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(54) Title: COMPOSITION FOR SEPARATION OF BITUMEN FROM ORE

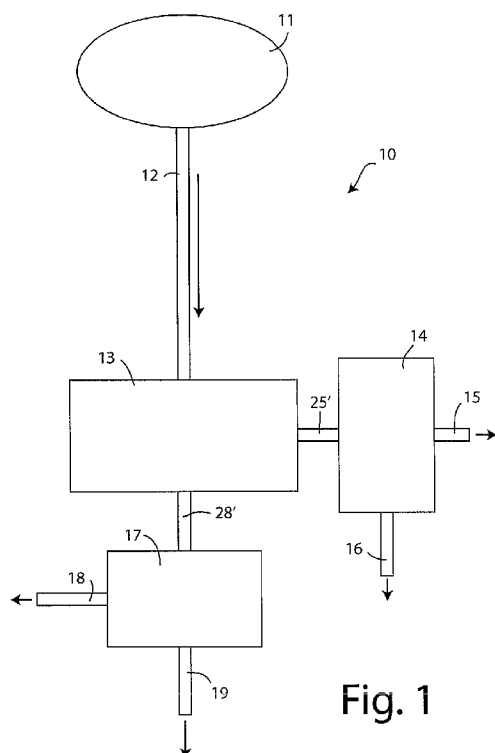


Fig. 1

(57) Abstract: An exemplary form of the invention relates to a composition useful, when mixed with water, for extracting bitumen from bituminous sand ore. The composition comprises the following ingredients, namely a citrus terpene (e.g. d-limonene), non-ionic liquid surfactant (e.g. an alcohol ethoxylate), acetone and isopropyl alcohol. The composition is mixed with water to form an aqueous mixture, and the aqueous mixture is then used for the extraction of bitumen upon being mixed and agitated with the bituminous ore. The bitumen extraction process may be carried out at ambient temperature, or at a temperature lower than that conventionally employed, thus making the process more energy efficient than hot-water extraction process, and causing fewer greenhouse gas emissions.

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COMPOSITION FOR SEPARATION OF BITUMEN FROM ORE

BACKGROUND OF THE INVENTION

I. FIELD OF THE INVENTION

- 5 This invention relates to the extraction of bitumen from bitumen ore. More particularly, the invention relates to compositions used in such extractions.

II. BACKGROUND ART

The term "bitumen" covers a variety of viscous or solid impure mixtures of hydrocarbons that occur naturally in asphalt, tar, mineral waxes, and the like. Bitumen deposits can be found in
10 many parts of the world and, where they occur in admixture with sand or clay, such deposits are often referred to as bituminous sands, oil sands or tar sands, and are regarded as unconventional petroleum deposits because the bitumen content can be upgraded to synthetic crude oils. Large deposits of bituminous sands are present in Canada and Venezuela and are currently being exploited commercially on a large scale in Canada. As the deposits lie at or close to the surface of
15 the ground in the major Canadian locations (e.g. the Athabasca oilsands), the bituminous ore can be strip-mined and transported to a central facility for treatment. However, before being upgraded to more fluid mixtures of hydrocarbons, the bitumen, which is extremely dense and viscous, must be separated from the balance of the bituminous ore (mainly sand, clay and water).

Currently, bitumen separation is normally carried out by mixing the ore with hot water and
20 caustic soda (NaOH) or other chemical bases. Bitumen itself has several naturally occurring water-soluble organic surfactants, but they do not become fully active until the pH and temperature conditions are appropriate. The resulting slurry is agitated so that the hydrocarbon content, assisted by a froth of air bubbles, rises to the surface where it skimmed off. This is referred to as the Clark hot water extraction process. The skimmed hydrocarbon product is then
25 treated further to remove residual water and fine solids, and is mixed with lighter petroleum or chemically split so that it can be transported by pipeline for upgrading into synthetic crude oil. The mined bituminous ore is often transported from the mine to the extraction plant by means of hydrotransport. This involves mixing the ore with warm water at high pH at the mine and moving it by pipeline to the plant. During the transport to the plant, the bituminous ore is conditioned,
30 causing complex physical and chemical changes to occur that break the surface tension between oil and water components.

