

The effect of sawdust, cow dung, and CaC₂ wastes on properties of refractory bricks

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Abstract

The effect of sawdust, cow dung, and CaC₂ waste on fireclay was studied. The X-ray Fluorescence (XRF) analysis of the fireclay showed that it contains about 14.328% and 44.382% of Al₂O₃ and SiO₂, respectively. The result of the scanning electron microscope (SEM) showed that the fireclay contained a mixture of coarse and fine grain particles. The analysis on the fireclay was; fireclay with cow dung only, sawdust only, Cow dung & CaC₂ waste, and sawdust & CaC₂ waste. In each phase, fireclay was combined in the ratio of 100, 90, 80, and 70%, for the additives used. All these properties were studied at 1000^oC and 1200^oC for all the additive combinations. The sample containing 30wt% of Sawdust and CaC₂ waste had the highest apparent porosity of 74% and 74.48% at 1000^oC and 1200^oC, respectively. The lowest apparent porosity was seen in the sample containing 10wt% of cow dung which were 15.29% and 9.06% at 1000^oC and 1200^oC, respectively. In this work, apparent porosity, water absorption, apparent density, and bulk density showed similar graphical trends for all the samples produced. On the other hand, linear shrinkage decreased at constant temperature but increased as the temperature varied from 1000^oC to 1200^oC for all samples produced. Analysis of Variance (ANOVA) carried out on the samples showed that most of the properties evaluated were significant.

Keywords: Refractories, fireclay, additives, biowastes, mechanical properties, firebrick

1. Introduction

Refractories are used in ovens, furnaces, and boilers to achieve heat transfer efficiency whilst minimizing cost. Generally, non-metallic inorganic materials which are both chemically and physically stable over a wide temperature range of 600-1200^oC are referred to as refractory materials (Antonovič et al., 2017; Shuaib-Babata et al., 2018). They occur in different forms and can resist heat as high as 1500^oC hence retaining their physical and chemical properties (Antonovič et al., 2017; Chima et al., 2017; Zaidan & Omar, 2018). In Nigeria, there are about seven clay deposits that have proven refractory properties and hence good candidates for refractory brick production (Amkpa et al., 2017b; Oke et al., 2015). Amkpa et al. (2017b) stated that there are possibly clay deposits in all the 36 states of the federation which until now are yet to be studied. This implies that Nigeria could become one of the world's leading producers of refractory materials if the potentials of these clay deposits are exploited. Irrespective of the large clay deposits found in Nigeria, she is yet to meet her refractory needs in both quality and quantity. The major reason for this is that most of the refractories used to wear out after several usages. This is because their chemical and physical properties become weakened over time leading to failures and fractures (Kazemi, 2019). Given this, mixtures of clay with another clay or with additives have been used to further extend the shelf life of the refractory materials by improving properties like thermal shock resistance, cold crushing strength, bulk density, porosity, etc. (Chima et al., 2017). Additives used are mainly biowastes which enable proper management of these wastes and reduce disposal costs (Cultrone et al., 2020). 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 20wt% and 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 owing to the fineness of the additives (Rahman et al., 2015). Elsewhere, (Amkpa et al., 2017a) worked on Barkin-

Ladi clay and determined its suitability for refractory application. It was discovered that the clay is refractory having a thermal conductivity of 0.03W/mK, a specific heat capacity of 0.07J/g⁰C, refractoriness of 1665⁰C, thermal shock resistance of 24 cycles, and high energy absorption. In the present work, efforts have been made to study the effects of local additives namely; sawdust, cow dung, and calcium carbide (CaC₂) waste on the refractory properties of local clays.

2.0 Material and methods

2.1 Materials sourcing and characterization

The fire clay sample selected for investigation was obtained from Isiagu, Awka South Local Government Area of Anambra State. Definite fire clay of precisely grey to light brown colour was collected. The colour may be due to the high amount of Fe₂O₃ in it. The analysis of fire clay was done in a chemical laboratory using both X-ray Fluorescence and Scanning Electron Microscope (SEM). Consequently, cow dung, sawdust, and calcium carbide (CaC₂) waste were sourced from cow stalls, timber, and panel beating shop all in Awka.

