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THE EFFECT OF PARTICLE SIZE AND PARTICLE SIZE DISTRIBUTION ON THE MODULUS OF RUPTURE OF SOME SOUTH EAST NIGERIA CLAYS

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Abstract: The modulus of rupture of three clay samples were investigated to determine the effect of particle size distribution on them. The samples were collected from natural deposits at Nsu-Ehime; Ohia- Umuahia and Awo-Omamma, all in south eastern Nigeria. The particle size distribution of the clays were determined by laser diffraction technique. Thermo physical and thermo-mechanical parameters of the clays were also tested. Comparison of the modulus of rapture of the three clay samples with their particle size distributions revealed that a high proportion of the larger sized particles, (21 μm-51μm) will lower the modulus of rupture of the clay. Awo-Omamma clay contains 16.14% of these size fractions. Nsu and Ohia clays contain 4.05% and 6.65% of these size fractions respectively. Correspondingly, Nsu clay has better modulus of rupture results than Ohia clay and Awo-Omamma clay. Nsu clay and Ohia clay will perform well in most structural and technical ceramic applications. Awo-Omamma clay would require proper blending with other ingredients to produce a similar performance.

Keywords: Clay, Particle Size, Porosity, Bulk Density, Modulus of Rupture, Volume Mean Diameter (VMD), Laser Diffraction, Sieve Aperture, Shrinkage, Thermo-Physical, Thermo-Mechanical.

INTRODUCTION

Clay minerals are hydrous alumino-phyllosilicates, sometimes with variable amounts of iron, magnesium, alkali metals, alkaline earths and other cations, found on or near the earth's surface. They are formed over long periods of time, by the gradual weathering of mainly silicate bearing rocks, by low concentrations of carbonic acid and other diluted solvents, (Bailey 1980). They are usually, (but not necessarily), ultra fine grained. Most clay minerals are found in nature with particle sizes in the range of < 4μm. Clays are the chief raw material for the ceramic industry. The optimum utilization of local clay deposits is very vital for the economic growth of many developing nations. However, this utilization is often hampered by poor technical characterization of these local clay deposits.

PARTICLE SIZE AND PARTICLE SIZE DISTRIBUTION OF CLAYS

Particle size is a notion introduced for comparing the dimensions of solid particles, (flecks), liquid particles, (droplets), or gaseous particles, (bubbles). The dictionary of earth sciences define particle size, as the diameter or volume of the grains in a sediment or sedimentary rock. It goes further to say that particle size can be determined by sieving, by measuring the settling velocity or by direct measurement of individual clasts, (Allaby, 1999). The particle size distribution of a powder, or a granular material, or particles dispersed in fluid, is a list of values or a mathematical function that defines the relative amount, typically by mass, of particles present according to size, (Jillavenkatesa 2001). Particle size distribution of a material can be important in understanding its physical and chemical properties as well as its technical behavior. For clays, it is thought that the composition of the parent rock as well as the dominant weathering mechanism contribute to the particle size distribution. There are several methods of measuring the particle sizes of colloidal substances like clay minerals. They can be broadly classified into direct methods and indirect methods. Direct methods include mechanical sieving, optical techniques, microscopy and laser light scattering techniques. Indirect methods include sedimentation technique, elutriation and centrifugation. Laser light scattering technique was used to determine particle sizes and size distributions for this work. Ordinarily the various methods of particle sizing have their pros and cons. The choice of which method to use would depend on a number of factors including:

- Nature of the material to be sized, e.g estimated particle size ranges; solubility; ease of handling; toxicity; flow parameters; intended use.
- Costs including initial equipment costs and running costs.
- Specification requirements
- Time constraints, (Dallavalle 1948).

From the point of view of laboratory efficiency, accuracy and reproducibility, the laser diffraction technique is a far superior method of particle size distribution analysis of colloidal substances compared to sieving and sedimentation techniques, (Konert and Vandenberghe 1997). In the laser diffraction technique, the particles are made to pass through a laser beam and the light scattered by them is collected over a range of angles in the forward direction. The angles of diffraction are inversely related to the particle size. The particles pass through an expanded and collimated laser beam, in front of a lens in whose focal plane is positioned a photo sensitive detector, consisting of a series of concentric rings. The distribution of the scattered intensity is analyzed by a computer to yield the particle size distribution, (Healy 2010).

