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# Study of effects of Groundnut shell and Grog additives on the refractory Properties of Isiagu Clay

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**Abstract:** This research aimed at finding out the impacts of groundnut shell and grog additives on the refractory properties of Isiagu Clay. The raw materials were collected locally, processed and analysed using Scanning Electron Microscope/Energy Dispersive X-ray (SEM/EDX). The additives were added to the raw clay in the ratio of 2.5,5.0,7.5 and 10 wt% and fired at 900°C and 1100°C, respectively. The refractory properties measured were; linear shrinkage, apparent porosity and bulk density. The result of the SEM/EDX analyses showed that the clay is a fire clay since it contains 61.68% Alumina ($Al_2O_3$) and 34.97% Silica ($SiO_2$) while the grog additive contains 65.5% $Al_2O_3$ and 34.33% $SiO_2$, respectively. The groundnut shell itself contains 1.55% $Al_2O_3$ and 2.91% $SiO_2$, respectively. Groundnut shell also contains 75.1% $Nb_2O_5$ which imparts good heat conductivity to samples containing it. SEM/EDX morphological analysis results showed that both grog and groundnut shells contain tiny to coarse particles, however, grog has greater strength for refractory application than groundnut shell due to its high alumina and silica contents. For groundnut shells, linear shrinkage increased from 7.477% to 7.881% at 900°C while grog decreased from 3.383 to 3.509 % at 900°C. The decrement in linear shrinkage in grog-containing samples is due to the high amount of $Al_2O_3$ and $SiO_2$ present in the grog which makes it desirable for refractory application. The apparent porosity measurement showed that the samples containing groundnut shell are suitable for insulating application since it contains a high amount of organic matter. ANOVA results confirmed that temperature and additive percentages were not significant for linear shrinkage measurement.

**Keywords:** Groundnut shell, grog, Clay, fireclay, insulating, and refractory

## 1. Introduction

Heat transfer is paramount in the process industry of which heating, boiling and roasting of metal ores are classical examples [1]. In these processes, the aim is to achieve efficient heat transfer in process equipment such as ovens, furnaces and boilers by lining the interior with high-temperature materials [2]. In Nigeria, seven clay deposits exist which are of good refractory properties and hence good feedstock for the production of high-temperature bricks at scale [3, 4]. According to Olalere,*et al* [5] and Oke,*et al* [4], the Nigerian government is set to rehabilitate her obsolete steel industries in the Kogi and Delta states which would require 68,000 tons of refractories which is only a small fraction of the refractory demands of other energy industries totalling 300,000 tons in the year 2000. Sadly, about $229 million is spent annually to cushion the unavailability of refractory materials for use in kilns and furnaces [6]. Although, some efforts have been made to meet this need, yet, there is a need to provide these high-temperature materials with improved mechanical and thermal properties. This must be done since weakness is usually due to chemical attacks and mechanical abrasions in the process of utilizing them, hence decreasing their life span to as low as 1 to 2 years [7]. Local clays can be used together with additives to improve the various properties of the refractory clay-like; thermal shock resistance, cold crushing strength, bulk density, porosity etc [8, 9]. Some researchers have made efforts to improve the quality of high-temperature materials for furnaces, boilers and oven linings. Improvement of thermal performance of environmental friendly bricks was studied by Kazmi, *et al* [10] in which they used agro-waste sugarcane bagasse ash (SBA) and Rice husk ash (RHA) as additives. This additive was incorporated in various dosages (5%, 10% and 15%) by the weight of clay. After various tests were carried out it was discovered that refractory brick incorporating agro-waste had lightweight and low compressive strength. In this present study, grog from clay and groundnut shell were used to improve the refractory properties of Isiagu clay. The Analysis of Variance (ANOVA) was achieved using Excel 2013 and the degree of significance of the factors was determined.

