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Performance Evaluation of Insulating Firebricks Produced from Hydrometallurgically Purified Termite Hill Clay Reinforced with Alumina

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ABSTRACT : *The performance of insulating firebricks produced from hydrometallurgically purified termite hill clay admixed with varying percentages of alumina cement has been qualitatively evaluated. A large quantity of termite hill clay was mined from a location on the campus of The Federal University of Technology, Akure (FUTA), Nigeria. The bulk of clay was washed in water, the deleterious shafts decanted, the slurry dried in sun for three days and later in the oven at 90 °C for eight hours. The dried clay was then crushed and ground to a fine size of 100 µm, being the average particle size upon the sieve size analysis. Sieved clay was purified hydrometallurgically at a predetermined condition; 1.6 mol/dm*^{3} *of oxalic acid at 90 °C for 150 min. and 200 rev/min agitation. Raw and purified clays were characterized using X-ray diffraction, Scanning Electron Microscopy and Transmission Electron Microscopy. Purified clay samples containing 5 – 20 % alumina were again fired at varying temperatures of 900 °C, 1100 °C, 1300 °C and 1500 °C and tested for some important refractory properties such as permanent linear change, modulus of rupture and permeability. Sample (purified clay + 10 % alumina fired at 1500 °C) that exhibited the best combination of these properties was examined under scanning electron microscope to see the effect of heat and analyzed chemically using the X-ray fluorescence machine to know the precise compositions.*

Keywords: *Insulating firebricks, Hydrometallurgy, Termite hill clay, Purification and Predetermined condition.*

I. INTRODUCTION

The insulating firebrick is a class of bricks which consists of adequately porous kaolin or fireclay. They are low in thermal conductivity, lightweight and sufficiently resistant to temperature which enables them to be used successfully on the hot side of the furnace wall. This therefore permits the use of thin walls of low thermal conductivity and low heat content. The low heat content is particularly valuable in saving fuel, time on heating up and allows rapid changes in temperature to be made, and also permits rapid cooling [1]. Even though substantial amount of research has been carried out in the area of production and development of good refractory materials for more than two centuries, the outcome of which has resulted in the varieties of refractory materials available in the world market today [2], most developing nations that are consumers of refractory materials still have to spend their hard-earned foreign reserves on the importation of these materials to meet their needs. In the light of this situation, there has been a continuous upsurge of interest in the imperative of looking inward, to develop good refractories using locally sourced materials. Consequently, a number of researches have been carried out on the thermophysical and thermochemical behaviour of some Nigerian refractory raw materials [3]. However, two major factors are still 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 [2].

Despite the observation, that the behaviour of termite hill clay when subjected to refractory tests is in good comparison with the firebricks used for refractory applications [4], further improvement may still be obtained when an additive such as alumina cement is added in appropriate proportions or other forms of rectification are made. This notion has therefore informed the need for this research effort.

II. MATERIALS AND METHODS

The materials and equipment used were raw termite hill clay, oxalic acid (99.8% purity), alumina cement (Secar 71), atomic absorption spectrometer (AAS) machine (model Spectre AA 220 FS), X-ray fluorescence (XRF) machine (model ARL 8410), scanning electron microscope (SEM) model JEOL 840 and coupled with an EDS analyzer, X-ray diffraction (XRD) machine (model Philips PW 3710 with PW 1752 graphite monocromator), sieve size analyser (Microtrac FLEX 10.5.4), Labcon shaking incubator (models 3081U and 5082U), Carbolite furnace, Rawwley Sussex jaw crusher and grinder.

2.1 Preliminary Preparations for Analysis

A large quantity of termite hill clay was mined from FUTA campus in Nigeria. It was then washed in water in order to remove the deleterious particles by decantation. Water was then drained from the clay slurry using a plaster of Paris (P.O.P.) mould. The recovered clay was then dried in the sun for three days and again in the Carbolite furnace at 90 °C for 8 hours. The dried clay was finally jaw crushed and ground in a Rawwley Sussex grinder to 100 µm.

