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FLOTATION

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This invention relates to an improved process for the concentration of ores, more particularly it relates to a method for improving the efficiency of froth flotation processes employing cationic surface active compounds as flotation agents. Still more particularly it relates to the use of assisting agents in froth flotation processes employing cationic surface active compounds as flotation agents; and still more particularly it relates to the use of assisting agents comprising water-soluble protective colloids of the carbohydrate type in froth flotation processes for the concentration of negatively-charged minerals employing cationic surface active compounds as flotation agents.

This invention has as an object the development of an improved method for separating or concentrating negatively charged minerals especially quartz and silicate minerals. A further object is to increase the selectivity of cationic surface active compounds as flotation agents in such processes. Another object is to provide a water-soluble protective colloid of the carbohydrate type as an assisting agent which in combination with a cationic surface active agent produces a synergistic effect thereby imparting to the combination a collecting power which is greater than that of the sum of the elements of said combination. A still further object is the production of an improved process of concentrating metal carbonate and oxide minerals, using cationic surface active agents as flotation agents and water-soluble protective colloids of the carbohydrate type as assisting agents. Other objects will appear hereinafter.

The above and other objects appearing hereinafter are accomplished by the following invention which comprises adding to a pulp containing metal carbonate and/or oxide minerals and negatively charged gangue material a cationic surface active compound as a flotation agent and a water-soluble protective colloid of the carbohydrate type as an assisting agent and subjecting the same to a froth flotation operation. The valuable fraction of the ore may be found in the froth layer or it may remain in the cell. In this way a sharper separation of valuable mineral from gangue is effected, resulting in a purer product and greater recovery.

The ability of surface-active cationic reagents to float siliceous minerals by means of a froth in preference to the indifferent metallic oxides and carbonates has been utilized by this invention, but the performance of the cationic surface active compound as flotation agents has been improved, particularly in the separation of siliceous material from calcium carbonate, or limestone, by the use of protective colloids of the carbohydrate type as assistants. These assistants have been found to promote separation of more of the siliceous material for a given amount of flotation agent resulting in the separation of a higher grade of limestone with the same or higher recovery remaining behind as tailings. For example, when tannic acid is added as an assistant (Example II) to a flotation operation using crude dodecylamine hydrochloride, the grade of $CaCO_3$ from the flotation operation is improved from 79.02 to 87.81% with a recovery increase from 78.9 to 82%. Another way of expressing the same effect is to state that in the majority of cases for the production of a desired grade of mineral concentrate a substantial reduction in the required amount of collector is made possible by the addition of an assistant. In certain instances an improvement in the collecting power of cationic surface active compounds as flotation agents is effected by a preliminary acid bleach which serves to remove mineral stains from quartz particles and gives clean, readily floated quartz. Thus, in the case of the removal of silica from iron oxide minerals an improved performance in the collecting properties of cationic surface active compounds as flotation agents results from leaching the ore with a small amount of a mineral acid such as hydrochloric acid prior to the flotation step.

This invention will be further illustrated but is not intended to be limited by the following examples:

Example I

A sample of siliceous limestone used in the production of Portland cement was employed in flotation experiments using assistants with cationic surface active compounds as flotation reagents. The mineral composition of this limestone has been described in detail by B. L. Miller and C. H. Breerwood (Am. Inst. Mining Met. Engrs., Tech. Pub. No. 606, p. 15, February, 1935). Calcite, mica and quartz constitute the bulk of

the rock. Pyrite, limonite, apatite, and amorphous carbon are present in small quantities.

The limestone was crushed in a small jaw crusher and then reduced to minus 40 mesh by means of a Braun disc pulverizer. Slurries for use in flotation experiments were prepared by grinding 1 kg. of the minus 40 mesh ore for 70 minutes in a one-gallon porcelain jar mill with one liter of water and 3 kg. of flint pebbles. The ore particles were then about 84–86% minus 200 mesh.

The slurry prepared according to the above procedure was placed in a 1 kg. Denver Equipment Company flotation cell of the sub-aeration type. Sufficient water was then added to make a pulp containing approximately 20–25% solids. The agitator was then started and 14 cc. of an 0.18% water solution of "crude dodecyl" amine hydrochloride was added. Seven hundredths of a gram of the frother hereafter referred to as frother *b*, was added to induce frothing. The frothing agent consisted of the branched-chain, oxygenated organic compounds prepared by the catalytic hydrogenation of carbon oxides under elevated temperatures and pressures as described in U. S. Patents 1,844,129, 1,844,857, and 1,939,708. The boiling range of the particular fraction of these oxygenated organic compounds used was 147–157° C.

