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[54] LIQUID CATIONIC FLOTATION COMPOSITION

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[58] **Field of Search** 209/166, 167; 212/61

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[57] **ABSTRACT**

A liquid, temperature-stable, cationic ore concentration flotation composition prepared from an aliphatic alcohol and the reaction product of a fatty acid, diethylene triamine, and glacial acetic acid.

3 Claims, No Drawings

LIQUID CATIONIC FLOTATION COMPOSITION

The present invention relates to the beneficiation or concentration of ores. More particularly, the present invention relates to a method for the concentration of phosphate minerals by froth flotation. Specifically, the present invention relates to a froth flotation process to separate silica from phosphate minerals. In another of its aspects, this invention relates to a reagent effective in the concentration and beneficiation of ores.

Phosphate rock, a naturally occurring ore which is a crude phosphatic material, consists of more or less impure noncrystalline calcium fluorophosphate. Phosphate rock occurs widely in nature and normally is in association with silica as a major gangue constituent. In addition to the silica, other gangue constituents present include silicates, calcium carbonate, carbonaceous material, heavy minerals, and the like, all of which contain no phosphorus.

Beneficiation and concentration of phosphate rock and other phosphatic materials is a rather old and well-developed art. In general, most processes include slurrying the naturally occurring ore in water, separating the debris from the slurry and classifying the solids according to particle size. The large particles which are frequently referred to as "pebble" phosphate can be used directly in the manufacture of phosphoric acid or triple superphosphate. The intermediate sized fraction, generally considered to be about −14 mesh, however, contains a substantial amount of siliceous material and fines which must be separated from the phosphate if recovery of a product of satisfactory grade is to be obtained.

To this end, the intermediate fraction is generally deslimed to remove fine clay and the like, then sized into about +35-mesh and −35-mesh fractions. The separate fractions are then treated with an anionic flotation reagent, such as fatty acid, kerosene or fuel oil and an alkaline reagent such as sodium hydroxide. When an aqueous suspension of phosphatic rock is reagentized with a composition containing this composition and then agitated and aerated in a flotation cell, the phosphatic values of the rock are found to rise and become segregated in the upper portion of the suspension while the siliceous values tend to settle or deposit in the lower portion of the suspension. Although the floated product, generally referred to as "rougher concentrate," is substantially improved as to phosphate content, nevertheless, it still contains from about 8 to about 20 percent undesirable silica and further processing is required. In the past this processing has generally involved three steps. First, acid scrubbing of the concentrate is utilized to remove oil and fatty acid. Secondly, the concentrate is conditioned with a cationic flotation reagent and finally, flotation removes the reagentized silica. The depressed or sink product from this latter treatment comprises essentially phosphatic material and constitutes final concentrate. Some of the factors which affect the recovery and the grade of the phosphate flotation product are the properties of the ore, the pH of the slurry, the type and quantity of reagents, and the type and quantity of aeration. Because of the large scale character of the business of producing phosphate rock concentrates, and the highly competitive market existing in such business, it is important that the reagentizing composition be relatively inexpensive. Additionally, it is also important that the concentrate recovered be of relatively high purity; that is, relatively free of solids other than the desired phosphatic material.

Although, heretofore, amines have been used as cationic flotation reagents, in practice it has been found that many are viscous pastes which require a substantial amount of heat to render and maintain them sufficiently fluid for use. Some are extremely difficult to disperse in water, especially cold water normally used during winter months. Others form unstable emulsions which create handling, use and recovery problems, and all have apparently required the coincidental use of substantial amounts of fuel oil, kerosene or the like to obtain satisfactory separation and recovery of phosphatic minerals from the undesirable siliceous materials in admixture therewith.

It is therefore an object of the present invention to provide a reagent effective in the concentration and beneficiation of phosphate mineral ores.

It is another object of the present invention to provide a cationic flotation composition which is readily dispersible in water, fluid over a wide temperature range and at temperatures as low as 0° C. and is stable in storage over an extended period of time.

