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Characterization of Clays from Adamawa State, Nigeria, for 3D Printing Applications

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Abstract

The Sustainable Development Goal 6 aims to ensure availability and management of sustainable water and sanitation, and this is particularly poignant for rural communities that rely heavily on sources such as streams, rivers, hand dug wells and in rare cases boreholes. In Nigeria, only a fraction of the population use improved drinking water sources and sanitation facilities. This makes access to safe and clean drinking-water in the rural areas of Nigeria practically impossible, leading to several health complications. This plays a significant role in diarrhoea related cases in Nigeria, in both adults and children. This research assessed the viability of locally sourced clay from Adamawa State as candidate material for 3D ceramic printed water filter. The assessment involves testing the physical, microstructural and mineralogical properties of the local materials obtained from different areas of the State. A prototype water filer was developed following geometric designs in Autodesk Fusion 360, and trial mix designs of composite materials using 3D printing extrusion process. The result indicates that the clays satisfied minimum plasticity requirements and exhibited satisfactory extrusion property. Heavy metals were not detected in all the clay materials, and composition displayed high amounts of silica and alumina content. The application of 3D printing technology will go a long way towards improving state-of-the-art and refinining the process as well as provide real time opportunity for correcting and editing flaws detected at each stage of prototype development as oppose to the traditional pottery process.

1. Introduction

Nigeria is blessed with large deposits of clay that are not properly harnessed due to lack of awareness on the significance, mineral contents, and how it can be utilized for industrial, laboratory and agriculture [1]. Considering the above, the complex physical, chemical and mineralogical characteristics of clays with unique properties related to their own natural diagenesis, characterization and quality control of each clay is important for the technical performance of local products [2–4]. The microstructure and properties of any ceramic depends on the characteristics of the raw materials and processing parameters [5]. Clay minerals are important inorganic components in soil because they are widely available and have excellent sorption properties due to their large surface area and exchange capacities. The charge present in the structure of clay minerals allows clay to effectively attract metal ions.

In recent years, there has been a surge in global research focused on low-cost filtration and decontamination systems. This is through ceramic filters shaped as a flowerpot, disc, or candle [6]. Filtration process involves the passage of water through a permeable ceramic substance using local resources. This cost-effective approach offers potential for decreasing the rising incidence of waterborne illnesses.

Ceramic water filter (CWF) has been reported [7–11] to provide practical options for effective removal of microorganisms and other contaminants from contaminated water in low - income settings. This filtration medium utilizes the filtering potential of the microscopic pore spaces within clay mineral to filter dirt, sediment, and pathogens out of water. The microbial removal efficiency, water discharge rate, and decontamination capability of ceramic water filters are all contingent upon the firing temperature, particle size, ramming (forming) pressure, and the reaction that takes place during firing [12]. In this set-up, clay is the skeleton element of CWF, and its quality and characteristics directly influence filter quality. Filters produced from a mixture of clay and sieved combustible material can be fabricated into different shapes and it is effective in the removal of microorganisms and reduction of turbidity in contaminated water is achieved by gravity filtration [10]. In the same vein, hybrid ceramic pot water filters (HCPWFs) provide practical options for effectively removing microorganisms and other contaminants from contaminated water in low - income settings. Hybrid water filters combine microscopic pore sizes in ceramics and organic materials to filter dirt, sediment, and pathogens out of water, making this technology appropriate for usage in rural settlements. It is worth mentioning that clay minerals are important inorganic components in soil because of their abundance in nature and their excellent sorption properties because of their large surface area and exchange capacities. They are structured in layered units with single or double tetrahedral silica sheets ($Si_2O_6(OH)_4$) bonded to octahedral aluminum sheets ($Al_2(OH)_6$), commonly at 2:1 or 1:1 ratio [13]. Most minerals are negatively charged, thus exhibiting affinity for cationic species of contaminants including heavy metal ions [14].

There has been a growing interest in the use of clay minerals to remove both inorganic and organic molecules [15-16]. Many recent research investigations have explored the possibilities of using diverse clay – based water filters to enhance the quality of drinking water. The objective was to investigate various alternative materials for treatment, including adsorbents such as clays. Thus, the purpose of this study was to characterize and develop ceramic water filters from clay in Adamawa State – Nigeria for use in 3D printing technology.

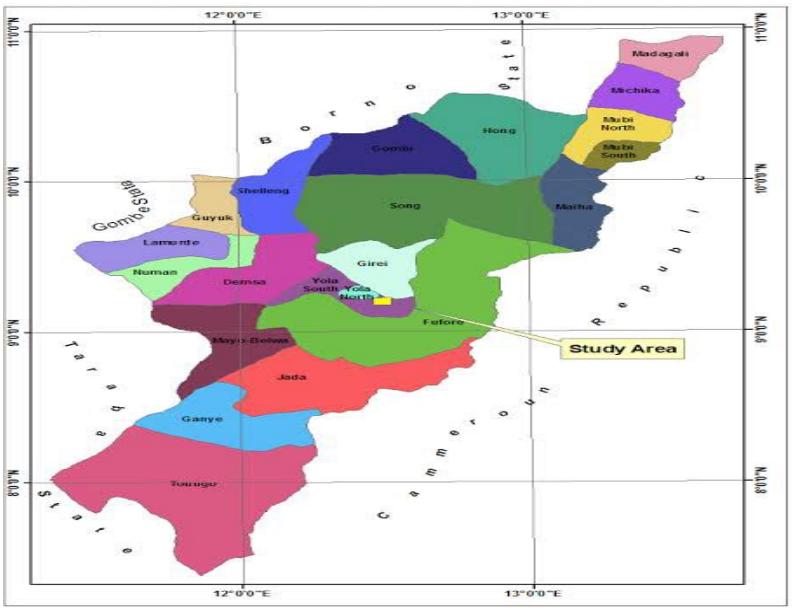


Fig. 1 *Map of Adamawa State showing location of clays (ADSUPDB)*

2. Materials Characterization and Methods

The study location, Adamawa State has an annual average temperature range of 17°C – 43°C, with the coldest and hottest months being January and April respectively, and relative humidity of 16.82% - 77.87% in February and August. The survey of the locations for the sources of clay to be utilized was based on information regarding pottery industrial areas statewide, and the following areas were identified: Fufore, Girei and Hong Local Government Areas. Ngurore, in Yola South Local Government was also identified as per Figure 1, and coordinates of locations is presented in Table 1. Sample collection was done in March with an average monthly temperature of 34°C and relative humidity of 20.06%.