The main disadvantages of this conventional extraction procedure is that large amounts of energy are required for heating the hot water used for bitumen separation from the ore, and that the caustic soda employed in the process is highly toxic and requires careful and expensive regeneration or disposal procedures. The energy required for the hot water process results in
5 the production of large amounts of carbon dioxide (as hydrocarbons are normally burned to generate the required heat) and this is becoming increasingly unfavorable because of concerns of the "greenhouse" effects of such gases on the world climate.

There is consequently a need for an improvement that overcomes these disadvantages, or at least reduces their unwanted effects.

10 The above process is often referred to as primary extraction. Secondary extraction is sometimes carried out on the residues of primary extraction (the so-called "middlings", i.e. the mainly aqueous phase formed between the bituminous upper layer and the sand-containing lower layer during the primary extraction). Secondary extraction generally involves a froth flotation process employing surfactants to raise the bituminous residues to an upper layer where they can be
15 skimmed off and treated. The hydrocarbon product of primary extraction may also be subjected to similar secondary extraction to reduce its water content since the water generally contains undesirably high levels of chlorides (present in bituminous ores).

SUMMARY OF THE INVENTION

20 One form of the present invention relates to a composition (liquid admixture) that can be used, when mixed with water, for extracting bitumen from bituminous sand ores as a full or partial replacement for the hot water/caustic soda solution conventionally used for such purposes. The composition may also be used for secondary bitumen extraction. Another form of the invention relates to a process of extracting bitumen from bituminous sand ores using the aforesaid
25 composition.

An exemplary embodiment of the invention relates to a composition useful, when mixed with water, for extracting bitumen from bituminous sand ores. The composition comprises a mixture of a citrus terpene (e.g. limonene, generally d-limonene), non-ionic liquid surfactant (e.g. alcohol ethoxylate), acetone and isopropyl alcohol as the main ingredients.

Another exemplary embodiment provides an aqueous mixture useful for extracting bitumen from bituminous ore, the aqueous mixture comprising water and the composition described above.

Another exemplary embodiment provides a process of extracting bitumen from bituminous ore.

- 5 The method comprises: a) mixing bituminous ore with an aqueous mixture to form a slurry; b) agitating the slurry and allowing a hydrocarbon-containing phase to rise to the surface of the slurry; and c) separating the hydrocarbon-containing phase from the remainder of the slurry; wherein the aqueous mixture is the mixture described above.
- 10 While the relative amounts of the four ingredients of the above mixture may vary widely, one embodiment employs these ingredients in the following proportions (by volume):

	<u>Ingredient</u>	<u>Parts by Volume</u>
	Citrus terpene	10 to 30
	Non-ionic liquid surfactant	20 to 40
15	Acetone	5 to 20
	Isopropyl alcohol	30 to 50.

The term "citrus terpene" means a terpene of the kind found in citrus trees, plants, fruits or oils.

- 20 However, such terpenes do not have to be citrus extracts for use in the present invention because synthetically-produced versions of these compounds, or such compounds from other natural sources, are equally effective. The citrus terpenes are generally monoterpenes, e.g. limonene.

- The composition may also contain one or more other ingredients, e.g. colorants or scents to improve the appearance and odor of the composition, rust-inhibitors to protect the extraction apparatus, and the like. Such additional ingredients, when present, are normally used in minor amounts, e.g. just the amounts necessary to provide the desired effects.
- 25

It is desirable, although not always essential, to mix the main ingredients by first mixing the citrus terpene with the isopropyl alcohol while stirring or agitating to form a fully blended admixture, then adding the acetone and finally adding the non-ionic liquid surfactant. This ensures that a fully mixed liquid composition is formed.

In one embodiment, the resulting composition is neutral or nearly neutral in terms of acidity/alkalinity, e.g. it has a pH of about 6.5 (i.e. $6.5 \pm 10\%$), and a density of about 0.92 g/cc.

