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SIMULTANEOUS FLOTATION OF SILICA PHOSPHATES AND CARBONATE MINERALS

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ABSTRACT OF THE DISCLOSURE

Process for flotation of phosphates, carbonates and silica with suppression of metallic oxides without desliming by dispersion of the pulp at an elevated pH within a controlled range and subsequent flotation.

This invention relates to flotation and more particularly to the flotation of carbonates, phosphates and silica.

Flotation procedures employed to date in connection with the concentration of the above materials have been characterized by the necessity for desliming the pulp prior to flotation and by the inability of the circuits to achieve a successful separation of the above materials from various materials particularly metallic oxides.

The process of the invention has as its principle object the elimination or minimization of the above two deficiencies.

According to the invention the pulp which contains the ore, comminuted to a particle size suitable to substantially liberate the minerals which it is desired to recover is adjusted to an elevated pH generally in the region of pH 10 or higher in the presence of a dispersing agent and a collecting agent, e.g. of the fatty acid type, petroleum sulphonate type or a combination of those two types either by themselves or with the addition of fuel oil. The conditioning is continued for a sufficient length of time to insure saponification of the fatty acid and/or petroleum sulphonate. The pH is then adjusted, e.g. with the use of sulfuric acid to the optimum pH for flotation of the mineral to be recovered. After a period of conditioning at the new pH, usually in the presence of additional quantities of dispersant a generally conventional flotation results in a surprisingly clean differential float of the desired material with substantially complete depression of the metallic oxides. In the case of pulp containing more than one of the above-mentioned minerals the pH may be adjusted a second time to accomplish differential flotation in respect to a second of such minerals with good control of the flotation.

I have found that where silica is the mineral to be floated the initial conditioning stage can be carried out within the optimum pH for flotation in which case the second conditioning stage with additional dispersing agent may be dispensed with, the flotation being conducted following the end of the initial conditioning period. The pH which I have found to be most satisfactory for the flotation of silica is the range of from about 11.0 to 12.0.

Where the mineral to be floated has an optimum pH range for flotation which is below the range of pH 11.0 to 12.0 and it is accordingly necessary to adjust the pH downwardly following the initial conditioning period, it is desirable that additional dispersing agent be added for a second period of conditioning prior to flotation. I have found that for the flotation of phosphates and carbonates the optimum pH range for flotation is from about 9.5 to 10.5.

While according to my invention it is possible by determining the optimum pH for the flotation of each of these minerals to float each differentially there are many instances where it is desirable to float all of these materials in the same float. It is an outstanding feature of the process of my invention that by prolonging the second conditioning stage with additional dispersing agent to an extent which is readily predetermined, a single flotation operation can be made to float all of those materials whose optimum pH range for flotation lies at or above the pH at which the flotation is carried out. Thus, for the first time so far as I am aware it has become possible to float carbonates, phosphates and silica together in a single float while effectively depressing the metallic oxides and without the need for desliming prior to flotation.

As flotation reagents any of the conventional reagents normally used for the flotation of the above mentioned materials may be employed and there is nothing unconventional in the flotation per se which may be conducted in any known manner and accommodated to the conventional practice of any particular flotation plant.

The preferred dispersing agent is sodium silicate by virtue of its being an effective dispersant and currently the most inexpensive. It has the added advantage of being almost universally available on a commercial scale. Other dispersing agents may however be used, the criteria of usefulness being simply the ability of the particular agent to accomplish effective dispersal of the particular materials which are present in the pulp without deleterious effects on the floatability of the materials which it is desired to float and without causing substantial activation of the metallic oxides which it is desired to suppress within the pH ranges of operation.

The reagents used for adjusting the pH are also conventional and suitably sulfuric acid is used as an acid reagent for lowering the pH and sodium hydroxide for raising the pH.

The following examples illustrate the operation of the process of the invention.

Example 1

A sample of niobium ore containing approximately 0.38% Nb_2O_5 and the remainder consisting essentially of approximately 40% calcite, 30–35% apatite and the remainder silica and iron oxides in the form of hematite and magnetite was prepared in a commercial grinding unit at a grind of −65 mesh at which grind nearly 30% of the ore, due to the natural slimes in the material, was finer than 20 microns. A pulp was prepared containing approximately 60% solids with 7.3 lbs. per ton of semi-refined tall oil and the pH was adjusted to 11.4 with the addition of sodium hydroxide and 7 lbs. per ton of sodium silicate. The pulp was conditioned for 90 minutes.

The pH was then adjusted to 10.1 with the addition of sulfuric acid. 3 lbs. per ton of sodium silicate was added with the pulp diluted to about 40% solids and the pulp was further conditioned for 15 minutes and then subjected to flotation in an open circuit consisting of a rougher float followed by three cleaners. Methyl isobutyl carbinol was used as a frother.

The metallurgical results were as follows:

Rougher tailing was 10.8% by weight and contained 2.10% Nb_2O_5.

