Investigative Study of effects of *Gallus domesticus guano shell ,* *Cucumeropsis mannii shell* and Elaeis *Guineensis shell* additives on the refractory Properties of Isiagu Clay

Abstract

This research investigated the effects of chicken dropping, melon shell and palm kernel 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 $900^{o}C$ and $1100^{o}C$, 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 fireclay since it contains 61.68% Al_2O_3 and 34.97% SiO_2 while chicken dropping, melon shell, and palm kernel shell contained 37.41% SiO_2, 84.62% P_2O_5, and 48.31% Nb_2O_5, respectively. Chicken dropping was responsible for strength, melon for toughness and palm kernel shell for conductivity. SEM/EDX morphological results showed that the clay sample contains tiny to coarse particles. The bulk density measurement confirmed the strength and toughness of melon shell, the only additive which met the international standard for refractory application. XRD analysis results showed the presence of two crystalline phases at 21.4^{o} and 22.4^{o} namely, quartz and calcite in the additives used which are both responsible for strength in the refractory brick. Sudden variation in properties was observed for chicken droppings due to the presence of a reasonable amount of P_2O_5. From the study, melon shell and palm kernel shell are suitable for refractory brick production and further studies are recommended for chicken dropping. ANOVA results confirmed that for melon shell and apparent porosity only temperature was significant.

Keywords: Melon shell, Palm kernel shell, Clay, fireclay, insulating, and refractory

INTRODUCTION

Heat transfer is essential in the process industry of which heating, boiling and roasting operations are good examples (Ogunsemi *et al.*, 2018). In these operations, the objective is to achieve energy conservation in process equipment such as ovens, furnaces and boilers by lining the inner sections with high-temperature materials (Hussein, 2019). Nigeria has a strong refractory production capacity since there are seven clay deposits that exist which are of good refractory properties (Amkpa, 2017; Oke *et al.*, 2015). According to the works of Olalere *et al.* (2019) and Oke *et al.* (2015), the Nigerian government is ready to renovate her moribund steel industries in Kogi, 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. It is sad to note that Nigeria spends about $229 million annually to arrest the shortage of refractory materials for use in the kilns and furnaces (Esezobor *et al.*, 2015). Until now, efforts have been made to address this imbalance, yet, there is a need for refractories with improved mechanical and thermal properties. This is important since chemical attacks and mechanical abrasions in the process of utilizing them, decrease their life span to as low as 1 to 2 years (Antonovič *et al.*, 2017). Some researchers have used local clays together with additives to improve the various properties of the refractory clay namely; thermal shock resistance, cold crushing strength, bulk density,

porosity etc (Ahmad *et al.*, 2017; Iyasara *et al.*, 2016). Some researchers have made efforts to improve the quality of high-temperature materials for furnaces, boilers and oven linings. The relationship between mechanical properties and thermal conductivity of an insulating fire brick using petroleum coal dust as an additive was studied by (Rahman *et al.*, 2015). The additive was incorporated in the range of 5wt% to 20w%. It was discovered that the finer coal dust particles of size less than 20µm performed best in terms of the measured mechanical and thermal properties. This was attributed to the fineness of the coal dust particles. Bulk density decreased from 1.48 to 1.1 while porosity increased from 46.50 to 58.48% and cold crushing strength decrease from 22.7MPa to 5.5MPa. Also, Amkpa *et al.* (2017) worked on Barkin-Ladi clay and determined its suitability for a refractory application. It was discovered that it is a good feedstock for refractory application having a thermal conductivity of 0.03W/mK, a specific heat capacity of $0.07J/g^0C$, refractoriness of 1665^0C, thermal shock resistance of 24 cycles and high energy absorption. In this present study, chicken dropping (*Gallus domesticus guano*), melon shell (*Cucumeropsis mannii shell)* and palm kernel shell (*Elaeis Guineensis shell*) were used to improve the refractory properties of Isiagu clay. The Analysis of Variance (ANOVA) was done and the degree of significance of the factors were determined using *Excel 2013*.

