# Beneficiation and Evaluation of Mutaka kaolin

John Baptist Kirabira<sup>1</sup>, Stefan Jonsson<sup>2</sup>, Joseph K. Byaruhanga<sup>3</sup>

<sup>1</sup>Department of Mechanical Engineering, Faculty of Technology, Makerere University, P.O. Box 7062, Kampala, Uganda Corresponding author email: jbkirabira@tech.mak.ac.ug <sup>2</sup>Department of Materials Science and Engineering, Royal Institute of Technology (KTH), Brinellvägen 23, SE-100 44 Stockholm, Sweden

# ABSTRACT

Kaolin is an important industrial mineral which often needs upgrading for special applications. The upgrading process, commonly known as beneficiation, is aimed at removing impurities like feldspar, quartz, iron oxide, etc. In this study, a kaolin sample was collected from a deposit located at Mutaka, South-Western Uganda, – the biggest kaolin deposit in the country. With the aim of upgrading this sample, it was processed by a laboratory hydro cyclone to produce a kaolinite concentrate and this has been characterized to ascertain its use as an industrial raw material. Characterization of the beneficiated sample was carried out by XRD, SEM, particle-size distribution, density and volume measurements, chemical analysis, whiteness index test, thermal analysis (DTA/TG) and surface area measurements. Results show that the laboratory beneficiation exercise improves the kaolin sample to a very high grade with a chemical composition close to that of ideal kaolinite. The major impurity after beneficiation, iron oxide, was further reduced by acid leaching. Results show that the properties of the final product are close to that of ideal kaolin.

Keywords: Beneficiation, Kaolin, Mutaka kaolin, Particle size distribution.

## **1.0 INTRODUCTION**

Kaolin is a soft, white, plastic clay consisting principally of kaolinite, which is a well ordered hydrated aluminium silicate  $Al_2Si_2O_5(OH)_4$ . It is formed by the alteration of granitoid rocks, mainly feldspar and muscovite. Naturally, kaolin occurs with impurities, which have to be removed for most of its commercial application, particularly in the paper filling and coating applications. The two principal objectives of kaolin beneficiation are the removal of impurities and production of a desired particle size distribution. Mineralogically and chemically, kaolins contain quartz as the major impurity and iron (in form of Fe<sub>2</sub>O<sub>3</sub>) and titania (in form of anatase) as minor impurities, particularly iron and titania bearing minerals, imparts colour to kaolin (low brightness) and are detrimental in most kaolin applications.

Basically, two different processes are used in beneficiating of kaolin – a dry process and a wet process. The dry process is the simpler and less expensive of the two but yields a lower quality product. In the dry process, kaolin is crushed, dried, pulverized and air floated. The air floating process removes most of the coarse particles from the fine ones, but in order to really obtain a high quality product, the wet process must be used. Consequently, the latter process was used in the present work.

### 2.0 MATERIALS AND METHODS

### 2.1 Sample Preparation and Examination

Mutaka is situated approximately on latitude 9922000N and longitude 171000E within sheet 85/1, Bushenyi, USD series. Approximately 4 tonnes of raw kaolin was collected from Mutaka

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south-western Uganda. A representative sample weighing 130 kg was used for the beneficiation process. Visual examination of the raw sample revealed the presence of fragments of quartz, muscovite, weathered iron oxide and clay. The lot was screened at 2.36 mm in order to remove the larger particles. Thus all further investigations were performed on the remaining size fraction below 2.36 mm weighing approximately 100 kg. Using riffles and following ISO/CD 14488 procedures, a uniformly distributed representative sample of 0.6 kg was split from the big sample. The small sample was used for determining the particle size distribution by sieve analysis. The big sample was beneficiated, as described below.

#### 2.2 Size classification

A combination of wet screening and hydro cycloning was used. The 100 kg sample with size fractions less than 2.36 mm was attrition scrubbed to make it ready for the hydro cyclone test rig. The fraction was fed into a receiving cone of a laboratory 2-inch stub hydro cyclone test rig and water was added to make a pulp of 16% solids. The pulp was left to run for ten minutes to allow for scrubbing action on the material before collection of the cyclone products. The pressure was set to 0.21 MPa (30 Psi) and the spigot was 6.3 mm. The vortex finder was set to 11.1 mm. The under size from the screening operation was fed into a hydro classifier (rougher-stage). From its overflow, a product rich in kaolin, was collected and fed to another cleaner hydro classifier. There it was further processed in order to obtain a cleaner product. The underflow fraction normally consists of sand and other heavy mineral impurities but also contains an appreciable amount of kaolin. In order to recover this kaolin it was fed into a scavenger which completes the kaolin extraction from the waste. The extracted kaolin from the scavenger was fed into the rougher hydro classifier stage together with the underflow fraction from the cleaner stage.

