

STRENGTH CHARACTERISTICS OF NIGERIAN QUARTZITIC AND KAOLINITIC CLAYS IN WALL TILE COMPOSITION

***¹O.S. OLOKODE, ¹P.O. AIYEDUN, ²G.O. OLUWADARE
³Y.T. OYELEKE AND ⁴W.E. LEE**

^{1,3} Department of Engineering, Federal University of Agriculture, Abeokuta, Nigeria.

²Bells University, Ota, Ogun State, Nigeria.

⁴Materials Department, Imperial College, London United Kingdom.

Corresponding author: engr_olokode@yahoo.com

Tel: +23408169677160

ABSTRACT

Clays from four different deposits in Abeokuta, Nigeria (Abiola Way 1 (AW1), Abiola Way 2 (AW2), Asero (AS), Mysis (MY)) have been investigated. Their chemical compositions were determined by Atomic absorption spectroscopy and the most crystalline phases were identified by X-ray diffraction. Wall tile specimens were formulated with clay, wood ash (flux) and quartz (filler). The compositions of the wood ash and quartz were kept constant in the wall tile composition. The results revealed that the wall tile composition containing higher amount of quartzitic clays, exhibited lower shrinkage, higher water absorption rate while lower strength and the wall tile containing higher amount of kaolinitic clay showed higher shrinkage, higher strength due to better densification and lowest water absorption rate in the temperature range of 1050-1150°C.

Keywords: Kaolinitic clay; Quartzitic clay; Wall tile; Wood ash; Low shrinkage; composition.

INTRODUCTION

Raw materials used in the traditional ceramics industry can be classified as clay (plastic) and non-clay (non-plastic) minerals. Both types are used to manufacture triaxial wall tiles (F.H. Norton, 1973) which comprise clay, flux and filler system. Clay is 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). 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 (Nnuka *et al*, 2001). 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. Recently, the increasing demand of ceramic tiles for both the floor and wall has made researchers to become interested in

developing this field (Das *et al.*, 2005; Olgun *et al.*, 2005).

The formation, structure, mineralogical and other physico-chemical properties of various types of clay minerals are widely studied subject discussed in literature (Hinkley, D.W, 1962; Kingery, W.D 1976; Murray *et al.*, 1993; Carty *et al.*, 1998). Other important materials that are traditionally used in making wall tiles are carbonates, which are commonly selected from chalk, limestone, marble and dolomite. These carbonate materials form a fusible eutectic with alumina and silica (Yatsenko *et al.*, 1998) and also act as fusible minerals. New multi purpose bodies containing various clays, wollastonite and calcium carbonate with other pyrophyllite, feldspar and sand for both wall and floor tiles had been reported (Brusa *et al.*, 1995; Dana *et al.*, 2002). Effect of partial substitution of clay by flash ash in the tile production has also been studied (Das *et al.*, 1996; Kumar *et al.*, 2001; Shah *et al.*, 2001; Moore, D.M *et al.*, 1997) reported an increase in strength up to 25-30% fly ash addition, beyond which the strength decreased. Because fly ash is calcined material, it has very low shrinkage which is beneficial in the wall tile compositions. In the present investigation, four different clay deposits were characterized with respect to their chemical, mineralogical, fired, and physical properties. These clays were incorporated in different proportions in the wall tile compositions keeping other raw materials the same. The effect of these clays on the properties of wall tile body was studied by measuring their linear shrinkage, bulk density, water absorption and flexural strength.

MATERIALS AND METHODS

Material

Representative samples (50kg each) of four

clay samples from Abeokuta, Ogun State, Nigeria was taken from three different geological formations namely (Abiola-way 1 & 2, Asero, and Mysis). Wood ash was used as fluxing material and quartz sand as filler material. Wood ash and quartz were got from Camp area, in Abeokuta, South West, Nigeria

METHODOLOGY

Chemical analyses of the materials were determined using Atomic Absorption Spectroscopy (AAS).

