Sept. 17, 1935. F. WEED 2,014,405

CONCENTRATING IRON ORES BY FROTH FLOTATION

Filed Oct. 12, 1932

Fig. 2.

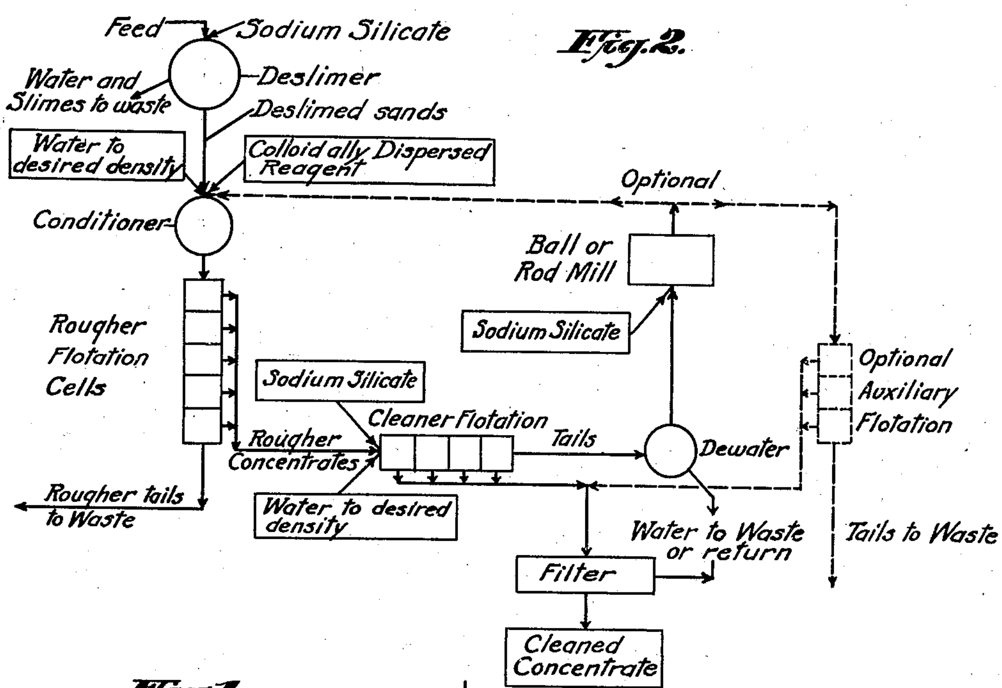
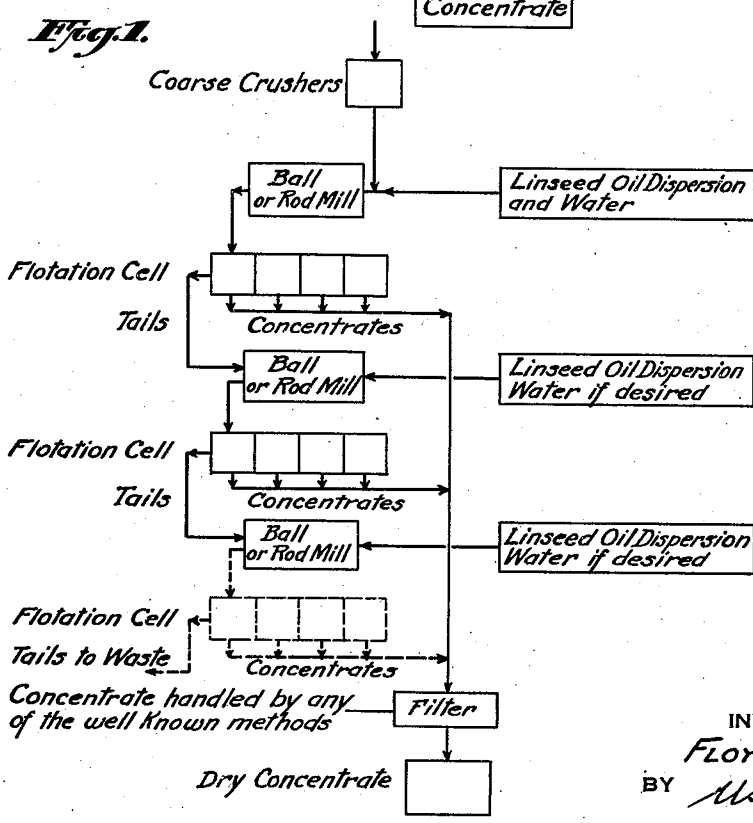


