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FROTH FLOTATION OF SILICIOUS GANGUE
FROM AN ALKALINE MAGNETIC IRON ORE
PULP WITH AN AMINEFred D. De Vaney, Hibbing, Minn., assignor to
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poration of MinnesotaNo Drawing. Application December 30, 1943,
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3 Claims. (Cl. 209-166)

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The present invention relates to the art of beneficiating iron-bearing materials by froth flotation. The invention is particularly concerned with an improvement in the froth flotation method of separating magnetite-bearing particles from naturally associated particles of silicious gangue material by the aid of a flotation reagent of the cationic type. The improved process is applicable to the beneficiation of magnetite concentrates per se and generally if iron-bearing materials containing magnetite in association with other oxidic iron minerals. It provides an advantageous mode of raising the grade of magnetic concentrates obtained from iron-bearing materials, e. g. ores, containing silicious gangue.

It heretofore was known that by the use of certain flotation reagents, known as "cationics" or "cationically acting flotation reagents," it was possible to float "acidic" particles, e. g., silica, away from associated particles of hematite and some other iron oxide minerals, the former being recovered in the froth product and the latter in the underflow of the flotation cell. However, it also was known that with use of the usual cationic collectors magnetite has a much greater tendency to float than has hematite, this "floating tendency" on the part of magnetite being akin to, although of a somewhat lower order than, that of silica. Thus, when a mixture of particles of silica and particles of magnetite is subjected to cationic froth flotation, more than half of the magnetite floats with the silica. For this reason, separations between the two had been relatively poor, and it had been believed that froth flotation of the cationic type was practically ineffectual for separating silica from magnetite.

It is an object of the present invention so to adapt the cationic type of froth flotation process as to make the same effectual in the separation of silica (and similarly acting gangue) particles from particles of magnetite, or from particles of magnetite admixed with other oxidic iron minerals. It is an object of the invention, also, to provide a method by which artificially to increase the difference between the floatabilities of silica and of magnetite to such extent that a desirably close approach to clean separation between the two may be made possible. Another inventive object is the provision of an improved cationic froth flotation method by which the "floatability" of magnetite particles may be depressed without,

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however, at the same time materially depressing the floatability of the associated silicious gangue particles.

I have found that the above, and other, inventive objects can be realized, in an advantageous manner, by the use of a suitable conditioning agent in the pulp. I have found that magnetite particles can be depressed into the underflow, without materially affecting the "floatability" of the silicious gangue particles, by conditioning the pulp with a small addition of sodium silicate. Other operable magnetite-depressing conditioning agents are the alkaline-acting alkali-metal compounds, e. g., caustic soda, sodium carbonate, caustic potash and potassium carbonate, and ammonium hydroxide (or ammonium carbonate). Use of the conditioning agent does not adversely affect the course of the hematite (and other iron mineral) particles, and does not materially depress the silica particles: so far as has been observed, its effect is selective with respect to the magnetite particles. This mode of effecting desirable depression of magnetite has been found to be particularly effective when the cationic collector being employed is a primary or secondary aliphatic amine, especially primary and secondary aliphatic amines having aliphatic chains corresponding in length to those of coconut oil fatty acids.

The following specific examples are illustrative but non-limitative of the improved process of the present invention.

Example 1

The starting material was a sample of Mesabi taconite containing about 45% of gangue material (mostly silica) and about 35.25% Fe as magnetite, hematite, martite, siderite and greenalite, of which minerals the first three were the major components. This material, crushed and ground to minus 100 mesh, was subjected to magnetic separation whereby there was obtained a magnetic concentrate assaying 63.25% Fe and 9.43% silica.

The magnetic concentrate, after having been passed through a demagnetizing coil, was made up into an aqueous pulp of about 25% solids content. The pulp was conditioned with 2.0 lbs./ton of sodium silicate and a small amount of AM 1120 (the collector employed) for 5 minutes, and thereupon was treated in a 500 g. me-

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chanically agitated flotation cell. As frother there was used methyl amyl alcohol, 0.05 lb./ton. During the course of the flotation treatment additional amounts of the collector were added to the pulp, in small increments. The total amount of collector used (in the conditioning step plus during the flotation operation) was 0.2 lb./ton. The results were as follows:

Product	Per Cent Wt.	Assay Per Cent		Per Cent of Total Fe
		Fe	SiO ₂	
Froth.....	29.15	58.12	15.16	25.61
Underflow (Conc.).....	70.85	65.36	6.80	74.39
	100.00	63.25	9.24	100.00

The collector referred to above as "AM 1120" is a product of Armour & Company and is understood to consist substantially of mono-n-dodecylamine. Because of the poor water-solubility of the amine per se, the latter was employed, in the above test, in the form of the corresponding hydrochloride.