It is a further object of this invention to provide a cationic flotation composition which improves phosphate recovery from phosphatic minerals while significantly reducing the amount of fuel oil, kerosene or the like, required for flotation of siliceous materials.

Additional objects and advantages of the present invention will become apparent from the description which follows.

In the production of phosphate ore material, a conventional procedure in the beneficiation and concentration of the ore is to first treat the raw ore to a flotation process involving the use of an anionic agent such as fatty acids and fatty acid soaps, either alone or in admixture with hydrocarbon materials such as kerosene or crude oil, in order to produce what is known as the rougher concentrate. This rougher concentrate which contains upgraded phosphatic materials, is then subject to a second beneficiation step in which a cationic agent such as long chain primary fatty amines and/or their acid salts are used as the essential flotation reagent to effect a further upgrading of the ore material to separate silica and other acid insolubles present therein. Conventional agents which have been used for this purpose in the past, in addition to those mentioned above, include primary rosin amines, primary tall oil amines, mixed crude primary amines and water soluble or dispersible acid salts of the foregoing.

In accordance with the present invention, it has been discovered that good and improved results can be obtained in ore concentration processes by use of a novel cationic flotation composition which demonstrates substantially uniform operating characteristics and good stability when used in normal mining field operation, temperatures and conditions.

Briefly, the process for the preparation of the compositions of the present invention involves the steps of treating a fatty acid, preferably a tall oil fatty acid, with diethylene triamine at an elevated temperature of about 100° to about 300° C., cooling the thus formed product and treating said product with acetic acid, preferably glacial acetic acid, to neutralize or partially neutralize the formed product. Thereafter the neutralized or partially neutralized product is treated with from about 20 to 40 percent and preferably 25 to 35 percent by weight of an aliphatic alcohol having from six to nine carbon atoms and a boiling point between 131° and 161° C.

More particularly, the compositions of the present invention can be prepared by a three-step process as follows:

1. A fatty acid is reacted with diethylene triamine at a temperature between about 100° to 300° C. and preferably about 200° and 250° C., said fatty acid being characterized by (A) an acid value of 195–199, (B) a saponification value of 197–200, (C) an iodine value of 129–131 and containing from about 0.3 to 4.2 percent rosin acid, from 0.4 to 1.6 percent unsaponifiables and from about 95 to 99.3 percent total fatty acids. Ratios of fatty acid to diethylene triamine, in equivalents, of about 1:1.5 to 1:2.5 are the preferred but equivalents of 1:1 to 1:3 may be employed. The fatty acids useful in the preparation of the compositions of the present invention may be further characterized as containing 5–8 percent conjugated polyunsaturates determined as percent linoleic, 36–39 percent nonconjugated polyunsaturates determined as percent linoleic and 50–52 percent oleic determined by difference.

2. The reaction is followed by cooling of the formed product to between about 70° and 90° C. and then treating said product with from about 0.25 and 1.0 equivalent, and preferably about 0.3 to 0.6 equivalents, of glacial acetic acid.

3. The product from step 2 is treated with from about 20 to 40 percent and preferably 25 to 35 percent by weight of an aliphatic alcohol or mixture of aliphatic alcohols having from six to nine carbon atoms and a boiling point between 131° and

161° C. and preferably selected from the group consisting of methylisobutyl carbinol, heptanol and mixtures thereof.

Although there are a substantial number of alcohols which one might employ as a solvent for the above characterized partially neutralized amines, we have discovered that only those meeting the definition set forth above can be used for the preparation of the compositions of the present invention.

Lower alcohols, while initially providing the fluidity desired for flotation reagents, have vapor pressures which permit escape of alcohol vapors from the finished reagent and create combustion and odor problems in the use, storage or handling thereof. Moreover, it has been found that the reagents prepared with such alcohols do not provide the improvement in separation of silica and phosphate rock achieved with the compositions of the invention. Additionally, we have found that treatment of the above-mentioned partially neutralized amines with higher alcohols such as polyalkylene glycols and the like, give compositions which do not exhibit the enhanced performance nor have the desired fluid properties, under diverse temperature conditions, which are obtained with the inventive composition.