#### 2.3 Removal of Iron Oxide

According to chemical analysis results, the beneficiated kaolin contained iron oxide as the main impurity. In order to explore the possibility of reducing the iron oxide content, about 200 g were leached with oxalic acid of concentration of 40 kgm<sup>-3</sup>. The mixture was maintained at 80°C for 5h while stirring. It was then filtered and dried at 110°C.

#### 2.4 Characterization of the beneficiated sample

The chemical composition was determined by Analytica AB, Luleå, Sweden according to the standard analysis package G-2. The powders were first dried at 105°C according to Swedish Standard SS 028113. Then, 0.125 g dried powder was smelted with 0.375 g LiBO<sub>2</sub> and dissolved in 5% HNO<sub>3</sub> before the metal content was determined using Induced Coupled Plasma-Atomic Emission Spectrometry (ICP-AES). The loss on ignition was determined by heating to 1000°C for two hours. For mineralogical properties, a randomly oriented powder-mount was scanned over the range 12 to 60° 20 using a Philips X-ray Diffractometer, PW 1130/90, with Ni-filtered  $K_{\alpha}$  Curadiation, operated at 40 kV and 30 mA. An energy dispersive detector of type Kevex was used. The collimator and receiving slits were set to 1 and 0.1°, respectively. The sample morphology was observed by a Field Electron Gun Scanning Electron Microscope (FEG-SEM), LEO 1530 with a GEMINI column. A thin layer of carbon was coated on to the powders in order to enhance conductivity and avoid charging when exposed to the electron beam. Particle size distribution was determined using a BI-90 particle sizer. Portions from the powder samples were ultrasonically mixed with water for 2 minutes in order to crush agglomerates. A small amount of a surfactant, Dispex A40, was added to keep the particles in suspension. 2 ml of the suspension was filled in a transparent container and analyzed for particle size distribution. The multipoint Brunauer, Emmett and Teller (BET) technique was used to measure the total surface area with a Micromeritics Gemini 2370 system. The gas adsorptions at multipoint partial pressures of nitrogen gas were determined. The density determination was carried out on a Pycnometer, Accu Pyc 1330. The samples were first weighed and dried in the sample holder at 120°C for 30

minutes. Then, the samples were weighed again and the volumes were measured by pumping in He gas to a pressure of 2 bar. For each sample, five cycles were taken and an average was automatically calculated by the Pycnometer.

Thermoanalytical characterization was done using a NETZSCH STA 449C Jupiter<sup>©</sup> DTA/TG calorimeter in a temperature interval of 20 - 1620°C in static air.

Whiteness index tests were carried out in accordance with SCAN-P 89:03 using a Lweropho reflectometer at STFI-Packforsk AB, Stockholm, Sweden. According to the standard, the investigated powders were milled, dried and pressed into tablets. For each sample five tablets were prepared and characterized. The average values were reported.

# **3. RESULTS AND DISCUSSIONS**

#### 3.1 Chemical analysis

The chemical composition of raw, of beneficiated and of beneficiated and leached samples as determined by ICP-AES is summarized in Table 1. The major oxides detected are the components of the kaolinite formula i.e.  $Al_2O_3$ ,  $SiO_2$  and  $H_2O$  (LoI). Only small amounts of other oxides, predominantly  $Fe_2O_3$  and  $K_2O$  were detected. It can be seen that the beneficiation process improves the quality of the raw kaolin to almost pure kaolin. The beneficiated sample has a deficit of only 0.30%  $Al_2O_3$ , 1.35%  $SiO_2$  and 0.25%  $H_2O$ . The sum of the main impurities,  $K_2O$ ,  $Fe_2O_3$  and CaO is limited to 1.3%, only. It is unfortunate that the composition of  $Fe_2O_3$  increases from 0.238 to 0.417% but still this is a low value. Typically, kaolin with less than 1%  $Fe_2O_3$  is considered to be a good quality for a variety of applications. Thus, most important is the increase of the  $Al_2O_3$  content from 36.0 to 39.2% and the  $SiO_2$  decrease from 48.8 to 45.2%. These changes bring the beneficiated product very close to pure kaolinite. The reason for increasing iron oxide during beneficiation may be associated to the impurity bearing compounds that are part of the fine fraction of the beneficiated sample and cannot be removed mechanically. Consequently, acid leaching was attempted for reducing iron oxide from the beneficiated kaolin. The chemical analysis confirms a substantial reduction as is shown in Table1.

