EVALUATION OF REFRACTORY PROPERTIES OF SELECTED CLAY SAMPLES FROM SOKOTO STATE, NORTH WEST NIGERIA

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Abstract

This study presents the results of characterization of the refractory properties of some selected clay deposits from Sokoto State in Nigeria. Characterization of the clays was carried out to determine its physical properties and chemical composition. The result of analysis shows that the clay samples were of high silicate content with Wamakko and Illela having a composition of 54.27 and 58.31 %. The clays were also found to have moderate alumina content of 23.57 and 27.56 % respectively and low ferrous oxide content (< 3 %). It also possesses very low contents of other metal oxides. The low iron content gives the clay rather whitish appearances suggesting its suitability for paint, chalk, and earthenware manufacturing. All the clay samples had refractoriness above 1500 °C establishing the potential for handling of ferrous metal and lining of furnace wall. The thermal shock resistances within the range of 23 and 26 cycle Wamakko and Illela clay sample further establish their suitability for use as fire clay refractory's. The clays samples investigated have quality characteristics for their potential application in the production of floor tiles, fire refractory brick, paint, chalk, and earthenware manufacturing.

Keywords: Refractories, Clay, Northwest, Fireclay, Characterization, Wamakko, Illela

Introduction

Refractories are mostly made up of inorganic compounds which are usually derived from clay. It is characterized by the ability to withstand physical, chemical and corrosive attack without deterioration at elevated temperatures (Musa et al., 2012). These materials are mostly used in metallurgical industries for the linings of internal furnaces wall, melting, holding and transportation of metal and slag. It is also widely used in varieties of industries such as glass manufacturing, cement, petroleum and ceramics industries (Waing et al., 2008).

In the last few years, the possibility and significant application of refractory materials in the industrial growth of Nigeria have been receiving serious attention (Abolarin et al., 2004). According to Amuda et al. (2005), there will be a high demand for refractory materials with the anticipated completion and subsequent commissioning of Ajaokuta Steel industry which have a production capacity of 1.3 million tonnes of liquid steel. Ndanusa et al. (2005) added that the industry will require approximately over sixty million naira for about 36,000 tonnes of refractory bricks for lining furnace wall only. The authors further stated that fired clay constitute over 80 % of the bricks that will be required for this purpose. It was also reported that between 1997 and 2002 about $850 million dollar was expended on the four Nigeria indigenous refineries during its turn around maintenance (TAM). Fluid Catalytic Cracking (FCC) is the most vital unit in the TAM process. This unit is lined with large number of different grades of refractory bricks (Borode et al., 2002). The Chemical, hardware, cement and glass industries are also in serious need of these refractory materials. The demand of these industries is far greater than 300,000 tonnes as at the year 2000 (Ndaliman, 2007). These refractory materials are sourced by importation (Borode et al., 2002). Meanwhile the primary raw materials for the production of refractory are magnesites, dolomites-chrome ores, spinel and clay (Waing et al.,2008).

In Nigeria abundant deposits of clay have been reported across major geological belt (Musa & Aliyu, 2011). But it is surprising that the growth of refractory industries in the country is still at its infancy stage. The present economic meltdown in the nation and uncertainty facing the petrochemical, metallurgical and other allied industries has necessitated the need to source for local raw materials to nurture the growth of these industries. This will lead to the production of high quality commercial refractory which will eventually substitute for importation and help to save the much needed foreign exchange (Musa et al., 2012).

This has stimulated the recent efforts to explore and improve the properties of Nigeria clay for relevant industrial application (Musa & Aliyu, 2011). Several researches have been documented on the properties and application of Nigeria clay for relevant industrial application. However most of the clay studied reported are limited to clays deposit from the following geological belt; East (Nnuka & Agbo,2000; Chukwudi, 2008; Ameh & Obasi,2009; Osaremwinda & Abel ,2014; Odewale et al., 2015), southwest (Amuda et al.,2005; **Omotoyinbo & Oluwole**., 2008; Oloruntola et al., 2010), North central (Isah, 2010; Musa et al., 2012, Manukaji, 2013), North East (Abolarin et al., 2004; Abdullahi & Samaila, 2007; Aremu et al., 201), South South (Osaremwinda & Abel, 2014).