2.2 Sieve Analysis

Size analysis was carried out on the ground clay using Microtrac FLEX 10.5.4 Filter enabled analyzer. 20 g of the clay was fed into the machine, with a loading factor of 0.0173, transmission rate of 0.851 and allowed to run for 10 seconds. The average sieve size was found to be about 100 µm. The bulk of the clay was therefore sieved to 100 µm for subsequent analyses carried out on it, in order to ensure uniformity.

2.3 Clay Characterization

Analyses of the clay were carried out using XRD, TEM, SEM/EDS and XRF according to the standard procedure [5]. The results are presented in Figures 1, 2, 6 and Table 1 respectively.

2.4 Hydrometallurgical Processing of Termite Hill Clay

The clay was treated hydrometallurgically by putting a large quantity of clay and 500 % by volume of 1.6 mol/dm^3 of prepared oxalic acid solution in a flat-bottom glass container and kept inside the Labcon shaking incubator, models 3081U and 5082U for agitation at 200 rev/min at 90 °C for 150 min [6]. On the expiration of the set time, the container was removed and allowed to cool to room temperature. The filtrate was decanted and the residue washed several times with deionized water, until all the acid content was completely expelled. This was achieved by testing intermittently with a blue litmus paper, until no further change in colour was noticed. The clay residue was then dried in the Carbolite furnace at 90 °C, crushed, ground and sieved to 100µm for subsequent tests.

2.5 Preparation of Samples for Performance Evaluation

Measures (150g each) of the purified clay with varying quantities (5, 10, 15 and 20%) of high alumina cement were formed into cylindrical (50mm diameter x 50 mm high) specimens after mixing with about (10-15)% deionized water. The samples were dried in air for 24 hrs and later in the oven at 110 ºC for 48 hrs. Some selected samples, after drying at 110 ºC were again taken for firing in the furnace at 900 ºC, 1100 ºC, 1300 ºC and 1500 ºC. The fired samples were subsequently tested in accordance to the American Standard for Testing and Materials [7] for the following properties:

2.5.1 Permanent Linear Change (PLC)

Permanent linear change tests were carried out in accordance with the [7] standard. The heights of the firebrick test samples were measured with vernier callipers before firing at various temperatures. Three different linear measurements were taken in each case and the average value calculated. The test samples were then fired at temperatures of 900 °C, 1100 °C, 1300 °C and 1500 °C for 2 hours at the rate of 4 °C per min, after which they were slowly cooled to the room temperature. The heights of the fired test specimens were again measured in order to determine the changes in heights. The results are as presented in Figure 3.

2.5.2 Modulus of Rupture (MOR)

According to [7], modulus of rupture is defined as the maximum stress a rectangular test piece (150 mm x 25 mm x 25 mm) can withstand in a 3-point bending test until it breaks. In doing this, test specimens were prepared, dried at 110 °C, allowed to cool to the room temperature and fired to 1500 °C. Some of the samples were tested after cooling from 1500 °C to the room temperature (30 °C) and the others were tested while the samples were right inside the MOR furnace at 1500°C. In each case, three samples were tested and the average values taken. The results are as presented in Figure 4.

2.5.3 Permeability

The permeability of the samples were measured to standard [7], using test specimens which had been dried in an oven at 110 °C to a constant weight within 0.1g accuracy and cooled to room temperature. The results are presented in Figure 5.