Table 1 *Coordinates of the sources of clay minerals*

S/No	Location	Latitude (N)	Longitude (E)
1.	Fufore	9.20808°	12.6539°
2.	Girei	9.30544°	12.4799°
3.	Hong	10.2360°	12.9483°
4.	Ngurore, Yola South	9.30455°	12.2464°

Samples were collected in plastic buckets, sealed and transported to the laboratory in accordance with BS 5930 for disturbed samples collected in the field. Initial assessment was done on samples collected from the field as per Clause 2 of BS 1377 – 1: 1990, with sample from Hong subjected to further classification under BS 5930. A representative sample was collected for natural moisture content (NMC) determination, the rest were air – dried in the laboratory at a temperature not more than 50°c in line with BS 1377-1 and subsequently placed in an oven at 105 °C for 24 hours. Clay lump samples were placed in trays and air – dried for 72 hours and then hand crushed with mortar and pistil to smaller size particles. This was subsequently dried at 60°C for 24 hours, and a representative sample was collected for the purposed of classification tests for engineering purposes using BS EN 1377 – 2 for the following: natural moisture content, dry density, specific gravity, liquid limit, plastic limit, linear shrinkage, and grain size distribution. Distilled water was used throughout the classification which complied with the requirements of BS 1377 – 3 of not more than 5mg of total dissolved solids per liter of water. Weighing balances used were 2kg capacity readable to 0.1g and 10kg readable to 1g. A drying oven capable of maintaining temperatures of 105°C to 110°C was used. To grind the samples to testing seize, they were placed individually as per location in a Los Angeles Abrasion machine and subsequently sieved to pass No. 200 BS sieve.

Samples were then prepared for chemical and mineralogical characterization. Atomic Absorption Spectroscopy (AAS) was carried out using Accusys 211, Buck Scientific to determine the metallic elements present after digestion of the samples. Sequential Extraction of Heavy Metals from Soil was used for Cd, Cr, Cu, Pb, Fe, Mn, As, Co, Hg, Cu, Ag and Zn. Homogenous and grounded bulk soil samples (0.1 g) after sieving to 75µm are treated with 4 ml of an oxidizing mixture of HNO_3: HCI in ratio 3:1, and 6 ml HF in a Teflon recipient put in a microwave oven (800 w, 4 min; 400 w, 4 min, 800 w, 4 min; 20 min. of ventilation). Recovered sample is then treated with 5.6 g HNO_3 to avoid silica evaporation and diluted to 100 ml by deionized water. Metal concentrations in solution were determined by AAS. Clay characterization was done through X-ray Fluorescence Spectrometry (XRF) with Genius IF Xenemetrix. Fourier transform infrared spectroscopy, ATR-FTIR was also done with Casy 630 Agilent Technologies in the range of 650 – 4000 cm^{-1} to obtain information about the transition of vibrations due to small structural changes. Results were subsequently superimposed on the same graph using Essential FTIR Software for analysis.

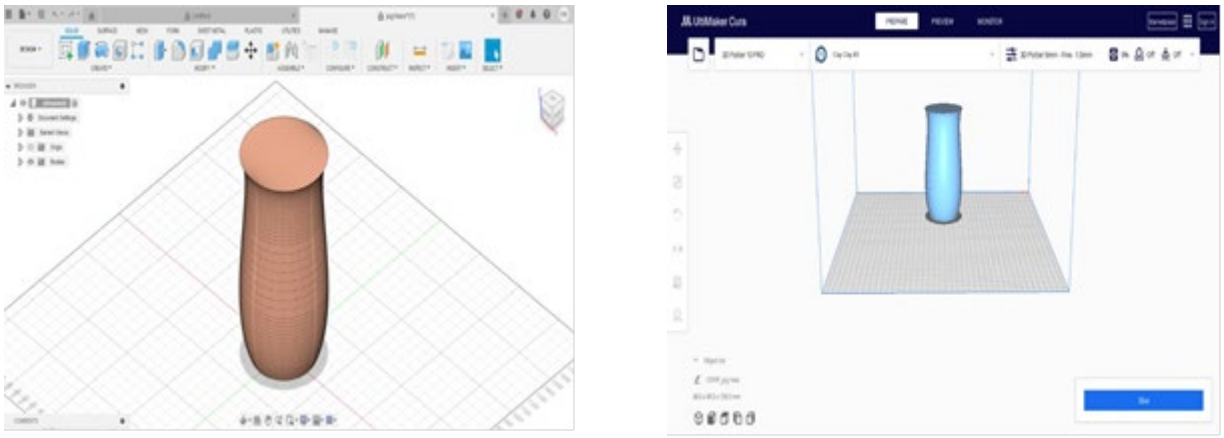
Microstructural analysis was done by energy dispersive spectroscopy (EDS) and using ProX PhenomWorld spectrometer, as well as other aspects relating to its granulometry and density. Rigaku Miniflex 600 was utilized for X – ray diffractometry (XRD) to determine their chemical composition, where the scanning regions of the diffraction were 5–80° on the 2θ angle. This is to allow the development of the blend to be studied over time, by characterization of the crystalline phases.

A representative sample of clay passing No 200 BS sieve was wetted by adding water 50 – 55% by weight to obtain a smooth homogeneous mixture. Clay material was molded into a "throw – up" clay and then remolded until the required mixture for use in 3D printing is obtained. The "throw – up" was fed into a single use 200ml capacity syringe (Figure 2a) for testing before feeding into the extruder of the 3D Printer Potter. This continued until an appropriate mix was obtained and the wall thickness for the vessel was adjusted to prevent collapse by its own weight after printing (Figure 2b). This was subsequently fed to the 3D printer potter. The potter is a commercially available 3D Printer Potter Micro 10 manufactured by 3D Potter, Inc. USA, and comes with duet Wifi, 1000ml extruder, and an aluminum nozzle with size range from 3 – 6mm in diameter. It is operated through a computer interface using a Wifi network "3DP – 10" with an IP address provided by the manufacturer. Input into the printer is made in form of G – Code generated after design of the geometry of choice in CAD

software (Autodesk Fusion 360 in this case) and saved in STL format, and generate the code using a slicer software, where CURA Ultimaker a free software was used as presented in Fig. 3.

(a) **(b)**

Fig. 2 *Test prints using (a) 200ml syringe; (b) 3D potter*



(a) **(b)**

Fig. 3 *Clay filter (a) in CAD software; (b) Cura slicer software*

3. Results

3.1 Physical Characterization and Plasticity

The particle size distribution for clay passing through standard BS sieve as presented in Figure 4 where the clay is well distributed. With the exception of the Hong clay mineral, which shows a noticeable proportion of coarse to medium sand composition, all the rest have fine sand characteristics which are distributed with a significant portion of fine material. Apart from Hong Clay where percentage passing No. 200 sieve is 41.3%, all others are above 80%, and with LL below 50%, it places the material in Borderline cases Low Plasticity (L). Thus, the Unified Soil Classification System (USCS) is SCL classification for Hong Clay.

Table 2 presents the physical characterization results, and Atterberg limits. The results indicated that the soils are highly plastic, except Hong clay, which is Low Plasticity Sand, however, all were A-7-6 by American Association of States Highways Transportation Officials (AASHTO) classification. The result is further assessed using the Holts and Kovacs diagram presented in Figure 5 - 6 based on the classification 0 – 30% Low Plastic Clays, 30 – 50% Moderately Plastic Clays, and 50 – 100% Very Plastic Clays. Fufore, Girei and Hong all fall within the moderately plastic clays region, while Ngurore can be classified as a very plastic clay based on the plot. This can further be interpreted using the clay workability chart where Hong clay can be said to have acceptable

moulding properties with a potential application for Bricks due to low percent passing No. 200 BS sieves. On the other hand, Fufore, Gerei and Ngurore clays displayed sticking consistency and possess optimal moulding properties with a potential application for pottery making. Ngurore clay has a high plasticity index compared to the others.

