PERFORMANCE ENHANCEMENT OF REFRACTORY PROPERTIES OF CLAY MATERIALS WITH LOCAL CONTENT INPUT

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ABSTRACT

Combination of local agro waste additives-(groundnut shell, sawdust and rice husk) mixed at equal proportions was used to enhance the performance of refractory properties of blended clay. The chemical composition of the clays and additives were determined with the help of x-ray fluoresce while the micro structural examination was done using scanning electron microscope. Two clay samples from Nguzu Edda and Amaiyi Edda deposits were blended at ratio 40:60 to get the control sample. Combination of the three different local waste additives was blended with the control sample at four different formulations (5, 10, 15 and 20%). The composite material was moulded and fired at 1200^{0}C and after that tested for the refractory properties. The refractory properties of bricks made with the control sample and the composite sample had respective values of (6.59 and 5.49%), (1.77 and 1.73 g/cm^3), (15.38 and 36.36%), (32.7 and 34.3N/mm^2), (30 and 32 cycles), (0.00318 and 0.00294 W/mm^{0}C) and (1550 and 1670^{0}C) for linear shrinkage, bulk density, apparent porosity, modulus of rupture, thermal shock resistance, thermal conductivity and refractoriness respectively. Hence, the results obtained show that percentage enhancement of (16.7, 72, 4.7, 6.2, 7.5 and 7.2 %) were achieved in linear shrinkage, apparent porosity, modulus of rupture, thermal shock resistance, thermal conductivity and refractoriness respectively. However, it was observed that bulk density deteriorated by 2.2%. It was also noted that the percentage composition of the additive had significant effect on the properties of the composite brick. Therefore, it was concluded that the use of combination of agro additives as secondary raw materials in brick production enhances the properties of the material. The additives are also good raw material for the production of insulating and refractory brick product of high quality.

***KEYWORDS**: Performance enhancement, refractory properties, additives, composite, clay*

1.0 INTRODUCTION

Refractories are non-metallic organic substances, mainly mixtures of oxides which are capable of withstanding high-temperature condition usually above 1580^{0}C without losing their chemical and mechanical integrity. Among the oxides found in refractory mixtures, those of silicon and alumina are the most common. Clay is the raw material used for refractory production. It is classified as low melting, high melting and refractory according to Nnuka and Agbo (2000).

The properties of refractory products depend on the oxide composition, processing method and particle size. These properties could be enhanced with the use of local content constituting of waste material. These materials have oxides that are used for the enhancement of the refractory properties. Besides, they are pore formers. When they are burnt off under high temperature, they leave larger pores in the structure of the clay. These pores enhance the insulating behavior of the material. It also impedes the movement of crack thereby increasing the resistance to thermal fluctuations of the material.

The use of refractory products with inadequate properties in high temperature engineering service is a major problem facing some local industries. This gives rise to frequent maintenance services and energy loss which increases production cost. The above stated problems led to the use of local additives on refractory brick production. They enhance the properties which improve the quality of the products. Therefore, this research study aims at using a combination of three different agro industrial wastes to improve the properties of refractory product for wider engineering applications.

Previous study like Fatai (2012) showed that agro waste like saw dust could improve strength at 10-15% composition and that porosity could be controlled by varying percentage composition of the saw dust mixture. The porosity on its own varies inversely to thermal conductivity, cold crushing strength and bulk density. Others stated that the addition of saw dust and groundnut shell to the clay sample could improve properties like porosity, thermal conductivity, linear shrinkage and solid density. Also the percentage composition could be varied to suit the particular insulating property desired. Manukaji, (2013a) and Manukaji, (2013b) Chima *et al*. (2017) found that combination of groundnut shell and rice husk improved refractoriness and the insulating property of clay material significantly while thermal shock resistance had an insignificant effect. Folarami, (2009) found that in the blend of sawdust, some properties improved, some remained stable while others deteriorated. Odo *et al.* (2013) found that rice husk as a combustible material can create a needed porosity of (25-50%*)* which is recommended for insulating bricks.

2.0 MATERIALS AND METHODS

2.1 Materials

The materials used in the research work were two clay samples. The deposits are located in Nguzu and Amaiyi Edda, Ebonyi State. The agro additives used include rice husk from Abakaliki rice mill, groundnut shell and gmalina sawdust. The equipment used were 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, thermal conductivity testing machine, model TK04, pyrometric cone, meter rule, venier caliper.