Fig. 1.



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CONCENTRATING IRON ORES BY FROTH FLOTATION

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9 Claims. (Cl. 209—166)

This invention relates to ore concentration methods and more particularly to the concentration of iron ores by froth flotation methods.

Heretofore many attempts have been made to concentrate iron ores by froth flotation. Low grade iron ores or iron ores associated either with minerals which are deleterious during subsequent smelting operations or which if not deleterious minerals are present in such excessive amounts as to tend to increase the costs of subsequent smelting operations through requiring the additions of inordinately large amounts of coke, lime and the like materials to the smelting ore. Such attempts at froth flotation of iron ores as have heretofore been made however have not been productive of commercially practical results.

One of the objects of the present invention is to provide a commercially practical and economical method of concentrating iron ores by froth flotation.

Another object of the present invention is to provide an improved reagent having a selective affinity for the iron content of an iron ore for use in the froth flotation of iron ores.

Another object of the present invention is to facilitate the manufacture of iron and steel from iron ores heretofore considered economically impractical to smelt.

Other objects and advantages will become apparent as the invention is further disclosed.

In accordance with the objects of the present invention I have discovered that the iron content of iron ores generally may be economically segregated from associated minerals by froth flotation by using a colloidally dispersed selective affinity reagent in accordance with the invention defined and claimed in copending application entitled "Method of concentrating non-sulfide minerals by froth flotation", Serial No. 637,542 filed October 12, 1932 by Floyd Weed and Edwin E. Ellis The said Floyd Weed, coinventor of the said copending application, is identical with the Floyd Weed of the present invention.

In applying the invention of the said copending application to iron ores, it has been found that the reagent showing a selective affinity for iron ores is preferably a mineral and/or vegetable oil. It has been found that linseed oil specifically is the preferred specific mineral and/or vegetable oil indicating the most marked and economical selective affinity for the iron content of iron ores. A coal tar creosote product known to the trade as Barrett #4 flotation oil, however, may be equally as well employed. Admixture of mineral and/or vegetable oils also may be utilized.

Heretofore in the art the chief difficulty in applying froth flotation to non-sulfide ores has been to properly and economically incorporate the reagent evidencing a selective affinity for the desired mineral values of an ore within the flotation pulp. It has heretofore been proposed to mechanically incorporate the reagent within the ground ore, either during grinding or subsequent to grinding. It has also been proposed to incorporate the reagent as a water soluble soap compound or as a fluid compound miscible with the water of the flotation pulp. Each of these proposed methods is marked by an inordinate use of reagent tending to make the process economically inoperative especially when applied to low grade ores.

In accordance with the invention of the above identified application I propose to add the reagent evidencing a selective affinity for the iron content of iron ores to the flotation pulp as a colloidal dispersion in water containing a small proportion of a water soluble soap compound. The addition of the colloidally dispersed reagent may be made to the ore during the final grinding operations, or to the flotation pulp just prior to subjecting it to flotation or at any point therebetween.

