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FROTH FLOTATION REAGENT

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This invention relates to the concentration of ores by froth flotation and has for its objects the utilization of a novel class of reagents for use as collectors for the more positive particles of the ore. In a particular aspect the instant invention relates to the use of chlorinated saturated fatty acids as collectors for phosphate rock particles in the flotation of phosphate rock from siliceous gangue.

The prior art recognizes the use of unsaturated fatty acids in the flotation of electro-positive particles from less positive constituents of an ore. Specifically, the prior art teaches the use of fatty acids in the flotation of phosphate rock from siliceous materials, and in the flotation of halite from sylvinite ores. The preferred fatty acid for such flotation operations is oleic acid. However, mixtures of fatty acids rich in unsaturated fatty acids of about 18 to 20 carbon atoms, and particularly mixtures of fatty acids rich in oleic acid, are used. The prior art has avoided the use of saturated fatty acids, except as they occur naturally in mixtures rich in unsaturated fatty acids, because the saturated fatty acids have been found to be ineffective, or only marginally effective, as collectors. For example, in the froth flotation of phosphate rock from siliceous gangue more than 10 times as much stearic acid as oleic acid must be employed to obtain the same distribution of bone phosphate of lime, and even then the assay of the concentrate obtained is poor when this much larger quantity of stearic acid is used.

It has now been found, and this invention is based in part upon this discovery, that saturated fatty acids which are unsatisfactory collectors can be converted by simple halogenation to provide collectors having an efficiency comparable to that of oleic acid. The collectors useful in this invention are halogenated saturated fatty acids having 12 to 22 carbon atoms per molecule. The halogenation is carried out readily by techniques described

in the prior art, and the reaction is continued to the point at which the product halo-acids display melting points in the range of −10 to +40° C. and more preferably in the range of 0 to 20° C. Particularly preferred is the use of chlorinated saturated fatty acids having 16 to 20 carbon atoms per molecule, which contain 20 to 50% by weight of chlorine, and which display melting points in the range of 0 to 20° C. Especially preferred is the use of chlorinated palmitic acid containing about 25% by weight of chlorine, and chlorinated stearic acid containing about 48% by weight of chlorine.

In accordance with this invention, the flotation operation is carried out in the usual manner. In the froth flotation of phosphate rock from a siliceous gangue-containing ore, the ore is conditioned by treatment with a hydrocarbon oil and a collector consisting essentially of halogenated C_{12} to C_{22} saturated fatty acids. As is typical of the techniques of the prior art, the hydrocarbon oil preferably consists of a mixture of hydrocarbon oils such as a 1:1 mixture of kerosene and bunker "C" oil. Other hydrocarbon materials and other ratios can, of course, be used. The hydrocarbon oil is preferably employed in the amount of about 2 lbs. per ton of ore. The halogenated fatty acid collector is employed in the amount of about 0.4 to 2.5 lbs. per ton of ore treated, and preferably in the amount of 1 to 2 lbs. per ton of ore treated. During the ore conditioning operation, which may require as little as 2 minutes, the pH is controlled in the range of about 7 to 9 by the addition of an appropriate caustic, such as aqueous sodium hydroxide. The pH in the flotation cell will ordinarily be somewhat lower, in the range of 6 to 8.5.

No marked difference has been observed between the use of chlorinated and brominated saturated fatty acids. The degree of halogenation is, however, critical in that it has been found that the melting point of the product acids varies in inverse proportion to the extent to which the halogenation is carried, and that to impart characteristics desirable in the use of the materials as collectors the halogenation must be carried out to produce a product having a melting point in the range of −10° C. to 40° C., and more preferably in the range of 0 to 20° C.

Random halogenated products have been prepared by passing chlorine gas through the warm acid and have

