



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁵ : B01D 17/05, C02F 1/56	A1	(11) International Publication Number: WO 92/21421 (43) International Publication Date: 10 December 1992 (10.12.92)
(21) International Application Number: PCT/AU92/00250 (22) International Filing Date: 29 May 1992 (29.05.92) (30) Priority data: PK 6407 29 May 1991 (29.05.91) AU (71) Applicant (for all designated States except US): HOEFER, Dawn, Annette [AU/AU]; Trustee of Modern Environmental Service Trust, Lot 5, O'Biren Road, Gidgegannup, W.A. 6555 (AU). (72) Inventor; and (75) Inventor/Applicant (for US only) : BROWNE, Geoffrey, Robert [AU/AU]; Lot 26, Orchard Road, Gidgegannup, W.A. 6555 (AU).		(74) Agent: MUNT, Gregory, Richard; Griffith Hack & Co., 601 St. Kilda Road, Melbourne, VIC 3004 (AU). (81) Designated States: AT (European patent), AU, BE (European patent), CA, CH (European patent), DE (European patent), DK (European patent), ES (European patent), FR (European patent), GB (European patent), GR (European patent), IT (European patent), JP, LU (European patent), MC (European patent), NL (European patent), SE (European patent), US. Published <i>With international search report.</i>
(54) Title: TREATMENT OF EMULSIONS (57) Abstract <p>A process for separating the components of an emulsion. The process comprises, adjusting the pH of the emulsion to 4 or less, adding a coagulant to the emulsion, forming a substantially uniform dispersion of inert particulate carrier in the emulsion, adding a polyelectrolyte flocculent either during or after the step of forming the substantially uniform dispersion of inert particulate carrier in the emulsion to form flocs of the inert particulate carrier, the dispersed liquid phase, and the contaminants, allowing the flocs to settle, and separating the relatively clarified continuous liquid phase from the settled flocs.</p>		

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- 1 -

TREATMENT OF EMULSIONS

The present invention relates to the separation of the components of an emulsion.

The term "emulsion" is herein understood to mean a dispersion of a liquid phase in a continuous liquid phase, with either one or both of the liquid phases containing contaminants.

- 2 -

One specific example of a relatively complex emulsion is abattoir waste. Abattoir waste comprises a dispersion of a liquid phase of oils, fats and/or grease (hereinafter referred to as "the oil phase") in an aqueous phase, and further comprises contaminants:

- (a) dissolved in the aqueous and/or oil phases;
- (b) in particulate form suspended in the aqueous and/or oil phases; or
- (c) in colloidal form in the aqueous and/or oil phases.

The contaminants include bacteria count, nutrients, BOD, sand, grit and faeces.

Abattoir waste does not satisfy the environmental requirements for disposal into river and stream systems and thus has to be treated to separate the oil phase from the aqueous phase and to remove the contaminants from the aqueous phase before disposal of the aqueous phase is possible. Generally, such treatment includes transferring the abattoir waste through a series of aerobic and anaerobic dams. This is not an altogether satisfactory treatment regime in view of the set-up and on-going costs involved. A further disadvantage is that the treatment is not adapted to recover economically valuable contaminants, such as dissolved protein, in the abattoir waste.

Another specific example of a relatively complex emulsion is mineral oil waste from de-greasing units

- 3 -

widely used in industry. Typically, in such units suitable solvents are sprayed onto equipment to dissolve the oil/grease on the equipment, water is then sprayed onto the equipment to remove the dissolved oil/grease and remaining solvent, and the resultant emulsion, which by this stage includes grit and other contaminants, is collected in a sump and pumped to a settling pond.

Mineral oil waste does not satisfy the environmental requirements for disposal into river and stream systems and thus has to be treated to separate the oil/grease phase from the aqueous phase and to remove the solid and liquid contaminants from the aqueous phase before disposal of the aqueous phase is possible. At present there is no altogether satisfactory cost-effective treatment regime for this purpose.

