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Effects of Beneficiation on the Characteristics of Alkaleri Kaolin

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ABSTRACT

Effect of beneficiation on characteristics of a local clay Alkaleri village in Bauch State, Nigeria is presented. The raw clay was beneficiated by wet beneficiation method. Mineralogical characterization of the clay was carried out using X-ray diffraction (XRD) machine. Physicochemical characterization of the clay was carried out using X-ray fluorescence (XRF) machine, pH meter and specific gravity analyzer. Morphological characterization of the clay was conducted using scanning electron microscopic (SEM) machine. The qualitative XRD analysis showed presence of kaolinite peaks at Bragg's angle values of 12.4°, 20.5°, 24.9°, 35.1°, 55.4° and 62.3° and presence of quartz peaks at Bragg's angle values of 26.6° and 38.5°. The XRD analysis further showed that the raw clay possessed 84% kaolinite and 16% quartz impurity. The XRF result of raw Alkaleri clay showed that the clay contained 53 wt% silica and 42.4 wt% alumina making the silica-alumina ratio of the 1.25. Other metallic oxides present in the clay as impurity were iron oxide, titanium oxide, magnesium oxide, potassium oxide, zirconium oxide, sulfur oxide and cesium oxide, present at 0.9, 2.2, 0.7, 04, 0.2, 0.1 and 0.1 wt%, respectively. The specific gravity values of the raw and beneficiated clay were 2.18 and 2.16, respectively. The average pH values of the raw and beneficiated were 4.9. and 6.0, respectively. The SEM results revealed that Alkaleri clay possessed dispersed lump-like morphology with tetrahedral or hexagonal imperfect crystal shapes and the average particle size of the clay was estimated to be 500 µm.

Keywords: Alkaleri clay; Kaolinite; XRD; SEM; pH; Specific gravity

1. INTRODUCTION

Clay is a naturally occurring aluminosilicate mineral formed as a product of weathering of rocks, especially igneous, granite and feldspathic rocks [1]. Clay minerals are chemically inert as they are hardly reactive to most chemicals. Clay minerals possess platelet morphology owing to the sheets of alumina and silica which form the secondary building units (SBUs) of clay structure [2]. They are commonly defined by geologists as hydrous layer aluminosilicates with a particle size range of 1–2 µm [3]. Clay minerals are formed in various categories each having a distinctive mineral and crystal

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structural pattern. These categories include kaolinite, montmorillonite, illite, vermiculite and chlorite. Each classification of clay possesses a set of unique structural and physical properties which differentiate it from other members of the clay family. Clay possess small size and large surface area to volume ratio and this attribute of clay is responsible for most of the impressing properties which make clay suitable for wide range of industrial applications. These properties include plasticity, chemical and temperature resistance, malleability, and complex composite formulations [4, 5].

Kaolin, also called China clay, is a highly demanded raw material used in production of variety of industrial products which include housewares, building materials ceramics, porcelain, paint, paper, white incandescent light bulbs, energy storage device, skincare products etc [5, 6]. The kaolinite group is chemically represented as $Al_2Si_2O_5(OH)_4$. Structurally it possesses one silica and one alumina unit stacked in alternating fashion known as 1:1 lattice type [7]. Owing to its relatively large particles and low specific surface, kaolinite exhibits less plasticity, cohesion and swelling as compared to other clay minerals. Nigeria has been reported to have billions of tonnes of kaolin [8, 9]. In Bauchi state where Alkaleri clay was mined it has been reported that 18 million tonnes of kaolin reserves are obtainable across different deposits which include Alkaleri, Damban, Darazo, Ganjuwa, Kirfi, Misau and Ningi [9]. This huge kaolin reserves can only be harnessed for greater economic benefits if their characteristics are studied. The aim of this work was to investigate the effects of beneficiation on the mineralogical and physicochemical characteristics of Alkaleri kaolin.

2. MATERIALS AND METHODS

2.1 Materials and Equipment

Raw Alkaleri clay sample was mined from Alkeleri village in Alkeleri Local Government Area of Bauchi State - Nigeria. The GPS coordinate of the deposit is 10°16.662’ N, 10°18.405’ E. Equipment used include, pH Meter (Model; 3510 pH meter), XRD machine (Model; BRUKER S2 RANGER) and SEM machine (Phenom-world Einhoven; Model-Phenom ProX). Apparatus used include weighting balance, glassware and density bottle.