2.2 Methods

2.2.1Chemical Analysis of Raw Materials

Samples of the two clay materials and the local additives (groundnut shell, sawdust and rice husk) were analyzed to determine their chemical constituent and composition using X-ray fluorescence (XRF) equipment.

2.2.2 Raw Material Processing and Moulding

The raw clay samples were air dried and crushed to finer grain sizes. They were further soaked in water for three days. This was to ensure proper dissolution and removal of soluble alkalis and dead organic matter. The presence of alkali oxide retard mullite formation which affects the refractoriness and strength of clay brick.

The dissolved clay was then filtered through a 0.425 mm mesh and sieved with 0.18 mm sieve to get rid of unwanted particles and to obtain finer particles respectively. The excess water content of the filtrate was decanted off while the clay slip was sun dried and then oven dried at 100^0C. The processed mixture of Nguzu- Amaiyi clays were further ground, sieved and blended at ratio 40:60 to produce moulding samples.

Combination of groundnut shell sawdust and rice husk which constitute the local additives were mixed at equal ratio and introduced into the blended clay at 5%, 10%, 15% and 20% formulations to develop different test samples. The composite mixtures were moulded to different shapes suitable for their respective standard tests and further fired to 1200^0C.

2.2.3 Micro Structural Examination

The internal structure of the clay brick was observed using scanning electron microscope. This examination was to reveal pore sizes and their distributions in the clay brick which affect most of the properties. The test samples were ground using abrasive papers of different sizes, polished with different sizes of diamond polishing paste ranging from 6 – 0.25µm. The scanning electron microscope was used to scan a focused beam of electrons across the specimen. The signal from the electron beam interaction with the specimen revealed the image. Computer based image processing was used to make the image visible.

2.2.4 Determination of the Refractory Properties of the Materials

The tests for the determination of the properties were done in line with ASTM standard experimental procedures.

Determination of Linear Shrinkage

ASTM C-326 standard for testing linear shrinkage of refractory material was used for this test. 50 mm mark was made on the surface of the dried sample (L_D). The same dimension was measured when the sample was fired to get the fired length L_F. The dry-fired shrinkage was calculated as the linear shrinkage represented as:

$$\left(\frac{L_D-L_F}{L_D}\right) \text{x } 100 \qquad 1$$

Determination of Bulk Density and Apparent Porosity

ASTM C 20-80a, standard test method for testing apparent porosity, water absorption and bulk density was used to determine these two properties. The long rectangular shaped test sample measuring 9.5cm length, 2cm width and

5cm height was used for these two experiments. The weights of the dry samples in air were taken as (W_1). The samples were transferred into a vessel of boiling water for 30 minutes after which the boiling was discontinued. The specimen samples were allowed to cool to room temperature in the vessel of water for four hours. They were tied to a string on a spiral balance suspended in a beaker of water, to get the suspended weight (W_2). The specimens were removed from the water and gently cleaned and reweighed in air to get the soaked weight (W_3).

From the data, the physical parameters were calculated using the formula:

$$\text{Bulk density} = \left(\frac{\text{Weight in Air } (W_1)}{\text{Soaked Weight}(W_3) - \text{Suspended Weight } (W_2)}\right) \qquad 2$$

$$\text{Apparent porosity} = \left(\frac{\text{Soaked Weight}(W_3) - \text{Weight in Air } (W_1)}{\text{Soaked Weight}(W_3) - \text{Suspended Weight } (W_2)}\right) \text{x } 100 \qquad 3$$

Determination of Refractoriness

The refractoriness or softening point was determined using the method of pyrometric cone equivalence (PCE) in accordance with ASTM C24-79 standard. 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 until the tips of the test cones had bent over the level with the base. 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.

Determination of Thermal Shock (Spalling) Resistance

The thermal shock resistance was determined by prism spalling test method according to ASTM C-484 standard. The spalling resistance was measured by the number of thermal cycles of heating and cooling withstood before cracking. The test pieces were 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 using a pair of tongs after 10 minutes and cooled in air for 10 minutes, and then observed for cracks. The number of complete thermal cycles withstood in the absence of crack (or fracture) was recorded as the thermal shock (spalling) resistance.

Determination of Thermal Conductivity

The test was conducted using heat conduction equipment. Circular test specimen 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.

