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Characterization of Alkaleri Clay and Its Suitability for Ceramic Filter Membrane

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ABSTRACT

This study focuses on the characterization of Kaolin clay found in commercial quantities in Alkaleri in Alkaleri Local Government Area (LGA) of Bauchi State, North-East Nigeria for its use as Ceramic Filter Membrane (CFM). The mineral composition is evaluated by X- Ray Fluorescence (XRF), crystalline structure by X-Ray Diffraction (XRD), Scanning electron microscopy (SEM)-EDX and Fourier Transform Infrared Spectroscopy (FT-IR The content in fluxing oxides (Na_2O, K_2O, CaO, and MgO) is very low and this may result in low vitreous phase formation during firing and The presence of TiO_2 is indicative of low anatase contents in all samples. The infra-red spectra are typical of kaolinite as indicative of the XRD of the investigated samples and the presence of kaolinite with quartz, corundum syn, montmorilonite and ilmenite as pure clay fractions in the clay mineral. The presence of relatively low less than 1 wt% of other oxides such as Fe_2O_3, K_2O, and TiO_2; can be considered as acceptable in the elaboration of clay for ceramic membranes.

1.0 INTRODUCTION

Clay defined as a natural, earthy, fine-grained material, largely of a group of crystalline hydrous silicate minerals known as clay minerals is composed mainly of silica, alumina, and water, but may also contain appreciable quantities of iron, alkalis, and alkaline earths. Clay is formed by the mechanical and chemical breakdown of rocks.

Clays have grown in importance and have interested the scientists in this area due to its abundant raw material requires low sintering temperatures, lower than that required for pure oxide materials and low temperatures during sintering process (Costa da Silva et al. 2015; Kamoun et al. 2020). However, the chemical and mineralogical composition of the samples bears enormous influence on the physical characteristics. Membrane separation processes extend more and more every day in industrial uses, with new requirements concerning the materials and preparation procedures. Due to their potential application in a wide range of industrial processes such as water and effluent treatments, drink clarification, milk pasteurization, biochemical processing, inorganic membrane technology grows in importance. The use of ceramic membranes according to Rekik et al. (2016) has many advantages as high thermal and chemical stability, pressure resistance, long lifetime, good resistance to fouling, and ease of cleaning.

Similarly, unlike the polymer-based filtration systems the use of ceramic membranes in water treatment applications is associated with several advantages such as high-temperature stability, fouling resistance, and low maintenance requirements contributing to low lifecycle costs in such systems. However, the high production costs of most commercially available ceramic membranes, stemming from raw materials and processing, are uneconomical for such systems in most water treatment applications. For this reason, there is a growing demand for new ceramic membranes based on low-cost raw materials and processes (Abdullayev et al. 2019). According to Abuh et al. (2014), indigenous clays with industrial potentials need to be investigated due to the challenges posed by high demand for clay base materials in Nigeria; and a kaolin deposit is of considerable economic potential but since its mineralogical composition controls ceramics processing properties as put forward by Shuaaibu et al. (2020); the qualities of raw clays are evaluated merely by their chemical composition and grains size distribution. The objective of this work is to study the natural clays from the Alkaleri clay mine, in the northeast of Nigeria, and the possibility of producing ceramic filters from it. The choice of this material is based on its natural abundance, low cost, high plasticity and thermal behavior for the manufacture of ceramic membranes.

2. MATERIALS/METHODS

2.1 Materials

The Kaolin material used for this research was sourced directly from the mine site in Alkaleri Local Governemtn Area (LGA) of Bauchi State, Nigeria. Clay sample was dug out from five different locations at a depth of 1.52m in order to get a good representation of the site. The mined clay samples were mixed properly and using the cone and quartering system by American Society for Testing and Materials (ASTM) a representative sample of 10kg was produced and stored for analysis.

