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Chemical characterization of Ijapo clay and its additives towards production of refractory bricks

Oladunni Oyelola ALABI[1✉], **Miracle ADEGUN**[2], **Temitope OLATUNJI**[3]

[1]Metallurgical and Materials Engineering Department, School of Engineering and Engineering Technology, Federal University of Technology, Akure, Ondo state, Nigeria; E- mail: oladunni69alabi@yahoo.com
[2]Metallurgical and Materials Engineering Department, School of Engineering and Engineering Technology, Federal University of Technology, Akure, Ondo state, Nigeria; E- mail: adigunmiracle@gmail.com
[3]Metallurgical and Materials Engineering Department, School of Engineering and Engineering Technology, Federal University of Technology, Akure, Ondo state, Nigeria; E- mail: olatunjitope@gmail.com

✉**Corresponding Author:**
Metallurgical and Materials Engineering Department, School of Engineering and Engineering Technology, Federal University of Technology, Akure, Ondo state, Nigeria
E- mail: *oladunni69alabi@yahoo.com
Phone: +234 803844739

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ABSTRACT
Clay is one of the important industrial minerals whose application depends on its structure and chemical composition. Chemical characterization of Ijapo clay bricks and its additives (charcoal and rice husk) was carried out via Energy Dispersive X-Ray Fluorescence Spectrometer (ED-XRF). The clay, rice husk and the charcoal were sourced from Akure metropolis, Ondo State. The effect of these additives on the physical and mechanical properties of Ijapo clay was investigated towards determining the suitability

and profitability of utilizing this clay for the production of refractory bricks. The clay was blended with the additives at varying weight percent ranging from 2-10 wt% to produce brick samples subjected to chemical and physical tests. ED-XRF analysis reveals that Ijapo clay assayed53.7% SiO_2 and 36.1% Al_2O_3 as the major constituents and with other trace in their compounds. The presence of high silica and alumina content affirms that Ijapo clay is an alumino-silicate in nature and also accounts for the characteristic hard texture of the clay. Chemical analysis of the additives reveals that charcoal contains CaO (63.0%); K_2O (11.2%), Fe_2O_3 (7.56%) and SiO_2 (5.9%); while the rice husk is of high silicate content assaying 83.9% SiO_2 other constituents are K_2O (3.72 %), CaO (2.84%) and Fe_2O_3 (5.83%). The refractoriness of Ijapo clay was found out to be 1350^{0}C. Moisture content, porosity, permeability and linear shrinkage were found to increase with increasing weight percent of charcoal and rice husk having values meeting the required standard for a refractory brick while the refractoriness and dry compressive strength decreases having values below required standard. On the premise of these findings, it was therefore recommended that 100 % Ijapo clay is unfit refractory for heavy duty applications at high temperature, however on account of economic viability; Ijapo claybrickofcomposition5 wt% charcoal and 10 wt% rice husk is best opt for as refractory for low duty work.

Keywords: Characterization; Ijapo clay; Additives; Refractory bricks; Production.

1. INTRODUCTION

Clay is a complex naturally occurring mixture of fine-grained minerals whose composition varies depending on the geographical location [1]. Clay is generally plastic with appropriate water contents and it becomes harden when dried or fired [2]. The presence of phyllosilicates and other materials have been attributed to impart the plasticity and hardness of clay when dried or fired [3]. It is an important industrial mineral whose application depends on its physical and chemical properties which are function of the mineral composition and structure [4]. Clay has been utilized for various purposes in industries and agriculture, and also for construction. In the metallurgical industry, clays are mostly used as refractories. Refractories are used in almost every industry in which heat is employed such as in metallurgical, chemical, cement, glass and petrochemicals industries [5].

The performance level derived from these refractory materials have been reported to be dependent on the type of reinforcements combined, the weight ratio of both reinforcing materials in the composite, the nature of the bonds between the additives and the refractory materials, processing route selected, and the metallurgical characteristics of the refractory materials [6]. Several materials have been used in the production of refractory bricks. Notwithstanding, the choice and suitability of a specific material depends mainly upon its availability, nature of the project, individual preference, durability, proximity and economic considerations [7]. Agricultural by-products and other wastes have been investigated as additives in production of refractory bricks and researchers are still beaming their search lights in this area [8]; this is because the introduction of additives can help improve the properties of clay. For instance, refractory bricks used as insulators must have more pores in order to reduce its thermal conductivity. Hence, combustible pore-forming materials can be used for such purpose.

