Plasticity characterization of certain Nigeria clay minerals for their application in ceramic water filters

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Abstract
Plasticity is an essential property of clay that determines its suitability for water filtration. There are no published works on the plastic behavior of clays from the study locations. The plastic behavior of seven Nigerian clays was examined using plasticity indices and compressive stress parameters in relation to chemical compositions and moisture content. The objective is to determine plastic behavior of some Nigerian clays and their suitability in production of Expanded Clay Aggregates (ECA) for water filters. Compressive stresses and deformation parameters were determined experimentally and compared theoretically. Atterberg limits (D 4318) were used to determine the plasticity indices. Chemical compositions of the samples were examined with XRF and correlated with plasticity and mineral contents of the clays. The clays are aluminosilicates with SiO₂/Al₂O₃ ratio of 1.61 to 3.03 and plastic indices of 8 to 49. Low plastic indices (8–11) and low compressive stresses parameters were observed for kaolinite clays (0.002 MPa) due to their low affinity for water while zeolite rich clays showed high plastic indices (46 and 49) for Obowo and Minna and sharp difference in their compressive stresses parameters (0.15 and 0.03 MPa) at optimum moisture contents of 57% and 53%, respectively. Despite varying moisture content, chemical and mineral compositions, all curves showed similar trends apart from

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kaolinites at 40% moisture content. Relationships exist among microstructural properties, chemical composition, moisture content, compressive strength, and plasticity indices of the clays. The plastic behaviors show they are suitable for development of ECA for water filters.

**Keywords**
Nigerian clay, Atterberg limits, compression test, plasticity, water purification, filtration media

**Introduction**

Plasticity is an essential property of clay that makes it workable or moldable when tempered with water.\(^1,2\) Clay can be transformed into shapes without any tendency to return to its elastic state after moderate applied pressure is withdrawn.\(^2-4\) The study of plasticity of clay and other porous materials is of great value to engineering practices and industrial applications.\(^5\) Artisans and potters rely on long aged experience and expertise during determination of clay plasticity. This is usually done by making a coil with clay and rolling it around one’s finger. Plastic clay will not break while non-plastic clay will break easily.\(^6\) The behaviors of clay, feel at touch or the potter’s wheel are unreliable and do not give dependable plasticity values. Although work experience is valuable, however relying much on experience based learning is arbitrary and not in tune with pace and age of information technology. It is pertinent that systematic approaches which depend on practical measurements based on quantitative expressions be adopted to test the plasticity of clay and categorize different grades of clay samples with different values. Such information becomes an asset to the user who relies on the specifications before purchasing for use or trial purpose.

Different clay plasticity measurements have been modeled or studied and reported by researchers though with discrepancies in concepts. Perhaps that is why there is no one acceptable technique that can be crystallized into a definition.\(^7\) The Atterberg method uses the plasticity index which is determined by varying physical behavior of clay with different moisture content. This method has recorded fair success in comparison with other methods that have been reported although with much judgment to the person conducting the test.\(^4,6-9\) Clay plasticity is influenced by lots of factors such as the nature of clay (primary or secondary formation), aging, weathering, mineral compositions and their crystal structures, moisture content, and particle size distribution.\(^2,10-12\) Different constituents, structures, and wide range of application make it difficult to have a generic reference on clay science.\(^6\) Plasticity measurements and behavior of extruded clay together with plastic behavior of porous material in any given situation is best analyzed by yield criterion (stress at which plastic deformation is initiated or onset of plastic flow) and constitutive relations.\(^5\) Khlystov et al.\(^13\) described the uniaxial tension and compression of metals, alloys, and ceramics to determine their Young’s Modulus, Yield Stress, Ultimate Tensile Strength, and Elastic Strain which are used to determine the elasto-plastic behaviors of materials. Measuring and modeling the plasticity of clays subjected to compression loads have been examined and insights on extrusion up-scales/important guidelines and properties were obtained.\(^2,7,14-16\)
Geotechnical properties of some of Nigerian lateritic soils and clay minerals have been reported although with major applications in building and construction, recharge sites for regional aquifer, bacterial removal from water, and adsorption studies of heavy metals.\textsuperscript{17–25} Good results were obtained when the materials are modified with additives.

