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Characterization and Industrial Applications of Wushishi Clay Deposit

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Abstract: The Wushishi clay deposit in Niger State, Nigeria, was examined to understand its possible industrial uses. The clay samples were randomly selected from different locations. They Clay samples appeared dry and had a dark gray color. The research focused on properties like drying and firing behavior, apparent porosity, bulk density, water absorption capacity, plasticity, modulus of rupture, shrinkage, and chemical composition. The Chemical screening revealed a composition of 59.8% SiO_2, 17.08% Al_2O_3, 2.54% Fe_2O_3, 0.3% MgO, 4.39% Na_2O, 2.54% K_2O, and 1.5% CaO. The clay exhibited moderate plasticity of approximately 2.83 kgf/cm^2, moderate shrinkage of 10.5%, and a modulus of rupture (strength) ranging from 22.56 to 34.86kgf/cm^2 at different temperatures. The clay deposit equally demonstrated strong positive significant correlation (r=0.993, p=0.007) between apparent porosity and water absorption capacity of clay. The higher the apparent porosity of the clay deposit the greater the clay's ability to absorb water. Additionally, the clay's color changed from gray to red upon firing. These properties indicate that Wushishi clay can be classified as stoneware clay. It has potential applications in the production of flowerpots, as a silica source for floor tiles and brickmaking, and as a binder in the absence of standard binders like phosphoric acid.

Keywords: Clay, Deposit, Characterization, Industrial Applications, Investigation

I.Introduction

Clay, a fine-textured earth with remarkable plasticity when wet but hardening upon drying, is a abundant component of the Earth's crust (McGraw-Hill, 1992; Ekosse & Adebayo, 2018). It is primarily found in soils, sediments, sedimentary rocks, and hydrothermal deposits (Temitayo, 2023). Clay minerals are categorized into two main groups: expandable and non-expandable (Ahmed and Onaji, 1986). Expandable clays, such as smectites, exhibit the ability to swell when exposed to water, even transforming into a liquid state under sufficient hydration conditions. Non-expandable clays, including bentonites, find widespread applications in the petroleum industry as drilling mud components. Additionally, non-expandable clays play a crucial role in the ceramics industry, serving as the primary material for crafting bricks, tiles, pottery, and porcelains (Hongming*et al*., 2023).

The properties of clay that make it particularly valuable for various applications include its plasticity, color, clay strength, and drying and firing shrinkages (Nnuka and Enejor, 2001). The percentage of mineral oxides, such as Fe_2O_3, MgO, CaO, and Na_2O, present in clay significantly influences its suitability for specific applications. For instance, the abundance of alkali metal oxides, including Na_2O, K_2O, and CaO, indicates the clay's potential for ceramic production (Nwosu & Ogueji, 2016).

Nigeria boasts a significant presence of industries engaged in metal and process operations, necessitating a steady supply of raw materials to support their growth (Oniseije & Olaniyi, 2018). Clay-based products, such as ceramic wares, burnt bricks, roofing, and floor tiles, offer economical and durable alternatives to cement, particularly in tropical climates (Nnuka and Enejor, 2001). Utilizing these clay-based products can effectively reduce reliance on cement, especially in the Nigerian context.

Clay deposits are widely distributed across Nigeria, with varying properties from region to region due to geological differences (Olusola, 1998). Despite this abundance, a substantial portion of Nigeria's clay requirements are imported from the United Kingdom, the United States, and Japan (Iwanyawu, 1990). The current economic climate underscores the need for internal sourcing of raw materials to meet the ever-increasing demand.

Exploiting the clay deposit would generate foreign exchange for the country and create employment opportunities for unemployed youths through the establishment of associated industries, as observed by Olokode and Aiyedun (2011) who noted the emphasis placed on exploiting Nigeria's abundant solid minerals to diversify the economy, boost GDP, and increase industrial activity.

Presently, the Whushishi clay deposit in Niger State is primarily utilized for earthenware pots. This paper aims to expand the applications of this clay deposit by characterizing its properties, focusing on the following objectives:

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1. Determination of physical properties
2. Analysis of the chemical composition (Al_2O_3, SiO_2, Na_2O, K2O, MgO,CaO, Fe_2O_3, and loss on ignition)

II.Materials and Method

2.1 Collection of Clay samples

Random sampling was employed to collect clay samples from the various locations, and these samples were dried and dark gray coloured in appearance.