The composition is a combination of surfactants and solvents designed to affect the surface tension of the bitumen in bituminous sand ores to facilitate the separation of the bitumen from the other materials present. The composition also helps to liquefy the bitumen and to maintain the bitumen in a suspended form to facilitate the separation process.

Before being used to extract bitumen from ore, the composition is mixed with water. In one embodiment, the composition is used at a concentration of 7 to 20% by volume of the resulting aqueous mixture, and is more usually used at a concentration of 7 to 12% by volume, and most normally at about 10% by volume. The composition may be mixed with the water in any way, e.g. by adding the ingredients together and stirring them at ambient temperatures (e.g. 0 to 30°C, more generally 15 to 25°C, and usually about 20°C, depending on the location).

The water may be of neutral pH (about 7), or may have a higher pH (e.g. an alkaline pH up to about 10, e.g. between pH 9 and 10). For example, the water used for mixing with bituminous ore is generally recycled from primary extraction and is often referred to as Process Effluent Water (PEW). Such water normally has an elevated pH due to prior caustic soda additions. PEW may be used in the present invention, thus giving the aqueous mixture an alkaline pH. When the water has a neutral pH, it is found that more of the composition of the invention is required for bitumen extraction than when the water has a higher, i.e. alkaline, pH. However, fresh additions of caustic soda or any other chemical base are generally unnecessary and can be avoided.

When fresh (neutral) water is used for form the aqueous mixture, one embodiment of the invention employs about 0.1 to 0.2 kg of the aqueous mixture (20% solution of the liquid composition) per tonne of mined ore. When PEW is used to form the aqueous mixture (again a 20% solution), the required amount of the aqueous mixture may be less than this.

The resulting aqueous mixture may be used for primary extraction of bitumen from ore in much the same way as is done in the conventional Clark extraction process with an alkaline solution, with the important difference that the extraction may be carried out at ambient temperature (e.g. 10 to 30°C, more usually around 20°C), i.e. without any pre-heating of the aqueous mixture or mined ore. Of course, in some embodiments, the aqueous mixture may be pre-heated prior to use if the extraction location is particularly cold and/or if a more rapid extraction is required, but temperatures below those used for the Clark extraction process (e.g. 80°C) are normally

employed. Generally, the higher the temperature of the aqueous mixture, and of course the higher the temperature of the bitumen ore itself, the higher will be the speed of extraction of the bitumen from the ore. However, the economies and advantages enjoyed as a result of a more rapid rate of extraction have to be weighed against the extra cost of heating the aqueous mixture and the disadvantages caused by emissions of greenhouse gases thereby generated. In general, in many situations, it is desirable to use the aqueous composition at ambient temperature without any pre-heating whatsoever.

The aqueous composition may be used for hydrotransport, i.e. to condition and supply bituminous ore from a mine to an extraction plant via pipeline. During such transport, the aqueous composition commences the separation of bitumen from the ore. Alternatively, the aqueous composition may be mixed with the ore at the extraction plant.

In one embodiment, the extraction process using the aqueous mixture is carried out with agitation of the bituminous ore, but the introduction of air or other gases to assist the separation may be avoided. The agitation may be the same as or similar to that used for the Clark extraction process, although a shorter duration of such agitation may be employed. The extracted bitumen, after a suitable time under agitation, forms a liquid hydrocarbon layer that floats on top of the residual water from the aqueous mixture. The hydrocarbon layer may be skimmed off or otherwise separated and the residual aqueous mixture may be recycled for further extraction, if necessary after replenishment of the extraction composition to allow for any loss of ingredients to the hydrocarbon layer. After a number of re-uses in this way, the entire residual aqueous mixture may be discarded, or a small proportion may be discarded and replaced with fresh mixture continuously or after each use. Since the ingredients of the liquid composition are not environmentally hazardous in the concentrations remaining in the discarded aqueous mixture, the mixtures may be discarded directly into pools or rivers. However, for additional protection of the environment, the used mixtures may first be treated to remove any remaining chemicals from the liquid composition or contaminants from the bitumen ore. Conventional filtering and content stripping techniques may be used for this purpose. It will be apparent that the liquid composition and the aqueous mixture lack caustic soda and other caustic ingredients, at least when fresh water is used to form the aqueous mixture, and are thus easier to use and dispose of in a safe manner.

