin converted to PAA

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(assuming that all three acetyl groups on triacetin are available for conversion to PAA) for each of the tests in Table XV.

[00164] Table XVI

Test #	% Hydrogen Peroxide Converted Overall	% Hydrogen Peroxide Converted to PAA (at maximum conversion time)	% Triacetin Converted to PAA (3 acetyls)
1	67.8	88.1	69.7
2	68.8	94.4	85.8
3	71.6	94.3	85.7
4	66.1	88.1	73.8

[00165] The data in Tables XV and XVI indicate that far higher amounts of PAA are possible if the 50% sodium hydroxide solution is the last component of the mixture to be introduced. Further, up to 7.1% PAA was generated, although this solution got hot despite chilling the source water. Generation of the 6.3% PAA solution was very efficient because it represented a high 85.7% utilization of triacetin converted into PAA. There was clearly a linear relationship between Test 2, which generated 2.1% PAA, and Test 3, which utilized three times more of the reactants and generated 6.3% PAA. The data in Table XV indicate that the time of maximum conversion of triacetin was between one and four minutes. Thereafter, the amount of PAA in solution is reduced because it undergoes high pH degradation slowly over the following 10 minutes.

[00166] Example 13

[00167] Fig. 1 is a schematic representation of an example of a system 10 that was used to perform an embodiment of one of the methods described above to prepare a non-equilibrium solution of PAA on a site having a point-of-use. Triacetin was used as the acetyl precursor and 50% sodium hydroxide was used as the aqueous source of an alkali metal hydroxide.

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[00168] A container 120 of a hydrogen peroxide-acetyl precursor solution, which was a hydrogen peroxide-triacetin solution, and a container 140 of 50% sodium hydroxide were each equipped with chemical delivery diaphragm pumps 115 and 135, respectively. Inlet water 90, which was softened, was provided from a water source on the site having the point-of-use. Inlet water 90 was directed through a pressure regulator 100 and flow meter 105, then into a section of pipe where the hydrogen peroxide-triacetin solution was introduced through injection quill 110. Mixing was accomplished using static mixer 125. Then, the sodium hydroxide solution was added through injection quill 130 to form a reaction medium.

[00169] The hydrogen peroxide-triacetin solution and the sodium hydroxide solution may be added in a sequential manner as described, where the hydrogen peroxide-triacetin solution was added first, or they may be added to the water simultaneously through a "T" fitting placed before static mixer 125. If a T fitting is used, the hydrogen peroxide- triacetin solution and the 50% sodium hydroxide solution are introduced to opposite ends of the T fitting and the mixture is injected into the pipe of water. In other embodiments, the hydrogen peroxide and triacetin may be added separately, or sequentially, with either one first, or simultaneously, with the sodium hydroxide added either simultaneously with, or after, the hydrogen peroxide and triacetin.

[00170] The reaction medium was introduced to a residence chamber 145 which provided reaction time and contained a packing material to promote mixing. Residence chamber 145 was designed to be of a volume such that at a total flow rate of one gallon per minute, by the time the reaction medium reached sampling port 160, 30 seconds had elapsed since the sodium hydroxide solution had been added. A pH probe 155 monitored the pH of the mixture.

[00171] A controller 150 was interfaced to the flow meter 105 of the inlet water 90, and also to the chemical delivery diaphragm pumps 115 and 135. The controller 150 monitored the

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rate of the inlet water 90 and governed the rate at which the hydrogen peroxide-triacetin solution and the NaOH solution were introduced. If the flow of inlet water 90 decreased because of lower PAA requirements at the point-of-use 195, the rates at which the hydrogen peroxide-triacetin solution and the NaOH solution were pumped decreased accordingly. If the flow of inlet water 90 increased because of higher PAA requirements at the point-of-use 195, the rates at which the hydrogen peroxide-triacetin solution and the NaOH solution were pumped increased accordingly. If the flow of inlet water 90 stopped, pumps 115 and 135 stopped. Thus, the generation of PAA was both continuous and intermittent, and was tailored to the PAA requirements at the point-of-use.

[00172] The rate of the reaction between the hydrogen peroxide and the triacetin to form PAA is dependent upon the temperature of inlet water 90. If the temperature of inlet water 90 is high (for example, about 70°F), the maximum conversion of triacetin into PAA may occur after 30 seconds, in which case the entire reaction medium was directed to the point-of-use 195. However, if the temperature of inlet water 90 is low (for example, about 32°F), longer reaction times may be necessary to maximize the conversion of triacetin into PAA. Then, the reaction medium was directed through one or more residence chambers 165, 175, and 185 which also contained a packing material to promote turbulence and cause thorough mixing. Residence chambers 165, 175, and 185 were designed to be of volumes such that at a total flow rate of one gallon per minute, the reaction medium reached sampling port 170 in one minute, sampling port 180 in two minutes, and sampling port 190 in five minutes, to permit sampling at different time intervals.

[00173] Thus, depending on the temperature of the inlet water, the time to achieve the maximum conversion of hydrogen peroxide and triacetin into PAA was determined by sampling and analyzing the solution at sample ports 160, 170, 180, and 190. The entire flow was then

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directed from the sampling port with the highest amount of PAA to the point-of-use 195. In practice, the PAA generated from the reaction of hydrogen peroxide with triacetin is typically diluted at the point-of-use 195.

[00174] Point-of-use 195 may be recirculating cooling water; municipal wastewater; poultry chill tank water; water used to sanitize meat, poultry, or seafood products; fruit and vegetable rinse water; or water used to clean and sanitize equipment used in the dairy, food, or beverage processing industries.

[00175] Example 14

[00176] Referring again to Fig. 1, a 55-gallon drum container 120 of a hydrogen peroxide-triacetin solution was prepared by mixing triacetin (251.5 lbs) with 50% hydrogen peroxide (298.4 lbs). The resulting 550 lbs of hydrogen peroxide-triacetin solution had a specific gravity of 1.19 g/mL (10 lbs/gal) and contained 54.3% hydrogen peroxide and 45.7% triacetin. Thus, the mole ratio of hydrogen peroxide:triacetin was 3.8:1. The apparatus depicted in Fig. 1 was used to continuously and intermittently prepare an approximately 1% PAA solution.