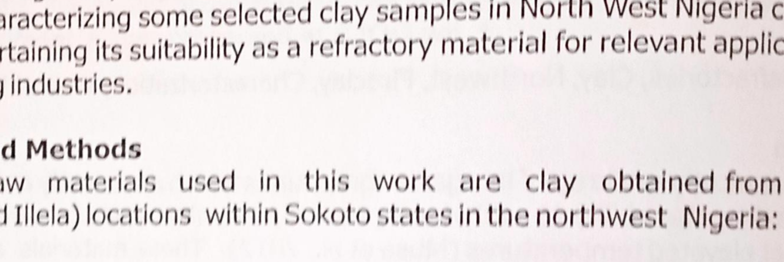
It is evident from literature that no attempt has been made to characterize Clay Deposits in the North Western Nigeria specifically Sokoto state to the best of the authors knowledge. This work is aimed at characterizing some selected clay samples in North West Nigeria clay deposits with a view of ascertaining its suitability as a refractory material for relevant application in Nigerian manufacturing industries.

Materials and Methods

The basic raw materials used in this work are clay obtained from two different (Wamakko and Illela) locations within Sokoto states in the northwest Nigeria: Sokoto state.

Sample Preparation

The clays from the various deposits were crushed and grounded to fine grains. The samples were moulded with adequate amount of water and then moulded into test laboratory sizes of rods of length 85 mm by 45 mm diameter after which they were air dried for 24 hours and oven dried at a temperature of 110 °C for 12 hours. The rods made from this samples were then fired in an electric muffle furnace at 1000 °C for 8 hours. The firing was done at a slow rate to avoid the cracking of the rods. The rods were then cooled at the rate of 15 °C per minute to room temperature making the rods ready for testing (Plate II).



Characterization

Atomic Absorption Spectrophotometer (AAS)

The chemical composition of the raw clay samples in weight % of (SiO_2, Al_2O_3, CaO, MgO, Na_2O and Fe_2O_3) was determined using AAS (model 210 VGP) at the Chemical Engineering Laboratory of Federal Polytechnic, Bida, Niger state.,

Determination of Loss on Ignition (LOI)

The water content of the clay was determined by measuring the weight loss of mass of a sample after firing in the furnace at 1000 ^{0}C for 1 hour thirty minutes. Loss on ignition was calculated using this relation:

$$LOI = \frac{W_i - W_f}{W_i} \times 100\ \% \qquad (3.1)$$

Where W_i and W_f are initial and final weight of the clay respectively.

Determination of B ulk Density

Test sample of 85 mm x 45 mm were prepared for all the clay under investigation. Each of the samples was air dried at a temperature of 110 ^{0}C in oven for 24 hours. The samples were cooled after drying and weighed accurately. The samples were th en heated for another 30 minutes to ensure that all trapped air were appreciably evacuated and the second weight reading of sample (w) are taken after cooling and soaking the samples in water. The samples were then suspended in water and the susp ended weight (S_s) taken. The bulk density was calculated

$$B_D = \frac{D_g}{W_s - S_s} \qquad (3.2)$$

Where D - dried weight, W_s soaked weight and S_s suspended weight all in gram (g)

Determination of Thermal Shock Resistance

The test samples were inserted into a furnace that was kept at 1100 ^{0}C. This temperature was maintained for about 10 minutes. The samples were removed one after the other with tongs and then cooled for ten minutes. The samples were returned to the furnace again for a further 10 minutes. The process was continued until there is conspicuous crack on the clay rods as shown in plate III. The number of cycles withstand before fracture were recorded for each sample.

