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Enhancement of Refractory Properties of Blended Clay with Groundnut Shell and Rice Husk Additives.

Chima, O.M[1], Nwoye, C.I[2], Nnuka, E.E[2].

[1](Department of Mechanical Engineering, College of Engineering and Engineering Technology, Michael Okpara University of Agriculture, Umudike, Nigeria)

[2](Department of Metallurgical and Material Engineering, Faculty of Engineering, Nnamdi Azikiwe University Awka, Nigeria)

Abstract : *Enhancement of the refractory properties of blended Elu-uhu Nguzu and Amaiyi Edda clays were achieved with combination of two local agro additives incorporated into the blended clay at different formulations. The chemical and mineralogical compositions were determined using x-ray fluoresce and x-ray diffraction techniques respectively, while the micro structural examination was done using scanning electron microscope. The two clays were blended at the ratio of 40:60 and further incorporated with groundnut shell and rice husk at four different formulations of 5, 10, 15 and 20%. The test samples produced were fired at 1200^{0}C and thereafter subjected to physico-mechanical tests using standard procedures. Results obtained show that Nguzu and Amaiyi clays have low alumina contents of 21.8% and 22.9% respectively and suggest the need for increasing the oxide contents to enhance the refractory properties. Groundnut shell has 7.6% alumina while rice husk has 21.3% phosphorus oxide. These oxides are useful for refractory purposes. Linear shrinkage and apparent porosity of the blended clay increased from 6.59% to (8.4 - 9.68) % and 15.38% to (23.91 – 42.85) % respectively for the various formulations while bulk density showed decrease from 1.77 to 1.39 and 1.43 only on 15% and 20% additives respectively. Modulus of rupture showed decrease with percentage composition of the additive from 32.7N/mm^2 to (30.3 - 26.5) N/mm^2 Thermal shock resistance slightly decreased from 30 cycles to 29-28 cycles with the additives. Refractoriness increased from 1550^{0}C to 1580^{0}C while thermal conductivity improved from 3.18W/m^{0}C to 0.86 W/m^{0}C. Hence, it was concluded that good insulating and refractory properties required for high temperature application could be obtained in clay material when blended with groundnut shell and rice husk additives*.

Keywords: *Refractory, blended clay, formulation, enhancement, composition*

I. INTRODUCTION

Refractory materials are materials that are capable of withstanding high temperature both physically and chemically. High quality refractory materials resist high temperature fluctuations between 1000^{0}C- 1500^{0}C. They conserve heat within the system and support the structural frame work of the furnace. They are expected to be thermal insulators with low thermal conductivity and provide temperature control [1]. Refractories are utilized in high temperature engineering applications. They possess material stability; wear resistance, excellent thermal shock tolerance and other high temperature related properties. They are durable and usually their usefulness is affected either by bending, cracking or occasionally by attack of carbon II oxide and slag in steel making [2]. Previous research works in industrial applications of refractories have proved that no single clay material has the capacity of providing all these properties required for most applications. Deficiency in the properties of clay materials limit its use and pose a lot of problems in brick manufacturing industries. This necessitates blending of clay materials, so as to exploit the good properties of the different deposits to improve those of low quality. It has also led to the use of blends of different clays and some local agro waste materials, as additives to clay material with the aim of improving the desired properties of the refractory materials. The wastes are hence converted into useful additives by incorporating them into clay to form a high quality refractory material that will meet international industrial standards. This will add value to the clay material and thereby widen its areas of application and also give the agro waste material some economic value and also clean up the environment. This constitutes the major objective of this study.

Many researchers have attempted to use local agro waste materials to improve the refractory properties of materials. Some used both controlled and uncontrolled rice husk ash and ground rice husk to improve refractory properties of clay and found that uncontrolled rice husk ash has more porous structure due to high carbon content which limited it to some applications [3]. Others blended rice husk with clay to develop refractory brick with plenty pores after heat treatment due to burning out of organic material [4]. Further studies were centered on the effect of rice husk and groundnut shell on the properties of clay and the conclusion was that properties like porosity, thermal conductivity, linear shrinkage and density of clay can be improved significantly to suit a desired insulating property on the addition of rice husk and groundnut shell separately [5], [6]. Recent research studies on the use of rice husk additive on clay material have proved that porosity and thermal shock resistance improved by 35% and ten folds respectively while linear shrinkage and bulk density reduced from 3.89 to 3.00 and1.98 to 1.52 respectively [7].

