EFFECT OF BENEFICIATION ON THE CHARACTERIZATION OF GETSO KAOLIN

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Abstract: *In their raw forms, clay minerals are found with a number of inherent impurities which make them unsuitable for most industrial applications. In order to overcome this problem and add value to clay minerals, beneficiation process is an indispensable solution. This study investigates effect of wet beneficiation process on the characteristics of a local clay mined from Getso village of Kano State, Nigeria. Mineralogical characterization of the clay was carried out using X-ray diffraction (XRD) analyzer. Chemical characterization of the clay was carried out using X-ray fluorescence analyzer. Physicochemical characterization of the clay was carried out using pH meter and density analysis conducted using density bottle. XRD analysis of Getso clay showed that the raw clay had 8 wt% kaolinite and 51% quartz. Wet beneficiation resulted into 53% improvement of the kaolinite content and 47% reduction of quartz impurity. The XRF analysis has shown that silica-alumina ratio of the raw Getso clay was 1.55 and this reduced to 1.49 after beneficiation. The physicochemical characterization of the clay has shown that Getso clay is neutral, the raw clay and beneficiated clay had average pH values of 7.5 and 7.3, respectively. Specific gravity values of the raw and beneficiated clay were 2.24 and 2.04, respectively. The beneficiation process had been effective as substantial increase in kaolinite content was observed and a reasonable decrease in the impurity contents was observed from the raw to the beneficiated clay. The Garnet content was completely reduced to zero while quartz, clinocllore and orthoclase were reduced by 24%, 9% and 13% respectively. The clay obtained after the beneficiation be serve as good raw material for production of whitewares, high grade ceramics in synthesis of zeolitic materials.*

1. Introduction

Clay is a layered structures of fined-grained minerals which occur naturally [1]. Clay material has particle size of about 1 μm [2]. Clay is a fine textured material produced by the weathering process of granite and feldspathic rocks [3,4]. The small particle size and huge surface to volume ratio properties of clay impact on them excellent properties such as high cation exchange capacities, catalytic properties, and plastic behavior needed for their industrial applications [5]. Clay is formed in various classifications each having a distinctive mineral and crystal structural pattern. Each classification of clay possesses a set of unique structural and physical properties which differentiate it from other members of the clay family. They various classifications include; kaolinite, montmorillonite, illite, vermiculite and chlorite. Clay is known for its long use by human for production of ceramic materials as far back as the early stage of human civilization [6]. Modern applications of clay include ceramics application in producing products such as whitewares, high temperature porcelains, sanitary ware and electrical insulators [7].

Kaolinite has been the major class of clay used in the production of ceramic and whiteware porcelain. Kaolin also called china clay is a high demanded raw material used in production of variety of industrial products which include housewares, building materials ceramics, porcelain, paint, paper, white incandescent light bulbs, skincare products etc [8]. In addition, kaolin I also applied in pharmaceutical, and composite

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materials industries [9]. Kaolinite group is chemically represented as $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$. Structurally it possesses one silica and one alumina unit molecules stacked in alternating fashion known as 1:1 lattice type [10]. The choice of kaolin for industrial applications is largely dependent on its purity [11]. Nigeria has been reported to have huge reserves of kaolin mineral which can be harnessed for great economic benefits [12].

This work is aimed at investigating effect of beneficiation on the characteristics of Getso kaolin with the objective of improving the suitability of the clay for industrial applications.

2. Materials and methods

2.1. Materials and Equipment

The raw clay was mined from Getso village in Gwarzo Local Government Area, Kano State, Nigeria. The deposit's GPS coordinate was $11^\circ 53' \text{N}$ north and longitude $7^\circ 58' \text{E}$. Equipment used include, pH Meter (Model; 3510 pH meter), XRD machine (Model; BRUKER S2 RANGER). 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 [13]. 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 clay-water mixture was plunged by stirred for 3 h then left to settle. After letting, 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. XRD analysis

The clay sample was pulverized, homogenized and a wafer of the clay was made. The clay wafer was 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 $4^\circ - 75^\circ$ 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. X-ray fluorescence analysis

The clay sample was pulverized, homogenized and a wafer of the clay was made. The clay wafer was mounted on the sample stage in the XRF cabinet for the determination of the metallic oxides compositions.