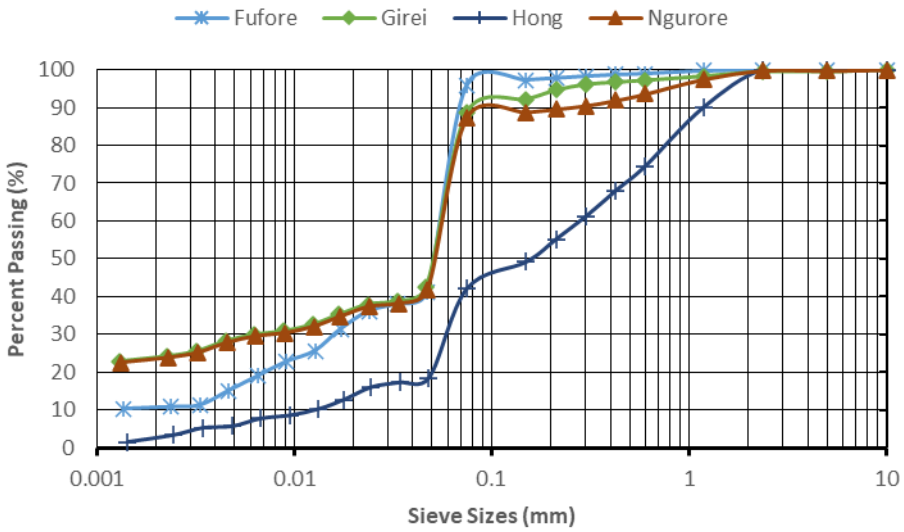


Fig. 4 *Particle size distribution for clay minerals*

Table 2 *Physical characteristics*

Property	Fufore	Gerei	Hong	Ngurore
Percent Passing No. 200 (%)	95.80	88.60	41.30	87.30
Natural Moisture Content (%)	19.87	11.17	4.21	10.92
pH	7.08	4.45	5.68	6.47
Liquid Limit (%)	42.60	50.00	42.60	53.30
Plasticity Limit (%)	14.78	18.65	24.76	13.85
Plasticity Index (%)	27.82	31.35	17.76	39.45
Linear Shrinkage (%)	12.14	11.17	13.04	16.25
Specific Gravity	2.50	2.48	2.42	2.44
Maximum Dry Density(g/cm^3)	1.70	1.67	1.87	1.70
AASHTO Classification	A-7-6	A-7-6	A-7-6	A-7-6
USCS Classification	SC	SC	SCL	SC
Dominant Phase	Quartz	Quartz	Quartz	Quartz

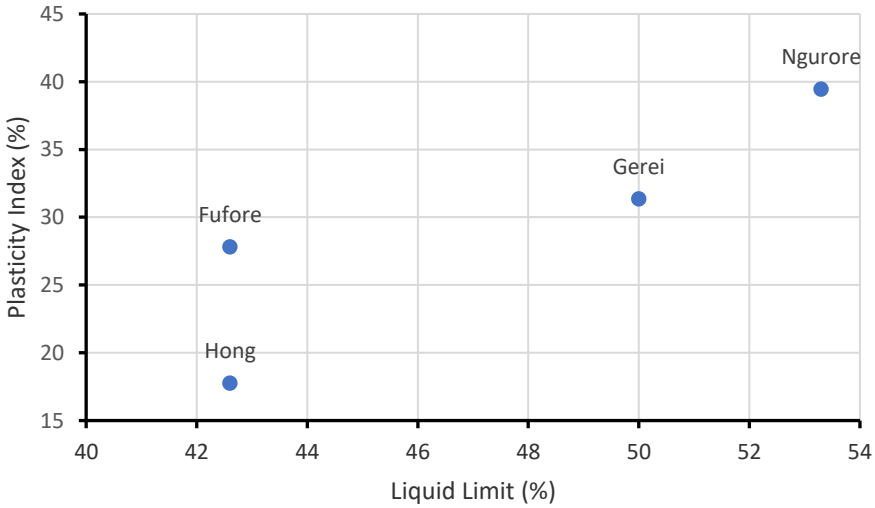


Fig. 5 *Holts and Kovacs Diagram and position of clays studied*

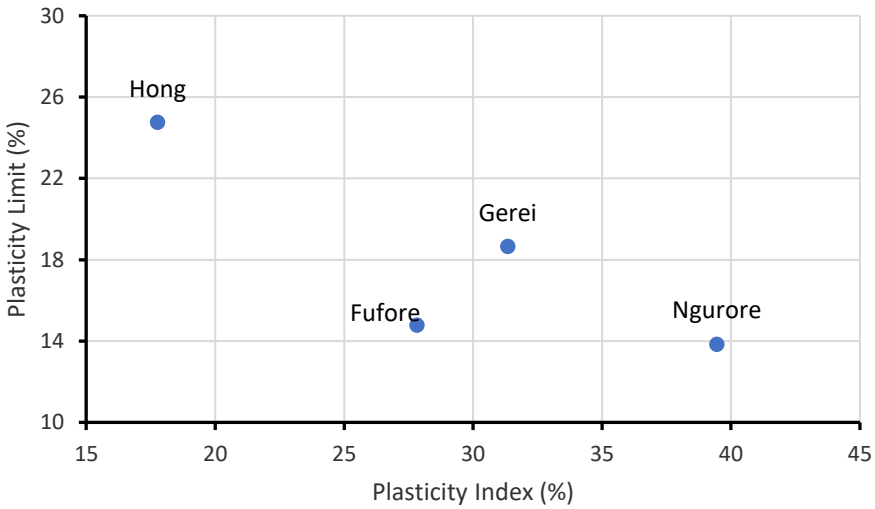


Fig. 6 *Clay workability chart*

3.2 Elemental Analysis

The elemental analysis of some elements for the clay samples is presented in Table 3 compared with World Health Organization (2021) Standards for permissible limits in soils. There is no indication of source of contamination in the parent source of the clays to avoid health complications in the intended product. Silver was not detected, while cadmium, chromium and arsenic were either negligible or in trace quantities. Lead and Chromium are heavy metals with potential toxicity, understanding their concentrations in clay samples is essential, especially if these clays are used for applications involving human exposure (e.g., pottery, building materials).

In Table 4, a summary of quantified EDS results for the elements is presented depicted in Fig. 7–10.

Magnesium content in Hong clay can be advantageous for use in ceramics, cement production, or as a component in refractory materials. This could be due to the geological composition of these regions, thus, having the potential to influence the suitability of these clays for different industrial and agricultural applications based on their elemental composition. High magnesium levels can be used to improve soil quality, particularly in areas deficient in this nutrient. Elements such as Mg, K, Ca, Na and Fe are associated with clay minerals such as quartz and feldspars [17–19].