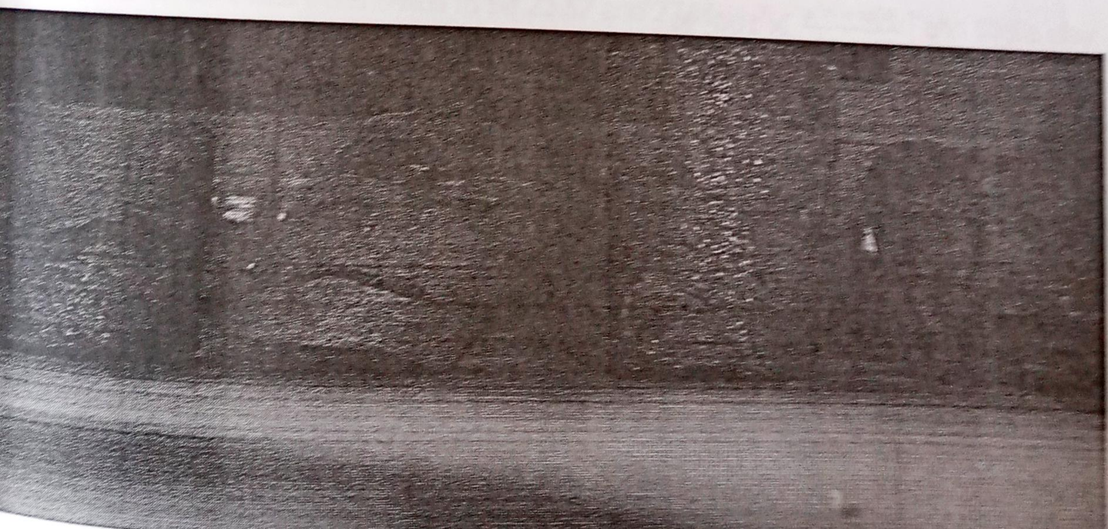


Plate III: Cracked Clay Rods

Determination of Cold Crushing Strength

The samples were placed on a compressive testing machine. Load was then applied axially by turning t he hand wheel at a uniform rate until failure occurs. Using a well calibrated scale, the maximum load was taken as the crushing load. The cold crushing strength was then determined using the formula;

$$\text{Cold Crushing Strength (CCS)} = \frac{Maximum}{Cross\ sectional\ area} \quad (3.3)$$

Determination of Refractoriness

The refractoriness of the rods could not be determined experimentally because the rods were fired to limited temperatures between 900 – 1100 °C but was estimated using Shuen's formula (Odo et al., 2009).

$$K = \frac{360 + AL_2O_3 - Ro}{0.228} \quad (3.4)$$

Determination of Linear Shrinkage

Linear shrinkage was determined by measuring the dimensional changes on both the wet and fired rods using a Vernier calliper. The linear dried dimensions were measured after air drying overnight. The linear shrinkage was determined using the relationship shown in equation

$$LS = \frac{\Delta L}{L} \times 100\ \% \quad (3.5)$$

Where ΔL is change in length of the rod and L is the original length of the rod.

Determination of Apparent Porosity

The 85 mm rod was dried in an oven at 105 °C for 1 hour to dry the samples. The weight of the dried weight in air (Wa) was taken, before sample was put into boiling water for 15 minutes to remove trapped air bubbles. It was then allowed to cool after which the weight of the soaked sample (Ws) was taken. The sample was removed from water and cleaned up and weighed again in air and th e saturated weight (Wss) determined. Apparent porosity was evaluated using the formula:

$$AP\ (\%) = \frac{volume\ of\ water\ absorbed\ after\ boiling}{bulk\ vuolume}$$

$$= \frac{Wss - Wa}{Wss - Ws} \times 100\ \% \quad (3.6)$$

Results and Discussions

Table 1 shows the results of chemical composition respectively for the clay samples:

