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(54) Title: COLLECTOR COMPOSITION FOR REVERSE FLOTATION

(57) Abstract: The invention relates to a reversed froth flotation process wherein as a collector a composition comprising a. a salt of an alkyl aromatic sulfonic acid and b. a fatty acid or a salt thereof is used, and to specific collector compositions that are of use in such a process.



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COLLECTOR COMPOSITION FOR REVERSE FLOTATION

The invention relates to a process of reversed froth flotation of ores and to a composition suitable for use as collector in such flotation processes of ores.

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US 4,364,824 describes a reverse flotation process for removing alkaline earth metal carbonate impurities from a phosphate concentrate obtained from phosphorite/dolomite ores, including the steps of (a) reagentizing the phosphate concentrate with a phosphate depressant, a carbonate collector and a pH regulator, and adjusting the solids content to about 20-30% with water to make a flotation feed; and (b) subjecting the flotation feed to flotation to cause alkaline earth metal carbonate mineral impurities to float, and the phosphate-containing materials to sink to thereby remove said alkaline earth carbonate mineral impurities as an overflow tail to waste, and to collect as an underflow a phosphate concentrate containing substantially less alkaline earth metal carbonate impurities, wherein the carbonate collector is selected from the group consisting essentially of sulfonated fatty acids having a linear carbon chain of 8-22 carbon atoms and in which the sulfur moiety is attached directly to a carbon atom in the fatty acid.

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There is a need for collectors which cause a higher efficiency of the flotation process, in particular a better selectivity in separation of desired components and impurities, and hence an improved and higher recovery of desired ores.

25 The invention now provides a process of froth flotation of ores wherein the process is a reversed flotation process and wherein as a collector a composition is used comprising:

- a. a salt of an alkyl aromatic sulfonic acid and
- b. a fatty acid or a salt thereof.

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Additionally, the present invention provides a composition suitable for use in such process comprising:

a. a salt of an alkyl aromatic sulfonic acid and

b. a fatty acid or a salt thereof,

wherein at least one of the components a. and b. is present as an ammonium salt.

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It has been found that performing the process according to the invention results in a higher efficiency, in particular in a better selectivity in separation of desired components and impurities, and hence in an improved and higher recovery of desired ores.

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The flotation process of the invention is a reversed flotation process. Reversed flotation means that the desired ore is not concentrated in the froth, but in the residue of the flotation process. The process of the invention is preferably a reversed flotation process for phosphate ores, more preferably for calcite-containing phosphate ores. In a reversed flotation process for concentration of calcite-containing phosphate ores, the pH during flotation in a preferred embodiment is suitably in the range of 4 to 6, preferably in the range of 4.8 to 5.5, especially in embodiments where a depressant is used that is a phosphate or phosphoric acid.

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The reversed froth flotation process of the invention, in an embodiment, comprises the steps of

- mixing a ground ore with an aqueous medium, preferably water;
- optionally, conditioning the mixture with a depressant;
- 25 - optionally, adjusting the pH;
- conditioning the mixture with collector as defined herein;
- introducing air into the conditioned water-ore mixture;
- skimming off the froth formed.

30 The composition of the present invention is a composition that is very beneficially used in a reversed froth flotation process as claimed, especially in a reversed froth flotation process of phosphate ores. When using either one or

both of the components a. and b. as the ammonium salts, it was found possible to make the collector in a high concentration while still being handleable as a liquid. This has as an advantage that solvent (including water that can be a solvent) does not have to be transported.

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It should be noted that a number of documents disclose the use of aromatic sulfonic acid and/or fatty acid-containing collector compositions, but all these documents relate to direct, i.e. not reversed, flotation processes. For example, *Indian Chem Eng.*, Vol. 52, No. 3, July 2010 discloses a comparison of several soap emulsions as flotation collectors. In the Examples the soap emulsions that are based on either tall oil or a mixed fatty acid are mixed with optional further ingredients which in Experiments 3 and 7 are a linear alkyl benzene sulfonate (LABS). As it is called a sulfonate, it appears that a salt of sulfonic acid has been used, but the salt is not explicitly disclosed. The ore for the flotation tests is a phosphate ore.