Table 3 *AAS results summary for some toxic elements (mg/kg)*

Element	Fufore	Gerei	Hong	Ngurore	WHO (2021) Standard
Cadmium (Cd)	0.04	0.02	0.03	0.04	3.00
Chromium (Cr)	0.07	0.02	0.08	0.08	100.00
Iron (Fe)	4,680.00	1,215.00	3,321.00	1,773.00	50,000.00
Lead (Pb)	0.76	0.44	0.60	0.71	100.00
Zinc (Zn)	2.31	0.60	3.64	2.84	300.00
Manganese (Mn)	11.00	3.00	5.04	7.68	2,000.00
Arsenic (As)	0.00	0.00	0.00	0.00	20.00
Cobalt (Co)	0.14	0.06	0.10	0.03	50.00
Mercury (Hg)	0.00	0.00	0.00	0.00	-
Copper (Cu)	0.58	0.14	0.33	0.35	100.00
Silver (Ag)	ND	ND	ND	ND	-

ND = Not Detected

Table 4 *EDS results summary*

	Weight Concentration (%)										
Clay	Si	Al	Fe	K	Ti	Na	Ca	Mg	P	S	Cl
Fufore	55.17	19.32	17.84	3.84	1.52	0.81	0.64	0.63	0.23	T	T
Girei	65.39	27.49	T	5.23	T	0.31	T	1.10	T	T	0.48
Hong	43.64	24.07	22.51	4.06	2.01	0.29	0.91	2.50	T	T	T
Ngurore	54.94	22.39	12.82	5.13	1.32	0.58	1.40	1.42	T	T	T

T = Trace

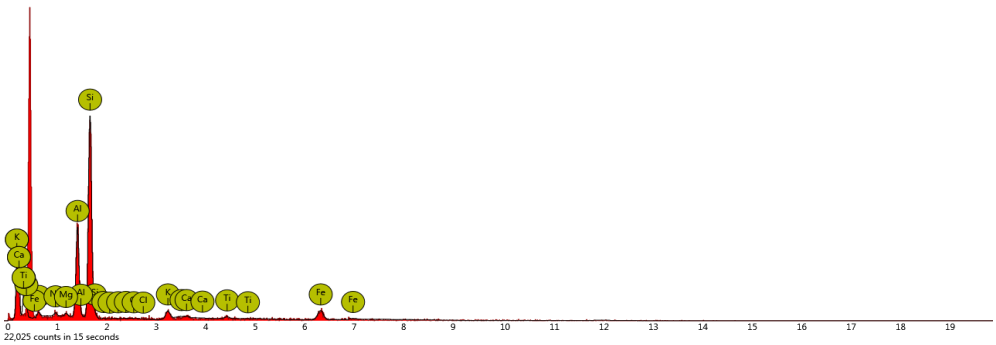


Fig. 7 *EDS at 500x of Fufore clay*

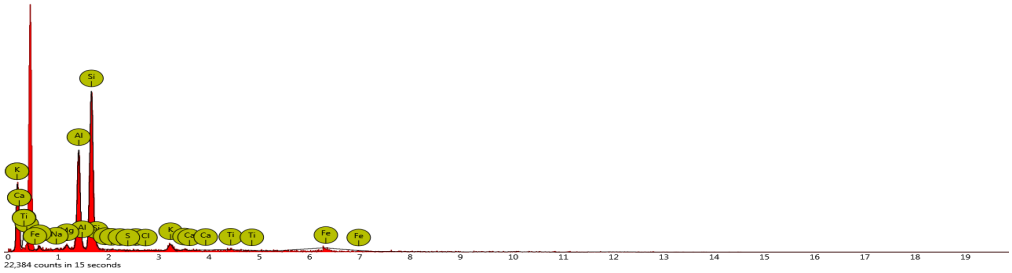


Fig. 8 *EDS at 500x of Gerei clay*

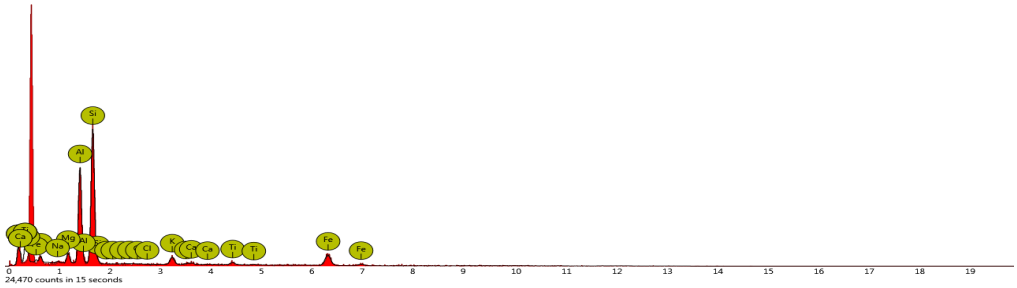


Fig. 9 *EDS at 500x of Hong clay*

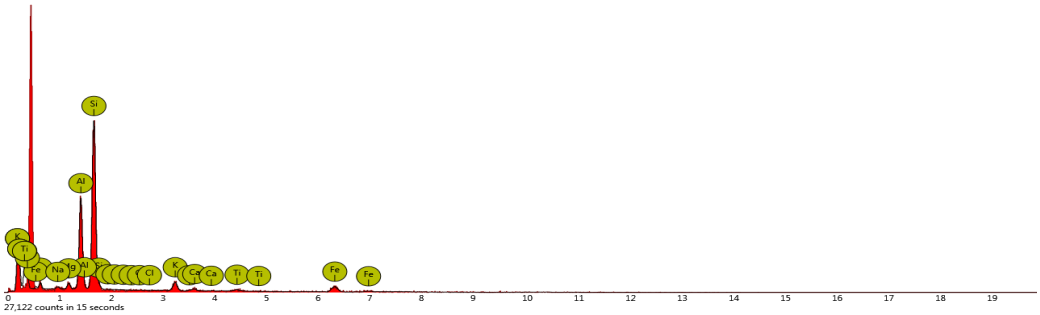


Fig. 10 *EDS at 500x of Ngurore clay*

3.3 Chemical Analysis

The chemical composition of each sample is presented in Table 5 as determined by using EDXRF spectrometer and confirms the high concentration of S_iO_2 and Al_2O_3 in clay. High SiO_2 content may be associated with crystalline phase quartz, and when combined with alumina form alumino silicate [20]. Alumina content is usually associated with Kaolinite, halloysite, and gibbsite [21]. This can be confirmed by XRD.

The classification of residual soils based on silica-sesquioxide ratio (Se) {(S_1O_2/ Al_2O_3 + Fe_2O_3)} is such that laterite with Se values of 1.33 or less, lateritic soil with Se values ranging between 1.33-2.00 and non-lateritic soil with Se values over 2.00 [22]. The Se value was highest in Fufore clay with 3.35 and lowest in Hong clay with value of 2.15. Based on this classification, all the soils are non-lateritic soils.

Table 5 *Oxide composition of clay mineral*

Sample	Fufore (wt.%)	Gerei (wt.%)	Hong (wt.%)	Ngurore (wt.%)
SiO_2	66.97	69.11	57.31	63.13
Al_2O_3	12.93	18.06	18.13	12.76
Fe_2O_3	7.06	3.74	8.57	8.31
CaO	3.35	0.83	2.84	5.77
MgO	0.00	0.00	3.86	0.00
K_2O	4.23	3.49	4.41	4.48
MnO	0.35	0.05	0.24	0.20
SO_3	0.27	0.29	0.34	0.20
Cl	1.68	1.68	1.47	1.60
ZrO_2	0.13	0.12	0.25	0.12
SnO_2	0.15	0.08	0.00	0.00
TiO_2	2.51	2.22	2.20	2.84
Sesquioxide ratio (S_1O_2/ Al_2O_3 + Fe_2O_3)	3.35	3.17	2.15	3.00
Molar Ratio (S_1O_2/ Al_2O_3)	5.18	3.83	3.16	4.95

The molar ratio was highest in Fufore clay and lowest in Hong clay, the least value being 3.16. This is an indication of a kaolinite clay sample with free silica in the form of quartz present [23]. High molar ratio greater than 3 is an indication of the contribution of quartz. Molar Ratio of 3 – 5 % is related to large amount of quartz [21, 24]. High SiO_2 content and low Al_2O_3 is consistent with kaolinite presence, and SiO_2 will be useful in finished products [25]. Silica content above 50% and alumina below 25% is generally consistent with "weak kaolin" [18]. Ngurore clays have a high amount of CaO which is an indication of the presence of limestone, but in combination with Na_2O and K_2O, it can be associated with cations of smectite [15, 26]. In the same way, the presence of K_2O in large amounts indicates the presence of phase containing illite and K-feldspar, which is consistent with what has been observed in [21]. Qualitative analysis by XRD is needed to confirm the mineral phases. K_2O is considered flux agent in clay minerals [25].