Therefore, refractory properties like shrinkage, porosity and bulk density are interrelated. The more a material shrink, the less porous it becomes and the density rises [8], [9]. Bulk density, porosity and shrinkage depend largely on the air voids in the clay. When the void is reduced, it results to shrinkage, less porosity and high density [10]. Therefore this research study aims at using a blend of groundnut shell and rice husk additives to enhance the refractory properties of a blend of two clays and to determine the percentage of the additives that give maximum performance enhancement when compared with standard values. This will add value to the clay and also give the agro additives industrial exposure for exploration and thereby convert waste to wealth.

II. MATERIALS AND METHODS

2.1 MATERIALS.

The materials used in the research work were two clay samples from Elu-uhu Nguzu Edda deposit and Amaiyi Edda deposit both from Ebonyi State Nigeria, rice husk from Abakaliki rice mill and groundnut shell from local market of the same state.

2.2 EQUIPMENT

The equipment and machines used in the work include Empyrean X-ray diffraction equipment made by panalytical of Netherland, minipal 4 ED- X-ray fluorescence made by panalytical of Netherland, Scanning Electron Microscope (SEM) Zeiss, model EV010, electric furnace, (Thermodyne 46200) and ceramic kiln, model 88FC2468, electrical transversal strength machine, model 235, digital weighing machine, spiral balance, sieves, mortar, pestle, moulds, pair of tongs, strong thread, heat conduction testing machine, model H9406/02877 PA Hilton, Pyrometric cone, meter rule, venier caliper.

2.3 METHODS

2.3.1 Chemical and mineralogical phase analysis of raw materials

Samples of the two clay materials and the local additives (groundnut shell and rice husk) were analyzed to determine their chemical constituents and mineralogical composition. The chemical analysis was done using X-ray fluorescence (XRF) analysis machine while the mineralogical composition was done using computerized X-ray diffractometer that uses Cu Kα radiation at a scan speed of 4^0/min.

2.4 RAW MATERIAL BENEFICIATION/PREPARATION

The raw clay samples were air dried before processing. The raw dry clay samples were crushed in a mortar to small grain sizes. The samples were soaked in a plastic container of water and allowed to soak for three days. The clay was dispersed in excess water in a pre-treated plastic container and stirred vigorously to ensure proper dissolution. Mineral constituents, soluble alkalis and dead organic matter were removed by washing during the soaking. This treatment was done since the presence of alkali oxides (Na_2O and K_2O) tend to retard mullite formation, hence lowers refractoriness and strength of clay brick. The dissolved clay was then filtered through a 0.425 mm mesh sieve to get rid of unwanted particles and plant materials. The filtrate was filtered further by the use of a mesh sieve of size 0.18 mm in order to obtain finer particles. The filtrate was allowed to settle for three days after which excess water was decanted off. The clay slip obtained was sun dried for 2 days and then dried in an oven at 100^0C.

The processed dried clay was pulverized and then passed again through a 0.18mm mesh sieve. Each of the clay samples was mixed with water and molded using different mould size that suited the respective tests they were to be used for. Nguzu and Amaiyi clays were later blended in the ratio of 40:60. The second phase of the experiment was carried out with the incorporation of groundnut shell and rice husk as the local additives into the clay. These additives at equal proportion were incorporated in combination into the Nguzu-Amaiyi blended clay at 5%, 10%, 15% and 20% formulations to develop different test samples.

2.5 MOLDING OF THE TEST SAMPLES

Each of the samples was mixed with 35% of water to make the clay plastic for molding. The clay was then molded into three different shapes using metallic moulds with the application of engine oil lubricant to the surface of the moulds to prevent the test pieces from sticking to the surface. The moulds were used for forming the bricks. An improvised wooden material was prepared which was used for transmitting the molding pressure of 2MPa to the mould when the required quantity of plastic molding mass was put into the mould. After pressing, a suitable wooden plunger was used to extrude out the green brick from the mould. The extruded green bricks were given a 50 mm mark using venire caliper and thereafter weighed to take the individual green weight. The test samples were sundried, oven dried to 110^0C and finally fired to 1200^0C before testing for the respective properties.

