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FROTH FLOTATION PROCESS

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4 Claims. (Cl. 209—166)

This invention relates to a process of ore beneficiation and, more particularly, to a froth flotation process for concentrating nonmetallic and some metallic minerals.

In the flotation of minerals from ores to which this invention is applicable, i.e., Florida phosphate rock, barite, fluorspar, cement rock, iron ore, ilmenite and so on, it is standard practice to use anionic collecting agents to float the valuable mineral from the gangue. The collectors commonly used for this purpose are tall oil, tall oil fatty acids, petroleum sulfonates, red oil, oleic acid and similar fatty acids, soaps and analogous surface-active agents. Other flotation agents, such as frothers and modifying agents, may be used along with the collector to enhance its activity.

A principal object of the present invention is the provision of an efficient and economic froth flotation process of the indicated type for recovering valuable minerals from ores.

Another and more specific object of the invention is the provision of a froth flotation process in which a new, efficient and low cost collecting agent is used to recover valuable minerals from ores.

The novel collecting agent employed in the process of the present invention is saponified tall oil pitch. Tall oil pitch is the residue or still bottoms resulting from the fractional distillation of crude tall oil whereby the fatty and resin acids and other distillable materials are removed leaving the pitch. It is a tarry, dark brown substance, the specific characteristics of which vary somewhat depending on the composition of the crude tall oil, the conditions of treatment and so on. The following is an analysis of a typical sample of tall oil pitch obtained by the fractional distillation of tall oil:

Color (Gardner—50% in benzene)	18
Saponification No.	124.9
Acid No.	70.4
Iodine No.	167.4
Rosin acids (Wolff method, percent)	29.8
Total fatty acids,[1] percent	40.9
Lignin, percent (about)	10.0
Other unsaponifiables, percent	19.3
Ash, percent	0.37
Water, percent	0.00

[1] Free and esterified.

The process of the invention may be carried out using conventional procedures except for the inclusion of the novel collecting agent. In accordance with such procedures, the ore is first ground, usually in the presence of water, to the proper particle size for liberation of the mineral component from the gangue. The ground material is then screened to remove the oversized particles and the resultant pulp, in most cases, deslimed to remove the colloidal particles which are harmful to the separation by the flotation process. The deslimed pulp, at 20–75% solids, is then usually conditioned for a short time with the chemical reagents to be used, particularly the collector and the frother and, in some cases, modifying agents, following which flotation is carried out in any of the various types of machines commonly used for this purpose. Customarily the valuable mineral is floated.

However, the procedure may be reversed with the valuable mineral concentrated in the tailing.

The following examples will serve to illustrate the invention. In these examples the tall oil pitch used for the saponification was obtained by the fractional distillation of crude tall oil and had the following typical analysis:

Acid Number	40–60
Rosin acids, percent	15–20
Fatty acids	7–12
Saponification number	115–125
Unsaponifiables	20–25

The saponification was carried out by reacting the tall oil pitch with 7–8 pounds of caustic soda per 100 pounds of pitch in an autoclave at 30–45 pounds per square inch pressure for about one hour. The pitch was heated first to 150–180° C. before addition of the caustic as a 20% aqueous solution. The average reaction temperature was 145° C. with 40 pounds per square inch pressure. The final saponified product, in each case, was water-soluble, had a solids content of about 80% and contained from about 0.5–2.0% free alkali as NaOH.

Example 1

Six hundred fifty (650) grams of wet Florida phosphate rock of about 28% B.P.L. (tricalcium phosphate) was scrubbed for 2 minutes in a 500 gram Denver flotation machine. The scrubbed ore was mixed with fresh water which was decanted off carrying with it the slimes. This operation was repeated twice to remove all the slimes. The deslimed sample was then conditioned at 70% solids for 2 minutes with the following:

NaOH	0.19 lb./ton of original ore.
No. 20 gravity fuel oil	1.38 lb./ton.
Saponified tall oil pitch	0.81 lb./ton.

The conditioned ore was transferred to the flotation machine, diluted with water to about 25% solids, and the phosphate mineral floated off. A concentrate of 42.2% of the original weight was obtained which analyzed 61.9% B.P.L. (tricalcium phosphate). The tailing contained only 2.6% B.P.L., showing a good recovery of the mineral.

Example 2

Six hundred fifty (650) grams of a different wet phosphate rock of about 28% B.P.L. was scrubbed and deslimed as described in Example 1. The deslimed sample was conditioned at 70% solids for 2 minutes with the following reagents:

NaOH	0.19 lb./ton of ore.
No. 20 gravity fuel oil	1.40 lbs./ton.
Saponified tall oil pitch	0.72 lb./ton.

The conditioned ore was floated at about 25% solids which gave a concentrate of 37.4% by weight. It contained 15.7% hydrochloric acid insoluble material. The tailing analyzed 95.2% HCl insoluble showing that practically all the phosphate had been recovered in the flotation concentrate.

Example 3

Six hundred (600) grams of specular hematite iron ore was ground to minus 65 mesh in a ball mill in the presence of about the same amount of water. To the slurry was added 0.5 lb./ton of sodium silicate and the ore was deslimed by diluting with water and decanting. This was repeated twice to remove the slimes which amounted to about 10%. The deslimed ore was conditioned at 65–70% solids for 5 minutes with the following reagents:

No. 2 fuel oil	1.2 lbs./ton.
Saponified tall oil pitch	0.70 lb./ton.