3.4 Mineralogical Characterization

FTIR analyses in Figure 11 - 14 shows band from 3697.6 cm^{-1} to 3623.0 cm^{-1} corresponding to O – H stretching bands observed in Kaolinite based clay [27 – 28]. Bands at 3696 cm^{-1} and 3621 cm^{-1} correspond to the presence of kaolinite and Illite clay [29]. This is consistent with the result of qualitative analysis in Table 6 – 9 indicating the presence of these minerals. At 1979.2 cm^{-1} to 1636.3 cm^{-1} the presence of water – bending mode is observed, an indication of water coordinating to the surface of kaolinite [28]. This is characterized by bending hydration water [27, 30]. Al – O bending is seen around the region of 900 cm^{-1} characteristics of aluminosilicate minerals [27, 30]. In Figure 15, the analyses from the different sources were merged and superimposed together, and it shows that they all have the same pattern and characteristics.

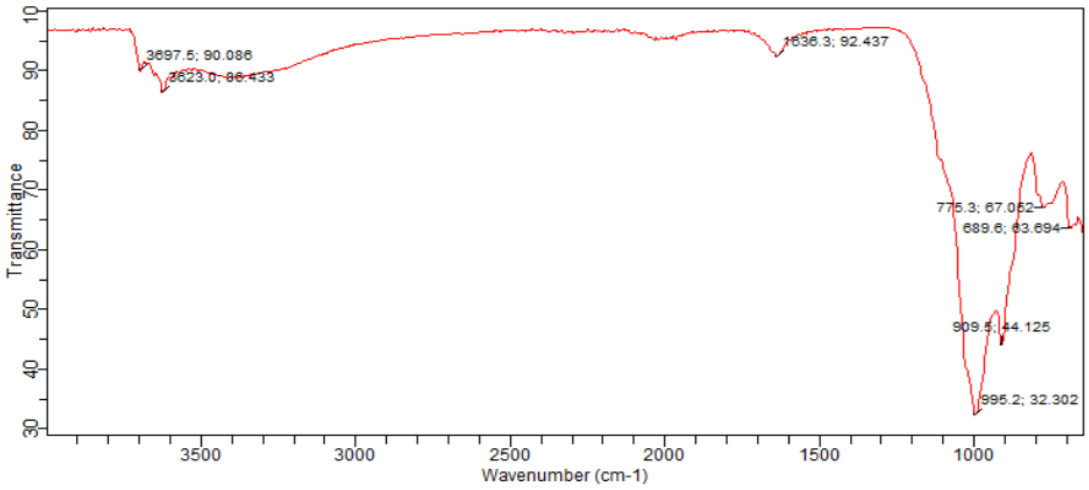


Fig. 11 *FTIR of Fufore clay*

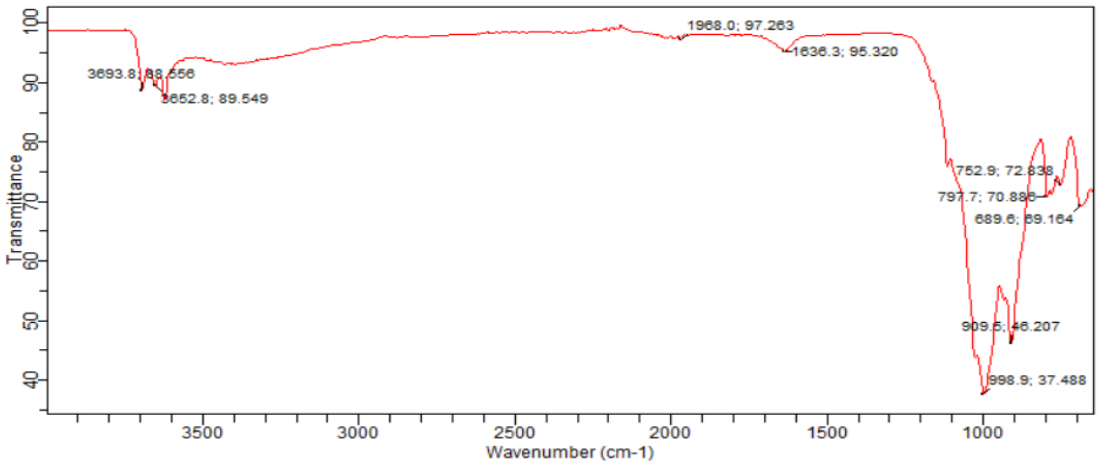


