Studies on the Five (5) Selected Clays in Abeokuta, Nigeria.

O.S. Fakolujo[1*], O.S. Olokode[2], P.O. Aiyedun[2], Y.T. Oyeleke[2], B.U. Anyanwu[2], and W.E. Lee[3].

[1]Metallurgical and Materials Department, Yaba College of Technology, Lagos, Nigeria.
[2]Mechanical Engineering Department, University of Agriculture, Abeokuta, Nigeria.
[3]Materials Department, Imperial College, London, UK

E-mail: samfakolujo_1@yahoo.com

ABSTRACT

Clays from five (5) deposits in Abeokuta, South-West Nigeria have been investigated with a view to determining their suitability for various uses. The samples were collected from different commercial centers in Abeokuta; namely, Asero 1 (AS1), Fajol (FA), Agba-Akin 1 Onibode 1 (AA1), Ile-Ise Awo 1 (IA1), and Ajebo 1 (AJ1). The clay samples were crushed, pulverized, and sieved. Their chemical compositions and X-ray diffraction were determined. The mineralogical characteristics of these samples revealed kaolinite as the principal mineral but other minerals are present in small quantities: illite, quartz, muscovite, microcline, cristoballite, and zeolite. Experimental tests for refractoriness, water absorption, bulk density, linear shrinkage, percentage dry shrinkage, percentage total shrinkage, percentage warpage compressive strength breaking load/flexural tests and crack formation were carried out on each of the five selected clays.

(Keywords: clays, kaolin, mineral, bulk density, porosity, shrinkage)

INTRODUCTION

Raw materials used in the traditional ceramics industry can be classified as clay (plastic) and non-clay (non-plastic) minerals. Clays are the chief raw material for many commercial structural ceramic products such as wall tiles, roofing tiles, building bricks, and white wares. Chemically, clay minerals are phyllosilicates with ions arranged in parallel planes forming layers (G. Brown, 1984). Clays occur in deposits of greatly varying nature. No two deposits have exactly the same clay and frequently different samples of clay from the same deposit differ.

The geomorphology of the township, Abeokuta and environs is made up of flat topped hills, escapements and lowlands. The hills geologically are the olden granite masses and other rocks such as gneisses and migmatites, commonly referred to as crystalline basement or basement complex. This escapement is of cretaceous age. Alluvial terraces are well dissected by the Ogun and where underlying rock is deeply weathered. The laterite caps on these rocks resistant to the environs and it is believed that they have contributed largely to the development of the escapements and crestas [3].

According to [6], the sedimentary rocks are not well exposed. There are, however, outcrops of coarse, ill-sorted, clayey brown sandstone occurring at about 200ft. Similarly, these are outcrops of cross bedded femiginious sandstone and pebble sandstone among others in south eastern part of Abeokuta. There are also fallen blocks of conglomerates, consisting of quartz cobbles in a femiginious and cretaceous sandstone matrix.

The basic rocks from which clays are formed are complex aluminosilicates. During the weathering these become hydrolysed, the alkali and alkaline earth ions form soluble salts and are leached out. The remainder consists of hydrated aluminosilicates of varying composition and structure, and free silica. It is the orientation of this predominantly silica structure that gives clay its unique plastic property. Nigeria has appreciable distribution of industries engaged in metal and process industries hence the need for adequate and appropriate raw materials to support their growth.

Clay products such as wall tiles, ceramic wares, burnt bricks, roofing, and floor tiles are cheaper and durable building materials than cement especially under tropical conditions [7]. An optimum combination of various clays is the essential ingredient in ceramic wall tile compositions, which provides plasticity and green strength during forming stages and contributes substantially to the color of the fired products depending upon the impurity of oxides present.