Thus in accordance with the usual processes for beneficiation or concentration of phosphate materials, using the composition of this invention, Florida pebble phosphate made into a rougher concentrate upgraded to about 68 percent measured in bone phosphate of lime (BPL), and preferably deoiled and deslimed, is introduced into a conventional flotation cell for amine flotation of the remaining silica. Approximately 0.25 to 1.0 pound of the composition of the invention per ton of feed is charged to the cell, and the feed conditioned in the usual manner. Aeration of the reagentized feed brings the silica to the surface of the cell and is thus removed from the feed slurry. Dewatering of the slurry yields a high phosphate concentrate of about 78 percent BPL.

Contrary to previously available partially neutralized amine compositions which are usually viscous pastes and thus require addition of heat for fluid use and are difficult to disperse in water under outdoor operating conditions, the compositions of the invention are fluid at temperatures as low as 0° C. and are readily dispersed in the cold aqueous solutions employed in the flotation systems of phosphate plants during the winter mining and processing operations. Thus in the past, these amine flotation reagents have, of necessity, had to be heated to a fluid state, dispersed in water, kerosene, pine oil, or mixtures thereof and pumped to the flotation cells. Steam jacketed lines have been employed to prevent blockage during periods of cold weather. But with the cationic flotation composition of the present invention, these requirements and difficulties are eliminated.

As mentioned previously, in the process of removing silica from phosphate rock, the conditions are such that particularly complete removal of the silica must be accomplished in order to produce a saleable phosphate material. It is, therefore, an advantage of this invention that the reagents of the present invention not only effect satisfactory removal of the silica but are economic in the amounts used. The total quantities of the inventive combination required range from about 0.25 to about 1.0 pound per ton depending upon the particular quality of the ore. The invention is not, however, limited to the use of such quantities.

It is also an economic advantage of the composition of this invention that a reduction of approximately 50 percent is achieved in the necessary amount of hydrocarbon oil necessary for the silica flotation while improving the product grade and total phosphate recovery.

But perhaps the most significant improvement of the present invention over previously available amine acetates is the provision of a cationic flotation composition which is fluid over a wide temperature range which includes from about 0° to about 55° C. and even higher, the temperature range normally encountered in the field.

Compositions of the present invention can be used alone or in admixture with other promotors, cooperating materials, such as, for example, conditioning agents, activators, frothing agents, dispersing agents, oily materials such as hydrocarbon oils, and the like.

Additionally, the compositions of the present invention are also adaptable for use in any of the ordinary concentrating processes, such as film flotation, tabling, but they are particularly useful in froth flotation.

When the compositions of the present invention are employed as a promotor in the froth flotation of silica from phosphate rock, the conditions may be varied in accordance with procedures known to those skilled in the art. The compositions can be employed in the form of aqueous solutions, emulsions, mixtures, or solutions in organic solvents and the like. The compositions can be introduced into the ore pulp in the flotation without prior conditioning, or they can be conditioned with the ore pulp prior to the actual concentration operation. They can also be stage-fed into the flotation circuit.

The following examples are intended to illustrate the underlying principles of the present invention and are not to be construed as limiting thereof.

EXAMPLE 1

14.5 equivalents of a tall oil fatty acid having an acid value of 195, a saponification value of 197 and an iodine value of 131, and containing 4.2 percent rosin acid, 1.6 percent unsaponifiables, 1.6 percent total fatty acids, 8 percent conjugated polyunsaturation (as percent linoleic), 36 percent nonconjugated polyunsaturation (as percent linoleic) and 52 percent oleic, is treated with 29 equivalents of diethylene triamine (85 percent) and heated to about 230° C. The reaction mixture is then cooled to about 80° C. and treated with 7.2 equivalents of glacial acetic acid to yield the 0.5 acetate (i.e., the partially neutralized amine).