Fig. 12 *FTIR of Girei clay*

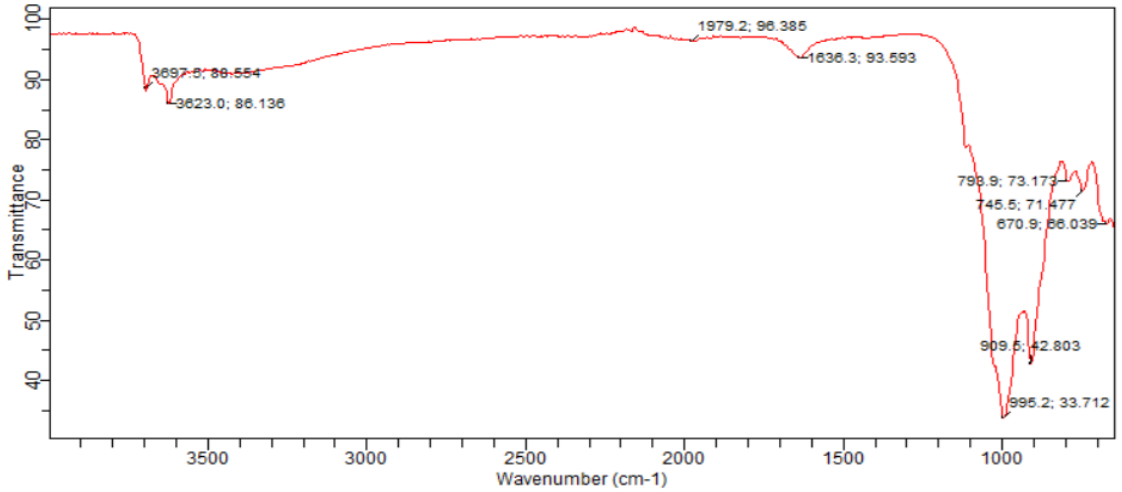


Fig. 13 *FTIR of Hong clay*

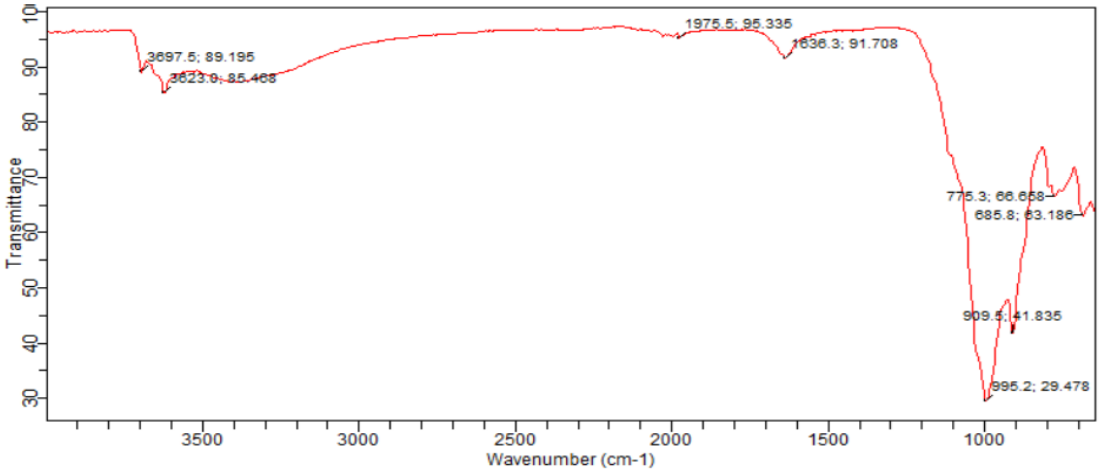


Fig. 14 *FTIR of Ngurore clay*

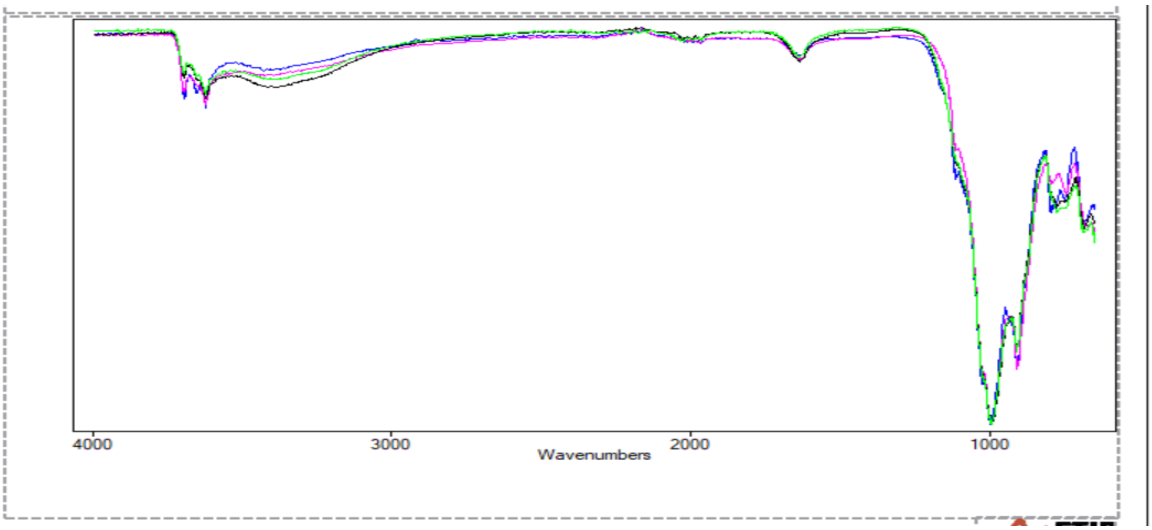


Fig. 15 *Superimposed FTIR analyses for the clay minerals*

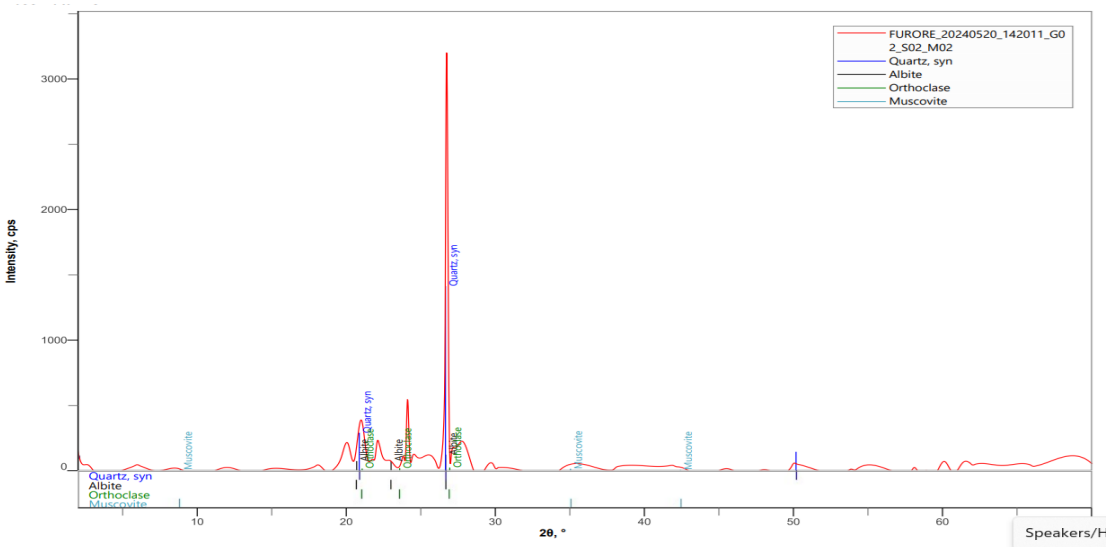


Fig. 16 *XRD analysis for Fufore clays*

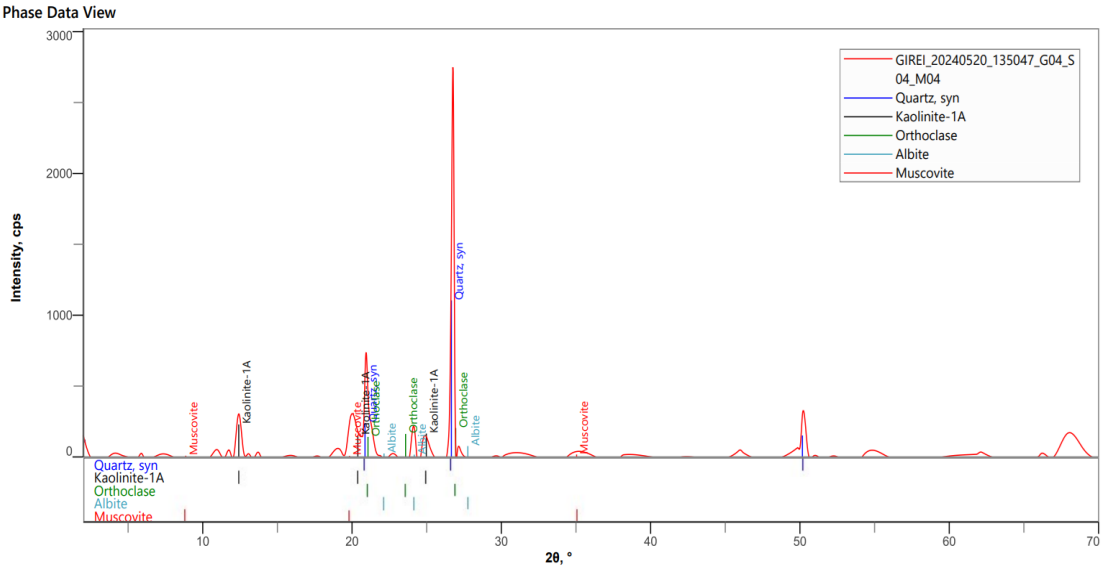


Fig. 17 *XRD analysis for Girei clays*

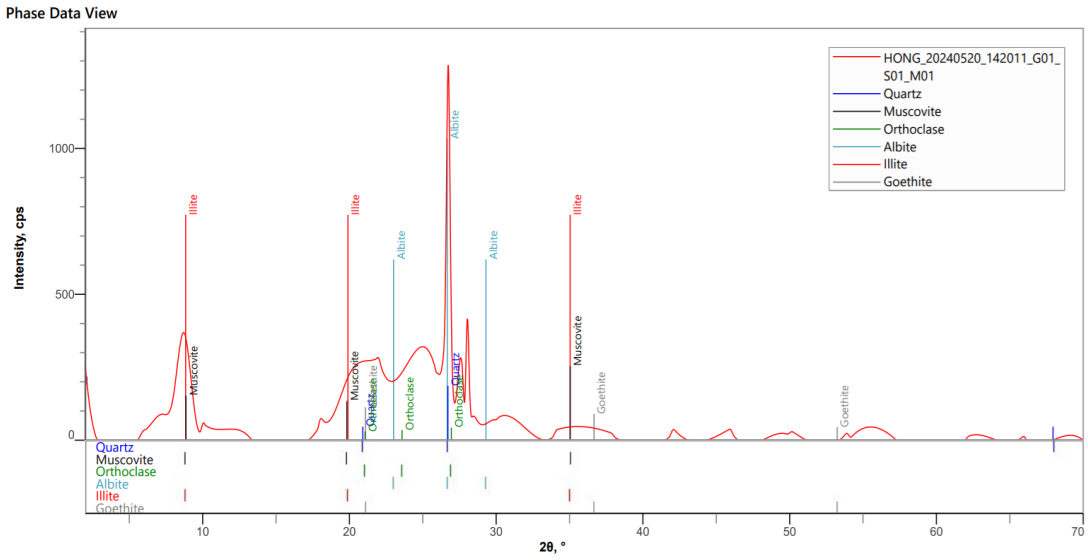


Fig. 18 *XRD analysis for Hong clays*

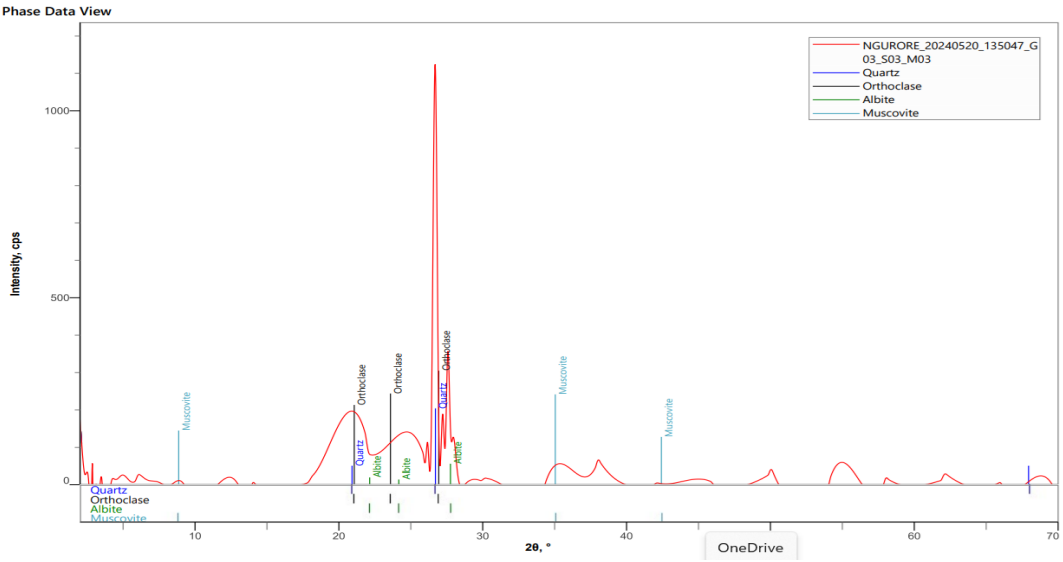


Fig. 19 *XRD analysis for Ngurore clays*

Table 6a *Qualitative analysis of Fufore clay mineral*

No.	Phase Name	Formula	Weight Fraction, (wt. %)
1.	Quartz, syn	SiO_2	66
2.	Albite	$Na\ Al\ Si_3\ O_8$	17
3.	Orthoclase	$K\ Al\ Si_3\ O_8$	16
4.	Muscovite	$H_2\ K\ Al_3\ (SiO_4)_3$	1.1

Table 6b *Peak list for Fufore clay mineral*

No.	2θ°	d, Å	Size, Å	Phase Name
1.	20.8	4.26	24	Quartz, Syn: 1 0 0, Albite 0 3 0