2.2 Materials processing

The clay was obtained in lumps. It was sun-dried to remove its moisture. The clay was reduced to smaller sizes mechanically with the aid of a mortar and pestle (crusher) after which they were sieved to the required particle size with the aid of a 600µm sieve. The ratio of the mixture samples for the production is shown below in Table 1.

Table 1: Percentages of additives and clay in the test samples

Sample	Clay (%)	Cow dung only	Sawdust only	Cow dung and CaC ₂ waste		Sawdust and CaC ₂ waste	
A	100	0	0	0	0	0	0
B	90	10	10	5	5	5	5
C	80	20	20	10	10	10	10
D	70	30	30	15	15	15	15

2.3 Materials mixing and molding

Based on the research design, the various compositions of clay, sawdust, cow dung, and CaC₂ waste were weighed out and mixed properly. Water was then added to the samples (100%clay, 90%clay, 80%clay, and 70%clay). Due to the addition of water, a sticky mass was formed. With the aid of the mould (solid materials made of wood used to form clay samples into different shapes), the sticky mass was formed into different shapes (cylindrical, rectangular) (Chima et al., 2017). The application of lubricants to the surface of the moulds was done to prevent the test pieces from sticking to the surface (Chima et al., 2017). The first shape is cylindrical with a width of 3.5cm and height of 3.7cm, the second is a rectangular piece with a length of 8cm, width 4cm, and height of 1.5cm, while the third has a long rectangular shape with a length 9.5cm, width 2cm and height 1.5cm

2.4 Drying and firing

The moulded test pieces were then allowed to dry by exposing them to air for some time. After air-drying the test pieces, they were then moved into the oven where they were dried at a temperature of 200⁰C for two days. They were thereafter moved into a furnace where they were fired at high temperatures (1000⁰C, 1200⁰C). There is a temperature gradient in the furnace which enabled the firing of the various clay compositions at various temperatures. The various test pieces were allowed to stay in the furnace for a week after which the furnace was unloaded.

2.5 Measurement of Refractory Properties

a. 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). The brick was oven-dried at 110°C to constant weight (D). After which it was transferred to a beaker and boiled with distilled water for 1.5hrs to assist in releasing the trapped air. It was soaked and the saturated weight free of water (W) was obtained. Finally, the specimen was suspended in the water by a rope tied to a spring balance and the suspended weight (S) was obtained when it is completely immersed in water.

$$\text{Apparent Porosity} = \%P_a = \frac{W-D}{W-S} \times 100 \quad (1)$$

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)

b. Determination of Water Absorption

The fired test pieces obtained after firing were then weighed and the weight recorded as dry weight, M_1 (g). Thereafter, the test pieces were soaked in water for one hour, then removed, cleaned, and weighed immediately, and recorded as soaked weight, M_2 (g). The water of adsorption was then calculated.

$$\text{Water Absorption (\%)} = 100 \times \frac{M_2 - M_3}{M_1} \quad (2)$$

Where;

M_2 = soaked weight, M_1 = dry weight, M_3 = the weight in air

c. Bulk Density (BD) and Apparent Density

The Bulk Density (BD) of a refractory indicates whether the refractory was well fired and thus the degree of densification and it was obtained by dividing the test brick mass by the exterior volume and multiplying with a density of water as recommended by (ASTM-C20-00, 2015).

$$\text{Bulk Density} = \frac{\text{Dry weight of sample} \times \text{Density of water}}{\text{saturated weight in air} - \text{suspended weight in water}} \quad (3)$$

$$\text{Apparent Density} = \frac{\text{Dry weight of sample} \times \text{Density of water}}{\text{suspended weight} - \text{suspended weight in water}} \quad (4)$$

d. Linear Shrinkage

This evaluates the linear changes that occur in brick samples when heated. The formula is as given in the work of (Adeosun et al., 2016). This is calculated based on the original length (L_o) before drying and the final length (L_f) after firing to a certain temperature

$$\text{Percentage Total Shrinkage} = \frac{L_o - L_f}{L_o} \times 100 \quad (5)$$

