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Removal of the Discoloring Contaminants of an East Georgia Kaolin Clay and its Dewatering

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Abstract

The present study deals with flotation, selective flocculation, leaching and dewatering tests conducted on East Georgia kaolin clay. Flotation and selective flocculation tests were performed on the samples to remove titanium impurities (TiO_2) using different reagents at pH 9.5. The leaching tests were applied to the treated samples to dissolve the iron impurities (Fe_2O_3) in the presence of sodium hydrosulfite ($Na_2S_2O_4$) and alum ($Al_2(SO_4)_3.18H_2O$) at pH 3.0. The test results showed that the brightness of the samples improved from approximately 82% to 91% . After that, the leached samples were conditioned with dodecylamine, and then a newly developed dewatering aid was added to the slurry to make the fine clay particles super hydrophobic. It was found that the moisture content of the clay sample decreased from 42.3% to 31.4% . As a result, it can be concluded that the high quality and low moisture content of the kaolin clay can be easily used for many industries.

Key words: Kaolin clay, Titanium and iron impurities, Flotation, Coagulation, Selective flocculation, Leaching, Surface hydrophobicity and moisture content.

Introduction

Kaolin clay is an aluminum hydrous silicate ($Al_2O_3.2SiO_2.2H_2O$) used in many industrial applications due to its unique physical, physiochemical and chemical properties (Yıldırım, 2001). These include paper, paint, rubber, ceramic, glass, refractory, agriculture, waste treatment, the cosmetic applications and for nanocomposites as coating, pigment, acid/base regulator and filler materials. The kaolin samples known as a platelets minerals, originally exhibit white color with high clay brightness, but it mainly contains various amount, of discoloring elements, such as anatase (TiO_2), mica and iron oxides (Fe_2O_3), which give low brightness, and are detrimental in final use (Basilio, 1997). In addition, the anatase (titaniferrous) and mica can also contain iron contaminants (Milledgeville *et al.*, 1997).

Recently, several research programs have been conducted on clay samples to determine the surface properties and interfacial interactions of kaolin clay. Geise *et al.* (1996) and Wu (2001) developed a thin layer wicking method to measure the advancing contact angle of the powder without using a microcalorimeter or Goniometer, and determined the hydrophobicity/hydrophilicity and acid/base characteristics of the material. Using this information, one can provide some information on the flotation, flocculation, coagulation, dispersion, wetting and adsorption characteristics of the clay samples. For example, if water molecules wet the surface of a solid, this surface is called a hydrophilic surface, while if not, it is called a hydrophobic surface. Kaolin clay is usually hydrophilic, and has negative zeta potential values. Some of the properties of a kaolin clay are given in Table 1 (Yıldırım, 2001; Wu, 2001).

The USA (mainly Georgia and North Carolina) is the world's kaolin clay producer with more than 9.5 million tons of production worth some $ 1 billion annually (Basilio, 1997; Milledgeville *et al.*, 1997). Many recent studies in the USA have focused on the treatment of fine the kaolin clay and its dewatering. Because of the size of kaolin particle and its platelet structure, the beneficiation and dewatering of the material are the main interests of many plants. In order to improve the quality of kaolin clay for industry, the discoloring impurities must be removed from the samples by suitable techniques. However, it is also known that the impurities are finer than the clay minerals, which, in turn, presents difficulties in the separation processes. These separations generally include magnetic separation, froth flotation, selective flocculation, size separation by hydrocyclone, and leaching (Basilio, 1997; Yoon and Shi, 1986). Finally, the dewatering is conducted on the processed sample by vacuum or pressure filter, and then high moisture content filter cake is spray dried to the desired moisture levels (less than 3%). The physical and chemical properties of the run-of-mine (ROM) Middle and East Georgia clay samples are given in Table 2 (Yordan *et al.*, 1994; Luz *et al.*, 2000).

Table 1. Properties of a Kaolin Clay.

Units	Values
Density (g/cm^3)	2.6
Luster	Dull
Surface Energy (mN/m)	45
Zeta Potential (mV)	-47
Tot. Interaction Energy (kT)	10200
Water Contact Angle (°)	44
Loss on Ignition (%)	13

In kaolin clay processing, the first step is to prepare the sample before the separation and dewatering processes. The ROM clay sample is usually blunged for approximately 10 min in a baffled container with a solid content of 35-65% at over 6000 rpm. To disperse the particles, generally sodium silicate or soda ash can be used in the blunger. This process is critically important to separate the fine clay and gangue minerals (less than 10 μm) in the separation steps. The blunged clay slip is then conditioned in the presence of collectors for another 10 min before the discoloring impurities (anatase) are removed at an appropriate pH value (Yordan *et al.*, 1994).