Refractory bricks made from various additives combined with refractory clay have been scientifically reported. Rice husk is a low-cost agricultural by-products from which silicon carbide "whiskers" can be manufactured. The silicon carbide (SiC) whiskers are then used for reinforcement, increasing the strengths of the resulting products such as cutting fork, tiles, bricks and others. Charcoal on the other hand is a carbonaceous material obtained from the destructive distillation of wood or organic matter. This research aims at determining the constituents of the Ijapo clay and its additives, and studies how these constituents influence the mechanical properties and thus, consequently determine the suitability and profitability of utilizing Ijapo clay reinforced with charcoal and rice husk for the production of refractory bricks.

2. MATERIALS AND METHOD

The raw clay was obtained from its alluvial deposit at Ijapo Estate, Akure, Ondo State, Nigeria with dimensional area of about ten (10) hectares of land with an estimate reserve of about 600 million tonnes of clay. 50 kg of clay was obtained from this deposit. The clay was jaw crushed, screened using 1000 μm sieve and soaked in water for three days. This treatment was necessary to remove attached impurities such as dead organic matters and silts. The clay particles were allowed to settle by free sedimentation and the overlaying water was decanted. The settled almost impurity free clay was dried in open air for a week, after which it was crushed using Laboratory Denver Jaw Crusher (Model: BDA 15561), and ground using Bico Ball Milling Machine (Model: 69012) to 100 % passing 300 μm. Sample was then measured out of the prepared clay and mixed with proportion of additives (coal, rice husk) as in

Table 1 which contains the percentage composition of mix design to produce refractory samples used for other test at the course of this research.

Sample Preparation

Samples were prepared based on the mix ratios as shown in Table1 with clay as the balance to make 100 percent by weight.

Table 1: Percentage Composition Mix of Samples

Percentage composition mix (%)					
Charcoal →	1	2	3	4	5
Rice Husk ↓	2	2	2	2	2
	4	4	4	4	4
	6	6	6	6	6
	8	8	8	8	8
	10	10	10	10	10

The additives – charcoal and rice husk, were also ground to 300 µm and blended at varying weight percent ranging from 2-10 wt% with the clay and 2% water. The blend was poured into a cylindrical mould of dimension 3 cm × 3 cm x 3 cm and was pressed using hydraulic press at a pressure 100 KN for 10 minutes to enhance compaction, homogeneity and surface smoothness of the cylindrical samples produced. The shrinkage samples were moulded using a 10 cm × 5 cm x 2 cm wooden pattern as mould. The moulded bricks were air-dried for three days to enable natural setting to take place before firing. Bricks produced were then oven-dried for two (2) hours at 110^{0}C to expel more of the moisture left in the bricks and to avoid crack during firing.

Chemical Characterization of the Materials

Chemical analysis of Ijapo clay, rice husk and charcoal were determined using Mini Pal compact Energy Dispersive X-Ray Spectrometer (ED-XRFS). The X-ray diffractometer was taken using Cu Kα radiation at scan speed of 3 per min. The pulverized samples were rotated at precisely one-half of the angular speed of the receiving slit, so that a constant angle between the incident and reflected beams is maintained. The receiving slit was mounted in front of the counter on the counter tube arm, and behind it is usually fixed a scatter slit to ensure that the counter receives radiation only from the portion of the specimen illuminated by the primary beam. The intensity diffracted at the various angles was recorded automatically for the samples: Ijapo clay, Rice husk and Charcoal respectively.