TABLE I.—COMPARISON OF FLOTATION RESULTS OBTAINED WITH HALOGENATED AND UNHALOGENATED FATTY ACIDS

Run	Product			#/T	Concentrate			Tail
	Name	M.P.°	Percent Halogen		Wt. Percent	Percent BPL		Percent BPL Assay
						Assay	Dist.	
1	Alpha-Bromo Palmitic	55	4.7	2.8	36.1	62.8	82.2	7.7
				2.4	34.3	63.4	78.9	8.9
2	Alpha-Chloro Palmitic	37	9.5	.72	42.0	59.5	91.2	4.1
				.60	33.8	65.0	77.2	9.8
3	Alpha-Bromo Stearic	34.9	18.8	1.6	44.6	57.5	92.8	3.6
				1.4	38.1	59.0	81.2	8.4
4	9-10 dichlorio stearic	30	21.0	1.4	42.4	62.7	92.9	2.8
				1.1	39.9	63.0	92.0	3.7
5	Chlorinated Stearic #1	14.6	21.8	0.56	44.9	57.8	93.9	3.2
				0.48	41.7	61.1	91.4	4.2
6	Chlorinated Palmitic	9.0	25.4	0.38	33.4	63.1	76.7	9.6
				0.44	41.0	63.9	92.3	3.7
7	Chlorinated Stearic #2	7.9	48.2	0.39	38.0	64.3	90.5	4.3
				0.57	46.4	55.7	93.8	3.2
8	9-10 diBromo Stearic	−13	36.0	0.47	44.0	58.6	93.5	3.2
				0.38	39.3	61.3	85.5	6.8
9	Stearic acid	69.4	0	0.9	42.5	61.1	93.9	3.0
				0.68	39.2	63.2	89.2	4.9
10	Palmitic	64.0	0	3.8	31.2	62.3	69.8	12.2
				2.3	43.6	57.0	89.5	5.2
11	Soap skimmings		0	1.9	39.1	59.6	85.3	6.6
				0.49	40.6	63.0	94.8	2.4
				0.38	34.6	66.7	89.5	4.8

been found to be effective as specific compounds, such as dihalogenated stearic acid. Also, it has been found that products formed by the halogenation of random mixtures of saturated fatty acids provide excellent results in the method of this invention. The fatty acid materials may contain minor proportions of unsaturated fatty acids without detrimental effect. Metallurgical experiments were carried out using Florida pebble phosphate flotation feed of about −35 +150 mesh using a conditioning time of 2 minutes and 2 lbs. per ton of a 1:1 ratio of bunker "C" oil and kerosene. The fatty acids were converted to their sodium soaps and added as the sodium soaps, rather than by separate addition of the acid and caustic to the conditioner. The pH was not regulated but was generally about 8 in the conditioner and about 7 in the flotation cell. The results of the experiments are recorded in Table I.

It will be noted that the halogenated saturated fatty acids provide much higher collector efficiency than the saturated fatty acids from which they were derived. It will also be noted that chlorinated palmitic acid and chlorinated stearic acid of Runs 6 and 7 which display melting points in the range of 0 to 20° C. are outstanding collection agents, and show efficiencies comparable to the soap skimmings of Run 11. These soap skimmings consist pricipally of the sodium soap of oleic acid.

This invention has been illustrated by the description of the flotation of phosphate rock from siliceous gangue. It will be understood, however, that the halogenated saturated acids of this invention can be employed in other froth flotation separations where unsaturated fatty acids, specifically oleic acid, has hereto been used. The invention is useful in the flotation of more positive particles from less positive constituents of the ore. Examples of such operations include the flotation of halite from sylvite, and the flotation of langbeinite from sylvinite.

While the instant invention has been described with particular emphasis upon the several preferred embodiments thereof, and has been illustrated by detailed examples, it will be understood that within the scope of the appended claims the invention can be practiced otherwise than has been specifically described.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. In the beneficiation of phosphate ore containing siliceous gangue and phosphate rock by froth flotation, the improvement comprising conditioning said ore prior to flotation at a pH in the range of 6 to 9 in the presence of hydrocarbon oil and 0.4 to 2.5 lbs. per ton of said ore of a collector consisting essentially of C_{12} to C_{24} halogenated saturated fatty acids having melting points in the range of −10° C. to 40° C.

2. The method in accordance with claim **1** in which said halogenated fatty acids display melting points in the range of 0° C. to 20° C.

3. The method in accordance with claim **1** in which said collector consists essentially of chlorinated saturated fatty acids.

4. The method in accordance with claim **3** in which said chlorinated fatty acids contain 16 to 20 carbon atoms and display melting points in the range of 0° to 20° C.

5. The method in accordance with claim **4** in which said chlorinated fatty acids contain 20 to 50% by weight of chlorine.

6. The method in accordance with claim **5** in which said collector is employed in the amount of about 1 to 2 lbs. per ton of ore.

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