This paper understudies the constitutive plastic behaviors of clay samples in Nigeria under compression loads and the plasticity indices in relation to their moisture content, chemical compositions, and mineral contents. Although some of the samples from the same study areas have been characterized by physio-chemical means, there are no published works on the plasticity of the samples of study. The presented results are extension of multiple characterization results which have already been published by Ihekweme et al.\textsuperscript{26} The insights and possible adjustment in the development of Expanded Clay Aggregates for water purification purposes was also presented.

**Materials and methods**

**Theoretical considerations**

The clay is considered an isotropic and porous material. The loading condition is an axial symmetry under compression. Every small element deforms homogenously to the same degree at the same instant. De Andrade et al.'s\textsuperscript{2} theoretical parts equations (equations (1) and (2)) were adopted for these measurements to evaluate related properties such as force, instantaneous radius, and other variables which affect the plasticity of given ceramic bodies.

\[
\sigma_z = \gamma \exp \left( \frac{2\mu}{h} \left( \frac{r_f - r_0}{2} \right) \right)
\]

\[
F = \int_0^{r_f} 2\pi \sigma_z \partial_r
\]

Where \(\sigma_z\) is the axial stress component; \(F\), axial force; \(\mu\), coefficient of friction; \(\gamma\), effective compressive stress; \(r_f\), radius after compression; \(r_0\), radius before compression; \(h\), height of sample; and \(r\), radial direction.

**Experimental methods**

Raw clay samples were collected from seven locations within the south-east, south-south, and north-central of Nigeria. Samples were named after their locations, that is Afuze, Ihitte, Kutugi, Minna, Nsu, Oboro, and Obowo for ease of identification. Initial preparations were done as stated by Ihekweme et al.\textsuperscript{26} X-ray result used in discussion was adapted from the earlier publication on the same samples characterization. The chemical compositions were determined by X-ray fluorescence spectroscopy (XRF), (model: XRFEDX3600B).
For the plasticity experimental procedure, 40 g of each sample was mixed with varying milliliter of water according to its moisture content\textsuperscript{26} and 40% moisture content for comparative analysis. The mixtures were stored in zip lock poly bags for 24 h for moisture homogeneity. A polyvinyl chloride (PVC) mold of dimensions 20.42 mm diameter and 30.06 mm height was constructed and used to produce the ceramic bodies. Measurements were taken using a vernier caliper (Model: Mitutoyo series 530). The ceramic bodies were loaded non-eccentrically and subjected to compressive forces at the rate of 3 mm/min with Instron model 3345 and a 5 kN load cell. Digital photographs were taken at intervals with Nikon D3400 digital camera and the concurrent diameter measurements were determined using image analysis tool (Image Tool). The instantaneous radius and applied forces were determined using equations (1) and (2). Experimental data was plotted and fitted non-linearly to produce the theoretical curves. The coefficient of friction is assumed to be constant because of the difficulty in its determination and it poses no significant threat in the analysis.\textsuperscript{2} The pictorial setup and deformation step is shown in Figure 1. Standard test methods D 4318\textsuperscript{27} were used to determine the Atterberg limits.

Results and discussion

Chemical and mineral compositions

The samples are predominantly aluminosilicates as shown in Table 1. Ihitte and Minna have very high content of iron while the SiO\textsubscript{2}/Al\textsubscript{2}O\textsubscript{3} ratio for all the samples ranged from 1.61 to 3.03. Kaolin is the most prevalent clay mineral for Afuze and Kutigi and also present in reasonable quantities in Ihitte and Nsu (Table 2). Quartz is the non-clay mineral in Nsu and Oboro and also high in Afuze. Chlorite clay mineral is prevalent in Ihitte. Illite and zeolite minerals were discerned for Minna. The loss on ignition is highest in Obowo and lowest in Kutigi. This may be attributed to their organic matter and impurities contents which may be traced to their formation and weathering. The ratio of silica to sesquioxides (Fe, Mn, Al, Ti) are

![Figure 1. Compressive deformation on Minna clay sample at 53% moisture content: (a) point of load, (b) intermediate load, and (c) high load.](image-url)
1.91, 1.36, 1.50, 0.99, 2.50, 1.69, and 1.31 for Afuze, Ihitte, Kutigi, Minna, Nsu, Oboro, and Obowo, respectively. According to Baver, the adsorptive capacity of water molecules on colloidal surfaces decreases with decrease in silica/sesquioxide ratios, however this was not observed in all cases for samples of study. Other factors like the mineral compositions, high content of organic matters, structure, formation, and weathering might have contributed in the differences.