Refractoriness Test on produced bricks

Refractoriness is the ability of a material to withstand the operation temperature without softening. The samples employed can be in cubic or cone form. The cone form was used in accordance with the pyro-metric cone equivalence approach to determine if the apex of the specimens will bend at 3'0 clock relative to the reference sample. The firing was carried out in a resistance heating furnace preset at heating rate of 7^{0}C per minute. The firing procedure used involved heating at various temperatures: 250^{o}C and held for 1 hour; 500^{0}C held for 1 hour; 750^{0}C held for 1 hour, 950^{0}C held for 1 hour, 1200^{0}C for 1 hour and 1500 ^{0}C for 2 hours. After firing the bricks were allowed to cool in the furnace, after which they were observe for refractoriness responds.

Percentage Moisture Content

The percentage moisture content of the samples was evaluated using equation 2.1.

$$\% \text{ Moisture content} = \frac{W2-W1}{W1} \times 100 \ldots\ldots\ldots\ldots\ldots\ldots (2.1)$$

The cylindrical samples were fired to 110^{0}C in the oven and soaked for one hour to homogenize the samples and to remove any moisture present therein. The samples were allowed to cool at room temperature, weighed and the weights were recorded as W_1. The samples were suspended in water for 10minutes, removed and the edges cleaned. The wet samples were weighed and the weights recorded as W_2.

Permeability and Porosity Test

Permeability and percentage porosity of the samples were calculated using equation 2.2 and 2.3 respectively as shown.

$$\text{Permeability} = \frac{W5-W4}{W4} \quad \text{.................................. (2.3)}$$

$$\text{\% Porosity} = \frac{W3-W1}{W1} \times 100 \quad \text{.......................... (2.2)}$$

The samples were fired to 900^{0}C in the oven and soaked for two hours to remove any moisture and burn off the organic matters present in the samples in order to create pores within the samples. The samples were allowed to cool at room temperature, weighed and the weights were recorded as W_1. The samples were suspended in water until bubbles formed disappears. At this point, the samples were removed, the edges cleaned, and weighed. The weights were recorded as W_3.

Shrinkage Test

This was done to assess the dimensional stability of the samples at high temperature. Equation 2.4 and 2.5 show the formula adopted to evaluate the percentage linear and volume shrinkage of the samples respectively.

$$\text{\% Linear Shrinkage} = \frac{L1-L2}{L1} \times 100 \quad \text{............................. (2.4)}$$

$$\text{\% Volume Shrinkage} = \frac{V1-V2}{V1} \times 100 \quad \text{........................... (2.5)}$$

Where: V_1/L_1= Green volume/length before firing; and

V_2/L_2 = Dry volume/length after firing

Rectangular samples were employed. The linear shrinkage of the fired bricks was determined by measuring the linear dimensions of the bricks before and after firing. The samples were initially heated to 110^{0}C and the green dimensions such as diagonal (D_1), length (L_1) and breadth (B_1) were taken. The samples were then fired in a resistance heating furnace preset at heating rate of 7 ^{0}C/min. The samples were fired at different temperatures; 250 ^{0}C and held for 1hr; 500^0 C held for 1hr; 750^0 C held for 1hr and 950^0 C held for 2hrs. After firing, the bricks were allowed to cool in the furnace; the dimensions measurement were re-taken as D_2, L_2 and B_2, and the percentage linear and volume shrinkages were evaluated using.

Green and Dry Compressive Strength

Fired bricks were tested for crushing strength using hydraulic strength testing machine and evaluated using equation 2.6.

$$\text{Compressive strength} = \frac{\text{Forceapplied}}{\text{Area}} \; (N/M^2) \quad \text{...................... (2.6)}$$

For the green compressive strength, the samples were heated to 100^{0}C and held for 1 hour. While for dry compressive strength, samples were heated to 950^{0}C and subsequent procedures were similar to that of refractoriness test.

3. RESULTS

The results obtained at the course of this research are presented in Table 2; showing the chemical composition of Ijapo Clay, Charcoal and Rice Husk.

Table 2: Chemical Analysis of Ijapo Clay and Charcoal and Rice Husk (Additives)

Samples/Composition	Ijapo Clay	Charcoal	Rice Husk
Al_2O_3	36.1	BDL	BDL
SiO_2	53.7	5.9	83.9
SO_3	0.09	0.85	0.91
K_2O	1.89	11.2	3.73

TiO_2	2.16	0.2	0.55
Fe_2O_3	5.007	7.56	5.83
CaO	BDL	63.0	2.44
RuO_2	BDL	6.95	1.71

BDL = below detectable level.