Table 1.0: Chemical Composition of Clay Samples

Clay	Al_2O_3	MgO	SiO_2	CaO	Fe_2O_3	Na_2O
Wamakko	23.57	0.006	54.27	0.010	0.015	0.168
Ilela	27.26	0.059	58.31	0.013	2.730	0.720
[1]Onibode, Ogun State	37.40	-	50.80	-	5.80	-
[2]Yamarkumi, Borno State	19.68	0.98	41.80	1.51	8.89	-
[3]Oghara, Delta State	30.46	0.50	50.92	0.19	2.70	0.70
[4]Tammah, Plateau State	27.48	0.11	56.43	0.03	-	
[5]Ekebedi, Abia State	28.03	-	58.53	0.2	1.41	0.03
*Fire Clay	25 – 45	< 2	55-75	< 1	0.5 – 2.0	-
**Refractory brick	25 - 44		51.70	0.10 - 20	0.5 – 2.4	-
#High melting clay	16 -29	0.5 – 2.6	53 - 73	0.5 – 2.6	1 – 9	-

[1]Apeh et al.,(2011) [2] Abdullahi and Samaila, (2007) [3,5]Osaremwinda and Abel (2014) [4]Musa et al. (2012) [5]Odewale et al., 2015 *Grimshaw (1971) **(Grimshaw, 1971; Abubakar et al., 2014) *# Nnuka and Agbo (2000)

The result for chemical analysis in Table 1.0 shows a high silica content ranging of 54.27 and 58.31 % for both clay samples. The high silica content shows approximate consistency with the standard for fire-clay and quite with within the range for high melting clays as shown in Table 1. The result was impressively higher than 50.80, 41.80 and 50.92 % reported for Onibode, Yamarkumi and Oghara clay in Ogun, Borno and Delta state of Nigeria. The result show that all the clay samples exist as quartz. The result suggests the suitability of these clays as a source of silica for the production of floor tiles.

The alumina content was determined to be 23.57 and 27.56 % for Wamakko and Ilella clay samples. The results are appreciably with the standard specification of 25-45, 25 -44 and 16 - 29 % for fireclay, refractory bricks and high melting clays (Grimshaw, 1971; Abubakar et al., 2014). These values are also relatively consistent with the report of literature of clay sample analyzed in other region of the country as clearly depicted in Table 1. According to Abolarin et al. (2004) aluminium and silicon oxides are the important materials used in the manufacture of fire clay refractories. The sample analyzed in study has the requisite potential to be used for such purpose.

It is important to state that the percentages of basic oxides (CaO, MgO and Na_2O) were low. This explains plasticity characteristics of the clay. It was observed in this study that the compositions of all the major oxides do not sum up to 100 %. This discrepancy may be due to the presence of impurities in the clay. It can as well be attributed due to the traces of other ions (trace elements) such as copper, sulphur and zinc in the samples. Another possible reason for this deficiency could be due to the presence of materials too infinitesimal for detection, such materials are expected to be removed during pre treatment stage.

Table 2.0: Physical Characteristics of Clay Samples

Clay Sample	Linear Shrinkage (%)	Bulk Density (g/cm³)	Apparent Porosity (%)	Thermal Shock Resistance (cycles)	Refractories (°C)	Cold Crushing Strength (MPa)	Loss on Ignition (%)
Wamakko	3.52	2.16	12.63	23	1669.07	12.56	15.07
Illela	13.53	3.14	23.26	26	1697.94	8.28	6.00
[1]Onibode, Ogun State	2.0	2.23	28.18	+30	1700	16	1.67
[2]Yamarkumi, Borno State	1.0	2.06	22.26	5	1400	15.43	14.05
[3]Ogbara, Delta State	5.0	1.74	31.44	-	1500	9.88	12.18
[4]Tammah, Plateau State	7.41	3.10	30.69	22	1696	11.21	4.75
*Fire Clay	7-10	1.7-2.1	20-30	20-30	1500 - 1700	15	<15
**Siliceus fire clay	7-10	2.0	23.7	1	1500- 1600	15	
##Refractory brick		2.30	10 - 30	20 - 30	1430 - 1717	15	8 -18