[00177] Softened local city water was directed through pressure regulator 100 at a flow rate of one gallon per minute as measured by flow meter 105. The container 120 of hydrogen peroxide-triacetin solution was equipped with a draw down tube so that the flow rate of the mixture could be measured. Diaphragm pump 115 injected the hydrogen peroxide-triacetin solution through injection quill 110. The hydrogen peroxide-triacetin solution was then mixed with the softened water using static mixer 125. A container 140 of 50% NaOH was also equipped with a draw down tube to measure the flow pumped by diaphragm pump 135 into injection quill 130.

[00178] The percent of PAA generated was measured by quenching samples drawn through the sampling ports in mineral acid, followed by use of the ceric sulfate–iodometric

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titration method. Table XVII summarizes the results of several trials where it was sought to maximize the conversion of triacetin into PAA and minimize the amount of 50% NaOH used.

[00179] Table XVII

Trial #	Sample Port	% PAA Generated	% Triacetin Converted to PAA	Hydrogen Peroxide-Triacetin Mix Flow Rate (mL/min)	NaOH Flow Rate (mL/min)	pH (PAA solution)
1	160	1.083	65.42	115.73	112.11	~13
2	170	1.073	64.82	115.73	112.11	13.19
3	160	0.955	57.32	116.79	122.12	13.37
4	160	1.058	63.39	116.79	114.93	13.11
5	160	1.061	63.3	116.79	98.1	12.9
6	160	1.102	68.96	110.82	85.01	12.91
7	170	1.055	55.59	132.64	99.77	12.95
8	160	0.978	59.91	115.5	74	12.62
9	180	0.936	56.7	114.25	78	12.64

[00180] It can be seen that the amount of PAA generated, and hence the conversion of triacetin into PAA, increased with the increasing flow rate of NaOH. For this mole ratio of hydrogen peroxide:triacetin of 3.8:1, the maximum amount of PAA was generated between 30 seconds (sample port 160) and two minutes (sample port 180) from the time the NaOH was added at injection quill 130. The flow rate of 74-78 mL/min of 50% NaOH was the optimum flow rate as it resulted in an efficient utilization of triacetin converted into PAA, consumed only a moderate amount of 50% NaOH, and had a lower pH that would have less impact on the pH of the receiving water.

[00181] Example 15

[00182] *E. coli* O157:H7 bacteria (ATCC 35150) was cultured in nutrient broth (Sigma, St. Louis, MO) by incubation for two days at 35°C. The bacteria were separated from the nutrient broth by centrifugation and carefully resuspended in two liters of sterile phosphate buffer, which was then split into two one-liter test solutions.

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[00183] *Salmonella* typhimurium bacteria (ATCC 14028) was cultured in nutrient broth (Sigma, St. Louis, MO) by incubation for two days at 35°C. The bacteria were separated from the nutrient broth by centrifugation and carefully resuspended in two liters of sterile phosphate buffer, which was then split into two one-liter test solutions.

[00184] The amount of *E. coli* O157:H7 and *Salmonella typhimurium* bacteria were measured by serial dilution and plating on 3M *E. coli* plates and 3M *Enterobacteriaceae* Petrifilms, respectively.

[00185] A 1000 ppm stock of PAA was made by weighing 0.6386 g of Perasan MP-2 (5.83% hydrogen peroxide and 15.66% PAA) and adding water up to 100 g. Perasan MP-2 is an equilibrium product of PAA (Enviro Tech Chemical Services, Inc., Modesto, California). Side-by-side, one of the *E. coli* and one of the *Salmonella* test solutions were treated with a nominal dose of 1.0 ppm PAA by adding one mL of the 1000 ppm PAA stock solution to each sample. After one minute, approximately 100 mL of each test solution was removed, and sodium thiosulfate (0.5 g) was added to neutralize the PAA and hydrogen peroxide oxidants. After five minutes, sodium thiosulfate (0.5 g) was added to the remaining test solution to neutralize the PAA and hydrogen peroxide in the rest of the test solutions. The amount of viable *E. coli* O157:H7 and *Salmonella typhimurium* bacteria remaining in the test solutions at the one-minute and five-minute contact times were measured by serial dilution and plating on 3M *E. coli* Petrifilms and 3M *Enterobacteriaceae* Petrifilms, respectively.

[00186] A 1% solution of PAA was prepared by combining 30.90 g of a mixture of 50% hydrogen peroxide and triacetin (54.22% hydrogen peroxide and 45.78% triacetin) with 929.4 g of water, and then adding 50% NaOH (40.53 g). The PAA solution had a pH of 12.79. The mole ratio of this mixture was 3.8:7.8:1 NaOH:hydrogen peroxide:triacetin. One minute after preparation, the second one-liter *E. coli* solution and the second one-liter *Salmonella* solution

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were treated with a nominal dose of 1.0 ppm PAA by adding 0.10 g of the 1% PAA solution to each sample. The test solution contact times, neutralization, and plating procedures were replicated for those that employed PAA from Perasan MP-2.

[00187] All 3M *E. coli* Pertrifilms and 3M *Enterobacteriaceae* Petrifilms were incubated at 35°C for 24 hours. After incubation, the plates were enumerated.

[00188] Table XVIII shows the microbiological results of the *E. coli* O157:H7 test solutions before treatment with PAA, one minute after the samples were dosed with a nominal 1.0 ppm PAA, and five minutes after being dosed with PAA. The *E. coli* test solution started with $\log_{10}$ 6.99 CFU/mL prior to being treated with 1.0 ppm of PAA from Perasan MP-2 or with 1 ppm of PAA from the 1% PAA solution.