Following the above procedure but terminating the synthesis prior to partial neutralization with glacial acetic acid yields the free base.

The fully neutralized amine (i.e., the 1.0 acetate) is also prepared by the process as described above but 14.2 equivalents of glacial acetic acid are used. Treatment of the partially or fully neutralized amine, as prepared above, with from about 20 to 40 percent by weight of an alcohol selected from the group consisting of methylisobutyl carbinol, heptanol and mixtures thereof yields the flotation compositions of the present invention.

EXAMPLE 2

14.5 equivalents of a tall oil fatty acid having an acid value of 195, a saponification value of 197 and an iodine value of 131, and containing 4.2 percent rosin acid, 1.6 percent unsaponifiables, 1.6 percent total fatty acids, 8 percent conjugated polyunsaturation (as percent linoleic), 36 percent nonconjugated polyunsaturation (as percent linoleic) and 52 percent oleic, is treated with 29 equivalents of diethylene triamine (85 percent) and heated to about 230° C. The reaction mixture is then cooled to about 80° C. and treated with 7.2 equivalents of glacial acetic acid to yield the 0.5 acetate (i.e., the partially neutralized amine).

The 0.5 acetate is then divided into two equal parts. Thirty percent by weight of methylisobutyl carbinol is thoroughly admixed with one portion of the 0.5 acetate at a temperature of 90° C. In like manner, the remaining portion of 0.5 acetate is admixed with 30 percent by weight of methylisobutyl carbinol at a temperature of 25° C. An inspection of these two samples shows that both have a similar brown, transparent characteristic appearance and are of similar consistency.

These compositions prepared at 25° and 90° C. were then blended and subjected to a stability test. The sample was divided into two portions and one was held at a temperature of −15° C. for a period of 12 hours. During this time it remained fluid and stable. The second half was stored at room temperature for a period of 3 days. At the end of this time, it too remained fluid and stable.

EXAMPLE 3

To evaluate the compositions of the present invention as to fluidity under diverse temperature conditions and emulsion stability on storage, approximately 900-gram samples of a variety of amines and amine compositions, including the compositions of the invention, commercially available amines, the free base, and a variety of related compositions were placed in quart mason jars and stored at 0° C., 25° C. and 30° C. The samples were examined daily for fluidity and emulsion stability and the results recorded. These results are reported in Table I below where it can be seen that the compositions of the invention remained fluid at 0° C. and provided stable emulsions.

TABLE I

Composition	25° C.	0° C.	Emulsion
1. 0.5 acetate, polyethylene glycol 20%	Fluid	Solid	Excellent.
2. Delamine 80 [1]	do	do	Do.
3. Delamine 80 [1]	do	do	Do.
4. Amine free base 90%, emulsifier plus ethyleneoxide	do	Viscous	Unsatisfactory.
5. Amine free base 50%, pine oil 50%	do	do	Do.
6. Amine free base 50%, pine oil 50%	do	do	Do.
7. Amine free base 84%, MIBC 16%	do	Fluid	Do.
8. Amine free base 80%, MIBC 20%	do	do	Do.
9. 0.5 acetate 69%, 2-ethylisohexanol 31%	Solid	Solid	Satisfactory.
10. 0.5 acetate 66.6%, pine oil 33.3%	Fluid	do	Do.
11. 0.5 acetate 78.3%, 6.2% MIBC, 15.5% kerosene	do	Plastic	Good.
12. 0.5 acetate 78%, 6% MIBC, 16% No. 5 fuel oil	do	do	Do.
13. 0.5 acetate 66.6%, C_6-C_9 alcohol B.P. 131-161° C. 33.3%	do	Fluid	Satisfactory.
14. 0.5 acetate 66.6%, MIBC 33.3% (2 replicates)	do	do	Do.
15. 0.5 acetate 68%, MIBC 32%	do	do	Do.
16. 1.0 acetate 83%, MIBC 17%	do	do	Do.
17. 1.0 acetate 80%, MIBC 20%	do	do	Do.
18. 0.5 acetate 66.6%, heptanol 33.3%	do	Highly Fluid	Good.
19. 0.5 acetate 66.6%, heptanol 33.3%	do	do	Do.
20. 0.5 acetate 66.6%, MIBC-heptanol [2] 33.3%	do	do	Do.
21. 0.5 acetate 66.6%, MIBC-heptanol [2] 33.3%	do	do	Do.
22. 0.5 acetate 66.6%, heptanol 33.3%	do	do	Do.

