ORIGINAL ARTICLE

REFINEMENT OF NIGERIAN KAOLIN BY WET PROCESSING AND OXALIC ACID TREATMENT FOR INDUSTRIAL APPLICATION

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ABSTRACT

Background: Generally, the quality of kaolin is measured as a function of iron content since this element gives an undesirable reddish colour to this type of mineral. If present in excess of threshold level, these impurities affect the commercial value of products derivable from kaolin. Nigeria is blessed with abundant deposits of kaolin in various parts of the country which are under-utilized for the production of kaolin based products because of iron-bearing impurities associated with the mineral. **Methods**: To address this challenge, this study was conducted to assess the effect of wet processing and oxalic acid treatment in the removal of iron oxide bearing impurities from Alkaleri and Onibode kaolin to meet industrial application. Kaolin sample from two major deposits, Alkaleri and Onibode was subjected to wet processing and leaching using 0.5M concentration of oxalic acid treatment. The total mineralogical composition of the two deposits, their chemical and morphological properties were studied using XRD, XRF and SEM as well as the effect of wet processing and oxalic acid treatment in the removal of iron-bearing impurities. **Results:** The results showed significant removal of iron oxide using oxalic acid from 1.92 to 0.60 % for ferric oxide and 2.62 to 1.05 % for titanifferous oxide in Alkaleri and 3.45 to 1.17% for ferric oxide and 4.28 to 1.98 for titanifferous oxide in Onibode kaolin. **Conclusion**: It can be inferred that the use of the two treatment processes can effectively reduce the iron-bearing impurities in Alkaleri and Onibode kaolin to acceptable threshold level for industrial application.

***Keywords**: Kaolin, Alkaleri, Onibode, wet processing, leaching, iron-bearing impurities.*

1. INTRODUCTION

Kaolinitic clay is a versatile industrial mineral. The oldest known use of the clay is as a ceramic raw material. Chemically, kaolin is a 1:1 layer sheet structured hydrated aluminum silicate with a very fine particle size with one silicon oxygen (SiO4) tetrahedral layer and one alumina [Al(O, OH)$_6$] octahedral layer (or expressed in other way, [Si_2O_5]$_2$– sheet and [$Al_2(OH)_4$]$_2$ sheet) with pseudo-hexagonal symmetry, bonded together through sharing of apical oxygen and exists alternately. Its theoretical formula is $Si_2Al_2O_5(OH)_4$ (other formulas are $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$ and $Al_2O_7Si_2 \cdot 2H_2O$), which has a molecular weight of 258.071 g/mol [1,2,3].

The main characteristic, which determines the utility of the clay for various applications, is its purity. Pure kaolinite is white in colour and its chemical composition is 46.54% SiO_2, 39.50% Al_2O_3 and 13.96% H_2O [4,5,6,7]. The Kaolin particles are usually hexagonal and 0.05-10 µm in diameter (0.5 µm on average) [8]. Presence of impurities, particularly iron- and titanium-bearing materials, imparts colour to kaolin. The mined kaolin is usually associated with various impurities like quartz, anatase, rutile, pyrite, siderite, feldspar, etc., depending on the origin and depositional environment [9]. These impurities impair the characteristics of a kaolin and effect its utilization for various end applications. Major Industrial application of kaolin are: paper filling and coating (45%); refractories and ceramics (31%); fiberglass (6%); cement (6%); rubber and plastic (5%); paint (3%); [10]. It also finds application as a filler for insecticide, formulation of medicine, cosmetics, [9].

However, presence of iron-bearing impurities in kaolin lowers its economic value and prevents its used in the production of ceramic and paper products and more advanced applications such as calcined kaolin. The major process of attaining high quality kaolin for industrial application is by the use of physical and/or chemical processes. Studies on of some of these processes are sieving, magnetic separation [11], selective flocculation [12], leaching with chemicals like oxalic and other organic acids [13,14,15,], organic acids in the presence of a fermented medium [16], leaching containing microbial-produced oxalic and hydrochloric acid [17], EDTA [18], sodium dithionate-H_2SO_4 mixtures [19], and so on, have been employed to lower the content of these impurities in the kaolin clays.

