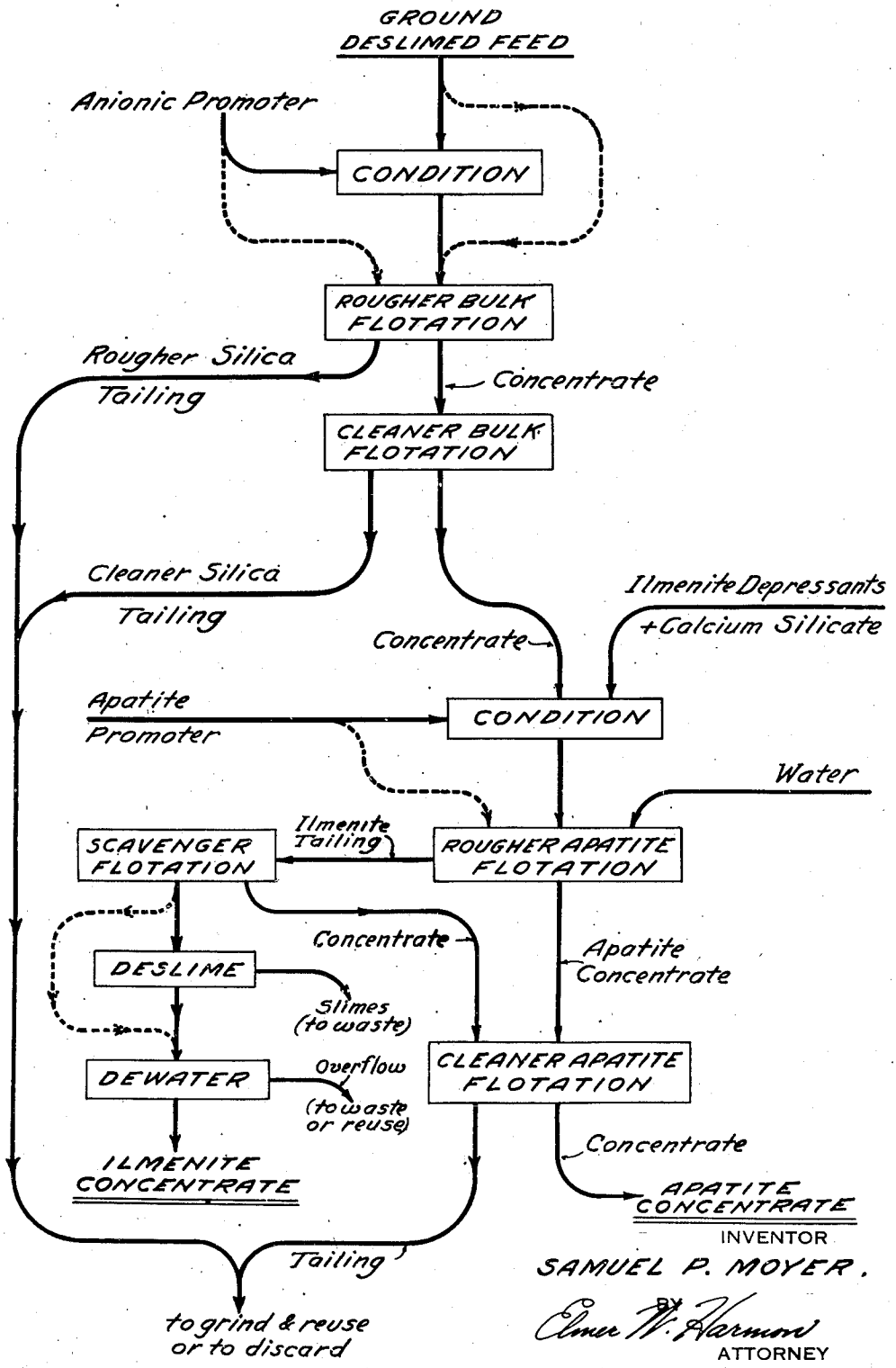
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FROTH FLOTATION OF APATITE USING CALCIUM SILICATE

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FROTH FLOTATION OF APATITE USING CALCIUM SILICATE

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This invention relates to the beneficiation by froth flotation of those metallic oxide ores containing excessive amounts of phosphate minerals. More particularly, it is concerned with the separation of phosphorus-bearing minerals such as apatite from metallic oxide minerals such as ilmenite, rutile, magnetite, hematite, and the like.