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CN101791591 discloses a direct froth flotation process of a phosphate ore using a collector of cottonseed oil, fatty acid methyl ester and dodecyl benzene sulfonic acid sodium salt. Finally, US 5,173,176 discloses dialkylated aryl monosulfonic acid salts useful as collectors in direct froth flotation processes of especially oxide ores. In some Examples the sodium sulfonic acid salts are used in combination with e.g. oleic acid or a fatty acid mixture.

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It should be noted that apart from not disclosing a reversed froth flotation process, none of the above documents discloses a collector composition in which either one of the components a and b as defined above is present as its ammonium salts.

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EP 498 231 discloses formulations of a triethanolamine salt of C10-C12 alkyl benzene sulfonic acid to which rape seed oil is added. However, these formulations are used as biocidal or agrochemical compositions and contain as additional components a toxicant and a silicone defoamer. Toxicants are highly

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undesirable in froth flotation processes because of environmental issues and therefore banned by law for this application in the majority of countries. A silicone defoamer will inherently make a froth flotation process unfeasible, as it will prevent the formation of the needed froth. Hence, the disclosed formulations
5 are inherently not suitable for use in a reversed froth flotation process

As mentioned above, in the process of the invention the collector composition comprises a salt of an alkyl aromatic sulfonic acid. Aromatic sulfonic acids are compounds wherein a sulfonic acid group is directly linked to a carbon atom of
10 an aromatic ring system. Suitable aromatic ring systems include single ring systems, as well as fused aromatic rings and multiple aromatic ring structures. Examples of suitable aromatic structures include benzene, naphthalene, indene, anthracene and phenanthrene. It is preferred that the aromatic structure is a derivative of benzene. In addition to the sulfonic acid group directly linked to a
15 carbon atom of an aromatic ring system, at least one alkyl group is linked to the aromatic ring system. Typically, the alkyl group has 4 to 24 carbon atoms. The alkyl group may be a linear alkyl group, a cycloaliphatic alkyl group, or a branched alkyl group. Suitably, the alkyl group is branched. The aromatic ring and the alkyl group may have further substituents, if so desired. Generally, the
20 alkyl aromatic sulfonic acid is a monomeric compound and is not part of a repeating unit of a polymer. Typically, the aromatic ring is substituted by one alkyl group and by one sulfonic acid group.

Alkyl aromatic sulfonic acids can be prepared by sulfonation of alkyl aromatic
25 precursors with concentrated sulfuric acid or sulfur trioxide. The reaction mechanism of sulfonation corresponds to an electrophilic aromatic substitution. Alkyl benzenes which are subjected to sulfonation are predominantly sulfonated in the *para* and *ortho* positions.

30 The alkyl aromatic sulfonic acid is present as a salt, which means that the sulfonic acid group has been at least partially neutralized by a base. The sulfonic acid may be fully or partially neutralized. The sulfonic acid is preferably

neutralized with a sub-stoichiometric (i.e. 90-99% on molar basis) amount of base. In one embodiment, the counter ion of the sulfonic acid salt is a metal ion, for example an alkali or alkaline earth metal ion, such as sodium or potassium. Alternatively and preferably, the counter ion is an ammonium cation. The ammonium cation is a positively charged polyatomic ion with the chemical formula NR_4^+ . It is formed by the protonation of ammonia or an amine. Ammonium is also a general name for positively charged or protonated substituted amines and ammonium cations (NR_4^+), where one or more hydrogen atoms are replaced by organic radical groups, indicated by R.

10

It is more preferred that the alkyl aromatic sulfonic acid salt in the composition used in the process of the invention is the salt of an alkylamine, i.e. an alkyl ammonium salt. More preferably, the aromatic sulfonic acid salt is a branched alkyl ammonium salt, such as the salt of isopropyl amine. In one embodiment, the aromatic sulfonic acid salt is the isopropyl amine salt of a branched C_{12} alkyl benzene sulfonic acid.

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The composition used in the process of the invention further comprises a fatty acid or a salt thereof. A fatty acid is a carboxylic acid with a long aliphatic chain, which is either saturated or unsaturated. Most naturally occurring fatty acids have a chain of an even number of carbon atoms, from 4 to 28. Fatty acid chains differ by length, often categorized as short to very long.