2.2 Preparation of the Samples

Approximately 2 kg of the dried samples underwent manual grinding using a mortar and pestle. Due to the small particle size, the ground material wasn't sieved through a 30-mesh British test sieve. The collected ground portion served as the analysis sample.

2.3 Analysis of the Physicalproperties of the Clay Samples

2.3.1 Determination of porosity, shrinkage tests, bulk density and colour

The ground sample was placed in a plastic basin and moistened with approximately 396 mL of water until it reached the wedging point, where moistened clay material forms a packed ball-in-hand until intentional vibration causes it to flow. The wedged sample was then cast into brass moulds coated with a thin film of machine oil, facilitating easy removal when dry. Eight test clay bars, measuring 7.5 × 3.5 × 1.5 centimetres, were prepared. Using verniercallipers, two points at a 5-centimetre interval were marked on each moulded clay. After allowing the bars to air-dry for 24 hours, the weight of each sample was recorded. Subsequently, the bars were dried in an oven at 110°C for 5 hours, and the marked distance was documented as the dry length. The expression for wet-dry shrinkage was then derived, as indicated in equation 1.

$$\mathrm{Wet-DryShrinkage} = \frac{\mathrm{OriginalLength} - \mathrm{DryLength}}{\mathrm{OriginalLength}} \cdot 100 \quad (1)$$

Eight clay bars were then placed into an electric furnace, alongside four American standard pyrometric cones of 900°C, 1000°C, 1100°C and 1200°C. The firing duration was approximately 10 hours. After reaching each specified temperature, samples with matching temperatures were taken out, cooled, and then assessed for color alteration, cracks, fired length, and fired weight.

The dry-fired shrinkage, percentage total shrinkage, apparent density, bulk density, percentage apparent porosity and percentage water absorption were determined from the relation given below in equations 2,3,4,5,6 and 7 respectively.

$$\mathrm{Dry-FiredShrinkage} = \frac{\mathrm{DryLength} - \mathrm{FiredLength}}{\mathrm{DryLength}} \cdot 100 \quad (2)$$

$$\mathrm{PercenctageTotalShrinkage} = \frac{5 - \mathrm{FiredLength}}{5} \cdot 100 \quad (3)$$

$$\mathrm{ApparentDensity} = \frac{\mathrm{DryWeight}}{\mathrm{SoakedWeight} - \mathrm{SuspendedWeight}} \quad (4)$$

$$\mathrm{BulkDensity} = \frac{\mathrm{DryWeight}}{\mathrm{DryWeight} - \mathrm{SuspendedWeight}} \quad (5)$$

$$\mathrm{PercentageApparentPorosity} = \frac{\mathrm{SoakedWeight} - \mathrm{DryWeight}}{\mathrm{SoakedWeight} - \mathrm{SuspendedWeight}} \cdot 100 \quad (6)$$

$$\mathrm{PercentageWaterAbsorption} = \frac{\mathrm{SoakedWeight} - \mathrm{DryWeight}}{\mathrm{DryWeight}} \cdot 100 \quad (7)$$

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Next, the specimens underwent immersion in water, with bubbles appearing as the pores filled. After 8 hours, the samples were weighed, and the soaked weights were duly recorded. The subsequent expressions were employed to derive the results as follows:

2.3.2Modulus of rupture (MOR)

Eight test clay bars measuring 10 x 1.5 x 1.2 cm dimension were prepared. The initially dry samples were moistened, mixed to a workable state, and cast in wooden molds coated with a thin film of machine oil. After air-drying for 48 hours, six temperature-marked bars were individually placed into an electric furnace with American standard pyrometric cones of 900°C, 1000°C, and 1200°C, firing for approximately 10 hours. Post-firing, the bars were cooled and then subjected to breaking at the center with a 7.0 cm span on a Denison strength testing machine. The Modulus of Rupture (MOR) was calculated using the expression provided by Chester (1973) as indicated in the equation 8:

$$MOR = \frac{3PL}{2bh^2} \quad (8)$$

Where L = Distance between support; P= breaking load in Kgf; b = Breath, h = Height.