Each of these samples was initially sun dried in the open air, and was manually crushed in a mortar agate. The crushed sample was sieved using 80 mesh. The sample of the dry ground powder was homogenized and granulated in a high intensity mixer with moisture content of 10% (moisture mass/dry mass). After reducing the moisture content to 5%, the granulates were kept in a desiccator for 24h. to homogenize their moisture content. The granulated powder was sent to the sieve to eliminate agglomerates greater than 2 μ m. Fired characteristics of all the clays were studied separately by preparing cylindrical rods (30 \times 40mm) from the prospective clay powder followed by oven drying at 110 $^{\circ}$ C and firing at 1000 $^{\circ}$ C with 2h soaking in an electric furnace. The fired clays cylindrical rods were tested for linear shrinkage, bulk density, and water absorption. A non clay material, wood ash was also chemically analyzed, and XRD identified. The ceramics formulation prepared were characterized by X-ray diffraction (XRD) MD 10 Radicon, version 2.00.1 using CuK α radiation from 5 $^{\circ}$ <2 θ <80 $^{\circ}$, exposure time 1200/1200sec. Microstructural features of the fractured specimens were examined by SEM (JSM-5000).

Mineralogical analysis by X-RAY Diffraction (XRD)

The mineralogical composition of the unfired individual four clay samples, wood ash and quartz were determined on powdered samples using XRD instrument; diffractometer MD10. Radicon MD100.000UM, Version 2.00.1 using $\text{CuK}\alpha$ radiation from $5^\circ < 2\theta < 80^\circ$, exposure time of 1200/1200sec.

Mechanical Properties

Cylindrical rod of dimension (30×40mm) was prepared for each of the four clay samples. Using die mold, uniaxial compaction at a pressure of 40MPa. The compacted cylindrical rod dried at 110°C till the moisture content was reduced to less 0.5 wt% were dried in the temperature range of 1050-1150°C for a soaking time of 2h. in an oxidizing electric furnace.

Linear Shrinkage

Linear shrinkage (LS) was determined by measuring the diameter of the dried rod L_d , and that of the fired pellets L_f .

The linear shrinkage was obtained according to the following equation:

$$LS = \frac{(L_d - L_f)}{L_d} \times 100 \quad (1)$$

where LS=Linear shrinkage

L_d = Length of dried sample,

L_f =Length of fired sample

The shrinkage along the disk diameter gave more accurate results than that of the height.

Water Absorption

Water absorption measurements were carried out by measuring fired sample weight

and soaked sample weight. Fired sample of each of the four clays were weighed, W_f . Fired samples were then soaked in boiling water. The wet weights, W_w of the samples were then measured.

Water absorption (WA) was computed as

$$WA = \frac{(W_w - W_d)}{W_d} \times 100 \quad (2)$$

where WA=Water absorption

W_w =Wet weight,

W_d = Dried weight

Flexural Strength

Flexural strength was determined for all the four clay samples (each 30mm length by 40mm diameter) using universal testing machine (Zwisch/Roell) in a 4 point bending fixture, adjusted to 25mm support span, and with a crosshead speed of 5mm/min. The average flexural strength, σ was calculated via the least squares method and linear regression analysis, adopted as probability estimation of failure.

$$P_N = (i-0.5)/N$$

RESULTS AND DISCUSSION

Table 1 show the chemical composition of the samples which have been determined by Atomic Absorption Spectroscopy (AAS). Concentrations of SiO_2 greater than the theoretical value can be explained by the presence of various amount of quartz in all the samples indicated by X-ray diffraction to (hydr)oxides with water release. Chemical analysis revealed that Abiola Way 1&2 (AW1&2) were rich in quartz while Asero (AS) and Mysis (MY) were rich in alumina. Figs. 1-4 showed that clay deposits Abiola

Way 1&2 (AW1&2) are quartzitic, while Asero (AS) and Mysis (MY) are kaolinitic clays. The presence of alkali oxide in these clay deposits indicates their excellent fluxing ability during firing at comparatively low temperature, to form glasses of complex composition towards giving a vitreous structure to ceramic bodies. Clay deposit of Mysis showed the highest lime compositions while clays from the other three deposits have lower lime composition. These oxides in Mysis clay deposit act as fluxes to

form a fusible eutectic with alumina and silica (Yatsenko *et al.*, 1998). The wall tile composition contained 50 mass content of Abiola way (AW1) with 25 mass content of Asero clay, 15 mass content of quartz and 10 mass content of wood ash as M1, while Abiola way 2 (AW2) has 25 mass content of clay, 50 mass content of Mysis clay, 15 mass content of quartz and 10 mass content of wood ash as M2.