2.	26.715	3.3342	594	Quartz, Syn: 0 1 1, Albite 2 -2 -1
3.	68.5	1.369	32	Quartz, Syn: 0 3 1, Albite 4 1 -5

Table 7a *Qualitative analysis of Girei clay mineral*

No.	Phase Name	Formula	Weight Fraction, (wt. %)
1.	Quartz, syn	SiO_2	56
2.	Kaolinite -1A	$Al_2\ Si_2\ O_5\ (OH)_4$	23
3.	Orthoclase	$K\ Al\ Si_3\ O_3$	17.4
4.	Albite	$Na\ Al\ Si_3\ O_8$	2.7
5.	Muscovite	$H_2\ K\ Al_3\ (SiO_4)_3$	0.5

Table 7b *Peak list for Gerei clay mineral*

No.	2θ°	d, Å	Size, Å	Phase Name
1.	12.45	7.104	391	Kaolinite -1A: 0 0 1
2.	19.94	4.449	153	Kaolinite -1A: 1 -1 0
3.	20.905	4.246	264	Quartz Syn: 1 0 0
4.	26.762	3.3284	494	Quartz, Syn: 1 0 1
5.	50.268	1.8136	512	Quartz, Syn: 1 1 2
6.	68.24	1.3732	104	Quartz, Syn: 2 1 2

Table 8a *Qualitative analysis of Hong clay mineral*

No.	Phase Name	Formula	Weight Fraction, (wt. %)
1.	Quartz, syn	SiO_2	28
2.	Muscovite	H_2 K Al_3 $(SiO_4)_3$	24
3.	Orthoclase	K Al Si_3 O_8	24
4.	Albite	Na Al Si_3 O_8	10.5
5.	Illite	2 K_2O .3Mg .Al_2O_3 .24…	6.8
6.	Goethite	Fe_2 O_3 .H_20	7.4