In the forming of the colloidal dispersion I prefer to use for most iron ores a soap compound comprised of an amine and oleic acid for the reason that most iron ores are comprised in addition to silica of water soluble calcium and magnesium salts which tend to react with ionized soap compounds comprised of fatty acids and alkali metals to form insoluble soap compounds thereby removing from the flotation pulp the beneficial frothing agency of the soap compounds. Amines generally are less basic than the alkali metals and the primary and tertiary amines are estimated to have a basicity approximating that of ammonia from which they are derived. It is preferable, therefore, to utilize the primary or tertiary amines in the forming of the soap compound and specifically I prefer to employ a commercial amine product known to the trade as "Triethanolamine" which is comprised in major part of triethanolamine and in part of secondary and primary amines.

Triethanolamine will react with oleic acid to form a water soluble salt in the proportion of 2.7 parts of oleic acid to 1 part (by volume) of triethanolamine. The soap compound is completely soluble in water. This water soluble soap may be obtained by adding the triethanolamine (2 c. c.) to water (70 c. c.) and then slowly adding 5.4 c. c. of oleic acid to the water-triethanolamine solution while aerating and agitating the solution. Instead of oleic acid other fatty acids may be employed to form a soap compound in an analogous manner but in view of the superior frothing properties of oleic acid soaps over other soap compounds and in view of the favorable basicity of the same above noted I prefer to use oleic acid soaps. By using oleic acid soaps I avoid the use of additions of froth modifying agents such as pine oil, crysylic acid and the like during subsequent froth flotation.

In the preparation of a colloidal dispersion of the agent exerting or evidencing a selective affinity for the iron content of iron ores, I add the reagent to water containing a desired proportion of the desired soap compound and aerate and agitate the resulting admixture until the desired emulsion or colloidal dispersion has been obtained. The specific amounts of reagent added to form the emulsion will depend upon the specific soap compound utilized and the quantity thereof, and with any given soap and water solution the amount of reagent to be added will vary with respect to the specific reagent desired.

Accordingly, as a specific embodiment of the practice of the present invention with respect to the forming of a colloidal dispersion I will disclose the same as it is applied to the preparation of a colloidal dispersion of linseed oil and of a coal tar creosote product known to the trade as Barrett #4 mineral oil in a water solution of a soap comprised of triethanolamine and oleic acid, and will further disclose the method of froth flotation of iron ores and materials that I have devised using these specific colloidally dispersed reagents.

A linseed oil emulsion or colloidal dispersion which is stable and which may be further diluted with water and still remain stable can be formed by slowly adding about 96.5 parts (by weight) linseed oil to about 150 parts (by weight) distilled water containing 3 parts (by weight) oleic acid and ½ part (by weight) triethanolamine, and aerating and agitating sufficiently during the addition of the linseed oil and for a time interval thereafter necessary to form the desired emulsion of colloidal dispersion.

As above indicated it is immaterial for the purposes of the present invention whether the triethanolamine soap be formed first and added to the water or whether the triethanolamine be first added to the water and the oleic acid added thereto. It is also immaterial whether the oleic acid be admixed with the linseed oil and the linseed oil-oleic acid admixture then added to the water-triethanolamine admixture. A stable emulsion suitable for the purposes of the present invention will be obtained in each case.

The specific proportions of linseed oil, triethanolamine soap and water may be varied widely and still obtain an emulsion of colloidal dispersion suitable for the purposes of the present invention and it may be varied widely depending upon the extent of dispersion desired, the specific ores being treated therewith, the relative proportion of selective affinity reagent to soap compounds desired within the flotation pulp and the presence of or absence of froth modifying agents or pulp conditioning agents within the flotation pulp or within the dispersion. When froth modifying agents are desired or required they may be added to the flotation pulp before or after the addition of the colloidally dispersed reagent or they may be colloidally dispersed together with the reagent in the water-soap solution. The same may be done with reagents tending to facilitate the segregation of the iron content of the ore from the gangue minerals.