With the addition of frother *b* a heavily loaded froth, dark gray in color, formed rapidly and was collected for 9 minutes, after which time there was no further collection of minerals or production of froth. The flotation concentrate and tailings were filtered, dried, weighed, and analyzed for their calcium carbonate content. The results are summarized in the following table:

Product	Weight g.	$CaCO_3$		
		Per cent	Weight g.	Per cent recovery
Heads	1,000.7	[1] 75.5	755.6	
Overflow	245.7	64.70	159.0	21.1
Underflow	755.0	79.02	596.6	78.9

[1] Calcd.

Reagents.—Crude dodecylamine hydrochloride, 0.025 g. or 0.05 lb./ton; frother *b*, 0.07 g. or 0.14 lb./ton.

It will be noted from the table that 78.9% of the calcium carbonate remained in the cell in the "underflow," commonly called the "tailings" in ore dressing terminology. Since the desired mineral concentrated in the underflow I prefer not to use the term "tailings" as that term generally implies a waste product or gangue. Similarly I use the term "overflow" to designate the product carried out of the cell by the froth. Generally the product frothed out of the cell is called the "concentrate" but I prefer to avoid that term, since in its usual use it connotes a purification or segregation of valuable minerals and in my process the froth carries out the undesired minerals. The terms "underflow" and "overflow" are therefore used to designate my flotation products rather than the commonly used terms "tailings" and "concentrate." In the above experiment the grade of limestone was increased from 75.5% to 79.02% of calcium carbonate with a recovery of 78.9%.

By crude dodecylamine hydrochloride as used herein is meant the mixture of hydrochlorides of the higher primary aliphatic amines in which the alkyl groups correspond in carbon content and composition to the fatty acids occurring naturally in coconut oil.

Example II

The process of Example I was repeated using, in addition to the same quantities of crude dodecylamine hydrochloride and frother *b*, 0.5 gram of tannic acid. The flotation fractions were filtered, dried, weighed and analyzed as before. The results of this experiment, using tannic acid as an assistant, are summarized in the following table:

Product	Weight g.	$CaCO_3$		
		Per cent	Weight g.	Per cent recovery
Heads	990.9	[1] 76.6	759.8	
Overflow	281.7	48.66	137.2	18.0
Underflow	709.2	87.81	622.6	82.0

[1] Calcd.

Reagents.—"Crude dodecyl" amine hydrochloride, 0.025 g. or 0.05 lb./ton; frother *b*, 0.07 g. or 0.14 lb./ton; tannic acid, 0.50 g. or 1.0 lb./ton.

Comparison of the above results with those of the control run (Example I) shows that a marked improvement has been made by the addition of tannic acid as indicated by the substantial increase in the grade of the limestone, represented by the underflow, from 79.02% to 87.81% of calcium carbonate in addition to an increase in recovery from 78.9% to 82.0%.

Example III

The process of Example I was repeated using, in addition to the same quantities of crude dodecylamine hydrochloride and frother *b*, 0.50 g. of pectic acid corresponding to 1.0 lb./ton of heads. The flotation fractions were filtered, dried, weighed and analyzed as before. The results of this experiment are summarized in the following table:

Product	Weight g.	$CaCO_3$		
		Per cent	Weight g.	Per cent recovery
Heads	997.7	[1] 75.7	755.4	
Overflow	272.0	43.07	117.2	15.5
Underflow	725.7	88.57	638.2	84.5

[1] Calcd.

Reagents.—Crude dodecylamine hydrochloride, 0.05 lb./ton frother *b*, 0.14 lb./ton; pectic acid, 1.0 lb./ton.

The addition of pectic acid increased the grade of underflow from 79.02%, obtained by the control run, to 88.57% calcium carbonate with an increase in recovery from 78.9% to 84.5%.