[00189] The *E. coli* test solution treated with 1.0 ppm PAA from Perasan MP-2 contained a $\log_{10}$ 6.74 CFU/mL after one minute. This corresponds to 43.48% reduction. After five minutes, $\log_{10}$ 6.34 CFU/mL remained (77.50% reduction). The *E. coli* solution treated with a nominal 1.0 ppm PAA from the 1% PAA solution contained a $\log_{10}$ 6.78 CFU/mL after one minute and 6.63 CFU/mL after five minutes, respectively, corresponding to 38.02% and 56.12% reductions, respectively.

[00190] Table XVIII

***E. coli* O157:H7**

Description	$\log_{10}$ CFU/ml remaining	$\log_{10}$ reduction	% reduction
Control	6.99	N/A	N/A
Perasan MP-2 (1 min)	6.74	0.25	43.48
Perasan MP-2 (5 min)	6.34	0.65	77.50
1% PAA solution (1 min)	6.78	0.21	38.02
1% PAA solution (5 min)	6.63	0.36	56.12

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[00191] Table XIX shows the microbiological results of the *Salmonella typhimurium* test solutions before treatment with PAA, one minute after the samples were dosed with a nominal 1.0 ppm PAA, and five minutes after being dosed with PAA. The *Salmonella* test solution started with $\log_{10}$ 7.07 CFU/mL prior to being treated with PAA from Perasan MP-2 or with the 1% PAA solution.

[00192] The *Salmonella* solution treated with a nominal 1.0 ppm PAA from Perasan MP-2 contained a $\log_{10}$ 1.76 CFU/mL after one minute and 0.70 CFU/mL after five minutes, corresponding to >99.999% reduction at both contact times. The *Salmonella* test solution treated with 1.0 ppm PAA from the 1% PAA solution contained a $\log_{10}$ 6.69 CFU/mL after one minute. This corresponds to 58.31% reduction. After five minutes, $\log_{10}$ 4.95 CFU/mL remained (99.24% reduction).

[00193] Table XIX

Salmonella typhimurium

Description	$\log_{10}$ CFU/ml remaining	$\log_{10}$ reduction	% reduction
Control	7.07	N/A	N/A
Perasan MP-2 (1 min)	1.76	5.31	99.9995
Perasan MP-2 (5 min)	0.70	6.37	>99.9999
1% PAA solution (1 min)	6.69	0.38	58.31
1% PAA solution (5 min)	4.95	2.12	99.24

[00194] The data in tables XVIII and XIX demonstrate that the 1% PAA had antimicrobial efficacy. After five minutes, the efficacy of this PAA was 99.24%, about equal to that of the Perasan MP-2, which was > 99.9999%. The difference in efficacy at one minute was likely not of statistical significance, given that the comparison was only at the one-minute interval.

[00195] **5. Solid Compositions for Bleaching and Stain Removal**

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[00196] A freely-flowable, solid peroxygen bleaching and stain removal composition comprises a liquid acetyl precursor, a water-soluble solid source of hydrogen peroxide, and a water-soluble solid source of alkalinity. A preferable liquid acetyl precursor is triacetin. The water soluble solid source of hydrogen peroxide is preferably sodium percarbonate, although sodium perborate monohydrate or sodium perborate tetrahydrate may be used. The water-soluble solid source of alkalinity acts as a pH control agent, and may be an alkali metal or earth alkali metal bicarbonate, carbonate, sesquicarbonate, borate, silicate, or hydroxide. Examples include sodium bicarbonate, sodium carbonate, sodium sesquicarbonate, sodium borate, sodium borate decahydrate (borax), sodium metasilicate, and sodium hydroxide.

[00197] In an embodiment, the composition comprises about 0.99% to about 8.45% triacetin, about 13% to about 54% sodium percarbonate, and about 24% to about 44% solid source of alkalinity.

[00198] The composition may optionally contain other components to improve performance. These components may prevent soil redeposition, sequester water hardness, increase dispercency, and lower surface tension.

[00199] Metal ions present in the bleaching medium can have a tendency to deposit on the substrates being bleached. Therefore, the solid bleaching composition may optionally contain a metal chelating agent such as a polyacrylate, a phosphonate, a maleic acid, a salt of ethylenediaminetetraacetic acid, a salt of nitrilotriacetic acid, or a salt of gluconic acid.

[00200] Surfactants reduce the surface tension of the bleaching medium and enhance the process of cleaning the soiled article. Surfactants also disperse the soil particles removed from the article and keep them suspended. Therefore, the solid bleaching composition may optionally contain a surfactant, such as an anionic, cationic, amphoteric, or non-ionic surfactant.

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[00201] The solid bleaching composition may also optionally contain a solid zeolite, such as sodium zeolite, which acts to capture calcium and magnesium ions that may be present in the bleaching medium that would otherwise interact with an anionic surfactant, causing the surfactant to precipitate and removing it from the bleaching medium.

[00202] The composition may also optionally contain an inert, solid filler, such as anhydrous sodium sulfate, sodium sulfate decahydrate, or sodium chloride. The filler increases the total ionic strength of the aqueous phase, but imparts no chemical properties to the bleaching medium.

[00203] To make the solid peroxygen bleaching and stain removal composition, the water soluble solid source of hydrogen peroxide, the water-soluble solid source of alkalinity, any chelating agent, any sufactant, and any filler are blended with the liquid triacetin to form a solid bleaching composition. The components may be added in any order. Any blending equipment may be used. Suitable examples of equipment include a ribbon and paddle blender, a V and double-cone blender, a Nauta screw mixer, and a rotating cement mixer. Alternatively, the ingredients may be blended by milling using a pulveriser mill, a ball mill, a hammer mill, a pin mill, an air mill, or a roller mill. Blending is performed for sufficient time to create a homongenous mixture. The mixture is a freely-flowable solid which is readily discharged from the blender.

[00204] Example 16

[00205] A series of tests were performed to determine whether triacetin, a liquid at room temperature, could be blended with a solid source of hydrogen peroxide to form a solid bleaching and stain removal composition that did not clump or turn into a sticky, non-flowable mass. The components listed in Table XX were mixed together to form a solid peroxygen bleach precursor.