The formation, structure, mineralogical and other physico-chemical properties of various types of clay minerals are widely studied subject discussed in literatures [2, 4, 5, and 8]. Two factors are accentuating the development of good refractories using the local raw materials. The first one is the growing number of metallurgical industries that are in dire need of these refractories, while the other factor is the advent of foreign exchange market, a situation that has led to higher and unaffordable cost of procuring the refractory materials needed by these industries. Some of the refractories materials usually employed are fireclay, quartz sand, magnesite, sillimanite, berylia, alumina, chromite, zirconia, boron, nitride, graphite and carbide.

This study investigate the working properties of the Asero, Fajol, Onibode, Ile-Ise Awo, and Ajebo clay deposits in Nigeria with the view to propose other possible uses such as microfiltration membranes in water treatment, food, and beverage production, chemical engineering and refractory processes.

MATERIALS AND METHODS

Raw Materials

Representative samples of the five (5) different selected clays from Abeokuta in Ogun-State were taken by hand (as received) from five (5) different geological formations at their respective depths in the pits of Asero 1, Fajol, Onibode 1, Ile-Ise Awo 1, and Ajebo 1.

Characterization of the Five Different Clay Deposits in Abeokuta

The physical, chemical, and mineralogical properties of the natural kaolins from Abeokuta clay was examined in the Materials Laboratory of Imperial College, London.

The results were compared with results published in literature, to ascertain if these five clays are suitable for the manufacturing of floor tiles, ceramics, stoneware, and firebricks.

Sample Preparation

All five collected clays, in the form of agglomerated lumps of five (5) kilograms, were initially dried at 110°C over night for 24 hours, in an oven MOV-112 of Imperial College London, and were grounded in a porcelain mortar and pestle and the resulting fine powder was termed the ″un-treated clay″.

Chemical Analysis

Chemical analyses of the materials were determined using inductively coupled plasma. atomic emission microscopy (ICP- Varian Vista-Pro ICP-AEP), where all sample were dried at 110°C for eight (8) hours prior to analyses.

Samples were analyzed by fusion with Lithium Barium ($LiBO_2$) into the plasma flame to produce characteristic emission of major and minor elements.

Mineralogical Analysis X-Ray Diffraction (XRD)

Each of these 5 clay samples was dried in an oven, crushed, ground to a fine powder, and was determined in the laboratory section of Materials Department, Imperial College London. The mineralogical data were obtained on powdered samples using XRD instrument Phillips PW1700 series automated powder diffractometer using CuK_α radiation at 40 Kv/40mA from $5° < 2\theta < 85°$ with the step size 0.04 at the counting time of 2 seconds. Both random and oriented powder samples were studied and were compared with International card for diffractometer data (ICDD).

Refractoriness

Each of the five test cones were prepared by mixing clay deposits aggregate with sufficient quantity of water to make the clay plastic and moulded by hand into cone shape. Each clay samples were dried and fired to a temperature of 900^0C in a muffler furnace. Pyrometric cones designed to deform at 1250, 1300, 1350, 1400, 1500^0C were placed round all the samples and the temperature raised to above 1000^0C at 10^0C per min. The heating was discontinued when the test cone bent over and leveled with the base of the disc. The Pyrometric Cone Equivalent (P.C.E) of each clay samples recorded to the number of standard pyrometric cone corresponding in the time of softening to the test cone.

Pressing

Dry pressing is commonly used to produce ceramic particles thicker than 0.5mm. Stages of dry pressing include: filling of the die, compaction, shaping and ejection.

