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PROCESS FOR CONCENTRATING ORES

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The invention relates to a froth flotation process for the separation of mineral raw materials, particularly sulphidic ores, for example, for the separate concentration of galena and sphalerite.

It has already been proposed to add alkyl polysiloxanes as flotation oil to an aqueous suspension of galena and quartz containing dissolved a froth-stabilizing component and to produce the galena in a froth layer separately from the depositing quartz sand by blowing in air. An industrially applicable process according to this suggestion and especially a process by which the sulphides of several metals may be separated from one another has not yet become known. Xanthates have hitherto preferably been used as so-called collectors in the flotation of sulphides.

The invention provides a flotation process for the selective separation of sulphidic ores with the use of conventional frothing agents such as terpene alcohols or those based on pine oil, and organopolysiloxanes as collectors, characterised by using the collector in the form of an emulsion of liquid organopolysiloxanes or solid organopolysiloxanes dissolved in a water-immiscible liquid, and using as emulsifier for this emulsion a surface-active nitrogen-containing organic compound. Especially advantageous organopolysiloxanes have proved to be those which contain on an average at least one alkyl radical with more than two carbon atoms per 4 Si-atoms, and among those again the organopolysiloxanes containing 3–10 siloxane units in an unbranched open chain. In this connection, a hydrocarbosiloxane may be used consisting of linear unbranched chains of more than 2 and less than 11 siloxane units per molecule wherein 75% of the hydrocarbon radicals of the hydrocarbosiloxane are methyl and 25% are radicals having more than 2 carbon atoms. Besides, for example, an octahydrocarbotrisiloxane may be used having one hydrocarbon radical of more than 2 carbon atoms, the other hydrocarbon radicals being methyl, such as an octaalkyltrisiloxane which has one alkyl radical of more than 2 carbon atoms, the other alkyl radicals being methyl. More specifically, octadecyl-(heptamethyl)-trisiloxane has been used with effective results. It is not necessary to produce the emulsion serving as collector prior to its addition to the suspension; it is also sufficient to add directly the siloxane liquid and the emulsifier to the pulp and to effect the emulsification and the froth production in the pulp.

It has further been found that it is advantageous to carry out the flotation of sulphidic lead-zinc ores in such a manner that the galena is initially floated out from an alkaline pulp, preferably at pH values between 8 and 10 and then sphalerite after acidification of the pulp to pH values of about 4 and after the addition of copper sulphate, preferably in amounts between 50 and 250 g. per 1000 kg. of raw ore to be concentrated. For a better depression of the sphalerite during the flotation of galena it is advantageous to add to the pulp before the beginning zinc sulphate in an amount between 300 and 600 g. per 1000 kg. of raw ore of the pulp.

The emulsion applied according to the invention contains between 0.5 and 2 percent by weight of the nitrogen-containing emulsifier and expediently about 40 percent by weight of organosiloxane; an amount of between 2 and

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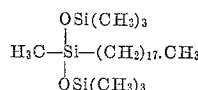
100 g., preferably between 7 and 30 g. of this emulsion is added to the pulp per 1000 kg. of raw ore. As emulsifiers which may be used in accordance with the invention are, quaternary alkyl ammonium salts, such as dodecyl-(dimethyl)-benzyl-ammonium chloride, and acyl-hydroxyalkylamide sulphates, such as especially the sodium salt of stearyl-hydroxyethylamide sulphate



The following example of operation demonstrates the advantages of the process according to the invention compared with an analogous process in which xanthates are used as collector.

Example

For producing the collector component according to the invention, 400 g. of polysiloxane of the formula



were emulsified with 12 g. of the sodium salt of stearyl hydroxyethylamide sulphate in 588 g. of water.

1 kg. of a West German lead-zinc ore having a grain size below 0.1 mm. containing 2.5 percent by weight of Pb and 9.5 percent by weight of Zn was placed in a 2.5 litre experimental flotation cell. To this there were added

- 10 cc. of an aqueous solution of sodium meta silicate containing 3.5 percent by weight of Na_2SiO_3
- 3 cc. of an aqueous solution of zinc sulphate containing 10 percent by weight of ZnSO_4
- 10 mg. of the aforescribed polysiloxane emulsion
- 1 drop (34 mg.) of a commercial synthetic terpene alcohol
- 272 cc. of a saturated aqueous caustic lime solution (1.18 g. CaO per litre)

After making up with water to 2.5 litres, air was beaten into the resultant pulp, the pH-value being 9.4. The froth layer thus formed on the surface of the pulp, rich in galena, was extracted for 2 minutes; it yielded the first concentrate of 31 g. with a content of 60 percent by weight of Pb and 10 percent by weight of Zn, equal to 74% Pb and 3% Zn of the total of the raw ore used. After a further 3 minutes a second concentrate was extracted, i.e. 21 g. with 18 percent by weight of Pb and 16 percent by weight of Zn (15% Pb and 3% Zn of the total used).