2.2 Sample Processing: The unprocessed clay brought into the laboratory was in the form of agglomerated lumps of 10kg. The clay was then crushed, screened, pulverized (size reduction), sieved using 140mm mesh. The sample was there after soaked in water and allowed to stand for 20mins for the suspended particles mostly organic impurities to be decanted. The resultant mixture was then poured into a blunger and blunged for 10mins for homogeneity and to reduce their particles size. The slurry was removed from the blunger, sieved using electro-magnetic shaker fitted with a <140mm mesh. The now processed sample was poured into Plaster of Paris (P.O.P) baths to dewater and was allowed for 4 days to dry up and separate out of the baths. The resultant dried processed clay was put into the dryer (Fisher isotherm oven model 175) and set at 109^0C for 3 hrs.

The dried cake sample was then removed from the dryer, allowed to cool at room temperature and then crushed again using porcelain mortar and pestle, sieved using 100mm mesh and the fine powder obtained was used for the physical, chemical analysis and FTIR. (Shaaibu et al. 2020; Abuh et al. 2014)

2.3 Characterization of Clays

The Kaolin clay samples were characterized using a variety of techniques as follows:

X-ray fluorescence (XRF) tests were performed to quantitatively determine the chemical composition of the samples. The tests were conducted using PANalytical equipment (Axios Max) equipped with a rhodium X-ray tube at 4 kv.

X-ray diffraction (XRD) experiments were performed to identify the glass and mineral phases in the samples. The tests were conducted on PANalytical equipment EMPYREAN with Celerator detector and copper tube at a 2θ range of 3.5° to 70°. X' Pert High Score plus was used to interpret the results of XRD analysis.

Scanning electron microscopy (SEM) with a 10-kV beam voltage was used to estimate the particle sizes and morphologies of samples (Tescan, LMU; model, Vega 3).

Fourier Transform Infrared Spectroscopy (FTIR) Analysis, an analytical technique was used to identify organic, polymeric, and, inorganic materials. The particle size distribution of the as-received samples was obtained by laser scattering in aqueous suspension (Mastersizer S. Malvern, England).

SEM-EDX, XRF, and XRD were performed in the Laboratory of Analysis of Minerals and Rocks at the Steel Raw Material Research Agency, Kaduna; while the FTIR analyses was conducted at the Defence Industries Corporation (DICON), Kaduna, Nigeria.

3. RESULTS AND DISCUSSION

The characterizations of Alkaleri clays using techniques of Granulometric analysis, (SEM-EDX), XRD, XRF and FTIR are presented:

3.1 Mineralogy and Chemical Composition

XRF Analysis

The quantitative compositions of the clay were determined by XRF analysis and are presented in Figure 1:

XRF revealed SiO_2 49.42%; Al_2O_3 42.12% and total fluxes content of 1.36%. XRD revealed the present of kaolinite, 40.1%, quartz, 33.6%, corundrum, 14.7%, montmorilonite, 9.5% and ilmenite, 2.08%. According to Ajenifuja et a. (2017), The basic properties which mostly determines the use of the kaolinites for various applications, is its purity. Pure kaolinite ($Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$) is white in colour and its chemical composition is 46.54% SiO_2, 39.50% Al_2O_3 and 13.96% H_2O.

Figure 1: shows that the clays and were mainly composed of SiO_2 and Al_2O_3, as well

as other oxides in small amounts. These oxides are characteristic components of clay minerals.

Table 1: Mineralogy and Chemical Composition (ALKALERI) Clay

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
14	Si	Silicon	49.42	46.65
13	Al	Aluminium	42.12	38.19
39	Y	Yttrium	1.11	3.33
26	Fe	Iron	1.36	2.55
47	Ag	Silver	0.57	2.07
19	K	Potassium	1.18	1.55
41	Nb	Niobium	0.48	1.51
20	Ca	Calcium	0.78	1.05
12	Mg	Magnesium	0.99	0.81
17	Cl	Chlorine	0.65	0.77
16	S	Sulfur	0.44	0.48
11	Na	Sodium	0.49	0.38
23	V	Vanadium	0.21	0.35
22	Ti	Titanium	0.19	0.31
15	P	Phosphorus	0.00	0.00