2.6 MICRO STRUCTURAL EXAMINATION

The micro structure of the clay brick was observed using scanning electron microscope (SEM), Zeiss, model EV010. The samples were placed on the holder and the images were captured under various magnifications and particle size diameters. The SEM was operated at an accelerating voltage of 15kV.

2.7 DETERMINATION OF THE REFRACTORY PROPERTIES OF THE MATERIALS

2.7.1 Determination of linear shrinkage

This test was done to determine dimensional stability of the sample after a given period of time and temperature change and the testing method is in line with ASTM C-326 standard for testing linear shrinkage of refractory material. Measured 50 mm points were marked on the surface of the test green sample. The green test sample which was given a 50 mm mark (L_o) on the surface was dried in the kiln to a temperature of 110^0C and thereafter the sample was brought out. The same previous 50 mm mark was measured to get the new length of the points after drying (L_d). The same specified length on the test sample was used when the sample was fired and the new dimension of the length was taken as fired length L_f. The dry-fired shrinkage was calculated as the linear shrinkage represented as:

$$\left(\frac{L_d - L_f}{L_d}\right) \times 100 \qquad (1)$$

2.7.2 Determination of bulk density and apparent porosity

The method of test used to determine these properties is in line with ASTM C 20-80a, standard test method for apparent porosity, water absorption and bulk density. The long rectangular shaped test sample measuring 9.5cm length, 2cm width and 5cm height was used for these two experiments. The specimens were fired to the required temperatures in preparation for the test. The dry weight (W_a) in air was taken using digital weighing balance. They were transferred into a vessel of boiling water for 30 minutes after which the boiling was discontinued. The specimens were allowed to cool to room temperature in the vessel of water for four hours. After 30 minutes in cold water, the specimens were tied to a string on a spiral balance suspended in a beaker of water, to get the suspended weight (W_{sp}). The specimens were removed from the water and gently cleaned before weighing it again to get the soaked weight (W_{so}).

From the above data, the physical parameters stated above were calculated:

$$\text{Bulk density} = \left(\frac{\text{Weight in Air } (W_a)}{\text{Soaked Weight}(W_{so}) - \text{Suspended Weight } (W_{sp})}\right) \qquad (2)$$

$$\text{Apparent porosity} = \left(\frac{\text{Soaked Weight}(W_{so}) - \text{Weight in Air } (W_a)}{\text{Soaked Weight}(W_{so}) - \text{Suspended Weight } (W_{sp})}\right) \times 100 \qquad (3)$$

2.7.3 Determination of refractoriness

The refractoriness or softening point was determined using the method of pyrometric cone equivalence (PCE) in accordance with ASTM C24-79. The test pieces were mounted on the refractory plaque along with some standard cone whose softening points are slightly above or below those expected of the test cones. The plaque was then inserted into the electric furnace. The temperature was raised at the rate of 5^0C per minute during which softening of Orton cone occurred along with the specimen test cone. The temperature was further raised up to 1400^0C and samples that with-stood the temperature were soaked in the furnace at that same temperature for 5 and 6 hours for the blended clay and the composite clay respectively, until the tips of the test cones had bent over the level with the base. Then the plaque bearing the specimens was removed from the furnace and the test cones examined when cold. The test cones were then compared with the standard cones and the test materials were said to have the pyrometric cone equivalent (PCE) of the standard cone that it resembled most in bending behavior. The refractoriness of each test cone is the number of the standard pyrometric cone that has bent over to a similar extent as the test cone. The temperature corresponding to the cone number was read off from the ASTM Orton series. The minimum (PCE) for intermediate and high are 24 and 27 which corresponds to the following fusing temperatures 1400^0C (5hrs), 1400^0C (6hrs) respectively. The soaked time in minutes was converted to equivalent temperature value by dividing with a factor of two.