Where; L_o = the green length, L_f = the fired length

3.0 Results and Discussions

3.1 Chemical Analysis of fireclay

The fireclay was analyzed using both XRF and SEM and the result indicated that the clay is a fireclay as it has refractoriness of about 1,460°C as was calculated using Shuen's formula (Iyasara et al., 2016) (Eqn 6). However, this is lower than that recommended by Madugu (2016). Although Elakhame et al. (2016) found that fireclays have an alumina percentage of 39.50%, the fireclay used in the current has some admirable properties especially as the additives were incorporated (Elakhame et al., 2016).

$$\text{Refractoriness (K)} = \frac{360 + Al_2O_3 - RO}{0.228} \quad (6)$$

Where RO implies other oxides minus Al_2O_3 and SiO_2

The result of the mineral content of the fireclay was presented in Table 2.

Table 2: Result of X-ray Fluorescence showing the concentration of various minerals in the Isiagu fire clay sample

Element	Concentration (Wt%)
Na ₂ O	1.315
MgO	1.273
Al ₂ O ₃	14.328
SiO ₂	44.382
P ₂ O ₅	1.115
K ₂ O	1.938
CaO	0.550
TiO ₂	1.004
Fe ₂ O ₃	4.936
TOTAL	100

The result of the SEM micrograph indicated that the clay is a mixture of micro and macroparticles therefore at different wavelengths namely; 536µm, 268µm, and 44.7µm as shown in Figure 1.

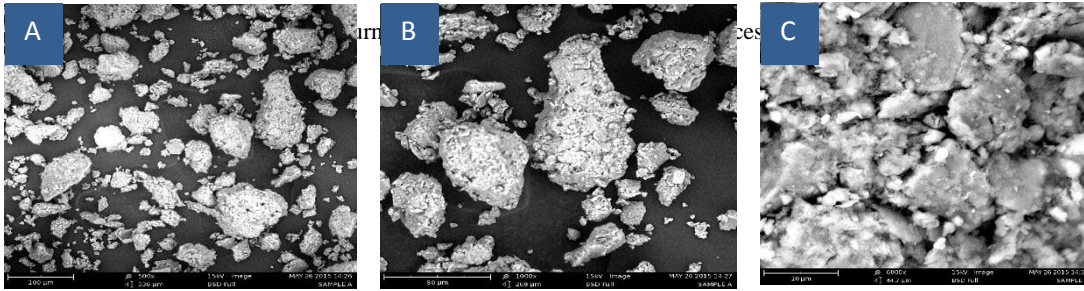


Figure 1: SEM micrograph of Isiaagu Clay; A) at 536µm; B) at 268µm; C) at 44.7µm

3.2 Refractory analysis results

a. Apparent porosity

The pore formers, cow dung, and sawdust are known to increase apparent porosity as they were removed when the brick material is fired (Babaso & Sharanagouda, 2017). There was a steady increase in the apparent porosity with additive amount as compared with the control (Fig 2.0) which were 12.95 and 5.33 wt% at 1000°C and 1200°C, respectively. The highest apparent porosity (74.48%) was recorded by the sample containing both sawdust and CaC₂ waste at 1000°C and 1200°C. This is way off the standard as was given by Chima et al. (2017) which is in the range of 2 to 30%. This may be as a result of the air pockets in the sample as (Keter, 2016) observed. Based on Chima et al. (2017) the reference provided samples containing cow dung only and sawdust only fell within the limit (Chima et al., 2017).

