

ANALYSIS AND CHARACTERIZATION OF SELECTED CLAY FROM SOUTH-WEST NIGERIA FOR CERAMIC FILTERS APPLICATION

ABSTRACT

This present study is designed to investigate the suitability of selected Nigeria clay deposit for Clay based ceramic water filters (CWFs). Clay from eight (8) randomly selected locations within south-west Nigeria have been investigated with a view to determine their suitability for Clay based ceramic water filters (CWFs). The samples were collected from the following locations; Ijero (IJ) in Ekiti state, Ijapo (IJ), Barrack (BR), and Ondo (ON) in Ondo state, Ibafo (IB) in Lagos state, Ilesha(IL) and Idoka (ID) in Osun state and Ajebo (AJ) in Ogun state. Experimental analysis for Liner shrinkage, water absorption, bulk density, compressive strength X-ray diffraction (XRD) and Energy Dispersive X-ray Fluorescence (EDXRF) were carried out on each of the eight (8) samples. Test results reveals that all the clays contain high contents of alumina (Al_2O_3) and silica (SiO_2) with minor contents of P_2O_5 , Fe_2O_3 , MgO , K_2O , MnO and TiO_2 . The average crystal sizes of the clay were between 8.961nm and 26.875nm, lattice structure indicates that the sample were Monoclinic, Anorthic and orthorhombic. Compressive strength varies from 7.11664Mpa to 19.6583Mpa, moisture content varies from 0.66% to 2.59% and linear shrinkage varies from 6.9% to 13.70% while water absorption varies from 15.55% to 35.23%. The research indicated that Idoka, Ijapo and Ilesha Clay with specification of some industrial clays shows that the samples can be recommended for use in Clay based ceramic water filters production.

Keywords: : Clay; south-west Nigeria; X-ray Fluorescence; X-ray diffraction

1. INTRODUCTION

Clay is a powdery form of a rock which has been broken into fine particles by the action of millions of years of decomposition by the forces of nature. It is a product of the continuous weathering of the earth surface (Agbalajobi and Omoijuanfo 2012). Clay minerals are the most important industrial minerals. Millions of tons are utilized yearly in various applications. These applications include uses in geology, the process industries, agriculture, environmental remediation and construction. The physical and chemical properties of a particular clay mineral are dependent on its structure and composition, that reason for utilization of certain clay minerals in specific application (Fatai et al. 2014).

The complex nature of clay makes its study and findings an ever fresh area of interest especially to the world of science. Clay and its minerals have played major roles in anthropogenic activities. The low cost of clay and its relative abundance in nature, high sorptive /electric charge properties, plus ion exchange ability and compatibility with several materials, gives it a wide range of application (Barbel and Kurnianwan 2003) and (Costanzo, 2001).

The physical, chemical and mineralogical characteristics of clay deposits from different location in south west Nigeria state of Ekiti (Folorunso *et al.*, 2014), Ondo (Aramide *et al.*, 2014), Osun (Fakolujo *et al.*, 2012), Ogun (Fakolujo *et al.*, 2012) has been reported, The report revealed that their properties are related to several applications.

Ceramic water filtration as defined by Brown *et al.* (2007), is the process that makes use of a porous ceramic (fired clay) to filter microbes or other contaminants from water. CWF, have been used for water treatment for several centuries. According to Plappally (2010), water purification and filtration methods must be low in cost, socially and culturally acceptable and should be technologically feasible for the local environment. And, that there is a need to provide economic feasibility for small water supply systems.

Ceramic water filters are most frequently associated with the poor rural areas of developing nations. But in Nigeria, with the increase in rural-urban migration and urban development stretching into villages, towns and communities that were previously referred to as rural, it has become imperative and essential to study the general water and health needs of the population as a whole. Brown *et al.* (2007), believes that ceramic filters have become a promising lowest cost option of providing improved potable water at point of use.