MODULUS OF RUPTURE

Modulus of rupture is a common strength parameter used in the ceramic industry. It is the maximum transverse breaking stress that a material will withstand before fracture. Ceramic raw materials as well as body recipes are often tested for their modulus of rupture. Various ceramic products require raw materials with specific ranges of modulus of rupture values. For example porcelain would require clays with much higher modulus of rupture than table ware. The transverse bending test is used to determine the modulus of rupture of a specimen. In this test, a bar specimen having either a rectangular or circular cross-section is bent until fracture occurs, using a three point or four point loading configuration. The stress at fracture is known as the flexural stress or the modulus of rupture. For a rectangular specimen in three point loading configuration, modulus of rupture is given by

$$MoR = \frac{3PL}{2BH^2} \quad \text{------ Eq. 2.1}$$

Where

P = load at fracture
L = length between supports (support span)
B = specimen width
H = specimen height

For a rectangular beam in 4 – point loading configuration, where the loading span is one third of the support span,

$$MoR = \frac{PL}{BH^2} \quad \text{---------- Eq. 2.2}$$

Where

L = length of the support span
P = load at the fracture point
B = specimen width
H = specimen height or thickness.

For 4- point loading configuration in which the loading span is half of the support span, then

$$MoR = \frac{3PL}{4BH^2} \quad \text{---------- Eq. 2.3}$$

If the loading span is nighters $^1/_3$ or ½ the support span in a 4-point load configuration, then

$$MoR = \frac{3P\,(L - Li)}{2\,BH^2} \quad \text{---------- Eq. 2.4}$$

where L is the distance between supports and Li is the load span, (ASTM 2008).

EXPERIMENTAL PROCEDURE

The clay samples were collected in lumps from their natural deposits at Nsu, Ohia and Awo-Omamma respectively. The lumps were crushed and the powder dispersed in excess distilled-deionized water in pre-treated plastic containers. The mixture was stirred vigorously to ensure proper dissolution. The dissolved clay was then filtered to get rid of dirt and organic material. The filtrate obtained was allowed to settle for 48 hours after which excess water was decanted. The settled clay samples were sun dried for four days to get rid of excess water molecules. 100 grams of each sample was weighed out and sent for chemical analysis and laser diffraction analysis to determine the particle size distribution of the samples. A second portion of the clay samples were weighed out and mixed with appropriate amount of water to make it plastic for the molding process. From each sample, rectangular test pieces were molded which had a length of 9.5cm, a width of 2cm and height of 1.5 cm. The test pieces were air dried for seven days after which they were oven dried at 110^0c until a constant weight was obtained. The three test pieces, one from each clay sample were fired in a laboratory kiln to a temperature of 1200^0c. After cooling, the test pieces were subjected to modulus of rupture test in a three point loading configuration, the breaking load P was determined using the electrical transversal strength machine at PRODA Emene, Enugu State Nigeria. A venire caliper was used to determine the distance between support L (cm) of the transversal strength machine. The height, H (cm) and the width, B (cm) of the broken pieces were determined and the average value obtained from the two broken parts were recorded. The modulus of rupture were than calculated as,

$$\text{MoR (kg/cm}^2\text{)} = \frac{3PL}{2BH^2} \quad \text{--------Eq. 3.1,}$$

(Akwilapo and Wiik 2003)

Other test pieces were prepared from the clay samples for the determination of other thermo-physical parameters in accordance with standard ASTM test procedures. The thermo-physical parameters determined for the clay samples include, drying and firing shrinkage, bulk density, apparent porosity and relative plasticity.

RESULTS AND DISCUSSION

Table 4.1 below shows the oxide composition of the three clay samples, obtained using x-ray fluorescence spectroscopy. Figures 4 a, b and c shows the results of the particle size analysis using laser diffraction technique. The dispersion medium was deionized water. Also table 4.2 shows the results of the modulus of rupture tests for the specimens. Two specimens were tested for each clay sample and the average values obtained. The modulus of rupture of the specimens were calculated using equation 3.1. Table 4.3 shows the values obtained in the shrinkage test of the clay samples.

Wet-Dry shrinkage was calculated as

$$\left(\frac{L_o - L_d}{L_o}\right) \times 100$$

Dry- fired shrinkage was calculated as

$$\left(\frac{L_d - L_f}{L_d}\right) \times 100$$

Total shrinkage was calculated as

$$\left(\frac{L_o - L_f}{L_0}\right) \times 100$$

Lynne etal (1980)

Table 4.4. shows the values obtained from the porosity tests of the clay samples.