[00206] Table XX

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Component	%
Sodium carbonate, anhydrous	26.3
Sodium percarbonate	58.9
Sodium sulfate decahydrate	13.8
Surfactant (60% sodium dodecylbenzene sulfonate)	1.0
Total	100

[00207] This solid peroxygen bleach precursor was then blended with incrementally increasing amounts of liquid triacetin. At each addition, the product was examined for flowability and for the odor of PAA. Table XXI summarizes the findings when up to 9.1g of liquid triacetin was blended into 100 g of the solid peroxygen bleach precursor set forth above.

[00208] Table XXI

Wt. of triacetin added to solid peroxygen bleach precursor (g)	Freely-flowable solid or sticky and clumping	Odor of PAA
1	Freely- flowable solid	None
2	Freely- flowable solid	None
3	Freely- flowable solid	None
4	Freely- flowable solid	None
5	Freely- flowable solid	None
6	Freely- flowable solid	None
7	Freely- flowable solid	None
8	Freely- flowable solid	None
8.45	Freely-flowable solid	None
9	Sticky and clumping	None
9.1	Sticky and clumping	None

[00209] Surprisingly, even though triacetin is a liquid, the solid peroxygen bleach precursor (100 g) could be blended with up to 8.45g of the triacetin to form a freely-flowable solid composition, without becoming a sticky, non-flowable product which would not pour easily or would become a solid mass upon storage inside a container.

[00210] The solid peroxygen bleaching and stain removal composition was calculated to contain the following percentage of components, listed in Table XXII.

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[00211] Table XXII

Component	%
Sodium carbonate, anhydrous	24.14
Sodium percarbonate	53.90
Sodium sulfate decahydrate	12.56
Surfactant (90% sodium dodecylbenzene sulfonate)	0.95
Triacetin	8.45
Total	100

[00212] Another unexpected result was that after storing the composition for over one year at ambient temperature, the composition still had not developed an odor of PAA.

[00213] These surprising results are in contrast to the current belief that a liquid acetyl precursor, such as triacetin, could not be utilized with a solid source of hydrogen peroxide in a non-chlorine peroxygen bleaching and stain removal composition, for at least two reasons. First, when a non-chlorine peroxygen bleaching composition is used in residential and commercial laundering applications, the product must be a freely-flowable solid. Typically, the acetyl precursor in a non-chlorinated peroxygen bleach is present in amounts up to 10%. It was believed that blending a liquid at this level with the other dry components of the laundry bleach would create a sticky, clumping mass, rather than a freely-flowable solid, and would therefore not be acceptable to the user. Second, it was assumed that a liquid acetyl precursor would interact with the solid source of hydrogen peroxide and any free water or water of crystallization associated with other components, and thus form PAA in the product during storage, before being introduced to the aqueous bleaching medium. The high volatility and strong odor of the PAA would be unacceptable to the user of such a product.

[00214] Example 17

[00215] Two solid peroxygen bleaching and stain removal compositions were prepared with the components listed in Table XXIII.

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[00216] Table XXIII

Components of Composition A	%	Charge Weights, g
Premix 1		
Sodium Percarbonate	13	195
NANSA HS 90	1.5	22.5
Sodium Metasilicate (Anhydrous)	19.5	292.5
Sodium Sulfate (Anhydrous)	41	615
Triacetin	4	60
Premix 2		
Mineral Oil	1	15
Sodium Hydroxide Beads	4	60
Soda Ash	16	240

Components of Composition B	%	Charge Weights, g
Premix 1		
Sodium Percarbonate	13	195
NANSA HS 90	1.5	22.5
Sodium Metasilicate (Anhydrous)	19.5	292.5
Sodium Sulfate (Anhydrous)	43	645
Triacetin	4	60
Premix 2		
Mineral Oil	1	15
Sodium Hydroxide Beads	2	30
Soda Ash	16	240

[00217] In both cases, Premix 1 was prepared by kneading the NANSA HS 90 (a sodium dodecylbenzenesulfonate surfactant) with the sodium sulfate. The sodium sulfate helped break up the sticky surfactant efficiently. Then, the sodium percarbonate, the sodium metasilicate, and the triacetin were added and mixed by hand.

[00218] In both cases, Premix 2 was prepared by mixing the sodium hydroxide beads with the mineral oil to afford a well-coated mixture, with some free-standing mineral oil remaining. Soda ash was blended in portions to take up the excess liquid and to provide a free-flowing solid mixture.

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[00219] Premix 2 was blended in portions into Premix 1, and the final solid composition was mixed well by hand.

[00220] Both compositions A and B, which were held in uncovered bowls at ambient temperature after preparation, exhibited slow weight gain over time, most likely due to absorbance of water from the air. Each composition had gained 2.4% of its initial weight over a period of three days.

[00221] Neither composition exhibited significant exothermic behavior. However, composition A was hotter at the end of preparation and cooled more slowly than composition B. The temperature of composition A was 85°F immediately after preparation, rose to 88°F over the next ten minutes, and then fell slowly to 74°F. The temperature of composition B was 80°F immediately after preparation, rose to 81°F over the next twenty minutes, and then fell slowly to 65°F.

[00222] The solid peroxygen bleaching and stain removal composition described above may be used as follows.

[00223] First, the solid peroxygen, composition is introduced to an aqueous medium that is used for bleaching or stain removal of at least one article contained within the aqueous medium. For bleaching of laundry articles, about 5g of bleaching composition is introduced for every liter of water used in the bleaching cycle. For home use, the bleaching composition can be added manually at the start of the wash. For commercial laundry systems, the bleaching composition may be introduced just before the bleaching cycle, toward the end of the wash and following the soil removal cycle. In most commercial and residential laundry machines, bleaching cycles are typically about 10-15 minutes.

[00224] Second, the composition is allowed to dissolve and react to form the bleaching medium containing PAA. The solid source of hydrogen peroxide, such as sodium percarbonate,

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and the solid source of alkalinity, such as sodium hydroxide, dissolve in the laundry water and cause the pH of the water to increase to above about 10.5.