2 MATERIALS AND METHODS
2.1 Sourcing and Characterization
The raw fireclay was collected from Isiagu Awka South Local Government Area, Anambra State following the standard specified by the American Society for Testing and Materials (ASTM) code ASTM-D4700-15 (2015). Awka city is located directly north of Portharcourt, Rivers State, Nigeria. The location of Isiagu as found by google earth lies on Longitude, $6^o10^{'}54^{''}$ N, Latitude, $7^o06^{'}54^{''}$ E and Altitude, 6.94Km (Fig 1). Definite fire clay of precisely grey to light brown colour was collected. The colour may be due to the high amount of Fe_2O_3 in it. All the additives namely chicken droppings, melon shell and palm kernel shell, were sourced in Awka. All the raw materials used were characterized using Scanning Electron Microscope /Energy Dispersive X-ray (SEM/EDX).
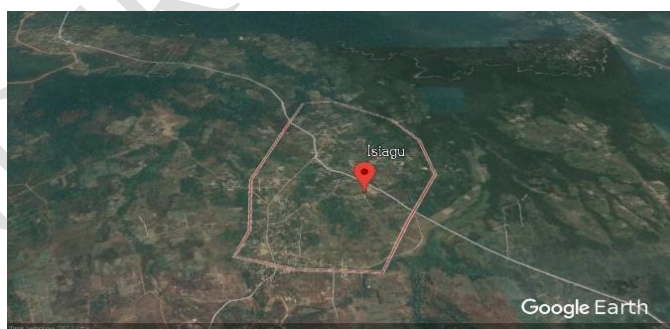

Fig 1. Google Earth Picture of Isiagu Awka South Local Government area

2.2 Material Processing and Moulding of Samples
The raw clay sample was air-dried under shade to allow removal of water and other volatile matter and afterwards crushed to small grain size in a mortar to increase the surface area of the clay sample (Amkpa, 2017; Chima *et al.*, 2017). To get rid of unwanted particles and plant materials, the slurry was filtered through a 0.425mm mesh sieve (Chima *et al.*, 2017). The clay slip obtained was sun-dried for two days and then oven-dried at 100^oC and fired at 600^oC

to remove carbonate and other organic matters (Chima *et al.*, 2017). The processed clay was pulverized and sieved through a 425μm sieve which is recommended by ASTM (Ajala & Badarulzaman, 2016). 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.

2.3 Mixing and Moulding

The clay sample was combined individually with the additives namely; chicken droppings, melon shell and palm kernel shell (2.5%wt, 5.0wt%, 7.5wt%, and 10%wt). Due to the addition of water, a sticky mass was formed and with the aid of the mould, the samples were formed into rectangular and cylindrical shapes for different tests to be conducted (Chima *et al.*, 2017). The moulds were rubbed with lubricating oil for easy release of the test pieces from the mould when they dry (Chima *et al.*, 2017). 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 linear changes that occur in 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.* (2016). This is calculated based on the original length (Lo) before drying and the final length (Lf) after firing to a certain temperature

$$Percentage\ Total\ Shrinkage = \frac{Lo-Lf}{Lo}\ x\ 100 \qquad (2.1)$$

b. Apparent Porosity

Apparent porosity is the percentage relationship between the volume of the open space and the total volume of the material as given by ASTM-C20-00 (2015). This will be determined using the boiling method as was used by Adeniyi *et al.* (2018). A moulded brick specimen (rectangular) will be used. The brick will be oven-dried at 110°C 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.

$$ApparentPorosity = \%Pa = \frac{W-D}{W-S}\ x\ 100 \qquad (2.2)$$

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 indicates whether the refractory was well fired and thus the degree of densification will be determined by dividing the test brick mass by the exterior volume and multiplying with the density of water as recommended by ASTM-C20-00 (2015). It has correlation with apparent density and apparent porosity.This can be obtained from the equation (2.3) below

$$Bulk\ Density = \frac{Dry\ weight\ of\ sample * Density\ of\ water}{saturated\ weight\ in\ air - suspended\ weight\ in\ water}$$ (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 putting them alongside pyrometric sega cones (standard cones with definite deformation temperature) into the kiln and firing to a very high temperature of about 1650°C. 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 (Iyasara *et al.*, 2016).

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

Where RO is all oxides minus Al_2O_3 and SiO_2

3. RESULTS AND DISCUSSION
3.1 Characterization of Clay and Additives

The results obtained from the characterization of clay and additive samples using SEM/EDX were given 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 (Elakhame *et al.*, 2016). The result presented in Table 2 shows that the chicken dropping is high in Silicon, Calcium and Nitrogen elements which is typical of its source and can impart strength to the refractory material. Melon shell on the other hand is very high in carbon metal confirming that is all organic matter. The oxide form of the results in Table 2 was presented in Table 3 which shows that the clay sample contains 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). Also, chicken dropping, melon shell, and palm kernel shell are high in SiO_2, P_2O_5 and Nb_2O_5 which will translate to increased strength, toughness and conductivity in the Isiagu clay sample.