In a comparison test the sodium silicate was omitted, this omission being the only deviation from the conditions set out in the above example. The comparative results were as follows:

Product	Per Cent Wt.	Assay Per Cent		Per Cent of Total Fe
		Fe	SiO ₂	
Froth.....	84.16	63.21	9.42	84.03
Underflow (Conc.).....	15.84	63.85	8.54	15.97
	100.00	63.31	9.28	100.00

It will be observed that in the absence of the sodium silicate the separation (as between silica and iron minerals) was relatively very poor, considerable of the magnetite having floated with the silica.

Example 2

In a repetition of the test described in Example 1 above, using the same starting material, cationic collector and frother, and observing the same technique, 0.5 lb./ton caustic soda was substituted for the 2.0 lbs./ton sodium silicate, with the following results:

Product	Per Cent Wt.	Assay Per Cent		Per Cent of Total Fe
		Fe	SiO ₂	
Froth.....	12.05	55.51	18.67	10.58
Underflow (Conc.).....	87.95	64.31	7.94	89.42
	100.00	63.25	9.24	100.00

Example 3

The starting material for this test was a demagnetized magnetic concentrate made from the metamorphosed taconite from the Eastern Mesabi district. The material contained considerable iron silicate mineral, and the iron oxide particles (magnetite and other oxides) were very finely disseminated through the matrix, which latter circumstance had necessitated grounding the taconite to minus 150 mesh in order to unlock as much as possible of the iron oxide particles. The magnetic concentrate analyzed 62.12% Fe and 10.97% silica.

This starting material was made up into an aqueous pulp of about 25% solids content, and

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was pre-heated for about 5 minutes with sodium silicate in an amount corresponding to 1.4 pounds per ton of ore. The resulting pulp was then treated in a 500 g. mechanically agitated flotation cell by the use of 0.15 lb./ton of "AM Coco" as cationic collector and 0.05 lb./ton of du Pont No. 60 frother. The "AM Coco" collector, a product of Armour & Company, is understood to consist essentially of a mixture of primary and secondary aliphatic amines corresponding in alkyl chain length to fatty acids of coconut oil. It was employed, in this test, in the hydrochloride form and in relatively dilute aqueous solution; the solution was added to the cell contents in small increments during the flotation operation. The results were as follows:

Product	Per Cent Wt.	Assay Per Cent		Per Cent of Total Fe
		Fe	SiO ₂	
Froth.....	13.76	42.56	28.95	9.44
Underflow (Conc.).....	86.24	65.24	8.10	90.56
	100.00	62.12	10.97	100.00

The relatively large iron content of the froth product was attributable to the large amount of iron silicate in the latter.

It will be observed that the rejection of this froth product yielded a concentrate of high grade. The process had effected a desirably sharp separation between the magnetite (and the other iron oxides associated therewith) and the iron silicate as well as the silica.

The improved process of the present invention is not limited to operating upon magnetic concentrates. It is equally applicable in other froth flotation processes involving the separation of silicious gangue material from the magnetite (and other associated oxides of iron) of iron ores or ore-like iron-bearing materials. The following specific example is illustrative of the application of the improved process to the beneficiation of a magnetite-containing silicious iron ore which had not been subjected to magnetic concentration.

Example 4

The starting material was a sample of silicious iron-bearing material, taken from the Majorca mine of the Mesabi Range and known locally as "oxidized taconite." This material contained 30.62% iron, of which 3.1% was present as magnetite, the balance being hematite with relatively small amounts of limonite and siderite. The magnetite content was determined by making a magnetic concentration test on ore crushed to minus 100 mesh.