The bitumen extraction process employing the aqueous mixture may be carried out batch-wise or continuously. Carrying out the process continuously is more efficient. For a continuous process,

the pH of the aqueous mixture may be monitored and fresh aqueous mixture injected to maintain the pH when it changes by more than a predetermined amount. The aqueous mixture may thus be employed in a closed loop system, with injection of fresh aqueous mixture as indicated above, and removal of an equivalent amount of the recycled mixture to maintain a constant volume. The scale of such operations may vary, but are generally the same as those used for the Clark extraction process.

The liquid composition and aqueous mixture of at least some of the exemplary embodiments may have at least one of the following advantages:

- 1) They can improve the recovery yield of bitumen and saleable synthetic crude oil above the present level;
- 2) They can improved thermodynamic efficiency and reduce the amount of heat required in bitumen extraction operations; and
- 3) They can reduce, or remove and eliminate, any causes of environmental impact on the land, water and air.
- 4) The composition does not affect the total petroleum hydrocarbon (TPH) concentrations in the bitumen.

While the liquid composition and aqueous mixtures discussed above may be used for primary bitumen extraction in the ways indicated, they may also be used for secondary bitumen extraction. For example, aqueous middlings from primary extraction may be mixed with the liquid composition or the aqueous mixture and then subjected to agitation by air injection to cause flotation of an organic phase to the surface where it can be skimmed off or otherwise removed. Alternatively, the bitumen-containing layer from primary extraction may be mixed with the liquid composition or aqueous mixture, optionally with froth flotation, to create a hydrocarbon layer and an aqueous layer containing residual water and dissolved salts (e.g. chlorides) from the primary extraction. The hydrocarbon layer is then separated and subjected to further upgrading or use.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a schematic drawing of a bitumen mining and separation system; and

Fig. 2 is a vertical cross section of a separation tank that may be used as part of the system illustrated in Fig. 1.

DETAILED DESCRIPTION

As mentioned above, the main ingredients of the liquid composition are a citrus terpene (e.g. d-limonene), a non-ionic liquid surfactant, acetone and isopropyl alcohol. These compounds are all commercially available and are discussed below.

D-Limonene

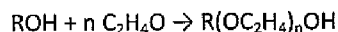
D-limonene is a cyclic terpene, available from fruits, having the formula $C_{10}H_{16}$ and the following structure:



(See Chemical Abstracts Service (CAS) No. 5989-27-5).

Non-Ionic Liquid Surfactant

Many non-ionic liquid surfactants are commercially available, e.g. alcohol ethoxylates and alcohol ethoxysulfates. These compounds are conventionally used in products such as laundry detergents, surface cleaners, cosmetics and for use in agriculture, textiles and paint. In the case of alcohol ethoxylates, the carbon chain length is 8-18 while the ethoxylated chain is usually 3 to 12 ethylene oxides in length. They are produced by reacting an alcohol with ethylene oxide usually in the presence of KOH as a catalyst: i.e.



wherein R is an alkyl group, and n is an integer of generally 3 or more.

One embodiment of the invention employs a blend of alcohol ethoxylates sold under the trademark BeroI® 609 by AkzoNobel Surface Chemistry, an international corporation with offices in the United States and Canada.

Acetone

- 5 Acetone is a well-known chemical having the formula C_3H_6O .

(See Chemical Abstracts Service (CAS) No. 67-64-1).

Isopropyl alcohol

Isopropyl alcohol is also a well-known chemical having the formula C_3H_8O .

(See Chemical Abstracts Service (CAS) No. 66-63-0).