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

Weight composition	Mg	Al	P	Si	K	Nb	Ca	Ta	C	O	N
Clay	0.3	17.37		54.01	2.4				24.44	1.47	
Chicken dropping	1.08		13.49	24.01	5.92		24.95		0.68	3.51	26.81
Melon Shell			5.57	1.25					87	0.25	5.94
Palm kernel Shell		3.66				36.43	24.92	26.7	0.57	6.67	1.05

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

Weight composition	MgO	Al_2O_3	P_2O_5	SiO_2	K_2O	Nb_2O_5	CaO	Ta_2O_5
Clay	0.27	34.97		61.68	3.08			
Chicken dropping	1.31		25.95	37.41	10.38		24.95	
Melon shell			84.62	15.38				
Palm Kernel Shell		6.27				48.31	15.83	29.58

3.2 SEM/EDX Morphological analysis result for Clay, Chicken droppings, Melon Shell and Palm Kernel Shell

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 sample indicates the presence of tiny to large coarse particles (Fig. 2a), however, Aboutaleb *et al.* (2017) obtained the same result but with refractory brick waste (RBW). Also, the melon shell sample showed the presence of tiny to large coarse particles in form of a sheet (Fig. 2 b) while both the melon shell and palm kernel shell contain more large particles than tiny particles.
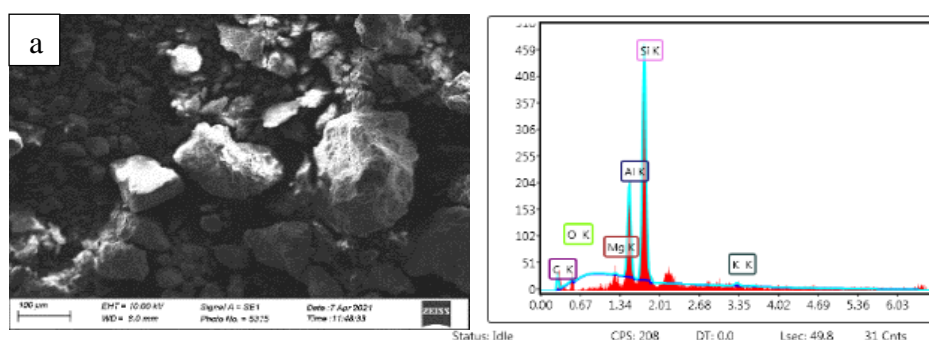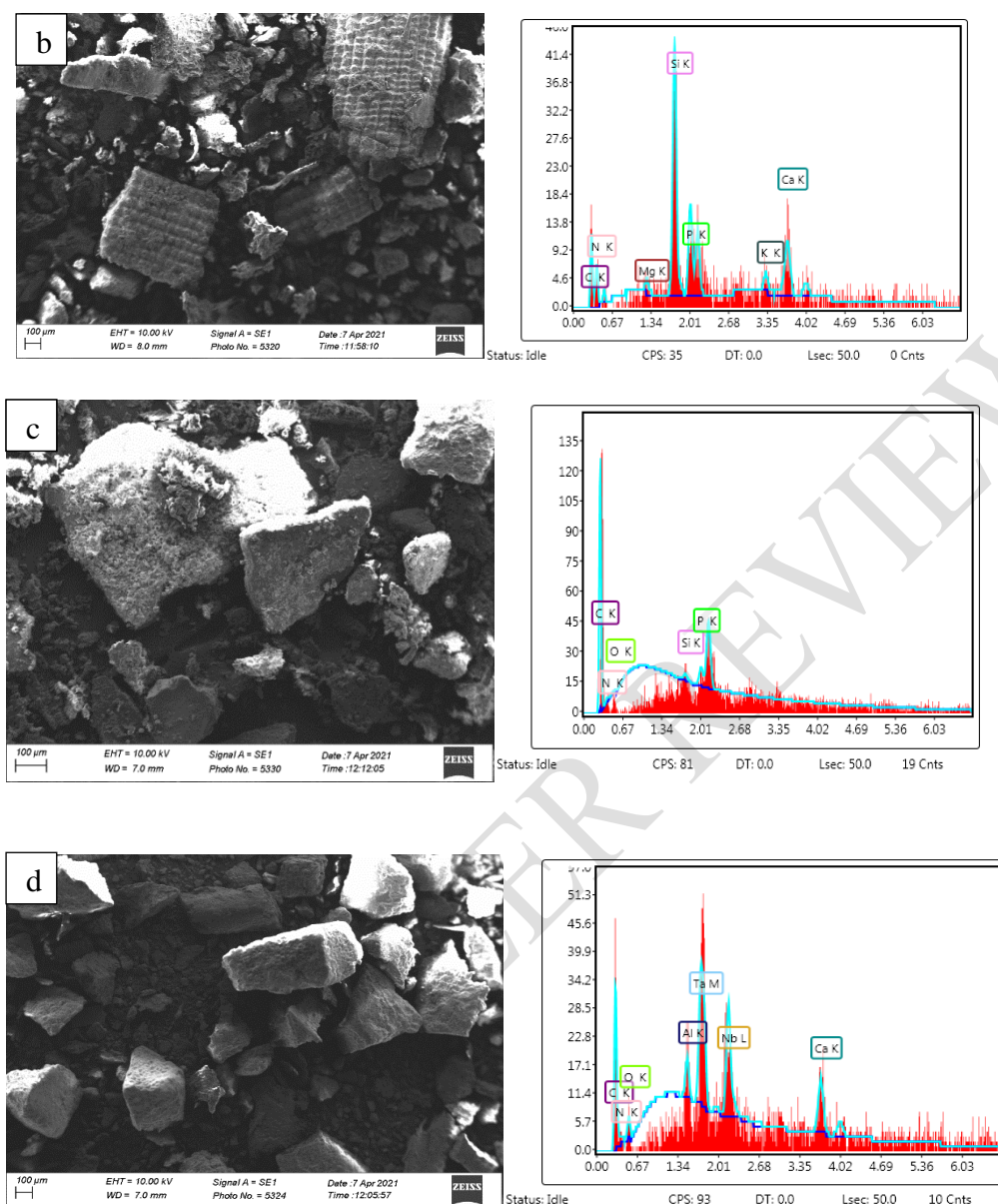
Fig 2. SEM/EDX results at EHT = 10.00kv and wavelength of 100μm for a.) Isiagu Clay
sample b.) Chicken droppings and c.) Melon Shell d.) Palm Kernel Shell