The alkali metal oxide, K₂O (flux material) is highest for Minna with 3.93 value followed by Oboro at 1.11 and lowest in Afuze at 0.55. The alkaline earth metal oxide, CaO (auxiliary flux material) during firing and cooling are low in all the samples. This may require an addition of fluxes when producing the expanded clay to bind the clay particles together to avoid disintegration. Transition metal oxides (TiO₂ and ZnO) are present in small quantities too. The predominant Kaolin samples (Afuze and Kutigi) recorded the highest values for TiO₂ which are necessary for photo catalytic processes during water treatment, however they do not contain MnO which aid the scavenging of heavy metals within an aqueous system especially in contaminated water.

| Oxides  | Afuze | Ihitte | Kutigi | Minna | Nsu | Oboro | Obowo |
|---------|-------|--------|--------|-------|-----|-------|-------|
| SiO₂    | 60.52 | 51.84  | 57.31  | 41.94 | 67.07 | 54.91 | 48.93 |
| Al₂O₃   | 24.80 | 27.07  | 34.74  | 24.51 | 22.16 | 28.38 | 30.48 |
| Fe₂O₃   | 5.19  | 10.13  | 2.36   | 17.14 | 3.91 | 3.17  | 5.85  |
| K₂O     | 0.55  | 0.60   | 0.67   | 3.93  | 0.56 | 1.11  | 0.80  |
| P₂O₅    | 0.35  | 0.22   | 0.24   | 0.19  | 0.29 | 0.30  | 0.24  |
| CaO     | 0.13  | 0.12   | 0.13   | 0.40  | 0.09 | 0.13  | 0.12  |
| TiO₂    | 1.72  | 1.02   | 1.19   | 0.54  | 0.80 | 0.98  | 0.89  |
| MnO     | –     | 0.02   | –      | 0.10  | 0.01 | 0.01  | 0.02  |
| ZnO     | 0.13  | 0.14   | 0.15   | 0.13  | 0.14 | 0.15  | 0.14  |
| LOI*    | 6.60  | 8.83   | 3.20   | 11.12 | 4.95 | 10.86 | 12.20 |
| Si/Al   | 2.4   | 1.92   | 1.65   | 1.71  | 3.03 | 1.93  | 1.61  |

*LOI: loss on ignition @ 1000°C.

Table 2. Mass % phase distribution of clays obtained by XRD.

| Name     | Afuze | Ihitte | Kutigi | Minna | Nsu | Oboro | Obowo |
|----------|-------|--------|--------|-------|-----|-------|-------|
| Kaolin   | 62.56 | 18.42  | 77.37  | –     | 19.86 | –     | –     |
| Illite/Mica | –     | –      | –      | 30.15 | –   | –     | –     |
| Quartz   | 37.44 | –      | –      | –     | 80.14 | 49.72 | –     |
| Chlorite | –     | 45.93  | –      | –     | –   | –     | –     |
| Zeolite  | –     | –      | 65.85  | –     | –   | 92.08 | –     |
| Others   | –     | 36.65  | 22.63  | –     | –   | 50.28 | 7.92  |

Adapted from Ihekweme et al. with permission from Elsevier.
Compressive stress distributions and plastic behavior

The plasticity results (Table 3) indicate that the liquid and plastic limits range from 29% and 21% for Afuze to 98% and 52% for Obowo, respectively. Minna has the highest plastic index of 49% while Afuze possessed the lowest plastic index of 8%. From the fore-going, Afuze and Kutigi can be classified as low plastic clays because their liquid limits (LL) are 30% and below.\textsuperscript{7,33,35–36} This result is not far-fetched as they are predominantly kaolin (Table 2) which has low affinity for water, low shrinkage, and swelling behavior. Similar findings have been reported.\textsuperscript{21,37} LL for Nsu is 50% which classifies it as having intermediate plasticity or plastic kaolin.\textsuperscript{35} Ihitte falls within the range of 50%–70% with classification as high plasticity. This is due to high content of chlorite and its high affinity for water.\textsuperscript{36} Oboro possessed very high plasticity for falling in between 70% and 90% while Minna and Obowo are classified as extreme high plastic clay for possessing LL above 90%. The Plasticity indices for all samples are between 8% for Afuze and 49% for Minna and increases in the order of; illite > chlorite > Kaolinite.