III. RESULTS AND DISCUSSION

3.1 X-ray Diffraction Analysis of Raw Termite Hill Clay

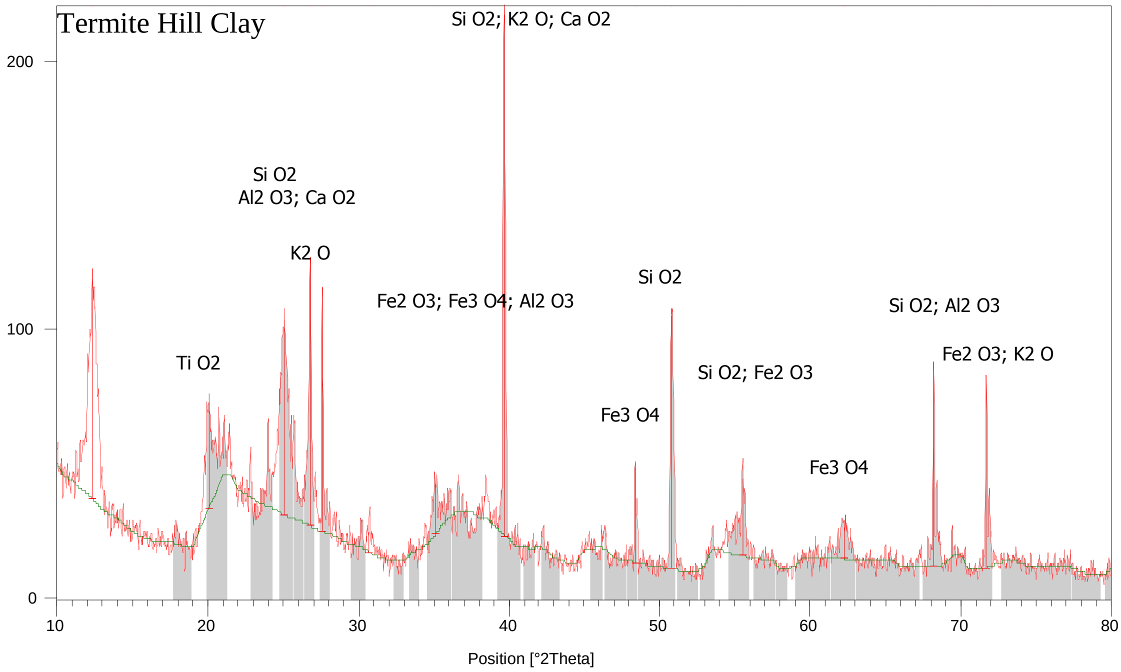
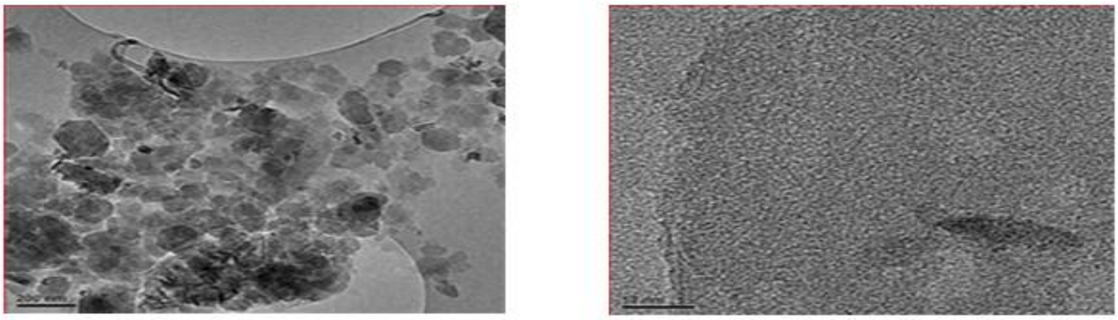


Figure 1: X-ray Diffraction Pattern for Termite Hill Clay

The pattern shows sharp high peaks for SiO_2, Al_2O_3, Fe_3O_4 and sharp but shorter peaks for K_2O, MgO, MnO, Na_2O and CaO. Some other oxides were also present but in very negligible proportions. The heights of the picks being directly proportional to the relative abundance of the various constituents of the clay.

3.2 Transmission Electron Micrograph (TEM)


(A) Raw clay before leaching

(B) Clay after leaching

Figure 2: Transmission Electron Micrographs of Raw and Leached Termite Hill Clay

Plates A and B in Figure 2 are the transmission electron micrographs of the raw and leached termite hill clays respectively. They both show the distribution of the minerals present in them. The minerals occurred as agglomerates in the raw clays but after undergoing the purification process, they became finely and uniformly distributed in the bulk. The uniform dispersion of the minerals in the processed clays enabled the oxalic acid to make direct contacts with virtually all the grains in the clays and hence able to remove the iron present in them [5].