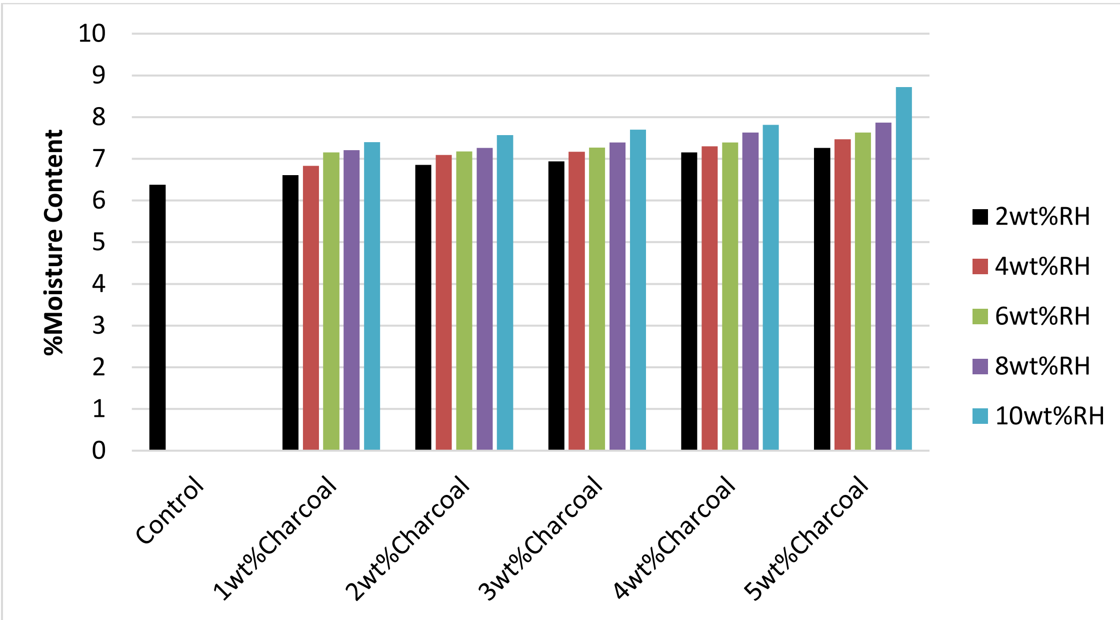

Figure 1: Moisture content of 100 % Ijapo clay (Control) and Ijapo clay plus varying weight % Additives (Charcoal and Rice husk)