3.4 XRD analysis results for Clay, Chicken droppings, Melon Shell and Palm Kernel Shell

Different diffraction peaks were recorded for the samples analyzed and the results are shown
in Fig. 3. It indicates that the samples analyzed are mixtures of polycrystalline and amorphous
broadband (Hossain & Roy, 2019). Similar peaks were recorded for melon shell and palm
kernel shell at 21.4^o which indicates the presence of quartz phase. Also, similar peaks at 22.4^o
for palm kernel shell and chicken droppings show the presence of calcite crystalline phase at

that peak which shows that both palm kernel and chicken droppings can both impart refractoriness to Isiagu clay.
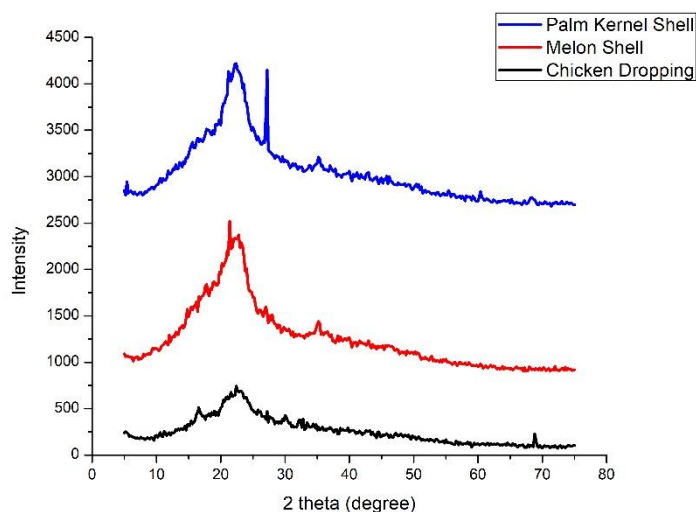

Fig 3. Results for XRD analysis for a.) Isiagu Clay sample b.) Chicken droppings and c.)