Apparent porosity of the specimens were calculated using the formula

$$\text{Apparent porosity (\%)} = \left(\frac{M_2 - M_1}{M_2 - M_3}\right) \times 100$$

$$\text{Apparent Density} = \left(\frac{M_1}{M_1 - M_3}\right)$$

$$\text{Bulk Density} = \left(\frac{M_1}{M_2 - M_3}\right)$$

(Akwilapo and Wiik, 2003).

The average values of the modulus of rupture for the three clay samples are 15.35 kg/cm^2, 15.58kg/cm^2 and 5.55kg/cm^2 for Nsu clay, Ohia clay and Awo-Omamma clay respectively. Similarly the average total shrinkage values for the clay samples are 8.5%; 9.6% and 11.7% for Nsu clay, Ohia clay and Awo – Omamma clay respectively. The samples yielded apparent porosity values of 36.6%; 39.7% and 38.7%; and bulk density values of 1.63g/cm^3; 1.7 g/cm^3 and 1.62 g/cm^3 respectively. Comparison of these thermo physical and thermo-mechanical results shows a similarity in values for the apparent porosity and bulk densities of the three samples. However sample C, Awo omamma clay had to undergo an 11.7%

shrinkage to reach this bulk density and porosity values compared to 9.6 % for Ohia clay and 8.5% for Nsu clay. Awo-omamma clay also showed a much lower modulus of rupture value of 5.5 kg/cm^2 compared to 15.58kg/cm^2 for Ohia clay and 15.35kg/cm^2. These behavior can be explained by considering the particle size distribution results of the three clay samples. The volume mean diameter (VMD) gives an indication of the average particle size of the clay samples. Awo-Omamma clay has a volume mean diameter of 11.29μm, compared to 8.93μm for Ohiya clay and 6.56 μm for Nsu clay. The X_{50} value gives an indication of the sieve aperture that will pass 50% of particles in a sample. It gives an idea of how large or how small half of the particles in a sample are. The X_{50} value for Awo-Omamma clay is 9.26μm compared to 7.20 μm for Ohia clay and 3.81μm for Nsu clay. Similarly, the X_{99} values gives an indication of the sieve aperture required to pass 99% of the particles in a sample. It gives an idea of the size of the largest particles in the sample. The X_{99} value for Awo-Omamma clay is 39.00 μm compared to 29.67μm for Ohiya clay and 29.54μm for Nsu clay. Thus, it can be seen that Awo – Omamma clay has a high percentage of larger sized fractions. This high proportion of large sized particles would result in poor particle packing which will adversely affect strength development as reported by (Sarkar 2011).

CONCLUSIONS AND RECOMMENDATIONS

It can be seen from the results generated in this research that particle size distribution of clays would affect particle packing during production of wares from such clays. This particle packing affects the strength development characteristics during firing. Very high proportion of large size fractions, typically above 21μm results in poor particle packing, which adversely affects strength development. This explains the poor mechanical behavior of Awo-Omama clay as indicated by the modulus of rupture value of 5.5 kg/cm^2. This particle packing phenomenon is thought to apply to any ceramic body composition. Thus care should be taken during raw material processing to ensure adequate grinding of raw materials and elimination of over sized particles. Nsu clay and Ohia clay will perform well in most structural and technical ceramic applications where slip casting methods are employed. Awo-Omama clay would require proper blending with other ingredients in order to produce a similar result. However Awo-Omama clay may be used effectively in applications where compaction pressure is applied in forming the ceramic body.

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COMPOSITION	WEIGHT PERCENT		
	Sample A NSU	Samples B Ohiya	Samples C Awo-Omamma[3]
SiO_2	46.01	48.23	50.42
Al_2O_3	31.32	29.45	26.01
TiO2	0.34	-	0.23
Fe_2O_3	4.78	3.58	5.68
MnO	2.66	-	-
MgO	1.45	1.49	0.96
CaO	-	0.22	-
Na_2O	0.98	0.29	1.53
K_2O	0.61	2.56	0.49
H_2O (ignition Loss at 1000^0C	10.82	13.40	14.68

Table 4.1: Oxide Composition of the Clay Samples.