Approximately 8mm diameter die were pressed using a laboratory hydraulic press Model 15010. The desired pressure applied, 40 MPa was maintained for about 60 seconds to allow for particle rearrangement. Freshly compacted test specimen were measured, weighed, marked for further identification and dried in an oven type MOV 112 at 110°C overnight prior to firing. Dry shrinkage, fired shrinkage and total shrinkage of these samples was determined at various temperatures of 600, 700, 800, 900, 1000, 1100, and 1200°C. A micrometer with accuracy of 0.01mm was used to measure the length of the samples directly after pressing, and the length of the dried samples. After firing at specific temperatures, the fired length for each temperature was measured. The formula for the calculation of dry shrinkage is:

$$\%DS = \frac{L_2 - L_3}{L_2} \times 100 \quad (1)$$

where, DS = Dry Shrinkage
L_2 = Length of sample directly after pressing (mm)
L_3 = Length of dry pressed sample (mm)

The formula used for the calculation of the fired shrinkage at each temperature is:

$$\%TS = \frac{L_2 - L_4}{L_2} \times 100 \quad (2)$$

where, TS = Total Shrinkage
L_2 = Length of sample directly after pressing (mm)
L_4 = Length of fired sample at a specific individual temperature (mm)

Water Absorption

Water absorption measurements were carried out according to British Standard EN 99 (1991). Fired pellets were soaked in boiling water in a beaker, and left to cool down to room temperature still soaked in water. Excess water was removed from the pellets surface using a moistened cloth prior to weighing. The wet weight (W_w) was then measured. Pellets were left drying in an oven at 110°C overnight, and the dry weight was measured (W_d). Water absorption (WA) was computed as:

$$WA = \frac{W_w - W_d}{W_d} \times 100 \quad (3)$$

As boiling water will penetrate open or interconnected pores of the specimen, the higher the water absorption the more porous is the material in terms of that specific type of porosity. Open pores are common in under fired stoneware, where not enough liquid has been produced to close the porosity of the ware on firing. In over fired specimens, gas escaping from inside the ware will create blistering on its surfaces.

Bulk Density

The bulk density of the fired samples was determined by water displacement using Archimedes principle. The experimental set-up allows the weights of the beaker partially filled with water, with and without the pellet immersed in water, to be measured. The difference gives weight of water displacement, according to the Archimedes principle. The weight of water displaced can be easily related to the volume of water displaced, as the density of water is known. The volume of immersing water was determined using a precision balance. Thus, the bulk density

(BD, g/cm³) is given in terms of sample mass (S_m) and the sample volume (S_v) as:

$$BD = \frac{S_m}{S_v} \quad \textbf{(4)}$$

Assuming that the composition of the glassy phase and that of the crystalline materials present in the samples fired at neighboring firing temperatures, and thus their theoretical density, are very similar, it is then possible to use bulk density measurements to give an idea of the total porosity of the ware: the higher the bulk density, the lower the total porosity.

Linear Shrinkage

Dry-to-fired contraction or linear shrinkage (LS) was determined by measuring the diameter of the dried pellet (D_d) and that of the fired pellet (D_f), according to the following equation:

$$LS = \frac{D_d - D_f}{D_d} \times 100 \quad \textbf{(5)}$$

The measurements were made using a digital caliper. As the ceramic body densities, it shrinks in all direction. The shrinkage along the disk diameter gave more results than that of the height.

Pressing Moisture

A representative batch of pressed test pieces were weighed (M2), dried at 110°C for 24 hours and then weighted (M1) again. The difference in mass was divided by the initial mass, multiplied by the initial mass, multiplied by the average pressing moisture of the sample is:

$$\%MC = \frac{M_1 - M_2}{M_2} \times 100 \quad \textbf{(6)}$$

where, MC = moisture content
M1 = weight of the sample after drying
M2 = weight of the sample after pressing.

Thermal Shock Resistance

Each of the five test specimens of area 0.002m^2 and thickness of 0.01m were cut from their respective bricks. A conical flask containing 50ml of water was placed directly above and in contact with specimen. A cork having a thermometer passing through it was used to cork the mouth of the conical flask. The thermometer reads the temperature changes of the water in the flask. The test section was then closed and the initial water temperature was noted. A second thermometer with the aid of a cork was inserted into the stream outlet pipe offset to monitor the stream temperature so as to ensure a constant base temperature of 100^{0}C.