10 **Examples of Uses of the Compound**

- Fig. 1 of the accompanying drawings illustrates one form of a bitumen mining and separation system 10 with which the liquid composition and aqueous mixture may be used. In the drawing, bituminous ore is mined at a strip mine 11 and, at that location, is mixed with an aqueous composition according to one form of the present invention at ambient temperature to make a
15 slurry. The slurry is then pumped from the mine 11 through tubing 12 to an extraction apparatus 13. This apparatus may take the form shown in Fig. 2, e.g. it may comprise a settling tank 20 into which the slurry is continuously pumped. The slurry in the tank may be agitated, e.g. by stirring or by introduction of air or other gas. The slurry separates into a lower sand layer, an aqueous middlings layer 22, and an upper bituminous layer 23. The tank 20 has a lower outlet 24 for
20 removal of sand, a central outlet 25 for removal of middlings and an upper outlet 26 for removal of the bituminous layer 23. The upper outlet is positioned to allow the bituminous layer 23 to overflow the tank without removal of middlings. The bituminous layer descends into a weir 27 and is removed from the apparatus for further processing via weir outlet 28. In the drawing of Fig. 1, the middlings layer is removed via tubing 25' connected to outlet 25, and the bituminous
25 layer is removed via tubing 28' attached to weir outlet 28.

In one embodiment, the middlings are subjected to secondary bitumen removal in a secondary treatment tank 14 where this primarily aqueous layer is mixed with a liquid composition as defined above and agitated with injected air or other gas to create an upper froth layer

containing residual bitumen. This is removed through piping 15 and the treated aqueous residue is removed through piping 16.

- Additionally, or alternatively, the bituminous layer from extraction apparatus 13 is subjected to a treatment with a liquid composition as defined above in treatment tank 17. This material is
- 5 largely hydrocarbon, but contains some water. The treatment with the liquid composition assists with the separation of the water from the hydrocarbon. The aqueous layer that forms at the bottom is removed through piping 18 and the hydrocarbon is removed through piping 19 for delivery to further apparatus for refining or other treatment.

CLAIMS:

1. A composition useful for extracting bitumen from bituminous sand ore, which composition comprises the following ingredients: citrus terpene, non-ionic liquid surfactant, acetone and isopropyl alcohol.
2. The composition of claim 1, wherein said ingredients are present in the following proportions:

<u>Ingredient</u>	<u>Parts by Volume</u>
Citrus terpene	10 to 30
Non-ionic liquid surfactant	20 to 40
Acetone	5 to 20
Isopropyl alcohol	30 to 50.

3. The composition of claim 1 or claim 2, wherein the citrus terpene is d-limonene.
4. The composition of claim 1, claim 2 or claim 3, wherein the non-ionic liquid surfactant is selected from alcohol ethoxylates and alcohol ethoxysulfates.
5. The composition of claim 1, claim 2 or claim 3, wherein the non-ionic liquid surfactant is an alcohol ethoxylate.
6. The composition of any one of claims 1 to 5, also including another ingredient selected from the group consisting of colorants, scents and rust inhibitors.
7. The composition of any one of claims 1 to 6, formed by first mixing said citrus terpene with said isopropyl alcohol to produce a first blend, then adding the acetone to the first blend to produce a second blend, and then adding the non-ionic liquid surfactant to the second blend to form said composition.

8. An aqueous mixture useful for extracting bitumen from bituminous sand ore, said mixture comprising water and the composition of any one of claims 1 to 7.
9. The mixture of claim 8, wherein said composition is present in said mixture at a concentration in the range of 7 to 20% by volume.
10. The mixture of claim 8, wherein said composition is present in said mixture at a concentration in the range of 7 to 12% by volume.
11. The mixture of claim 8, wherein said composition is present in said mixture at a concentration of about 10% by volume.
12. The mixture of any one of claims 8 to 11, wherein said water has a neutral pH.
13. The mixture of any one of claims 8 to 11, wherein said water has an alkaline pH.
14. The mixture of claim 13, wherein said water has an alkaline pH of less than 10.
15. The mixture of claim 13, wherein said water has a pH in a range of 9 to 10.
16. A process of extracting bitumen from bituminous ore, which method comprises:
 - a) mixing bituminous ore with an aqueous mixture to form a slurry;
 - b) agitating the slurry and allowing a hydrocarbon-containing phase to rise to the surface of the slurry; and
 - c) separating the hydrocarbon-containing phase from the remainder of the slurry;wherein said aqueous mixture is the mixture of any one of claims 8 to 15.
17. The process of claim 16, wherein said mixing and agitating are carried out at a temperature below 80°C.