2.7.4 Determination of thermal shock (spalling) resistance

This test was carried out with the help of an electrical furnace (Thermodyne 46200) heated at the rate of 5^0C/min. The thermal shock resistance was determined by prism spalling test method according to ASTM C-484 standard in which the spalling resistance was measured by the number of thermal cycles (heating, cooling and testing for failure). The test pieces of refractory bricks were thoroughly dried and placed in the cold furnace and heated at the rate of 5^0C/minute until the furnace temperature got to 1200^0C. The samples were then removed one after the other using a pair of tongs and cooled in air for 10 minutes, and then observed for cracks. In the absence of cracks (or fracture), the bricks were put back into the furnace and reheated for a further period of 10 minutes and then cooled for another 10 minutes. This process or cycle of heating, cooling and observing for cracks was repeated until cracks were observed. The number of complete cycles that produced visible cracks in each specimen was noted. This constituted the thermal shock (spalling) resistance.

2.7.5 Determination of thermal conductivity

The thermal conductivity was determined under steady state condition at room temperature. The test was conducted using heat conduction equipment. Circular test specimens measuring 40 mm diameter and 4mm thickness were used for the test. The specimens were inserted and clamped in between the heater and cooler faces of the equipment. 5Watts power input was selected and maintained for 30 minutes until steady state conditions were achieved. The temperature T at all six sensor points (three on the heater section and three on the cooler section) were recorded. The thermal conductivity was calculated using Fourier's law as:

$$K = \frac{Q\,dx}{A.dT} \qquad (4)$$

Where Q is quantity of heat supplied, x is the specimen thickness; A is the cross sectional area of the specimen sample and dT is the temperature difference between the two circular faces.

2.7.6 Determination of modulus of rupture

The modulus of rupture property tested in this work was done using 3 point bend tester in accordance with ASTM C-648 standard for testing modulus of rupture. The electrical transversal strength machine was used to determine the breaking load, P (kg). A venire caliper was used to determine the distance between supports L (cm) of the transversal machine. The height H (cm) and the width B (cm) of the broken pieces were determined. The modulus of rupture was then calculated as:

$$\text{Modulus of rupture Kg/cm}^2 = \frac{3PL}{2BH^2} \qquad (5)$$

Where P is load applied when the specimen failed, L is the distance between the centre lines of the lower bearing edges of the equipment, B is the width of the broken specimen and H is height of specimen (cm).

III. RESULTS AND DISCUSSION

The results of this study are shown in Tables 1-6, Figure 1 and 2 and Plates 1-5

Table 1 Chemical composition of Amaiyi clay

Oxide	Al_2O_3	SiO_3	K_2O	CaO	TiO_2	V_2O_5	Cr_2O_3	Fe_2O_3
Composition	22.9	48.9	5.15	2.79	1.63	0.098	0.037	10.51
Oxide/Element	CuO	ZnO	Ga_2O_3	MoO_3	Ag_2O	Eu_2O_3	Au	HgO
Composition	0.019	0.02	0.018	3.9	1.98	0.14	0.072	0.12

Table 2 Chemical composition of Nguzu Clay

Oxide	Al_2O_3	SiO_3	K_2O	CaO	TiO_2	V_2O_5	Cr_2O_3	Fe_2O_3	Re_2O_7	Bi_2O_3
Composition	21.8	54.4	1.77	0.49	1.89	0.10	0.033	14.62	0.08	1.0
Oxide/Element	CuO	ZnO	Ga_2O_3	MoO_3	Ag_2O	Eu_2O_3	SO3	MnO	IrO_2	
Composition	0.022	0.01	0.001	0.57	0.845	0.17	2.0	0.005	0.12	

Table 3 Results of chemical analysis of rice husk

Oxide	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	MnO
Composition	60.8	21.3	2.76	7.77	3.07	0.35	0.771
Oxide	Fe_2O_3	NiO	CuO	ZnO	BaO	Eu_2O_3	Re_2O_7
Composition	2.41	0.024	0.059	0.24	0.13	0.09	0.19

Table 4 Results of chemical analysis of groundnut shell

Oxide	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	MnO	Al_2O_3

Composition	16.0	8.0	5.1	10.6	30.4	1.5	0.95	7.6
Oxide	Fe_2O_3	NiO	CuO	ZnO	BaO	Eu_2O_3	CeO_2	Yb_20_3
Composition	13.0	0.2	1.2	0.3	1.5	2.2	0.6	0.9