Input power of 5 watts was selected and maintained for 30 minutes until steady state conditions were achieved. The temperatures (T) at all the sensor points were recorded. The thermal conductivity was calculated using Fourier's law as:

$$K = \frac{Qx}{A.dT} \qquad 4$$

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.

Determination of Modulus of Rupture

This test was done with 3 point bend tester in accordance with ASTM C-648 standard for testing modulus of rupture. The electrical transversal strength testing machine was used to determine the breaking load, P (kg). The distance between supports of the transversal machine was measured and noted as L (mm). The height H (mm) and the width B (mm) of the broken pieces were determined. The modulus of rupture was then calculated as:

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

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 (mm).

3.0 RESULTS AND DISCUSSION

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

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

Source: Chima *et al*. (2017)

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	

Source: Chima *et al*. (2017)

Table 3: Chemical analysis of saw dust

Oxide	**SiO_2**	**P_2O_5**	**SO_3**	**K_2O**	**CaO**	**TiO_2**	**MnO**
Composition	17.2	4.7	3.0	19.2	47.4	1.0	0.65
Oxide	**Fe_2O_3**	**NiO**	**CuO**	**ZnO**	**BaO**	**Eu_2O_3**	**Re_2O_7**
Composition	5.73	0.024	0.44	0.2	0.13	0.09	0.4

Table 4: 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 5: 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_2O_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 oxide composition of the agro additives showed that the additives have oxides like alumina and phosphorus oxide which improve the refractory properties of clay brick. The alumina content of 7.6% in groundnut shell increased that of the composite clay. This enhanced the refractoriness and strength of the composite material. Also, phosphorus oxide was found in all the three additives. This oxide forms alumino phosphate bond in the refractory material which had non wetting effect against molten metal and also increases bond strength. Beside these, it increases the resistance of refractory lining to crack and Carbon II oxide attack - a byproduct of the furnace activity which causes crack of the furnace lining.

3.2 Micro structural examination results

The micro structural images shown by SEM indicated a more homogeneous structure with small pore sizes in Nguzu-Amaiyi blended clay. However, in the composite clay structure, the pore sizes were relatively larger with wider distribution that showed a heterogeneous structure. The pores were indicated by dark spots in the structures. The increased pore sizes gave rise to increase in apparent porosity and improvement in the insulating property observed in the result. The large pores in the composite brick also assisted to impede crack propagation which improved the thermal shock resistance property.