Table 1: Chemical composition (wt %) of the four clays from Abeokuta

	SiO ²	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	P ₂ O ₅	ZnO	MnO	CuO	LOI
Abiola-way 1	74.7	12.8	0.10	0.7	0.2	0.1	0.7	n.d	n.d	0.1	0.6	9.97
Abiola-way 2	64.0	10.3	0.1	0.6	0.3	0.1	0.7	n.d	0.1	n.d	0.8	22.98
Asero	41.7	30.5	0.04	0.7	0.3	0.1	1.0	n.d	0.1	0.1	0.8	24.8
Mysis	47.5	35.4	0.1	3.4	0.1	0.03	0.1	0.01	0.10	0.02	0.20	13.04

n.d.= Not detected.

LOI- Loss on ignition

The characteristic of the clay samples after firing at 1000°C are given in Table 2. From Table 2 it was observed that percent linear shrinkage (%LS) of clays Abiola Way 1&2 (AW1&2) at 1000°C is significantly lower at (0.33%), compared to other clays (>5%) and this is very advantageous for wall tile compositions. This may be due to the presence of more quartz in clays of Abiola Way 1&2 (AW1&2) (Swampa *et al.*, 2005). Lower ranges of percentage absorption %WA)16-18) in clays (AW1) and (AW2) indicate bet-

ter vitrification at 1000°C compared to others chemical analysis of non clay materials are shown in Table 2. It is noted that the wood ash contains around 1.92 content Fe₂O₃ and 10.93 mass content losses on ignition as indicated in Table 4. This may be due to unburnt carbon, but the presence of low amount of iron oxide may be used as observed by many authors (Das *et al.*, 1996; Kumar *et al.* 2001; Shah *et al.* 2001). The oxide composition of the experimental wall tile bodies are given in Table 5. It is observed

that there is no significant variation in the oxide constituents between the bodies due to the optimal combination of different clays used in the present study keeping other raw materials the same. Fig.5 shows the results of linear shrinkage observed with the increase in firing. No significant increase in shrinkage observed for specimen M2 at temperatures 1100-1150°C. Fig.6 shows the variation in water absorption content with heating temperatures. The per-

cent water absorption results showed specimen M1 to be the higher at temperature 1050-1100°C whereas at temperature 1150°C, both M1 and M2 showed no significant variation with heating temperature. The result of flexural strength with increased with temperature in all the specimens (see Fig 7). Strength of the specimen M2 was found to be significantly higher compared with specimen M1 at all temperatures due to better densification.

Table 2: Characteristics of fired clay samples (1000°C, 2h soaking)

Clays	% Linear shrinkage	% Water absorption	Flexural strength (N/mm ²)
Abiola-way 1	0.33	16.2	22.60
Abiola-way 2	0.33	18.3	23.65
Asero	6.67	23.6	27.84
Mysis	5.00	22.5	26.70

Table 3: Composition of two batches of the wall tile specimen (wt. %)

Raw Materials	Wall Tile Specimen, M1 (%)	Wall Tile Specimen, M2 (%)
AW 1	50	-
AW 2	-	25
AS	25	-
MY	-	50
Quartz	15	15
Wood Ash	10	10

Table 4: Chemical composition by mass content of flux and filler used

Materials	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	P ₂ O ₅	ZnO	MnO	CuO	LOI
Wood ash	43.20	5.84	1.92	22.8	0.7	7.89	3.2	0.61	1.24	1.28	0.26	10.9
		6		5			2					3
Quartz sand	49.08	11.30	4.00	4.20	n.d	2.20	0.5	n.d	n.d	n.d	n.d	28.7
												2

n.d = Not detected

LOI – Loss on ignition

Table 5: Oxide composition of wall tile specimens (wt %)