3.1 CHEMICAL ANALYSIS

The results of the chemical analysis shown in Table 1 indicated the alumina content of Nguzu clay and Amaiyi clay to be 21.8% and 22.9% while the silica content was 54.4% and 48.9% respectively. This suggested a low alumina value and as well low alumina – silica ratio. The analysis of the agro additives showed that groundnut shell had 7.6% of alumina while rice husk had 21.3% phosphorus oxide. The alumina content of the groundnut shell supplemented that of the deficient clays to enhance the refractoriness and strength while the phosphorus oxide from rice husk formed aluminum phosphate bond in the refractory material which has non wetting effect against molten metal. Besides, it increases the resistance of refractory lining to crack and Carbon II oxide attack which is a byproduct of the furnace activity.

3.2 MINERALOGICAL ANALYSIS

The mineralogical phase analyses done with the aid of XRD and results interpreted using International Centre for Diffraction Data (ICDD) software revealed the main mineralogical phases in Amaiyi clay to be kaolinte ($Al_2Si_2O_5(OH)_4$), xonotlite ($Ca_6Si_6O_{17}(OH)_2$), chrysotile ($Mg_3[Si_2$-$xO_5](OH)_{4-4x}$, quartz (SiO_2), anhydrite ($CaSO_4$), os($KMg_2Al_3(Si_{10}Al_2)O_{30}$) and clinochryso ($Mg_3Si_2O_5(OH)_4$) while that of Nguzu clay were periclase (MgO), Sep($Mg_4Si_6O_{15}(OH)_2.6H_2O$), antigorite ($Mg_3Si_2O_5(OH)_4$), sanid (NaK) ($(Si_3Al)O_8$), os($KMg_2Al_3(Si_{10}Al_2)O_{30}$), mu($KAl_2(Si_3Al)O_{10}(OH)_2$), riebeckit $(Na,Ca)_2$ (Fe,Mn)3 $Fe_2(Si,Al)8O_{22}(OH)$) and chrysotile ($Mg_3[Si_2$-$xO_5](OH)_{4-4x}$).

3.3 MICRO STRUCTURAL EXAMINATION RESULTS

The micro structural images shown by SEM indicated a more homogeneous structure in Nguzu-Amaiyi blended clay than in the structure that contained groundnut shell and rice husk which contained some agglomeration of composite material with more pore sizes. This is indicated by dark spots in the structures. The increased pore sizes gave rise to increased apparent porosity observed in the result. The image also revealed the presence of mullite shown by whitish needle like shape. This phase is responsible for good value of refractoriness and other related properties.