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- Short-chain fatty acids (SCFA) are fatty acids with aliphatic groups of fewer than six carbons (i.e. butyric acid).
- Medium-chain fatty acids (MCFA) are fatty acids with aliphatic chains of 6–12 carbons.
- Long-chain fatty acids (LCFA) are fatty acids with aliphatic chains of 13 to 21 carbons.
- Very long chain fatty acids (VLCFA) are fatty acids with aliphatic tails longer than 22 carbons.

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Any of the above-mentioned types of fatty acids as well as mixtures thereof can be used according to the invention. Fatty acids having aliphatic chains of 6 to 22

carbon atoms are preferred. Tall oil fatty acid, which consists mainly of palmitic acid, oleic acid and linoleic acid, may be specifically mentioned as a suitable mixture of fatty acids.

- 5 It has been found that the efficiency of the flotation process can be improved when the fatty acid is at least partially neutralized by a base. The fatty acid may be fully or partially neutralized. Preferably, the fatty acid may be neutralized with a just below stoichiometric (i.e. 90-99% on molar basis) amount of base. In one embodiment, the counter ion of the sulfonic acid salt is a metal ion, for example
10 an alkali or alkaline earth metal ion, such as sodium or potassium. Alternatively and preferably, the counter ion is an ammonium ion, as defined above. In one more preferred embodiment, the fatty acid is neutralized with ethanol amine.

- The weight ratio of alkyl aromatic sulfonic acid salt a. to fatty acid b. in the
15 composition as used in the process of the invention may vary widely. Generally, the weight ratio of a. to b. is from 10:90 to 90:10, such as 30:70 to 70:30.

- The composition as used in the process of the invention is preferably liquid at ambient temperature, i.e., at least in the range of 15 to 25°C but preferably in
20 the whole range of between 10 and 50°C. It has been found that concentrated liquid compositions can be obtained, which require only a low amount of water to be liquid. Generally, the amount of water in the composition is in the range of 0 to 25% by weight of the composition, or 0 to 15% by weight.

- 25 The invention also relates to certain preferred collector compositions that are advantageously used in the process of the invention. As indicated, these are compositions suitable for use in such process comprising:
- a. a salt of an alkyl aromatic sulfonic acid and
 - b. a fatty acid or a salt thereof,
- 30 wherein at least one of the components a. and b. is present as an ammonium salt.

The compositions of the invention comprise a salt of an alkyl aromatic sulfonic acid as component a. Aromatic sulfonic acids are compounds as described above for the process. The composition further comprises a fatty acid or a salt thereof as component b. The fatty acid in component b. is a fatty acid as
5 described above when describing the process.

As said, the counter ion for at least one of either components a. or b. or for both is an ammonium cation. The ammonium cation is a positively charged polyatomic ion with the chemical formula NR_4^+ . It is formed by the protonation of
10 ammonia or an amine. Ammonium is also a general name for positively charged or protonated substituted amines and ammonium cations (NR_4^+), where one or more hydrogen atoms are replaced by organic radical groups, indicated by R.

In the ammonium cation of the formula NR_4^+ , throughout this document, at least
15 one R group is hydrogen and up to 3 R groups are organic radical groups. More preferably, the ammonium cation is the salt of an alkylamine compound containing 1 to 3 alkyl groups (i.e. a primary, secondary or tertiary amine), wherein each alkyl independently has between 1 and 6 carbon atoms, even more preferably 1 to 4 carbon atoms, and wherein each alkyl group optionally
20 may contain heteroatoms such as oxygen or nitrogen, like an alkanolamine or aminoalkylamine (alkyleneamine).

In another more preferred embodiment, the aromatic sulfonic acid salt in the composition is a branched alkyl ammonium salt, such as the salt of isopropyl
25 amine. In one particular embodiment, the aromatic sulfonic acid salt is the isopropyl amine salt of a branched C_{12} alkyl benzene sulfonic acid.

In another more preferred embodiment, the fatty acid and/or aromatic sulfonic acid is neutralized with 90 to 99%, most preferably 95 to 98%, of the
30 stoichiometric amount of amine. In yet another more preferred embodiment, the fatty acid is neutralized with ethanol amine.

In a more preferred embodiment in the composition of the present invention both component a. and component b. are present as ammonium salt.

5 The weight ratio of alkyl aromatic sulfonic acid salt a. to fatty acid b. in the composition of the invention may vary widely. Generally, the weight ratio of a. to b. is from 10:90 to 90:10, such as 30:70 to 70:30.

