SEPARATION OF MICA FROM CLAY BY FROTH FLOTATION OF CLAY

Inventor: David C. Yang, Edison, N.J.
Assignee: Engelhard Minerals & Chemicals Corporation, Township of Woodbridge, N.J.

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Field of Search ................................. 209/160, 167, 5

References Cited
UNITED STATES PATENTS
2,162,494 6/1939 Trotter .............................. 209/166
2,249,570 7/1941 Lane .............................. 209/166
2,303,962 12/1942 Tantaroff ......................... 209/166
2,759,962 8/1956 Zenftman .......................... 209/166 UX
2,857,051 10/1958 Noblitt .......................... 209/167
3,259,237 7/1966 Schueld ......................... 209/166 X
3,278,028 10/1966 Millsups ....................... 209/160
3,329,265 7/1967 Browning ....................... 209/166
3,462,013 8/1969 Mercade ......................... 209/166 X
3,480,143 11/1969 Mitzmagel ..................... 209/166
3,567,636 3/1971 Katzenstein ................... 252/32.5

FOREIGN PATENTS OR APPLICATIONS
1,044,733 11/1958 Germany .................... 209/166

Primary Examiner—Robert Halper
Attorney, Agent, or Firm—Melvin C. Flint; Inez L. Moselle

ABSTRACT
Mica, present as an impurity in kaolin clay, is removed from the clay particles by stage-wise froth flotation of the clay in a dispersed alkaline pulp, using as a collector for the clay a complex phosphate ester of a non-ionic surfactant of the ethylene oxide-adduct type.

3 Claims, No Drawings
1 SEPARATION OF MICA FROM CLAY BY FROTH FLOTATION OF CLAY

BACKGROUND OF THE INVENTION

High purity kaolin clay is widely used as a coating pigment for paper and paints. Such pigments must be free from undesired impurities, of which mica is an example. The presence of mica in more than trace quantity may render the clay unsatisfactory for use as a pigment.

Mica can be removed from clay with reasonable ease by gravimetric means or flotation when the clay is coarse. However, when the clay particles and micaceous impurities are fine, the separation is very difficult. Froth flotation with conventional ionic collectors is usually unsuccessful because mica and kaolin, both silicate minerals, have remarkably similar flotation properties.

THE INVENTION

An object of the invention is to provide a novel method for floating finely divided kaolin clay from a micaceous impurity.

A specific object is to provide a novel flotation method for floating kaolin clay from mica.

Briefly stated, in accordance with the present invention, a micaceous impurity, especially muscovite mica, is removed from kaolin clay by defloculating a pulp of the impure clay and subjecting the defloculated ore pulp to stage-wise froth flotation in an alkaline flotation circuit in the presence of a complex phosphate ester, or a salt thereof, of a nonionic surface active ethylene or propylene oxide adduct, producing a froth which is a concentrate of purified clay and a tailings which is a concentrate of mica. No activator for mica (e.g., an aluminum salt) is used. When an aluminum salt is employed in conjunction with the complex phosphate ester, as disclosed and claimed in my copending application, Ser. No. 266,159, filed June 26, 1972, the froth product will be a concentrate of mica.

PRIOR ART

Various combinations of anionic and cationic reagents have been suggested as flotation collectors for micaceous minerals. Reference is made to the following U.S. patent Nos. 3,214,018 to Neal; 3,278,028 to Milsap et al; 3,329,265 to Browning et al. U.S. Pat. No. 2,249,569 to Phelps suggests the flotation of kaolin clay from impurities, of which mica is mentioned as an example, by frothing a slip of impure clay with sulfite waste liquor.

DETAILED DESCRIPTION

In putting the invention into practice, the impure clay is initially blunged in water and coarse grit and indispersible agglomerates are removed in conventional manner, e.g., by sedimentation or screening. Preferably soda ash, in amount sufficient to adjust pH to a value within the range of 8 to 11 and partially disperse the clay, is added before degritting.

The deflocrated pulp of impure clay should be well-defloculated before flotation is carried out. The dispersant (defloculating agent) used may vary with the nature of the clay. In some cases soda ash, added before or after degritting, will suffice to provide a well-defloculated pulp suitable for flotation. In most cases, however, a more powerful dispersant will be required. The most satisfactory dispersant may vary with the nature of the clay. In many cases, sodium lignosulfonate will give best results. Other strong clay dispersants include sodium silicate and sodium condensed phosphates.