3.3 Permanent Linear Change (PLC)

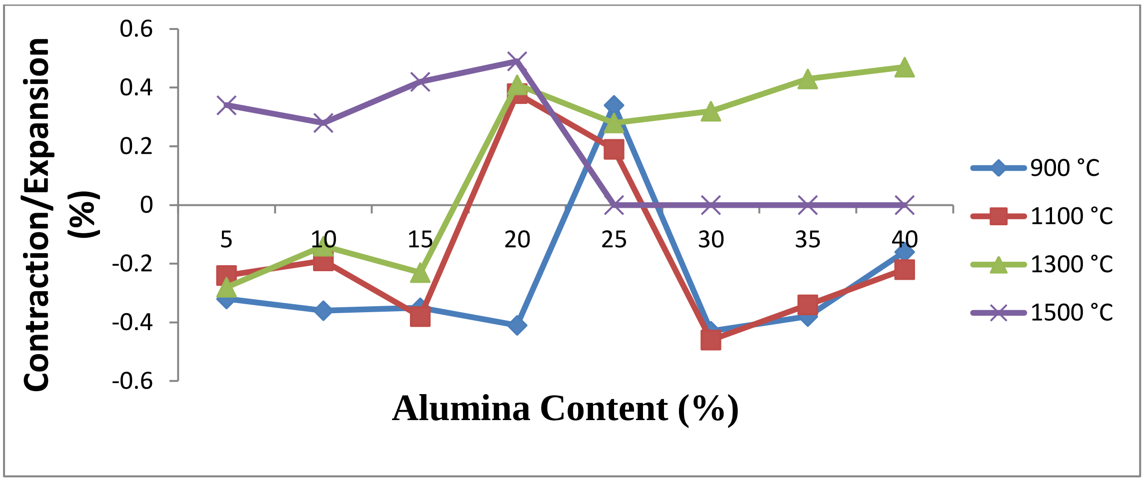


Figure 3: Permanent Linear Changes at Different Firing Temperatures

The results of the variation of permanent linear changes with alumina contents and firing temperatures are shown in Figure 3. It was observed that firebricks produced from the processed termite hill clay underwent linear contractions for firing temperatures of 900 ºC, 1100 ºC and 1300 ºC for low additions of alumina. For alumina contents between 20 and 30% the firebricks suffered permanent linear expansion after which the bricks suffered permanent linear contraction again. This is indicative of variations in the clay-alumina reactions at different alumina contents. Firing at 1500 ºC caused the firebricks to undergo a high permanent linear expansion and hence suffered dimensional instability.

When fired at 1500 ºC, the bricks suffered permanent linear expansion for alumina additions up to 20 %, beyond which the bricks became dimensionally unstable and crumbled under its own weight. This accounted for the reason samples with 25 % alumina and above had zero contraction/expansion on the graph (Figure 3)