*Grimshaw (1971) ** Omowumi (2000) ## (Grimshaw, 1971; Abubakar et al., 2014)
[1]Apeh et al.,(2011) [2] Abdullahi and Samaila, (2007) [3,5]Osarenwinda and Abel (2014) [4]Musa et al. (2012) [5]Odewale et al., 2015

The bulk density is an important property in the transportation or handling of a refractory material. Bulk density is affected by a number of factors such as particle size, treatment process and the composition of the clay sample (Omotoyinbo & Oluwole, 2008). The standard bulk density for general refractory falls within the range of 1.7 - 2.1 g/cm³ (Grimshaw, 1971) and 2.2 - 2.3 g/cm³ for high alumina refractories (Amuda et al. 2005). Wamakko and and Illela clay sample had a bulk densities of 2.16 and 3.14 g/cm³ respectively. Wamakko clay was consistent with this specification but Illela clay sample do not fall within these standard range as reported in Table 2. The high bulk density observed in these samples shows close proximity to 3.10 g/cm³ reported for Tammah clay. This high bulk density might be attributed to the presence of impurities.

Loss on ignition (L.O.I) is a measure of amount of combustible volatile matter present in a clay (Abdullahi & Samaila, 2007).This value is a probable reflection of the finess and structure of the refractory. The L.O.I for both clay samples was determined to be 6.0 and 15.07 % for Illela and Wamakko clay sample. These values fall within the standard value of less than 15 % except for Wamakko that show slight deviation from the set specificification. However the L.O.I for this clay falls within the limit of 8-18 % for standard refractory brick (Grimshaw, 1971; Abubakar et al., 2014). The result obtained in this study was higher than other reported work in some other part of the country as shown in Table 2.

Thermal shock resistance is the ability of sample to withstand heating and cooling several times before collapsing or the appearance of a visible crack (Folaranmi, 2009). Thermal shock of a refractory material must be high to withstand a wide variation in the operating temperature encountered in a furnace (Atanda et al., 2012). Wamakko and Illela clay samples were found to have very good thermal shock resistance of 23 and 26 cycles respectively. This result shows appreciable consistency with 22 cycles reported for Tammah Clay (Musa et al., 2012) and higher than 5 reported for Yarmarkumi clay (Abdullahi & Sumaila, 2007). The results also fall within the 20 -30 cycles specified for fireclay and refractory brick (Grimshaw, 1971; Abubakar et al., 2014). The result revealed that both clay samples can withstand high temperatures before collapsing or losing it properties and therefore can be used for furnace linings.

The cold crushing strength is a measure of a material to withstand abrasion and load without breaking or disintegration (Muhammadu, 2013). The cold crushing strength were found to be 12.56 and 8.28 MPa for Wamakko and Illela, clay samples respectively. These values fall short of the standard values of 15 MPa for fireclay, refractory brick (Grimshaw, 1971).The values is also less than 15.93 and 16 MPa for Onibode and Yarmakumi clay samples reported in literature (Abdullahi & Sumaila, 2007; Apeh et al., 2011). Wamakko clay sample however exhibit a higher value of CCS than 9.88 and 11.21 MPa reported for Oghara and Tammah clay. This shows that the Wamakko clay samples can be used to transport slag.

Apparent porosity is the ability of the clay to be impermeable to gases and liquids. Porosity is used for measuring the degree of vitrification and it is directly related to shrinkage volume (Oloruntola et al., 2010). The apparent porosity of 23.26 and 12.61 % were obtained for Illela and Wamakko clay samples respectively. The values for Illela were within the standard range of 20-30 % recommended for fireclay while Wamakko porosity of 12.61 % exhibits a shortfall of the stipulated standard. According to Aremu et al., 2013 these effect associated thermal losses at elevated temperature. The author added that the thermal conductivity of a refractory materials decreases as its porosity increases as the pores tends to act as non conducting media. Low porosity of clay makes it's a good conductor of heat which makes it application in construction of oven smelting furnace (Isah, 2010). The results obtained for Wamakko and Mafara clay samples suggest its suitability for such purpose.