The phosphate esters of nonionic surface active agents which are used as collectors in carrying out the invention are described in U.S. Pat. No. 3,567,636 to Katzenstein. The pertinent disclosures in the Katzenstein patent as to the composition of such esters and to their preparation are incorporated herein by reference. Briefly, these are complex phosphate esters (or salt thereof) of a nonionic surface active compound, the phosphate being selected from the group consisting of monoesters, diesters and mixtures thereof, the nonionic surface active compound being the condensation product of an organic hydroxy compound of from eight to 50 carbon atoms selected from the group consisting of alkyl phenol and alkoxylates with at least 1 mole of an alkylene oxide having from two to three carbon atoms, the nonionic surface active compound containing a maximum of about 50 percent by weight of alkylene oxide based on the weight of the nonionic surface active compound.

The phosphate esters are commercially available under the trade name “GAFAC” and are anionic surfactants. The commercial products are usually mixtures of mono- and di-esters. The product “GAFAC RM 510” is understood to be a mixture of monoester and diester, principally diester. The monoester constituent may be described by the formula:

\[
\text{RO} \left(\text{CH}_2\text{CH}_2\text{O}\right)_n \text{HO} = \text{R} \left(\text{CH}_2\text{CH}_2\text{O}\right)_n \text{OH}
\]

wherein \( R = \text{alkylaryl} \) and \( n = 5 \) to 7.

The diester constituent has the formula:

\[
\text{RO} \left(\text{CH}_2\text{CH}_2\text{O}\right)_n \text{HO} = \text{R} \left(\text{CH}_2\text{CH}_2\text{O}\right)_n \text{OH}
\]

wherein \( R = \text{alkylaryl} \) and \( n = 5 \) to 7.

Obviously the ester can be used in salt form (e.g., sodium, ammonium, calcium, magnesium). In use the acid would be converted to alkali metal salt form in the basic clay pulp.

An essential feature of the method of the invention resides in the stage-wise addition of the phosphate ester collector. Total amount of ester collector generally is within the range of 1.5 to 5.0 lb./ton, usually 2.0 to 3.0 lb./ton. Normally, no more than 1.0 lb./ton is added during any stage. A typical amount to add during each stage is within the range of about 0.1 to 1.0, preferably 0.2 to 0.8 lb./ton.

The phosphate esters normally will not provide adequate frothing when added stage-wise in small amount. Thus, a small amount of a conventional frother such as...
3 methyl isobutyl carbinol or pine oil is normally added to develop adequate froth.

All of the frother may be added during the first stage or additional frother may be required in one or more subsequent stages.

During the first stage, the deflocculated pulp is conditioned with phosphate ester, preferably using prolonged conditioning, followed by frothing and froth removal. The flotation tailings are then conditioned with additional phosphate ester, again followed by frothing and froth removal. This is repeated one or more additional times.

Froth flotation may be carried out at solids within the range of about 5 percent to 50 percent, preferably 20 percent to 45 percent solids.

After flotation the clay in the froth product may be re-dispersed, fractionated in conventional manner to recover one or more fine size fractions of beneficiated clay. In many cases, the fine fractions will be lower in residual mica than the coarse fraction of the flotation purified clay.

The following example is given for illustrative purposes and is not to be considered as limiting upon the invention.

The clay used in the test was an impure kaolin from France. The principal impurity in the clay was muscovite. The degreased minus 325 mesh (Tyler) portion of the impure clay analyzed 1.97 percent K₂O, corresponding to 16.7 percent muscovite.

A sample of the crude clay (9,000 gm., dry basis) was blunged in deionized water at 30 percent solids for 15 minutes. Soda ash was then added in amount of 2.0 lb./ton, the pH was adjusted to 9.0 using caustic soda, and the slip was conditioned for 15 minutes. The slip was screened on a Sweco vibratory screen and the minus 325 mesh fraction of the slip was recovered. A portion of the minus 325 mesh slip was fractionated with a minus 5 micron cutoff and the minus 5 micron fraction was analyzed for K₂O content for purposes of comparison.