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	P ₂ O ₅	ZnO	MnO	CuO	LOI
M1	59.46	16.31	0.85	3.45	0.26	1.17	1.00	0.07	0.15	0.20	0.53	16.59
M2	51.43	22.56	0.87	4.77	0.21	1.17	0.63	0.07	0.20	0.14	0.33	17.69

LOI- Loss On Ignition

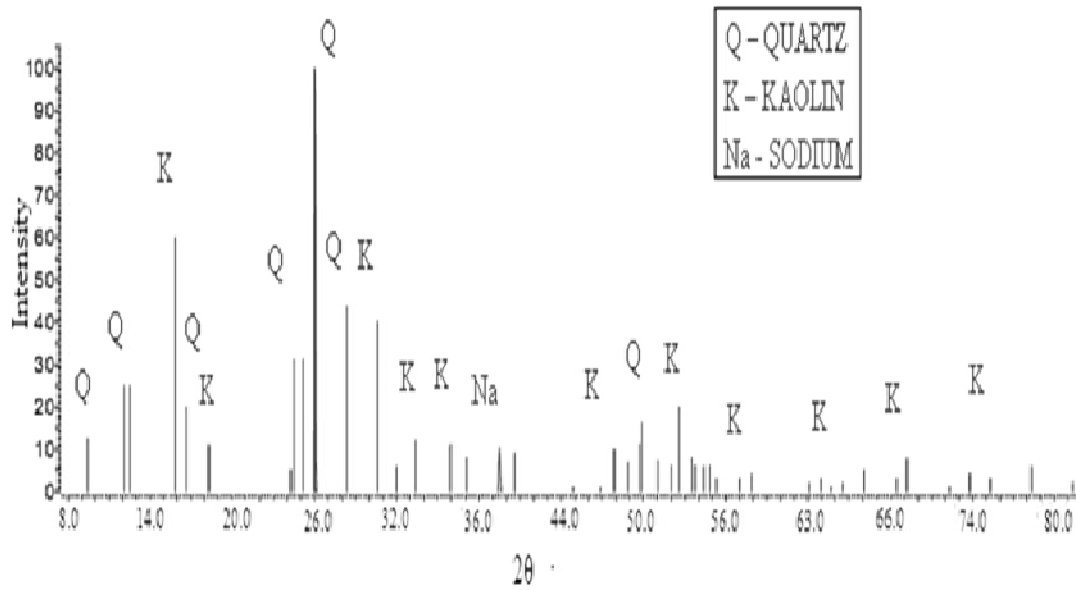


Figure 1: Peak analysis of XRD traces of Abiola-way 1