3.3.3 Modulus of Plasticity

A 50g clay sample was placed in a container and slightly moistened. The damp clay was shaped into a cylinder using a cylindrical mold. A flat-headed plunger, with a known weight, was dropped onto the molded clay from a fixed height. The distance traveled was measured using a graduated scale. The Modulus of Plasticity (MOP) for the clay sample was determined using the following expression:

$$MOP = \frac{\text{Original height}}{\text{Deformed hieght}} \quad (9)$$

Also, the percentage making moisture for the clay samples were obtained from the expression:

$$\%\ \text{Making Moisture}\quad MOP = \frac{\text{Wet Weight} - \text{Dry Weight}}{\text{Net weight}} \quad (10)$$

2.4 Analysis of the Chemical Properties of the Clay Samples

2.4.1 State Solution Preparation

A 0.1g sample was weighed into a Teflon crucible and dampened with Aqua regia (a mixture of HCl and HNO3 in a 3:1 volume ratio). To this, 15ml of hydrofluoric acid was added, and the mixture was covered, then heated in a fumed chamber at 100°C until the solution turned clear. After cooling, the solution was transferred to a 250ml plastic volumetric flask (due to hydrofluoric acid's corrosive effect on glass) and made up to the 250ml mark with distilled water. Various chemical standards, equipment, and analytical methods were employed to determine the percentage composition of SiO_2, Al_2O_3, Na_2O, K_2O, CaO, MgO, and $Fe2O_3$.

2.5 Loss on Ignition

A 0.5g portion of the clay sample was placed in a clean, dried platinum crucible and subjected to a furnace at 600°C for 3 hours. Subsequently, it was cooled in a desiccator and weighed. The weight lost during this process, attributed to ignition, is denoted in Equation 11 as follows:

$$LOI = \frac{\text{WeightLoss}}{\text{WeightofSampleMud}} \cdot 100 \quad (11)$$

2.6 Statistical Analysis

Statistical analyses such as standard deviations or statistical significance tests including statistical measures was performed on the data to determine the significance of observed differences and the reliability of the results.

2.6.1 Mean

Mean is a measure of central tendency denoted by$\bar{x}$. In other word, it is an average or expected value of an observation which is mathematically defined in equation 12 as follows:

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$$\bar{x} = \frac{\sum_i^n x_i}{n} \quad (12)$$

Where:

Σ = Summation

X=observation

n=number of observations

2.6.2 Standard Deviation (SD)

Standard deviation is a quantity measuring the extent to which a number of a group differ from the mean value of the group. It is usually denoted as "S" and its denoted in equation 13 as follows:

$$S = \sqrt{\frac{\sum(x_i - \bar{x})^2}{n}} \quad (13)$$

Where:

S = Standard Deviation

x_i= Each value from the sample

$\bar{x}$= Sample mean

n=sample size

2.6.3 Correlation Analysis

Correlation analysis is a statistical tools used in examining the degree or strength of linear relationship between two variables. The correlation coefficient ranges from +1 (positive relationship) to -1 (negative strong relationship). Meanwhile, the value of zero (0) implies no relationship between the variables. However, it is usually denoted by r and mathematically defined in equation 14 as follows:

$$r = \frac{\sum(x_i - \bar{x})(y_i - \bar{y})}{\sqrt{\sum(x_i - \bar{x})^2 \sum(y_i - \bar{y})^2}} \quad (14)$$

Where:

r=correlation coefficient

x_i=value of independent variable

$\bar{x}$=Mean of value of independent variable

y_i=value of dependent variable

$\bar{y}$=mean of value of dependent variable

III. Results and Discussions

3.1 Results

The results obtained for the above named tests and procedures are presented below.

Table 1: Shrinkage test Results

Temp ºC	Wushishi Clay					Total Shrinkage
	Original length (cm)	Dry length (cm)	Fired length (cm)	% Dry Fired Shrinkage	% Wet Dry Shrinkage	
900	5	4.71	4.57	4.25	5.8	8.6

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1000	5	4.76	4.51	5.25	4.8	9.9
1100	5	4.85	4.43	8.66	3.6	11.4
1200	5	4.92	4.41	10.37	3	11.9
Mean						**10.5**
S.D						**1.5**

Table2: Percentage Apparent Porosity, Percentage Water Absorption, Apparent and Bulk Density, Firing Behaviour and Colour Variation Results