A stable colloidal dispersion of Barrett #4 flotation oil may be formed by admixing the oil and oelic acid together and then slowly adding this admixture to water containing triethanolamine and aerating and agitating until the desired colloidal dispersion or emulsion has been obtained. If desired the oleic acid may be added to the water and triethanolamine solution first and the Barret #4 oil added thereafter but better and more efficient results may be obtained by forming the dispersion as first indicated. The preferred admixture contains:—

	Per cent by weight
Oleic acid	30
Triethanolamine	5
Barrett #4 (flotation oil)	8
Water	57

This colloidal dispersion may be diluted with water to form a stable emulsion. The amount of oleic acid employed in the forming of the Barrett #4 dispersion is higher than that used in forming a linseed oil dispersion as is also the amount of triethanolamine. This has been found to be necessary in order to obtain a stable emulsion of the Barrett #4 flotation oil which is a mineral oil specifically refined for flotation purposes. The additional oleic acid favorably acts upon the iron content of the ore and appears to favor the segregation of the same from some types of associated gangue materials. Instead of this specific colloidally dispersed reagent however I may employ colloidally dispersed linseed oil or any other type of mineral oil exerting a selective affinity for the iron. The amount of this reagent added approximates 1.5 pounds per ton of ore. No frothing agents or froth modifying agents are required. After the addition of the reagent the pulp should be conditioned for from .5 to 1.5 minutes for best results.

As one specific embodiment of the present invention, I will disclose the method I have devised for the concentration of iron ores known geologically as Birmingham Red Ores by froth flotation. Birmingham Red Ores are essentially hematite ores and are located in the Birmingham district of Alabama. There are millions of tons of these iron ores which are too high in insolubles to be economically smelted for the production of pig iron. The costs of additional fluxing materials and of coke to ensure the smelting and of reduced blast furnace output do not permit a smelter to use these ores when ores are available wherein the silica and the fluxing materials, lime and magnesia, are more nearly balanced for best fluxing.

The iron content of these ores is mostly present as hematite (Fe_2O_3) and the ore averages about 36% Fe. Associated with the hematite are varying amounts of so-called insoluble materials silica and alumina together with varying proportions of calcium and magnesium carbonates. In one section of the district an ore high in calcium and magnesium carbonates and low in insolubles is found while in another section of the district the reverse proportion of insolubles and calcium and magnesium carbonates is found. It is apparent, therefore, that an efficient and commercially practical method of concentrating these ores to obtain a hematite product of relative uniform composition and high iron content would be highly desirable. By the practice of the present invention this desirable product may be obtained.

Before further disclosing this specific embodiment reference should be made to the accompanying drawing wherein—

Fig. 1 is a flow sheet diagram of the process devised for the froth flotation of Birmingham Red Ores; and

Fig. 2 is a flow sheet diagram of the process devised for the froth flotation of washer tailings from the Canisteo district, Minnesota.

Referring to Fig. 1, the process disclosed comprises basically a coarse ore crushing step, a final grinding step during which the colloidally dispersed reagent is added, a first flotation concentration, a tailings grinding and flotation operation and a recovery step of the two flotation concentrates.

In the coarse ore crushing step the ore is reduced to a particle size approximating ½" in diameter by any heretofore well known means, such as a jaw or gyratory crusher followed by rolls or by a cone crusher. The crushed ore is then admixed with water and fed into a rod or ball mill. A quantity of colloidally dispersed linseed oil reagent (in water containing a proportion of a soap compound comprised of triethanolamine and oleic acid) sufficient to recover the iron content of the ore is then added and the crushed ore then is ground to the desired particle size. The specific particle size to which the ore must be ground will vary somewhat according to the quantity and kind of associated minerals. In practice the iron oxide, lime and magnesia carbonate particles tend to be reduced to small sized particles more rapidly than the silica, partly because of their greater softness and partly because of the nature of their occurrence as a blanket or mat around rounded grains of silica. I have found that a relatively light grinding is effective to obtain the necessary freeing of the iron oxide from the associated minerals.