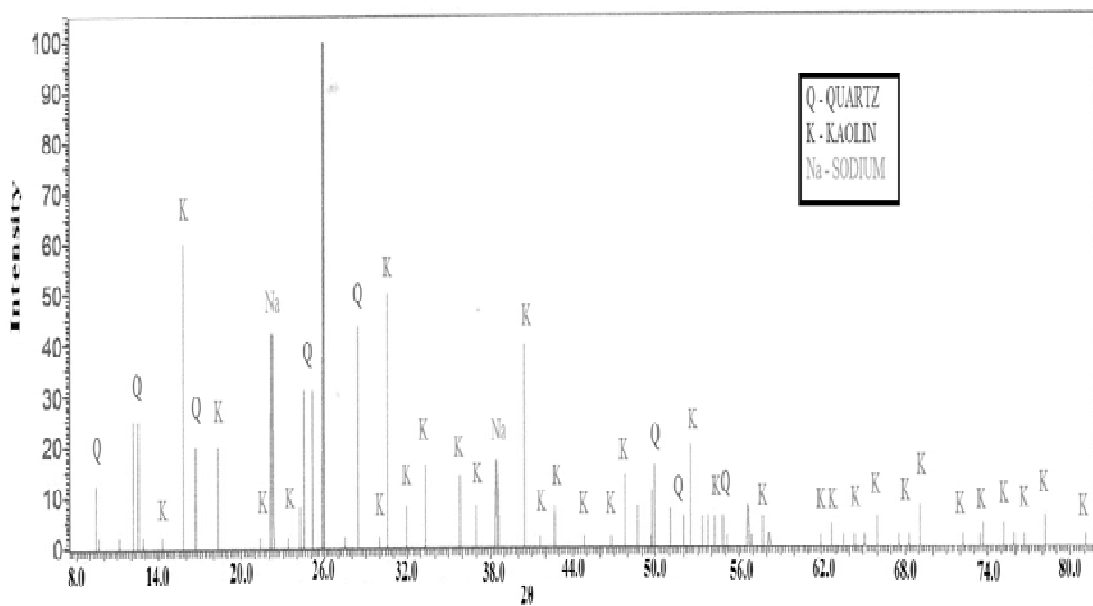


Figure 2: Peak analysis of XRD traces of Abiola-way 2 clay

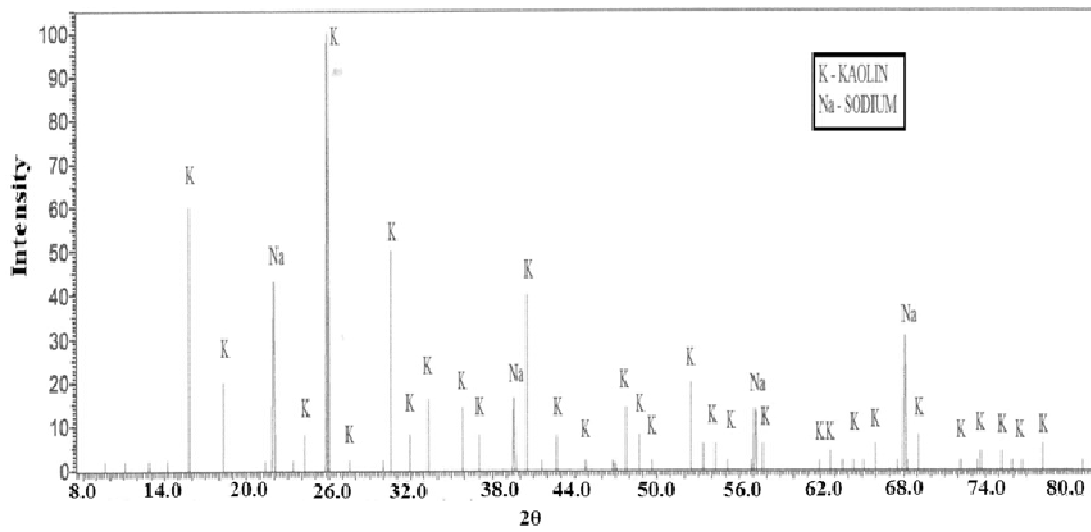


Figure 3: Peak analysis of XRD traces of Asero clay

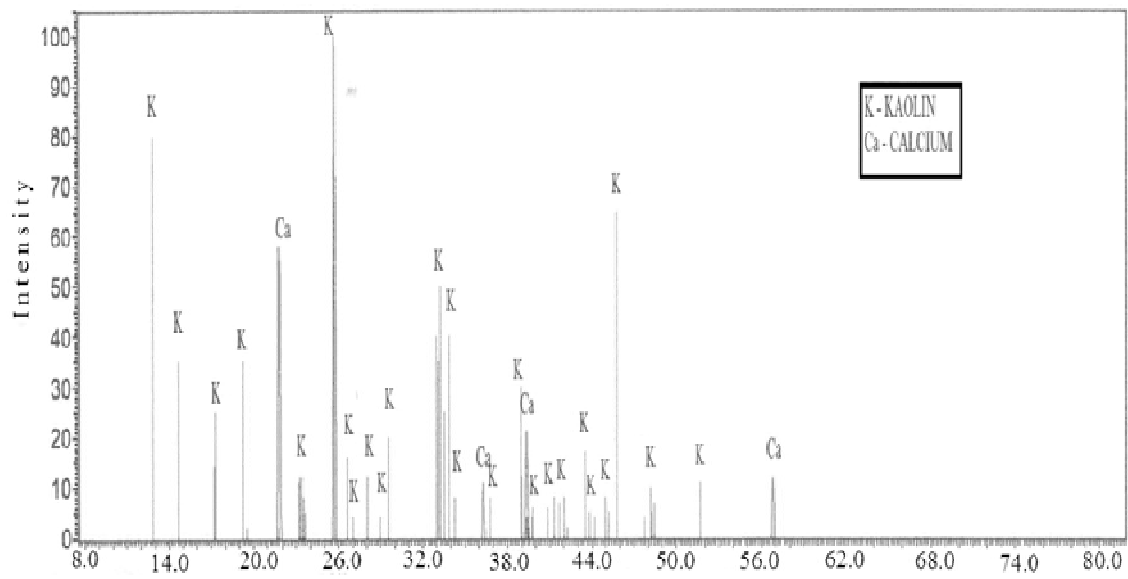


Figure 4: Peak analysis of XRD traces of Mysis

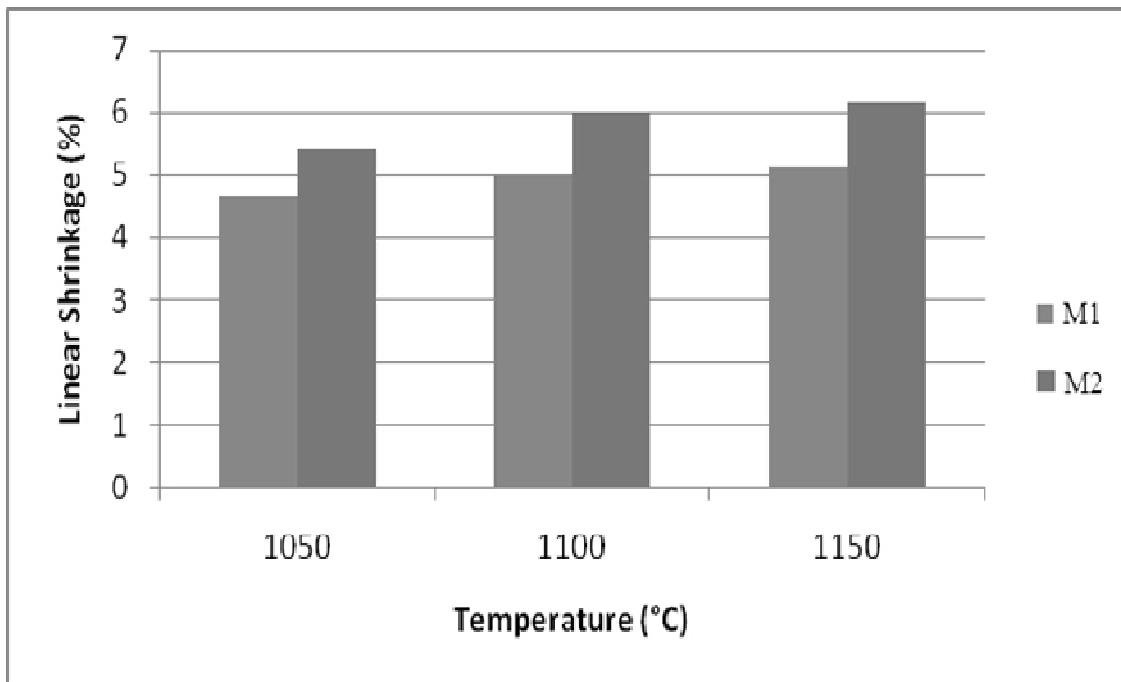


Figure 5: Variation in linear shrinkage with temperature