In order to solve the problem associated with the procesing of fine kaolin clay, flotation, selective flocculation, leaching and dewatering tests were applied East Georgia clay samples. The first three tests were carried out using classical treatment procedures. For dewatering tests, a new chemical was developed from a natural product, and successfully tested on the dewatering of the treated fine kaolin clay. This dewatering aid is code named Reagent TX101, which is a low Hydrophile-Lipophile Balance (HLB) nonionic surfactant (i.e., HLB numbers are less than 10). In the this article, the treatment and dewatering test results obtained with the clay samples will be discussed in view of brightness, yield (weight recovery) and moisture content values.

Processing of Kaolin Clay

High gradient magnetic separation

Kaolin clay consists of several magnetic discoloring impurities, such as anatase, hematite, pyrite, mica and rutile with the magnetic susceptibilities of approximately $10^{-6}/\mathrm{cm}^{-3}$ (Yoon and Shi, 1986). Conventional and superconducting magnetic separations are typically employed units with 1-2 Tesla and 2-5 Tesla magnetic fields, respectively. The prepared clay sample with a 15% -25% solid content is fed through a high gradient magnetic field created around the ferromagnetic stainless steel wool fiber. The magnetic separations are generally operated as a batch unit with 10-20 min cycles depending on the size of the units, feed rate, solid content, impurity content, applied filed, machine capacity, etc.

Table 2. Physical and Chemical Characteristics of ROM Georgia Kaolin Clays.

Locations	Finer than 2 μm (%)	TiO_2 (%)	Fe_2O_3 (%)	Color
M. Georgia (A)	55.8	1.72	0.28	Tan
M. Georgia (B)	55.8	1.56	0.31	Yellow
M. Georgia (C)	49.9	1.29	0.82	Red
East Georgia	90.0	2.35	0.70	Tan

Literature studies showed that the brightness of Brazilian kaolin clay was increased from 87% to 90% using superconducting high gradient magnetic separation 5 Tesla magnetic field (D'Assumpcao, 1995). The tests were conducted at 25.2% solid content after the samples were sized in a centrifuge (i.e., 90% is finer than 2 μm). It was also determined that high magnetic separators were effective for the nanosize discoloring elements to produce high brightness clay. Therefore, other processes (i.e., flotation and/or selective flocculation) can be incorporated to increase the brightness of kaolin clay (Basilio, 1997; Khalek *et al.*, 1996).

Flotation

The flotation of the anatase (titaniferrous) in fine kaolin clay is a difficult task in the clay industry. The reason may be that the bubbles generated in conventional flotation cells are too large to capture the ultrafine particles, which are usually less than 2 μm (Basilio, 1997). Therefore, treatment time of flotation is longer (between 25 min and 100 min) than other separation methods, such as magnetic field and flocculation. However, froth flotation has been used to remove anatase minerals from the kaolin clay due to the better efficiency (high yield and low impurity content) of the method compared to the other separation techniques (Luz *et al.*, 2000; Khalek *et al.*, 1996).

In the early flotation process (Yoon and Shi, 1986), the anatase minerals were floated using 3 to 4 kg/ton of tall oil or fatty acids as collectors at pH 9-10 after they were activated by divalent cations (i.e., Ca^{2+} and Pb^{2+}). It was determined that approximately 100-150 g/ton of calcite carrier (caustic soda) could activate the anatase minerals in the kaolin clay. In the early stage of flotation, the kaolin is dispersed using 3.5 kg/ton of sodium silicate, 3.5 kg/ton of ammonium hydroxide for the saponification of the collector and pH regulator and 1.5 kg/ton of petroleum sulfonate (Basilio, 1997).

In the recent flotation process (Yoon *et al.*, 1992; Yordan *et al.*, 1994), it was found that hydroxamates (i.e., S6973, Aero 6493 and Aero 6973) were excellent collectors at alkaline pHs (i.e., best at 9.5) for the removal of anatase minerals from the kaolin clay. It was reported that 0.5 to 1.5 kg/ton of the hydroxamate was sufficient for the removal of the titaniferrous minerals at approximately 20% solid content. It was also reported that the kaolin clay samples could be better dispersed at alkaline pHs compared to acidic pHs (Yoon *et al.*, 1992). In addition, no activations by the cations are necessary for the hydroxamate flotation before the flotation tests. The other advantages are that the yields and brightness of the hydroxamate flotation product are higher than that of the fatty acid flotation product. This may be attributed to the fact that the hydroxamate reagents can render the anatase minerals more hydrophobic and improve the selectivity and recovery of the clay samples (Luz *et al.*, 2000; Yordan *et al.*, 1994).