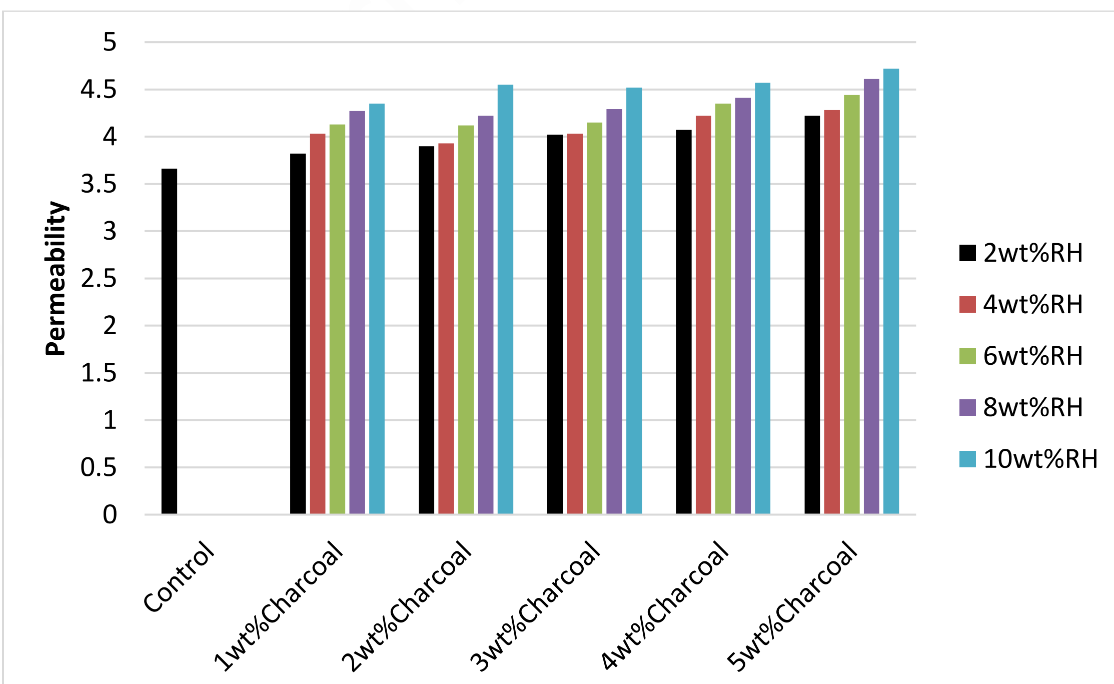

Figure 2: Permeability of 100 % Ijapo clay (Control) and Ijapo clay plus varying weight % Additives (Charcoal and Rice husk)

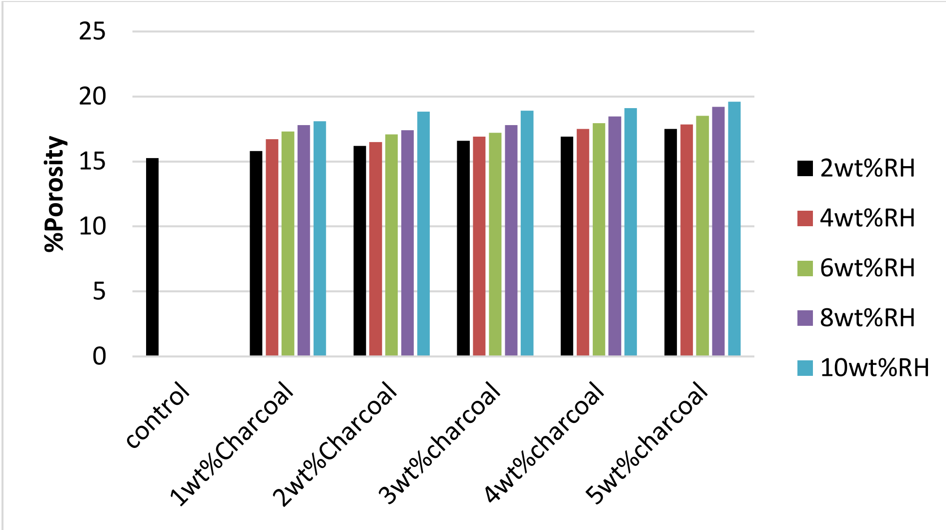

Figure 3: Apparent porosity of 100 % Ijapo clay (Control) and Ijapo clay plus varying weight % Additives (Charcoal and Rice husk)