The boiler water outlet valve was closed while 5 liters of water was measured and poured into the boiler. The stream inlet valve, outlet valve and condensate outlet valve were all closed. With the boiler cover remaining opened, the boiler was switched on. Immediately, the water started boiling, the boiler cover was closed, while the steam inlet valve was fully opened with all the remaining valves closed. Timing commenced with the aid of a stopwatch immediately the steam inlet valve was opened.

The testing was timed in each case for 10 minutes and final temperature of the water in the beaker was noted at the end of time. Each specimen was tested (experimented) twice and mean temperature value was obtained. At the end of each experiment, the steam outlet valve was opened to release steam. The water in the boiler was refilled to maintain 5 liters and the experiment was repeated as stated above for the other specimens.

The value of the thermal conductivity, K for each of the specimen was determined using the formula:

$$K = 2.303MCL/A\ [\log(\theta_1\theta_2)] \quad \textbf{(7)}$$

where: K = Thermal conductivity of the specimen,
T_1 = Temperature of steam K.
T_i = Initial temperature of water in conical flask,
T_4 = Final temperature of water in conical flask,
τ = Time (s),
A = Specimen area (m^2),
M = mass of water in conical flask (Kg)
C = Specific heat capacity of water in conical flask (J/KgK),

$L =$ Thickness of specimen (m),
$\theta_1 = T_1 - T_i$,
$\theta_2 = T_1 - T_4$.

Percentage Warpage

Warping is the measurement of curvature (convex or concave) of deviations of material surface from a true plane along the edges or the diagonals. The deviations were measured at the mid-length of an edge or diagonal and expressed as percentage of the length of the edge or diagonal. The diagonal method was used by initially setting a dial indicator to zero at the mid- centre of each of the five clay deposits and later moving the dial indicator to record deviations of points along the drawn diagonal lines on the moulded clay.

The percentage warpage were calculated using Equation (8):

$$W_L/D_L \times 100 \quad \textbf{(8)}$$

where, W_L is the amount of warpage in mm.

D_L is the gauge length or total length of the diagonal.

The recorded percentage warpage is based on average percentage warpage of at least two times of each of the five clay deposits.

Mechanical Properties

The mechanical properties, compressive strength and breaking load were conducted using Equations 8 and 9 in accordance with ASTM standards for testing structural clay materials.

Compressive Strength

Each specimen from the five selected clay deposit was placed on hardened steel plate and load was gradually applied by manually operated hydraulic press through an indenter or load applicator until there was a sign of crack. The compressive test was calculated using Equation 9.

$$б_C = W/A \quad \textbf{(9)}$$

where, $W=$ the maximum attained (N)
$A=$ the area of load allocation

The recorded compressive strength was an average of the two times of each of the five clay selected deposits.

Breaking Load/Flexural Test

This test method consisted of supporting the tile specimens on the ends of three cylindrical rods, arranged in an equilateral triangle form and applying a load, until it fail. The recorded flexural strength was an average of the two times of each of the fire clay deposits.

RESULTS AND DISCUSSION

Figures 1 to 5 showed the X-ray Diffraction (XRD) of the five clay samples. Asero 1 and Fajo 1 had the highest intensity of Q-Quartz at 10,000 intensity while Agba-Akin clay 1 had the least intensity of XRD at 27 position (^{0}2Theta) copper. The Ajebo1 clay XRD displayed K-Kaolin as the major content of the clay.

From Figure 6, the five selected clays samples had the same Experimental Physico-Mechanical Properties but deviated after the compressive length (MPa). Ile Ise Awo1 (IA1) had the highest Crack Formation while Agba-Akin1 (Onibode had the least Crack Formation). Ajebo1 (AJ1) and Ile Ise Awo1 (IA1) had almost the same Experimental Physico-Mechanical Properties.