Example IV

A number of assisting reagents in addition to those described in Examples II and III were tested under the same conditions outlined in the control run (Example I) using 0.05 lb./ton crude dodecylamine hydrochloride and 0.14 lb./ton frother *b*. For sake of brevity, typical results are listed in the accompanying table:

Assistant	Amt. used, lb./ton	Per cent $CaCO_3$		$CaCO_3$ per cent recovery
		Heads	Underflow	
None (control)		75.5	79.02	78.9
Sodium cellulose glycollate	0.20	73.0	86.93	88.8
	0.50	69.8	87.90	86.3
Mesquite gum	0.50	70.3	86.50	86.5
Cherry gum				

The above variations in the analyses of the heads are due to the different batches of limestone used for the tests.

Example V—(Control)

The process of Example I was repeated using the same reagents but a limestone of higher calcium carbonate content (80.81%) from another source. The experiment described herein is also intended to illustrate the results obtained in the absence of an assisting agent and for comparison with subsequent examples where an assisting agent is used. The collector crude dodecylamine hydrochloride, was increased to 0.10 lb./ton, and the frother *b* was used in a slightly smaller amount. For purpose of comparison, no assistant was used in this control run. The flotation fractions were filtered, dried, weighed and analyzed as usual. The results of this experiment are summarized in the following table:

Product	Weight g.	$CaCO_3$		
		Per cent	Weight g.	Per cent recovery
Heads	997.6	[1] 80.3	800.3	6
Overflow	206.6	56.05	115.8	14.7
Underflow	791.0	86.50	684.5	85.3

[1] Calcd.

Reagents.—Crude dodecylamine hydrochloride, 0.10 lb./ton; frother *b*, 0.11 lb./ton.

The above results indicate that the grade of the limestone (heads) was increased from 80.3% to 86.50% calcium carbonate with a recovery of 85.3%.

Example VI

The process of Example V was repeated with the various assisting agents enumerated in the table below, but using less than half the quantity of collector with the following results:

Assistant	Amt. used, lb./ton	Per cent $CaCO_3$		$CaCO_3$ per cent recovery
		Heads	Underflow	
Control (Example VI)		80.3	86.50	85.3
Gum tragacanth	0.50	81.0	97.28	81.8
Starch	0.50	81.8	91.50	84.8
Gum arabic	0.50	80.2	96.30	83.0
Citrus pectin	0.20	80.4	93.70	82.4
Irish moss	0.20	80.6	90.10	87.1
Ammonium alginate	0.20	80.1	93.20	88.3
Palmetto gum	0.20	78.1	94.97	88.0

Example VII

The process of Example I was repeated using a collecting agent of the cationic surface active type prepared from the chloracetate of the mixture of branched chain alcohols boiling at 160° to 195° C. at atmospheric pressure, produced by catalytic hydrogenation of carbon oxides, in combination with pyridine. Four cubic centimeters of a 5% solution of the above pyridine compound was added to the slurry described in Example I. A heavily loaded, gray froth was produced and collected for a period of 9 minutes. The fractions were filtered, dried, weighed and analyzed. The results are summarized in the following table:

Product	Weight g.	$CaCO_3$		
		Per cent	Weight g.	Per cent recovery
Heads	993	[1] 75.4	748.7	
Overflow	177	62.09	109.9	14.6
Underflow	816	78.28	638.8	85.4

[1] Calcd.

Reagents.—Collector, 0.40 lb./ton.

This experiment represents a control run which is to be compared with following Example VIII, in which an assistant is used.

Example VIII

The process of Example VII was repeated with the addition of 0.25 g. or 0.50 lb./ton of gum tragacanth as an assistant. The flotation fractions were filtered, dried, weighed and analyzed as before. The results are summarized in the following table:

Product	Weight g.	$CaCO_3$		
		Percent	Weight g.	Percent recovery
Heads	1,000	78.2	782.4	
Overflow	161	44.00	70.8	9.0
Underflow	839	84.82	711.6	91.0

Reagents.—Collector, 0.40 lb./ton; gun tragacanth, 0.50 lb./ton.

Example IX

A control run similar to that described in Example I was made using 0.20 g. of 2-ethylhexylamine hydrochloride as the collector and 0.05 g. of frother *b*. The resulting well-loaded, gray froth was collected for 9 minutes. The flotation fractions were filtered, dried, weighed and analyzed for calcium carbonate. The results are given in the accompanying table:

Product	Weight g.	$CaCO_3$		
		Percent	Weight g.	Percent recovery
Heads	986	[1] 75.4	743.3	
Overflow	342	63.56	217.3	29.2
Underflow	644	81.68	526.0	70.8

[1] Calcd.