18. The process of claim 16, wherein said mixing and agitating are carried out at ambient temperature.
19. The process of claim 16, wherein said mixing and agitating are carried out without prior heating of said aqueous mixture.
20. The process of claim 16, wherein said aqueous mixture, following step c, is re-used for extracting bitumen from further bitumen ore.
21. The process of claim 16 carried out continuously with injections of fresh said aqueous mixture and removal of equivalent amounts of said mixture over time.
22. The process of claim 21, wherein the pH of said aqueous mixture is monitored during said agitating and said injections of fresh said aqueous mixture are made when said monitored pH varies from a predetermined value by a predetermined amount.
23. A process of secondary recovery of bitumen from aqueous middlings derived from a primary extraction of bitumen from bituminous ore, said middlings containing residual bitumen, said process comprising mixing said aqueous middlings with a composition as defined in any one of claims 1 to 7, agitating the resulting mixture, allowing a hydrocarbon-containing layer to form over said aqueous middlings, and removing said hydrocarbon-containing layer for recovery of bitumen contained therein.
24. A process of removing water from a bitumen-containing stream derived from a primary extraction of bitumen from bituminous ore with an aqueous extractant, said process comprising mixing said bitumen-containing stream with a composition as defined in any one of claims 1 to 7, agitating the resulting mixture, allowing an aqueous layer to form below said bitumen-containing stream, and separating said bitumen-containing stream from said aqueous layer.