Table 1: Chemical composition in wt % of raw, of beneficiated and of beneficiated and acid
leached Mutaka kaolin compared to pure, ideal kaolin.

Oxide	Raw	Benefi-	Beneficiated	Ideal
		ciated	and acid	
			leached	
SiO <sub>2</sub>	48.800	45.200	47.600	46.550
$Al_2O_3$	36.000	39.200	39.100	39.500
CaO	< 0.090	0.135	< 0.100	-
Fe <sub>2</sub> O <sub>3</sub>	0.238	0.417	0.149	-
MgO	0.038	0.059	0.051	-
K <sub>2</sub> O	1.140	0.760	0.821	-
MnO <sub>2</sub>	0.028	0.012	0.011	-
Na <sub>2</sub> O	0.048	< 0.040	0.058	-
TiO <sub>2</sub>	0.004	0.012	0.016	-
P <sub>2</sub> O <sub>5</sub>	0.009	0.022	0.023	-
LoI [wt %]	12.600	13.700	13.500	13.950

## 3.2 Mineralogical analysis

Figure 1 shows the XRD patterns of the raw and beneficiated kaolin samples. The patterns reveal that the two samples are essentially dominated by kaolinite, with the strong peaks at  $2\theta = 12.68^{\circ}$ , d=6.98 Å; 25.1°, d=3.54 Å; 38.7°, d=2.33 Å. These patterns are typical for kaolin minerals, as shown by previous investigators on kaolins (Ekosse, 2000; Murray and Keller, 1993).



Figure 1: XRD scans of raw and beneficiated Mutaka kaolin. For clarity, the latter curve is vertically displaced by 4000 units.

#### 3.3 Microstructure of the kaolin concentrate

The morphologies of the raw and beneficiated samples from SEM investigations are displayed in **Error! Reference source not found.**-

Figure 3, respectively. As seen, the raw Mutaka kaolin shows a highly layered platy-structure of thin lamellae packed in larger aggregates. This layering is typical of many types of primary kaolin (Murray and Keller, 1993). The micrograph also reveals that the structure of Mutaka kaolin includes loosely agglomerated minute crystals, of rather consistent size and pseudo hexagonal shape. Apparently, the beneficiated kaolin sample displays similar lamellae compared to the raw kaolin. This shows that the beneficiation process essentially leaves the lamellar structure impervious. Instead, separation occurs on a larger length scale.



Figure 2: SEM image (SE) of raw Mutaka kaolin

Figure 3: SEM image (SE) of beneficiated Mutaka kaolin

#### 3.4 Sieve analysis and particle size distribution using BI-90

The accumulated weights passing through a series of sieves  $(2360 - 20 \ \mu m)$  are fitted to an Avrami equation i.e.

$$V_{acc} = 1 - \exp(-Kd^n) \tag{1}$$

where  $V_{acc}$  denotes accumulated weights and *d*, the sieve diameter. *K* and *n* are parameters used to fit the experimental information.

Figure 4 shows Avrami plots of the sieve analyses of the hydro cyclone products of Mutaka kaolin. The plots show good fitting to the experimental information with correlation factors of  $R^2 > 0.98$ . The intersection with the dashed line indicates the equivalent diameter for 90% volume fraction,  $d_{90}$ . At that diameter, 90% of the volume fraction has smaller particle diameter. The equivalent diameters are given in

Table 2. The sieve analyses show that the raw kaolin has the largest particle fraction followed by the intermediate hydro-cyclone products and finally the beneficiated kaolin. The beneficiation changes the  $d_{90}$  from 1157 to 48 µm. The slope of the beneficiated kaolin is somewhat less than for the raw kaolin, indicating that the size distribution has essentially moved to finer particles with a small widening in the distribution curve, only.