2.2 Methods

2.2.1 Beneficiation

Wet beneficiation of Getso clay was carried out as presented in our previous work [10]. The raw clay was crushed and ground. The ground clay was soaked overnight in water in a predetermined ratio of 0.1 g/L. The mixture was plunged by stirred for 3 h then left to settle for 1 h. The supernatant water was decanted and the sedimented clay was sieved using mesh size of #200. The fine clay filtrate known as the beneficiated clay was dewatered, dried, ground and weighted. The residue impurity was collected, dried and weighted.

2.2.2 X-ray diffraction analysis

The clay material was pulverized and homogenized. Wafer of the powdered sample was prepared using the sample preparation block. The clay was compressed in the flat sample holder to create a flat wafer that was later mounted on the sample stage in the XRD cabinet. The sample was analyzed using the reflection-transmission spinner stage at Theta-Theta settings. XRD scan was carried out at 2θ range of 4°-75° using 2θ step of 0.026261°

at 8.67 s/step. Tube current was set at 40 mA and the tension was 45 VA.

2.2.3 Scanning Electron Microscopy

The scanning electron microscopy (SEM) was carried out using Phenom ProX Desktop SEM. The sample was placed on double adhesive which was on a sample stub, it was coated with 5 nm of gold using sputter coater (Quorum technologies model: Q150R). The coated sample was placed in the sample chamber of the SEM machine and the SEM gun is focused on a selected area of the sample at certain magnification. Then the electron gun shot a beam of high energy electrons on the focused area to generate a SEM micrograph of the sample.

2.2.4 Physicochemical analysis

2.2.4.1 X-ray fluorescence analysis

The clay material was pulverized and homogenized. Wafer of the powdered sample was prepared using the sample preparation block. The clay was compressed in the flat sample holder to create a flat wafer that was mounted on the sample stage in the XRF cabinet for the determination of the metallic oxides compositions.

2.2.4.2 Specific gravity analysis

Specific gravity (SG) analysis was carried out using density bottle. Using a weighting balance, weight of empty density bottle was measured as W_1. Weight of the bottle plus clay filled to the bottle mark was measured as W_2. Weight of the bottle plus clay plus water was measured as W_3. Weight of the bottle plus only water filled to the bottle mark was measured as W_4. Equation (1) was used to determine the specific gravity of the clay sample [10,11].

$$SG = \frac{(W_2 - W_1)}{(W_4 - W_1) - (W_3 - W_2)} \tag{1}$$

2.2.4.3 pH analysis

pH analysis was carried out by preparing different samples of clay-water mixtures using 10 g constant weight of clay and varying weight of water so as to make 1:2, 1:4,1:6, 1:8 and 1:10; wt%-wt% of clay- to- water in each case, respectively [12]. At each experimental run three readings of pH value of the mixture were taken and the average reading was considered for the pH analysis result.

3. RESULTS AND DISCUSSION

3.1 Beneficiation Results

From the 1500 g raw clay beneficiated, the residue obtained after beneficiation was 108.3 g. This implies that the residue obtained was 7.2% of the raw clay beneficiated. The residue impurity is expected to be quartz [10]. This result suggests that the beneficiation process had reduced the quartz impurity in Alkalari clay by 7.2%.

3.2 X-ray Diffraction Analysis

Figure 1 shows the qualitative XRD patterns of raw Alkaleri clay. It could be observed that the mineral phases present in the clay are kaolinite and quartz. Kaolinite which could be observed as the dominant mineral phase was identified at Bragg's angle values of 12.4°, 20.5°, 24.9°, 35.1°, 55.4° and 62.3° [13, 14] and each corresponded to intensity reading of 1314, 569, 1749, 602, 577, 816 counts, respectively. The quartz phase present were identified at Bragg's angle values of 26.6° and 38.5° which correspond to intensity reading of 545 and 863 counts, respectively. Figure 2 shows the quantitative XRD analysis of Alkaleri clay. It could be observed that the kaolinite content of the clay was 84% and the quartz impurity composition was 16%. This further validate the dominance of kaolinite already observed in the analysis of the qualitative XRD analysis.