Clay based ceramic water filters (CWFs) are usually produced by mixing of clay, sawdust (woodchips) and water. Other combustible organic material, such as rice husk, coffee husk or flour can also be used (Oyanedel-Craver & Smith, 2008). When clay is fired, it undergoes chemical transformation to become a strong, slightly porous material that does not deteriorate in water (Hagan *et al.*, 2009). Filters produced from modified clays can possess a high surface area (Hwang *et al.*, 2006), be durable and reusable and exhibit thermal stability, chemical inertness and excellent mechanical strength (Velmurugan and Mohan, 2009; Alam *et al.*, 2010; Selvaganapathi *et al.*, 2010). One property that is typically improved in porous ceramics is permeability, or the ability of fluids to move in the pores of the material (Chilingarian, 1995). The permeability of unmodified clay is low because the inter-granular spaces are very small.

Thus, the present study is designed to analyse, characterise and investigate the working properties of clay deposits from selected site in five south west state of Nigeria with the view to propose other possible uses such as microfiltration membranes in water treatment.

2. MATERIAL AND METHODS

2.1 Sample Preparation

The samples were collected from eight randomly selected locations within south west Nigeria namely: Ijero (IJ) in Ekiti state, Ijapo (IP), Barrack (BR), and Ondo (ON) in Ondo state, Ibafo (IB) in Lagos state, Ilesha (IL) and Idoka (ID) in Osun state and Ajebo (AJ) in Ogun state.

The samples were dried in open air, after which, the samples were manually crushed into thin particles using a mortar and pestle, and then levitated using the water extraction method. The clay samples were mixed with distilled water and thoroughly stirred and allowed to hydrate for several hours and latter decanted. This process is repeated until purer clay is obtained. The samples were sun dried and subsequently dried in a laboratory oven at 110°C for 24 hours. The resulting dried clay samples were pulverised and sieve with a digital octagon sieve shaker BS/ISO 3310 to an average particle size of 150 µm, and sent for analysis.

2.2 Physico-Chemical Analysis

2.2.1 Moisture Content

Test analysis was carried out as specified in ASTM D2216. A representative sample was weight and place in a crucible of know weight and dried in an oven at 105°C, until a constant

weight was obtained and the percentage of moisture in each sample was calculated using the formula below;

$$W(\%) = \frac{M_w}{M_s} \times 100 \quad 1$$

$$M_w = M_{CM} - M_{CD} \text{ and } M_s = M_{CD} - M_C \quad 2$$

Where: , W= water content (%), M_c = mass of empty crucible, M_{CM} = mass of crucible and moist clay, M_{CD} = mass of crucible and dry clay, M_s mass of clay solids, M_w = mass of pore water

2.2.2 Bulk density and apparent porosity

Test samples were dried in laboratory air, (temperature of 105°C, humidity of 40%) for 4hours. After drying, the samples were sintered in a muffle furnace. The firing involved pre-heating of the sample to 500°C, followed by heating to the sintering temperature of 850°C and allow to cool overnight in muffle furnace. The dry weight (D), of the samples were recorded, then immersed in a beaker of distil water and their soaked weight (S) recorded. They were then suspended in a beaker using a sling and their suspended weights (W) recorded. The bulk density and apparent porosity were calculated using

$$\begin{aligned} \text{Bulk density} &= \frac{D}{S-W} \\ \text{Apparent porosity} &= \left[\frac{(S-D)}{S-W} \right] \times 100 \end{aligned}$$

2.2.3 Determination of Linear Shrinkage

Clay sample were made into flat bars of 12cmx3cmx1cm, and a line of 100mm long draw on each bar. The clay bar was dried at room temperature for 14 days, after which the final length was measured. Total percentage shrinkage was determined using the formula;

$$\% \text{ linear shrinkage} = \frac{\text{initial length} - \text{final length}}{\text{initial length}} \times 100\% \quad 4$$

2.2.4 Water Absorption

Flat bar of clay samples were first weighed using an electronic weighing balance, soaked in a bowl of water for 24 hours. Each was then removed from water, allowed to drip and the remaining was gently wiped to ensure that no water was attached to the surface and was re-weighed again. The difference in weight was then used in computing the percentage water absorption applying the formula below:

$$\% \text{ Water Absorption} = \frac{\text{soaked weight} - \text{dry weight}}{\text{Dry weight}} \times 100\% \quad 5$$

2.2.5 Compressive Strength

The compressive strength was determined using the Instron Universal Tester Model No: 3069, with a compressive load 25kN, applied at the rate of 70N/min.

