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DETERMINATION OF THE PARTICLE SIZE DISTRIBUTION OF SOME SOUTH EAST NIGERIA CLAYS BY LASER DIFFRACTION TECHNIQUE.

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ABSTRACT

The particle size distribution of three south east Nigerian clays were studied using laser diffraction technique .The clay samples were collected from their natural deposits at Nsu in Ehime Mbano, Awo-Omamma in oru west ,both of Imo state, and Ohia in Umuahia South of Abia state, all in south eastern Nigeria .The dispersion medium used was deionized water . It was discovered that the volume mean diameter of the clay samples were 6.56μm;8.93μm and 11.29μm for Nsu clay, Ohia clay and Awo-Omamma clay respectively . Also the mean surface area of the clay particles are 9560.93 cm^2/g, 6445.79 cm^2/g and 6277.40 cm^2/g for Nsu, Ohia and Awo-Omamma clays respectively .The cumulative distribution of the smallest particle size fractions in the samples, as indicated by X_{10} values are 0.88 μm, 1.32 μm and 1.22 μm for Nsu, Ohia and Awo-Omama clays respectively .

Keywords: *Particle size, Clays, Laser diffraction, Analysis, Dispersion, Volume mean, Mean surface area.*

INTRODUCTION

Clay minerals are hydrous aluminium 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). Clays are usually ,but not necessarily ultrafine grained .Most clay minerals are found in nature with particle sizes in the range of≤ 4 μm. This makes many clays to require special analytical techniques for their identification and study ,including XRD ;election diffraction methods ;various spectroscopic methods ;SEM methods ;Laser diffraction methods etc.

Clays are the chief raw material for 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. The particle size distribution of a clay sample is a list of values or a mathematical function that define the relative amount ,typically by mass, of particles present according to size, (Jillavenkatesa 2001). For clay minerals, knowledge of the particle size distribution can be important in understanding its physical and chemical properties ,for instance, the particle size distribution of a clay will affect the densification behaviour during firing, from which derives other thermo-mechanical properties of the fired material.

Methods of Particle Size Determination

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 techniques. The choice of which method of particle sizing to be used depends on a number of factors which include;
Nature of the material to be sized, e.g estimated particle size and particle size ranges; solubility, ease of handling ; toxicity; flow ability, intended use.

- Costs including initial equipment costs and running costs.

- Specification requirements
- Time constraints.

Thus the various methods of particle sizing have their advantages and their shortcomings, based on the above mentioned considerations.

Laser Diffraction Analysis

Laser diffraction technique is based mainly on Gustav Mies light scattering theory .It determines particle size distribution by measuring the angular variation in intensity of light scattered as a laser beam passes through a dispersed particulate sample. Mie has established a relationship between particle size and the angle and intensity of scattered light in his solutions to Maxwells equations for scattering of an electromagnetic plane wave by a homogeneous sphere. According to Mie's theory light scatters more intensely and at smaller angles off of larger particles than smaller particles. The laser diffraction analyser measures the angle and intensity of light scattered from particles in a sample. This information is then passed to an algorithm designed to use the Mie scattering theory which transforms the scattered light data into particle size information.

Another theory of light scattering often used is the Fraunhofer diffraction theory which states that the intensity of light scattered by a particle is directly proportional to the particle size, (ALena 1997). The angle of the laser beam has an inverse proportionality relationship with the particle size , where the laser beam angle increases as particle size decreases, (Mccave et al 1986).

The advantages of the laser diffraction method include

- Non-intrusiveness, as it uses a low power beam.
- Fast - it takes typically less than 3 minutes to take a measurement and analysis
- High precision and wide range
- High reproducibility

- Absolute measurement; no calibration is required as the instrument is based on fundamental physical properties.
- Simple to use and high versatility

However it requires a very expensive equipment and requires that a difference must exist in refractive index between particles and the suspending medium, (Healy 2010). According to Konert and Vandenberghe (1997), 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.

Experimental Procedure

The samples were collected in lumps from their natural deposits in Nsu, Ohia and Awo –omamma respectively. The lumps were collected at a depth of 1.5meters after the overburden had been removed. About 2 grams of each sample was put in labeled 250 ml beaker. The beakers were put onto a sand bath in a fume cupboard and the heat setting was switched to 2. A 30% solution of hydrogen peroxide was poured into each beaker until the sample was completely covered. Frothing of the samples was controlled by squirting small amounts of methylated spirit from wash bottles into the beakers. After the frothing subsided, the sand bath was turned up to heat level 6. More hydrogen peroxide was poured onto the samples until all organic substances have been oxidized and no more frothing observed. The heat was turned off and the samples allowed cooling. Distilled water was then added to the beaker to cover the samples and keep it moist.