## 2. Materials and Methods

### 2.1 Materials

The raw clay sample was air-dried under shade to allow removal of water and other volatile matter and afterwards crushed to a small grain size in a mortar to increase the surface area of the clay sample [3, 11]. To get rid of unwanted materials, the slurry was passed through a 0.425mm mesh sieve [11]. The clay slip obtained was sun-dried for two days and then oven-dried at 100oC and fired at 600oC to remove carbonate and other organic matter [11]. Afterwards, some reasonable part of the clay was fired up to 1000oC for 6 hours to produce grog which was an additive just as did [12]. The processed clay was pulverized and sieved through a 425µm sieve which is recommended by ASTM [13]. The clay sample was mixed proportionally with the additives and

moulded into different shapes namely; rectangular, circular, and conical for the respective tests to be carried out. The morphological analysis was carried out using the Zeiss EVO LS 10. A beam of electrons is produced at the top of the microscope using an electron gun. The beam was focused toward the sample via the magnetic field which ejected electrons and x-rays as they contacted. The detector read the signals and converted them to the final image.

### 2.2 Mixing and Molding

The clay sample was combined individually with the additives namely; grog, groundnut shell, melon shell, palm kernel shell, sawdust and chicken droppings (2.5%wt, 5.0wt%, 7.5wt%, and 10%wt). Following the addition of water, a sticky mass was formed and with the aid of the mould, the samples were formed for different tests to be conducted [11]. The moulds were rubbed with lubricating oil for easy release of the test pieces from the mould after it dries [11]. The formulation of the samples for analysis is shown in Table 1.

**Table 1. Design of experiment for the production of refractory**

| Input variables | Ranges |
|---|---|
| Temperature ($^0$C) | 900 – 1100 |
| Additive percentage (wt%) | 2.5 – 10 |

### 2.3 Measurement and Analysis

**a. Linear Shrinkage**

This evaluates the changes in the length of brick samples when heated. For this purpose, the brick samples will be made in cuboidal shapes. The formula is as given in the work of Adeosun, *et al* [14]. This is calculated via the original length (Lo) before drying and the final length (Lf) after firing to a certain temperature

$$\textbf{Percentage Total Shrinkage} = \frac{Lo - Lf}{Lo} \; x\, 100 \quad (2.1)$$

**b. Apparent Porosity**

Apparent porosity is the mathematical relationship between the volume of the void and the total volume of the material as given by ASTM-C20-00 [15]. This will be determined using the boiling method as was used by Adeniyi, *et al* [16]. A moulded brick specimen (rectangular) will be used. The brick will be oven-dried at 110ºC to constant weight (D). After which it will be transferred to a beaker and boiled with distilled water for 1.5hrs to assist in releasing the trapped air. It will be soaked and the saturated weight free of water (W) will be obtained. Finally, the specimen will be suspended in the water by a rope tied to a spring balance and obtain the suspended weight (S) when it is completely immersed in water.

$$\textbf{ApparentPorosity} = \%Pa = \frac{W-D}{W-S} \; x\, 100 \quad (2.2)$$

Where:

D = Constant Weight of the dry Sample

S= Weight of the sample suspended in the water

W= Weight of sample in the air including the moisture in its open pores (saturated weight)

**c. Bulk Density**

The Bulk Density (BD) of a refractory shows the degree to which it was fired and hence the level of densification will be determined by dividing the mass of sample by the outside volume and multiplying with the density of water as recommended by ASTM-C20-00 [15].

$$Bulk\ Density = \frac{Dry\ weight\ of\ sample * Density\ of\ water}{saturated\ weight\ in\ air - suspended\ weight\ in\ water} \quad (2.3)$$

**d. Refractoriness**

This is a thermal property of refractory brick materials that determines the degree of temperature the material can withstand. This will be achieved by making conical brick samples and putting them alongside pyrometric sega cones (standard cones with definite deformation temperature) into the kiln and firing to a very high temperature of about 1650ºC. After firing, the cones will be examined and the one that bent to the same extent as the test sample is said to have the equivalent temperature of deformation. Alternative, Shuen's formula can be used to roughly estimate the refractoriness of a clay sample [8].