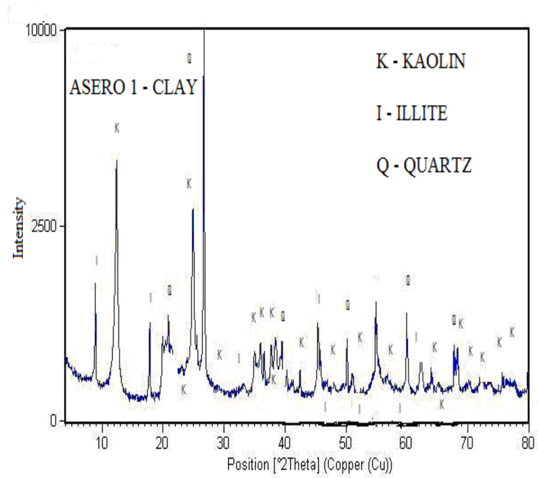

Figure 1: XRD Trace of Asero Clay.

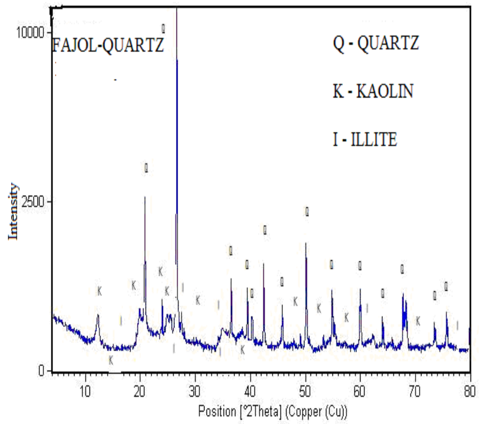

Figure 2: XRD Trace of Fajol Clay.

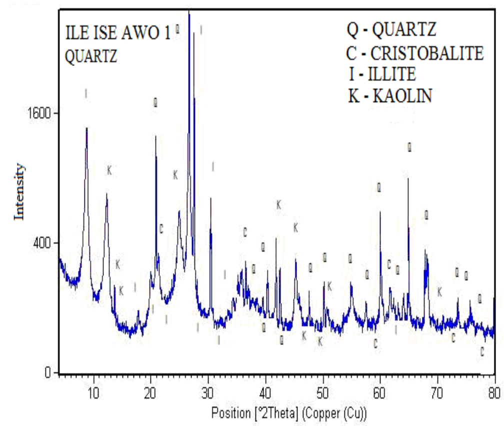

Figure 4: XRD Trace of Ile-Ise.

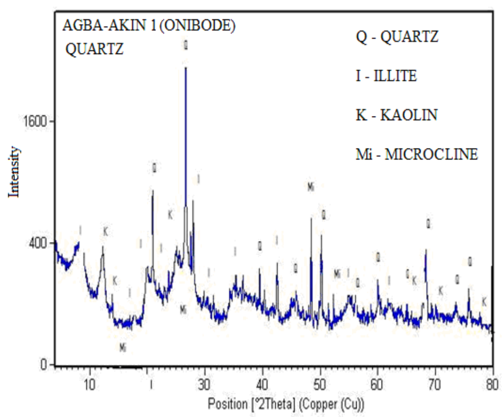

Figure 3: XRD Trace of Agba-Akin Clay 1 Awo 1.

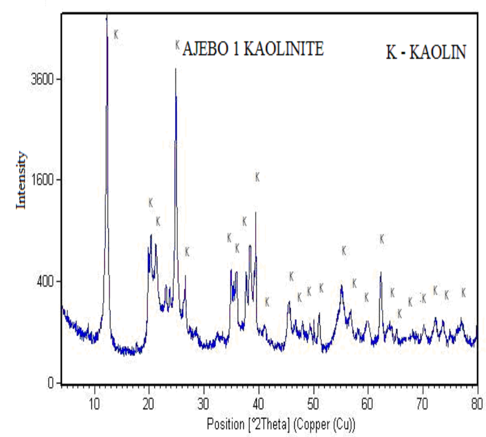

Figure 5: XRD Trace of Ajebo 1 Clay.