Then, there were added to the pulp:

- 10 cc. of 2 N sulphuric acid
- 1 cc. of an aqueous solution of copper sulphate containing 10 percent by weight of CuSO_4
- 10 mg. of the aforescribed polysiloxane emulsion
- 1 drop of terpene alcohol as above and another
- 10 cc. of 2 N sulphuric acid

A Zn concentrate of 126 g. with a content of 0.6 percent by weight of Pb and 53 percent by weight of Zn equal to 3% Pb and 70% Zn of the raw ore used were then extracted from the aerated pulp for 2 minutes. A second zinc concentrate was extracted during the following 3 minutes, namely 59 g. with 2 percent by weight of Pb and 33 percent by weight of Zn (4% Pb and 21% Zn of the total used).

The loss, i.e. the residue remaining in the flotation cell was 763 g. with a content of 0.1 percent by weight of Pb and 0.3 percent by weight of Zn.

The lead content of the raw ore was thus floated out after 5 minutes to 89% in the form of 52 g. of lead concentrates, the metal content of which amounting to 43 percent by weight of Pb and 12 percent by weight of Zn, and the Zn content of the raw ore upon flotation of the

residual pulp for 5 minutes to 91% in the form of 185 g. of zinc concentrates with a metal content of 47 percent by weight of Zn and 1 percent by weight of Pb.

For comparison, an experimental flotation was carried out according to the known art using xanthates as follows:

To the same amount of 1 kg. of West German lead-zinc ore having a grain size of below 0.1 mm. and containing 3.3 percent by weight of Pb and 9.8 percent by weight of Zn in the same cell as above there were added

- 3 cc. of an aqueous solution of sodium metal silicate containing 3.5 percent by weight of Na_2SiO_3
- 2.5 cc. of an aqueous solution of sodium carbonate containing 10 percent by weight of Na_2CO_3
- 20 cc. of an aqueous solution of potassium cyanide containing 1 percent by weight of KCN
- 4.5 cc. of an aqueous solution of zinc sulphate containing 10 percent by weight of ZnSO_4
- 10 drops (0.65 cc.) of an aqueous solution of potassium ethyl xanthate containing 10 percent by weight of $\text{C}_2\text{H}_5\text{OCS.SK}$
- 1 drop (34 mg.) of terpene alcohol as above

The mixture was made up with water, and air beaten in as described above. The resultant froth cover was withdrawn for 1 minute and yielded a first concentrate of 38 g. with a content of 55 percent by weight of Pb and 9 percent by weight of Zn equal to 63% of Pb and 3% of Zn of the total contained in the raw ore. During a further 3 minutes a second concentrate was withdrawn, namely 33 g. containing 20 percent by weight of Pb and 11 percent by weight of Zn (20% Pb and 4% Zn of the total used). In the course of 3 minutes each there was withdrawn a third and fourth concentrate:

- (3) 20 g. containing
 - 4 percent by weight of Pb and
 - 9 percent by weight of Zn
 - (2% of the total Pb and
 - 2% of the total Zn)
- (4) 9 g. containing
 - 3 percent by weight of Pb and
 - 8 percent by weight of Zn
 - (0.8% of the total Pb and
 - 0.7% of the total Zn)

After stopping the aeration there were added to the pulp:

- 5 cc. of an aqueous solution of copper sulphate containing 10 percent by weight of CuSO_4
- 11 drops (0.46 cc.) of an aqueous solution of potassium hexylxanthate containing 10 percent by weight of $\text{CH}_3(\text{CH}_2)_5\text{OCS.SK}$

From the re-aerated pulp a zinc concentrate of 27 g. with a content of 1 percent by weight of Pb and 56 percent by weight of Zn, equal to 0.9% Pb and 16% Zn of the total contained in the raw ore was then extracted in the course of 2 minutes. During the following 3 minutes a second concentrate was extracted, namely 87 g. containing 1 percent by weight of Pb and 53 percent by weight of Zn (3% Pb and 47% Zn of the total used).

A further drop of the aforesaid terpene alcohol was then added and, thereupon, a third zinc concentrate of 50 g. was extracted after 3 minutes containing 2 percent by weight Pb and 43 percent by weight of Zn, that is to say 3% of the total Pb and 22% of the total Zn. In the course of 3 minutes each there were extracted a fourth and fifth concentrate and, finally, a sixth concentrate during 7 minutes:

- (4) Zinc concentrate, 19 g.; 3%/w. Pb, 25%/w. Zn
 - 2% Pb of the total
 - 5% Zn of the total
- (5) Zinc concentrate, 15 g.; 3%/w. Pb, 3%/w. Zn
 - 1% Pb of the total
 - 0.4% Zn of the total

- (6) Zinc concentrate, 38 g.; 1%/w. Pb, 0.5%/w. Zn
 - 2% Pb of the total
 - 0.2% Zn of the total

The loss amounted to 664 g. containing 0.1 percent by weight Pb and 0.05 percent by weight Zn.