Melon Shell d.) Palm Kernel Shell.

3.5 Refractory Analysis Results

a. Linear Shrinkage
The results of the linear shrinkage measurement are shown in Fig. 4 below. The results show that there was a progressive increase in linear shrinkage for all samples at both 900^0C and 1100^0C and was within the acceptable limit of 7 to 10% (Chima *et al.*, 2017). This agrees with the SEM/EDX results which showed that chicken droppings, melon shells and palm kernel shells contain a quartz phase. However, there was a sharp decrement in chicken droppings at 10wt% which may be due to the closure of some pores of the refractory bricks because of the presence of P_2O_5 (Ajala & Badarulzaman, 2016).
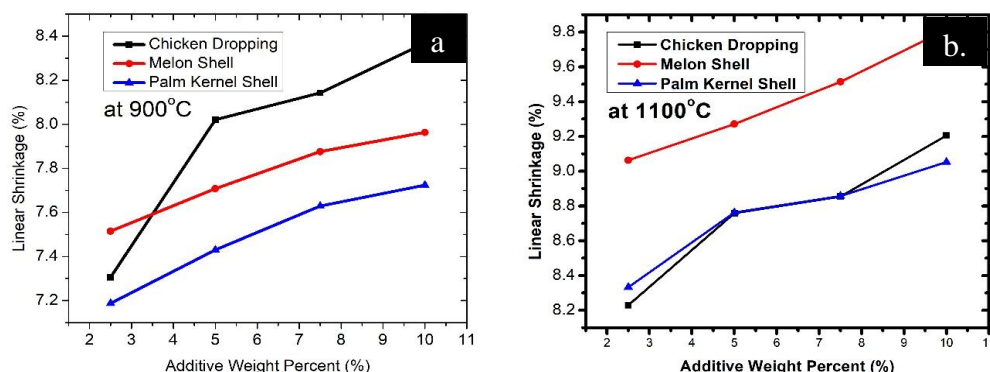

Fig 4. The plot of Linear Shrinkage of samples at a.) 900^oC and b.) 1100^oC

b. Apparent Porosity

There was a progressive increase in the apparent porosity for the melon shell and palm kernel shell as each had their peaks at 29.475% and 30.513% at 900°C, respectively according to Fig. 5. But chicken dropping showed an inconsistent trend both at 900°C and 1100°C. This is most likely due to the presence of P_2O_5 (Ajala & Badarulzaman, 2016). Based on the international standard of 2 to 30%, only the apparent porosity of the melon shell was within the specified limit (Chima et al., 2017).
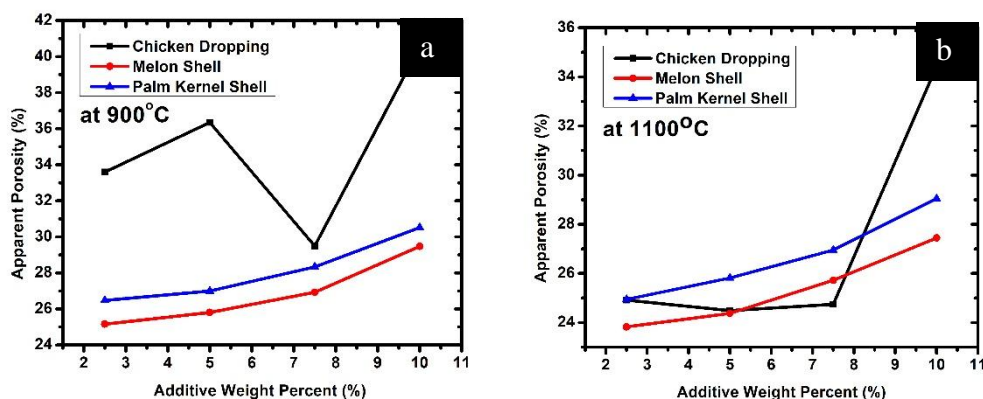
Fig 5. The plot of Apparent porosity of samples at a.) 900°C and b.) 1100°C

c. Bulk Density

There was a bulk density decrement for both melon shell and palm kernel shell according to Fig. 6 following previous works (Chima et al., 2017; Rahman et al., 2015; Velasco et al., 2015). All the samples containing melon shells at both 900°C and 1100°C were within the internationally acceptable range of 1.7 – 2.1g/cm^3 while some samples containing palm kernel shells and chicken dropping at 900°C and 1100°C were within the specified standard (Chima et al., 2017).