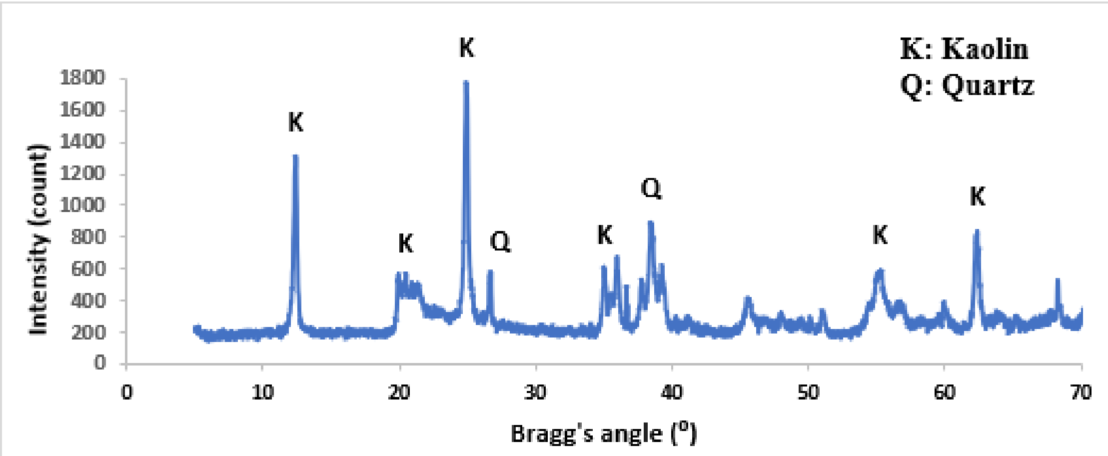

Figure 1 Qualitative XRD diffractogram of raw Alkaleri clay

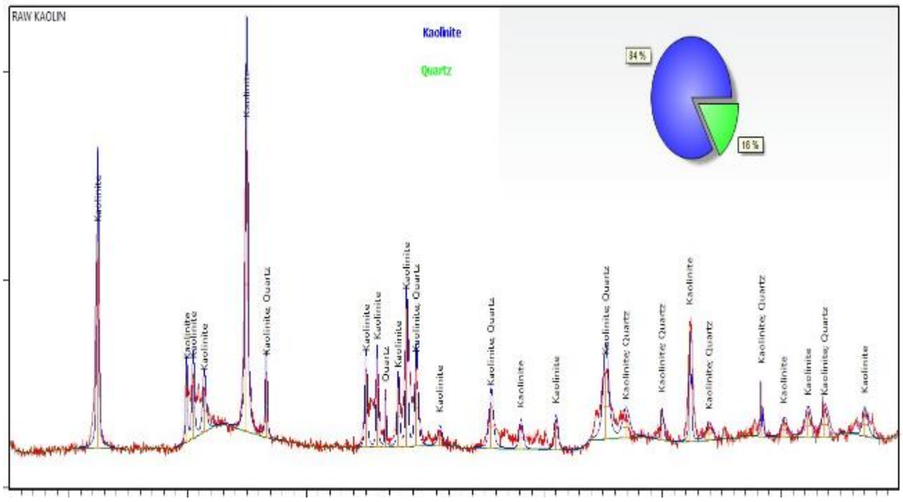

Figure 2 Quantitative XRD diffractogram of raw Alkaleri clay

Figure 3 shows the qualitative XRD patterns of the beneficiated Alkaleri kaolin. The intensity of the kaolinite peaks at 12.47°, 20.5°, 25.0°, 35.1°, 55.4° and 62.3° were 1235, 676, 1849, 731, 647 and 1014 counts respectively. The intensity of quartz peaks at 26.7° and 38.6° were 1402 and 987 counts respectively [13]. The quantitative XRD results shown in Figure 4 indicates that the kaolinite content of the beneficiated clay was 93% and the quartz content was 7%. Comparison of this result with the quantitative XRD of the raw clay has clearly shown that the beneficiation process resulted into reduction of the quartz impurity by 9% and improvement of the kaolinite content by the same amount.