3.4 Modulus of Rupture (MOR)

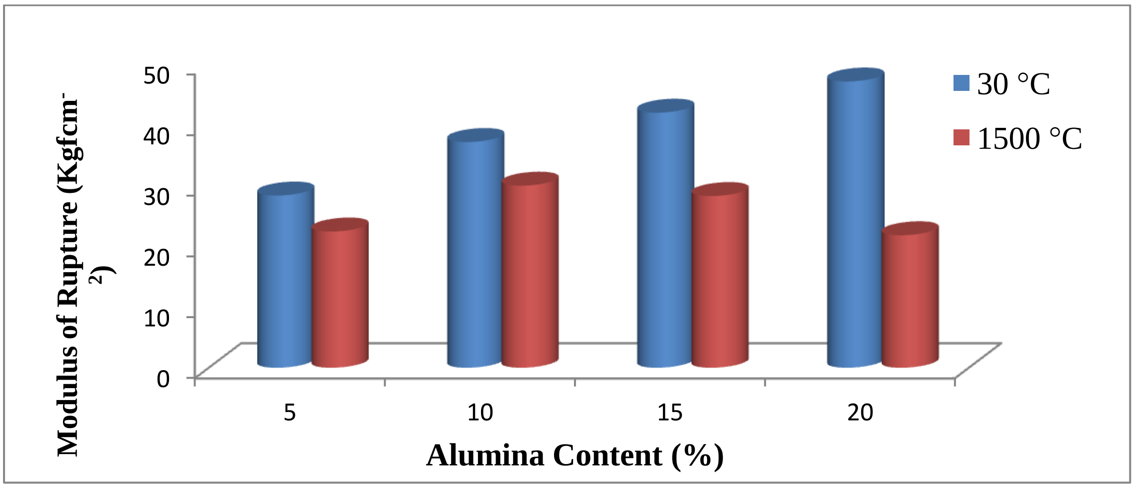


Figure 4: Modulus of Rupture at different alumina contents and temperatures

The modulus of rupture values at the room temperature (30 °C) and 1500 °C are shown in Figure 4. The values were very much lower when tested at 1500 °C than at 30°C. The MOR increased progressively with the quantity of alumina in the cold state while it only increased to the maximum at 10 % alumina and dropped again when tested at 1500 ºC. The trend was due to the fact that the liquid so formed when the samples were first heated and allowed to cool had solidified to form glass which further strengthened the samples. On the other hand, when the samples were rupture tested at the elevated temperature of 1500 °C, the samples were still in the molten state and thus the strength was comparatively lower than when it had solidified to form glass.

However, the MOR attained the maximum value of 29.97 Kgfcm^{-2} at 10 % alumina because the liquid phase formed when fired was adequately matched with the alumina present to form a solid mass and hence the relatively higher strength than at 15 % and 20 % alumina where the liquid phase was predominant.

3.5 Apparent Porosity

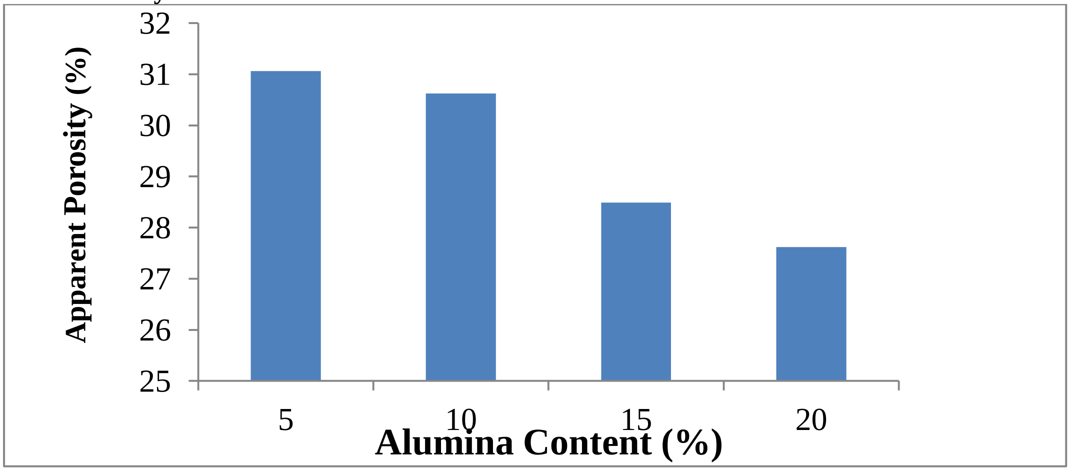


Figure 5: Apparent Porosities at different alumina contents

The permeability of the samples was measured as described in 2.5.4 above, for samples with 5 – 20 % alumina. The results are as presented in Figure 5. The permeability reduced with alumina contents because the pores were blocked as a result of the binding effect of alumina which reduces the interparticle distances between the grains. The maximum permeability obtained was 30.62 %, which was much lower than the 45 % required by standard [8] for insulating firebricks. It therefore becomes imperative for the introduction of a combustible material such as sawdust or rice husk which are expected to burn off at high temperature in order to further create pores for easy passage of the evolved gases.