Selective Flocculation

It was determined that the submicron anatase minerals can be effectively removed from the clay sample using a selective flocculation technique (Basilio, 1997). In this process, the dispersed clay slip is diluted to approximately 10% to 20% solid content by adding tap water, and then introduced to polyacrylamide type anionic or nonionic flocculants are added These include the trade names Sharpefloc 9131, NALCO 8872, NALCO 9825, Percol 730 or Percol 90L, which are commercially available. When the kaolin clay is flocculated with the polymer, the heavy flocs of impurities quickly settle at the bottom of the cup, and then the suspended clay is removed by either gravitational or centrifugal forces. Finally, the processed kaolin clay is dewatered to achieve marketable products (Milledgeville *et al.*, 1997; Luz *et al.*, 2000).

Recently, Luz *et al.* (2000) performed a set of flocculation tests on Brazilian kaolin clay using 125 g/ton NALCO 8872 flocculants at 12.5% solid content of pH 9.5. In the tests, a 500 g sample of bone-dry kaolin clay was blunged in a conditioner for 10 min at 50% solid content adding 3.75 kg/ton of sodium silicate, 3.75 kg/ton of sodium hexametaphosphate and 0.5 kg/ton of ammonium hydroxide. The tests were conducted in a 1 L graduated cylinder. The flocculants was poured into the cylinder, and it was hand shaken for a minute. The test results showed that the TiO_2 content of the sample was reduced from 1.9% to 0.8% with a 56% yield (weight recovery). However, it was also pointed out that the yield of the flocculation was lower than that of flotation, which could be attributed to fine clay particle entrapment in the settled flocs (i.e., mostly impurities).

Leaching

Due to the size of the anatase and iron contaminants in kaolin clays, magnetic separation, flotation and selective flocculation methods cannot be as efficient as a leaching method for achieving high brightness (+90) products for pigment and filler materials (Basilio, 1997). To be able to improve the brightness of kaolin clay, the sample is usually leached in the presence of sodium hydrosulfite, zinc hydrosulfite, sodium hypochlorite or hydrogen peroxide and alum at pH 2.5 to 3.5 (sulfuric acid) and 400 rpm agitation to remove the iron impurities. As is known, titanium compounds are very difficult to dissolve by acid leaching and mainly iron contaminants dissolved in this process (Yoon and Shi, 1986).

In the recent applications (Veglion *et al.*, 1998; Yoon *et al.*, 1992), the leaching is conducted on the pretreated sample obtained from the separation methods (flotation, flocculation, magnetic separation, hydrocyclone, etc). For example, the brightness of a kaolin clay treated using 350 g/ton of hydroxamate was increased from 87.3% to 90.6% at 1.5 kg/ton of $Na_2S_2O_4$, 2.5 pH and 20% solid content after 30 min of leaching time. It was also determined that when the temperature of the pulp was increased from ambient to 90°C, more iron could be removed at higher leaching kinetics.

Dewatering

The dewatering of kaolin clay is the most difficult task in preparation plants due to the surface area, structure and hydrophilicity of the material (Rushton *et al.*, 2000; San and Hosten, 1996; Svarovsky, 1990). The clean kaolin clay treated with magnetic separation, flotation, selective flocculation and/or leaching methods usually contains more than 80% moisture content. Rotary drum or hyperbaric pressure filters are commonly employed dewatering units to improve the solid content of the feed sample, which is approximately 35% to 55% by weight. Because of the size of the clay sample, it is mostly coagulated by an electrolyte (300 to 600 g/ton of Alum or NaCl), and then a flocculant (100 to 300 g/ton of NALCO 9765 or Superfloc 84) is added to the slurry before the filtration processes. From 60 kPa to 150 kPa of pressure is applied to the filter media to remove the free water from the filter cake. In addition, if the clay sample is intended for use in cosmetic, paper and agricultural industries, the filter cake is usually washed by clean spray water to decrease the harmful effects of the chemicals used during the clay processing. Finally, the filter cake is thermally spray dried to achieve less than 3% of finished product depending on the market requirements (Bassilio, 1997; Stanley *et al.*, 1988).