Reagents.—2-ethylhexylamine hydrochloride, 0.4 lb./ton; frother *b*, 0.10 lb./ton.

Example X

The process of Example IX was repeated with the same reagents but with 0.25 g. of tannic acid as an assistant. The flotation fractions were filtered, dried, weighed and analyzed as before. The results are summarized in the following table:

Product	Weight	$CaCO_3$		
		Percent	Weight g.	Percent recovery
Heads	986	[1] 74.1	731	
Overflow	452	51.50	233	31.9
Underflow	534	93.33	498	68.1

[1] Calcd.

Reagents.—2-ethylhexylamine hydrochloride, 0.40 lb./ton; frother *b*, 0.10 lb./ton; tannic acid, 0.50 lb./ton.

It is apparent that the addition of tannic acid increases the amount of overflow as compared with the control run in Example IX. The grade of the underflow was increased from 81.68% to 93.33% calcium carbonate with a slight decrease in recovery which could be increased by a cleaner operation at no appreciable decrease in grade.

Example XI—(Control)

The following example, in which no assistant was used, is presented with a view to demonstrating the effectiveness of an assisting agent which was used in the subsequent Example XII.

A slurry of 100 g. of Minnesota iron ore washer tailings was treated with 0.015 g. crude dodecylamine hydrochloride and 0.009 g. frother *b*. The resulting froth was collected for 6 minutes, broken down with a mixer and then recleaned with 0.003 g. crude dodecylamine hydrochloride and 0.009 g. frother *b*. The fractions were filtered, dried, weighed, and analyzed for the hydrochloric acid soluble content, which is a measure of iron content. The results are tabulated below:

Product	Hydrochloric acid-soluble content		
	Weight, g.	Percent	Distribution
Heads	100.0	31.0	100.0
Rougher overflow	35.0	10.6	11.9
Cleaner overflow	30.0	7.4	7.1
Rougher underflow	65.0	42.0	88.1
Cleaner underflow	5.0	29.0	4.8
Combined underflow	70.0	41.2	92.9

Reagents:
Rougher operation—
0.30 lb./ton dodecylamine hydrochloride
0.18 lb./ton frother *b*
Cleaner operation—
0.06 lb./ton dodecylamine hydrochloride
0.18 lb./ton frother *b*

Example XII

To demonstrate the improved grade of underflow obtained with an assistant, the process of Example XI was repeated using, in addition to the exact amounts of dodecylamine hydrochloride and frother *b*, 0.025 g. gum tragacanth. The fractions were filtered, dried, weighed, and analyzed. The results are summarized in the following table:

Product	Weight g.	Per cent	Distribution
Heads	100.0	31.5	100.0
Rougher overflow	48.5	10.3	15.8
Cleaner overflow	42.1	7.4	9.8
Rougher underflow	51.5	51.5	84.2
Cleaner underflow	6.4	29.9	6.0
Combined underflow	57.9	49.1	90.2

Reagents:
Rougher operation—
0.30 lb./ton dodecylamine hydrochloride
0.18 lb./ton frother *b*
Cleaner operation—
0.06 lb./ton dodecylamine hydrochloride
0.18 lb./ton frother *b*

The addition of gum tragacanth improved the flotation operation by giving rougher and combined underflows of higher soluble content as compared with those in Example XI. In other words, the quality of iron oxide in the combined underflow has been increased from that represented by 41.2% soluble content in the control (Example XI to 49.1% soluble content). It goes without saying that the lower the hydrochloric acid-soluble content, the higher the insoluble silica content. It is apparent from Example XI and XII that the silica rejection is increased when an assistant is used with a cationic collector.

In place of the water-soluble protective colloids listed in the above examples, others may be substituted with equally good results. Additional suitable agents include water soluble ammonium and amine salts of cellulose glycollic acid, glucamine, dextrine, methylstarch, agar agar, locust bean gum, water-soluble ethyl cellulose, hydroxyethyl cellulose, water-soluble methyl cellulose, water-soluble salts of pectic, alginic and tannic acid. Suitable salts include alkali metal, ammonium and amine salts, e. g. mono, di and trimethyl, ethyl, propyl, isopropyl, butyl, etc. amine salts.