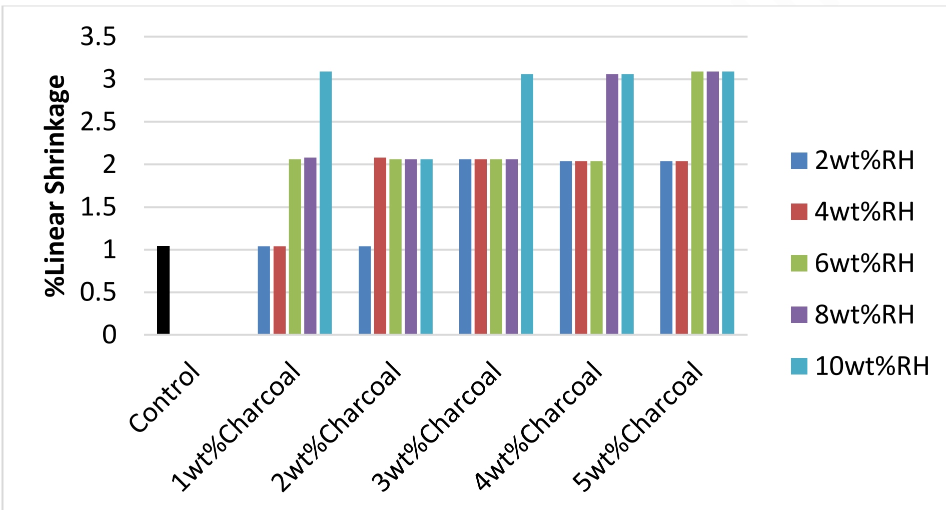

Figure 4: Linear shrinkage of100 % Ijapo clay (control) and Ijapo clay plus varying weight % Additives (Charcoal and Rice husk)

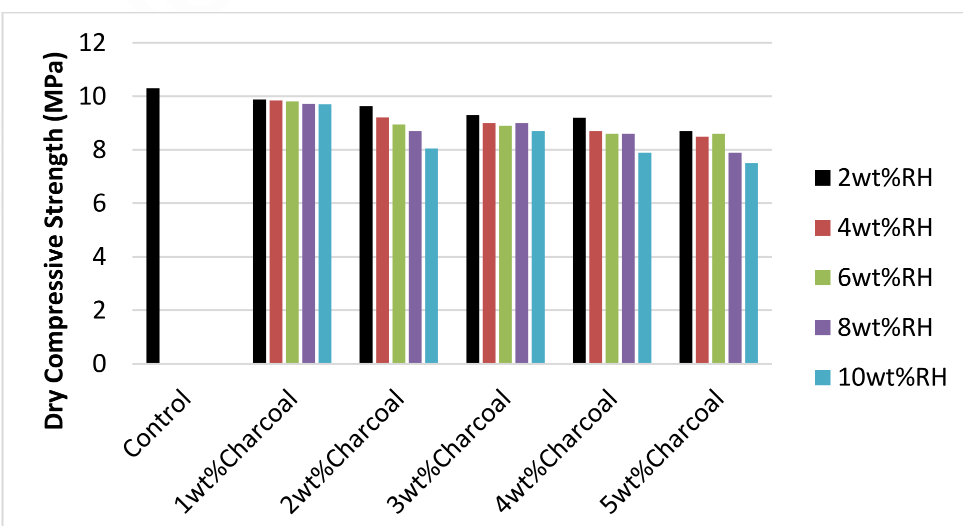

Figure 5: Dry compressive strength of 100 % Ijapo clay (Reference) and Ijapo clay plus varying weight % Additives (Charcoal and Rice husk)

4. DISCUSSION

Chemical Analysis of Ijapo Clay and Additives

Table 2, shows the chemical analysis of the Ijapo clay, rice husk and charcoal. From this, it can be deduced that Ijapo clay assayed 53.7% SiO_2 and 36.1% Al_2O_3 as the major constituents and other trace compounds. The presence of high silica and alumina content affirms that Ijapo clay is an alumino-silicate in nature and also accounts for its characteristic hard texture. Chemical analysis of the additives reveals that charcoal contains CaO (63.0%); K_2O (11.2%), Fe_2O_3 (7.56%) and SiO_2 (5.9%) and the amount of fixed carbon in the charcoal was determined using carbon analyzer and the value was 42.63% carbon; while the rice husk is of high silicate content to the tune of (SiO-83.9%) other constituents are K_2O (3.72), CaO (2.84%) and Fe_2O_3 (5.83%). However, this result shows that high percentage lime is present in charcoal which is a good binder to high silica contained in rice husk and also contains fairly high carbon - 42.63 %, which also aid in impacting refractoriness to the produced bricks in the presence of low iron sourced from the iron oxide present in both additives [9].

Refractoriness

The refractoriness of the Ijapo clay was found out to be 1350^{0}C. Hence, it can be used as refractory lining for non-ferrous extraction whose melting point are below 1200^{0}C since its softening temperature lies in the recommended range for fire-clay refractory bricks [9, 10]. The relatively high refractoriness can be attributed reasonably due to high alumina content of 36.1%, fairly low high carbon present in charcoal and low content of iron (low amount of impurity oxides)which when present in high amount can adversely affect the refractoriness.