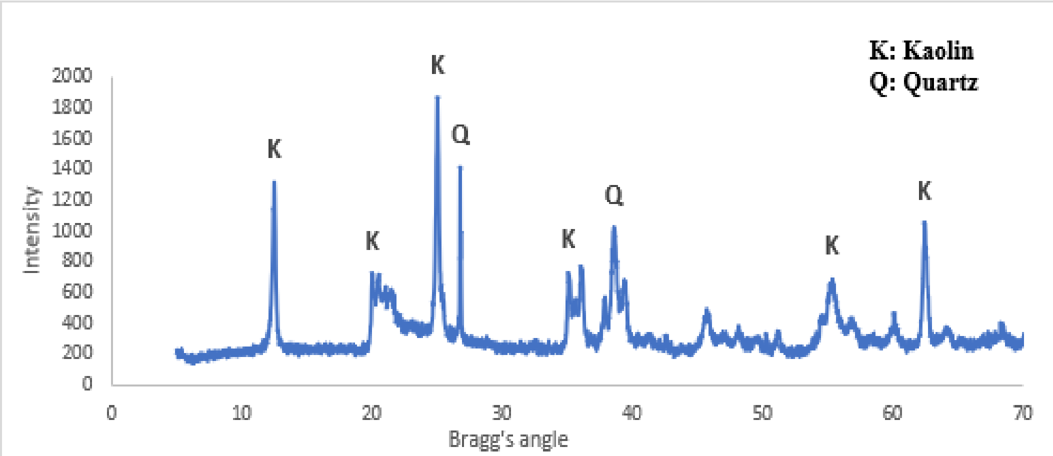

Figure 3 Qualitative XRD diffractogram of beneficiated Alkaleri clay

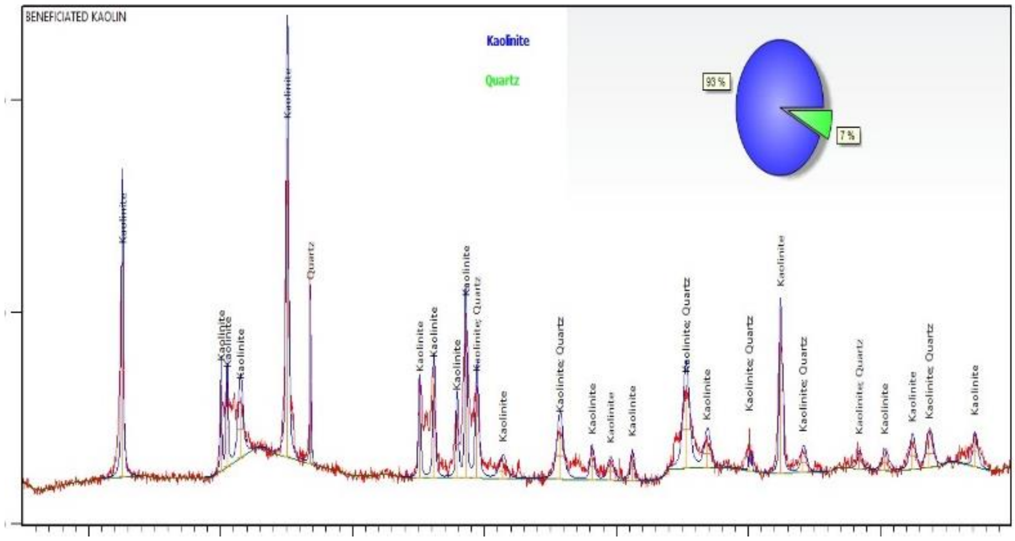

Figure 4 Quantitative XRD diffractogram of beneficiated Alkaleri clay

3.3 Scanning Electron Microscopy

Figures 5(A), 5(B) and 5(C) show the SEM morphological micrograph of raw Alkaleri clay at 500x, 1000x and 1500x magnifications respectively. It could be observed that the raw clay possessed dispersed lump-like morphology with tetrahedral or hexagonal imperfect crystal shapes. The average particle size was estimated to be 500 μm.