An object of the present invention is to provide a process for separating the components of an emulsion.

A more particular object of the present invention is to provide a process for separating the components of complex emulsions comprising an oil-based liquid phase dispersed in a continuous aqueous phase, such as abattoir waste, mineral oil waste, dairy waste and food waste, to allow the aqueous phase to be released into river and stream systems.

According to the present invention there is provided a process for separating the components of an emulsion, as described herein, comprising the following steps in sequence:

(a) adjusting the pH of the emulsion to be 4 or less;

- 4 -

- (b) adding a coagulant to the emulsion;
- (c) forming a substantially uniform dispersion of inert particulate carrier in the emulsion;
- 5 (d) adding a polyelectrolyte flocculent either during or after the step of forming the substantially uniform dispersion of inert particulate carrier in the emulsion to form floccs of the inert particulate carrier, the dispersed liquid phase, and the contaminants;
- 10 (e) allowing the floccs to settle; and
- (f) separating the relatively clarified continuous liquid phase from the settled floccs.

The basis of the present invention is that it has been found unexpectedly that adjustment of the pH of an
15 emulsion, such as abattoir waste, to 4 or less and the addition of a coagulant has the effect of conditioning the emulsion so that inert particulate carrier and flocculent can separate the dispersed liquid phase and the contaminants, including dissolved contaminants, from
20 the continuous liquid phase of the emulsion. The unexpected finding is derived from the combined effect of acid and coagulant being greater than the separate effect of acid and coagulant.

The term "inert" as used herein in relation to
25 "particulate carrier" is understood to mean that the particulate carrier is not substantially attacked by the

- 5 -

emulsion. In other words, the term "inert" means that the particulate carrier exhibits both suitable chemical and physical stability in the emulsion.

5 It is preferred that the flocculent is a polyelectrolyte flocculent. The term "polyelectrolyte flocculent" as used herein is understood to mean any suitable cationic, non-ionic and anionic flocculent.

It is preferred that the pH is adjusted to be 3.5 or less.

10 It is preferred that the pH is adjusted to 3 or less.

It is preferred that the process further comprises adjusting the pH of the emulsion to be 5 or more after the addition of the coagulant in step (b) above.

15 It is particularly preferred that the pH is adjusted to be 5.5 or more after the addition of the coagulant in step (b) above.

20 It is preferred that the process further comprises processing the flocs to separate the inert particulate carrier from the dispersed liquid phase and the contaminants. It is particularly preferred that the process further comprises classifying the contaminants into valuable components.

25 It is preferred that the emulsion comprises a dispersion of an oil-based liquid phase in a continuous aqueous phase.

- 6 -

It is particularly preferred that the emulsion comprises abattoir waste, mineral oil waste, dairy waste, or food waste.

5 It is preferred that the coagulant comprises aluminium salts, ferrous salts or ferric salts.

It is preferred that the inert particulate carrier is selected from the group comprising sand, alumina, garnet, magnetite, hematite, ilmenite and calcite.

10 The process of the present invention is described further with reference to the following examples.

Example 1

15 A series of samples of emulsified mineral oil waste from a de-greaser unit were treated in accordance with a preferred embodiment of the process of the present invention. The principal objective of the experimental work was to assess the extent to which the process of the present invention could separate the aqueous component from the mineral oil waste.

20 The mineral oil waste samples contained a dispersion of oil and grease in a continuous aqueous phase and the contaminants at the levels set out in Table 1 below.

- 7 -

Table 1

	mg/l
Total Suspended Solids	350
Benzene	< 1
5 Toluene	< 1
Xylene	< 1
Polynuclear Aromatic	
Hydrocarbons	230
Chemical Oxygen Demand	2570
10 Biological Oxygen Demand	110

The samples of the mineral oil waste were treated as follows.

1. Adjustment of the pH to between 2.5 and 3.5.
2. Addition of a coagulant-sodium aluminate.
- 15 3. Adjustment of pH to between 5.5 and 7.5.
4. Agitating the mineral oil waste and inert particulate carrier to form a uniform dispersion of inert particulate carrier in the mineral oil waste.