It is well established that oxalic acid is a potential leaching agent for dissolving heavy metals from various minerals including kaolin, and biohydrometallurgy groups are now considering processes for removal of heavy metals depending on this property of oxalic acid [20,21,22].

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Nigeria has a huge reserve of kaolin with an estimated reserve of about 3billion metric tonnes scattered in different parts of the country [23]. There are at least 45 known deposits of kaolin in Nigeria, there is no state in the country without at least one deposit [24]. The market for kaolin in Nigeria is large, sustainable and expanding because of the numerous applications of the product. However, kaolin as raw materials for industrial application must be free or significantly reduced to acceptable limits, from iron-bearing and other impurities for increased whiteness and refractory properties of the end products [13-25,26,27]. Therefore, considering the vast deposits of kaolin scattered in various parts of Nigeria versus industrial utilizations, this study was aimed at assessing the use of wet processing and oxalic acid treatment for the refinement of Alkaleri and Onibode kaolin to meet industrial specification and application.

2 MATERIALS AND METHOD

2.1 Materials

Kaolin was procured from miners in Alkaleri, Bauchi State situated at 10^{o} 48′ E and 9^{o} 88′ 98′′ N latitudes and Onibode in Ogun State situated at 3^{o} 27′ E and 7^{o} 7′ 60′′ N latitudes.

2.2 Wet processing

The crude kaolin samples were beneficiated by soaking in water for 2 days in a plastic drum, with periodic stirring using an automatic fast rotating stirrer, while the suspended impurities were decanted on daily basis. The resulting mixture was sieved using 75micron sieve and allowed to settle for 24hours. The supernatant was decanted, while the beneficiated kaolin clay was centrifuge, dried atmospherically for 48hrs, and heated thermally using a laboratory oven to 100°C for 12 h. The resulting beneficiated dry kaolin was grinded and sieved using a 200µm sieved.

2.3 Leaching process

The leaching experiments were performed following the method of Baba *et al.,* (2015) [22] with a slight modification. The leaching was conducted using a 500 mL glass reactor equipped with a magnetic stirrer under the following conditions: Oxalic acid concentration of 0.5M., Temperature maintained at 85°C., Time duration of 120 min and a solid to liquor ratio of 1:12.5. After the completion of leaching, leached residues were filtered and oven-dried.

2.4 Characterization techniques

The crude, wet processed and leached clay, were characterized by X-ray fluorescence spectroscopy (XRF), X-ray diffraction (XRD), and scanning electron microscope (SEM).

2.4.1 X-ray fluorescence (XRF): XRF analyses of the samples were done using a Model-PW2400 of Phillips make, with X-ray tube of rhodium anode and scintillation detector with a current 40mA and voltage 40 mV.

2.4.2 X-ray diffraction (XRD): The X-ray diffraction data was collected using a Philips analytical X-ray instrument, X'Pert-MPD (PW 3020 vertical goniometer and PW 3710 MPD control unit) employing Bragg–Brentano para focusing optics. The XRD patterns were recorded in the range of 10–70◦ with a scanning rate of 2◦/min.

2.4.3 Scanning electron micrographs (SEM): Scanning electron micrographs were taken on a JEOL-JSM 5600 LV microscope, equipped with a 6587 EDS (energy dispersive Xray spectrometry) detector, using an accelerating voltage of 15 kV. The samples were deposited on a sample holder with an adhesive carbon foil and sputtered with gold.

3 RESULTS AND DISCUSSION

3.1 XRF characterization

Table 1: The table presents the chemical Composition Analysis of Raw, Wet processed and leached kaolin samples.

OXIDES	RALK	RON	WPALK	WPON	LALK	LON
Al_2O_3	45.05	45.20	41.68	39.60	38.29	37.05
SiO_2	34.08	32.89	43.28	43.26	44.91	45.66
TiO_2	2.62	4.28	2.43	3.95	1.05	1.98
Fe_2O_3	1.90	3.45	1.79	3.02	0.60	1.17
CaO	0.08	0.10	0.025	0.07	0.01	0.04
CuO	0.035	0.10	0.02	BD	BD	BD
ZnO	0.014	0.02	0.012	0.01	0.01	BD
MnO	0.018	0.003	0.012	BD	BD	BD
LOI	12.24	11.89	11.49	10.79	14.38	13.87
Si/Al	2.23	2.23	1.80	1.90	2.00	2.10

RALK: Raw Alkaleri Kaolin; **RON:** Raw Onibode Kaolin; **WPALK**: Wet processed Alkaleri Kaolin; **WPON:** Wet processed Onibode Kaolin; **LALK**: Leached Alkaleri Kaolin; **LON**: Leached Onibode Kaolin.