In this case the lead content of the raw ore was floated out after a flotation period of 10 minutes to 86% in the form of 100 g. of lead concentrate the metal content of which amounted to 29 percent by weight Pb and 10 percent by weight Zn, and the zinc content of the raw ore after flotation of the remaining pulp for 21 minutes to 90% in the form of 236 g. of zinc concentrates with a metal content of together 37 percent by weight Zn and 2 percent by weight Pb.

The comparison teaches that by the process according to the invention a substantially higher concentration of the ores is attained in a shorter flotation period. If in each case the proportions of the metals in the concentrates obtained are compared with the ratio in the raw ore the process according to the invention results in a calculated factor of 13 for the lead concentration and a factor of 12 for the zinc concentration; in the experiment using xanthate instead of polysiloxane the respective factors were only 9 for lead and 8 for zinc.

We claim:

1. In a process for the concentration of sulphidic lead-zinc ores with the use of conventional frothing agents and organo-polysiloxanes as collectors by using the collector in the form of an emulsion of a member selected from the group consisting of liquid hydrocarbopolysiloxanes and solid hydrocarbopolysiloxanes dissolved in a water-immiscible liquid, and using as emulsifier for this emulsion a surface-active organic nitrogen compound selected from the group consisting of alkyl ammonium salts and acyl-hydroxyalkylamide sulphates, the improvement which comprises initially floating out galena from an alkaline pulp, at pH values between 8 and 10, and, then sphalerite after acidification of the pulp, to a pH value of about 4 and after the addition of copper sulphate, in amounts between 50 and 250 g. per 1000 kg. of raw ore to be concentrated.

2. Process according to claim 1 which comprises adding to the pulp zinc sulphate in an amount between 300 and 600 g. per 1000 kg. of raw ore prior to the extraction of the lead ore.

3. In a process for the concentration of sulfidic ores by blowing air into an aqueous suspension of the ore in the presence of a frothing agent and a hydrocarbopolysiloxane the step which comprises adding to said aqueous suspension of the ore (1) a linear alkylpolysiloxane of more than two and less than 11 siloxane units per molecule, 75% of the alkyl radicals of said alkylpolysiloxane being methyl and 25% of said alkyl radicals having more than 2 carbon atoms, and (2) a surface-active organic nitrogen compound selected from the group consisting of alkyl ammonium salts and acyl-hydroxyalkyl amide sulphates.

4. In a process for the concentration of sulfidic ores by blowing air into an aqueous suspension of the ore in the presence of a frothing agent and a hydrocarbopolysiloxane the step which comprises adding to said aqueous suspension of the ore (1) an octahydrocarbotrisiloxane having one hydrocarbon radical of more than 2 carbon atoms, the other hydrocarbon radicals being methyl, and (2) a compound selected from the group consisting of dodecyl-(dimethyl)-benzyl ammonium chloride and the stearyl hydroxyethylamide sulfates.

5. In a process for the selective concentration of sulfidic lead-zinc ores by blowing air into an aqueous suspension of the ore in the presence of a frothing agent and an alkylpolysiloxane, the step which comprises adding to said aqueous suspension of the ore (1) a linear alkylpolysiloxane of more than 2 and less than 11 siloxane units per molecule, one of said siloxane units per each 4 thereof having one alkyl radical of more than 2 carbon atoms, all

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remaining alkyl radicals being methyl, and (2) a surfactant selected from the group consisting of quaternary ammonium salts and acyl-hydroxyalkylamide sulfates.

6. In a process for the selective concentration of sulfidic lead-zinc ores by blowing air into an aqueous suspension of the ore in the presence of a frothing agent and an alkyl-polysiloxane, the step which comprises adding to said aqueous suspension of the ore (1) an octaalkyltrisiloxane having one alkyl radical of more than 2 carbon atoms, the other alkyl radicals being methyl, and (2) a compound selected from the group consisting of dodecyl-(dimethyl)-benzyl-ammonium chloride and the stearyl-hydroxyethylamide sulfates.

7. In a process for the selective concentration of sulfidic lead-zinc ores by blowing air into an aqueous sus-

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pension of the ore in the presence of a frothing agent and an alkyl-polysiloxane, the step which comprises adding to said aqueous suspension of the ore octadecyl-(heptamethyl)-trisiloxane and a stearyl-hydroxyethylamide sulfate.

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