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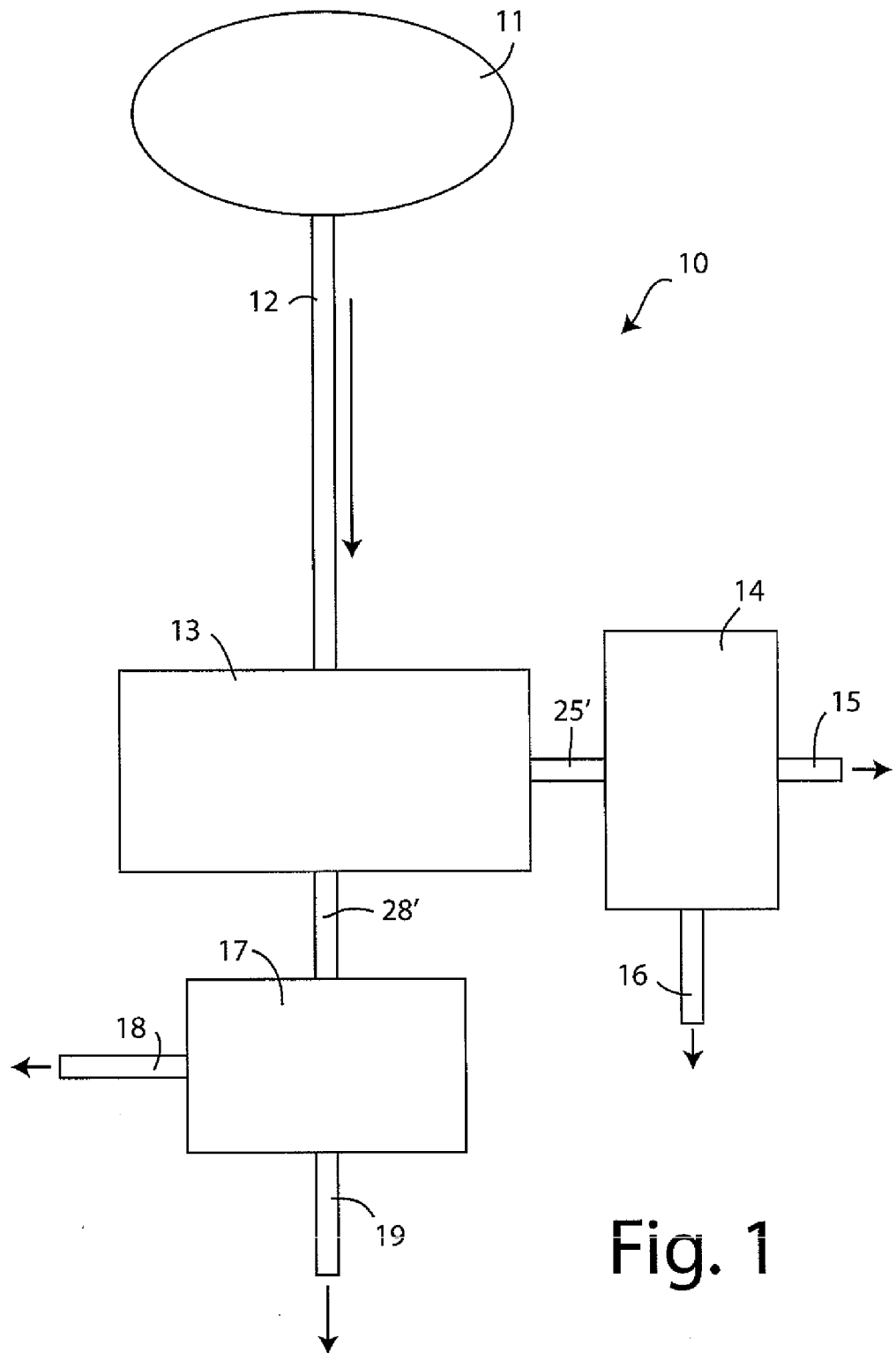
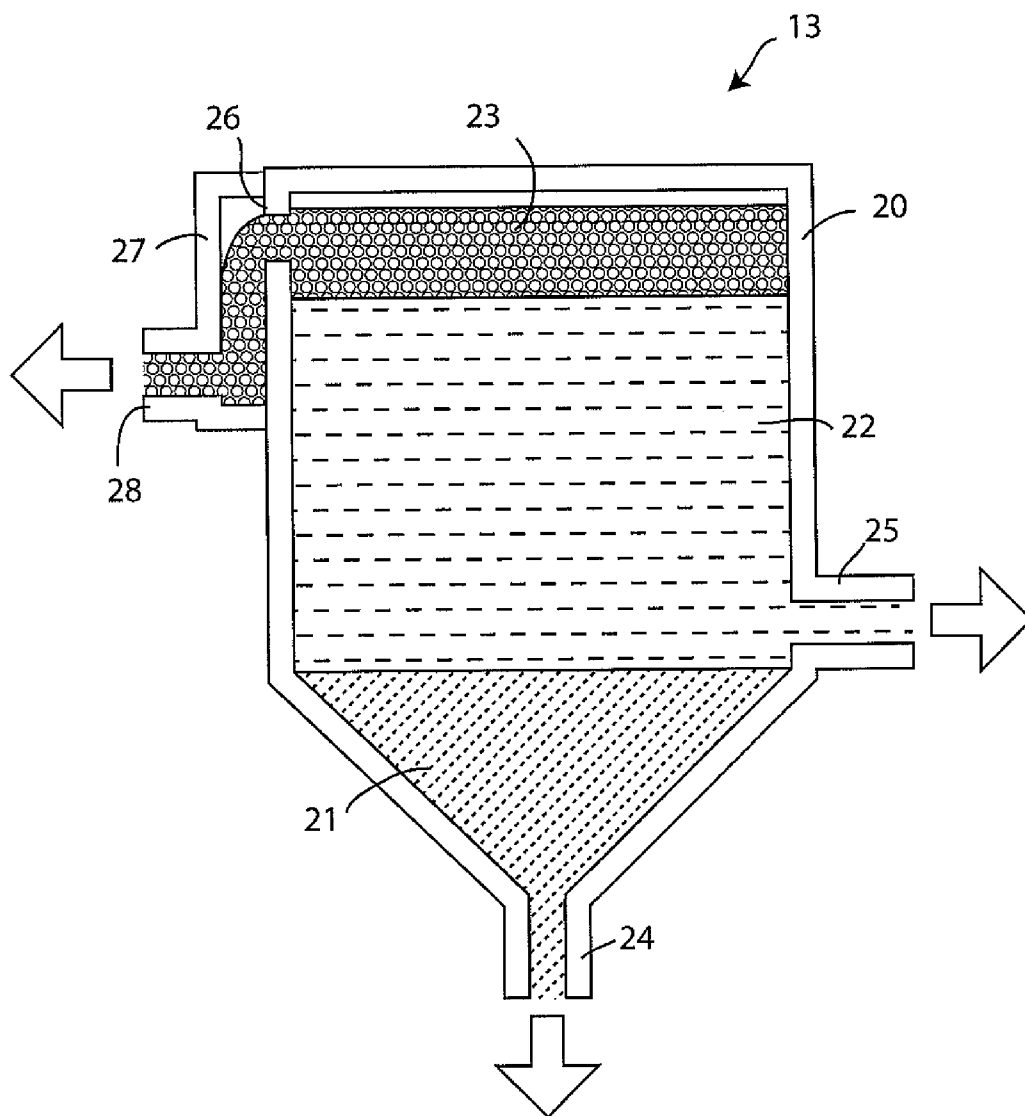