[00225] While not wishing to be bound by theory, it is believed that under elevated pH conditions, hydrogen peroxide dissociates according to the following equation:

$$H_2O_2 = H^+ + HO_2^-$$

[00226] The perhydroxyl anion (HO_2^-) then effects nucleophilic substitution reactions on the carbonyl groups of triacetin, in a perhydrolysis reaction, to form PAA and 1,2,3-propanetriol (glycerine). The laundry water would also contain unreacted hydrogen peroxide, unreacted triacetin, and sodium hydroxide.

[00227] Third, the aqueous solution of PAA and the unreacted hydrogen peroxide are mixed for sufficient time to effect bleaching or stain removal of the articles contained within the aqueous medium. Both commercial and residential bleaching cycles are about 10-15 minutes.

[00228] Fourth, the article is rinsed with fresh water so that it becomes substantially free of PAA and the unreacted hydrogen peroxide, the glycerine, the unreacted triacetin, and the sodium hydroxide. Generally, for a laundered article, the washing machine is programmed to empty the bleaching medium and replace it with fresh water. The machine can agitate the articles with the rinse water to enhance the rinsing process.

[00229] Fifth, the article is allowed to dry. Any suitable means of drying the bleached articles may be employed. This includes the use of centrifugal forces to mechanically dewater the articles prior to introducing the wet articles to another machine before the articles are tumbled in a stream of hot air. Alternatively, the articles may be allowed to air dry without the use of externally-applied heat.

[00230] Example 18

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[00231] Laundry swatches were made by cutting a tea towel into 50 equal 5 x 4 inch sheets. They were then separated into seven groups of seven swatches. Six of the groups were stained and the seventh group was left unstained for comparison purposes. Five of the groups that were stained were stained by soaking them in one of the following: black tea, black coffee, red wine, blood (pork), and acetone-extracted chlorophyll. The sixth group of swatches was stained by rubbing them on green grass. After allowing the stained swatches to dry, and before challenging the stains, the swatches were set aside for two months to allow the stains to set in the fabric.

[00232] A solid peroxygen bleaching and stain removal composition (200 g) was prepared by blending sodium percarbonate (104.0 g), triacetin (8.00 g), soda ash (80.01 g), and sodium hydroxide beads (8.00 g). The final product was a freely-flowable solid.

[00233] The composition was tested using 1:100 dilution at both 75°F and 104°F. The initial pH was measured and the concentrations of PAA and hydrogen peroxide were evaluated using the method of U.S. patent no. 7,651,724. At each temperature, the composition (1.0 g) was weighed out into a glass beaker (100 mL). To the beaker, city water (99 mL) was added while mixing on a stir plate. The pH and PAA generation profile was then monitored over the next 10-13 minutes to model the times of a typical low temperature and high temperature bleaching cycle. For city water at 75°F, the initial pH measured 11.2 and the PAA measured highest at 10.5 minutes (123 ppm PAA and 1594 ppm hydrogen peroxide). The test with city water at 104°F had the same initial pH of 11.2, but the PAA measured highest at 5.5 minutes (133 ppm PAA and 1494 ppm H_2O_2). Table XXIV shows the amount of PAA generated and the amount of hydrogen peroxide remaining over time at 75°F at a pH of 11.2. Table XXV shows the amount of PAA generated and the amount of hydrogen peroxide remaining over time at 104°F at a pH of 11.2.

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[00234] Table XXIV

Time (min)	ppm PAA	ppm Hydrogen Peroxide
1	61	1717
3	106	1673
5.2	113	1694
7.3	121	1571
10.5	123	1594
13.5	120	1547

[00235] Table XXV

Time (min)	ppm PAA	ppm Hydrogen Peroxide
0.5	72	1569
2.8	125	1426
5.5	133	1494
7.8	122	1427
10.3	119	1405

[00236] The stain removal ability of the composition was determined by comparing treated swatches to control swatches (swatches washed in just city water for the same amount of time and at elevated temperature) and non-stained swatches. The description of the swatches is set forth in Table XXVI. Three stained swatches were used for each test. The composition (10.0 g) was weighed into a beaker (2000 mL). To this beaker a measured amount of hard water (990.0 g) was added at ambient temperature (64°F). The swatches were added to the solution and agitated by mixing with a plastic utensil for 10 minutes to simulate a laundry bleaching cycle. After 10 minutes, the wash water was discarded and a water rinse was performed by adding fresh city water (1000.0 g) to the swatches and agitating for two minutes using the same plastic utensil. The swatches were immediately removed from the beaker after rinsing, wrung out by hand, and laid to dry. These steps were then repeated using water at an elevated temperature (120°F).

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[00237] A control swatch was obtained by taking one swatch at a time from each stain group and washing in city water at an elevated temperature (120°F) for 10 minutes in the same manner as the treated swatches. After the wash, the swatches were immediately removed, wrung out by hand, and laid to dry.

[00238] Table XXVI

Type of Swatches	Description
Treated Swatches	Swatches subjected to bleaching for 10 minutes using bleaching formulation with triacetin and a 2-minute rinse with hard water
Control Swatches	Swatches subjected to only an elevated temperature (120°F) hard water wash for 10 minutes
Non-Stained Swatches	Non-altered swatches

[00239] The bleaching ability of the test swatches was obtained by visual comparison of the treated swatches to the control swatches (treated with water only) and the non-stained swatches. The scale of bleaching ability of the composition was rated on a scale of 0 to 10, with 0 representing no apparent bleaching or stain removal properties of the swatches compared to the control and 10 representing bleaching of swatches to the original color (i.e., identical to the non-stained swatches). Three individuals rated the bleaching ability of the triacetin-containing formulation on the swatches independently. The average ratings from the three individuals are shown in Table XXVII.

[00240] Table XXVII

Type of Stain	Bleaching Formula at 64°F	Bleaching Formula at 120°F
Black Tea	5	8
Black Coffee	2	8
Red Wine	3	7
Blood (pork)	3	3
Extracted chlorophyll	0	0
Grass Stains	0	1

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[00241] It can be seen that, in general, the composition displayed enhanced stain removal properties at elevated temperature. Excellent stain removal properties were obtained for black tea and black coffee, and more moderate performance with red wine and blood. The composition had virtually no effect on grass stains or the chlorophyll pigment that was acetone-extracted from green grass.