$$Refractoriness\ (K) = \frac{360 + Al_2O_3 - RO}{0.228} \quad (2.4)$$

Where RO is all oxides minus $Al_2O_3$ and $SiO_2$

***RESULTS AND DISCUSSION***

***3.1 Characterization of Clay and Additives***

The results obtained from the characterization of clay and additive samples using SEM/EDX was carried out at an Electron High Tension of 10kV at different wavelengths of 100µm and 20µm and the result are shown in Table 2. SEM/EDX results for the weight composition of elements show that the clay contains principally, aluminium and silicon elements with a relatively high amount of carbon [17]. The result presented in Table 2 shows that the sawdust additive contains only carbon metal, this is since sawdust is very rich in organic matter compared with coconut shell and rice husk [18]. Grog on the other hand is very high in silicon as expected because it was gotten from a clay sample fired to 900ºC where all the carbonaceous materials are removed [12]. The result in Table 3 below was derived from the results in Table 2. The oxide forms of the elements present were obtained and the result showed that clay and grog had almost the same composition by weight of both $Al_2O_3$ and $SiO_2$. Since both samples contain a high amount of $Al_2O_3$ and $SiO_2$, they can be referred to as kaolinites, hence suitable for the production of refractory bricks (Chikwelu, et al, 2018; Ajala & Badarulzaman, 2016a).

**Table 2: SEM/EDX results for the percentage by weight composition of elements in clay and all additives**

| Weight composition | MgO | $Al_2O_3$ | $P_2O_5$ | $SiO_2$ | $K_2O$ | $Nb_2O_5$ |
|---|---|---|---|---|---|---|
| Clay | 0.27 | 34.97 | | 61.68 | 3.08 | |
| Grog | 0.17 | 34.33 | | 65.5 | | |
| Groundnut shell | | 5.40 | | 5.75 | 13.15 | 75.71 |

**Table 3: SEM/EDX results for the percentage by weight composition of oxides in the clay and all additives**

| Weight composition | Mg | Al | Si | K | Nb | C | O | N |
|---|---|---|---|---|---|---|---|---|
| Clay | 0.3 | 17.37 | 54.01 | 2.4 | | 24.44 | 1.47 | |
| Grog | 0.25 | 22.68 | 76.28 | | | | 0.78 | |
| Groundnut shell | | 1.55 | 2.91 | 5.92 | 28.11 | 50.39 | 1.64 | 9.49 |

**3.2 SEM/EDX Morphological analysis result for Clay, groundnut and grog**

SEM/EDX morphological analysis results for various samples are presented in fig 2. The analysis was carried out at an Electron High Tension of 10kV at different wavelengths of 100µm and 20µm, respectively. The SEM/EDX result for the clay and the grog samples indicates the presence of tiny to large coarse particles (fig2a & b), however, Aboutaleb,et al [19] obtained the same result but with refractory brick waste (RBW). Also, the groundnut shell sample showed the presence of tiny to large coarse particles in form of a sheet l (fig 2c).

**3.3 Refractory Analysis Results**

**a. Linear Shrinkage**

The results of the linear shrinkage measurement are shown in Fig. 3 below. The results show that while grog additive decreased progressively, groundnut shell increased for both 9000C and 11000C and was within the acceptable limit of 7 to 10% [11].