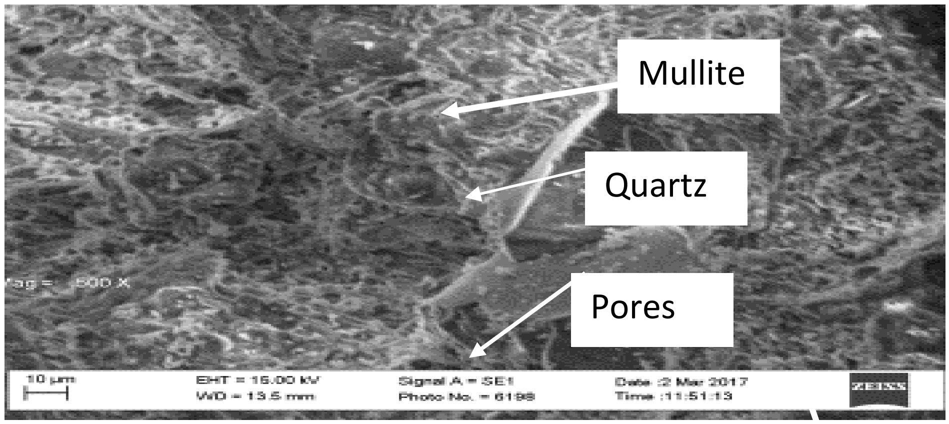

Plate 1: SEM for blended Nguzu – Amaiyi clay

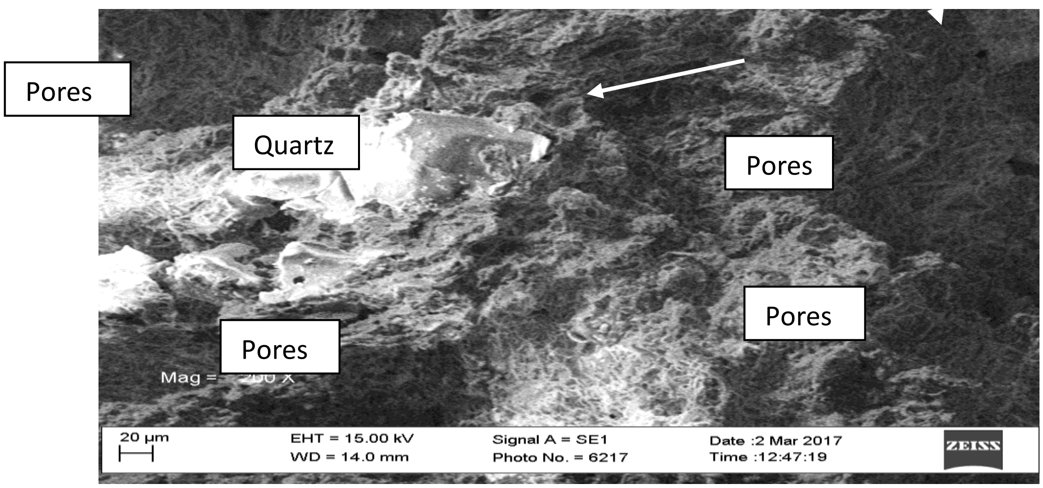

Plate 2: SEM for composite clay with groundnut shell sawdust and rice husk additive

3.3 Refractory properties

The refractory properties of both the blended clay (control sample), composite clay with combination of the admixture of agro additives were shown in Table 6. Comparison of the results obtained with the specified standard was also indicated.

Table 6: Comparison of Refractory Properties of the Blended Clay, Composite Clay with admixture of three agro additives and Specified Standard

Property	Blended clay	Composite clay with different composition of the additive				Specified standard
		5%	10%	15%	20%	
Linear Shrinkage (%)	6.59	5.49	10.42	10.64	10.64	4 – 10
Bulk density g/cm^3	1.77	1.73	1.48	1.25	1.32	1.71 – 2.1
Apparent Porosity (%)	15.38	36.36	43.18	54.17	54.69	25-50-Insulating brick 2-30-Dense brick
Modulus of Rupture (N/mm^2)	32.7	34.3	32.8	31.6	26.1	
Thermal Shock Resistance (cycles)	30	31	31	32	31	25 – 30
Thermal Conductivity (W/mm^{0}C)	0.00318			0.00294		
Refractoriness (^{0}C)	1550	1670	1670	1670	1670	1500 – 1700

Source: Grimshaw (1971)

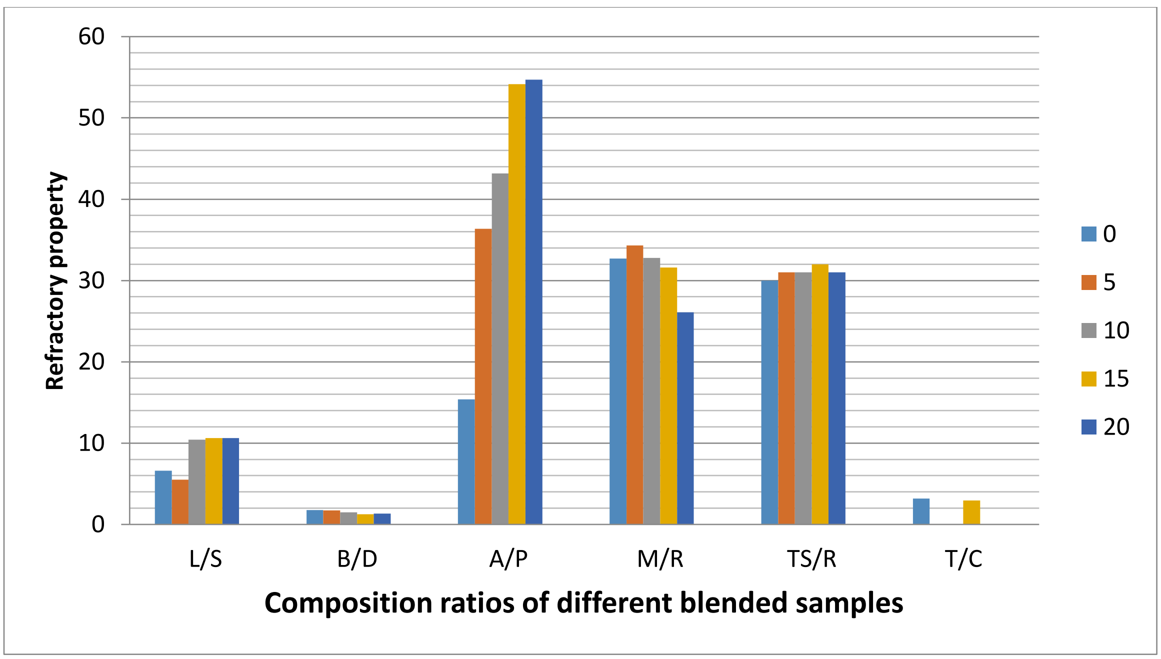

Figure. 1: Effect of composition of the additives on the enhancement of the refractory properties of composite clay samples

