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FLOTATION PROCESS

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This invention relates to the treatment of ores, metallurgical products, and like heterogeneous materials containing compounds of the useful or more or less valuable metals and has for its object to provide improvements in the separation of the compounds in question by flotation, and particularly by froth flotation from the components of the materials treated. This application is in part a continuation of my U. S. application Serial No. 676,681, filed on June 20, 1933.

In accordance with the present invention it has been discovered that in the flotation treatment of ores to separate the desirable constituents therefrom, liquid media containing the salts of sulfuric acid esters and/or the unsaponified esters, of the mixture of oxygenated organic compounds obtained from the catalytic hydrogenation of carbon oxides under elevated temperatures and pressures are effective in separating the gangue from the ore according to the well known principles of froth flotation. In this process the action of the mixture of sulfates and/or their salts may be either to concentrate the mineral in the froth or foam and to separate the mineral from the gangue, or the action may be to concentrate the desired mineral in the form of a dispersed gangue from which the other matter is separated. In general, the action of the sulfates and their salts of the mixture of compounds will be a soap-like action, and will involve the preferential wetting of different ores.

An object of the present invention is to provide a new and improved frothing agent. Another object of the present invention is to provide a frothing agent which will develop a brittle and impermanent froth of sufficient buoyancy to float the mineral particles and yet of such an impermanent nature that upon removal from the activating apparatus it rapidly and completely collapses, thus affording easy separation of the mineral particles therefrom. A still further object of the invention comprises a process for the froth flotation of minerals involving the use of salts of strong sulfonic acid derivatives and/or the sulfates of the higher boiling mixture of oxygenated organic compounds obtained from the catalytic hydrogenation of carbon oxides under elevated temperatures and pressures. Other objects and advantages will hereinafter appear.

The sulfonic acids, sulfates, and/or ester salts thereof used in accord with the present invention contain mixtures of organic radicles which are predominantly alkyl radicles having at least 3 carbon atoms, although these radicles generally contain 5 or more carbon atoms, which may be either of a saturated or unsaturated nature. The specific sulfate ester salts comprise the alkali metal salts, the ammonium, the sodium and potassium salts, alkaline earth metal salts, calcium and magnesium salts, and the other heavy metal salts, lead and aluminum salts of the sulfuric esters. The salts and esters herein designated are given only by way of example, and it will be understood that other salts and other esters within the range indicated and obtained by the process for the formation of the ester salts, which will be hereinafter more fully particularized and which have the same general properties, may be used in lieu thereof.

My preferred frothing agents are derived from the following source. In the catalytic hydrogenation of carbon oxides under elevated temperatures and pressures a variety of straight and branch chained organic compounds containing oxygen are made. These organic compounds have a boiling point ranging from about 50° C. to a temperature of 250° C. and higher. Various methods have been proposed for manufacturing these compounds, as, for example, the method described in the U. S. application of Roger Williams, now U. S. Patent 1,820,417. The fractions of the mixture of oxygenated organic compounds obtained by Williams and equivalent processes, from which fractions I generally prefer to prepare my flotation agents, are those boiling from a temperature of approximately 100° C. up.

The oxygenated organic compounds boiling from 100° C. up have been fractionated systematically, however, into a series of fractions, namely 100—130; 130—147; 147—157; 157—195; 195—240; and 240° up. My flotation agents are made from these fractions, each of which contain a plurality of organic compounds. The sulfates may be obtained, for example, by reacting any of these fractions or mixtures thereof with a strong sulfating agent, such as sulfuric acid of 96%–100% of strength, fuming sulfuric acid, chlorsulfonic acid, sulfur trioxide and the like; the sulfonic acid derivatives are obtained by treatment with 75%–90% sulfuric acid. The temperature during the reactions may vary widely, e. g. between 0° and 100° C., but a temperature of from 0° to 40° C. is preferred with the strong acid if it is desired to favor the formation of a maximum amount of true sulfate. Methods for forming these sulfated products have been fully described in the prior art, viz. Br. 307,709 and 317,039; Fr. 671,605 and others. The ester resulting from the sulfating reaction may, if desired, be reacted with a suitable compound of a

metal, for example, sodium hydroxide, lead oxide, calcium hydroxide, and the like, give a salt corresponding to the ester.