The first cleaner tailing was 5.8% by weight and contained 1.10% Nb_2O_5.

The second cleaner tailing 4.4% by weight containing 0.68% Nb_2O_5.

The third cleaner tailing was 5.4% by weight containing 0.35% Nb_2O_5.

The final cleaner concentrate was 73.6% by weight containing 0.05% Nb_2O_5.

This illustrates the high degree of differential flotation with approximately 74% by weight of original ore con-

taining 0.05% Nb_2O_5. Although analysis of the products was not made for iron, visually, hematite and magnetite reported in the rougher and cleaner tailings.

Example 2

A sample of the same ore was prepared in the same way as that used in Example 1. The same reagent balance was employed and the same procedure followed as in Example 1 with the exception of the period of conditioning at pH 10.1 to 10.3 was extended to 30 minutes. The metallurgical results were as follows:

The final cleaner concentrate after the same three stages of cleaning contained 82% by weight and analyzed 0.058% Nb_2O_5 with the head value being slightly higher that is 0.411% Nb_2O_5. Thus, by increasing the conditioning time from 15 to 30 minutes the cleaner concentrate was increased from 73.6 to 82% in weight with negligible increase in Nb_2O_5. This additional weight is mainly silica and illustrates the effect of prolonging the conditioning time at the lower pH.

Example 3

A sample of iron ore from Belle Island, Newfoundland was prepared in a laboratory rod mill at a grind of approximately 85% minus 325 mesh. The main deleterious mineral in this ore was phosphrous in the form of a phosphate. The original head value was approximately 0.8 to 1% as P_2O_5. The head assay in iron was approximately 48% with gangue materials consisting mainly of silica and alumina. The procedure in this case was to adjust the pH of the pulp to the range of 11.4 to 11.6 having preconditioned with the sodium hydroxide and approximately 2 lbs. per ton of sodium silicate as a dispersant with the pulp consisting of approximately 45% solids. Following this initial stage calcium chloride was added to the pulp to help in activating the free silica and gum together with a commercial reagent known as Palcotan was added as a further depressent for the iron minerals. To the final conditioning stage approximately 2½ lbs. per ton of a semi-refined tall oil was added to the pulp as a collector for the free silica and the phosphate minerals. Methyl isobutyl carbinol was used as a frother in an open flotation circuit consisting of a rougher float followed by two cleaners. The metallurgical results were as follows:

The first rougher tailing was 49.92% by weight and analyzed 58.15% iron and 0.09% P_2O_5 as against an original head value of 0.816% P_2O_5. This illustrates the high differential float obtainable on the phosphate minerals with an effective degree of depression of the hematite in the ore.

Example 4

In this example the pH was originally adjusted to 11.6 with sodium hydroxide and conditioned with approximately 2 lbs. per ton of sodium silicate and 1 lb. per ton of calcium chloride. This was followed by a second stage of conditioning with gum and Palcotan. The third and prolonged stage of conditioning was with approximately 2½ lbs. per ton of semi-refined tall oil. An initial rougher

float was made with a single cleaner stage. The pH of the rougher tailings was then adjusted down to a pH of approximately 7.8 with sulfuric acid and an additional approximate 3 lbs. per ton of sodium silicate was added as a dispersant. The rougher tailing was 42 to 43% by weight analyzing 55.15% iron and 0.076% phosphorous. The original float after the single stage of cleaning was 13.59% by weight analyzing 23.71% iron and a calculated approximately 2% P_2O_5. The No. 2 float was 18.42% by weight analyzing 46.21% iron and 0.233% phosphorous.

This illustrates the high degree of differential float of the phosphorous minerals and the ability of the circuit once controlled dispersion is reached at a specific pH to activate and float the phosphate minerals at other than the initial conditioning pH range in Example 3.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. A process for the broth flotation of phosphate, carbonate and silica minerals in the presence of slimes while substantially suppressing metallic oxides comprising; preparing a suitable pulp of the ore to be treated; conditioning the pulp at about pH 11–12 in the presence of sodium silicate dispersing agent and a fatty acid type collecting agent; and floating the said minerals at a pH which is optimum for flotation of the mineral to be floated having the lowest optimum flotation pH range.

2. A flotation process as defined in claim 1 wherein the mineral to be floated is silica and the flotation is carried out at about pH 11–12.

3. A flotation process as defined in claim 1 wherein the minerals to be floated are phosphates, carbonates and silica, wherein, after said conditioning, the pH of the pulp is reduced to about 9.5 to 10.5 and the pulp is further conditioned with the addition of further of said dispersing agent before simultaneous flotation of phosphate, carbonate and silica minerals.

4. A flotation process as defined in claim 2 wherein, after flotation of the silica, the pH of the pulp is adjusted to about pH 9.5–10.5, and the pulp is further conditioned with the addition of further of said dispersing agent, following which carbonates and phosphates are floated.

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