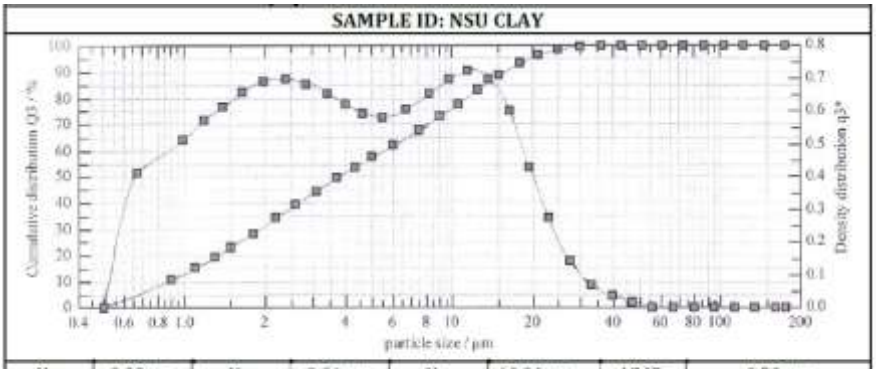
Sample	Breaking load P (kg)	Distance between supports Lo (cm)	width B. (cm)	Height H (cm)	MOR (kg/cm^2)
A_1	5.8	7	1.93	1.43	15.43
A_2	5.6	7	1.91	1.42	15.27
B_1	5.7	7	1.82	1.45	16.19
B_2	2.0	7	1.85	1.47	14.97
C_1	2.1	7	1.92	1.41	5.50
C_2	2.1	7	1.93	1.43	5.59

Table 4.3:The Results Obtained in the Shrinkage Test of the Clay Samples.

Sample No	Original length Lo (cm)	Dried Lo length u (cm)	Fired Lf length (cm)	Wet-Dry shrinkage (%)	Dry –fired shrinkage (%)	Total shrinkage (%)
A_1	5	4.77	4.58	4.6	3.98	8.4
A_2	5	4.78	4.57	4.4	4.39	8.6
B_1	5	4.69	4.51	6.2	3.84	9.8
B_2	5	4.70	4.53	6.0	3.62	9.4
C_1	5	4.64	4.42	7.2	4.74	11.6
C_2	5	4.65	4.41	7.0	5.16	11.8

Table 4.4: The Results Obtained from the Porosity Test of the Clay Samples.

Sample No	Soaked weight (g) M_2	Dried weight (g) M_1	Suspended weight (g) M_3	Apparent porosity %	Apparent density (g/cm^3)	Bulk density (g/cm^3)
A_1	88.68	72.47	44.34	36.55	2.58	1.63
A_2	85.56	69.85	42.78	36.72	2.58	1.63
B_1	74.13	58.32	37.07	42.64	2.74	1.57
B_2	76.05	60.26	33.03	36.70	2.21	1.82
C_1	78.89	63.12	39.45	39.97	2.67	1.60
C_2	80.56	65.51	40.28	37.36	2.60	1.63

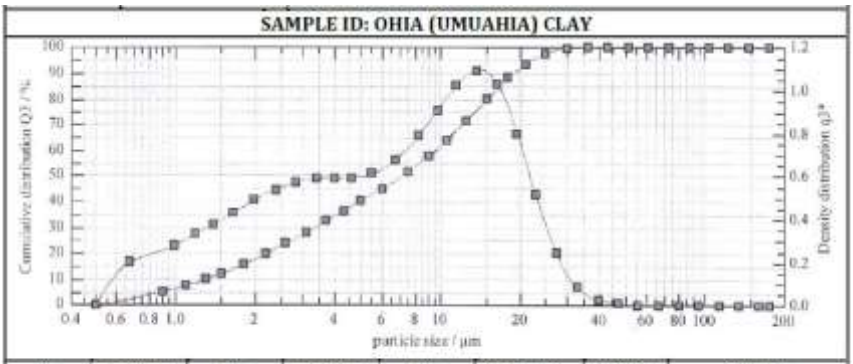


X_{10}	0.88 µm	X_{50}	3.81 µm	X_{90}	16.04 µm	VMD	6.56 µm
X_{16}	1.15 µm	X_{84}	13.03 µm	X_{99}	29.54 µm	S_m	9560.93 cm^2/g
Dispersion Medium		DI Water					