The values of compressive stresses parameters from the compression test at different moisture content are presented in Table 4. Obowo has the highest compressive stress of 0.15 MPa at 57% moisture while Afuze and Kutigi showed the lowest compressive stress of 0.02 MPa. However, at 40% moisture content, Minna exhibited the highest compressive stress of 0.06 MPa. Despite moisture content, chemical and mineral compositions, all curves showed similar trends apart from kaolinites at 40% moisture content. Such results were comparable to those reported in literature.\textsuperscript{2,38,39} The values presented showed that moisture content, chemical and mineral compositions exert much influence on clay plasticity (compressive stress parameter). Equipped with such correlations, possible adjustment can be made to obtain desired results.

The different forces with radial variations during compression test of the samples are presented in Figures 2 and 3. The dots are the experimental points which fitted into theoretical curves of plasticity.

During compression, internal stress distribution affects the physical structure of the samples with a frictional force between the two opposing plates and the clay sample (Figure 1(a)). Water molecules which are bi-polar are attracted to the tiny particles of clay which are negatively charged and keeps them together by means

### Table 3. Atterberg plastic parameters.

| Sample   | Liquid limits (%) | Plastic limits (%) | Plastic index |
|----------|-------------------|--------------------|---------------|
| Afuze    | 29                | 21                 | 8             |
| Ihitte   | 63                | 41                 | 22            |
| Kutigi   | 30                | 19                 | 11            |
| Minna    | 91                | 42                 | 49            |
| Nsu      | 50                | 36                 | 14            |
| Oboro    | 82                | 44                 | 38            |
| Obowo    | 98                | 52                 | 46            |
of capillary forces. These forces are overpowered upon exertion of more stress causing the water molecules to recede and allow the inflow of air into the pores of the ceramic body (Figure 1(b)). Deformation occurred gradually until crack was initiated (Figure 1(c)) on the side walls of the samples which propagated as the load increased (barreling). This was possible because the samples were not confined.

Table 4. Compressive stresses at different moisture contents.

| Clay   | Moisture (%) | Compressive stress (MPa) | Coefficient of friction |
|--------|--------------|--------------------------|-------------------------|
| Afuze  | 27           | 0.02                     | 0.15                    |
|        | 40           |                          |                         |
| Ihitte | 45           | 0.05                     | 0.15                    |
|        | 40           | 0.04                     | 0.15                    |
| Kutigi | 27           | 0.02                     | 0.15                    |
|        | 40           |                          |                         |
| Minna  | 53           | 0.03                     | 0.15                    |
|        | 40           | 0.06                     | 0.15                    |
| Nsu    | 44           | 0.04                     | 0.15                    |
|        | 40           | 0.03                     | 0.15                    |
| Oboro  | 52           | 0.11                     | 0.15                    |
|        | 40           | 0.03                     | 0.15                    |
| Obowo  | 57           | 0.15                     | 0.15                    |
|        | 40           | 0.03                     | 0.15                    |

The moisture contents of 27, 45, 27, 53, 44, 52, and 57 for Afuze, Ihitte, Kutigi, Minna, Nsu, Oboro, and Obowo were adapted from Ihekweme et al. with permission from Elsevier.

Figure 2. Curves of applied force versus radial changes; Rf-R0 are radius after and before compression respectively, the lines represent theoretical fitted curves while the points are experimental data (a) varying moisture content; Obowo has the highest plasticity while kutigi possessed the lowest plasticity (b) 40% moisture content; Minna possessed the highest plasticity curve, Ihitte was more plastic than the others while Obowo has the lowest plasticity outside Kutigi and Afuze that are non-plastic at 40% moisture content.
Figure 3. Comparative presentation of the clay minerals with varying and 40% moisture content of individual same clay mineral. Rf-R0 represents the radius after and before compression respectively; the lines are theoretical fitted curves while the dots are experimental data. Afuze and Kutigi (a and c) showed reasonable plasticity at 27% moisture content, (b) Ihitte exhibited more plasticity at 46% moisture content with lesser force than at 40% moisture content, (d) Minna showed more strength at 40% but lower plasticity, it exhibited high plasticity at 53% moisture content, (e) Nsu was more plastic at 44% than at 40%, (f) Oboro was more plastic at 52% than at 40%, and (g) Obowo was more plastic at 57% moisture content however it utilized higher force to achieve that.
in a die. Although soil exhibit both elastic and plastic behavior, elastic strain was excluded due to insignificant elastic limit which is of no interest in the study.