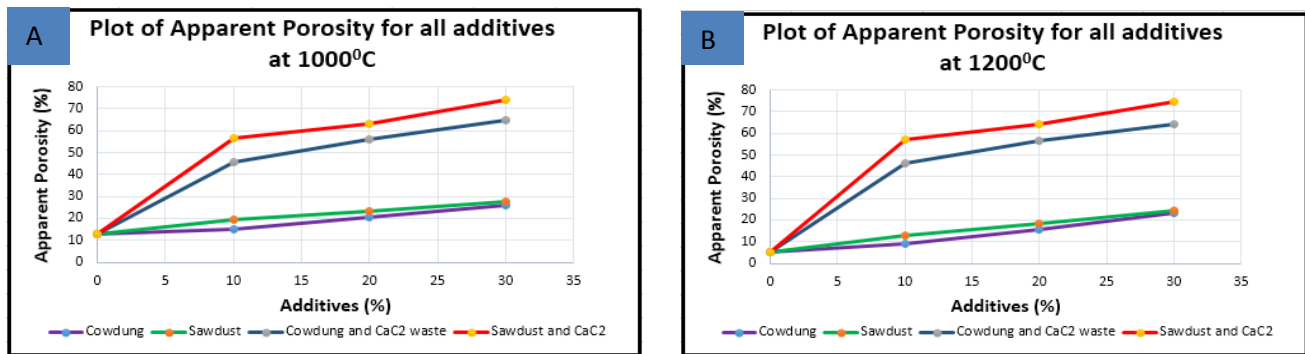


Figure 2: Plot of Apparent Porosity for all samples at A.) 1000°C and B.) 1200°C

b. Water absorption

Water absorption in its own right had the same trend as apparent porosity having the highest value of 64.06% for cow dung and CaC₂ waste combination at 1000°C and lowest value of 2.74% at 1200°C (Fig 3.0). Just like apparent porosity, water absorption increased steadily with the additive percentage at both temperatures (Ajala & Badarulzaman, 2016). Based on the international standard of 20 to 80%, we can conclude that the water absorption values for all the samples containing cow dung only or sawdust only fell below the limit while the ones containing cow dung & CaC₂ and sawdust & CaC₂ were within the acceptable limit and hence met the requirement for refractory application (Chikwelu et al., 2018).

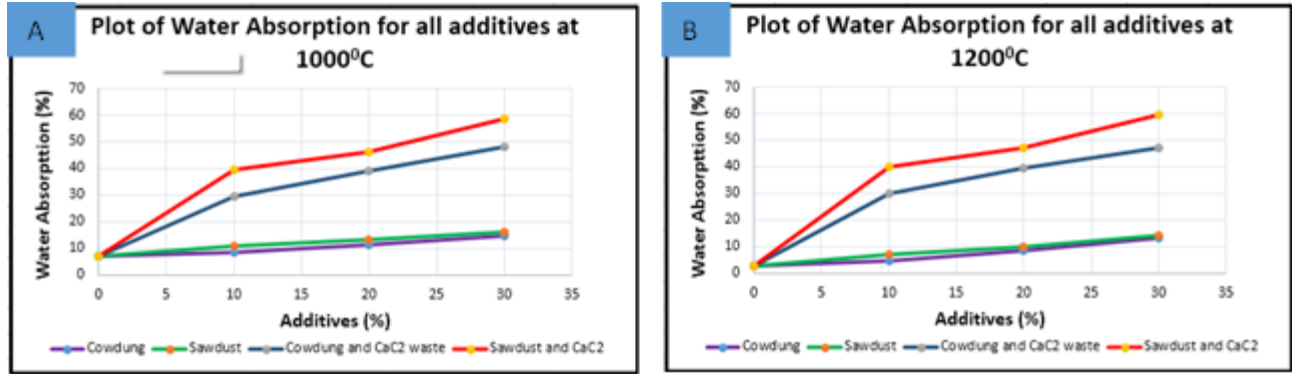


Figure 3: Plot of Water absorption for all samples at A.) 1000°C and B.) 1200°C

c. Apparent Density

The apparent density at both 1000°C and 1200°C increased steadily for sawdust & CaC₂ only and cow dung & CaC₂ only and a slight increase for cow dung only and sawdust only. The internationally accepted standard of 2.3-3.5g/cm³ was achieved in the samples containing cow dung only and sawdust only at 30% additive percentage while that of sawdust & CaC₂ only and cow dung & CaC₂ only was achieved at 10% additive percentage (Chikwelu et al., 2018).