2.2.4. Physicochemical analysis

Specific gravity 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 . The specific gravity of the clay sample was determined using Equation (1) [14].

$$\text{Specific gravity} = \frac{(W_2 - W_1)}{(W_4 - W_1) - (W_3 - W_2)} \quad (1)$$

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 1:1, 1:2, 1:3 and 1:4; wt%-wt% of clay-to-water in each case, respectively. 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

Starting with 1500 g raw clay sample for the beneficiation, 1027 g was recovered as the beneficiated clay, implying 68.46% recovery for the beneficiation process. The amount of residue obtained was 327g, implying 21.8% removal of impurity. The remaining 9.7% was loss likely due to soluble impurities, suspended impurities such as organic matters and debris in the raw clay. The residue was the dense mineral impurity which was likely to be quartz.

3.2. XRD Analysis

Figure 1 shows the qualitative XRD patterns of the raw Getso clay. It could be observed that the mineral phases present in the raw clay are kaolinite, quartz, orthoclase, clinocllore and garnet. Kaolinite peak which could be observed as the dominant mineral phase was identified at 2θ values of 12.4° , 20.5° , 24.9° and 35.1° each corresponding to intensity readings of 1500, 500, 1800 and 400 counts, respectively. The quartz phase present were identified at 2θ values of 26.6° and 38.5° which correspond to intensity readings of 9200 and 250 counts, respectively. Other impurities identified include clinocllore at 2θ values of 13° and 25° ; orthoclase at 2θ values of 36° and 52° and garnet at 2θ values of 39° and 56° .

Figure 2 shows the quantitative XRD analysis of raw Getso clay. It could be observed that the kaolinite content of the clay was 8% and the quartz composition was 51%. The of other impurities present were 18%, 16% and 7% for the orthoclase, clinocllore and garnet, respectively.

Figure 1. Qualitative XRD diffractogram of raw Getso clay

Figure 2. Quantitative XRD of raw Getso clay

Figure 3 shows the qualitative XRD patterns of the beneficiated Getso clay. The peaks of kaolinite at 2θ values of 12.4° , 20.5° , 24.9° and 35.1° became more prominent in the beneficiated clay. The corresponding intensity readings were 6200, 700, 4800 and 800 counts, for the 2θ values of 12.4° , 20.5° , 24.9° and 35.1° , respectively. The intensity of quartz at 2θ values of 26.6° had reduced drastically from the initial value of 9200 counts in the raw clay to 1500 counts in the beneficiated clay. The peaks for the other impurities could be observed to have reduced similarly.

Figure 4 shows the quantitative XRD analysis of the beneficiated Getso clay. It could be observed that the kaolinite content of the beneficiated clay had improved immensely from the initial value of 8% in the raw clay to 61% in the beneficiated clay. The quartz composition had reduced immensely from the initial value of 51% in the raw clay to 27% in the beneficiated clay. Garnet was completely absent in the beneficiated clay while orthoclase and clinocllore had each reduced by 5% and 7% respectively.

Figure 3. Qualitative XRD diffractogram of Beneficiated Getso clay

Figure 4. Quantitative XRD of Beneficiated Getso clay

Table 1 presents a tabular analysis of the XRD results of Getso clay before and after beneficiation. It could be observed that the major kaolinite peaks at 2θ values of 12.4° and 24.9° had intensities of 1500 and 1800 counts, respectively in the raw clay. These kaolinite intensities increased by 313% and 166%, respectively after the beneficiation process. Quantitatively this translated into 53% increase in the kaolinite content. The beneficiation process had been effective as substantial decrease in the impurity content could be observed from the raw to the beneficiated clay. The Garnet content was completely reduced to zero while quartz, clinocllore and orthoclase were reduced by 24%, 9% and 13% respectively.