In addition to the specific cationic surface active agents disclosed in the examples, we have found that a very large number of other agents of this type will give good results. Either single compounds or mixtures are useful. Among the agents which can be used to advantage in the separation of metal carbonates and oxides from siliceous gangues, the following may be mentioned

Hexadecyl trimethyl ammonium bromide or chloride
Dodecyl trimethyl ammonium bromide or chloride
Octadecyl trimethyl ammonium bromide or chloride
Hexadecyl pyridinium bromide or chloride
Dodecyl pyridinium bromide or chloride
Dodecylamine hydrochloride, hydrobromide, nitrate, acetates or chloroacetate
Hexadecylamine hydrochloride
Octadecylamine hydrochloride
Dodecyl dimethyl sulfonium methyl sulfate
Dodecyl dimethyl sulfonium bromide
Octadecyl methyl ethyl sulphonium iodide
Tetradecyl dimethyl sulphonium bromide
Dodecyl methyl cyclohexyl sulphonium iodide
Dodecyl benzyl methyl sulphonium chloride

A large list of useful ammonium, sulphonium and phosphonium compounds which have utility in the herein disclosed processes are set forth in Dunbar U. S. Patent 2,165,356.

The agents of our invention may be designated by the following general formula:

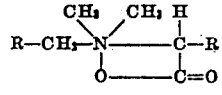
$$R—M(R')_x—Y$$

in which R is a non-aromatic (i. e. non-benzenoid) hydrocarbon radical of 8 to 22 carbon atoms; M represents pentavalent nitrogen, pentavalent sulfur, or tetravalent sulfur; R' represents hydrogen atoms or hydrocarbon radicals having not more than 5 carbon atoms which may be constituents of an alicyclic or of a heterocyclic ring; *x* is an integer less by two than the valence of element M, and Y is a negatively charged salt forming atom or group, for example, chlorine, bromine, nitrate, acetate, chloroacetate, etc. The long chain aliphatic radical R need not be entirely composed of carbon atoms in the chain but may contain ether linkages, ester groups, sulfur atoms, carbonyl groups, etc. provided these groups do not introduce new centers of polarity. For example, reagents such as the hydrochloride of the dodecyl ester of alpha-amino-isobutyric acid, the hydrochloride of beta-aminoethyl dodecyl ether, and the hydrochloride of the dodecyl ester of alpha-amino-isobutyric acid are good flotation reagents for use in the process of this invention; such substances, however, as the hydrochlorides of a long chain amine having a carboxyl group on the end removed from the amino group would not produce the desired effect, since another polar, solubilizing group has been introduced.

Two other types of cationic surface-active reagents which bear some mention are found useful in the practice of this invention. One of these types comprises salts of amino naphthenes derived from naphthenic acids of 12 to 20 carbon atoms and natural acidic resins, e. g. rosin, Congo, kauri, Manila, sandarac, damar, Pontianac, Zanzibar, etc. The aminonaphthenes can

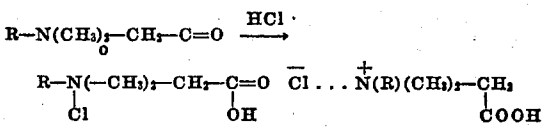
be made by catalytically dehydrating the acids in the presence of ammonia and subsequent reduction of the nitriles to the corresponding amines and the salts can be made by treating the amines with an acid, e. g. hydrochloric. The reactions involved are discussed below in connection with higher fatty acids.

The second class which appears to differ from the type formula are the betaines having long aliphatic chains. Some illustrative betaine type compounds apparently have the following formula, although there is some evidence in favor of their being non-cyclic compounds:



wherein one of the R's is a long chain aliphatic hydrocarbon radical and the other R hydrogen. The preparation of these compounds and specific examples of related compounds useful in this invention is given in Downing and Johnson U. S. Patent 2,129,264. Examples of such betaines are N-dodecyl betaine, N-cetyl betaine, N-decyl betaine, C-dodecyl betaine, C-octadecyl betaine, etc.

The betaines are particularly effective if used in the presence of small amounts of mineral or organic acids and this observation may furnish a clue to their manner of action. It is possible that the betaine ring tends to open by addition of acid and the resulting product tends to ionize yielding a surface active cation.