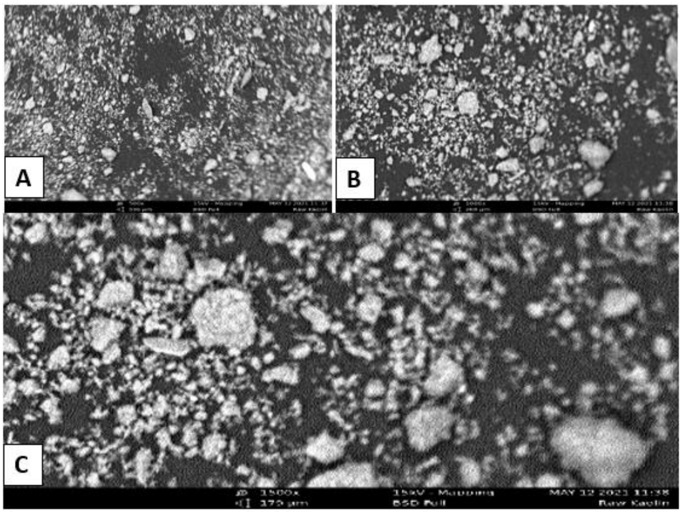

Figure 5 SEM image of raw kaolin at; (A) 500x magnification (B); 1000x; magnification (C); 1500x magnification

Figures 6(A), 6(B) and 6(C) show the SEM morphological micrograph of beneficiated Alkaleri clay at 500x, 1000x and 1500x magnifications respectively. It could be observed that the beneficiated clay retained a dispersed lump-like morphology with tetrahedral or hexagonal imperfect crystal shapes. Estimation of the average particle size remained 500 µm.

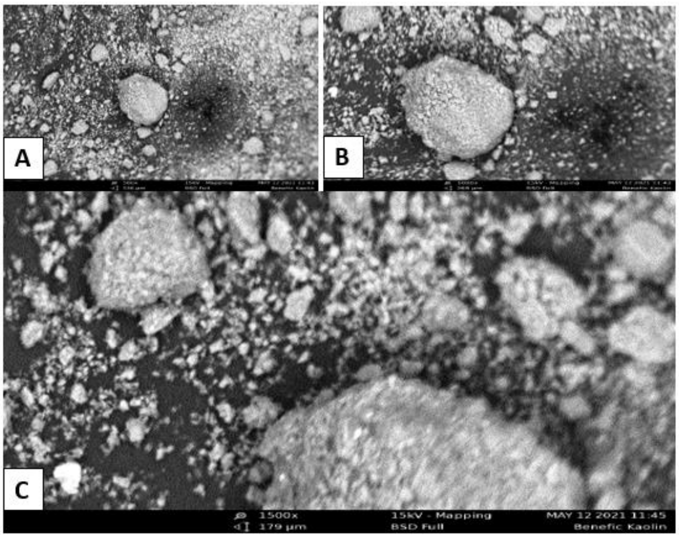

Figure 6 SEM image of beneficiated kaolin at;
(A) 500x magnification (B); 1000x; magnification (C); 1500x magnification

3.4 Physicochemical Characterization

3.4.1 X-ray fluorescence analysis

Table 1 presents XRF results of the raw Alkaleri clay. Being aluminosilicate mineral, the clay was majorly composed of alumina and silica. Silica was the highest metallic oxide constituent of the clay measuring up to 53 wt%. The alumina content of the clay was 42.4 wt%. The silica-alumina ratio of the clay was 1.25, this value falls within the general range of silica-alumina ratio for kaolin clay

[1, 12]. Compared to a very low silica-alumina ratio of 1.05 reported [15] for Alkaleri clay, 1.25 ratio determined in this study confirmed presence of substantial amount of kaolinite in Alkaleri clay [13]. This value also agrees closely with other reports on kaolin [16]. The metallic oxide impurities in Alkaleri clay were iron oxide, titanium oxide, magnesium oxide, potassium oxide, zirconium oxide, sulfur oxide and cesium oxide, they were present at 0.9, 2.2, 0.7, 04, 0.2, 0.1 and 0.1 wt%, respectively.