The linear shrinkage is a property of clay material which enables it's to resist structural changes and disintegration when subjected to heat (Folaranmi, 2009). Wamakko and Illela clay samples were found to have linear shrinkage of 3.52 and 8.24 % respectively. Illela clay samples conforms to the standard value of 7 to 10 % for fireclay. Wamakko donot conform to this standard but had higher value than 1 and 2 % reported by Apeh et al. (2011) and Abdullahi and Samaila, (2007) for Onibode and Yarmarkumi clay samples

Refractoriness is the maximum temperature a material can withstand after which it will fail (Folaranmi, 2009). The refractoriness of the rods could not be determined experimentally because the rods were fired to limited temperatures between 900 – 1100 °C. The values were estimated using Shuen's formula. The refractoriness of the clay samples was found to have softening temperature greater than 1500 °C which is in agreement with the relevant standard specification and literature.

Conclusion

The investigation carried out on the clay samples from Sokoto showed that the properties of the clays samples shows appreciable consistency with standard for fireclay and industrial refractory bricks except for some slight deviation. Results of chemical analysis clearly revealed that the clays are member of the alumina- silicate family and found to be suitable for use as fireclay refractories. The refractorines the, clay samples were well above 1500 °C establishing their potential usage melting metals and non-metals with melting points below its refractoriness. The clay rods have very good crushing strength and satisfactory apparent porosity. The physico-chemical test revealed that the entire clay samples can be used for refractory purposes. Geological survey should be carried out to know the quantity of the clay deposit. Further research should be carried out on blending and the use of local additives and binders such as asbestos, graphite and rice husk to improve the physical and chemical properties of Wamakko clay samples with low qualities.

References

Abdullahi, M. Y. & Samaila, U (2007). *Continental Journal Engineering Sciences*, 2, 30 – 35.

Abubakar, I., Birnin-Yauri, U. I., Faruq, U. Z., Noma, S. S. & Sharif, S. (2014).Characterization of Dabagi clay for its ceramic. *Potential African Journal of Environmental Science and Technology*, 8(8), 645-649.

Abolarin, M. S., Olugboji, O. A. & Ugwuoke, I. C., (2004). *Experimental investigation on local refractory materials for furnace construction.* 5th Annual Engineering Conference Proceedings, Minna, Nigeria, June 2004, Federal University of Technology, Minna.

Amuda, M. O., Lawal, G. I. & Majolagbe, F. O. (2005). Characterisation and evaluation of some Ccay deposit in South West Nigeria. *NSE Technical Transaction*, 40(4), 13 – 22.

Ameh, E. M. & Obasi, N. L. (2009). Effect of rice husk on insulating bricks producedfrom Nafuta and Nsu clays. *Global Journal of Engineering and Technology*, 2 (4), 661- 668.

Apeh, F. I., Esezobor, D. E. & Lawal, G. I. (2011). Characterization of Onibode and Owode-Ketu clays for use as refractory materials in foundry industry. *Journal of Engineering Research*, 16(3), 89-77.

Aremu, D. A., Aremu, J. O., Ibrahim, U. H. (2013). Analysis of Mubi clay deposit as a furnace lining. *International Journal of Scientific & Technology Research*, 2(12), 182-187.

Atanda, P., Adeniji, O. I. & Oluwole, O. (2012). Development of heat treatment refractory bricks using local Nigerian clays. *International Journal of Materials and Chemistry*, 2(5), 185-191. DOI: 10.5923/j.ijmc.20120205.01

Borode, J. O., Oluwade, G. O.& Olasupo, O. A. (2002). *Development of ramming mass.Proceedings of the Nigeria Material Congress* - NIMACON Materials Society of Nigeria, 93-98.