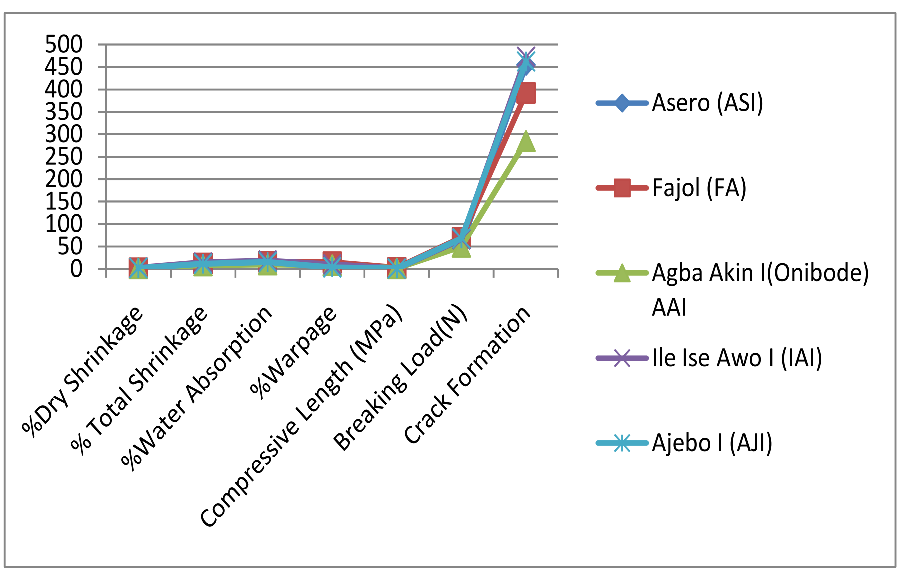

Figure 6: Experimental Physico-Mechanical Properties of the Five Selective Clays in Abeokuta at 1200^{0}C.

Table 1: Chemical Composition of five (5) clays deposits in Abeokuta, Nigeria (n.d.- Not detectable).

Raw Materials (RM)	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	LOI
Asero (AS1)	66.8	0.45	21.3	2.94	n.d	0.07	0.08	0.02	0.45	0.06	8.43
Fajol	78.3	0.87	9.1	2.94	0.02	0.31	0.26	0.24	1.01	0.03	6.92
Agba-Akin 1(Onibode) AA1	63.7	0.7	18.7.	5.6	0.02	1.0	1.1	1.9	0.04	0.03	5.89
Ile-Ise Awo 1 (IA1)	67.9	0.6	14.7	5.9	0.03	0.8	0.4	0.49	2.46	0.05	6.67
Ajebo1 (AJ1)	46.4	1.69	34.0	2.49	n.d	0.04	0.02	0.03	0.08	0.04	17.7

Table 2: Experimental Physico-Mechanical Properties of the Five Selective Clays in Abeokuta at 1200^{0}C.

Clay Deposits	Properties							
	Bulk density (g/cm^3)	%Dry Shrinkage	% Total Shrinkage	%Water Absorption	%Warpage	Compressive Length (MPa)	Breaking Load(N)	Crack Formation
Asero (ASI)	1.26	9.53	11.91	10.73	1.82	54.53	454.15	No Crack
Fajol (FA)	1.62	12.26	15.61	14.77	2.12	69.17	391.88	No Crack
Agba Akin I(Onibode) AAI	1.24	7.59	9.56	8.24	1.09	49.10	284.60	Slight Crack
Ile Ise Awo I (IAI)	2.46	14.57	18.56	9.94	0.95	65.51	472.50	No Crack
Ajebo I (AJI)	1.79	11.21	14.92	3.34	0.82	67.90	461.83	No Crack

Table 1, displayed the Chemical composition of the five selected clays, Ajebo1 (AJ1) had the highest amount of α-alumina (Al_2O_3) of 34.0% which made it the purest of all. Fajo I clay had the highest amount of silica $(SiO)_2$ while Ajebo1 had the least.