Cumulative Distribution

X_0/µm	Q_3/%	X_0/µm	Q_3/%	X_0/µm	Q_3/%	X_0/µm	Q_3/%
0.90	10.46	3.70	49.27	15.00	88.36	61.00	100.00
1.10	14.91	4.30	53.30	18.00	93.08	73.00	100.00
1.30	19.04	5.00	57.16	21.00	95.95	87.00	100.00
1.50	22.83	6.00	61.75	25.00	97.99	103.00	100.00
1.80	28.01	7.50	67.57	30.00	99.10	123.00	100.00
2.20	34.01	9.00	72.73	36.00	99.64	147.00	100.00
2.60	39.05	10.50	77.38	43.00	99.89	175.00	100.00
3.10	44.25	12.50	82.84	51.00	100.00		

Figure 4.a. Particle size analysis of samples A (Nsu Clay)



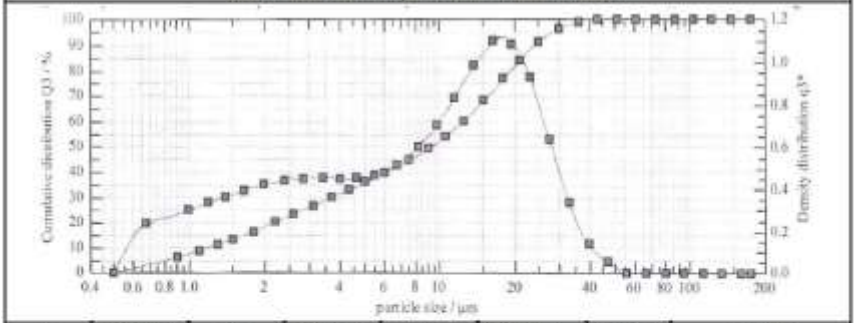
X_{10}	1.32 µm	X_{50}	7.20 µm	X_{90}	19.11 µm	VMD	8.93 µm
X_{16}	1.85 µm	X_{84}	16.51 µm	X_{99}	29.67 µm	S_m	6445.79 cm^2/g
Dispersion Medium		DI Water					

Cumulative Distribution

X_0/µm	Q_3/%	X_0/µm	Q_3/%	X_0/µm	Q_3/%	X_0/µm	Q_3/%
0.90	5.05	3.70	32.32	15.00	79.93	61.00	100.00
1.10	7.43	4.30	36.14	18.00	88.03	73.00	100.00
1.30	9.79	5.00	39.98	21.00	93.35	87.00	100.00
1.50	12.10	6.00	44.80	25.00	97.23	103.00	100.00
1.80	15.46	7.50	51.32	30.00	99.13	123.00	100.00
2.20	19.67	9.00	57.54	36.00	99.77	147.00	100.00
2.60	23.53	10.50	63.60	43.00	99.95	175.00	100.00
3.10	27.85	12.50	71.33	51.00	100.00		

Figure 4.b. particle size analysis of samples B. (Ohiya clay)

SAMPLE ID: UMUEJIKE AWOMAMMA CLAY



X_{10}	1.22 µm	X_{50}	9.26 µm	X_{90}	24.49 µm	VMD	11.29 µm
X_{16}	1.77 µm	X_{84}	21.08 µm	X_{99}	39.00 µm	S_m	6277.40 cm^2/g
Dispersion Medium		DI Water					

Cumulative Distribution

X_0/µm	Q_3/%	X_0/µm	Q_3/%	X_0/µm	Q_3/%	X_0/µm	Q_3/%
0.90	5.96	3.70	29.96	15.00	67.91	61.00	100.00
1.10	8.53	4.30	32.87	18.00	76.62	73.00	100.00
1.30	10.93	5.00	35.80	21.00	83.86	87.00	100.00
1.50	13.17	6.00	39.48	25.00	90.89	103.00	100.00
1.80	16.26	7.50	44.46	30.00	95.92	123.00	100.00
2.20	19.93	9.00	49.19	36.00	98.54	147.00	100.00
2.60	23.11	10.50	53.88	43.00	99.61	175.00	100.00
3.10	26.52	12.50	60.14	51.00	100.00		

Figure 4.c. particles size analysis of sample- C (Awo – Omamma Clay)

Reference to this paper should be made as follows: Osonwa Nobert Okey, et al (2017), The Effect of Particle Size and Particle Size Distribution on the Modulus of Rupture of Some South East Nigeria Clays. *J. of Sciences and Multidisciplinary Research,* Vol. 9, No. 3, Pp. 51-60