Table 1 showed the results of chemical analysis of the raw, wet processed and acid treated kaolinite clay. The XRF analysis was carried out to know the chemical compositions of the clay and the subsequent chemical changes that occurred due to wet processing and acid treatment. The parent clay contains alumina and silica which are in major quantities and high content of iron oxide and titanifferous oxide which appeared as titanium dioxide. Other oxides such as manganese oxide, calcium oxide, Copper oxide, and zinc oxide are present in trace amounts.

3.2 Leaching Analysis

Hernandez *et al* (2012) depicted the process of leaching reaction with the following stoichiometry [25]:

$$H_2C_2O_4 \longrightarrow H^+ + HC^2O_4^- \quad (1)$$

$$HC_2O_4^- \longrightarrow H^+ + C_2O_4^- \quad (2)$$

$$Fe_2O_3 + H^+ + 5HC_2O_4 \longrightarrow 2Fe\,(C_2O_4)_2^{2-} + 3H_2O + 2CO_2 \quad (3)$$

From equations (1) and (2) it is evident that dissolution of iron impurities from the kaolin samples were carried out by bi-oxalate as depicted in equation (3). The reduction of iron oxide was a result of metal ions migration from lattice position to exchange position which was proceeded by iron removal [28].

During the acid treatment it was observed that the alumina content of the wet processed clay decreased. The Al^{3+} content decreased as a result of the 0.5M Oxalic acid leaching of the beneficiated kaolin resulting also to a decrease in the Si/Al ratio. Panda *et al.,* (2010) assert the decrease in the alumina content in the acid treated sample to the leaching of the Al^{3+} ions from the octahedral layer due to hydrolysis under acidic conditions [35]. During acid treatment, it was observed that the concentration of CaO, MnO, ZnO and TiO_2 also reduced. The decrease in the aluminum content in the leached kaolin can be interpreted due to the shielding of aluminum cation by the silicon oxygen network and the presence of Al–O–Si bonds results in low solubility at low acid strength.

3.3 XRD analysis

The mineralogical content of the crude kaolin is shown in the XRD patterns of Figures 1 and 2. The sharp peaks of the pattern were an indication that all the kaolin clay from the two deposits were crystalline as also evident by the high intensity counts (Y-axis). The crystalline nature observed was because the mineral was formed by nature. The observable characteristic peaks of kaolinite at Bragg's angle of 12.4, 19.9, 20.4, 24.9, 34.9, 36.0, 36.1, 38.4, 45.2, 54.9 and 62.3° confirmed that the starting materials were kaolinite [29,30,31], with other associated major impurities such as Zircon, Anatase Quartz, Ilmenite and halloysite