Both Ajebo1 and Asero (ASI) did not have any significant traces of magnesite. The illite content displayed highest amount in Ajebo 1 (AJ1) sample while Agba-Akin 1 (Onibode) (AAI) had the lowest illite.

ASTM -Allowable Value for ceramic tiles on sale states the % warpage maximum should not be more than 2.1%, 15% maximum for total shrinkage and % water absorption should not be more than 16% maximum.

REFERENCES

1. Carly, W.M. and U. Senapati. 1998. “Porcelain Raw Materials, Processing Phase Evolution and Mechanical Behaviour”. *Journal of American Ceramic Society*. 81(1):3-20.

2. Das, S.K, K. Dana, K. Singh and V. Sarka. 2005. “Shrinkage and Strength Behaviour of Quartzitic and Kaolinitic Clays in Wall tile Composition”. *Applied Clay Science*. 29:137- 143.

3. Durotoye. 1975. *Quaternary Sediments in Nigeria*. Elizabethan Publishing Co.: Lagos, Nigeria. 347-362.

4. Hinkley, D.W. 1962. “Variability Crystallinity Values among the Kaolin Deposits of the Coastal Plain of Georgia and South Carolina”. *Proceedings of the 11th National Conference on Clays and Clay Minerals*. Ottawa, Canada. 229-235.

5. Kingery, W.D. 1976. *Introduction to Ceramics*. Wiley: New York, NY. 78-79:532-540.

6. Kogbe, C.A. 1975. *Soil Survey Report Bulletin*. No 46.

7. Nnuka, E.E. and C. Enejo. 2001. “Characterization Nahuta Clay for Industrial and Commercial Applications”. *Nigerian Journal of Engineering and Materials*. 2(3): 9-12.

8. Olgun, A., Erdogan, Y., and B. Zeybek. 2005. Development of Ceramic Tiles from Coal Fly Ash and Tincal Ore Waste”. *Ceramic International*. 31:153-158.

ABOUT THE AUTHORS

Engr. O.S. Fakolujo, is a qualified engineer in Metallurgy and Material Engineering. He is the Chief Lecturer and Head of Metallurgical Department, Yaba College of Technology. He is currently completing his Ph.D. (Materials) at the University of Ottawa in Canada.

Dr. Engr. O.S. Olokode, is a Senior Lecturer and Acting Head of Mechanical Engineering Department, Federal University of Agriculture, Abeokuta. He holds a Ph.D. from both Imperial College, London and Federal University of Agriculture, Abeokuta.

Prof. P.O. Aiyedun, is a Professor of Mechanical Engineering, Federal University of Agriculture, Abeokuta. He is a registered Engineer and a Fellow of the Nigerian Society of Engineers. He holds a Ph.D. in Metallurgy and Material Engineering. He was a former Dean, College of Engineering, Federal University of Agriculture, Abeokuta-Nigeria. His research interest is in the area of steel production and material engineering.

Engr. B.U. Anyanwu, is a Senior Staff in the Federal University of Agriculture, Abeokuta. He is a registered Member of the Nigerian Society of Engineers.

Y.T. Oyeleke, holds Bachelor in Engineering from the Federal University of Agriculture, Abeokuta.

Professor W.E. Lee, is a Professor and Doctor of Science in Materials at Imperial College, London and former Head of Materials Department.

SUGGESTED CITATION

Fakolujo, O.S., O.S. Olokode, P.O. Aiyedun, Y.T. Oyeleke, B.U. Anyanwu, and W.E. Lee. 2012. "Studies of Five (5) Selected Clays in Abeokuta, Nigeria". *Pacific Journal of Science and Technology*. 13(1):83-90.