Temp ºC	Apparent Porosity %	Water Adsorption %	Bulk Density g/cm^3	Apparent Density g/cm^3	Colour Formation	Crack Formation
900	24.72	15.48	2.25	1.78	Light red	Few crack
1000	20.28	11.28	2.26	1.80	More red	
1100	14.39	7.76	2.17	1.82	Very red	
1200	13.88	7.47	2.17	1.86	Dark red	
Mean			**2.21**	**1.82**		
SD			**0.05**	**0.34**		
Correlation coefficient (r=0.993, p=0.007) between Apparent Porosity and Water Adsorption						

Table 3: Modulus of Rupture

Temp (ºC)	Distance between Supports (cm)	Modulus of rupture kgf/cm^2
900	7	22.56
1000	7	32.63
1100	7	33.13
1200	7	34.86
Mean		**30.80**
S.D		**5.59**

Table 4: Modulus of plasticity and % Making Moisture

Wet Weight (g)	Dry Weight (g)	Deformation Height (cm)	Original Height (cm)	Modulus of Plasticity (kgf/cm^2)	% Marking moisture
62.68	51.21	3.62	10.27	2.837	18.61
63.97	52.22	3.64	10.31	2.832	18.37
64.5	52.91	3.64	10.30	2.829	17.96
Mean				**2.833**	
SD				**0.004**	

Table 5: Chemical Analysis of Wushishi Clay Deposit

Clay Samples	Percentage (%) Composition

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	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	LOI
Wushishi Clay Deposit	59.8	17.08	2.54	1.50	0.34	4.39	2.89	14.0

Table 6: Comparison of the chemical analysis of Wushishi clay deposit with termite hill and typical composition of imported refractory materials.

CONSTITUENTS	WUSHISHI CLAY DEPOSIT	TERMITE HILL	INDIAN CLAY	CORNISH CHINA CLAY	FIRE CLAY	SILCEOUS FIRE CLAY
SiO_2	59.8	68.08	55-70	45.46	60-5	59.0
Al_2O_3	17.08	28.12	25-38	38.33	33-8	25.0
Fe_2O_3	2.54	1.00	2-5	0.55	2-2	5.9
TiO_2	-	1.09	1-1.5	0.34	1-2	0.6
CaO	1.50	0.30	0.5-1	0.06	-	0.6
MgO	0.34	0.38	0.2-0.8	0.12	-	-
Na_2O	4.39	2.4	--	0.43	-	-
K_2O	2.89	-	1 – 2	2.22	1-2	2-3
L.O.I	14.0	14.3	--	-12.44	-	-

3.2 Discussion

3.2.1 Shrinkage

The shrinkage test results, with an average value of 10.5%, and standard deviation 1.5 indicate that the Wushishi clay deposit falls within the standard range 8-12% for brick clay reported by (Singer and Singer, 1971; Nwosu and Ogueji, 2016)). Shrinkage is a crucial property for brickmaking, and the suitability of Wushishi clay for brick production is evident from its shrinkage behavior.

3.2.2 Apparent Porosity and Water Absorption

Table 2 showed that Wushishi Clay deposit demonstrated strong positive significant correlation ($r=0.993$, $p=0.007$) between apparent porosity and water absorption capacity of clay. The higher the apparent porosity of the clay deposit the greater the clay's ability to absorb water, this agreed with (Ekosse and Adebayo, 2018). This water absorption capacity significantly influences the drying behavior of clay, as the absorbed water must be expelled during drying, often leading to high drying shrinkage.

3.2.3 Bulk Density

Table 2 showed that the bulk density of the clay was found to be 2.25, 2.26, 2.17 and 2.17 g/cm^3 at 900^{O}C, 1000 ^{O}C, 1100 ^{O}C and 1200 ^{O}C respectivelywhich indicated mean value of 2.21g/cm^3 and standard deviation of 0.05. The values compared favourably with the general standard value range of between 1.0 and 2.5 g/cm^3 recorded by (Das and Sivakugan, 2010; Oniseije, and Olaniyi, 2018). This makes the clay deposit suitable for brick making.

3.2.4Apparent Density

Table 2 revealed that the apparent density of the clay deposit was found to be 1.78, 1.80, 1.82 and 2.86 g/cm^3 at 900^{O}C, 1000 ^{O}C, 1100 ^{O}C and 1200 ^{O}C respectively with mean value of 1.82g/cm^3 and standard deviation of 0.35. The values compared favourably with the internationally acceptable range of 1.7 – 2.1g/cm^3 reported by (Olusola, 1998). This could be as a result of effective sintering during firing which makes the clay deposit suitable for brick making.