[1] Trademark of the Hercules Powder Co. for an amine, pine oil mixture.
[2] Mixture=35 pts./v. MIBC, 35 pts./v. heptanol, 40 pts./v. fuel oil No. 2.

EXAMPLE 4

425–475 g. samples of Florida pebble phosphate rougher concentrate, upgraded from about 30 percent BPL to about 68 percent BPL deoiled and deslimed, were charged to laboratory, Wemco-type flotation cells each filled with 2 liters of tap water. Test compositions were added to the cells in an amount sufficient to provide from about 0.20 to 0.50 pounds of composition per ton of feed. In runs designated as 1 and 2, 0.64 pound of kerosene per ton of feed was added. In runs designated as 3 through 20, 0.32 pound of kerosene per ton of feed was also added. The mixtures were agitated for about 5 seconds without air, then the air valves opened and the reagentized silica floated and skimmed from the surface of the cells, the unfloatable phosphate remaining in the cells.

Both the silica and the phosphate were removed, dried, weighed and analyzed for BPL content. The data obtained are reported in Table II below where it can be seen that with the compositions of the invention, phosphate losses in the tails were substantially reduced and total phosphate recovery was improved.

TABLE II

Composition	Rate	Feed BPL	Conc. BPL	Tails BPL	Silica float BPL recov.
1. 0.5 acetate, polyethylene glycol 20%	0.41	69.0	74.2	9.0	99.0
2. Delamine 80 [1]	0.40	69.0	75.4	35.4	91.9
3. Delamine 80 [1]	0.20	69.3	75.2	10.3	98.6
4. Amine free base 50%, pine oil 50%	0.36	68.7	73.7	18.3	97.6
5. Amine free base 84%, MIBC 16%	0.24	69.4	75.0	9.0	98.8
6. Amine free base 80%, MIBC 20%	0.26	68.9	74.6	11.2	98.5
7. 0.5 acetate 69%, 2-ethylisohexanol 31%	0.44	69.1	74.8	10.2	98.6
8. 0.5 acetate 66.6%, pine oil 33.3%	0.30	69.3	74.6	11.3	98.7
9. 0.5 acetate 78.3%, 6.2% MIBC, 15.5% kerosene	0.256	68.7	73.9	9.8	98.6
10. 0.5 acetate 78%, 6% MIBC, 16% No. 5 fuel oil	0.40	68.7	74.2	9.2	98.8
Average		69.0	74.6	13.4	97.9
Composition of the Invention					
11. 0.5 acetate 66.6%, C_6-C_9 alcohol B.P. 131-161° C. 33.3%	0.30	68.9	74.0	8.2	99.1
12. 0.5 acetate 66.6%, MIBC 33.3% (2 replicates)	0.30	69.0	74.0	7.5	99.2
13. 0.5 acetate 68%, MIBC 32%	0.44	68.5	73.9	10.8	98.6
14. 1.0 acetate 83%, MIBC 17%	0.36	68.8	73.4	6.0	99.4
15. 1.0 acetate 80%, MIBC 20%	0.25	68.7	71.1	7.8	99.6
16. 0.5 acetate 66.6%, heptanol 33.3%	0.30	69.5	74.9	15.2	98.0
17. 0.5 acetate 66.6%, heptanol 33.3%	0.45	68.9	74.6	16.3	97.8
18. 0.5 acetate 66.6%, MIBC-heptanol [2] 33.3%	0.30	68.0	73.3	9.9	98.8
19. 0.5 acetate 66.6%, MIBC-heptanol [2] 33.3%	0.45	68.5	74.1	10.9	98.5
20. 0.5 acetate 66.6%, heptanol 33.3%	0.30	68.1	73.5	8.5	99.0
Average		68.7	73.7	10.1	98.8

[1] Trademark of the Hercules Powder Co. for an amine, pine oil mixture. [2] Mixture = 30 pts./v. MIBC-35 pts./v. Heptanol-40 pts./v. fuel oil No. 2.