From the elemental compositions Table 1 sample contain more silicon than aluminum. This was appropriate for kaolinite clay as the result was consistent with that obtained by Yahaya et al. (2017). The main mineral component of kaolin according to Zsirka et al. (2015) is kaolinite, consisting of layers held together by H-bonds where each layer comprises of two-dimensional arrangement of Al-centred octahedra (O) and a two-dimensional arrangement of Si-centred tetrahedra (T). The ratio of SiO_2 to Al_2O_3 of 1.22 is higher than theoretical value of 1.18 (Ojo et al. 2017; Garcia-Valles et al. 2020) indicating the presence of impurities, the presence of coloring materials such as iron and titanium in kaolin, make it unusable for industrial applications while the presence of other oxide impurities like CaO, MgO etc. could only reduce the suitability of such clayey mineral samples for refractoriness (Refaei et al. 2017; Ombaka, 2016). However, a Silicon content of 46.65% with Aluminum content of 38% is suitable for ceramic production (Ojo et al. 2017). Chemical and mineralogical composition of the kaolin sample is similar to other kaolins with applications in the ceramics industry (Zsirka et al. 2015; Okunlola & Owoyemi, 2015; Garcia-Valles et al. 2020).

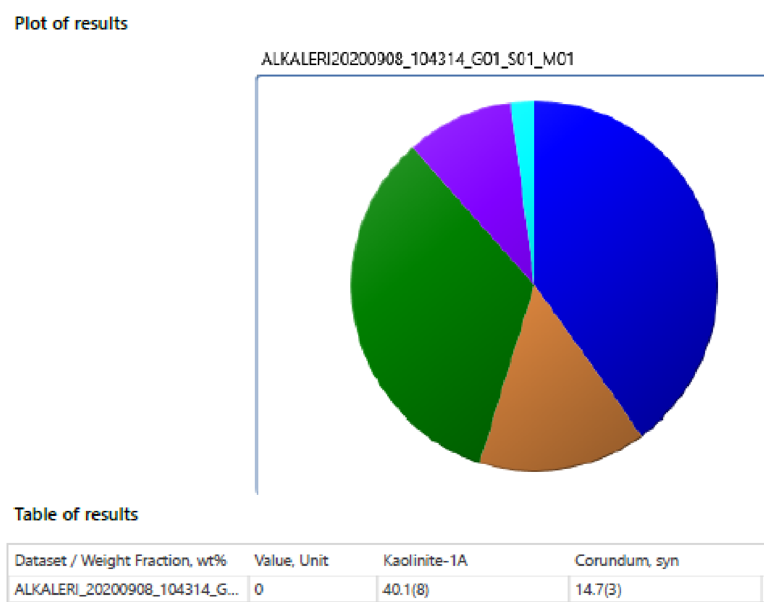


Figure 1: Plot of Compositions (ALKALERI) Clay Sample

3.4 X-ray Diffractrometry (XRD) Analysis

The clays and membranes were analyzed by XRD to identify the crystalline and amorphous phases. The XRD patterns of the clay sample (Figure 2) contain more expressive crystalline reflections that indicate Kaolinite, Quartz, Corundum, Montmorilonite and Ilmenite. The major mineral component of raw kaolin is kaolinite (K) with a small amount of quartz (Q) and illite (I) impurities, no other components were observed, because the impurities are in so tiny quantity (Figure 1) and most of them are probably incorporated into the crystal structure of kaolinite (Rekik et al. 2016). According to Garcia-Valles et al. (2020) high quality kaolin has low Fe, Ti, and alkaline earths contents.

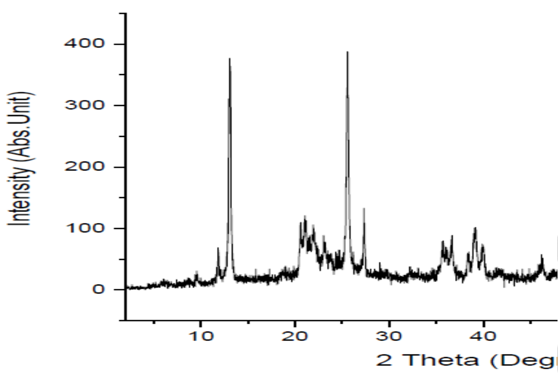


Figure 2: XRD patterns of Alkaleri Clay.

The XRD patterns of the sample (Figure 2) contain more expressive crystalline reflections that support the XRF result (Figure 1). The peak at 2Ɵ = 13.50^{o} is the distinctive XRD form of kaolin. Crude clay contains kaolinite and quartz as the major mineral constituents (Sengupta et al. 2008). The chemical requirements of the clay sample could be used as material for making ceramics (Annisa et al. 2019).