The typical problems involved as well as their solutions are found in the production of ilmenite concentrates. In commercial practice, the use of titanium oxides has become increasingly important and accordingly, the demand for high grade ore from which they may be recovered has steadily increased. Unfortunately, while there are many natural deposits of titanium minerals, the grade of these ores in many cases is not sufficiently high to make recovery of titanium oxides therefrom commercially feasible. Particularly is this true of most deposits within the United States. Therefore, the process of the present invention will be discussed in its application to ilmenite ores. Ilmenite, however, is used for illustrative purposes only and is intended as a representative of titanium-bearing oxide ores generally.

Natural deposits of titanium-bearing minerals are usually ores containing, in addition to a titanium-bearing mineral such as ilmenite, rutile, or the like, a silica-bearing gangue. Ordinarily, this silica and/or silicate content must be reduced to produce a concentrate meeting commercial standards, for example, to be used as raw material for the production of titanium oxide pigments.

In addition to the problems presented by silica or silicates, many ores, particularly in the United States, contain a certain amount of phosphorus-bearing minerals. In the illustrative case of ilmenite, this is frequently apatite which will therefore also be taken as illustrative of phosphorus bearing minerals generally. The presence of phosphorus is objectionable in a raw material for the production of titanium oxide pigments in their various crystal forms, such as rutile or anatase. Particularly is this true in the latter case. A commercially acceptable concentrate must have a low P_2O_5 content. Since the P_2O_5 assay may vary from about 1–2% in ilmenite concentrates obtained from ores which are worked primarily for other constituents to 12% or more, as found in ilmenite ores such as nelsonite, the reduction of the P_2O_5 content is essential. Those processes which in the past have been employed to reduce the silica or silicate content are not effective for this purpose.

Despite the demand therefor, there has been available no process of readily and effectively-producing concentrates of metallic oxides sufficiently low in P_2O_5 content. Neither have the

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concentrates previously obtainable been in a condition in which they can be readily dewatered and dried for further processing.

It is, therefore, a principal object of the present invention to provide a process whereby a satisfactorily low P_2O_5 content concentrate can be obtained. It is also an object to provide a process whereby this is readily and cheaply accomplished. It is a further object to provide suitable reagents for use in this process, which reagents are simple, readily obtainable, effective in use and economical in operation.

It is with the reduction in the P_2O_5 content that the process of the present invention is particularly concerned. In accordance with the present invention, separation of apatite, for example, from a metallic oxide such as ilmenite is made by conditioning a pulp of the minerals to be separated with a group of reagents including a novel ilmenite depressant and froth conditioning agent and then conducting a flotation with an anionic promoter for the apatite.

The process of the present invention is of general application to mixtures of various origin. This is true, whether the mixture to be beneficiated is a concentrate from an ore that is worked primarily for some mineral content other than the ilmenite; or is a natural apatite-ilmenite mixture; or is a bulk concentrate obtained, in accordance with the teachings of my copending application, Serial No. 636,199, filed of even date, by floating a mixed concentrate of the desired metallic oxide and apatite away from the silica, silicates and other acidic constituents with an anionic promoter. It is also applicable to mixtures variously obtained by magnetic or gravity separation.

It is highly desirable that the apatite separation be carried out using anionic reagents, if possible, since the latter are not only usually both simpler and more economical in use but produce a more readily-dispersible concentrate. Unfortunately, apatite and ilmenite react very much the same to most anionic-type promoters. It is necessary, therefore, that a depressant be found for one constituent or the other. As shown in my above identified copending application, it is possible to depress the metallic oxide minerals by the use of a suitable soluble alkali metal silicate in an alkaline circuit. If a pulp of the minerals to be separated is conditioned, for example, with a suitable amount of a sodium silicate and caustic soda, the apatite may then be selectively floated therefrom almost quantitatively with substantially any good anionic-type promoter such as a fatty acid, resin acid, talloel, petroleum sulfonates, or soaps thereof.