2.2.6 Elemental Characterization.

The quantitative analysis of chemical components of both processed and unprocessed clay was done using EDX 3600B Energy Dispersive X-ray Fluorescence (EDXRF) spectrometer.

2.2.7 X-Ray Diffraction Analysis

The X-ray diffraction (XRD) was monitored using X-ray diffractometer GBC EMMA, CuK α radiation using an acceleration voltage of 25 kV and current of 400 μ A. The diffraction angle was scanned from 10° to 65° 2 θ , at a rate of 4.00 °/min. Crystallite size was determined using the Debye Sherrer's formula.

$$D = \frac{k\lambda}{\beta \cos \theta}$$

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144 Where, D is the particle size (Å),

145 K = constant (usually k = 0.89)

146 λ = wavelength of the incident X-ray beam ($\lambda_{\text{CuK}\alpha} = 1.5418 \text{ \AA}$),

147 β = full width at half maximum of the X-ray diffraction peaks (rad)

148 θ = Bragg angle of X-ray diffraction peak

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3. RESULTS AND DISCUSSION

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3.1 Physical Parameters

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154 The result of Bulk density, moisture content, linear shrinkage, water absorption and compressive strength are shown in table 1. It was observed that clay from barrack has the lowest moisture content of 0.66% follow by clay from Ajebo while clay from Ibafo has the highest content 2.59%. Liner shrinkage results shows that clay from Ondo has the highest shrinkage of 13.7%, follow by Ilesha clay with 12.5%, while Ijero clay has the lowest shrinkage of 6.9%, follow by Ajebo with 8.9% which fell within the internationally accepted value of 7—10% value for Alumino-silicates, Kaolin and fireclays Zubeiru (1997).

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162 **Table 1: Physical parameters of clay**

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Property	Ijero	Idoka	Ijapo	Ilesha	Ajebo	Ibafo	Barrack	Ondo
Bulk Density (g/cm ³)	1.07	1.54	1.38	1.82	1.21	2.56	1.31	1.39
Apparent Porosity (%)	30.0	22.2	24.6	27.0	25.4	27.9	22.4	26.9
Moisture Content (%)	2.34	0.96	1.37	1.37	0.93	2.59	0.66	1.42
Liner Shrinkage (%)	6.90	11.70	10.40	12.50	9.47	7.31	10.98	13.70
Water Absorption (%)	35.23	22.77	26.6	22.77	31.22	15.22	25.67	27.41
Comp. Strength (Mpa)	7.12	15.69	10.07	19.74	19.11	19.66	9.11	19.26

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166 Water absorption content shows Lagos clay with the lowest value of 15.22%, while clay from Ijero has the lowest compressive strength value of 7.11664Mpa and Lagos clay has the highest compressive strength of 19.6583Mpa.

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3.1.1 X-Ray Diffraction Analysis

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172 X-Ray Diffraction Analysis the clay samples are shown in figure 1-3 below, while the average crystal size and lattice structure parameter are shown in table 2.

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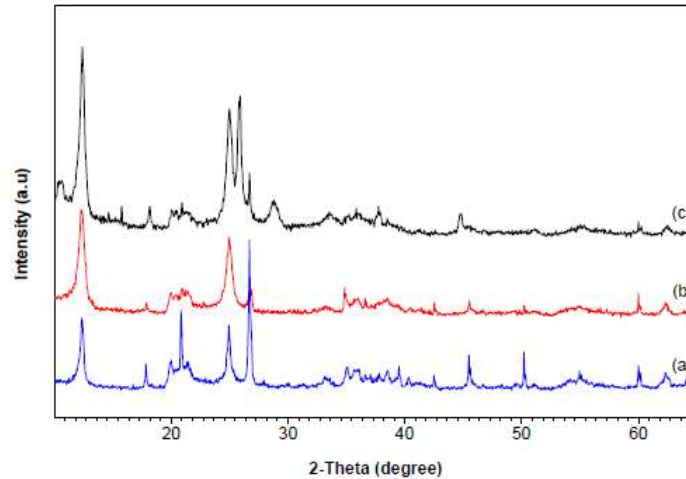