Moisture Content

Figure 1 reveals the percentage moisture content of 100 % Ijapo clay (Control) and Ijapo clay plus varying weight % Additives (Charcoal and Rice husk). Moisture content of Ijapo clay brick was roughly 6.38 % while that of additives vary from 6.61-8.0 %. This variation is mainly due to slight differences in amount of rice husk and charcoal added. The low moisture content can be attributed to low weight percent (wt %) of charcoal and rice husk. The moisture content was observed to increase with increasing weight percent of additives. This may be due to the pore forming ability of the rice husk which enables the samples to absorbed more water. It has been noted that the additives showed acceptable limit (≤20%) of water absorption in all cases. The highest moisture content of 8.72 % was observed at 5 wt% charcoal and 10 wt% rice husk addition which is still within the standard limit [11]. However, the moisture content of a good quality brick should not exceed 20 % of its dry weight when immersed in water for 24 hrs [11].

Permeability

The permeability of the Ijapo clay bricks also followed the same trend like the porosity since both permeability and porosity are closely related. Figure 2 shows the permeability of Ijapo clay brick (control) and brick with additives. The permeability of the fired samples was observed to increase with increasing weight percent additives. Optimum permeability was observed for composition 5 wt% charcoal and 10 wt% rice husk with values found to be within the internationally defined standard of 15-25% [12].

Apparent Porosity

Porosity refers to the proportion of voids (or pores) per unit volume of a porous solid. Usually porosity is related to mineralogy, internal brick structure and geometry [11]. Figure 3showed that the highest porosity was observed for compositions with 10% rice husk addition, and the lowest porosity of 18% with no additives (i.e. 100% Clay). The high values of porosity and moisture content caused high thermal resistance. The result demonstrated that fired bricks exhibited different apparent porosity values depending on the amount of additives and its ability to burn off at high temperature. The porosity of the fired samples strongly depends on the amount of additives which burns off during the firing and resulted in the observed porosity. The voids insulate thermal flow thereby causing a decrease in thermal conductivity of the samples as the amount of charcoal or wheat husk was increased.

Linear Shrinkage

This is an indicator of the firing efficiency of the clay samples. A range of 4-10% for fireclays has been recommended. Lower values were more desirable as this means the clay is less susceptible to volume change [13]. Ijapo clay has a comparatively lower linear shrinkage value of 1.04% and higher value of 3.09% but falls within the range for normal kaolin ($Al_2O_3.2SiO_2.2H_2O$) range which is between 7-10% [14]. This shows that the Ijapo clay can withstand high temperature with deformation (high dimensional stability) and thus regarded as refractory clay. It is evident from Figure 4 that the percentage linear shrinkage increases slightly with an increase in the amount of charcoal as well as rice husk addition. The increase in linear shrinkage has been attributed to the removal of residual and chemically combined water as well as conversion of additives into ashes during firing which evidently decrease the volume [15]. The increase in linear shrinkage with increasing rice husk is mainly due to the burning off of rice husk which occupied spaces in the composite bricks during firing thereby, leaving open spaces which caused shrinkage to occur [14]. These chemical reactions during firing along with the re-arrangement of grains/particles and re-orientation in the crystal lattice, form a more compact solid texture in comparison to the initial state which causes shrinkage.

Green and Dry Compressive Strength

Figure 5 shows the observed variation in mechanical strength of clay bonded brick at varying percentages of the additives-charcoal and rice husk. The result indicated that the strengths of test specimens depend on the percentage weight (wt %) of additives. It was deduced that the compressive strength of test specimens fired at 950^0C decreased with an increase in the quantity of charcoal and rice husk compared to the highest compressive strength of control sample. Though, the decrease in strength was minimal across the additive addition with the least compressive strength observed for composition of 5wt% charcoal and 10wt% rice husk. Generally, in clay based ceramic systems, strength decreases with an increase in porosity [14].