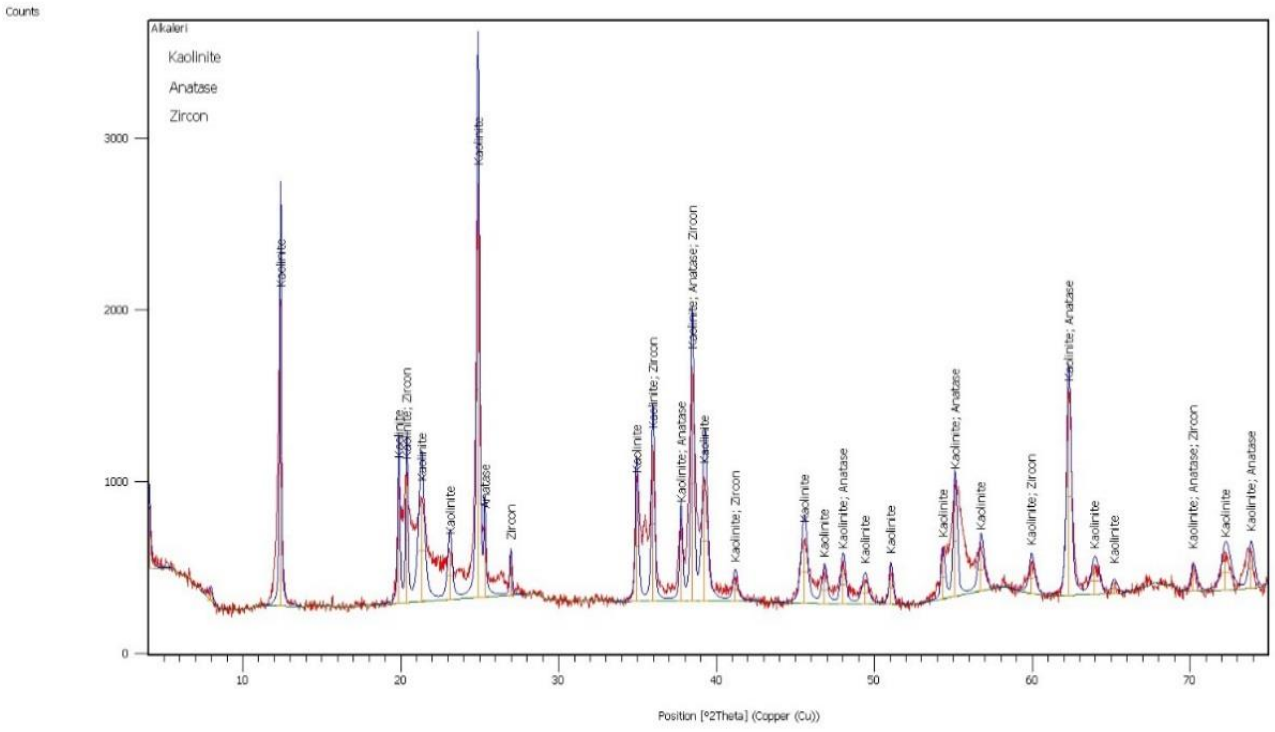

Figure 1: The figure presents the X-ray diffractogram of Raw Alkaleri kaolin.

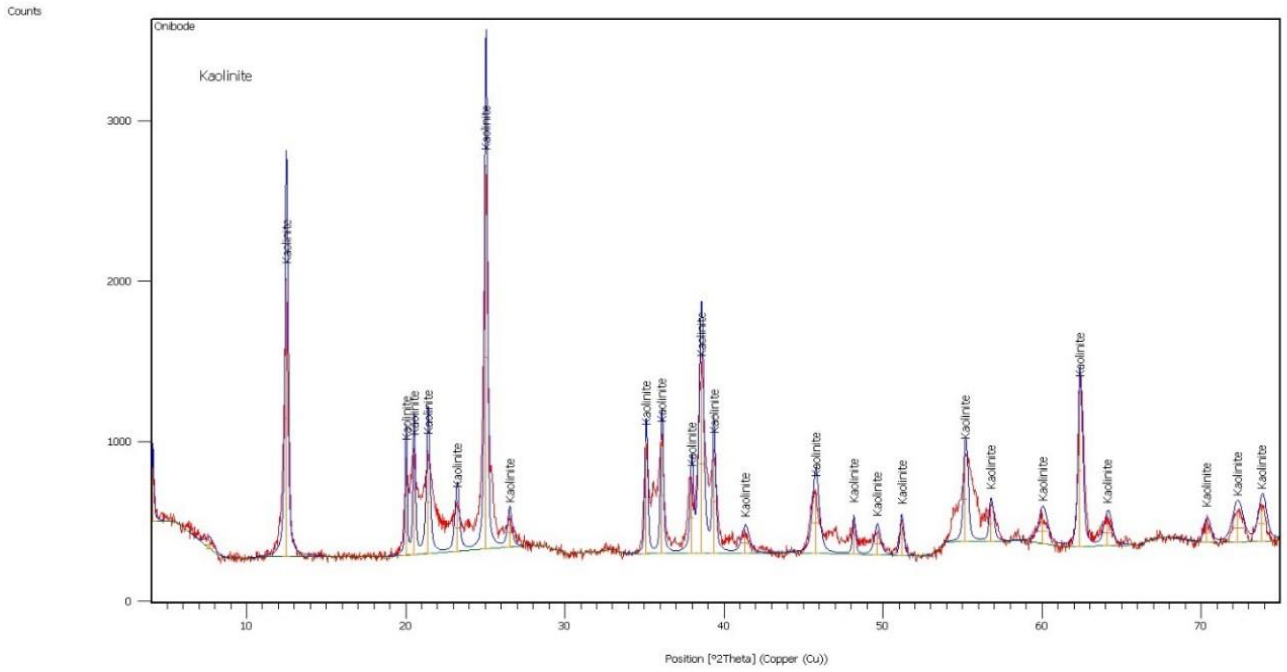

Figure 2: The figure presents the X-ray diffractogram of Raw Onibode kaolin.