Fig. 1

2/2

Fig. 2



INTERNATIONAL SEARCH REPORT

International application No.
PCT/CA2013/050262

A. CLASSIFICATION OF SUBJECT MATTER IPC: C10G 1/04 (2006.01) , C10C 3/08 (2006.01) 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) C10G 1/04 (2006.01) , C10C 3/08 (2006.01) Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Electronic database(s) consulted during the international search (name of database(s) and, where practicable, search terms used) Canadian Patents Database, TotalPatent (d-limonene, bitumen, surfactant, nonionic surfactant, acetone, isopropyl alcohol, citrus terpene)														
C. DOCUMENTS CONSIDERED TO BE RELEVANT <table border="1"> <thead> <tr> <th>Category*</th> <th>Citation of document, with indication, where appropriate, of the relevant passages</th> <th>Relevant to claim No.</th> </tr> </thead> <tbody> <tr> <td>X</td> <td>US2012234548 A1 (Dyer, R.) 20 September 2012 (20-09-2012) * Paragraphs [0036]-[0043] *</td> <td>1-6, 8-15</td> </tr> <tr> <td>X</td> <td>WO9909134 A1 (Levey, M.) 25 February 1999 (25-02-1999) * Page 4, lines 6-26; page 11, lines 7-10 *</td> <td>1, 3</td> </tr> </tbody> </table>			Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.	X	US2012234548 A1 (Dyer, R.) 20 September 2012 (20-09-2012) * Paragraphs [0036]-[0043] *	1-6, 8-15	X	WO9909134 A1 (Levey, M.) 25 February 1999 (25-02-1999) * Page 4, lines 6-26; page 11, lines 7-10 *	1, 3			
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☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.														
<table border="0"> <tr> <td>* Special categories of cited documents :</td> <td>"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</td> </tr> <tr> <td>"A" document defining the general state of the art which is not considered to be of particular relevance</td> <td>"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</td> </tr> <tr> <td>"E" earlier application or patent but published on or after the international filing date</td> <td>"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</td> </tr> <tr> <td>"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)</td> <td>"&" document member of the same patent family</td> </tr> <tr> <td>"O" document referring to an oral disclosure, use, exhibition or other means</td> <td></td> </tr> <tr> <td>"P" document published prior to the international filing date but later than the priority date claimed</td> <td></td> </tr> </table>			* Special categories of cited documents :	"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	"A" document defining the general state of the art which is not considered to be of particular relevance	"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	"E" earlier application or patent but published on or after the international filing date	"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	"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)	"&" document member of the same patent family	"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	
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Date of the actual completion of the international search 23 May 2013 (23-05-2013)		Date of mailing of the international search report 28 June 2013 (28-06-2013)												
Name and mailing address of the ISA/CA Canadian Intellectual Property Office Place du Portage I, C114 - 1st Floor, Box PCT 50 Victoria Street Gatineau, Quebec K1A 0C9 Facsimile No.: 001-819-953-2476		Authorized officer Chi Wing Hung (819) 994-7614												

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
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