5. CONCLUSION AND RECOMMENDATION

In this research, Ijapo clay and its additives have been characterized. The claybricks were reinforced with varying weight percent of charcoal and rice husk additives. It can therefore be concluded that:

1. chemical analysis showed that the Ijapo clay contains high silicate and moderate alumina content, low ferrous oxide content and possess very low contents of other metal oxides.
2. the refractoriness of the Ijapo clay brick was found to be 1350^0C.
3. moisture content, porosity, permeability and linear shrinkage were found to increase with increasing weight percent of charcoal and rice husk having values meeting the required standard for a refractory brick; and
4. the refractoriness and dry compressive strength decreases having values below the required standard.

Therefore, due to these ensuing properties, Ijapo clay may be unfit for heavy refractory applications, however, considering its low cost, availability and easy accessibility, Ijapo claybrick of composition 5 wt% charcoal and 10 wt% rice husk is best opt for as refractory for low duty application at low refractory temperatures.

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REFERENCE

1. Umaru, M., Aliyu, M. A., Aris, M. I. and Munir, S. M., (2012). A Comparative Study on the Refractory Properties of Selected Clays in North Central Nigeria. Academic Research International, 3 (1): 393 – 398.
2. Ameh, E. M., and Obasi, N. L., (2009) Effect of rice husk on insulating bricks produced with Nafuta and Nsu clays. Global Journal of Engineering and Technology. 1(1): 661-668.
3. Irabor P.S.A.,Physical and chemical investigation on some Nigerian kaolinite clays for use in the ceramics and allied industries, (2002).Nigerian Journal of Engineering Research and Development.1 (1): 54-59.
4. Aramide. F.O. Alaneme, K. K., Olubambi, P. A., and Borode, J.O., (2014). Characterization of some Clay Deposits in South West Nigeria. Leonardo Electronic Journal of Practices and Technologies. 1 (25): 46 – 57
5. Jock, A., Ayeni, F. A., Jongs, L. S., Kangpe, N.S., (2013) Development of Refractory bricks from Nigerian Nafuta Clay Deposit. International Journal of Materials, Methods and Technologies. 1(10): 189-195.
6. Misra, M. I., (1975). Refractories: Their manufacture, Properties and Uses. SMT, Lakshmi Misra, Krishna colony, Ghamapur, Jabalphur, 4(1): 8-13, 22-42.
7. Ahmad, S., Iqbal, Y., Muhammad, R., (2017). Effects of coal and wheat husk additives on the physical, thermal and mechanical properties of clay bricks. Boletindelassocie daadespanoladeceramicyvario, 5 (6): 131–138.
8. Lingling, X., Wei, G., Tao, W., Nanru, Y., (2005). Study on fired bricks with replacing clay by fly ash in high volume ratio, Constr. Build. Mater. 19 (1): 243–247.
9. Chesti, A.R., (1986).Refractories: Manufacture, Properties and Applications. Prentice-Hall of India private Ltd, New Delhi,1(1): 55-63.

10. Yami, A. M., and Umaru, S. (2007).Characterisation of some Nigerian clays as refractory materials for furnace lining. Continental J. Engineering Sciences. 1 (1): 30-35.
11. Olawuyi, B., Olusola, K., Ogunbode, E. and Kyenge, S., (2014). Performance assessment of Makurdi burnt Bricks, Civil Engineering. 1(1): 12 -16.
12. Mazen, N.A., (2009). Production of fire clay refractory bricks from local materials. European Journal of Scientific Research, 1(1): 386-392.
13. Omowumi, O.J., (2000). Characterization of some Nigerian clays as refractory materials for furnace lining. Nigeria Journal of Engineering Management. 2 (3): 1-4.
14. Abolarin, M. S., Olugboji, O. A., and Ugwoke, I. C.,(2004) Experimental investigation on local refractory materials for furnace construction. 5th Annual Engineering Conference Proceedings Minna, 1(1): 82-85.
15. Darweesh, H. and El-Meligy, M.,Non-conventional light-weight clay bricks from homra and kraft pulp wastes. Journal of Chemistry and Materials Resources. 4 (1): 123–129.