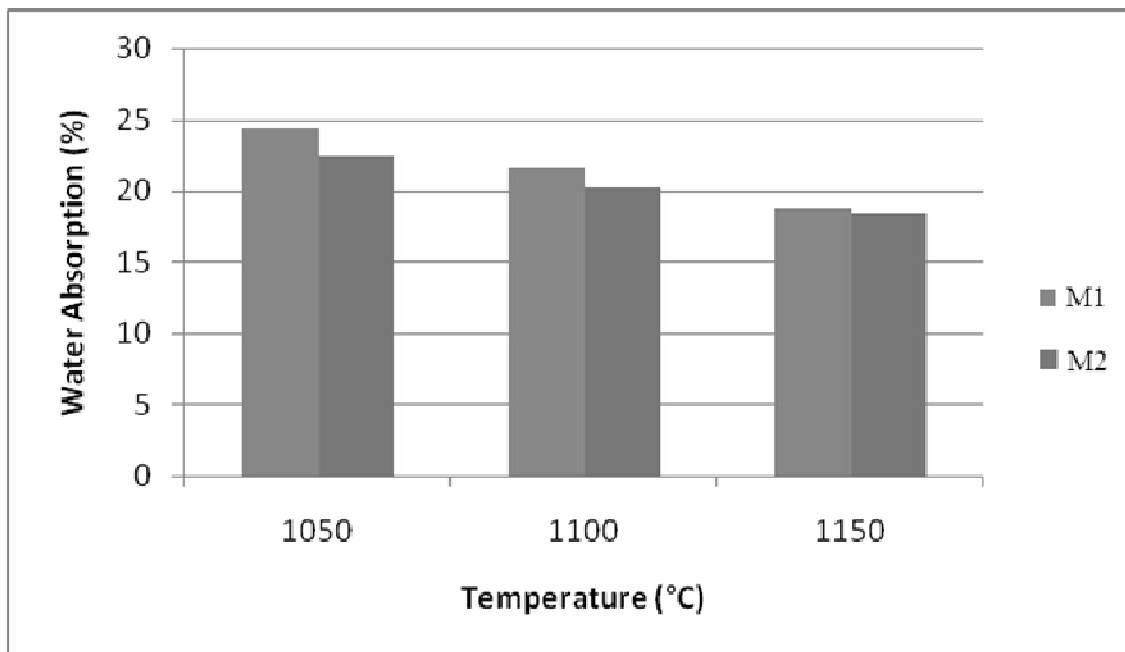


Figure 6: Variation in water absorption with temperature

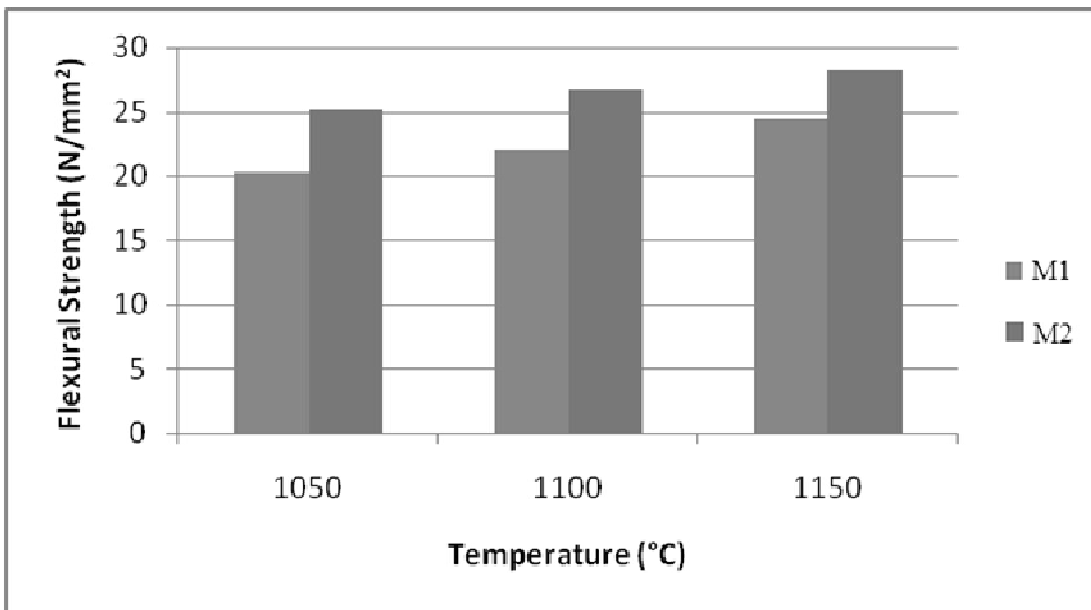


Figure 7: Variation in flexural strength with temperature

CONCLUSION

Four different clays from Abeokuta, South West Nigeria were used in formulating wall tile compositions along with other raw materials including wood ash and quartz. The tile composition with a combination of more quartzitic clays showed shrinkage, high absorption and adequate densification with low strength value, whereas the composition with kaolinitic clays showed higher shrinkage, lower water absorption and higher densification with strength values.