Table 8b *Peak list for Hong clay mineral*

No.	2θ°	d, Å	Size, Å	Phase Name
1.	8.90	9.93	127	Muscovite: 0 0 2
2.	21.1	4.21	22	Quartz: 1 0 0
3.	24.8	3.59	32	Quartz: 1 0 1
4.	26.711	3.335	565	Albite: 2 -2 1

Table 9a *Qualitative analysis of Ngurore clay mineral*

No.	Phase Name	Formula	Weight Fraction, (wt. %)
1.	Quartz, syn	SiO_2	62
2.	Orthoclase	K Al Si_3 O_8	15.6
3.	Albite	Na Al Si_3 O_8	11
4.	Muscovite	H_2 K Al_3 $(SiO_4)_3$	11

Table 9b *Peak list for Ngurore clay mineral*

No.	2θ°	d, Å	Size, Å	Phase Name
1.	20.6	4.31	23	Quartz: 1 0 0
2.	26.69	3.337	351	Quartz: 1 0 1

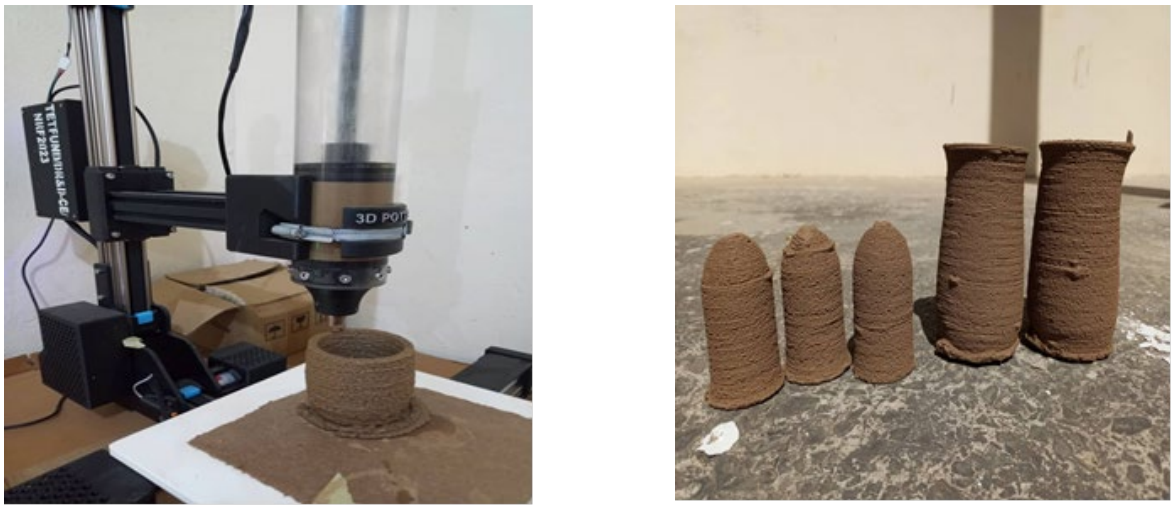
XRD analyses in Figure 16 - 19 show an excess amount of quartz with some alumina present, and Table 7 – 9 corroborates this assertion with quantitative XRD results. The major peaks in the XRD results correspond to quartz (SiO_2) phase, and feldspars in the form of orthoclase (K Al Si_3 O_8) in all the samples. Excess SiO_2 is attributed to the presence of quartz and is consistent with the molar ratios of 3 and above. Relatively high intensity of quartz peaks indicates significant presence of free silica [25]. Phase names, peaks and miller indices are consistent with what has been reported by Mitchell [31], and the results also show abundance of Al_2O_3, an indication of a kaolinite clay, confirmed from the XRD analysis in Table 7a in Girei clay. Hong clay exhibited higher Iron content in Table 4 and Iron Oxide content in Table 5. Similarly, quantitative analyses in Hong show the contribution of illite and Goethite. This is responsible for the lighter colour in ceramics, responsible for the reddish colour after firing [25]. High Iron Oxide content has been attributed to application in structural engineering because of mechanical strength [32].

3.5 Prototype 3D Printed Products

As highlighted in Section 2 and depicted in Figure 2, the consistency of the mix is tested by feeding the proportion into a 200ml syringe to test the extrusion process. This is followed by manual printing of a model shape on a ceiling board as seen in Figure 20 below. This is to monitor and adjust the consistency of the mix constituents in preparation for feeding into the 3D printer extruder. Preliminary batches of mixes fed into the 3D printer either collapsed under its own weight in which case the mix was too fluid, or the extruder could not print leading to the overheating, an indication the mix consistency was stiff (Figure 2b). Printing time depends on the number of layers the slicer software divided the model during generation of the G-code, and can last up to an hour, with the pauses intermittently to allow the printer cools down in the event of overheating, as would be indicated on the 3D Printer Software Interface (Figure 21). The operator monitors the printing process up to the finishing surface, and the prototype is allowed to air-dry (Figure 22a) inside the laboratory from 48 – 72 hours, before it is finally brought for sintering at 800°C. After firing, the distinct Reddish – Brown colour of the

prototype filter is evident in Figure 22b, consistent with the presence of Iron Oxide in Table 5 and Goethite phase in Table 8a mentioned earlier.

Fig. 20 *Manual extrusion process using a syringe to test consistency*



(a) **(b)**

Fig. 21 *Clay filter (a) during 3D printing; (b) candle and jug prototype*

(a) **(b)**

Fig. 22 *Clay filter (a) after air drying; (b) after firing at 800°c*

4. Conclusions

The characterization of locally sourced clay materials from Adamawa State as candidate material for 3D printing application was explored, and the following conclusions were drawn:

- All selected clays from four different locations in the study area were characterized as A – 7 – 6 based on AASHTO. The classification using USCS confirmed clay from three sources as SC and a clay from the fourth source was classified SCL due to significant percentage of coarse particle content.
- The clay minerals satisfied the minimum plasticity requirements. However, further qualitative analysis based on Holts and Kovacs criteria, confirmed that clay sample from Ngurore was "very plastic clay" while the rest are "moderately plastic."
- There was absence of heavy metals that could discredit all the clay materials as view of WHO standard permissible limits in soils.
- All the clay materials displayed high amounts of silica and alumina resulting in molar ratio above 3, an indication of kaolinite clay mineral with free silica (quartz).
- Clay samples from three sources including Fufore, Girei and Ngurore exhibited high extrusion properties, while clay sample from Hong exhibited satisfactory extrusion behavior due to its moderately plastic properties.
- Clay from Hong showed a reddish-brown coloration after firing indicating and confirming its high Iron Oxide content and Goethite phase.

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Conflict of Interest

Authors declare that there is no conflict of interest regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design:*** *A. U. Abubakar, I. A. Yahya;* ***data collection:*** *A. Idris, M. S. Nadro, A. T. Buni, H. Mamman, H. M. Yusuf, I. S. Diwa;* ***analysis and interpretation of results:*** *E. W. Gadzama, H. A. Mahmoud, I. A. Yahya, S. Bala, S. Abdullahi, A. U. Abubakar;* ***draft manuscript preparation:*** *I. A. Yahya, M. B. Yawale, Y. Aliyu, M. L. Tumba, T. Akcaoglu, E. W. Gadzama, A. U. Abubakar. All authors reviewed the results and approved the final version of the manuscript.*

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