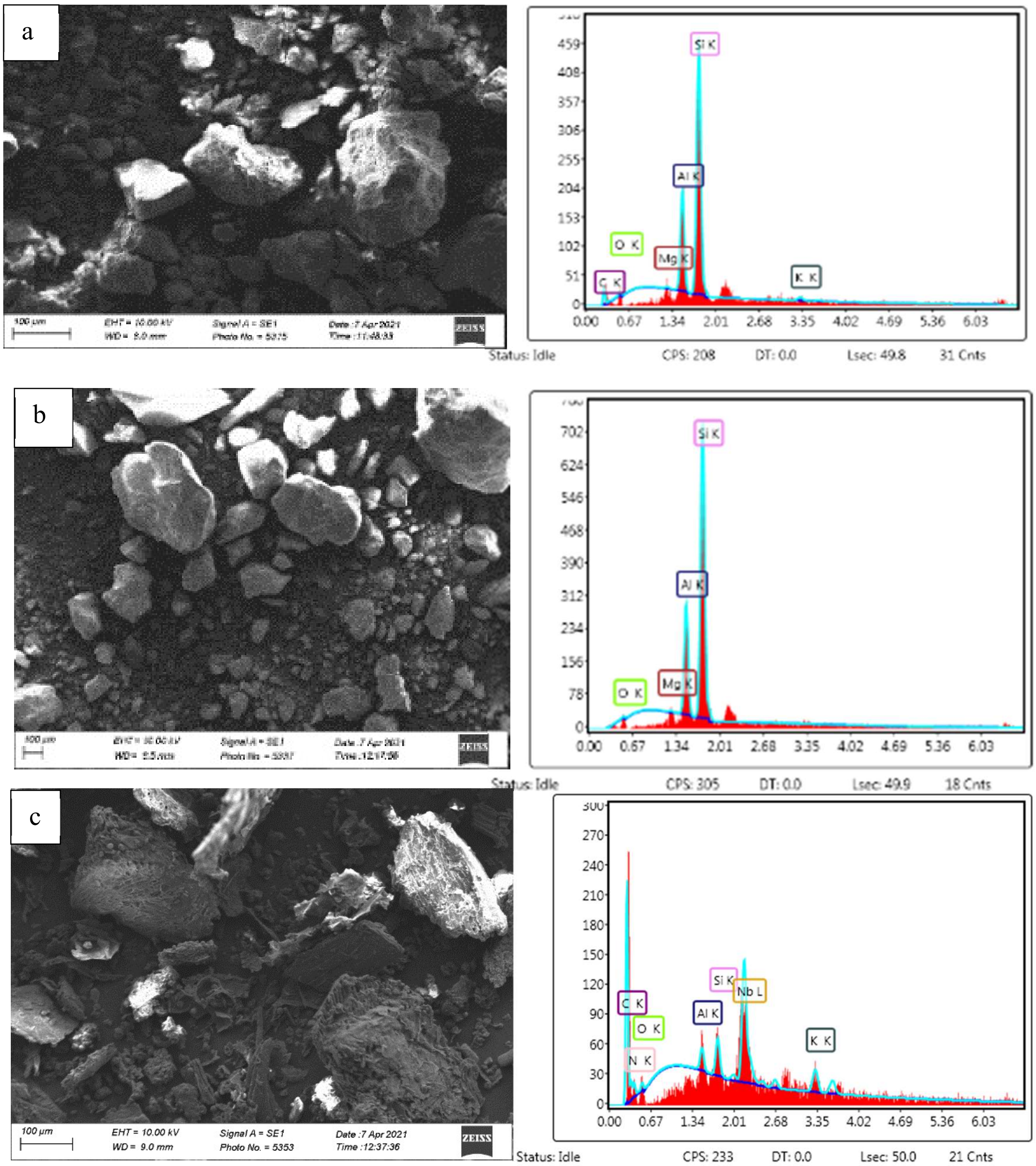
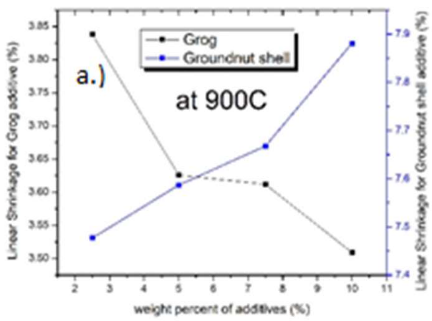



Fig 2. SEM/EDX results at EHT = 10.00kv and wavelength of 100µm for a.) Isiagu Clay sample b.) Grog and c.) Groundnut Shell

However, the variation of grog was the opposite of this trend and the least among the samples. This agrees with the SEM/EDX results which showed that grog contains more silicon than any other element. The result is shown in Fig 3. shows that grog and groundnut shells have comparable linear shrinkage at both 900oC and 11000C, that is, opposite trends were recorded for the additive percentage.



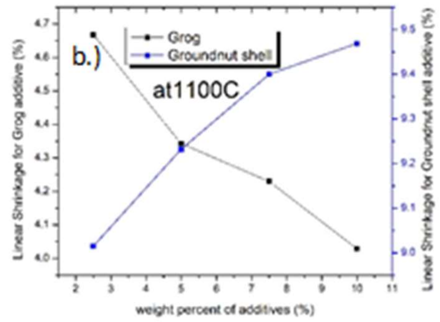



Fig 3. The plot of Linear Shrinkage for Groundnut shell and Grog at a.) 900°C and b.) 1100°C

**a. Apparent Porosity**

The apparent porosity increased steadily with the addition of groundnut shell additive but decreased with grog additives at both 900°C and 1100°C (Fig 4) [20]. Based on the international standard of 2 to 30%, the apparent porosity of the groundnut shell was within the specified limit [11].