The behavior exhibited (Figures 2 and 3) were in agreement with stress-strain curve for elasto-plastic deformation of porous ceramic materials. Straight line part of the graph showed elastic deformation while the curve portion showed plastic deformation. Early cracking was observed for Kutigi and Afuze samples at 27% moisture content which indicated their low-plastic nature. They are non-sedimentary kaolin and lack organic substances which improve the plasticity of clay. Kaolinite clay requires more time for water to penetrate some of the basal plane surfaces to a fix position, an act that aids the development of plasticity and bonding strength.41 However, time factor was not included in the analysis. More so, at moisture content above 27%, they exceeded their plastic limits and became difficult to mold corroborating the earlier report by Baran et al.42 and Barnes.43 Such was observed at 40% moisture (Figure 2(b)) content where they exhibited extreme humidity and do not possess enough green strength for the compression test. Obviously, their optimum water content had been exceeded causing aggregates saturation and pores to be partly filled by free water, hence are represented by the straight line. Minna and Ihitte showed high plastic strength which may be attributed to their high content of iron and swelling potentials due to dry density.44 Fe atoms are known to affect the hardening and shrinkage of clay minerals because they act as an internal source of heat and had effect on reaction kinetics. They also possessed high content of zeolites with special properties45 and chlorite respectively which has high affinity for water.

Different moisture content induce significant changes in plastic behavior of cylindrical clay bodies (Figure 2(a)) due to the rearrangement of clusters of adjacent grains guiding the laminar properties in a flow direction and the separation of attractive and repulsive forces between the clay particles. Obowo showed the highest plasticity, that is, highest curve while Kutigi showed the lowest plasticity with the lowest curve.14 This also conforms to the Atterberg and moisture content results.

Producing Expanded Clay Aggregates with the low plastic clay will require the addition of processing binders like sodium salt, Zusoplast C28 or any compatible plasticizers to extend the plastic region.2,39 This can however be overcome by having a blend of high plastic clay mineral with non-plastic clay mineral since it has been reported that, the adsorption of heavy metals depends on the plasticity of fine-grained soil.20

**Conclusion**

Direct measurement of clay plasticity by moisture content evaluation and the relationship between the applied force and the resultant deformation has been presented. Compressive test has proven to be suitable to characterize the plastic behavior of extruded clay. Good correlation between the experimental points and theoretical curves were obtained. Optimum water content of 27%, 46%, 27%,
53%, 44%, 52%, and 57% are recommended for Afuze, Ihitte, Kutigi, Minna, Nsu, Oboro, and Obowo, respectively based on the obtained result. Compressive stresses and plastic indices are parameters to predict plasticity in relation with chemical and mineral compositions and moisture content. Thus the clay material properties can be optimized using this veritable tool for wide industrial applications. Low plastic clay will require addition of plasticizers to bind the particles or working with optimum moisture content to extend their plastic regions. Good plasticity occurs when the material do not present early crack or extreme humidity. From the perspective of water filter application and in order to eliminate the use of chemicals in treatment plants, it is recommended that the low plastic clay (Kutigi) be mixed with high plastic clay (Minna) to obtain balanced plasticity that will withstand the frothing pressure during backwashing in treatment plants or water bottles when using Expanded Clay Aggregates. These two samples were also favored in the multiple characterizations previously reported by same authors. Future work will use the selected clay and associated minerals to produce Expanded Clay Aggregates for ceramic water filters. This will be tested on contaminants removal of microbes, organic contaminants, and adsorption studies of heavy metals.

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Data availability
The data that support the findings of this study is available from the corresponding author, Ihekweme GO, upon request.

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