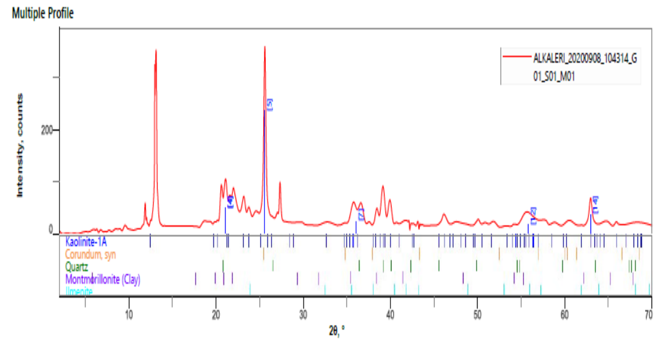
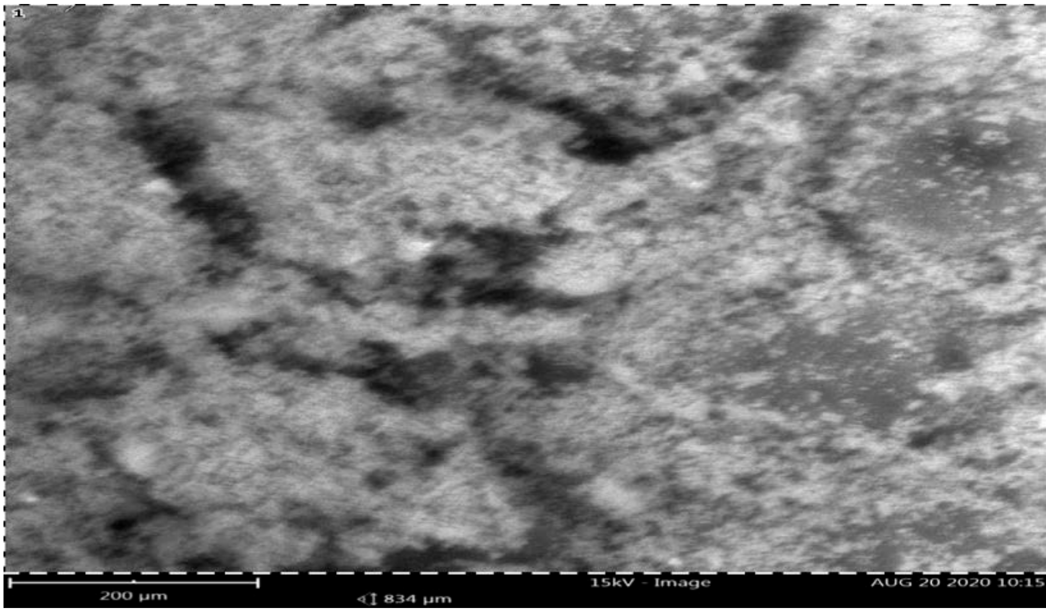


Figure 3: XRD Quantitative Analysis Plot (Alkaleri) Clay Sample

3.5 Scanning Electron Microscopy (SEM-EDX)

The morphologies of the clay were evaluated by SEM as shown in (Figure 4). Micrographies by SEM showed that the sample is essentially constituted by lamellar structures (Garcia-Valles et al. 2020; Elgamouz et al. 2019; Refaei et al. 2017; Hassan and Abdu, 2014). A larger number of pores were seen in the micrographs of the sample. The higher agglomeration may be associated with the organic matter contained in the sample, suggesting that the sample had a higher porosity mechanical strength. Different peaks observed correspond to the elements present in the samples. The accuracy of EDX spectrum can be affected by various factors since many elements will have overlapping peaks.