Linear shrinkage

The linear shrinkage of the composite clay decreased from 6.59% to 5.49% which indicated 16.7% enhancement of the property. However, it was noted that increase in the composition of the additive yielded higher value of shrinkage. This was caused by the rearrangement of the particles of the composite material during firing which led to the formation a more compact structure. Though, the shrinkage values increased with increase in the percentage composition of the additive, it was observed that the values were not too far from the recommended value of (4-10%) as reported by Omowumi (2001).

Apparent Porosity and Bulk Density

The apparent porosity was found to depend on the composition of the additive according to Fatai (2012) and Manukaji (2013). It was observed that the value rose to the recommended range of insulating brick of (25-50%). This shows that the bricks have been converted from conventional dense brick to insulating type with the additive. The same results correlate with values obtained in bulk density test. This proves that the larger pores in the composite brick gave rise to increase in porosity, decrease in bulk density, and better insulating property. The larger pores were caused by the burning off of the combustible materials which left pores in the structure of the clay brick. The values of bulk density decrease from 1.77 to 1.32g/cm^3. The decrease is associated with increase in the pore size. Hence, it was noted that apparent porosity, bulk density and other associated properties are interrelated. These properties were significantly affected by the composition of the additive mixture.

Modulus of Rupture

The results obtained in the test for modulus of rupture show that the transverse strength improved from 32.7N/mm^2 to 34.3N/mm^2 and 32.8N/mm^2 at (5 and 10%) formulations respectively. However at further increase in the composition of the additive, the strength decreased. Generally, in clay based material, strength decreases with increase in porosity. The observed decrease in the property is due to increase in porosity obtained with increase in composition of the additive. Therefore, the amount of the additive must be controlled to ensure that the strength is not impaired. The enhancement of 4.7% achieved in modulus of rupture at 5% formulation is in line with the conclusion of Fatai (2012) which stated that (10-15%) composition should not be exceeded when strength is desirable. It is also in line with the finding of Manukaji (2013) which stated that the composition could be varied to obtain desirable properties.

Thermal Shock Resistance

From the results shown in the spalling resistance test, it was found that the clay on its own has good thermal shock resistance property of 30 cycles. However, the composite brick showed better thermal shock resistance behavior of 31 and 32 cycles. The enhancement is traced to oxide composition of the additives which contributed to improve the property. Besides, the larger pores developed in the composite material impede crack propagation. Hence, yields improvement in the thermal shock resistance property.

Refractoriness

The refractoriness of the composite clay material was found to increase from 1550 to 1670^{0}C. The increase is an indication of the increase in alumina content from groundnut shell additive. This also increased the alumina/silica ratio which has capacity of increasing the refractoriness. It was also found that the refractoriness of the composite clay could match those used for furnace lining in ferrous material.

Thermal Conductivity

The insulating behavior of the material improved from 0.00318 W/mm^{0}C to 0.00294 W/mm^{0}C. The reason for the observed decrease is that the additive left larger voids and pores in the clay structure upon burning during firing. The pores decreased the concentration of thermal conduction pathways. More also, when the proportion of air inside the brick body is higher, the thermal insulating capacity of the material becomes better. This is because air is a good insulator in comparison to the solid material.

4.0 CONCLUSION

Based on the results and findings obtained in this study, it was concluded that;

The use of combined multiple additives like groundnut shell, rice husk and saw dust is more effective in the enhancement of refractory properties unlike the use of single ones which were used by previous researchers.

The refractory properties depend on the percentage composition of the additive.

Better and excellent properties of composite brick are achieved with composition of the additive ranging from 5 – 10%. Some properties could be impaired when this range of composition is exceeded.

Conventional dense brick could be converted to insulating brick of recommended standard without compromise to other properties with the use of this combination of additive at the specified composition.

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