The Helos Sympatech sizer was switched on and the water supply turned on. The machine's software was opened on the PC. The glass beaker in the sample suspension unit was filled to about 2/3 full with de-ionised water and the stirring head lowered into place

The laser sizer automatically checked the water in the bath, measured the background and other initializing operations. The background reading of the obscuration value of clean deionised water was displayed on the screen. A value of 1 % or less is usually acceptable.

The samples were then put in a whirl mixer and thoroughly mixed in a vortex to ensure no lumps are stuck to the bottom of the whirl mixer tube. A few drops of the sample was added to the beaker in the sample suspension unit with a pipette, care was taken to ensure that the particles were evenly distributed. The obscuration value on the display was observed. A value of 10-20% is usually acceptable. When a steady value of the obscuration was maintained for 6 seconds, the machine automatically proceeds with analysis of the sample. After the analysis, the results are automatically displayed on the screen as a chart and thereafter printed from a printer attachment. The sample in the beaker was then drained and the unit rinsed three times with plenty of water before analysis of the next sample.

DISCUSSION

Figures 4.1, 4.2 and 4.3 show the results of the particle size distribution analysis of Nsu, Ohia and Awo-Omamma clays respectively. The particle size is plotted against the cumulative distribution, as well as the density distribution, generating the two curves on the chart. From the results, the volume mean diameter of particles in the samples are 6.56μm, 8.93 μm and 11.9 μm for Nsu, Ohia and Awo-Omama clays respectively. The volume mean diameter is an indication of the average particle size of the samples. Also the mean surface area of the particles were determined as 9560.93cm^2/g, 6445.79cm^2/g and 6277.40 cm^2/g for Nsu, Ohia and Awo-Omamma clays respectively. The mean surface area would have a bearing on the packing tendencies of particles when formed into a ceramic body or slip. The lower the mean surface area, the denser the ceramic body. Similarly a higher surface area mean, will result in a more porous body.

The X_n values give an idea of the percentage composition of the samples by size fractions. If it were possible for instance, to arrange the particles of the clay samples in order of increasing particle size, and take an arbitrary cut off point, say 10%by weight (10w/w%), then Nsu clay sample will consist of particles that are 0.88 μm or less, Ohia clay will consist of particles that are 1.32 μm or less, while Awo-Omamma clay will consist of particles that are 1.22 μm or less. Similarly other X_n values show the outcome of a similar procedure at other weight percentages. These results indicate that the three clay samples are suitable for most ceramic applications. However, in applications that require a dense body, for instance in electrical porcelain, Awo –Omamma and Ohia clays will give a good result.

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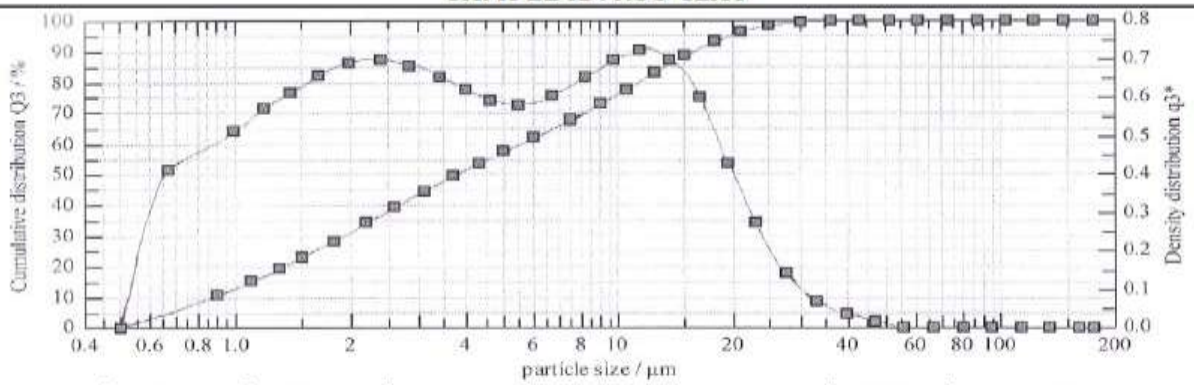
SAMPLE ID: NSU CLAY