Furthermore, the basal spacing at different angles indicates that the basal spacing of Alkaleri is sharper and intense than Onibode. This indicates that the kaolinite crystals in Alkaleri is more orderly than the kaolinite from Onibode. Bloodworth *et al.,* (1993) observed that the degree of order or disorder of kaolinite component may influence the potential end-use of the kaolinite [32]. Generally, a well ordered kaolinite has better crystal morphology and are usually non-plastic while the reverse is the case for a poorly ordered kaolin [7].

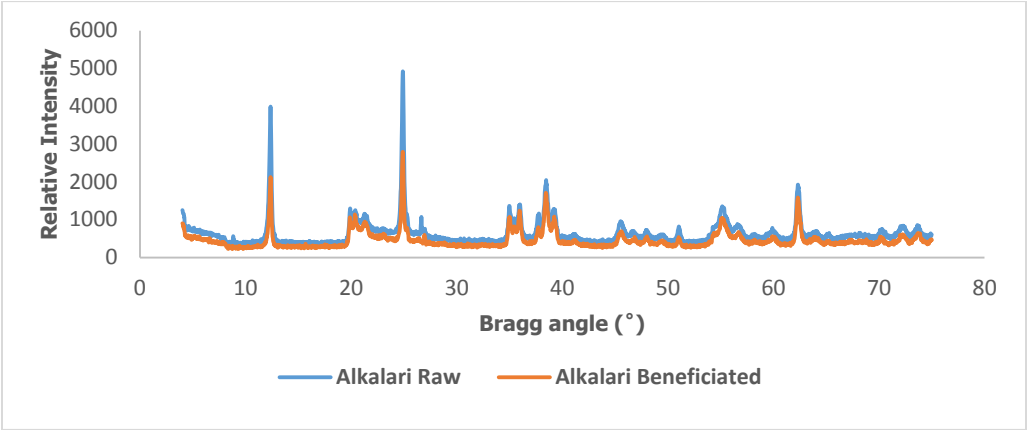

Figure 3: The figure presents the X-ray diffractogram comparison of raw and beneficiated Alkaleri kaolin.

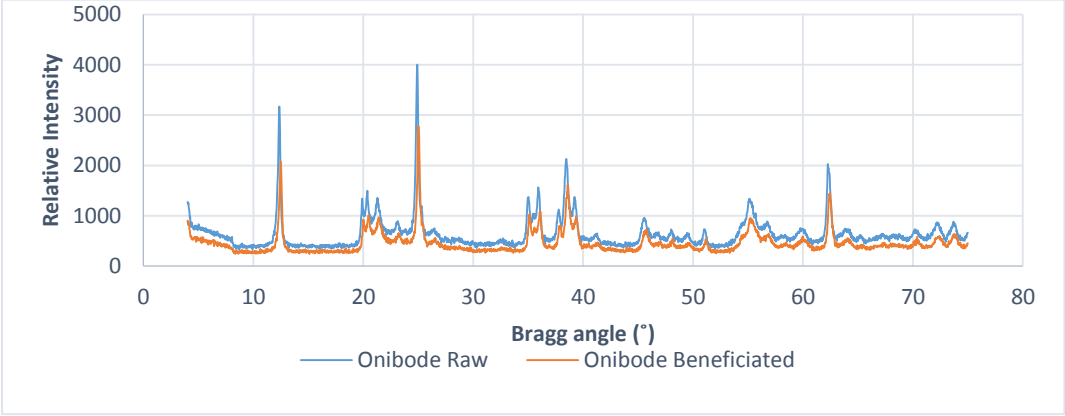

Figure 4: The figure presents the X-ray diffractogram of raw and beneficiated Onibode kaolin.