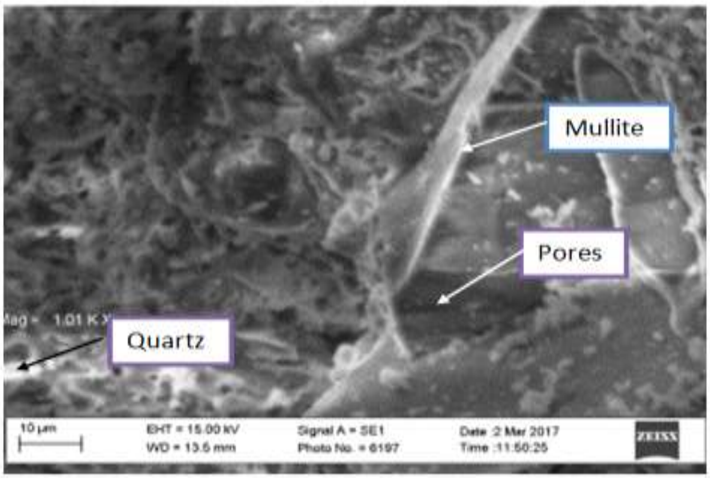

Plate 1 SEM for blended Nguzu-Amaiyi clay

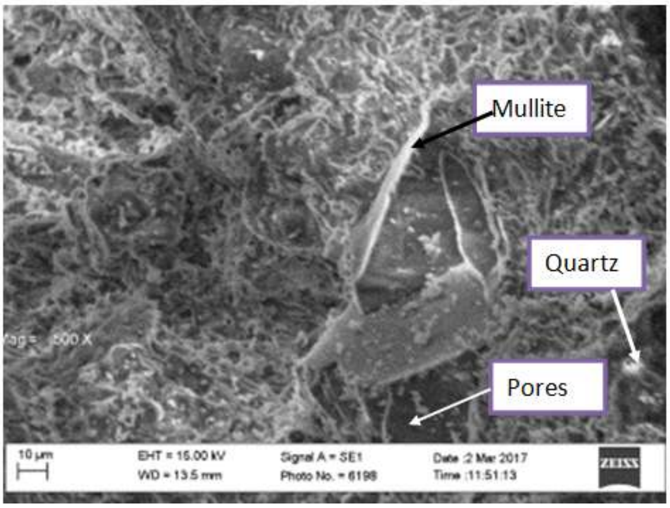

Plate 2 SEM for blended Nguzu – Amaiyi clay

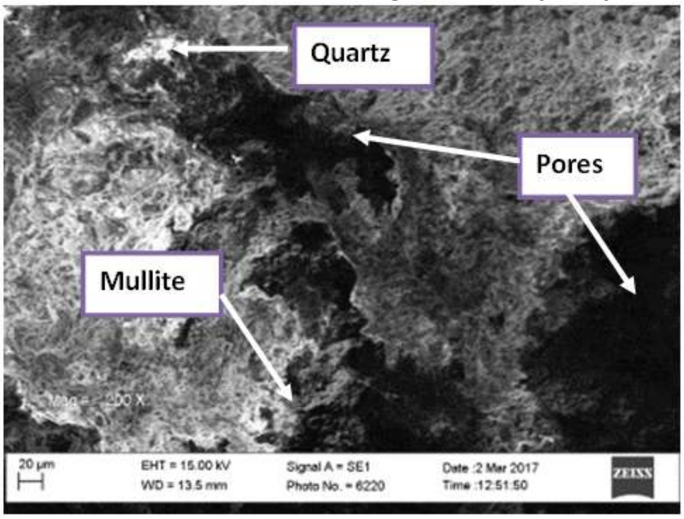

Plate 3 SEM for composite clay with groundnut shell and rice husk additive

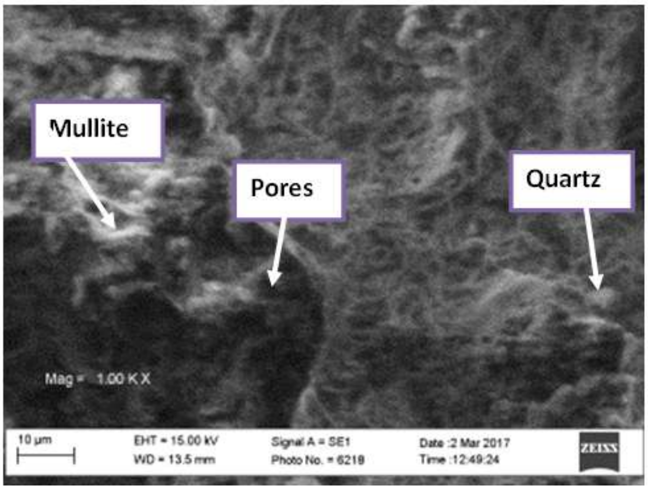

Plate 4 SEM for composite clay with groundnut shell and rice husk additive

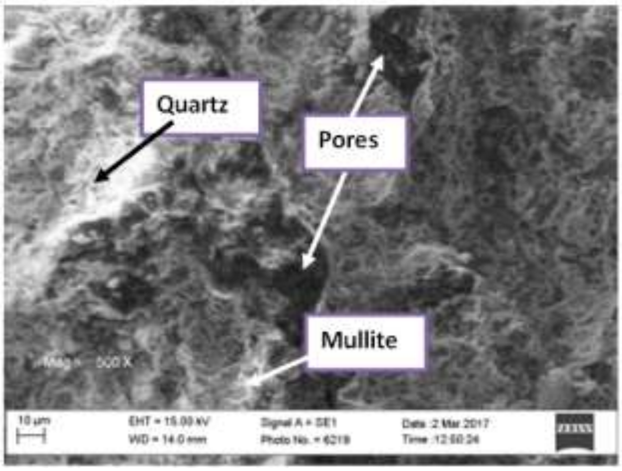

Plate 5 SEM for composite clay with groundnut shell and rice husk additive

Table 5. Refractory properties of blended and composite clays compared with internationally accepted standard values