Chukwudi, B. C. (2008). Characterisation and evaluation of the refractory properties of Nsu clay deposit in Imo State, Nigeria. *The Pacific Journal Science Technology*, 9(2), 487 – 494.

Grimshaw, R. W. (1971). *The chemistry and physics of clay and allied ceramic materials.* 4th Edition Revised. New York: Willey Inter Science Pp 16-89.

Isah, A. I. (2010). *Determination of the chemical and physical properties of some selected Benue clay deposits.* Unpublished B. Eng. Project, Chemical Engineering Department, Federal University Technology, Minna, Nigeria. 24- 28.

Joshua, F. (2009). Effect of sawdust additive on the properties of clay. *AU J.T*, 13(1), 53-56.

Muhammadu, M. M. (2013). An investigation on refractory clays properties for application in metallurgy industries in Nigeria. *International Journal of Advanced Research*, 3(6), 200-207.

Manukaji, J. U. (2013). Industrial potential of some clay deposits in Kogi State North Central Nigeria. *American Journal of Engineering Research (AJER)*, 2(4), 33-38. Available at www.ajer.us

Musa, U. & Aliyu, M. A. (2011). *Evaluation of refractory properties of Tatiko and Beji clay.* Proceedings of 2nd Bienial Engineering Conference, Federal University of Technology Minna, Niger State, Nigeria. 16th-18th November, 2011, 106-109.

Musa, U., Aliyu, M. A., Sadiq, M. M. & Mohammed, I. A. (2012). Investigation into refractory properties of some selected clays in North Central, Nigeria. *Academic Research International*, 3(1),393-398.

Ndaliman, M. B. (2007). Refractory properties of termite hills under varied proportion of additives. *AU-JT*, 10 (3), 159-162

Ndanusa, I. A., Thomas, D. G., & Usaini, M. N. (2005). *Suitability of blended lokongoma feldspar and Kankara clay for refractory bricks production. Fifth Annual Engineering Conference Proceedings*, Federal University of Technology Minna, 73-81.

Nnuka, E. E. & Agbo, U. J. E. (2000). Evaluation of the refractory characteristics of Otukpo clay deposits. *N.S.E Technical Transaction*, 35(1), 34-41.

Odewale, I., Olajide, O., Michael, B. & Tse, D. T. (2015). Production and characterization of Aluminosilicate refractory brick using Unwana Beach silica sand, Ekebedi and Unwana clays. *British Journal of Applied Science & Technology*, 5(5),: XX-XX, 2015, Article no.BJAST.2015.044 Available at www.sciencedomain.org

Odo, J. U., Nwajagu, C. O. & Nnuka, E. E. (2009). Characterization of Ezebunagu clay deposit and its potential for industrial applications. *Journal Metallurgy and Materials Engineering*, 4(2), 31-37.

Oloruntola, M. O., Bayewu, O. O., Mosuro, G. & Dauda, M. (2010). Evaluation of clay deposits in falafonmu and its environments, Ijebu-Ode, SW Nigeria, *Journal of Applied Sciences Research*, 6(12), 2026-2034.

Omotoyinbo, J. A. & Oluwole, O. O. (2008). Working properties of some selected refractory clay deposits in South Western Nigeria, *Journal of Minerals & Materials Characterization & Engineering*, 7(3), 233-245.

Omowumi, O. J. (2000). Characterization of some Nigeria clays as refractory materials for furnace lining. *Nigerian Journal of Engineering Management*, 2(3), 1-4.

Osarenmwinda, J. O., Abel, C. P. (2014). Performance evaluation of refractory bricks produced from locally sourced clay materials. *J. Appl. Sci. Environ. Manage*, 18(2), 151-157, Available Online at www.ajol.info and www.bioline.org.br/ja

Waing, W. K., Wut, H. A. & Thi, K. L. (2008). Study on the production of chromites refractory bricks from local chromites ore. *World Academy of Science, Engineering and Technology*, 46, 569 – 574.