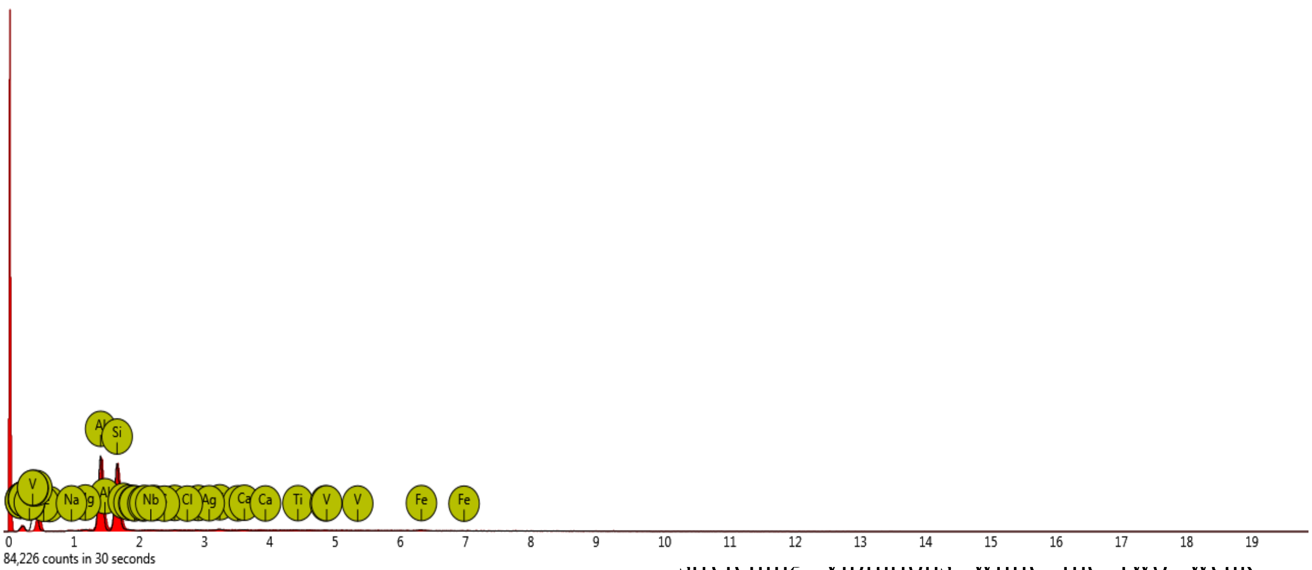


Figure 4: a)SEM-EDX (ALKALERI) Clay Sample

FTIR Analysis

An absorption band (Figure 5) was used for the identification of the clay mineral sample. Owing to the structural OH and Si-O groups obtained from the FT-IR spectrum, the types of cations directly linked to the hydroxyls has an influence on the OH absorption bands which is important in the determination of

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stretching vibrations while the two weak bands at around 2800-2400 and 1600-1400 cm-[1] were due to the out-of plane stretching vibrations.

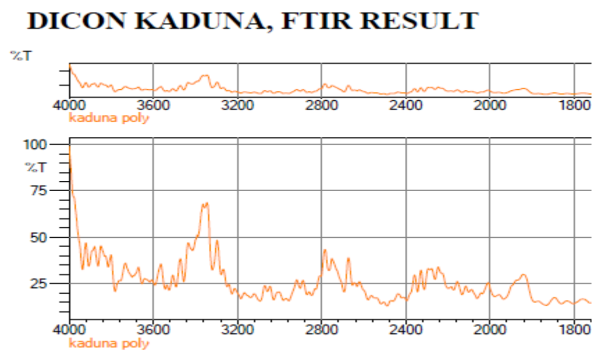

Figure 5: FTIR Pattern (ALKALERI) Clay Sample

Conclusions

The current work has studied the physic-chemical properties of Alkaleri natural clay sample via the elemental, chemical, and microstructural characterization techniques for its use as ceramic membrane filter. The results show that the analyzed sample is mainly constituted by kaolinite clay in major concentration, and quartz in minor concentration, which was confirmed by optical microscopy. The studied sample has a high SiO_2 content due to the presence of quartz, as shown by the diffraction peak. The presence of relatively low less than 1 wt% of other oxides such as Fe_2O_3, K_2O, and TiO_2; can be considered as acceptable in the elaboration of clay for ceramic membranes. In addition, the analysis by scanning electron microscopy revealed that the kaolin sample analyzed in this work is constituted of kaolinite clay mineral, as evidenced by the lamellar structure observed in the micrographies.

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