EXAMPLE 5

To evaluate the composition of the invention under standard operating conditions, flotation processing data as to percent BPL in the feed, tails and concentrate and amine and kerosene usage, were collected for a 10-day period immediately prior to substituting the compositions of the inven-

tion for the amine 0.5 acetate normally used in the system (i.e., prepared as described in paragraph 1 of Example 1). Following standard procedures 55 gallon drums of the amine 0.5 acetate (a viscous paste) is placed in a vessel of hot water maintained at about 170° F. and stirred for about 1.5 hours until the amine becomes fluid. The amine is then poured into a heated mixing kettle, dispersed in water and pumped to the flotation cells. For maximum efficiency of operation during the plant test period with water emulsion, as prepared above, 0.340 pound of amine 0.5 acetate per ton of concentrate and 0.785 pound of kerosene oil per ton of concentrate were required. At the end of the 10-day operating period the composition of the invention is substituted for the amine 0.5 acetate. No heating is necessary and the 55 gallon drums of composition are simply dispersed in water at ambient temperature and pumped to the flotation cells. During the test period with the amine composition of the invention 0.336 pound of composition per ton of concentrate and 0.470 pound of kerosene oil per ton of concentrate gave optimum efficiency of operation.

In both tests the feed (i.e., rougher concentrate) is deoiled and deslimed in conventional manner before subjecting it to amine treatment. The data obtained from these tests are reported in Table III below.

It can be readily seen that the composition of this gave improved phosphate recovery results while using substantially less kerosene.

EXAMPLE 6

Fluidity and emulsion stability determinations were made on a variety of amine 0.5 acetate, methylisobutyl carbinol and kerosene combinations under varying temperature conditions. Fluidity was determined at 85° F., 75° F., in an ice water bath, and at room temperature for 1 week. All compositions contained from 65 to 80 percent of amine 0.5 acetate as prepared in Example 1. The amount of the methylisobutyl carbinol and kerosene used in the preparation of the several compositions tested, was varied from 0 to 35 percent for each material. From the data obtained and provided in Table IV below, it can be seen that the compositions of the invention remained fluid at low temperature and readily pumpable after storage for 1 week. After storage at room temperature for 6 months, the compositions of the invention appear to be substantially unchanged from the 1 week reading. Compositions containing less than about 20 percent by weight of methylisobutyl carbinol became plastic or solid at low temperatures and separated to a significant degree on standing.

TABLE III

Amine 0.5 Acetate

	Feed, percent BPL	Tails, percent BPL	Concentrate: Percent BPL	Concentrate: Insol.	Percent recovery of total HPL	Amine usage, lbs./ton concentrate	Kerosene usage, lbs./ton concentrate
Days:							
1	32.06	11.09	78.00	2.99			
2	32.49	9.65	77.38	2.70			
3	30.27	9.06	77.40	2.56			
4	26.17	10.27	75.64	3.00			
5	31.36	9.51	77.97	3.30			
6	27.73	9.00	76.30	3.08			
7	35.59	9.67	78.95	2.47			
8	35.50	9.68	78.34	2.72			
9	33.87	8.64	78.58	2.60			
10	32.99	8.28	78.63	2.62			
Avg.	31.88	9.49	77.86	2.76	79.90	0.340	0.785
Amine 0.5 Acetate 70%—Methylisobutyl Carbinol 30% Composition							
11	32.47	8.08	78.50	2.45			
12	34.52	9.54	79.04	2.65			
13	36.37	6.71	78.16	2.83			
14	35.23	9.16	79.19	2.50			
15	31.50	9.78	78.60	2.64			
16	32.00	7.98	78.22	2.69			
17	34.43	10.03	78.21	2.47			
18	38.20	6.00	78.10	2.50			
19	35.38	8.80	77.54	2.86			
Avg.	34.26	8.67	78.37	2.64	83.99	0.336	0.470