Refractory properties	Linear shrinkage (%)	Bulk density g/cm^3	Apparent porosity (%)	Modulus of rupture N/mm^2	Spalling resistance (cycles)	Thermal conductivity W/m^{0}c	Refractorines s (^{0}C)
Blended clay	6.59	1.77	15.38	32.7	30	3.18	1550
Composite clay	8.4 - 9.68	2 - 1.39	23.91 - 44.89	30.3 - 26.5	29 - 28	0.86	1580
Internationally Accepted values	7 - 10	1.71 - 2.1	2 - 30	-	25 - 30	-	1500-1700

Source: [11]

3.4 REFRACTORY PROPERTIES OF BLENDED AND COMPOSITE CLAY BRICKS

The refractory properties of the blended clay and the composite clay are shown in TABLE 6.

Table 6. Refractory properties of blended and composite clay bricks with various formulations.

Refractory property	Blended clay	Blended clay with groundnut-rice husk additives (5%)	Blended clay with groundnut-rice husk additives (10%)	Blended clay with groundnut-rice husk additives (15%)	Blended clay with groundnut-rice husk additives (20%)
Linear Shrinkage (%)	6.59	8.40	8.51	9.68	9.68
Bulk density g/cm^3	1.77	2.00	1.87	1.39	1.43
Apparent Porosity (%)	15.38	23.91	34.09	44.89	42.85
Modulus of Rupture N/mm^2	32.7	30.3	28.1	27.0	26.5
Thermal Shock Resistance (cycles)	30	29	28	29	28
Thermal Conductivity W/m^{0}c	3.18	-	-	0.86	-
Refractoriness (^{0}c)	1550	1580	1580	1580	1580

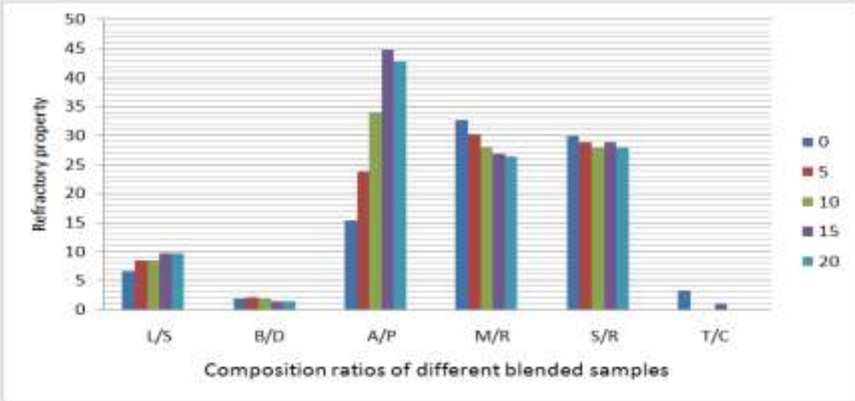

Figure. 1: Variation of refractory properties with composition for blended and composite clay samples

3.4.1 LINEAR SHRINKAGE.

The results of this test revealed that linear shrinkage for the blended clay was 6.59% while those blended with the additives were 8.40, 8.51, 9.68, 9.68% for 5, 10, 15 and 20% formulation respectively. The values of the composite clays were within the recommended values for internationally accepted standard of 7-10% for refractory bricks [11] while those of blended clays were within the recommended values of 4-10% for fire clay [12].The results obtained were also in agreement to what some other previous research works reported. [5], [6]. Hence, within the range of composition of additives investigated; it was found that the stability of the material was not impaired. It was also noted that linear shrinkage increased with increase in the composition of the additives.