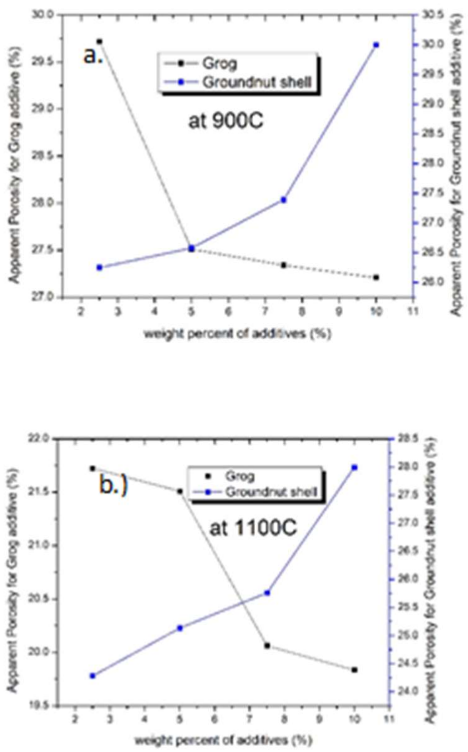



Fig 4. The plot of Apparent porosity for Sawdust and Grog at a.) 900°C and b.) 1100°C

**b. Bulk Density**

There was a bulk density decrement according to Fig. 5 following previous works [11, 21, 22]. A handful of the samples containing grog at 1100°C were within the internationally

acceptable range of 1.7 – 2.1g/cm$^3$ while the samples containing groundnut shell at 900°C and 1100°C were within the specified standard [11].

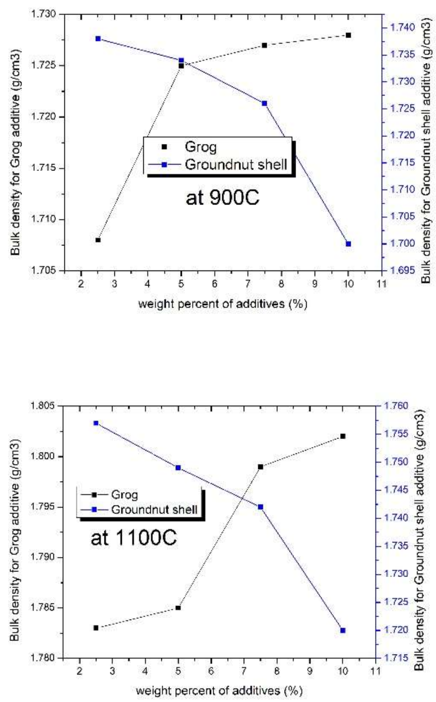



Fig 5. The plot of Bulk Density for Groundnut shell and Grog at a.) 900°C and b.) 1100°C

**d. Refractoriness**

This was determined using equation 2.4. The calculation indicated that Isiagu clay is fireclay and hence can withstand temperatures as high as 1447oC. This is very close to that recorded in the work of Harith and Hani [23] where a sintering temperature of 1500oC was observed for 70% kaolin and 30% metakaolin and above 1325oC recorded by Aşkın,et al [24] for Cordierite with waste magnesite.

### 3.4 ANOVA

Results for Grog and Sawdust Additives

Analysis of variance was done for all the samples with various additives and all the properties measured.

**Grog Only additive**

Analysis of variance was carried out for all samples containing grog additives. The result of two-way ANOVA without replication is presented in Table 4 to Table 6. The result shows that the effect of percentage additive for all properties was not significant (P>0.05), while the temperature was not significant only for linear shrinkage measurement [25, 26]. This is because organic matters have been removed from the grog by firing and hence increase in grog per cent had no impact on the properties measured.

Table 4: Linear Shrinkage

ANOVA

| *Source of Variation* | *SS* | *df* | *MS* | *F* | *P-value* | *F crit* | *Remark* |
|---|---|---|---|---|---|---|---|
| Temperature | 14.61114 | 2 | 7.305569 | 1.86858 | 0.233968 | 5.143253 | **Not significant** |
| Per cent Additiv | 8.061763 | 3 | 2.687254 | 0.687332 | 0.591915 | 4.757063 | **Not Significant** |
| Error | 23.45814 | 6 | 3.90969 | | | | |
| Total | 46.13104 | 11 | | | | | |