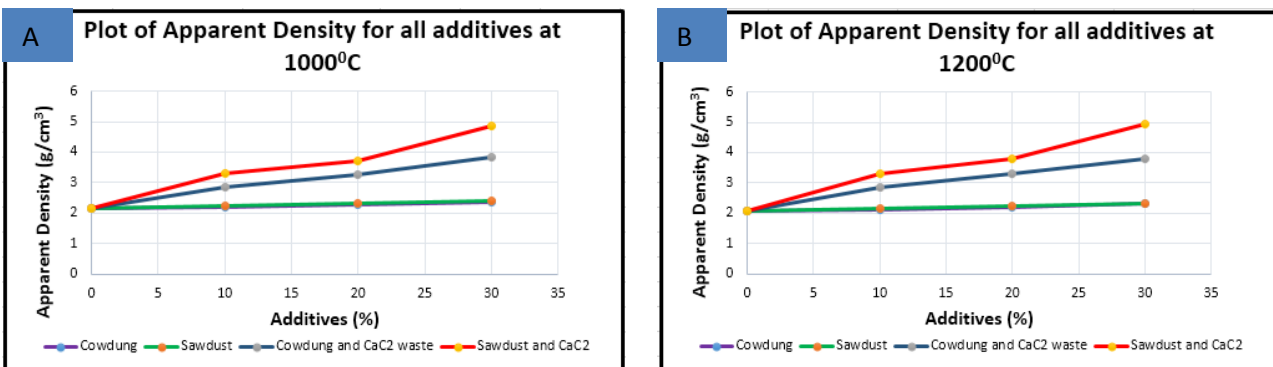


Figure 4: Plot of Apparent Density for all samples at A.) 1000°C and B.) 1200°C

d. Bulk density

Bulk density decrement is following previous works (Chima et al., 2017; Rahman et al., 2015; Velasco et al., 2015) (Velasco et al., 2015). It was seen that the samples containing sawdust & CaC₂ only and cow dung & CaC₂ only fell below the internationally permissible value of 1.7- 2.1 g/cm³. While the samples containing sawdust and cow dung only at 30% additive met the internationally permissible standard.

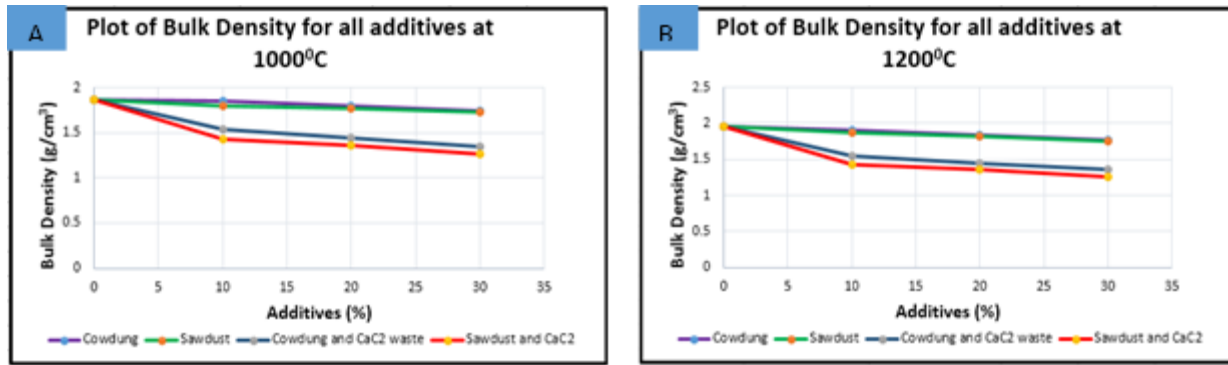


Figure 5: Plot of Bulk Density for all samples at A.) 1000°C and B.) 1200°C

e. Linear shrinkage

The graph in Fig 6.0 shows the variation of the linear shrinkage from 1000°C to 1200°C. The standard permissible limit for linear shrinkage was given by Chima et al, 2017 as 7-10% (Chima et al., 2017). In the present work, the range was met by the samples containing sawdust only as it ranged from 7.4 at 10wt% to 8.76% at 30wt%. Hence, the samples containing sawdust only can be applied in the production of refractory bricks.