In the first test 500 g. of minus 100 mesh ore, in the form of an aqueous pulp of about 25% solids, was treated in a mechanically agitated flotation cell with the usual cationic collector and frothing reagent; no sodium silicate or alkali was used to depress the magnetite. The results were as follows:

Product	Per Cent Wt.	Assay Per Cent		Per Cent Total Iron
		Iron	Silica	
Froth (Tailing).....	66.81	16.48	-----	35.97
Underflow (Conc.).....	33.19	58.95	11.05	64.03
	100.00	30.61	-----	100.00

Reagents: .35 lb./ton "AM Coco"; .05 lb./ton methyl amyl alcohol; pH of pulp 7.2.

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The magnetite content had a marked tendency to float, and much of it was lost with the silica in the overflow. This tendency was proved by making a magnetic concentration test on the flotation products.

When, in a repetition of the above test, the pulp was conditioned for a few minutes with a small amount (less than one pound per ton of ore) of caustic soda before the flotation operation, the magnetite was depressed into the underflow along with the hematite (and other non-magnetic oxidic compounds of iron), and the flotation results—tabulated below—were markedly improved.

Product	Per Cent Wt.	Assay Per Cent		Per Cent Total Iron
		Iron	Silica	
Froth (Tailing)-----	63.95	14.37	-----	29.99
Underflow (Conc.)-----	36.05	59.45	10.62	70.01
	100.00	30.62	-----	100.00

Reagents: Same as above, except ore conditioned 5 min. with 0.75 lb./ton caustic soda; pH of pulp 8.9.

It will be noted that the relative amount of concentrate was increased, and that the grade of the concentrate was improved. Magnetic concentration tests on the flotation products established the fact that the conditioning treatment influenced as sharp a separation of magnetite (from silica) as of the other oxidic iron minerals.

This improved process lends itself to incorporation into a magnetic separation method of beneficiating oxidic iron ores or ore-like material containing magnetite, in that the magnetic separation step may be so conducted that the concentrate resulting therefrom contains all or practically all of the magnetite of the starting material along with silicious middling particles, whereupon the latter may, by employment of the present process, be floated away from the magnetite leaving a clean magnetite concentrate. Such combination of steps is described and claimed in my application Serial No. 508,849, filed November 3, 1943, patented November 6, 1945, No. 2,388,471.

This application contains subject matter in common with my application Serial No. 500,002, filed August 25, 1943, now abandoned. Reference is made also to my application Serial No. 505,697, filed October 9, 1943, containing related subject matter.

I claim:

1. Process of beneficiating a ferruginous chert whose oxidic iron content is largely magnetite which chert has been finely subdivided to a particle size of the order of minus 100 mesh or finer, which comprises adding to an aqueous pulp of the chert an agent of the group consisting of alkaline-acting alkali metal compounds, ammonium

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hydroxide and ammonium carbonate in amount sufficient to make said pulp at least as alkaline as pH 8, and subjecting the alkaline pulp to cationic froth flotation treatment using a frother and as cationic collector a water-soluble form of an aliphatic amine whose aliphatic radical corresponds in length to that of a fatty acid of coconut oil, whereby to obtain a silicious froth product poor in magnetite and an underflow concentrate containing most of the magnetite.

2. Process of beneficiating a ferruginous chert whose oxidic iron content is largely magnetite which chert has been finely subdivided to a particle size of the order of minus 100 mesh or finer, which comprises adding to an aqueous pulp of the finely divided chert an agent of the group consisting of alkaline-acting alkali metal compounds, ammonium hydroxide and ammonium carbonate in amount sufficient to confer upon said pulp an alkalinity equivalent to a pH of at least 8 and subjecting the alkaline pulp to cationic froth flotation treatment using a frother and as cationic collector a water-soluble derivative of dodecylamine, whereby to obtain a silicious froth product poor in free magnetite and an underflow concentrate containing most of the free oxidic iron contents of the pulp.

3. Process of separating magnetite-bearing particles from associated silicious gangue particles in an aqueous pulp of a silicious iron ore material whose iron content is largely magnetite which material has been finely subdivided to a particle size of the order of minus 100 mesh or finer, which comprises adding to the pulp an agent of the group consisting of alkaline-acting alkali metal compounds, ammonium hydroxide and ammonium carbonate in amount sufficient to make said pulp at least as alkaline as pH 8, and subjecting the alkaline pulp to cationic froth flotation treatment using a frother and as cationic collector a water-soluble form of an aliphatic amine whose aliphatic radical corresponds in length to that of a fatty acid of coconut oil, whereby to obtain a silicious froth product poor in magnetite and an underflow concentrate containing most of the magnetite.

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