3.2.5 Colour

The firing process induces colour variations in the Wushishi clay deposit, ranging from gray to red, as shown in Table 2. This colour transformation is attributed to the presence of iron oxide, with a content of 2.54%. The ferrous iron imparts a red hue to the fired sample due to its conversion to ferric compounds. This Colour variation makes the clay suitable for manufacturing flowerpots (Rhodes, 1973; Olusola, 1998; Olufemi*et al., 2023*).

3.2.6 Modulus of Rupture (MOR)

MOR represents the load-bearing capacity of clay. Table 3 revealed that the average MOR value of Wushishi clay was 30.80 which ranges from 22.56 to 34.86 kgf/cm^2 as the firing temperature increases from 900°C to 1200°C with standard deviation value of 5.59. This strength enhancement is attributed to bond formation within the glassy phase. The soda content in the clay components likely contributes to the formation of low-temperature melting compounds, which contribute to the clay's bulk strength upon cooling. However, the MOR values fall within the acceptable range of 1.4 to 105 kgf/cm^2 reported by (Lawi, 2006).

3.2.7 Plasticity

Table 4 indicates that the Wushishi clay exhibits moderate mean plasticity(2.833 kgf/cm^2) and standard deviation (0.004). This plasticity property, coupled with its good workability, makes it suitable for various industrial applications. However, the presence of iron oxide and other impurities limits its use to stoneware production. The results are comparable to those of Ukpor clay, suggesting that Wushishi clay deposit can serve as a binder in the absence of conventional binders like phosphoric acid (**Singer and Singer, 1971;** Nwajugu and Aneke, 2001).

3.2.8 Chemical Composition

The chemical analysis results presented in Table 5 and 6 highlight the high silica content (SiO_2,59.8%) of Wushishi clay, suggesting its presence as quartz. Comparing these results to those obtained for Oshiele clay indicates their potential utilization as silica sources for floor tile production (Nwajugu and Aneke, 2001). The high percentage of alkali oxides (CaO, K_2O, and Na_2O) explains the clay's plasticity characteristics (Nnuka and Enejor, 2001). However, the elevated alkali oxide content, particularly soda exceeding 3%, along with the low aluminum content, renders the clay unsuitable for refractory, paper, and ceramic applications.

IV. Conclusion

Based on the outcome of the investigation it could be concluded that Wushishi clay possesses moderate shrinkage capacity with an average value of 10.5%, and standard deviation 1.5 which showed that the clay deposit falls within the standard range 8-12% for brick clay, moderate bulk density of 2.25, 2.26, 2.17 and 2.17 g/cm^3 at $900^{O}C$, 1000 ^{O}C, 1100 ^{O}C and 1200 ^{O}C respectively with mean value of 2.21 g/cm^3 and standard deviation of 0.05.

Wushishi clay deposit equally demonstrated strong positive significant correlation (r=0.993, p=0.007) between apparent porosity and water absorption capacity of clay. The higher the apparent porosity of the clay deposit the greater the clay's ability to absorb water. It also showed moderate plasticity with average value of 2.83 $kgf/cm^2$and standard deviation of 0.004. This plasticity property, coupled with its good workability, makes it suitable for various industrial applications.

Additionally, the clay exhibited good strength, with modulus of rupture (MOR) values ranging from 22.56 - 34.86 kgf/cm^2.The investigation results show that Wushishi clay possesses favorable properties for diverse industrial applications, offering potential economic benefits through export and job creation.

These favorable properties suggest that Wushishi clay holds promise for various applications, including:

1. **Flowerpot Production:** The clay's color change upon firing and its moderate plasticity make it well-suited for manufacturing flowerpots.
2. **Silica Source for Floor Tiles:** The high silica content (SiO_2) of Wushishi clay indicates its potential as a silica source for floor tile production.
3. **Binder in Moulding Sand:** The clay's plasticity and strength characteristics make it a viable binder in the production of moulding sand.
4. Exploiting the clay deposit would generate foreign exchange for the country and create employment opportunities for unemployed youths through the establishment of associated industries.

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