- 8 -

5. Addition of a polyelectrolyte flocculent (Zetag 92 - produced by Allied Colloids) to form flocs of oil/grease, contaminants listed in Table 1, inert particulate carrier, and polyelectrolyte flocculent.

6. Separation of the flocs and the liquor.

The samples of the liquor recovered from step 6 above were tested and found to be water with no visible oil/grease and with contaminants at the levels set out in Table 2 below.

Table 2

	mg/l
Total Suspended Solids	7
Benzene	< 1
Toluene	< 1
Xylene	< 1
Polynuclear Aromatic	
Hydrocarbons	< 0.1
Chemical Oxygen Demand	550
Biological Oxygen Demand	90

- 9 -

The results of the experimental work indicate clearly that the preferred embodiment of the process of the present invention is capable of separating the aqueous phase of the mineral oil waste from the oil/grease phase and substantially removing the contaminants from the aqueous phase.

Example 2

A series of samples of abattoir waste were treated in accordance with a preferred embodiment of the process of the present invention. The principal purpose of the experimental work was to assess the extent to which the process of the present invention could separate the aqueous component from the abattoir waste.

The abattoir waste samples contained a dispersion of 85 mg/l of oil and grease in a continuous aqueous phase and the contaminants at the levels set out in Table 3 below.

Table 3

	mg/l
Total Suspended Solids	2,700
Total Phosphorus	30.5
Chemical Oxygen Demand	29.9
Biological Oxygen Demand	1850
Total Kjeldahl Nitrogen	175

- 10 -

The samples of abattoir waste were treated as follows:

1. Adjustment of pH to between 2.5 and 3.5.
2. Addition of a coagulant - sodium aluminate.
- 5 3. Adjustment of pH to between 5.5. and 7.5.
4. Agitating the abattoir waste and inert particulate carrier to form a uniform dispersion of inert particulate carrier.
- 10 5. Addition of polyelectrolyte flocculent (Zetag 92 - produced by Allied Colloids) to form floccs of oil and grease, contaminants listed in Table 3, inert particulate carrier, and polyelectrolyte flocculent.
6. Separation of the floccs and the liquor.

15 The samples of the liquor recovered from step 6 above were tested and found to have only 5 mg/l oil and grease and the contaminants at levels set out in Table 4 below.

Table 4

	mg/l
Total Suspended Solids	45
Total Phosphorus	0.85
Chemical Oxygen Demand	*ND
Biological Oxygen Demand	220
Total Kjeldahl Nitrogen	30

*ND - not determined

The results of the experimental work indicate clearly that the preferred embodiment of the process of the present invention is capable of substantially separating the aqueous phase of the abattoir waste from the oil/grease phase and substantially removing the contaminants from the aqueous phase to a level at which the aqueous phase is safe for disposal in river and stream systems.

Many modifications may be made to the process of the present invention as described above without departing from the spirit and scope of the present invention.

In this regard, it is noted that the step of adjusting the pH to between 5.5 and 7.5 after the initial acidification and coagulant addition steps is not an essential step of the process of the invention and was necessary as part of the procedure in the examples in order to ensure total removal of the aluminium based coagulant and optimum performance of the particular polyelectrolyte used in the examples.

- 12 -

CLAIMS:

1. A process for separating the components of an emulsion, as defined herein, comprising the following steps in sequence:

5 (a) adjusting the pH of the emulsion to be 4 or less;

(b) adding a coagulant to the emulsion;

(c) forming a substantially uniform dispersion of inert particulate carrier in the emulsion;

10 (d) adding a flocculent either during or after the step of forming the substantially uniform dispersion of inert particulate carrier in the emulsion to form floccs of the inert particulate carrier, the dispersed liquid phase, and the contaminants;

15

(e) allowing the floccs to settle; and

(f) separating the relatively clarified continuous liquid phase from the settled floccs.