A class of substances which we have found to be particularly effective is composed of mixtures of salts of amines derived from the fatty acids obtained by hydrolytic splitting of natural fatty glycerides, e. g. palm oil, coconut oil, stearin, etc. For example, the mixture of amines derived from coconut oil fatty acids by the following series of reactions such as follows gives excellent flotation results after conversion to a water-soluble salt such as the hydrochloride or acetate.

$$R{-}COOH \rightarrow RCOONH_4 \rightarrow R{-}CONH_2 \rightarrow R{-}CN \rightarrow R{-}CH_2NH_2$$

The amount of cationic surface active agents required to produce the above effects varies somewhat with the reagent chosen and the particular ore being treated. In general, the amount needed will be between 0.05 and 2.0 pounds per ton of ore, but in some cases somewhat smaller or larger quantities may be required. The amount of protective colloid of the carbohydrate type used may also vary widely. In general from 0.01 to 5.0 pounds of assistant per ton of ore is satisfactory, but for most cases from 0.2 to 1.0 pound per ton is sufficient. Some of the reagents produce sufficient frothing so that an additional frothing agent is not required, while others have little or no frothing power. When greater frothing power is needed, we prefer to use a material such as pine oil, cresylic acid or frother *b*, since these frothers have no adverse effect on the selectivity of the collecting agents. Occasionally we find it advantageous to use mixtures of two or more of the cation-active reagents to produce flotation products of high quality. In carrying out a flotation process according to the teachings of this invention, any of the well known types of flotation cells may be employed. The ratio of pulp solids to water may vary from about 1:1 to 1:6. Ordinarily a neutral pulp is used, but in some cases it may be desirable to use an alkaline or acid pulp.

It will be clear from the above that one of the uses of this invention is the preparation of calcium carbonate of good quality from natural rocks whose calcium carbonate content is too low to be of technical value. By means of this process limestone rocks containing 40–70% calcium carbonate can be purified to such an extent that the product can be used in the manufacture of Portland cement. It is also possible to prepare calcium carbonate concentrates approaching theoretical purity from low-grade limestone by this process. The process of this invention also permits the recovery of iron carbonate, manganese carbonate, magnesium carbonate, and other metallic carbonates from ores containing these minerals with other siliceous materials as impurities. Conversely it may be used to prepare talc, mica, or quartz products of high purity and value when the principal impurities are alkaline earth or heavy metal carbonates or oxides. While the herein disclosed examples apply to the use of assistants in the beneficiation of calcium carbonate, the same principles apply to the concentration of oxidized iron minerals in which the cationic surface active collectors are benefited through the use of the water-soluble protective colloid assistants of the carbohydrate type.

The present process affords two outstanding advantages as compared with previously known methods for the removal of siliceous minerals by means of cationic surface active agents as collectors. First, it provides a process by which quartz and siliceous minerals can be separated from other minerals at a substantial reduction in the amount of collector through the use of assistants. Secondly, with a given amount of cationic surface-active agents, the use of an assistant increases the selectivity as a result of which a higher grade of mineral concentrate is obtained. In general, processes for the flotation of ores containing negatively charged minerals by means of cationic surface agents as collectors are improved by any one of the protective colloid assisting agents described herein.

This application is a continuation-in-part of co-pending application Serial No. 169,072, filed October 14, 1937.

While the preferred embodiments of my invention have been disclosed, it will be readily apparent to those skilled in the art that many variations and modifications may be made therein without departing from the spirit of the invention.

I claim:

1. A froth flotation process for concentrating ores containing negatively charged gangue materials taken from the group consisting of metal carbonate and oxide ores which comprises adding to an aqueous pulp of such ores a cationic surface active agent and a small amount of a water soluble substance taken from the group consisting of tannic, pectic, and alginic acids and their water soluble salts.

2. A froth flotation process for concentrating ores containing negatively charged gangue materials taken from the group consisting of metal carbonate and oxide ores which comprises adding to an aqueous pulp of such ores a small amount of an alkyl amine salt wherein said alkyl radical contains from 8–22 carbon atoms and

a small amount of a water soluble substance taken from the group consisting of tannic, pectic, and alginic acids and their water soluble salts.

3. A froth flotation process for concentrating ores containing negatively charged gangue materials taken from the group consisting of metal carbonate and oxide ores which comprises adding to an aqueous pulp of such ores a small amount of dodecylamine hydrochloride and a small amount of a water soluble substance taken from the group consisting of tannic, pectic, and alginic acids and their water soluble salts.

JAMES EMORY KIRBY.