While it is an object of the present invention to treat ores generally in the presence of my flotation agents, the process is applicable with particular efficiency to the treatment of non-sulfide bearing ores, for example, phosphate rock, limestone, coal, phosphite, pyrites, zircon, and ilmenite.

The quantity of the sulfonic acid derivatives, sulfates, or sulfate ester salts which may be used in the flotation for the treatment of a given gangue may vary within wide limits. Ordinarily, the amount used will be in the order of 100 parts of ester salts or more to one million parts of water.

I shall now illustrate one method of utilizing my class of flotation agents, but I am not to be limited by its details:

One thousand grams of bituminous slack which had been dry ground to pass a 40-mesh sieve was mixed with water to make a total volume of 4 liters and placed in a Denver Equipment Company laboratory flotation cell. There was then added 0.4 gram of a 32% aqueous solution of the sodium salt of the sulfate ester of the mixture of oxygenated organic compounds, predominantly alcohols boiling between 147–157° F. obtained from the catalytic dehydrogenation of carbon oxides under elevated temperatures and pressures. An excellent froth well loaded with coal particles was produced. Concentrate A was collected during a 30 minute flotation period. The coal was readily carried out of the cell by the froth without severe flocculation and very little mechanical raking was required. Another 0.1 gram of the same reagent was then added and concentrate B collected during another flotation period of about 10 minutes. The pulp water remaining in the cell was gray in color and obviously nearly free from coal particles. The results of the experiment are summarized in the following table:

Product	Weight (Gram)	Ash (%)	Weight ash (Gram)	Weight coal (Gram)
Concentrate A	383	5.48	21.0	362.0
Concentrate B	512	8.84	45.3	466.7
Tailings	71	47.46	33.7	37.3
Feed	966	10.35	100.0	866.0

Coal recovered in concentrates A plus B, 95.7%.

Calculated ash content of concentrates A plus B, 7.40%.

Ash rejected by flotation, 33.7% of total.

Reagent used, 1.0 pound of 32% solution per ton of coal or 0.32 pound on a dry basis.

In a control experiment 2.6 pounds of cresylic acid per ton of coal were required to effect as substantially a complete recovery of the coal.

The novel flotation agents described herein may be used as the sole flotation agents or may be used in combination with flotation agents already known to the art, for example pine oil, cresylic acid, xanthate, crude oil, and the like.

The fraction or mixture of fractions of oxygenated organic compounds which are sulfonated to obtain my flotation agent will to a certain degree determine the extent of the utility and the efficiency of the compounds obtained in the flotation of a given ore. By the selection of a given fraction or a mixture of fractions it is possible, therefore, to obtain flotation agents having varying degrees of chemical and physical properties with corresponding variation in flotation ability. The expert in this art, from this group of flotation agents, may then select those which are most efficient for application to his particular separation problem.

I claim:

1. In the froth flotation process of treating ores, the step which comprises utilizing as a flotation agent a sulfuric acid derivative of a mixture of the oxygenated organic compounds boiling above 100° C. obtained from the catalytic hydrogenation of carbon oxides under elevated temperatures and pressures.

2. In the froth flotation process of treating ores, the step which comprises utilizing as a flotation agent a sulfate of a mixture of the oxygenated organic compounds boiling above 100° C. obtained from the catalytic hydrogenation of carbon oxides under elevated temperatures and pressures.

3. In the froth flotation process of treating ores, the step which comprises utilizing as a flotation agent a salt of a sulfate of a mixture of the oxygenated organic compounds boiling above 100° C. obtained from the catalytic hydrogenation of carbon oxides under elevated temperatures and pressures.