10 The composition is preferably liquid at ambient temperature, i.e., at least in the range of 15 to 25°C. It has been found that when using at least one of the components a and b as their ammonium salts, concentrated liquid compositions can be obtained, which require only a low amount of water to be liquid. Generally, the amount of water in the composition is in the range of 0 to 25% by weight of the composition, or 0 to 15% by weight.

15 The process of the invention may involve other additives and auxiliary materials which are typically present in a froth flotation process that can be added at the same time or preferably separately added during the process. Further flotation aids that may be present in the flotation process are depressants (see below), frothers/froth regulators (such as pine oil, MIBC (methylisobutyl carbinol) and
20 alcohols (such as hexanol and alcohol ethoxylates/propoxylates, such as diethylene glycol mono methyl ether), and pH-regulators.

As indicated above in an embodiment, in a reversed froth flotation process oftentimes depressants are added, which in the event that phosphate ores are
25 treated can be phosphate mineral depressants. Examples of such phosphate depressants are polysaccharides, such as alkalized starch or dextrin; phosphates, such as phosphoric acid, sodium phosphate, poly phosphoric acid; sodium silicate (water glass) and soda ash. Preferred depressants are phosphoric acid, sodium phosphate and poly phosphoric acid When using
30 phosphate depressants it is furthermore preferred to ensure that the pH is in the range in which the depressant and/or the collector is performing best. For example, when using phosphoric acid, it is best to ensure that the pH of the

conditioned aqueous medium is between 4 and 6 and to optionally adjust the pH to within this range by adding an acid or base to the medium.

In another aspect, the present invention relates to a pulp comprising crushed
5 and ground phosphate ore, a collector composition as defined herein, and optionally a depressant and further flotation aids.

In a preferred embodiment the process of the invention is a flotation process wherein the ore is a phosphate ore. In a particular embodiment, the phosphate
10 ore contains calcite (CaCO_3) as impurity to be removed in the flotation process.

The amount of the collector used in the process of reversed flotation of the present invention will depend on the amount of impurities present in the ore and on the desired separation effect, but in some embodiments will be in the range of from 100 to 2,000 g/ton dry ore, preferably in the range of from 200 to 1,500
15 g/ton dry ore.

Examples

Raw materials:

ABS	Branched C12 alkyl benzene sulfonate isopropyl amine salt, 90% in water
TOFA	Tall oil fatty acid
FASK	Fatty acid sulfonate potassium salt, 50% in water
MEA	Mono ethanol amine

20 Preparation of compositions

The following compositions were prepared by mixing the components in the weight ratios indicated.

Collector composition 1

Component	Parts by weight
ABS	43.2
TOFA	39.0
MEA	7.8
Diethylene glycol mono ethyl ether	10.0

Collector composition 2

Component	Parts by weight
ABS	52.6
TOFA	47.4

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Comparative collector composition A

Component	Parts by weight
FAK	100

10 Comparative collector composition B

Component	Parts by weight
TOFA sodium salt	100

Comparative collector composition C

Component	Parts by weight
ABS	100

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The compositions were tested as collector for reversed flotation of calcite from a sedimentary phosphate ore from Saudi Arabia with about 24% P_2O_5 containing about 40% calcite.

320 g of the ore in about 1.3 l tap water of 20°C were conditioned for 30 seconds together with 1,500 g/t phosphoric acid. Then 2,000 g/t sulfuric acid were added and further conditioned for 30 seconds. Then the collector was added as 3.2% aqueous solution and conditioning continued for another 60
 5 seconds. The pH was kept at 5.0 – 5.2 during the conditioning. Subsequently flotation was performed by switching on an air inlet with air flow of about 3.5 l/min for 2 minutes.

Floated material and remaining in the cell were dried in an oven, weighed and analyzed for P₂O₅ content by X-ray fluorescence.

10

The results are summarized in the following Table I.