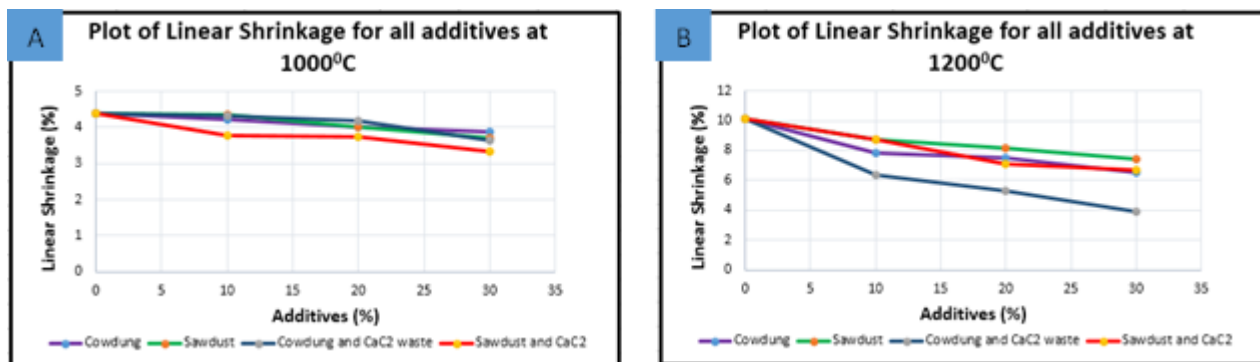


Figure 6: Plot of Linear Shrinkage for all samples at A.) 1000°C and B.) 1200°C

3.3 Analysis of Variance (ANOVA) for the brick samples

The two-way Analysis of Variance (ANOVA) without replication was carried out for all the brick samples using *Excel 2013*. The results of the analysis are given for the additive categories;

A. COW DUNG ONLY

Analysis of Variance was carried out on samples containing cow dung only. The results are as shown in Table 3-7. The analysis of the effect of temperature and cow dung additive using two-way ANOVA without replication was found to be significant ($P < 0.05$) for the properties tested except the linear shrinkage which had a P-value for cow dung additive (> 0.05) (Li et al., 2013; Shehu et al., 2018).

Table 3: Apparent Porosity ANOVA

APPARENT POROSITY ANOVA		COW DUNG ONLY			
Source of Variation	df	F	P-value	F crit	Remark
Temperature	1	24.29455	0.016008	10.12796	significant
Cow dung Percentage	3	39.82436	0.00646	9.276628	significant
Error	3				
Total	7				

Table 4: Water Absorption ANOVA

WATER ABSORPTION ANOVA		COW DUNG ONLY			
Source of Variation	Df	F	P-value	F crit	Remark
Temperature	1	33.49472	0.010261	10.12796	significant
Cow dung Percentage	3	58.13934	0.003714	9.276628	significant
Error	3				
Total	7				

Table 5: Apparent Density ANOVA

APPARENT DENSITY ANOVA		COW DUNG ONLY			
Source of Variation	Df	F	P-value	F crit	Remark
Temperature	1	72.08571	0.003431	10.12796	significant
Cow dung Percentage	3	131.9714	0.001105	9.276628	significant
Error	3				
Total	7				

Table 6: Bulk Density ANOVA

BULK DENSITY ANOVA		COW DUNG ONLY			
Source of Variation	df	F	P-value	F crit	Remark
Temperature	1	22.42373	0.017854	10.12796	significant
Cow dung Percentage	3	38.28814	0.006841	9.276628	significant
Error	3				
Total	7				

Table 7: Linear Shrinkage ANOVA

LINEAR SHRINKAGE ANOVA		COW DUNG ONLY			
Source of Variation	df	F	P-value	F crit	Remark
Temperature	1	33.27453	0.010356	10.12796	significant
Cow dung Percentage	3	1.726773	0.332351	9.276628	Not significant
Error	3				
Total	7				