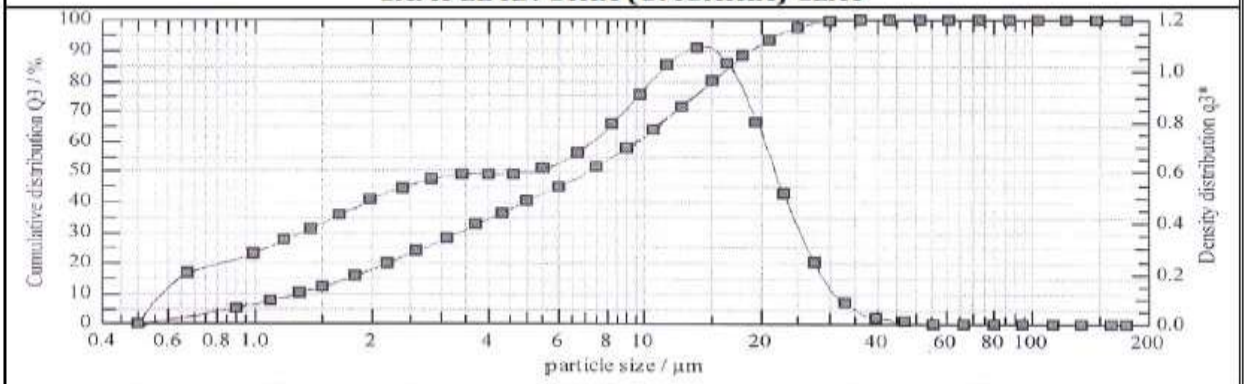
X_{10}	0.88 µm	X_{50}	3.81 µm	X_{90}	16.04 µm	VMD	6.56 µm
X_{16}	1.15 µm	X_{84}	13.03 µm	X_{99}	29.54 µm	S_m	9560.93 cm^2/g
Dispersion Medium	DI Water						

Cumulative Distribution

$X_0/\mu m$	$Q_3/\%$	$X_0/\mu m$	$Q_3/\%$	$X_0/\mu m$	$Q_3/\%$	$X_0/\mu m$	$Q_3/\%$
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. 4.1. Particle size analysis of NSU clay.

SAMPLE ID: OHIA (UMUAHIA) CLAY



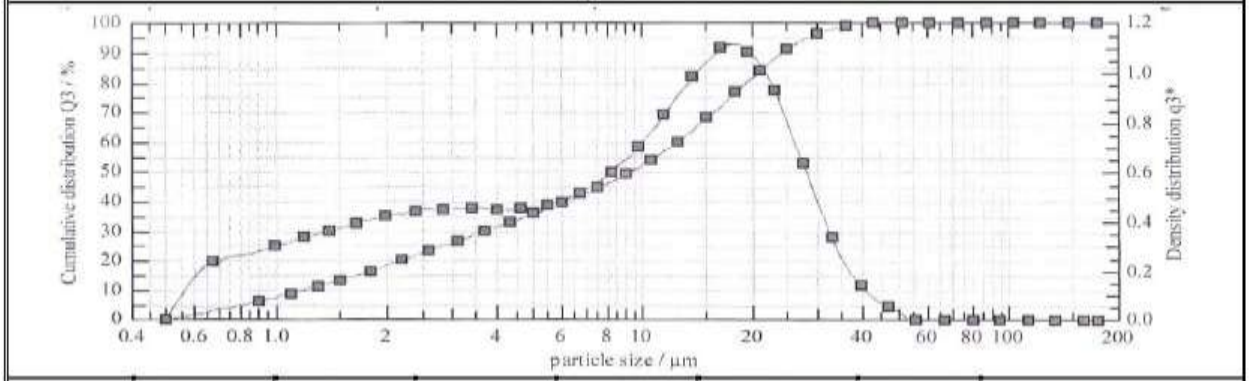
X_{10}	1.32 µm	X_{50}	7.20 µm	X_{90}	19.11 µm	VMD	8.93 µm
X_{16}	1.85 µm	X_{84}	16.51 µm	X_{99}	29.67 µm	S_m	6445.79 cm^2/g
Dispersion Medium		DI Water					

Cumulative Distribution

$X_0/\mu m$	$Q_3/\%$	$X_0/\mu m$	$Q_3/\%$	$X_0/\mu m$	$Q_3/\%$	$X_0/\mu m$	$Q_3/\%$
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. 4.2. Particle size distribution of OHIA 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

$X_0/\mu m$	$Q_3/\%$	$X_0/\mu m$	$Q_3/\%$	$X_0/\mu m$	$Q_3/\%$	$X_0/\mu m$	$Q_3/\%$
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. 4.3. Particle size analysis of UMUEJIKE AWO-OMAMA clay.

Reference to this paper should be made as follows Osonwa Nobert Okey, et. al.,(2017) Determination of the Particle Size Distribution of Some South East Nigeria Clays by Laser Diffraction Technique. J. of Physical Science and Innovation, Vol. 9, No. 2, Pp. 23 – 31