Table 1. Analysis of the XRD results for the raw and beneficiated Getso clay

Constituent Mineral	XRD Major Peak ($^\circ$)	Intensity (Count)		Quantitative XRD (%)	
		Raw Clay	Beneficiated Clay	Raw Clay	Beneficiated Clay
kaolinite	12.4 & 24.9	1500 & 1800	6200 & 4800	8	61
Quartz	26.6	9200	1500	51	27
Clinocllore	13	2000	750	16	7
Orthoclase	52	650	50	18	5
Garnet	39	650	200	7	0

3.3. Chemical Characterization

The XRF chemical compositions of the raw and beneficiated Getso clay are as presented in Table 2. The silica-alumina ratio of the raw clay which was 1.55 reduced to 1.49 after the beneficiation process. This was likely due to the removal of silica components such as quartz as already determined in the beneficiation and XRD results. A complete absence of MgO was observed after beneficiation. This was likely due to complete removal of magnesium-containing impurities during the beneficiation process. Other metallic oxides which reduced as a result of the beneficiation were P_2O_5 and MnO.

Table 2. X-ray fluorescence results of Getso clay

Oxide (wt%)	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	K ₂ O	P ₂ O ₅	MgO	MnO	CaO	TiO ₂
Raw	55.68	35.95	2.48	1.42	0.83	0.59	0.31	0.3	0.23
Beneficiated	55.51	37.30	3.13	1.47	0.59	0.0	0.20	0.43	0.30

3.4. Physicochemical Characterization

3.4.1. pH analysis

The pH analysis results of the various clay-water mixture are as presented in Table 3. The raw Getso clay had an average pH value of 7.5. Beneficiation of the clay reduced the pH value marginally as the beneficiated clay had pH value of 7.3. It could be inferred that either in the raw or beneficiated form Getso clay is neutral.

The neutrality was enhanced by the beneficiation process as the pH value of the clay was closer to 7.0 absolute neutral point after beneficiation.

Table 3. pH analysis of Getso clay

Clay to water ratio	1:1	1:2	1:3	1:4	Average pH of Sample
pH of raw clay	7.7	7.5	7.4	7.4	7.5
pH of beneficiated clay	7.2	7.3	7.3	7.4	7.3

3.4.2 Specific gravity

Table 4 presents the specific gravity analysis of Alkalari clay. Applying Equation (1) the specific gravity of the raw clay was determined as 2.24. After beneficiation the specific gravity of the clay reduced by 9%. This was likely due to the effect of quartz removal as quartz is the densest constituent of a clay [13]. This result further validates results of the beneficiation and XRD which earlier suggested removal of quartz during beneficiation.

Table 4. Specific gravity analysis of Alkalari clay

Weight (g)	Raw Clay	Beneficiated Clay
W ₁	29.3	29.3
W ₂	72.4	80.3
W ₃	102.2	104.4
W ₄	78.3	78.3
Specific gravity	2.24	2.04

4. Conclusion

Mineralogical characterization of raw Getso clay has revealed that the clay was a kaolin clay having high content of mineral impurities such as quartz, orthoclase, clinocllore and garnet. The raw clay contained only 8 wt% kaolinite and 51% quartz. However, the beneficiation process was quite effective as it improved the kaolinite content by 660% while the quartz impurity was decreased by 47%. The chemical characterization has shown that silica-alumina ratio of the raw Getso clay was 1.55 and this reduced to 1.49 after beneficiation due to reduction in quartz and other dense impurities. The beneficiation process resulted into complete removal of MgO and reduction of P₂O₅ and MnO impurities. The physicochemical characterization has shown that Getso clay is neutral having average pH values of 7.5 and 7.3 for the raw and beneficiated clay, respectively. The specific gravity of the raw Getso clay which was 2.24 reduced by 9% after beneficiation. It could be referred that good commercial grade china clay could be produced from Getso clay if it undergoes wet beneficiation process. The china clay produced could be serve as an excellent raw material for production of whitewares such as tiles, high grade ceramics and even used in synthesis of high-tech materials such as zeolite catalysts [15].

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