Table 5: Apparent Porosity

ANOVA

| *Source of Variation* | *SS* | *df* | *MS* | *F* | *P-value* | *F crit* | *Remark* |
|---|---|---|---|---|---|---|---|
| Temperature | 967.1841045 | 2 | 483.6 | 85.06908 | 3.95E-05 | 5.143253 | **Significant** |
| Percentage Additives | 2.52790825 | 3 | 0.843 | 0.148229 | 0.927107 | 4.757063 | **Not Significant** |
| Error | 34.1081895 | 6 | 5.685 | | | | |
| Total | 1003.820202 | 11 | | | | | |

Table 6: Bulk Density

ANOVA

| *Source of Variation* | *SS* | *df* | *MS* | *F* | *P-value* | *F crit* | *Remark* |
|---|---|---|---|---|---|---|---|
| Temperature | 53.83901 | 2 | 26.9195 | 7.794196 | 0.021468 | 5.14325285 | **Significant** |
| Per cent Additive | 10.52781 | 3 | 3.509272 | 1.016064 | 0.448761 | 4.757062663 | **Not Significant** |
| Error | 20.72273 | 6 | 3.453788 | | | | |
| Total | 85.08955 | 11 | | | | | |

This implies that the firing had little or no impact on the weight of the samples from 2.5wt% to 10wt.

**Groundnut shell only**

Analysis of variance was carried out for all samples containing groundnut shell additives. The result of two-way ANOVA without replication is presented in Table 7 to Table 9. The result shows that the effect of temperature and percentage additive for all properties were significant ($P<0.05$), except for the effect of percentage additive for bulk density [25, 26].

Table 7: Linear Shrinkage

| *Source of Variation* | *SS* | *df* | *MS* | *F* | *P-value* | *F crit* | *Remark* |
|---|---|---|---|---|---|---|---|
| Temperature | 207.5613 | 2 | 103.7807 | 68.81258 | 7.29E-05 | 5.14325285 | **Significant** |
| Per cent Additive | 30.4618 | 3 | 10.15393 | 6.732645 | 0.023907 | 4.757062663 | **Significant** |
| Error | 9.048983 | 6 | 1.508164 | | | | |
| Total | 247.0721 | 11 | | | | | |

Table 8: Apparent Porosity

| *Source of Variation* | *SS* | *Df* | *MS* | *F* | *P-value* | *F crit* | *Remark* |
|---|---|---|---|---|---|---|---|
| Temperature | 1118.426 | 2 | 559.2128 | 519.3603 | 1.89E-07 | 5.143253 | **Significant** |
| Per cent Additive | 40.99877 | 3 | 13.66626 | 12.69232 | 0.005228 | 4.757063 | **Significant** |
| Error | 6.460403 | 6 | 1.076734 | | | | |
| Total | 1165.885 | 11 | | | | | |

Table 9: Bulk Density

| *Source of Variation* | *SS* | *df* | *MS* | *F* | *P-value* | *F crit* | *Remark* |
|---|---|---|---|---|---|---|---|
| Temperature | 54.40336 | 2 | 27.20168 | 7.759389 | 0.021677 | 5.143253 | **Significant** |
| Per cent Additive | 10.21775 | 3 | 3.405917 | 0.971551 | 0.465523 | 4.757063 | **Not Significant** |
| Error | 21.03388 | 6 | 3.505647 | | | | |
| Total | 85.65499 | 11 | | | | | |

## 4. CONCLUSION

The effect of grog and groundnut shells on the refractory properties of Isiagu clay was studied. The ANOVA was also carried out using Excel 2013. The SEM/EDX result showed that groundnut shell additive contains 50% carbon metal and implies that samples containing groundnut shell additive can be used for insulating firebrick production. In the same vein, the Grog additive showed high content of $Al_2O_3$ and $SiO_2$ which makes it suitable for refractory applications. The SEM/EDX morphological results further confirmed the class of application of the two additives used in this work. Grog and groundnut shell both contains tiny to coarse particles which indicates their strength, however, grog has an advantage over groundnut shell because of the higher $Al_2O_3$ and $SiO_2$ content. Samples containing grog showed a reverse trend in all the properties measured due to the high content of Al2O3 and SiO2 and further confirming its suitability for a refractory application. ANOVA results confirmed that for grog and groundnut shell additives, the additive percentage was not significant for bulk density which implies that the bulk density is Acknowledgement

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