[00242] The invention has been described above with the reference to the preferred embodiments. Those skilled in the art may envision other embodiments and variations of the invention that fall within the scope of the claims.

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Claims

We claim:

1. A hydrogen peroxide-acetyl precursor solution, comprising:

    a. aqueous hydrogen peroxide;

    b. a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide;

    c. a trace amount of peracetic acid; and

    d. water.

2. The solution of claim 1, wherein said liquid acetyl precursor is triacetin.

3. The solution of claim 2, wherein said aqueous hydrogen peroxide is about 23% to about 40% and said triacetin is about 20% to about 53%, and further wherein the mole ratio of said hydrogen peroxide to said triacetin is about 2.98:1 to about 12.84:1.

4. The solution of claim 3, wherein said aqueous hydrogen peroxide is about 27% and said triacetin is about 46%, and further wherein said mole ratio of said hydrogen peroxide to said triacetin is about 3.8:1.

5. A method of preparing a hydrogen peroxide-acetyl precursor solution, comprising:

    a. introducing a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide to a solution of aqueous hydrogen peroxide; and

    b. allowing the liquid acetyl precursor and the solution of aqueous hydrogen peroxide to mix to form a hydrogen peroxide-acetyl precursor solution.

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6. The composition of claim 5, wherein the liquid acetyl precursor is triacetin.

7. A method of generating a non-equilibrium solution of peracetic acid, comprising:

a. providing water;

b. introducing a hydrogen peroxide-acetyl precursor solution to the water;

c. mixing the hydrogen peroxide-acetyl precursor solution and the water to form a mixture;

d. adding an aqueous source of an alkali metal or earth alkali metal hydroxide to the mixture; and

e. forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

8. The method of claim 7, further comprising, prior to step (b), a step of providing the hydrogen peroxide-acetyl precursor solution comprising: introducing a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide to a solution of aqueous hydrogen peroxide; and allowing the liquid acetyl precursor and the solution of aqueous hydrogen peroxide to mix to form the hydrogen peroxide-acetyl precursor solution.

9. The method of claim 8, wherein the liquid acetyl precursor is triacetin defining a hydrogen peroxide-triacetin solution.

10. The method of claim 9, wherein the hydrogen peroxide-triacetin solution has a mole ratio of hydrogen peroxide to triacetin of about 2.98:1 to about 12.84:1.

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11. The method of claim 7, wherein the provided water is an aqueous stream.

12. The method of claim 7, wherein the provided water is contained in a mixing tank or other vessel.

13. The method of claim 9, wherein the aqueous source of the alkali metal or earth alkali metal hydroxide is sodium hydroxide.

14. The method of claim 13, wherein the sodium hydroxide is about 1.82% to about 7.28%.

15. The method of claim 7, wherein step (d) is performed simultaneously with step (b).

16. The method of claim 7, further comprising allowing the reaction medium sufficient time to maximize the conversion of the hydrogen peroxide and the acetyl precursor into peracetic acid.

17. The method of claim 16, wherein the time to maximize the conversion of the hydrogen peroxide and the acetyl precursor into peracetic acid is about 30 seconds to about five minutes.

18. The method of claim 17, further comprising, after step (e), a step of sampling the reaction medium to determine the time required to maximize the amount of hydrogen peroxide and acetyl precursor that are converted into peracetic acid.

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19. The method of claim 16, wherein the acetyl precursor is triacetin and the percent of triacetin that is converted into peracetic acid is about 40.9% to about 85.7%.

20. The method of claim 14, wherein the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is about 4.2:3.8:1.

21. The method of claim 7, wherein steps (a) through (e) are performed on a continuous basis.

22. The method of claim 7, wherein steps (a) through (e) are performed on an intermittent basis.

23. The method of claim 7, wherein the peracetic acid solution is for immediate use.

24. The method of claim 7, further comprising, after step (e), a step of adding a source of acid to stabilize the non-equilibrium solution of peracetic acid for storing and using throughout a working day.

25. The method of claim 7, wherein the peracetic acid solution is prepared on a site having a point-of-use.

26. A method of generating a non-equilibrium solution of peracetic acid for use on a site having a point-of-use of peracetic acid, comprising:

a. providing water on a site;

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b. providing a hydrogen peroxide-acetyl precursor solution on the site having a point-of-use;

c. introducing the hydrogen peroxide-acetyl precursor solution to the water;

d. mixing the hydrogen peroxide-acetyl precursor solution and the water to form a mixture;

e. providing an aqueous source of an alkali metal or earth alkali metal hydroxide on the site having the point-of-use;

f. adding the aqueous source of the alkali metal or earth alkali metal hydroxide to the mixture; and

g. forming a reaction medium comprising a non-equilibrium solution of peracetic acid for use at the point-of-use of peracetic acid.

27. The method of claim 26, further comprising, prior to step (b), a step of providing the hydrogen peroxide-acetyl precursor solution comprising: introducing a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide to a solution of aqueous hydrogen peroxide; and allowing the liquid acetyl precursor and the solution of aqueous hydrogen peroxide to mix to form the hydrogen peroxide-acetyl precursor solution.

28. The method of claim 8, wherein the liquid acetyl precursor is triacetin defining a hydrogen peroxide-triacetin solution.

29. The method of claim 28, wherein the hydrogen peroxide-triacetin solution has a mole ratio of hydrogen peroxide to triacetin of about 2.98:1 to about 12.84:1.

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30. The method of claim 26, wherein the provided water is an aqueous stream.

31. The method of claim 26, wherein the provided water is contained in a mixing tank or other vessel.

32. The method of claim 28, wherein the aqueous source of the alkali metal or earth alkali metal hydroxide is sodium hydroxide.