2. The process defined in claim 1, comprising adjusting the pH to be 3 or less.

3. The process defined in claim 1 or claim 2, further comprising adjusting the pH of the emulsion to be 5 or more after the addition of the coagulant in step (b) above.

- 13 -

4. The process defined in claim 3, comprising adjusting the pH of the emulsion to be 5.5 or more after the addition of the coagulant in step (b) above.

5. The process defined in any one of the preceding claims, further comprising processing the floccs to separate the inert particulate carrier from the dispersed liquid phase and the contaminants.

6. The process defined in any one of the preceding claims, further comprising classifying the contaminants into valuable components.

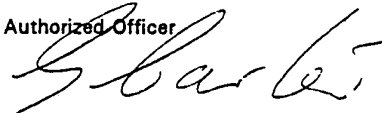
7. The process defined in any one of the preceding claims, wherein the emulsion comprises a dispersion of an oil-based liquid phase in a continuous aqueous phase.

8. The process defined in claim 7, wherein the emulsion comprises abattoir waste, mineral oil waste, dairy waste, or food waste.

9. The process defined in any one of the preceding claims, wherein the coagulant comprises aluminium salts, ferrous salts or ferric salts.

10. The process defined in any one of the preceding claims, wherein the inert particulate carrier is selected from the group comprising sand, alumina, garnet, magnetite, hematite, ilmenite and calcite.

INTERNATIONAL SEARCH REPORT

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) ⁶				
According to International Patent classification (IPC) or to both National Classification and IPC Int. Cl. ⁸ B01D 17/05 C02F 1/56				
II. FIELDS SEARCHED				
Minimum Documentation Searched ⁷				
Classification System	Classification Symbols			
IPC	B01D 17/05 17/04 C02F 1/56, 1/20			
Documentation Searched other than Minimum Documentation to the extent that such Documents are included in the Fields Searched ⁸				
AU: IPC as above				
III. DOCUMENTS CONSIDERED TO BE RELEVANT ⁹				
Category [*]	Citation of Document, ¹¹ with indication, where appropriate of the relevant passages ¹²	Relevant to Claim No ¹³		
A	US.A.4265770 (Thomas) 5 May 1981 (05.5.81)			
A	Patents Abstracts of Japan, 24C21, JP, A, 55-70307 (MATSUSHITA DENKA SANGYO K.K.) 21 November 1978 (21.11.78)			
A	AU-A 22673 /88 (GOLCONDA ENGINEERING 23 February 1989 (23.02.89)			
A	US.A.3350302 (Demeter) 31 October 1967 (31.10.67)			
A	EP.A 117586 (METALLGESELLSCHAFT A.G 5 September 1984 (05.09.84)			
<p>[*] Special categories of cited documents : ¹⁰</p> <table border="0"> <tr> <td style="vertical-align: top;"> <p>"A" Document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"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)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </td> <td style="vertical-align: top;"> <p>"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</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"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</p> <p>"&" document member of the same patent family</p> </td> </tr> </table>			<p>"A" Document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"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)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p>	<p>"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</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"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</p> <p>"&" document member of the same patent family</p>
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IV. CERTIFICATION				
Date of the Actual Completion of the International Search 6 August 1992		Date of Mailing of this International Search Report 18 Aug 1992 (18.08.92)		
International Searching Authority AUSTRALIAN PATENT OFFICE		Signature of Authorized Officer G.Carter 		

**ANNEX TO THE INTERNATIONAL SEARCH REPORT ON
INTERNATIONAL APPLICATION NO. PCT/AU 92/00250**

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report	Patent Family Member		
AU 22673/88	EP 377603 WO 8901357 NZ 224369 ZA 8802849	NZ 225853 AU 17053/88 WO 8808408	US 4997573 EP 355109 ZA 8806084
EP 117586	AU 24648/84 DE 3405451 US 4496374	BR 8400822 JP 59189923 ZA 8400969	CA 1239341 NZ 207073

END OF ANNEX