While this process is effective and has produced

commercially acceptable results, it is not as easily employed as is often desirable in a commercial operation. The froth characteristics, for example, are not always satisfactory, in that the froth tends to be thin and watery, and, as a result, the apatite floats more slowly. Attempts to use other modifying agents such as lignins or tannins does not improve the situation appreciably. Substituting other alkaline materials such as potassium or ammonium hydroxides produces effective results, but not appreciably better. Substitution of alkaline earth metal hydroxides for the sodium hydroxide does not produce any operable results, since selectivity is destroyed.

It has now been found that in addition to sodium silicate and caustic soda a relatively small amount of calcium silicate is used, the difficulties with froth control and rate of flotation are completely overcome. This is quite surprising, in view of the fact that an attempt to use sodium silicate and lime as separate reagents, as noted above, does not produce any useful separation.

An illustration of the use of the calcium silicate is shown in the accompanying drawing which is a flow sheet delineating a typical process of recovering an ilmenite concentrate from an original ore containing both apatite and silicate-bearing gangue.

If an original ore is to be treated it is prepared for use by grinding and desliming. Usually, grinding the ore to about the size of minus 65 mesh is found to be good practice, although somewhat larger or smaller sizes may be desirable in some cases. If the feed is a previously made concentrate, this may not be necessary.

Also in the case of an original ore some reduction in acidic gangue content is required. An excellent process for this is disclosed in my above-mentioned copending application in which an apatite-ilmenite concentrate is directly floated away from the acidic gangue with an anionic type promoter. The steps therefor are shown in the accompanying flow sheet. As shown in the drawing it is desirable, when possible to do so, to condition a pulp of the ore, preferably at 30–70% solids content, with the anionic promoter. Where this is not practical because of lack of space for conditioning, or other limitations, the reagent may be added directly to the pulp in the flotation cell. Preferably, the flotation is carried out in a neutral or slightly alkaline circuit since this appears to aid in obtaining a bulk concentrate of the apatite and ilmenite which is relatively free from silica or silicates. This concentrate is usually given at least one cleaner float, the tailings from both the rougher and cleaner flotations being discarded or, if desired, reprocessed by being returned to the grinding and desliming steps which form no part of the present invention.

The apatite-metallic oxide mixture, whether as a prepared bulk concentrate comprising the major portions of both the ilmenite and apatite of an original ore, or a mixture from some other source such as a table concentrate is then conditioned with the sodium silicate, sodium hydroxide and calcium silicate. It is not important that this conditioning be carried out at high solids. The time of conditioning required will vary, depending on the characteristics of the ilmenite-apatite concentrate, but commonly about 7 to 10 minutes is sufficient to insure good results in the subsequent flotation step.

An anionic promoter of the same general class used in obtaining the bulk concentrate is used to float a relatively ilmenite-free apatite concentrate. This promoter may be the same or different than the promoter chosen in the bulk flotation step. Talloel or talloel sodium soap is commonly found highly satisfactory for both purposes, and because of its low cost and availability it is probably preferable. In the bulk concentration step a sodium neutralized soap is usually found to give somewhat better results. Since the flotation of apatite from ilmenite is conducted in an alkaline circuit, the use of the unsaponified material probably only results in saponification, or at least a part thereof in situ.

The presence of the calcium silicate appears to overcome all the previously found objectionable characteristics of the froth. In addition, both the grade and recovery of the apatite concentrate is usually considerably improved.

As shown in the flow sheet, the apatite concentrate preferably should be given a cleaner flotation. If so desired, the cleaner tailings may be either discarded or recycled to the grinding operation. As a practical matter, it is usually desirable that the ilmenite tailing be passed through a scavenger cell to insure against occasional inequalities in operation. The concentrates from this cell, if any, are passed to the apatite cleaner flotation and the tailings constitute the ilmenite concentrate. The latter is in a particularly well-dispersed condition so that it is easily deslimed and subsequently easily filtered or dried, as may be desired for use in further processes.