33. The method of claim 32, wherein the sodium hydroxide is about 1.82% to about 7.28%.

34. The method of claim 26, wherein step (d) is performed simultaneously with step (b).

35. The method of claim 26, further comprising allowing the reaction medium sufficient time to maximize the conversion of the hydrogen peroxide and the acetyl precursor into peracetic acid.

36. The method of claim 35, wherein the time to maximize the conversion of the hydrogen peroxide and the acetyl precursor into peracetic acid is about 30 seconds to about five minutes.

37. The method of claim 36, further comprising, after step (e), a step of sampling the reaction medium to determine the time required to maximize the amount of hydrogen peroxide and acetyl precursor that are converted into peracetic acid.

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38. The method of claim 35, wherein the acetyl precursor is triacetin and the percent of triacetin that is converted into peracetic acid is about 40.9% to about 85.7%.

39. The method of claim 33, wherein the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is about 4.2:3.8:1.

40. The method of claim 26, wherein steps (a) through (e) are performed on a continuous basis.

41. The method of claim 26, wherein steps (a) through (e) are performed on an intermittent basis.

42. The method of claim 26, wherein the peracetic acid solution is for immediate use.

43. The method of claim 26, further comprising, after step (e), a step of adding a source of acid to stabilize the non-equilibrium solution of peracetic acid for storing and using throughout a working day.

44. The method of claim 26, wherein the peracetic acid solution is prepared on a site having a point-of-use.

45. A non-equilibrium solution of peracetic acid made by the method of claim 7 for use as a disinfectant or sanitizer.

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46. The solution of claim 45 for clean-in-place pipeline and equipment cleaning.

47. The solution of claim 45 for use in washing fruits and vegetables.

48. The solution of claim 45 for use in the treatment of meat, poultry, and seafood.

49. A method for sanitizing a surface, which comprises generating a non-equilibrium solution of peracetic acid comprising:

peracetic acid, hydrogen peroxide, triacetin, 1,2,3-propanetriol, a source of an alkali metal or earth alkali metal hydroxide, and water.

50. A non-equilibrium solution of peracetic acid, comprising:

a. peracetic acid;

b. hydrogen peroxide;

c. triacetin;

d. 1,2,3-propanetriol;

e. an aqueous source of an alkali metal or earth alkali metal hydroxide; and

f. water.

51. The solution of claim 50, wherein said solution has a pH of about 11.2 to about 13.37.

52. The solution of claim 50, wherein said peracetic acid is about 1% to about 7.1%.

53. A method of generating a non-equilibrium solution of peracetic acid, comprising:

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a. providing water;

b. introducing a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide and aqueous hydrogen peroxide to the water;

c. mixing the liquid acetyl precursor and the aqueous hydrogen peroxide with the water to form a mixture;

d. adding an aqueous source of an alkali metal or earth alkali metal hydroxide to the mixture; and

e. forming a reaction medium comprising a non-equilibrium solution of peracetic acid.

54. The method of claim 53, wherein the liquid acetyl precursor is triacetin defining a hydrogen peroxide-triacetin solution.

55. The method of claim 53, wherein the liquid acetyl precursor and the aqueous hydrogen peroxide are introduced simultaneously.

56. The method of claim 53, wherein the liquid acetyl precursor and the aqueous hydrogen peroxide are introduced sequentially with either one first.

57. The method of claim 53, wherein the provided water is an aqueous stream.

58. The method of claim 53, wherein the provided water is contained in a mixing tank or other vessel.

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59. The method of claim 54, wherein the aqueous source of the alkali metal or earth alkali metal hydroxide is sodium hydroxide.

60. The method of claim 59, wherein the sodium hydroxide is about 1.82% to about 7.28%.

61. The method of claim 53, wherein step (d) is performed simultaneously with step (b).

62. The method of claim 53, further comprising allowing the reaction medium sufficient time to maximize the conversion of the hydrogen peroxide and the acetyl precursor into peracetic acid.

63. The method of claim 62, wherein the time to maximize the conversion of the hydrogen peroxide and the acetyl precursor into peracetic acid is about 30 seconds to about five minutes.

64. The method of claim 63, wherein the acetyl precursor is triacetin and the percent of triacetin that is converted into peracetic acid is about 40.9% to about 85.7%.

65. The method of claim 60, wherein the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is about 4.2:3.8:1.

66. The method of claim 53, wherein said steps (a) through (e) are performed on a continuous basis.

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67. The method of claim 53, wherein said steps (a) through (e) are performed on an intermittent basis.

68. A method of generating a non-equilibrium solution of peracetic acid on a site having a point-of-use, comprising:

a. providing water on a site;

b. providing a liquid acetyl precursor that is soluble in aqueous hydrogen peroxide and aqueous hydrogen peroxide on the site having a point-of-use;

c. introducing the liquid acetyl precursor and the aqueous hydrogen peroxide to the water;

d. mixing the aqueous hydrogen peroxide and the liquid acetyl precursor with the water to form a mixture;

e. providing an aqueous source of an alkali metal or earth alkali metal hydroxide water on the site having the point-of-use;

f. adding the aqueous source of the alkali metal or earth alkali metal hydroxide to the mixture; and

g. forming a reaction medium comprising a non-equilibrium solution of peracetic acid for use at the point-of-use.

69. The method of claim 68, wherein the liquid acetyl precursor is triacetin.

70. The method of claim 68, wherein the aqueous source of the alkali metal or earth alkali metal hydroxide is sodium hydroxide.

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71. The method of claim 70, wherein the sodium hydroxide is about 1.82% to about 7.28%.

72. The method of claim 68, further comprising allowing the reaction medium sufficient time to maximize the conversion of the hydrogen peroxide and the acetyl precursor into peracetic acid.

73. The method of claim 72, wherein the time to maximize the conversion of the hydrogen peroxide and the acetyl precursor into peracetic acid is about 30 seconds to about five minutes.

74. The method of claim 68, further comprising, after step (g), a step of sampling the reaction medium to determine the time required to maximize the amount of hydrogen peroxide and acetyl precursor that are converted into peracetic acid.

75. The method of claim 72, wherein the acetyl precursor is triacetin and the percent of triacetin that is converted into peracetic acid is about 40.9% to about 85.7%.