Collector	Dosage	Weight recovered	P ₂ O ₅ recovery	P ₂ O ₅ content in recovered material
	g/ton ore	%	%	%
1	663	67.7	92.8	32.8
1	771	68.3	92.8	33.4
1	879	65.6	91.7	34.1
2	806	68.2	91.5	32.6
2	901	66.4	91.1	33,3
A	1,224	64.5	86.2	32.7
A	1,535	57.0	79.7	34.4
B	1,270	88.5	Too few solids float	
C	700	No particles in the foam, but a lot of foam		

From Table I it can be inferred that the process of the invention provides an
 15 improved balance of P₂O₅ recovery and P₂O₅ enrichment in the recovered material.

When FASK (comparative composition A) is used as collector, the recovery of P₂O₅ is not satisfactory, meaning that too much P₂O₅ is lost.

When TOFA (comparative composition B) is used as collector alone, some froth is formed but not much material is floated even at quite high dosage (1,270 g/ton). Weight recovery has to be in the range of 60-75% in order to float enough calcite to increase the P_2O_5 grade in the cell product (concentrate). The
5 weight recovery using TOFA became 88.5%, which means too little calcite is floated.

When ABS (comparative composition C) is used as collector alone, a lot of foam is formed but the foam does not contain any particles, meaning no flotation occurs at all.

10 Furthermore, it is shown that compositions using both the alkyl aromatic sulfonic acid and the fatty acid component at least partly as their ammonium salts (composition 1, the monoethanolamine will neutralize the tall oil fatty acid to its monoethanolammonium salt almost stoichiometrically) are slightly better than compositions using one of them as their ammonium salt (composition 2)

Claims

1. A process of froth flotation of ores, wherein the process is a reversed flotation process and as a collector a composition is used comprising:
 - 5 a. a salt of an alkyl aromatic sulfonic acid and
 - b. a fatty acid or a salt thereof.
2. The process according to claim 1, wherein the ore is a phosphate ore.
- 10 3. The process according to claim 1 or 2, wherein component a. is an alkyl ammonium salt of an alkyl aromatic sulfonic acid.
4. The process according to any one of preceding claims 1 to 3, wherein component a. is a branched alkyl ammonium salt of an alkyl aromatic sulfonic acid.
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5. The process according to any one of preceding claims 1 to 4, wherein the alkyl group of the alkyl aromatic sulfonic acid is a branched alkyl group.
20
6. The process according to any one of preceding claims 1 to 5, wherein the alkyl aromatic sulfonic acid is an alkyl benzene sulfonic acid.
7. The process according any one of preceding claims 1 to 6, wherein
25 component b. is present in the salt form.
8. The process according to claim 7, wherein component b. is an ammonium salt of a fatty acid.
- 30 9. The process according to any one of preceding claims 1 to 8, wherein the amount of water in the composition is in the range of 0 to 15% by weight of the composition

10. A composition suitable for use in the process of preceding claims 1 to 9 comprising:
- a. a salt of an alkyl aromatic sulfonic acid and
 - b. a fatty acid or a salt thereof,
- 5 wherein at least one of the components a. and b. is present as an ammonium salt.
11. The composition according to claim 10, wherein component a. is an alkyl ammonium salt of an alkyl aromatic sulfonic acid.
- 10 12. The composition according to claim 10 or 11, wherein component a. is a branched alkyl ammonium salt of an alkyl aromatic sulfonic acid.
- 15 13. The composition according to any one of preceding claims 10 to 12, wherein the alkyl group of the alkyl aromatic sulfonic acid is a branched alkyl group.
14. The composition according to any one of preceding claims 10 to 13, wherein the alkyl aromatic sulfonic acid is an alkyl benzene sulfonic acid.
- 20 15. The composition according to any one of claims 10 to 14, wherein component b. is an ammonium salt of a fatty acid.

INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER INV. B03D1/008 B03D1/012 B03D1/02 ADD. B03D101/02 B03D103/06		
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) B03D		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) EPO-Internal, WPI Data		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
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Y	page 4, line 30 - line 34; claims 1,11,16 column 3, line 43	8,15
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A	US 5 173 176 A (KLIMPEL RICHARD R [US] ET AL) 22 December 1992 (1992-12-22) claim 1; example 6; table VI -/-	1-15
☒ Further documents are listed in the continuation of Box C. ☒ See patent family annex.		
* Special categories of cited documents : "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier 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 22 June 2016		Date of mailing of the international search report 01/07/2016
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016		Authorized officer Roider, Josef

INTERNATIONAL SEARCH REPORT

International application No

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