3.4.2 Porosity and bulk density

The value of the apparent porosity of the blended clay was 15.38% while those with the additives were 23.91, 34.09, 44.89, and 42.85 for 5, 10, 15 and 20% formulations respectively. It was found that only 5% formulation of the additives yielded values within the acceptable range of (2-30%) as suggested for refractory clay [13]. It was observed from the results of bulk density test that the values of bulk density significantly decreased at 15 and 20% formulation of the additive. This decrease in bulk density can be traced to increase in apparent porosity and linear shrinkage. This is in agreement with some research conclusions that noted the correlation between bulk density, apparent porosity and linear shrinkage and hence, stated that denser clay materials are less porous and less likely to shrink. [8]

3.4.3 Modulus of rupture

The result of modulus of rupture test shown in Table 2 indicated that with the introduction of the additives, the value of modulus of rupture of the blended clay initially at 32.7N/mm^2 decreased with increase in the composition to 30.3, 28.1, 27, and 26.5 N/mm^2 for 5, 10, 15 and 20% formulations respectively. The decrease in the value may be traced to reduction in clay inter particle bonding strength due to the presence of the agro material additive in the composite material.

3.4.4 Thermal Shock Resistance

Results obtained from thermal shock resistance test showed that the thermal shock resistance of the material slightly decreased from 30 cycles to 29 and 28 cycles respectively. However, the values are still within the acceptable range of 20 -30 cycles as recommended by some researchers. [13].

3.4.5 Refractoriness

From the results of the refractoriness test, it was noted that for the blended clay, the softening point occurred at 1550^{0}C while for those blended with the additives, at all the four different formulations, it was 1580^{0}C. This suggests an enhancement in the refractoriness value which may be traced to the presence of useful oxides found in the additives. The oxides of alumina and phosphorus though in trace quantity have improved the refractoriness of the composite clay materials to the value required for good refractory material. It was also found that combination of two additives yielded better refractoriness value than single one which was done in previous research works.

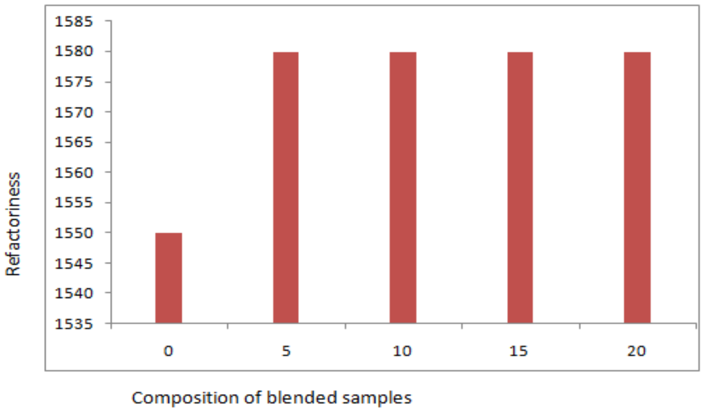

Figure.2: Variation of refractoriness with composition for blended and composite clay samples

3.4.6 Thermal conductivity

The insulating property of the material observed at 5 Watts input power showed that the value of thermal conductivity of the blended clay was 3.18 W/m^{0}C while that of the composite clay with groundnut and rice husk additive was 0.86 W/m^{0}C. This suggested that the composite material blended with the additives were more insulating than the blends of the two original clays. This is due to the larger pores produced which contained air that is a poor conductor of heat, hence lowering the thermal conductivity value.

IV. CONCLUSION

Based on the results obtained in this work, it was concluded that;
-The clay material has low alumina value which suggests the need for enhancement.
-The agro additives contained some useful oxides like Al_2O_3 and P_2O_5 which improved the refractory properties of the clay material, therefore the waste material could be used to develop porous refractory bricks with combination of desirable properties.
-Linear shrinkage and apparent porosity generally increased while bulk density and modulus of rupture decreased with increase in the percentage composition of the additives. However, 5% composition gave optimum value of the properties investigated.
-Refractoriness and the insulating property of the material significantly improved with the additive while thermal shock resistance had an insignificant effect.
The insulating refractory properties of the clay material could be improved with the addition of combination of agro additives, example groundnut shell and rice husk additives.

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