Fig 6. The plot of Bulk Density for Groundnut shell and Grog at a.) 900°C and b.) 1100°C

d. Refractoriness

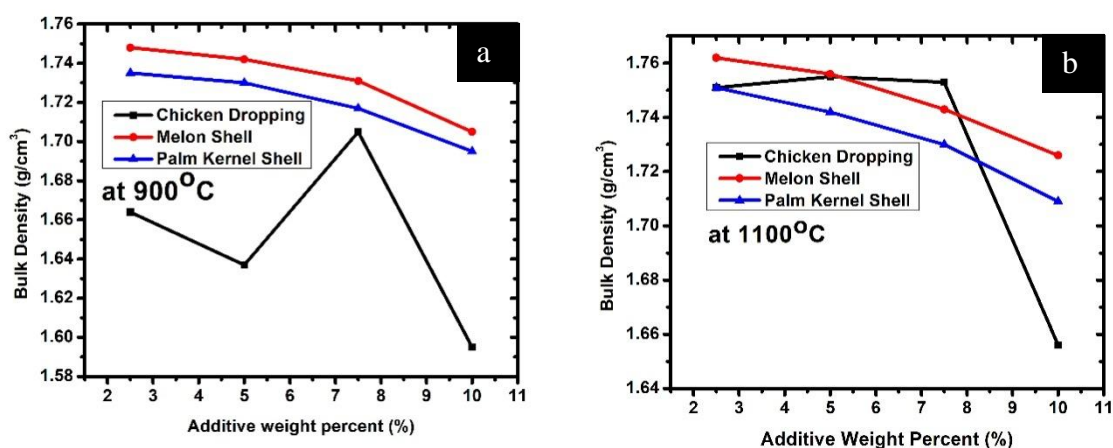This was determined using equation 2.4. The calculation indicated that Isiagu clay is fireclay and hence can withstand temperatures as high as 1447°C. This is very close to that recorded in the work of Harith and Hani (2017) where a sintering temperature of 1500°C was observed for 70% kaolin and 30% metakaolin and above 1325°C recorded by Aşkın *et al.* (2017) for Cordierite with waste magnesite.

3.3 ANOVA Results for Grog and Sawdust Additives

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

For Chicken Dropping additive

Analysis of variance was carried out for all samples containing Chicken dropping additive. The result of two-way ANOVA without replication is presented in Table 4-6. The result shows that the effect of temperature for all properties was significant (P<0.05), except for linear shrinkage (Li *et al.*, 2013; Shehu *et al.*, 2018). In the same vein, the effect of the percentage additive was not significant (P>0.05) except for apparent porosity.

Table 4: Linear Shrinkage for Chicken dropping

Source of Variation	*SS*	*Df*	*MS*	*F*	*P-value*	*F crit*	*Remark*
Temperature	13.16812	2	6.584058	2.47782	0.164263	5.143253	**Not Significant**
Percent Additive	16.42558	3	5.475193	2.060513	0.207006	4.757063	**Not Significant**
Error	15.94319	6	2.657198				
Total	45.53688	11					

Table 5: Apparent Porosity for Chicken dropping

Source of Variation	*SS*	*Df*	*MS*	*F*	*P-value*	*F crit*	*Remark*
Temperature	1766.228	2	883.114	131.8173	1.1E-05	5.143253	**Significant**
Percent Additive	126.7681	3	42.25604	6.307313	0.027621	4.757063	**Significant**
Error	40.19718	6	6.69953				
Total	1933.193	11					

Table 6: Bulk Density for Chicken dropping

Source of Variation	SS	df	MS	F	P-value	F crit	Remark
Temperature	55.47409	2	27.73704	7.852409	0.021124	5.143253	**Significant**
Percent Additive	10.0697	3	3.356566	0.95025	0.473812	4.757063	**Not Significant**
Error	21.19378	6	3.532297				
Total	86.73756	11					

For Melon additive

Analysis of variance was carried out for all samples containing melon shell additives. The result of two-way ANOVA without replication is presented in Table 7-9. The result shows that the effect of temperature for all properties was significant ($P>0.05$) except for linear shrinkage, while the temperature was significant ($P<0.05$) only for apparent porosity measurement (Table 4.5) (Li *et al.*, 2013; Shehu *et al.*, 2018). This confirms the increment in apparent porosity as temperature increased from $900^{o}C$ to $1100^{o}C$ and is hence suitable for insulating firebrick application.