Figure 1: X-Ray diffraction of Ekiti and Osun state (a) Idoka (b) Ilesha (c) Ijero

Mineralogical compositions of Ekiti and Osun state clay as determined by X-ray diffraction in figure 1, shows that kaolinite is the dominant clay mineral in all samples. Other mineral constituents detected include free silica or quartz (SiO_2), illite $\{\text{KAl}_2(\text{Si}_3\text{Al})\text{O}_{10}(\text{OH})_2\}$. The major non-clay mineral constituent in these samples were quartz. The percentage composition of quartz ranges from 8% to 20%.

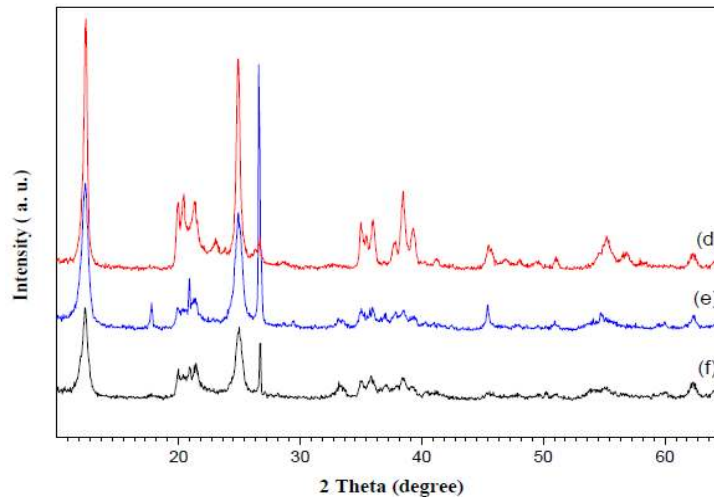


Figure 2: X-Ray diffraction of Ondo state clays (d) Ijapo (e) Barrack (f) Ondo

X-ray diffraction pattern of Ondo state clay shown in figure 2, identified major mineral phases includes silica, alumina, hematite and titania. Kaolinite content in sample d was higher than other clays. Feldspar's $\{(K, Na)\text{AlSi}_3\text{O}_8\}$, composition in sample e, was however higher than any other phases present in the clay samples.

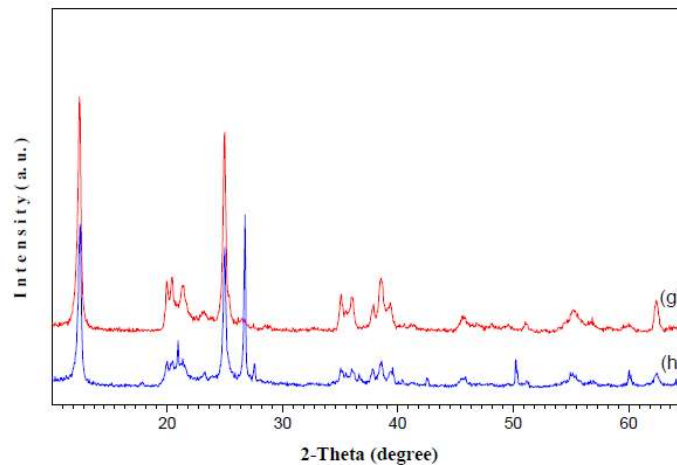


Figure 3: X-Ray diffraction of Ogun and Lagos clay (g) Ajebo (h) Ibafo

XRD results in figure 3, identified major mineral phases of silica, alumina, hematite and trace of titania. Sample h is observed to contain high feldspar in comparison with samples g. According to Brown *et al.* [Brown *et al.*, 2006] feldspars are the most abundant mineral group found in Earth's crust. Albite ($\text{NaAlSi}_3\text{O}_8$) anchors the two main feldspar compositional series: the alkali feldspars (Na, K) AlSi_3O_8 and the plagioclase series (Na, Ca) $\text{Al}(\text{Si, Al}) \text{Si}_2\text{O}_8$.