3.6 Scanning Electron Microscopy (SEM)

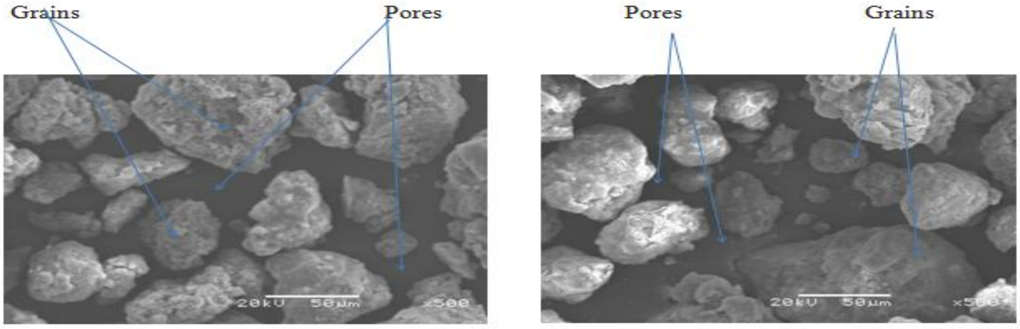


Plate D1 (5 % alumina) **Plate D2 (10 % alumina)**

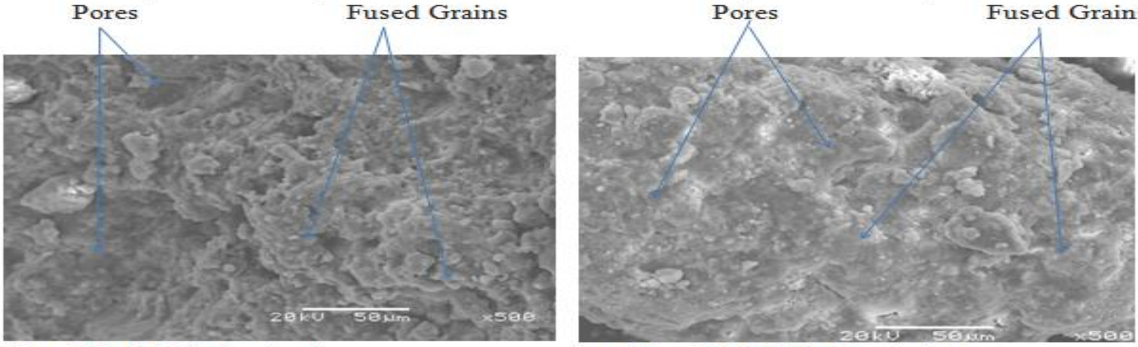


Plate D3 (15 % alumina) **Plate D4 (20 % alumina)**

Figure 6: SEM micrographs (X500) of refractory bricks fired at 1500 °C

The Scanning Electron Microscopy of samples containing 5% – 20 % alumina was done in order to see the effect of high temperature firing on the samples. The results are as presented in Figure 6, Plates (D1 – D4). Plates D1 and D2 show the presence of large pores between individual clay particles loosely bonded together. Plates D3 and D4, on the other hand, show an evidence of melting and fusing together of the clay particles. This is even more pronounced in Plate D4. The fusing together of the clay particles in fired bricks containing 15% and 20% alumina resulted in pore closure which will ultimately lead to reduction in permeability. The firebrick therefore had comparatively high permeability when the alumina content was ≤ 10 %.
These observations account for the low permeability which the samples exhibited when the alumina contents were above 10 % and the moderate porosity when the alumina content was between 5 and 10 %. These observations further confirmed that the samples maintain phase stability when fired at temperature as high as 1500 ºC provided the alumina content is not more than 10 %.

3.7 XRF Analysis of the Constituents of chosen Brick

The brick that combined the desired properties of a good insulating firebrick (processed clay + 10 % alumina) was finally analyzed chemically in order to know the exact constituents. The results are presented in Table 1 below.