The XRD pattern shown in Fig 3 and 4, was dominated by the kaolinite peaks after wet processing and leaching of the kaolin samples and also, the peak attributed to quartz. This is a confirmation that the quartz material (especially the chemically combined form of it) was not completely removed through wet processing and leaching.

3.4 SEM/EDX analysis

The scanning electron micrographs of the two kaolin clay samples are presented in Fig 5 and 6 which showed the morphological features of raw and processed.

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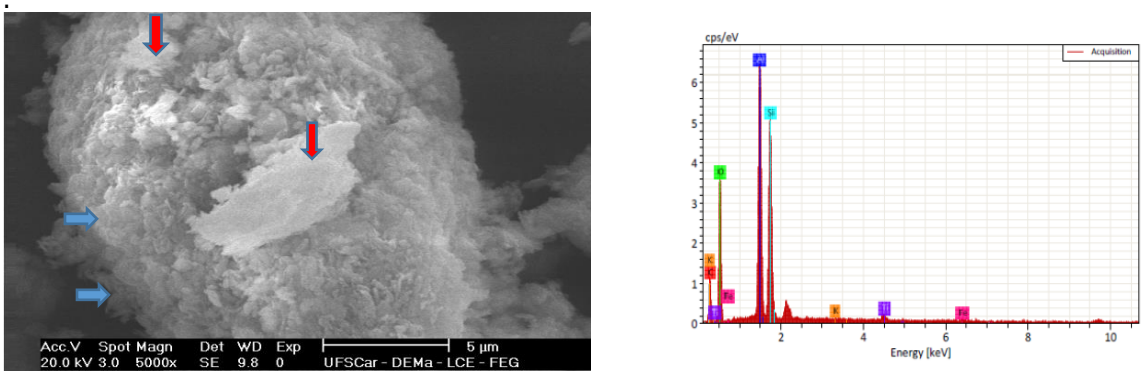

Figure 5: SEM/EDX of raw Alkaleri kaolin

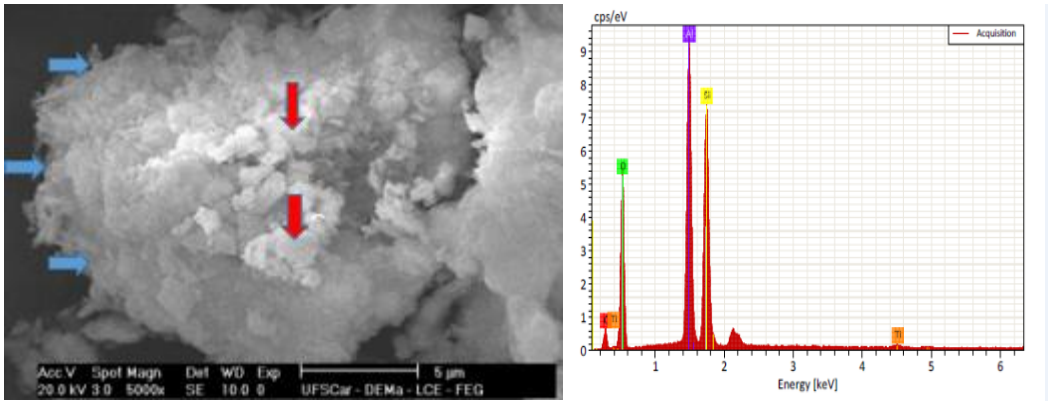

Figure 6: SEM/EDX of raw Onibode kaolin.

The SEM micrograph showed a moderately ordered crystalline kaolinite flakes in both Alkaleri and Onobode kaolin with poorly developed hexagonal outlines. The edges of the particles are bevelled, somewhat ragged and irregular. Some wedge-edged patches associated with wedge shaped minerals such as titanifferous minerals, which coat kaolinite particles as impurities are also noticeable [33]. This is in total confirmation with the diffractogram in Fig 1 and 2 and the XRF result in Table 1 further buttress the level of crystallinity and purity. The kaolin EDX spectrum for both kaolin as shown in Fig 5 and 6 were characterized by nearly equal peak height of Al and Si, another means to confirm the predominance of kaolinite