By adding the colloidally dispersed reagent evidencing a selective affinity for the iron oxide to the ore during grinding, the particles of iron oxide, lime and magnesia carbonates are contacted with the reagent as they are separated from the silica and alumina, and it is therefore unnecessary to grind these particles to such a fine particle size as would otherwise be necessary. Alternatively, the colloidally dispersed reagent may be added to the pulp after grinding and classifying and prior to flotation provided that a suitable conditioning operation be made prior to flotation to insure the contacting of the reagent with all of the iron particles. The addition of the reagent during light grinding however is preferred.

The pulp from the ball or rod mill including the reagent then may be passed directly into a flotation cell and a first concentrate obtained therefrom which is about 40% of the heads and carries about 60 to 65% of the total iron content of the heads with about 10% to 12% of the total insolubles in the heads. This concentrate may be recovered in any convenient manner as by filtering and is of a sufficiently high grade to be economically smelted directly to pig iron.

The tails from the first flotation cell still carry a large proportion of iron and may be economically treated again by froth flotation for the recovery of the iron content. The tails should first be admixed with water to form a flotation pulp of the desired density, and reground in a ball or rod mill in the presence of a suitable amount of the colloidally dispersed linseed oil reagent. After grinding the pulp may be again passed through a flotation cell as before and the concentrate recovered may be combined with the first concentrate for smelting. If necessary or desired, and as indicated in dotted lines the flotation may be repeated a third or more times until economically impractical.

The amount of colloidally dispersed reagent to be added to the pulp during grinding depends primarily upon the amount of iron present and the estimated efficiency of the grinding. It is undesirable to grind too fine as the slime material thus produced hinders subsequent flotation and contaminates the recovered concentrate thus lowering the grade of the product. I have found, for example, that with an iron ore of the type described averaging about 31 to 32% Fe with about 30% insolubles, balance lime and magnesia carbonates, the addition of sufficient colloidally dispersed reagent, usually approximately 5 pounds of the reagent, and containing 1.9 pounds of linseed oil, .15 pound of oleic acid, and .1 pound of triethanolamine is adequate to obtain a recovery on the first flotation of about 60 to 65% of the total iron of the ore. Where Fe recoveries higher than this are sought the relative proportion of insolubles becomes excessive. Where the tailings are reground and subjected to a second flotation it is necessary to add only about 3 pounds of linseed oil emulsion per ton of ore. A third flotation operation would require about the same amount of linseed oil as is used in the second.

The specific colloidal disperson preferred is that having the composition and relative proportion of linseed oil, oleic acid, triethanolamine and water hereinbefore identified as this dispersion is stable for prolonged periods and dilutable with water to form stable dilutions thereof. Other proportions of materials forming colloidal dispersions of lesser or greater stability may be used however without departing essentially from the nature and scope of the present invention.

It is not necessary in this specific embodiment to add to the pulp frothing agents or froth modifying agents.

As a second specific embodiment of the present invention I disclose in Fig. 2 a flow sheet diagram of a process utilizing the present invention as applied to the recovery of the iron content of washer tailings from iron ores of the Canistea district of Minnesota. These tailings contain about 20% iron and are essentially comprised of finely divided ore and silica particles. By the practice of the present invention and with modifications to adapt the same to the type of material involved a concentrate averaging 60 to 62% Fe and about 10 to 12% insolubles can be obtained.

In Fig. 2 the flow sheet diagram of the process I have devised for the froth flotation of this material is disclosed. The washer tailings must first be deslimed and dewatered in any heretofore known manner. To facilitate desliming sodium silicate is added in an amount varying with the amount of slimes to be removed. I have found that generally from 1 to 2 pounds of sodium silicate per ton of ore is sufficient where the slimes are not excessive. A moderate excess of sodium silicate is not harmful during subsequent flotation as it there may be utilized as an inhibiting reagent for silica. The slimes lost are about 3 to 4% of the total tailings and contain about 6% of the total iron of the feed. The removal of the slimes facilitates the subsequent flotation and reduces the amount of colloidally dispersed reagent that is necessary for flotation.