Table 1 XRF chemical compositions of raw Alkaleri clay

Metallic Oxide (wt%)	Raw Alkaleri Clay
Al_2O_3	42.4
SiO_2	53.0
Fe_2O_3	0.9
TiO_2	2.2
MgO	0.7
K_2O	0.4
ZrO_2	0.2
SO_3	0.1
CeO_2	0.1

3.4.2 Specific gravity analysis

Table 2 presents the specific gravity analysis of the raw and beneficiated samples of Alkalari clay. Applying Equation (1) the specific gravities of the raw and the beneficiated clay samples were 2.18 and 2.16, respectively. These values fall within specific gravity range reported for kaolin by other researchers [11].

Table 2 Specific gravity analysis of Alkalari clay

Weight (g)	Raw Clay	Beneficiated Clay
W_1	26.8	26.8
W_2	70.4	71.1
W_3	99.8	100
W_4	76.2	76.2
Specific gravity	2.18	2.16

3.4.3 pH analysis

Table 3 presents the pH analysis of Alkalari clay using clay-water mixture at varying mixture ratios. The raw clay was slightly acidic having average pH value of 4.9. The 1:2 sample had pH of 5.1 and the pH value of the clay-water mixture decreased marginally as the water content of the mixture increased. The 1:10 sample had the least pH value of 4.7. The slightly acidic pH of the clay confirmed that it contains substantial kaolinite and this further suggests that the clay originated from weathering of igneous rock [17]. The clay also contains soluble acidic salt which may be sulphate salt as the XRF result indicated presence of sulphate in the raw clay. The beneficiated clay was less acidic and the average pH was approaching neutrality having value of 6.0. The trend of variation in value of pH with increase in water content of the clay-water mixture was similar to what was observed in the raw clay. It could be inferred that the beneficiated clay was relatively neutral due to washing off of the soluble acidic salt impurity during the beneficiation process.

Table 3 pH analysis of the raw and beneficiated samples of Alkalari clay

Clay to water ratio	pH of Raw Clay	pH of Beneficiated Clay
1:2	5.1	6.7
1:4	4.9	5.9
1:6	4.8	5.8
1:8	4.8	5.7
1:10	4.7	5.7
Specific gravity	4.9	6.0

4. CONCLUSION

Alkaleri clay was mined from its deposit in Bauch State, Nigeria. The raw clay was beneficiated by wet beneficiation method in order to purify the clay and make it suitable for industrial applications. Physically the beneficiation process resulted into 92% recovery of the clay and 7.2% impurity collected as residue of the beneficiation process. Mineralogical analysis of the raw clay carried out using qualitative XRD analysis showed presence of kaolinite peaks at Bragg's angle values of 12.4°, 20.5°, 24.9°, 35.1°, 55.4° and 62.3° and presence of quartz peaks at Bragg's angle values of 26.6° and 38.5°. The quantitative XRD analysis further showed that raw clay possessed 84% kaolinite and 16% quartz impurity. Beneficiation process resulted into improvement of the kaolinite content of the clay by 9% making the kaolinite content 93% after beneficiation and reducing the quartz content to 7%. Morphological characterization of both the raw and beneficiated Alkaleri clay have shown that the clay possessed dispersed lump-like morphology with tetrahedral or hexagonal imperfect crystal shapes and the average particle size of the clay was estimated to be 500 μm. Chemical analysis of Alkaleri clay revealed that the clay contained 53 wt% silica and 42.4 wt% alumina making the silica-alumina ratio of the 1.25. Other metallic oxides present in the clay as impurity were iron oxide, titanium oxide, magnesium oxide, potassium oxide, zirconium oxide, sulfur oxide and cesium oxide, present at 0.9, 2.2, 0.7, 04, 0.2, 0.1 and 0.1 wt%, respectively. Other physicochemical characterization of the clay revealed that the raw clay had 2.18 specific gravity which reduced marginally to 2.16 after beneficiation. The average pH of the raw clay was slightly acidic at 4.9. The average pH approached neutrality at 6.0 after beneficiation process. The pH values further confirmed presence of kaolinite and suggested that the clay originated from igneous weathering. Therefore, it could be concluded that Alkaleri clay is a kaolinite clay having some quartz impurity. The wet beneficiation process had improved the kaolinite purity of the clay substantially, reduced its quartz impurity substantially but only improved the physicochemical properties of the clay marginally.

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