Generally, the XRD result revealed a high degree of crystallinity in all the samples. Careful investigation reveals that the samples were composed essentially of SiO_2 , Al_2O_3 , and to very limited extent of K_2O and Fe_2O_3 . Some other oxides were also present but in very negligible proportions. All the samples showed Kaolinite ($\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$) as the predominant mineral content with main reflections of silicon oxide and kaolinite.

Table 2 shows that Ilesha clay samples have the smallest average crystal sizes of 8.961nm, while Ijapo clay has the largest average crystal sizes of 26.875nm. Lattice structure shows that Idoka, Lagos, Barrack and Ondo are all Monoclinic, Ijapo, Ilesha and Ajebo are Anorthic, while Ijero is orthorhombic.

Table 2: Average Crystal Size and Lattice Structure Parameter of Clay Sample

Property	Ijero	Ijapo	Ilesha	Ajebo	Lagos	Barrack	Ondo
Lattice Structure	Orthorhombic		Anorthic			Monoclinic	
Average Crystal Size (nm)	20.35	26.88	8.96	19.65	19.68	20.78	14.88

3.2 Chemical Analysis

The results as showed in Table 3 indicated that the observed large amount of silica, alumina and iron contents suggests that the clays could be used for a variety of purposes. The value of Al_2O_3 in the samples ranges between 37-51%, which is higher than that from other location within Nigeria (Mark U. 2010, Kunlola and Owoyemi 2015). The presence of iron in

the clay samples is clearly observed although in various amounts; this has been supported by various researchers as a rule rather than exception [Badraoui and Bloom 1990, Boiabid *et. al.*, 1991, Badaut *et. al.*, 1992].

Table 3: Elemental Characteristics of Clay Sample

Element	Ijero	Idoka	Ijapo	Ilesha	Ajebo	Lagos	Barrack	Ondo
Al_2O_3	37.828	51.752	46.189	48.596	47.424	51.468	37.547	44.668
SiO_2	47.997	33.87	41.132	36.66	39.954	30.663	48.631	40.732
P_2O_5	0.147	0.157	0.162	0.175	0.128	0.162	0.171	0.117
SO_3	0.398	0.698	0.539	0.705	0.508	0.573	0.567	0.432
K_2O	1.419	1.258	1.142	1.098	1.299	1.118	1.071	1.406
CaO	0.087	0.048	0.064	0.056	0.084	0.051	0.351	0.060
TiO_2	0.776	0.684	0.785	1.057	0.141	0.948	0.898	0.859
V_2O	0.027	0.038	0.025	0.021	0.028	0.02	0.033	0.029
Cr_2O_3	0.027	0.031	0.022	0.016	0.047	0.006	0.031	0.042
MnO	0.099	0.072	0.04	0.032	0.183	0.003	0.062	0.128
Co_2O_3	0.346	0.316	0.046	0.087	0.36	0.009	0.306	0.447
Fe_2O_3	1.903	0.969	0.381	0.594	0.866	0.850	0.648	1.694
Ni_2O	0.053	0.076	0.05	0.046	0.066	0.08	0.041	0.054
CuO	0.036	0.064	0.04	0.056	0.061	0.063	0.049	0.035
ZnO	0.063	0.091	0.072	0.087	0.069	0.121	0.069	0.063
Mo	0.203	0.184	0.213	0.187	0.231	0.15	0.206	0.219
LOI	8.591	9.692	9.098	10.527	8.551	13.715	9.319	9.015

*LOI-loss on ignition

4. CONCLUSION

Selected clay from south west Nigeria state of Ekiti, Ondo, Ogun, Osun and Lagos in view of its physical and chemical properties has been characterized. Results obtain suggest that the sample contain high contents of alumina (Al_2O_3) and silica (SiO_2) with minor contents of P_2O_5 , Fe_2O_3 , MgO , K_2O , MnO and TiO_2 . The average crystal sizes of the clay were between 8.961nm and 26.875nm. Compressive strength varies from 7.11664Mpa to 19.6583Mpa.

A comparison results obtain from the analysis and charaterisation of all clay used in the research indicated that Ilesha Clay can be recommended for use in ceramic filter production

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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