Table 7: Linear Shrinkage for Melon shell additive

Source of Variation	SS	df	MS	F	P-value	F crit	Remark
Temperature	20.05284	2	10.02642	3.407164	0.102652	5.143253	**Not Significant**
Percent Additive	14.02444	3	4.674812	1.588588	0.287713	4.757063	**Not Significant**
Error	17.65648	6	2.942746				
Total	51.73375	11					

Table 8: Apparent Porosity for Melon shell additive

Source of Variation	SS	Df	MS	F	P-value	F crit	Remark
Temperature	1054.415	2	527.2075	597.3286	1.25E-07	5.14325285	**Significant**
Percent Additive	44.58738	3	14.86246	16.83923	0.002511	4.757062663	**Significant**
Error	5.295654	6	0.882609				
Total	1104.298	11					

Table 9: Bulk Density for Melon shell additive

Source of Variation	SS	df	MS	F	P-value	F crit	Remark
Temperature	54.26178	2	27.13089	7.732732272	0.021839	5.143253	**Significant**
Percent Additive	10.20038	3	3.400128	0.969090084	0.466472	4.757063	**Not Significant**
Error	21.05146	6	3.508577				
Total	85.51363	11					

For Palm Kernel Shell additive

Analysis of variance was carried out for all samples containing palm kernel shell additive. The result of two-way ANOVA without replication is presented in Table 10-12. The result shows that the effect of percentage additive for all properties was not significant, that is, P>0.05, while the temperature was not significant only for linear shrinkage measurement (Li *et al.*, 2013; Shehu *et al.*, 2018).

Table 10: Linear Shrinkage Palm

Source of Variation	SS	Df	MS	F	P-value	F crit	Remark
Temperature	12.51518	2	6.257588	2.132542	0.199695	5.143253	**Not Significant**
Percent Additive	14.09016	3	4.69672	1.600609	0.285195	4.757063	**Not Significant**
Error	17.606	6	2.934333				
Total	44.21133	11					

Table 11: Apparent Porosity

Source of Variation	SS	Df	MS	F	P-value	F crit	Remark
Temperature	1195.078	2	597.539	740.7198	6.56E-08	5.143253	**Significant**
Percent Additive	45.56803	3	15.18934	18.82897	0.001865	4.757063	**Significant**
Error	4.840203	6	0.8067				
Total	1245.486	11					

Table 12: Bulk Density

Source of Variation	SS	df	MS	F	P-value	F crit	Remark
Temperature	54.5749	2	27.28745	7.774269	0.021587	5.143253	**Significant**
Percent Additive	10.19213	3	3.397376	0.967922	0.466923	4.757063	**Not Significant**
Error	21.05982	6	3.50997				
Total	85.82684	11					

4. CONCLUSION

The effect of chicken dropping, melon shell, and palm kernel shell on the refractory properties of Isiagu clay was studied. The ANOVA was also carried out using Excel 2013. The SEM/EDX result showed that chicken dropping, melon shell and palm kernel shell were high in SiO_2, P_2O_5 and Nb_2O_5, which translated to increased strength, toughness and conductivity in the Isiagu clay sample, respectively. The strength and toughness impartation was confirmed by bulk density measurement for melon shell as it is the only additive which met the international standard for refractory brick application. On the other hand, chicken dropping showed sharp changes for all the properties measured namely; linear shrinkage, apparent porosity and bulk density which is due to the presence of P_2O_5 and makes its suitability for refractory application doubtful. However, researchers can explore increased additive percentage and decreased grain size for the possibility of better results. Although XRD results confirm the capability of Chicken droppings and palm kernel shells in imparting strength to refractory bricks, more work should be done on chicken dropping usage. ANOVA results confirmed that for melon shell and apparent porosity only temperature was significant as this property increased from 900°C to 1100°C.

COMPETING INTERESTS DISCLAIMER:

Authors have declared that they have no known competing financial interests OR non-financial interests OR personal relationships that could have appeared to influence the work reported in this paper.

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