The rougher concentrate was cleaned once. The results obtained were:

	Percent Wt.	Percent Fe	Percent Fe Recovery
Head Sample	100.0	40.4	100.0
Rougher Tail	23.1	6.4	3.6
Cleaner Tail	14.3	15.2	5.4
Clean Concentrate	62.6	58.9	91.0

Thus, a high grade iron concentrate was obtained in good yield.

Example 4

In this example a commercial carbonaceous rock flotation feed of 20% solids was used. It contained about 67% $CaCO_3$. Nine hundred (900) milliliters of this slurry was transferred to a 200 gram flotation cell and the reagents added to produce the individual floats as shown.

Reagents	Lb./Ton		Percent Wt.	Percent $CaCO_3$	Percent $CaCO_3$ Recovery
No. 2 fuel Oil	0.09				
Methyl Amyl Alcohol	0.14	Float 1	23.9	67.5	23.1
Saponified Tall Oil Pitch	0.68				
Tall Oil Fatty Acids	0.18	Float 2	33.8	79.6	38.5
Saponified Tall Oil Pitch	0.23				
Tall Oil Fatty Acids	0.09	Float 3	31.0	75.4	33.5
		Tail	11.3	30.2	4.9

The first float contained the bulk of the carbonaceous material plus some of the calcium carbonate produced by the saponified tall oil pitch plus a small amount of the tall oil fatty acid.

In the preparation of the collecting agents utilized in the process of the present invention tall oil pitch is reacted with an alkali, i.e., the hydroxides or carbonates of the alkali metals. The reaction may be carried out at a temperature from about 100° C. to about 200° C. and at a pressure from about 1 pound per square inch to about 100 pounds per square inch. The preferred conditions of temperature and pressure are from about 150 to about 180° C. and from about 30 to about 45 pounds per square inch pressure. The alkali is preferably added to the molten tall oil pitch as an aqueous solution at a concentration from about 20 to 50% depending on the solids content desired in the product. The amount of alkali may be varied but should be sufficient to more than neutralize all the acidity of the pitch. It is preferred to utilize an amount of alkali such that the final saponified product contains from about 0.5–2.0% free alkali as NaOH. The saponified tall oil pitch, prepared as described, is water-soluble or at least water-dispersible. If desired, the paste product can be spray dried to give a free-flowing powdered material.

The objects of the invention are realized by employing saponifed tall oil pitch in amounts from about 0.1 to 5 pounds per ton of ore, and preferably 0.3 to 1.0 pounds per ton in flotation processes for beneficiation of ores. In most instances, it is advantageous to employ other

flotation agents from the class of collectors, frothers, and modifying agents along with the collector of this invention to achieve the most effective separation of the minerals from the ore. Typical collectors, frothers and modifying agents which may be used with the collectors of this invention are disclosed in U.S.P. 2,545,132.

The collector of this invention may be used generally in the flotation beneficiation of ores but is most advantageously employed on nonmetallic ores and oxide ores. For example, the flotation of barite, apatite, fluorite, calcite, gypsum, spodumene, ilmenite, hematite, magnetite, chromite, magnesite and so on are some of the minerals which may be concentrated by the collector of this invention.

An important advantage of this invention is the conversion of a cheap raw material, with limited use in flotation and most difficult handling properties, into a water-soluble product of valuable collecting properties for a wide variety of minerals. While preferred embodiments of the invention have been exemplified, the invention is not intended to be limited to the specific details described except as they may be included in the following claims.

What I claim and desire to protect by Letters Patent is:

1. The process of concentrating an ore selected from the group consisting of nonmetallic ores and oxide ores by froth flotation which comprises incorporating a water-dispersible saponified tall oil pitch prepared by reacting tall oil pitch with alkali at temperatures from about 150° C. to about 180° C. and at pressures from about 30 lbs./sq. in. to about 45 lbs./sq. in. in an aqueous ore pulp of the indicated type, agitating the pulp to form a froth, and separating the froth from the pulp.

2. The process of concentrating an ore selected from the group consisting of nonmetallic ores and oxide ores by froth flotation which comprises forming an aqueous ore pulp from such an ore, adding to said pulp from about 0.1 to 5 pounds per ton of ore of a saponified tall oil pitch prepared by reacting tall oil pitch with alkali at temperatures from about 150° C. to about 180° C. and at pressures from about 30 lbs./sq. in. to about 45 lbs./sq. in., agitating the pulp to form a froth, and separating the froth from the pulp.

3. The process of concentrating an ore selected from the group consisting of nonmetallic ores and oxide ores which comprises finely grinding such an ore and forming an aqueous ore pulp therefrom, adding to said pulp from about 0.1 to 5 pounds per ton of ore of a saponified tall oil pitch prepared by reacting tall oil pitch with alkali at temperatures from about 150° C. to about 180° C. and at pressures from about 30 lbs./sq. in. to about 45 lbs./sq. in., agitating the pulp to form a froth and separating the froth from the pulp.

4. The process of claim 3 in which the saponified tall oil pitch contains free alkali.

References Cited in the file of this patent

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UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 2,987,183 June 6, 1961

William T. Bishop

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 2, line 9, for "Rosin acids" read -- Free Rosin Acids (calculated as abietic acid) --; line 10, for "Fatty acids" read -- Free Fatty Acids (calculated as oleic acid) --; column 3, line 15, after "carbonaceous" insert -- cement --.

Signed and sealed this 7th day of November 1961.

(SEAL)
Attest:

ERNEST W. SWIDER
Attesting Officer

DAVID L. LADD
Commissioner of Patents

USCOMM-DC