TABLE IV

Test Number	1	2	3	4
Percent amine 0.5 acetate	65	70	75	80.
Percent MICB	35	30	25	20.
Mixture consistency:				
After heating at—				
85° F	Excellent, clear	Excellent, 1% solids separates.	Excellent, 3% solids separates.	Excellent, 1% solids separates.
75° F	Excellent	Excellent, 1% solids separates.	Excellent, 3% solids separates.	Excellent, 1% solids separtes.
In ice water	Definitely pumpable	Pumpable	Pumpable	Pumpable.
Test Number	**1-A**	**2-A**	**3-A**	**4-A**
Percent amine 0.5 acetate	65	70	75	80.
Percent MIBC	17.5	15.0	12.5	10.0.
Percent kerosene	17.5	15.0	12.5	10.0.
Mixture consistency:				
After heating at—				
85° F	Excellent, separated slightly.	Excellent, separated 2%	Excellent, separated 5–10%	Excellent, separated 25%.
75° F	Excellent, separated slightly.	Excellent, separated 2%	Excellent, separated 5–10%	Excellent, separated 25%.
In ice water	Plastic flow	Solid	Solid	Plastic.
After 1 wk. room temp	20% solid	Pumpable 80% solid	Pumpable 80% solid	Pumpable 50% solids.

Table IV—Continued

Test number	1-B	2-B	3-B	4-B
Percent amine 0.5 acetate	65	70	75	80.
Percent MIBC	11.7	10	8.3	6.7.
Percent kerosene	23.3	20	16.7	13.3.
Mixture consistency:				
After heating at—				
85° F	Excellent, separates 30% solids.	Excellent separates 5% solids.	Excellent, separates 30% solids.	Excellent, separates 60% solids.
75° F	Excellent, separates 30% solids.	Excellent, separates 5% solids.	Excellent, separates 30% solids.	Excellent, separates 60% solids.
In ice water	Separated	Slightly plastic flow	Plastic flow	Plastic flow.
After 1 wk. room temp	20% solid	70% solid	60% solid	Uniform plastic flow.

Test number	1-C	2-C	3-C	4-C
Percent amine 0.5 acetate	65	70	75	80.
Percent kerosene	35	30	25	20.
Mixture consistency:				
After heating at—				
85° F	Excellent, separates 10%	Excellent, separates 5%	Excellent, separates 20%	Excellent, separates 80%.
75° F	Excellent, separates 10%	Much worse	Worse	Almost solid.
In ice water		Solid	Solid	Solid.
After 1 wk. room temp	Plastic flow uniform	Plastic flow uniform	Plastic flow uniform	Plastic flow uniform.

EXAMPLE 7

To further evaluate the compositions of the invention, a 30 percent methylisobutyl carbinol, 70 percent amine 0.5 acetate composition, prepared in accordance with the invention and Example 1 above, was tested under normal operating conditions in the amine flotation circuit of a Florida phosphate mining operation and compared with the amine 0.5 acetate normally employed in said circuit. During the 17-day test period with the conventional amine (14 days prior to use of the inventive composition and 3 days after) from 0.53 to 0.62 pounds of amine per ton of concentrate was required for optimum efficiency of operation. During the 14-day test period with the composition of the invention an average of 0.48 pounds of composition per ton of concentrate produced, gave optimum efficiency. These data show a 10 to 20 percent reduction in reagent costs per ton of concentrate produced. The data recorded are provided in Table V below.