4. In the froth flotation process of treating ores, the step which comprises utilizing as a flotation agent a metal salt of the oxygenated organic compounds sulfates, the oxygenated organic compounds boiling above 100° C. from which the salts are made having been obtained from the catalytic hydrogenation of carbon oxides under elevated temperatures and pressures, the metal of the salt being selected from the group comprising the alkali metals, the alkaline earth metals, lead, aluminum, and the heavy metals.

5. In the froth flotation process of treating ores, the step which comprises utilizing as a flotation agent a mixture of the oxygenated organic compounds obtained from the catalytic hydrogenation of carbon oxides under elevated temperatures and pressures, boiling between 100–240° C., which has been converted into a sulfuric acid derivative.

6. In the froth flotation process of treating ores, the step which comprises utilizing as a flotation agent a mixture of the oxygenated organic compounds obtained from the catalytic hydrogenation of carbon oxides under elevated temperatures and pressures, boiling between 130–147° C., which has been converted into a sulfuric acid derivative.

7. In the froth flotation process of treating ores, the step which comprises utilizing as a flotation agent a mixture of the oxygenated organic compounds obtained from the catalytic hydrogenation of carbon oxides under elevated temperatures and pressures, boiling between 157–195° C., which has been converted into a sulfuric acid derivative.

8. In the froth flotation process of treating ores, the step which comprises utilizing as a flotation agent a mixture of the oxygenated organic compounds obtained from the catalytic hydrogenation of carbon oxides under elevated temperatures and pressures, boiling at 240° C. up, which has been converted into a sulfuric acid derivative.

9. In the froth flotation process of treating ores, the step which comprises utilizing as the flotation agent an alkaline earth salt of the mixture of oxygenated organic compound sulfates, the oxygenated organic compound boiling above 100° C. from which the sulfates were prepared, having

been obtained from the catalytic hydrogenation of carbon oxides under elevated temperature and pressure.

10. In the froth flotation process of treating ores, the step which comprises utilizing as the flotation agent an alkali metal salt of the oxygenated organic compound sulfates, the oxygenated organic compound boiling above 100° C. from which the sulfates were prepared having been obtained from the catalytic hydrogenation of carbon oxides under elevated temperature and pressure.

11. In the froth flotation process of treating ores, the step which comprises utilizing as the flotation agent a lead salt of the oxygenated organic compound sulfates, the oxygenated organic compound boiling above 100° C. from which the sulfates were prepared having been obtained from the catalytic hydrogenation of carbon oxides under elevated temperature and pressure.

12. In the froth flotation process of treating ores, the step which comprises utilizing as the flotation agent a salt of a sulfuric acid derivative of a mixture of oxygenated organic compounds boiling above 100° C. obtained from the catalytic hydrogenation of carbon oxides under elevated temperature and pressure.

13. As a new flotation agent, the heavy metal salt of sulfuric acid derivatives of the oxygenated organic compounds boiling above 100° C. obtained from the catalytic hydrogenation of carbon oxides under elevated temperatures and pressures.

14. As a new flotation agent, the heavy metal salt of sulfuric acid derivatives of the oxygenated organic compounds obtained from the catalytic hydrogenation of carbon oxides under elevated temperatures and pressures, said oxygenated organic compound prior to sulfonation boiling between 130–240° C. up.

15. As a new flotation agent, a heavy metal salt of sulfuric acid derivatives of an oxygenated organic compounds boiling above 100° C. obtained from the catalytic hydrogenation of carbon oxides under elevated temperatures and pressures, the metal of the salt being selected from a group comprising lead, aluminum, and the other heavy metals.

16. In the froth flotation of coal bearing slack the step which comprises utilizing as a flotation agent a sulfuric acid derivative of a mixture of the oxygenated organic compounds boiling above 100° C. obtained from the catalytic hydrogenation of carbon oxides under elevated temperatures and pressures.

17. In the froth flotation of coal bearing slack the step which comprises utilizing as a flotation agent a sulfuric acid derivative of a mixture of the oxygenated organic compounds obtained from the catalytic hydrogenation of carbon oxides under elevated temperatures and pressures the compounds prior to being converted to the sulfuric acid derivatives boiling between 147 and 157° C.

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