Figure 4: Avrami plot of sieve analysis of raw and hydrocyclone products of Mutaka kaolin.

Figure 5: Avrami plot of particle size distribution (BI-90) of raw and beneficiated Mutaka kaolin.

The BI-90 analyses show that the beneficiation process has little effect on the suspended particle distribution. A small increase in  $d_{90}$  from 1148 to 1329 nm has occurred. The slope is somewhat less indicating that the size distribution has widened moderately.

Property	Raw	OF-HC1	UF-HC2	OF-HC2 /benef.
Density, $(g \cdot cm^{-3})$	2.643	-	-	2.828
Sieve analysis $d_{90}$ , (µm)	1157	496	120	48
BI-90, <i>d</i> <sub>90</sub> , (nm)	1148	-	-	1329
Spec. surf. Area $(m^2g^{-1})$	16.498	-	-	14.330

**Table 2:** Density, equivalent diameters,  $d_{90}$ , and specific surface area

## 3.5 Specific surface area

The specific surface area measured by BET and taking a multipoint surface is 14.33  $m^2g^{-1}$  for the beneficiated sample, as compared to 16.498  $m^2g^{-1}$  for the raw kaolin sample. See

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Table 2. The decrease is difficult to explain since the morphology on the fine scale is quite similar. See **Error! Reference source not found.** and Figure 3.

# 3.6 Density

The density of the beneficiated kaolin is 2.828 gcm<sup>-3</sup> as compared to 2.643 gcm<sup>-3</sup> for the raw kaolin, see Table 2. This weight increase is most probably a direct result from the increase in  $Al_2O_3$  content accompanied by the simultaneous decrease in  $SiO_2$  content during beneficiation.

## 3.7 Thermal analytical analysis

Figure 6 reveals the DTA and TG curves for the beneficiated kaolin sample. In the current study, one endothermic peak is observed at around 530°C of the DTA curve, and an exothermic one at 1003°C. These results corroborate well with previous researchers investigating transformation of kaolin during heating (Kirabira, et al, 2004; Chen and Tuan, 2001; Chen, *et al*, 2000 (a); Chen, *et al*, 2000 (b); Chin-Yi and Wei-Hsing, 2002; Chandrasekhar and Ramaswamy 2002; Kirabira, *et al*, 2003; Yung-Feng *et al*, 2004; Pask and Tomsia, 1991; Chandrasekhar and S. Ramaswamy, 2006). The TG curve, shows a weight loss of about 13.0%, which corresponds to the endothermic peak at 530°C. The weight loss in the TG analysis is close to the corresponding LoI (13.7%) of the beneficiated sample, see Table 1.



Figure 6: DTA/TG signals of beneficiated Mutaka kaolin sample during heating

# **3.9 Whiteness Index**

The results from the whiteness index test are given in Table 3. As seen, the beneficiation reduces the brightness which confirms the increase in iron oxide reported by chemical analyses. However, acid leaching almost fully restores the brightness.

Sample powder	ISO brightness (R 457)	Y-value
Raw	84.53	89.44
Beneficiated	69.95	79.83
Beneficiated and acid leached	81.37	85.02

Table 3: Whiteness Index of Mutaka kaolin powders

#### **4.0 CONCLUSIONS**

Mutaka kaolin is a kaolinite-rich mineral that has muscovite and other feldsparthic materials as the major impurities. In the present work, a successful beneficiation of Mutaka kaolin was achieved through a mechanical process of particle separation based on a wet classification method. The iron bearing impurity, Fe<sub>2</sub>O<sub>3</sub>, seems to concentrate in the fine fraction of the kaolin mineral. The chemical composition of the kaolin has improved with the alumina content increasing from 36.0% to 39.2% and with that of silica decreasing from 48.8% to 45.2%. The beneficiation of the kaolin improves it to a suitable grade for filler material, especially in paints,

pharmaceuticals, ceramic porcelain and related traditional and engineering ceramics. It is demonstrated that acid leaching can improve the quality of the product further. However, for refractory applications, there is no need to beneficiate this kaolin because it can be used in its raw form and upgrading it further would only add an unnecessarily big production cost.

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STFI-Packforsk AB, Box 5604, SE-114 86 Stockholm, Sweden.

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