The deslimed material then is made up into a flotation pulp of about 25 to 30% solids and flowed to the conditioner where the desired amount of colloidally dispersed Barrett #4 flotation oil prepared substantially as above noted is added.

The conditioned pulp then is passed through a flotation cell and a rougher concentrate obtained. The tailings may be discarded. The rougher concentrate then is subjected to a second flotation after from .05 to .1 pounds of sodium silicate per ton of ore has been added thereto. The cleaner concentrate thus obtained will average about 60 to 62% Fe and may be economically smelted into pig iron.

To recover the remaining iron content of the tailings from the cleaner flotation, the tailings may be subjected to grinding in a rod or ball mill, conditioned with sodium silicate and either returned to the rougher flotation cell or subjected to concentration in a separate flotation cell as is indicated in dotted lines in Fig. 2. The concentrate from the third flotation cell may be admixed with the cleaner concentrate as is indicated in dotted lines in Fig. 2, and economically smelted therewith into pig iron.

Having broadly and specifically defined the present invention and having disclosed two specific embodiments of the practice of the same, it is apparent that many modifications and departures from the same may be made without departing from the nature and scope thereof as may be included within the following claims.

What I claim is:

1. In the concentrating of oxidized iron ores by froth flotation, a flotation reagent comprising a coal tar creosote product colloidally dispersed in an aqueous solution of a soap compound comprised of oleic acid and triethanolamine.

2. In the concentrating of oxidized iron ores by froth flotation, a flotation reagent comprising an admixture of coal tar creosote and linseed oil colloidally dispersed in an aqueous solution of a soap compound comprised of oleic acid and triethanolamine.

3. In the concentrating of oxidized iron ores by froth flotation, a flotation reagent comprising a linsed oil colloidally dispersed in an aqueous solution of a soap compound comprised of oleic acid and triethanolamine.

4. In the concentrating the oxidized iron ores by froth flotation the step of adding to the flotation pulp linseed oil colloidally dispersed in an aqueous solution of a soap compound comprised of oleic acid and triethanolamine.

5. In the concentrating of oxidized iron ores by froth flotation, the method which comprises crushing the ore to relatively small particle size, adding water thereto, incorporating therein a proportion of linseed oil colloidally dispersed in an aqueous solution of an alkyl amine soap compound, grinding the ore to the desired final particle size, and thereafter subjecting the same to froth flotation.

6. In the concentrating of oxidized iron ores by froth flotation, the method which comprises forming a flotation pulp of said ore, adding thereto a proportion of linseed oil colloidally dispersed in an aqueous solution of a triethanolamine soap compound, agitating and aerating the pulp to form a minerals bearing froth, and separating the said froth.

7. In the concentrating of oxidized iron ores by froth flotation, the method which comprises forming said ore into a flotation pulp, adding thereto a proportion of linseed oil colloidally dispersed in an aqueous solution of a fatty acid-triethanolamine soap compound, agitating and aerating the pulp to form a minerals bearing froth and separating the said froth.

8. In the concentrating of oxidized iron ores by froth flotation, the method which comprises forming said ore into a flotation pulp, adding thereto a proportion of linseed oil colloidally dispersed in an aqueous solution of a soap compound consisting of oleic acid and triethanolamine, agitating and aerating the pulp to form a minerals bearing froth and separating the said froth.

9. In the concentrating of oxidized iron ores by froth flotation, the method which comprises incorporating in the flotation pulp a proportion of oily reagent evidencing a selective affinity for the oxidized iron content of said ore, said reagent comprising at least one of a group comprising a coal tar creosote product and linseed oil and being colloidally dispersed in an aqueous solution of an alkyl-amine soap compound and thereafter agitating and aerating the pulp to form a minerals bearing froth and separating the froth.

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