Table 1: XRF analysis of the components of the insulating firebrick produced

Compound	Processed clay	Pure alumina (Secar 71)	Brick produced
SiO_2	65.18	0.36	50.23
Al_2O_3	24.56	68.78	34.36
CaO	6.27	30.3	13.42
Fe_2O_3	1.28	0.07	0.74
MgO	0.7	0.22	0.13
K_2O	0.09	0.03	0.62
Na_2O	0.84	0.24	0.30
TiO_2	0.68	0.02	0.36
Cr_2O_3	0.03	0.04	0.04
P_2O_5	0.02	0.00	0.00
ZrO_2	0.1	0.00	0.00
Total	100.75	100.06	100.2

The brick that combined the desired properties of a good insulating firebrick (processed termite hill clay + 10 % alumina) was finally analyzed chemically in order to know the exact constituents. The results are presented in Table 1. The Table shows the chemical analysis of the various components that make up the insulating firebrick. The silica content of the brick produced was 50.23 %, the alumina content was 34.36 %, the calcium oxide content was 13.42 % , the iron oxide content was 0.74 % (less than 1 %) while the sum of MgO, K_2O and Na_2O contents was 1.05 % (less than 2 %).

However, the constituents of a good insulating firebrick are expected to be about 50 % silica, 30 % alumina and 15 % calcium oxide [9,10]. The brick produced with the processed termite hill clay and alumina can therefore operate satisfactorily as an insulating firebrick.

IV. CONCLUSION

The characterization by XRD affirmed that termite hill clay is mainly constituted by silica and alumina in major quantities and oxides of iron, calcium, magnesium and other elements in minor quantities. Hydrometallurgical purification of termite hill clay improves the efficiency of insulating firebricks by reducing the iron oxide content of the clay which is responsible for lowering the refractoriness. The addition of alumina cement accounts for improving the binding strength of the brick and also aid in raising the refractoriness.

Termite hill clay is therefore a promising material for the production of insulating firebricks at a very reduced cost without compromising quality, provided the appropriate quantity of alumina cement is added.

REFERENCES

[1] E. A. Avallone and T. Baumeister III, “*Marks standard handbook for mechanical engineers”*, New York, Mc Graw-Hill International Edition, 1996.

[2] Omotoyinbo and Oluwole, **“**Working Properties of Some Selected Refractory Clay Deposits in South Western Nigeria”, Vol.7, No. 3, pp. 233-245, 2008.

[3] M. S. Abolarin et al., **“**Experimental investigation on Local Refractory Materials for Furnace Construction”.5th Annual Engineering Conference, Federal University of Technology, Minna, Niger State. pp. 82-85, 2004.

[4] M. B. Ndaliman, “Refractory Properties of Termite Hills under Varied Proportions of Additives”, Leonardo Electronic Journal of Practices and Technologies, Issue 9, July-December, pp. 161-166, 2006.

[5] D. O. Folorunso et al., “Characterization and Qualitative Analysis of Some Nigerian Clay Deposits for Refractory Applications”, IOSR Journal of Applied Chemistry, e-ISSN: 2278. Vol. 7, Issue 9, Ver. I, pp. 40-47, Sept. 2014.

[6] D. O. Folorunso et al., “Effect of alumina cement on the refractory properties of leached Ipetumodu clay”, Leonardo Electronic Journal of Practices and Technologies. Issue 20, pp. 25-38, 2012

[7] ASTM C 133-84, “Standard test method for Cold Crushing Strength and Modulus of Rupture of refractory brick and shapes”, Pennsylvania: American Society for Testing and Materials, 1984.

[8] ASTM C 201-86, “Standard test method for thermal conductivity of Refractories”. Pennsylvania: American Society for Testing and Materials, 1986.

[9] R. K. Rajput, “*Heat and mass transfer”*, Ram Nagar, New Delhi, S. Chand and Company Ltd, 2003.

[10] A. R. Chesti, “*Refractories: manufacture, properties, and applications”,* Delhi, Prentice-Hall of India Private Limited, 1986.