The invention will be illustrated in conjunction wtih the following example which is illustrative only, and not by way of limitation.

Example

A sample of nelsonite ore containing, in addition, apatite silica-bearing gangue and small quantities of other metallic oxide minerals was reduced to about minus 10 mesh, agitated at about 40% solids with 3.60 lbs./ton and deslimed, ground to about minus 65 mesh in the presence of about 0.60 lb./ton of sodium silicate and the gangue deslimed. The deslimed sands were conditioned to 0.40 lb./ton of saponified talloel, and the concentrate therefrom given a cleaner flotation with 0.05 lb./ton of saponified talloel. The cleaner concentrate was conditioned with 1.20 lb./ton of sodium silicate, 0.30 lb./ton of NaOH and about 0.60 lb./ton of calcium silicate. This was followed by a short conditioning with 1.20 lbs./ton of saponified talloel and the conditioned pulp subjected to froth flotation. The concentrate therefrom was given a cleaner flotation, the product therefrom constituting the final apatite concentrate. The apatite tailing was deslimed, the sands therefrom constituting the ilmenite concentrate. The metallurgical results are shown in the following table.

Metallurgical results

Product	Weight, percent	Assay		Distribution, percent	
		Percent TiO_2	Percent P_2O_5	TiO_2	P_2O_5
Feed	100.00	22.40	9.03	100.0	100.0
Clean Ap. Ct.	15.79	7.60	34.35	5.4	60.0
Clean. Ap. Tail	2.78	41.31	2.07	5.1	0.6
Ilm. Ct. (Sand)	26.68	45.85	0.02	54.6	0.1
Ilm. Ct. (Slime)	2.67	43.06	0.49	5.1	0.2
Rough Tailing	20.01	2.87	0.93	2.6	2.0
Cl. Talling	32.07	19.02	10.44	27.2	37.1

It will be noted that the procedure described in the example resulted in a final ilmenite product

analyzing 0.02% P_2O_5. Even without the final desliming step on the ilmenite product (tailing) from the apatite float, the P_2O_5 content is only 0.063% P_2O_5.

The cleaned bulk concentrate from the first flotation step contained 11.48% P_2O_5 and this was reduced to 0.063% P_2O_5 in the apatite flotation step. In other words 99.67% removal of the apatite was obtained in the retreatment of the cleaned bulk concentrate obtained in the first flotation operation.

It should be pointed out that the small amounts of rutile, magnetite and hematite occurring with the ilmenite in this sample of nelsonite ore were recovered with the ilmenite.

Calcium silicate is not particularly soluble in water. Because of this fact it is somewhat difficult to feed in the proper concentrations. A simple solution to the difficulty is to dissolve about 20 to 40 parts of the calcium silicate to a million parts of water, and to use this treated water in making up the aqueous pulps in which the presence of the calcium silicate is desirable.

While the above discussion has been principally concerned with the separation of apatite from ilmenite, calcium silicate has been found effective as a froth modifier in various other fatty acid, resin acid or soap type flotations. It is particularly useful in obtaining well-flocculated concentrates not only of apatite but of other phosphate minerals such as phosphate rock. In addition, it has been found useful in many separations of valuable minerals in an anionic flotation from silicious gangue minerals.

I claim:

1. In the separation of apatite from mixtures thereof containing at least one titanium-bearing oxide mineral, the improved procedure which comprises forming an aqueous, substantially slime-free, pulp of the mixture, conditioning the aqueous pulp with sodium silicate, an alkali, and calcium silicate, subjecting the conditioned pulp to froth flotation in the presence of an anionic promoter for the apatite, and collecting the resultant flotation concentrate as an apatite concentrate.

2. In the separation of apatite from mixtures thereof containing ilmenite, the improved procedure which comprises forming an aqueous, substantially slime-free, pulp of the mixture, conditioning the aqueous pulp with sodium silicate, an alkali, and calcium silicate, subjecting the conditioned pulp to froth flotation in the presence of an anionic promoter for the apatite, and collecting the resultant flotation concentrate as an apatite concentrate.

3. A process according to claims 1 and 2 in which about 20–40 parts of calcium silicate per million parts of water is added to the process water.

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