TABLE V

Days	Reagent	Avg. lbs. reagent/ ton concentrate	Percent BPL feed	Concentrate, percent BPL	Concentrate, percent Insol.	Tails, percent BPL	Percent recovery total BPL
14	Amine 0.5 acetate	0.53	30.00	79.87	3.01	3.67	91.70
14	Amine-MIBC composition	9.48	25.40	78.55	2.82	3.63	90.10
3	Amine 0.5 acetate	0.62	21.35	78.50	2.80	3.66	87.20

In similar tests using the conventional amine and the inventive compositions described above, but a different flotation circuit and a phosphate feed analyzing approximately 15 percent BPL 8.8 pounds of the conventional amine yielded 13.44 tons of concentrate analyzing 73.0 percent BPL, 3.81 percent insol. and gave a total percent BPL recovery of 67.9 percent. Using the 30 percent methylisobutyl carbinol 70 percent amine 0.5 acetate composition prepared in accordance with the process of the invention, it was found that 8.8 pounds of said composition yielded 16.75 tons of concentrate analyzing 72.6 percent BPL, 3.50 percent insol. and gave a total BPL recovery of 71.4 percent. Data obtained are reported in Table VI below.

TABLE VI

Reagent in lbs.	Percent BPL feed	Concentrate Tons	Concentrate Percent BPL	Concentrate Percent insol.	Total, percent BPL recovery
8.8 Amine	14.35	13.44	73.0	3.81	67.9
8.8 Amine-MIBC	15.55	16.75	72.6	3.50	71.4

From these data it is evident that the composition of the invention increased concentrate recovery more than 24 percent over that obtained with an equivalent amount of the conventional amine tested.

While the foregoing invention has been described and exemplified in terms of its preferred embodiments, those skilled in the art will readily appreciate that variations can be made without departing from the sphere and scope of the invention.

We claim:

1. A liquid cationic ore separation flotation composition comprising from about 20 to 40 percent by weight of an alcohol having a boiling point between about 131° and 161° C. selected from the group consisting of methylisobutyl carbinol, heptanol, and mixtures thereof, and about 60 to 80 percent by weight of the product obtained by reacting approximately one equivalent of a tall oil fatty acid with from about 1.5 to 2.5 equivalents of diethylene triamine at a temperature between about 100° and 300° C., and thereafter treating the thus formed product with from about 0.2 to 1.0 equivalents of glacial acetic acid to at least partially neutralize the product.

2. A process for the preparation of a liquid cationic flotation percent comprising: reacting, at a temperature of between about 100° and about 300° C., a tall oil fatty acid having an acid value of 195–199, a saponification value of 197–200, an iodine value of 129–131 and containing 0.3 to 4.2 percent rosin acid and 0.4 to 1.6 percent unsaponifiables, with diethylene triamine, cooling the thus formed product and treating said product with glacial acetic acid, the ratio of fatty acid to diethylene triamine to acetic acid being about 1:1.5–2.5:0.2c–1.0, respectively; cooling the product from said reaction and treating said product with from about 20 to 40 by weight of an alcohol having a boiling point between about 131° and 161° C. selected from the group consisting of methylisobutyl carbinol, heptanol, and mixtures thereof.

3. A liquid cationic ore separation flotation composition comprising from about 20 to 40 percent by weight of an aliphatic alcohol having a boiling point between about 131° and 161° C. selected from the group consisting of methylisobutyl carbinol, heptanol, and mixtures thereof, and about 60 to 80 percent by weight of the product obtained by reacting approximately one equivalent of a tall oil fatty acid having an acid value of 195–199, a saponification value of 197–200, an iodine value of 129–131 and containing from about 0.3 to 4.2 percent rosin acid and 0.4 to 1.6 percent unsaponifiables with from about 1.5 to 2.5 equivalents of diethylene triamine at a temperature between about 100° and 300° C. and thereafter treating the product with from about 0.2 to 1.0 equivalents of glacial acetic acid to at least partially neutralize the product.

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