Experimental

Sample

A ROM kaolin clay sample (90% is lower than 2μm) obtained from East Georgia was subjected to a series of flotation, selective flocculation, leaching and dewatering tests. Two buckets of the sample were mixed and homogenized in the laboratory, and then 500 g of bone-dry samples were representatively divided and stored until required. The assay results obtained using an XRF spectrometer showed that the kaolin clay contained 2.18% TiO_2 and 0.92% Fe_2O_3.

Methods

Initially prepared kaolin clay samples were dispersed in a kitchen blender at 6200 rpm and 40% solid content using 3.5 kg/ton of sodium silicate and 3.5 kg/ton of hexametaphosphate at pH 9.5 (using ammonium hydroxide) for 10 min. After the dispersion, the clay slip was diluted to 13% solid content by adding tap water. A known amount of collectors was added to a 4 L standard Denver D-12 flotation cell and conditioned at 1600 rpm for another 10 min. The flotation time was 45 min to remove mostly anatase minerals from the kaolin clay in the presence of a hydroxamate type collector (Aero 6973).

In the selective flocculation, the dispersed clay sample (13% solid content) was conditioned with 0.5 kg/ton of hydroxamate (Aero 6973), and then transferred to a 1 L graduated cylinder 6 cm in diameter and 36 cm in length. The aim of the hydroxamate addition was to make the anatase particles hydrophobic enough to selectively adsorb the anionic flocculants (NALCO 8872) on these minerals. Afterwards, the reagents were added to the cylinder, and then a couple min of hand shaking was given for flocculant adsorption on the anatase particles. The flocs were then allowed to settle at a rate of 3 min per 1 cm of slurry depth, which was about 100 min for each test. It was assumed that most of the flocs were anatase and they were rarely iron oxide minerals. Finally, the unsettled clay sample was siphoned by means of a rubber tube and used for the next steps.

The samples received from the flotation and selective flocculation methods were separately leached in the presence of sodium hydrosulfite and alum at pH 3.0 and 400 rpm agitation to remove the iron contaminants at room temperature. In the final procedure, the leached clay samples were dewatered in an air pressure filter at 100 kPa pressure using a newly developed non-ionic dewatering aid (Reagent TX 101). The dewatering aid was dissolved in light oil (diesel) or alcohol (butanol) at a 1:2 ratio before use. Details of the air pressure filter are given elsewhere (Asmatulu, 2002). After the treatment and dewatering processes, the brightness of the clay sample was determined using a Technical Association of Pulp and Paper Industry (TAPPI) standard brightness technique (Basilio, 1997).

Results and Discussion

Treatment Tests

The dispersed kaolin clay was subjected to flotation tests at pH 9.5 using 0.75 kg/ton of hydroxamate (Aero 6973) as a collector and 120 g/ton of polypropylene glycol (PPG) as a frother. The role of the hydroxamate was to improve the hydrophobicity of the anatase minerals for the flotation process. The results showed that after 45 min of flotation time, the titanium content was decreased from 2.18% to 1.27% TiO_2 with a yield (weight recovery) of 72.6% (see Table 3) and, hence, increased the GE brightness (used for pigment and filler brightness in the USA) from 82.2% to 87.3% . Experimental results also showed that further increases in hydroxamate dosage did not change the titanium content; however, the clay yield (weight) was gradually reduced, which may be due to the fact that the higher dosages of hydroxamate can float the clay minerals.

For the next treatment tests, the same dispersed clay slip was also utilized in the selective flocculation experiments in the presence of 120 g/ton of NALCO 8872 type flocculant. The test results, given in Table 3, showed that the titanium content of the clay sample was reduced from 2.18% to 0.93% TiO_2 content with a clay yield of 57.2% and that brightness improved up to 88.5% . It was seen that the titanium content of the flocculated product was less than that of the floated product; in contrast, the yield of the former method is approximately 16% lower. This is because the fine clay particles are entrapped in the settled flocs of the impurities, and overall clay yield was decreased.

The leaching tests were conducted on the precleaned samples using 2.5 kg/ton of sodium hydrosulfite and 0.5 kg/ton of alum at pH 3.0 and 30 min of leaching time. Diluted sulfuric acid was used to adjust the pH values. The test results approved that the brightness of the flotation and selective flocculation samples were improved respectively from 87.3% and 88.5% to 90.2% to 91.3% with $Na_2S_2O_4$ addition. The reason might be that the $Na_2S_2O_4$ addition dissolved the iron contaminants at a lower pH, and then this contaminant was removed during the filtration as effluent. Table 3 shows the leaching results of the flotation and selective flocculation products. It is seen that all test results are in excellent agreement with results in the literature (Basilio, 1997; Shi, 1986; Yoon *et al.*, 1992). As a result, this high quality clay product can be used for coating, filler, acid-base regulator and pigment purposes in many industrial applications.

