1

3,477,809 KAOLIN TREATMENT

Wayne M. Bundy, Westfield, and Joseph P. Berberich, Rahway, N.J., assignors to Georgia Kaolin Company, Elizabeth, N.J. No Drawing. Filed Dec. 30, 1966, Ser. No. 606,034 Int. Cl. C01b 33/26

U.S. Cl. 23-110

5 Claims

## ABSTRACT OF THE DISCLOSURE

A method is provided for improving the whiteness, brightness and printability of kaolinite by the steps of forming a slurry of degritted crude clay, bleaching the clay with hydrosulfite bleach, attrition grinding the 15 bleached clay, fractionating the ground clay to recover a fraction having more than 90% below  $2\mu$  spherical dimeter, redispersing and selectively flocculating the recovered fraction at an alkaline pH, recovering the flocculated portion, treating the recovered flocculated por-  $^{20}$ tion with a peroxide bleaching agent at an elevated temperature, and drying the peroxide bleached fraction at an elevated temperature to provide a kaolinite product.

This invention relates to kaolin treatment and particularly to a method of treating kaolinite to provide improved whiteness and printability for paper coatings. Large quantities of kaolinite are used for coating papers for printing to provide high gloss and whiteness along 30 with a superior surface for printing. It is well known in the trade that the secondary kaolinites of Georgia, which are the principal source of such material in the United States, have a generally lower whiteness index than the primary kaolinites of England. The Georgia clays are generally contaminated with a buff or yellow color resulting from iron and titanium impurities, as well as others, deposited with the kaolinite at the time of formation. Bleaching of kaolinites has been practiced for many years with limited success as have also certain floatation techniques designed to remove the titanium impurities. In general the methods used have been of limited utility or excessively expensive and complex.

We have discovered a process of treating clay which improves both the whiteness and printability to a degree equal to any and superior to most prior art techniques and by steps which are simpler and less expensive.

We have found that crude clay which has been degritted and bleached can be improved in both whiteness and printability by subjecting it to attrition grinding in a dispersed phase, fractionation to recover a fraction containing about 90% below two microns equivalent spherical diameter, redispersing the recovered fraction, selectively flocculating the redispersed fraction at an alkaline pH, recovering the flocculated fraction, treating the recovered flocculated fraction with a peroxide bleaching agent at elevated temperature and drying at elevated temperature to break down any remaining flocculate. Preferably, we selectively flocculate by the practice described in Rowland Patent No. 2,981,630. The preferred selective flocculant is a polyacrylamide such as Nalco 670. The peroxide bleaching agent is preferably hydrogen peroxide used in aqueous solution at a treating temperature of about 60° to 70° C. The resulting clay is finally dried at a temperature of about 90° C., although this temperature may vary somewhat depending upon the specific selective

2

flocculating agent used. Preferably the grinding is carried out with Ceramedia.

The invention may perhaps be better understood by reference to the following examples.

#### EXAMPLE I

A crude kaolinite from the Sandersville, Ga., area was blunged in water with sodium hexametaphosphate (about 2% by weight) and degritted in the usual manner. A sample of the degritted kaolinite was bleached with sodium hydrosulfite after acidification with H<sub>2</sub>SO<sub>4</sub> to a pH of about 3 to 3.5, filtered and washed. The bleached clay was then redispersed with sodium hexametaphosphate (about 1% by weight) and ground in a sand grinder. Following grinding the clay was fractioned by centrifuge to recover a fraction between CPS .09 and CPS .11 at 10 minutes. The recovered fraction was again dispersed with sodium hexametaphosphate (1% by weight) and the pH raised to about pH 8 with ammonia and diluted to about 10% solids. To the slurry was added, with strong agitation, 10 ml. of a 0.25% solution of Nalco 670 per 100 mg, of clay. The precipitated or flocculated fraction was recovered, heated to 60° C. and hydrogen peroxide added (4% by weight of a 3% solution). The thus treated material was dried at 90° C. The resulting material had a whiteness index of 11 as compared with 16 for a like clay fractionated bleached, and produced by usual procedures. This is comparable to good English primary clay.

#### EXAMPLE II

In a pilot plant operation a Georgia kaolinite was blunged with 4.5 lbs. per ton of a mixture of sodium hexametaphosphate and soda ash (50-50) and degritted. A batch of 3.4 tons of this degritted kaolinite was heated to 140° F., bleached with 10 lbs./ton of zinc hydrosulfite and iron free alum to a pH of 3.5 and allowed to stand for one hour. The kaolinite was rinsed with water and redispersed with 0.3% of sodium hexametaphosphate. The dispersed clay was then ground on an attrition grinder at 132 r.p.m. and a feed rate of 10 g.p.m. The feed was at a specific gravity of 1.164 and CPS of 1/.10, 3/.17, 10/.25 and 15/.27. The product has CPS values of 1/.10, 3/.15, 10/.22 and 15/.25. The product was then fractionated by centrifuging on a Bird centrifuge to recover a product of 90% below 2µ average spherical diameter. The recovered fraction was dispersed with 1% sodium hexametaphosphate and the pH raised to 8.3 with 10% ammonia and then diluted to 10% solids after which it was selectively flocculated with Nalco 670. The flocculated portion was recovered, slurried in water, flocculated to pH 3 with sulfuric acid, heated to 140° F., and treated with about 4% of a 3% solution of hydrogen peroxide. The resulting precipitate was recovered on a De Laval centrifuge and washed. The material was again heated to 140° F., treated with 2 lbs./ton zinc hydrosulfite and alum to a pH of 3.3, recovered on a De Laval centrifuge and dried at 195° F. The brightness of the resulting kaolinite was 89.9 and the whiteness index 11. The same kaolinite bleached but without the added treatment of this invention showed a whiteness index of only 16.

In the foregoing specification we have set out certain preferred practices and embodiments of our invention. It will be understood that this invention may, however, be otherwise practiced and embodied within the scope of the following claims.

We claim:

1. The method of providing a kaolinite of improved whiteness, brightness and printability comprising the steps of

(a) forming a slurry of degritted crude clay,

- (b) bleaching the clay with a hydrosulfite bleach,
- (c) subjecting the bleached clay to attrition grinding,
   (d) fractionating the ground clay to recover a fraction having more than 90% below 2μ spherical
- diameter,

  (e) redispersing and selectively flocculating the recovered fraction at an alkaline pH,

(f) recovering the flocculated portion,

- (g) treating the recovered flocculated portion with a peroxide bleaching agent at an elevated tempera- 15 ture, and
- (h) drying the peroxide bleached fraction at an elevated temperature.
- 2. The method as claimed in claim 1 wherein the peroxide bleached clay is further treated with a hydrosulfite bleaching agent at an acid pH prior to drying.

3. The method as claimed in claim 1 wherein the se-

4

lective flocculation is carried out by the addition of a polyacrylamide.

4. The method as claimed in claim 1 wherein the peroxide bleaching agent is hydrogen peroxide.

5. The method as claimed in claim 1 wherein the attrition grinding is carried out in a sand grinder.

## References Cited

# UNITED STATES PATENTS

1,588,956	6/1926	Feldenheimer 23—110
2,524,816	10/1950	Lyons 23—110
2,955,051	10/1960	Maloney 23—110 X
2,981,630	4/1961	Rowland 23—110 X

## OTHER REFERENCES

Mitchell et al.: Soil Science, vol. 77 (1954), pp. 173-183.

## EDWARD J. MEROS, Primary Examiner

U.S. Cl. X. R.

106---72