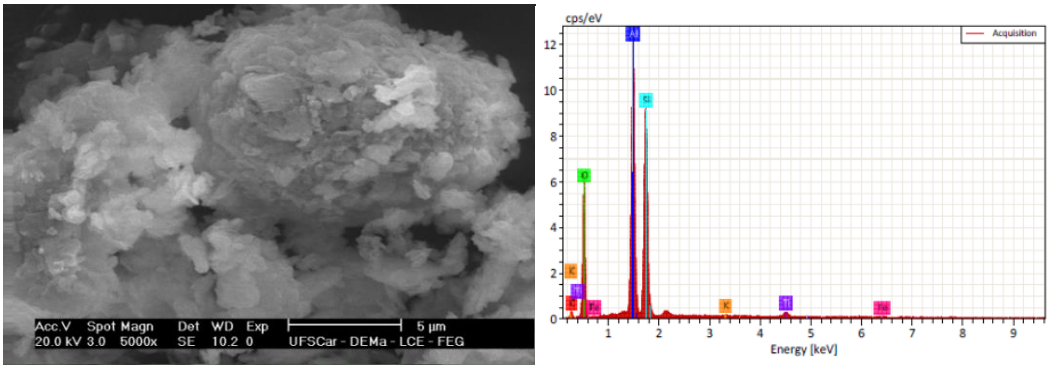

Figure 7: SEM/EDX processed Alkaleri kaolin.

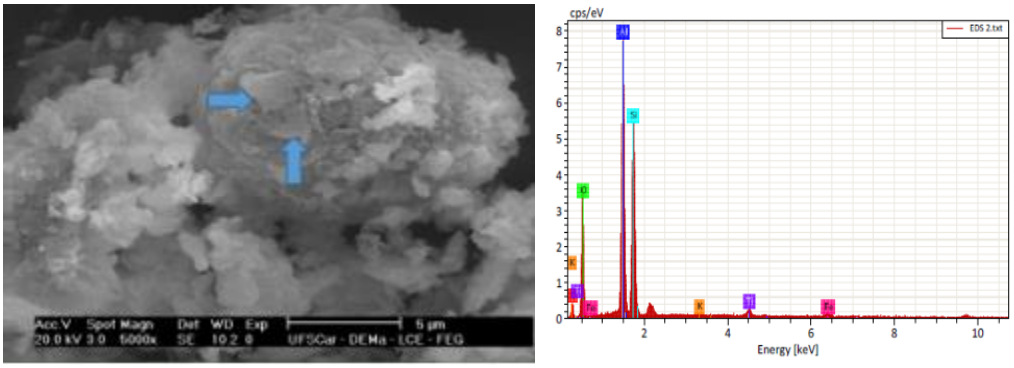

Figure 8: SEM/EDX processed Onibode kaolin.

The SEM micrograph of the processed kaolin samples indicated the retainment of kaolinite structure with small aggregate particles of silica-alumina plate. Spongy surface observed in the raw kaolin was not observed in the SEM for the processed kaolin samples. It was however observed that the processed kaolin was lean than the earlier identified fibrous materials, pointing to removal or reductions in the inherent impurities. The EDX showed less silica retainment in Onibode than Alkaleri which conformed to the XRF result in Table 1. Additionally, the silica-alumina plate for kaolinite clay was observed to be better arranged with little broken edges in Alkaleri than Onibode kaolin.

5. CONCLUSION

In this work, we have studied the effect of wet processing and acid treatment on the structural and chemical properties of kaolin clay. The XRF and SEM clearly showed the changes in the chemical composition and morphological changes on the kaolin samples upon wet processing and acid treatment. As the processing progressed, the iron oxide bearing impurities decreased as follows: Alkaleri (TiO_2) 2.62 – 2.43 – 1.05., (Fe_2O_3) 1.90 – 1.79 – 0.60. While for Onibode (TiO_2) 4.28 – 3.95 – 1.98., (Fe_2O_3) 3.45 – 3.09 – 1.17 Similarly, as the processing progressed from wet processing to acid treatment, Al_2O_3, SiO_2, MnO, CaO and ZnO contents in the Kaolin material decreased progressively. XRD studies also showed the structural transformation of the kaolin clay from crystalline in the raw state to an amorphous state. It can be therefore be concluded that a combination of wet processing and leaching is required for a high purity grade Nigerian kaolin for industrial application.

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