Dewatering Tests

It is known that kaolin clay is the most difficult material to dewater in preparation plants. Vacuum or high pressure filters are the only dewatering methods before the thermal dryers, which is a spray drier performed in a natural gas flame. It is reported that the overall dewatering cost is higher with these procedures to obtain a high solid content material for the market (Yoon and Shi, 1986; Rushton *et al.*, 2000).

Table 3. Test Results of Flotation, Selective Flocculation and Leaching Tests Conducted on the East Georgina Kaolin Clay Sample.

Separation Methods	TiO_2 (%)	Clay Yield (%)	GE Brightness (%)	
			0 kg/ton $Na_2S_2O_4$	2.5 kg/ton $Na_2S_2O_4$
Flocculation Product	0.93	57.2	88.5	91.3
Flotation Product	1.27	72.6	87.3	90.2
Feed	2.18	100.0	82.2	-

Table 4. Effect of Reagent TX101 on the Dewatering of the East Georgia Kaolin Clay (-2 micron) at 100 kPa Air Pressure

Reagent Dosage (kg/ton)	Selective Flocculation Product		Flotation Product	
	Moisture Content (%)	Cake Formation Time (min)	Moisture Content (%) Time	Cake Formation (min)
0	42.3	19.41	42.0	19.24
3	39.6	18.57	38.0	18.28
6	37.5	17.02	36.3	16.45
9	35.2	16.34	32.7	15.09
15	33.1	15.44	31.4	14.51

In the present study, a series of filtration tests were conducted on East Georgia kaolin clay samples, which were separately treated using flotation and selective flocculation methods before the leaching tests. The clean leached samples were conditioned with 1000 g/ton of dodecylamine at pH 3.0 to make the surface of the clay sample slightly hydrophobic. This is called a first hydrophobization step for hydrophilic clay particles. The treated products were then subjected to pressure filter tests at 100 kPa air pressure and 2.5 mm cake thickness in the presence of Reagent TX 101, called a second hydrophobization step. In this step, it was assumed that close packed monolayer formation with the surfactant molecules occurred on the surface of the particles to make the particles super hydrophobic. The conditioning time of the dewatering was 5 min for each test. The test results obtained at 3 min of cake dewatering time are given in Table 4.

Base line cake moistures of the flocculation and flotation products were 42.3% and 42.0% , and the cake formation times were 19.41 min and 19.24 min when no dewatering aid was used. At 15 kg/ton TX101 addition, the moisture contents of selective flocculation and flotation products were reduced to 33.1% and 31.4% with 15.44 min and 14.51 min of cake formation time, respectively. As seen in Table 4, the flotation product gave approximately 1% to 2% lower moisture content than the selective flocculation product. This may be attributed to the fact that in the selective flocculation tests, the surface of the clay particles was contaminated by the flocculants, and increased the moisture content of the filter cake. More importantly, in the presence of the dewatering aids more than a 9% moisture difference was achieved, which may be due to the hydrophobicity improvement of the clay particles.

Conclusions

A series of flotation and selective flocculation tests were conducted on kaolin clay samples (2 μm x 0) from the East Georgia using appropriate reagents at pH 9.5 in order to remove the titanium and iron minerals. After the sample treatment, the clean clay products were leached with $Na_2S_2O_4$ and alum at pH 3.0 to decrease the iron bearing contaminants. Titanium and iron minerals are the main discoloring impurities in kaolin clay. The test results showed that after the treatment of samples, more than 91% brightness (GE brightness for pigment and filler materials) kaolin product could be achieved with 72.6% clay yields (weight recovery).

The dewatering aid used in the present studies works well with hydrophobic particles. To be able to improve the hydrophobicity of the clean kaolin clay samples obtained from treatment tests (flotation, selective flotation and leaching), the samples were first hydrophobized by dodecyl amine at pH 3.0. After that, a series of filtration tests conducted on the hydrophobized clay samples using Reagent TX 101 as a dewatering aid at 100 kPa air pressure and 2.5 mm cake thickness. The tests results confirmed that the novel dewatering aid could significantly decrease the moisture content of kaolin clay. In addition, the experimental results also showed that the kinetics of mechanical dewatering were gradually improved with the addition of dewatering aid, which would greatly increase the throughput of dewatering devices in plants.

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