ACKNOWLEDGEMENT

This research at Imperial College, London for Dr. Olokode O.S was supported by the commonwealth scholarships commission programme. He gratefully acknowledge the Material Department of Imperial College for providing the equipment used and extends his thanks to Mr. Richard Sweeny and

Dr. Mahmood Adrakani for their experimental assistance.

REFERENCES

- Brusa, A., Bresciani, A.** 1995. Using a multipurpose tile body. *Am. Ceram. Soc. Bull.* 74(9): 59-63.
- Carty, W. M., Senapati, U.** 1998. Porcelain; raw materials, processing, phase evolution and mechanical behavior. *Journal of American Ceramic Society*, 81(1): 3 – 20.
- Dana, K., Das, S.K.,** 2002. Some studies on ceramic body compositions for wall and floor tiles. *Trans. Indian Ceram. Soc.* 61(2): 83-86.
- Das, S. K., Dana, K., Singh and V. Sarkar, R.** 2005. *Shrinkage and Strength behaviour of quartzitic and kaolinitic clays in wall tile composition*, *Applied Clay Science*, 29: 137 - 143.

- Das, S.K., Kumar, S., Singh, K.K., Ramachandra Rao, P.** 1996. Utilization of fly ash in making ceramic wall and floor tile. Proc. RVBIS, IIM. Ghatshila, India. Pp.7-10
- Norton, F.H.** 1973. Introduction to Ceramics Technology, Edgard Blucher, Brazil.
- Brown, G.** 1984. Crystal structures of clay minerals and related phyllosilicates in clay mineral: Their structure, behaviour and use, edited by L. Fowden, R.M. Barrer, and P.B. Tinker. The Royal Society, London, Pp.1-20.
- Hinkley, D.N.** 1962. Variability crystallinity values among the kaolin deposits of the costal plain of Georgia and South Carolina. Proceedings of the 11th National Conference on Clays and clay minerals (Ottawa, Ontario, Canada), pp.229-235.
- Kingery, W.D.** 1976. Introduction to Ceramics, Wiley, New York, pp. 78-79, pp. 532-540.
- Kumar, S., Singh, K.K., Ramachandra Rao, P.** 2001. Effect of fly ash additions on the mechanical and other properties of porcelainised stoneware tiles. *J. Mater. Sci.*, 36: 5917-5922.
- Moore, D.M., Reynold, R.C** 1997. X-ray Diffraction and the Identification and Analysis of Clay Minerals, 2nd ed., Oxford University Press, New York, pp.227-260.
- Murray, H.H., Keller, W.D.**, 1993. Kaolin genesis and utilization. In: Murray, H.H., Bundy, W., Harvey, C. Clay Minerals Society, Special publication, 1: 1-24.
- Nnuka, E.E., Enejo, C.** 2001. Characterization Nahuta Clay for Industrial and Commercial Applications. *Nigerian Journal of Engineering and Materials*, 2(3): 9 -12.
- Olgun, A., Erdogan, Y., Ayhan Y., Zeybek, B.** 2005. Development of ceramic tiles from coal fly ash and tincal ore waste, *Ceramics International*, 31: 153 - 158.
- Shah, H.M., Maity, K.N.** 2001. Development of glazed tile through optimal utilization of fly ash. *Trans. Indiana Ceram. Soc.*, 60(3): 145-149.
- Swanpa, Kr Das., Kausik Dana., Nar Singh., Ritwik Sarkar.** 2005 Shrinkage and strength behavior of quartzitic and kaolinitic clays in wall tile compositions. *Applied Clay Science*, 29: 137-143.
- Yatsenko, N.D., Zubekhin, A.P., Rakova, V.P.** 1998. Low shrinkage ceramic tiles. *Glass ceram.*, 55 (7-8): 255-257.

(Manuscript received:25th February, 2011; accepted: 25th May, 2011).