The degreased slip (minus 325 mesh fraction) was charged to a Fagergren conditioner. Sodium lignosulfonate was added in amount of 1.75 lb./ton, followed by conditioning for 6 minutes.

“GAFAC MO-510,” 0.5 lb./ton, was incorporated, followed by 16 minutes’ conditioning. The pulp was then conditioned for 1 minute with a drop of methyl isobutyl carbinol. pH was 9.0.

The pulp was transferred to a Denver flotation machine and froth was collected for 10 minutes. After removing froth from the first stage of flotation, an additional 0.5 lb./ton of “GAFAC” was added, followed by 2 minutes’ conditioning and reflation (second stage) for 10 minutes without further addition of frother. This was repeated a total of four times (total collector used was 2.0 lb./ton “GAFAC MO-510”).

The combined froths (“FP 1-4”) were combined and a portion was analyzed for K₂O to estimate mica content of the purified clay. “FP 1-4” was then re-dispersed and fractionated at 5 microns. The minus 5 micron cut was analyzed for K₂O. In similar manner the tailings product was analyzed for K₂O before and after fractionation to minus 5 microns. The results are summarized in table form along with K₂O analyses and estimated mica contents of the impure clay before and after fractionation.

Metallurgical results in the table show that stage-wise froth flotation using a phosphate ester complex as the collector for kaolin reduced the K₂O content of the minus 5 micron fraction of the clay from 1.53 percent, corresponding to 13.0 percent mica, to 1.14 percent K₂O, corresponding to 9.66 percent mica. Thus, about one-third of the mica was removed. Overall clay recovery was 41.4 percent, with an 18.9 percent recovery of a minus 5 micron fraction.

### TABLE

<table>
<thead>
<tr>
<th>Product</th>
<th>K₂O</th>
<th>Muscovite</th>
<th>Distribution of Mica, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control (no flotation)</td>
<td>1.97</td>
<td>16.7</td>
<td>100.0</td>
</tr>
<tr>
<td>-5 micron fraction</td>
<td>1.53</td>
<td>13.0</td>
<td>43.0</td>
</tr>
<tr>
<td>Froth Product</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FF 1-4 unfractionated</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>-5 micron fraction</td>
<td>1.14</td>
<td>9.66</td>
<td>18.9</td>
</tr>
<tr>
<td>Tailings</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>MD 1-4 unfractionated</td>
<td>2.43</td>
<td>20.6</td>
<td>58.7</td>
</tr>
<tr>
<td>-5 micron fraction</td>
<td>1.76</td>
<td>14.9</td>
<td>18.9</td>
</tr>
</tbody>
</table>

I claim:

1. In a process for removing a micaceous impurity from kaolin clay, the steps comprising dispersing an aqueous pulp containing kaolin clay and micaceous impurity with sodium lignosulfonate, conditioning the pulp with a complex phosphate ester, or salt thereof, of a nonionic alkylene oxide adduct as a collector for the kaolin clay, the pulp being free from an activator for the micaceous impurity, and subjecting the pulp to froth flotation at a pH in the range of 9 to 11, producing a froth which is a concentrate of purified clay and a tailings enriched in micaceous impurity, said phosphate ester collector being a nonionic surface active agent which is selected from the group consisting of monoester, diester and mixtures thereof, said nonionic alkylene oxide adduct being the condensation product of an organic hydroxy compound which contains from 8 to 50 carbon atoms and is selected from the group consisting of alkyl phenol and alkanols with at least 1 mole of an alkylene oxide containing from two to three carbon atoms, said nonionic alkylene oxide adduct containing a maximum of about 50 percent by weight of alkylene oxide.

2. The process of claim 1 wherein said tailings is re-conditioned with additional phosphate ester collector and subjected to froth flotation at least one more time, and combining the froth products to produce a purified clay product.

3. The process of claim 2 in which said phosphate ester is a mixture of monoester and diester, principally diester, wherein the monoester has the following formula:

$$RO(CH_2CH_2O)_nO$$

wherein R = oleyl; and n is 4 to 5; and the diester has the following formula:

$$RO(CH_2CH_2O)_mO$$

wherein R = oleyl; and n is 4 to 5.