76. The method of claim 71, wherein the mole ratio of the sodium hydroxide to the hydrogen peroxide to the triacetin is about 4.2:3.8:1.

77. An on site non-equilibrium peracetic acid generation system, comprising:

a first container comprising a first solution wherein said first solution comprises:

aqueous hydrogen peroxide;

a liquid acetyl precursor that is soluble in said aqueous hydrogen peroxide;

a trace amount of peracetic acid; and

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water;

a second container comprising a second solution wherein said second solution comprises:

an aqueous source of an alkali metal or earth alkali metal hydroxide; and

means for mixing the first solution, water, and the second solution to generate a non-equilibrium peracetic acid solution.

78. A freely-flowable solid composition, comprising:

a. a liquid acetyl precursor;

b. a water-soluble solid source of hydrogen peroxide; and

c. a water-soluble solid source of alkalinity.

79. The composition of claim 78, wherein said liquid acetyl precursor is triacetin.

80. The composition of claim 79, wherein said triacetin is about 0.99% to about 8.45%.

81. The composition of claim 78, wherein said solid source of hydrogen peroxide is sodium percarbonate and is about 13% to about 54% of the total.

82. The composition of claim 78, wherein said solid source of alkalinity is about 24% to about 44% of the total.

83. A method of making a solid composition for bleaching and stain removal, comprising:

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forming a homogenous freely-flowable solid by blending a liquid acetyl precursor, a water-soluble solid source of hydrogen peroxide, and a water-soluble solid source of alkalinity.

84. The method of claim 83, wherein the liquid acetyl precursor is triacetin.

85. The method of claim 84, wherein the triacetin is about 0.99% to about 8.45%.

86. The method of claim 83, wherein the solid source of hydrogen peroxide is about 13% to about 54%.

87. The composition of claim 83, wherein the solid source of alkalinity is about 24% to about 44% of the total.

88. The method of claim 83, further comprising adding a metal chelating agent.

89. The method of claim 83, further comprising adding a surfactant.

90. The method of claim 83, further comprising adding a compound to sequester water hardness.

91. The method of claim 83, further comprising adding an inert solid filler.

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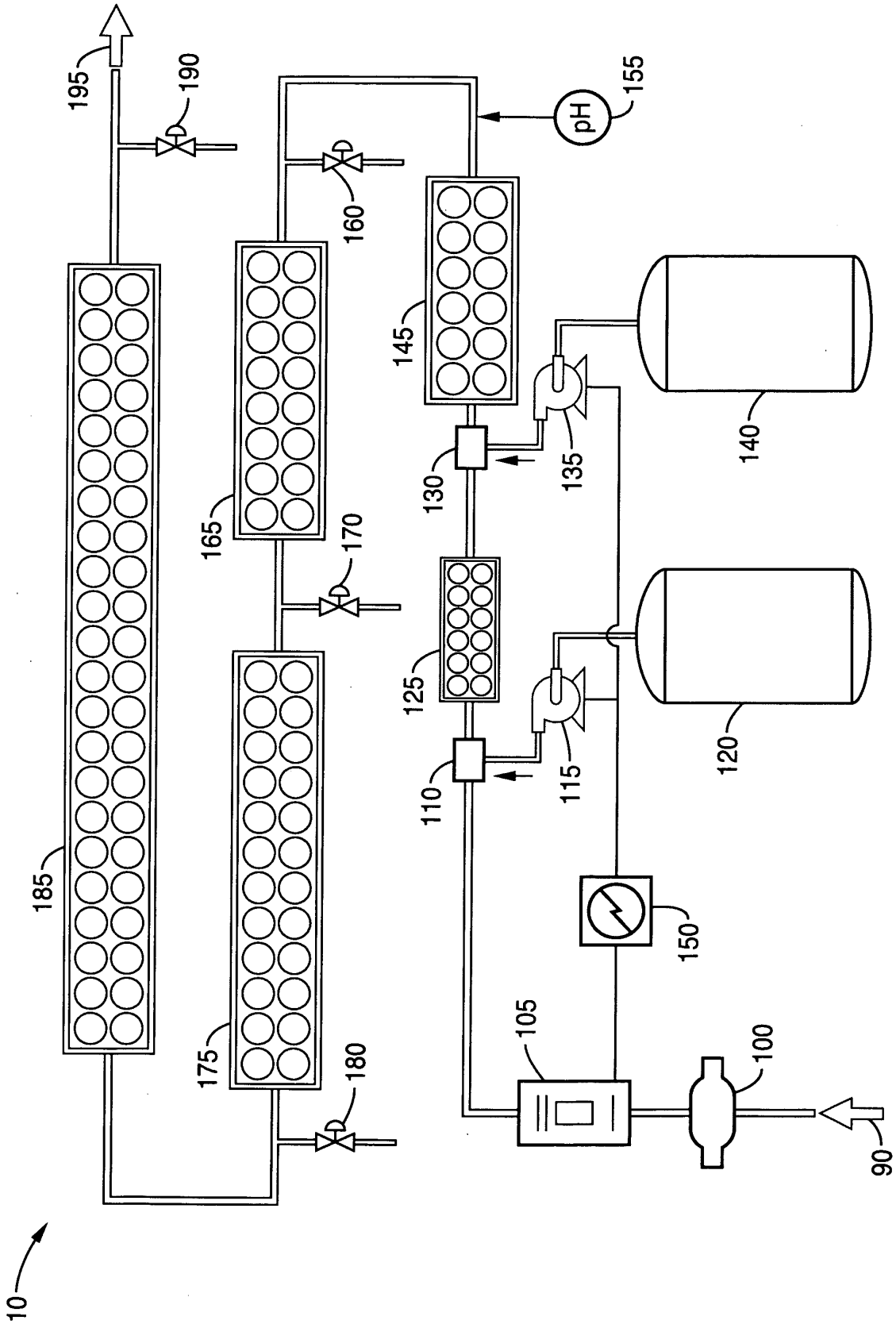

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FIG. 1