B. SAWDUST ONLY

Analysis of Variance was carried out on samples containing sawdust only. The results are as shown in Table 8-12. The analysis of the effect of temperature and sawdust additive using two-way ANOVA without replication was found to be significant ($P < 0.05$) for the properties tested except the linear shrinkage which had a P-value for sawdust additive (> 0.05) (Li et al., 2013; Shehu et al., 2018)

Table 8: Apparent Porosity ANOVA

APPARENT POROSITY ANOVA			SAWDUST ONLY		
<i>Source of Variation</i>	<i>df</i>	<i>F</i>	<i>P-value</i>	<i>F crit</i>	<i>Remark</i>
Temperature	1	30.17257	0.011872	10.12796	significant
Sawdust Percentage	3	50.36757	0.004584	9.276628	significant
Error	3				
Total	7				

Table 9: Water Absorption ANOVA

WATER ABSORPTION ANOVA			SAWDUST ONLY		
<i>Source of Variation</i>	<i>Df</i>	<i>F</i>	<i>P-value</i>	<i>F crit</i>	<i>Remark</i>
Temperature	1	44.20659	0.006934	10.12796	significant
Sawdust Percentage	3	75.25756	0.002539	9.276628	significant
Error	3				
Total	7				

Table 10: Apparent Density ANOVA

			SAWDUST ONLY		
<i>Source of Variation</i>	<i>df</i>	<i>F</i>	<i>P-value</i>	<i>F crit</i>	<i>Remark</i>
Temperature	1	67.04651	0.003811	10.12796	significant
Sawdust Percentage	3	124.1628	0.001209	9.276628	significant
Error	3				
Total	7				

Table 11: Bulk Density ANOVA

BULK DENSITY ANOVA			SAWDUST ONLY		
<i>Source of Variation</i>	<i>df</i>	<i>F</i>	<i>P-value</i>	<i>F crit</i>	<i>Remark</i>
Temperature	1	17.28571	0.025302	10.12796	significant
Sawdust Percentage	3	29.2381	0.010108	9.276628	significant
Error	3				
Total	7				

Table 12: Linear Shrinkage ANOVA

			SAWDUST ONLY		
<i>Source of Variation</i>	<i>df</i>	<i>F</i>	<i>P-value</i>	<i>F crit</i>	<i>Remark</i>
Temperature	1	105.6048	0.001965	10.12796	significant
Sawdust Percentage	3	2.649703	0.222365	9.276628	Not significant
Error	3				
Total	7				

C. COW DUNG AND CaC₂ ONLY

Analysis of Variance was carried out on samples containing cow dung & CaC₂ only. The results are as shown in Table 13-17. The analysis of the effect of temperature and cow dung & CaC₂ additive using two-way ANOVA without replication was found to be significant for properties such as apparent porosity, water absorption, apparent density, and bulk density ($P < 0.05$) for the various percentages of additives used. However, the results were not significant ($P > 0.05$) for the same set of properties at various temperatures (Li et al., 2013; Shehu et al., 2018). This may be attributed to the high loss of matter as the samples were fired. The result of the linear shrinkage showed that both temperature and additive percentage were not significant. This may also be attributed to the high loss of matter as the samples were fired.

Table 13: Apparent Porosity ANOVA

APPARENT POROSITY ANOVA		COW DUNG AND CaC ₂			
Source of Variation	df	F	P-value	F crit	Remark
Temperature	1	0.899957	0.41278	10.12796	Not significant
Additive percentage	3	156.4839	0.000857	9.276628	significant
Error	3				
Total	7				

Table 14: Water Absorption ANOVA

WATER ABSORPTION ANOVA		COW DUNG AND CaC ₂			
Source of Variation	df	F	P-value	F crit	Remark
Temperature	1	0.907268	0.411111	10.12796	Not significant
Additive Percentage	3	284.2538	0.000352	9.276628	significant
Error	3				
Total	7				

Table 15: Apparent Density ANOVA

APPARENT DENSITY ANOVA		COW DUNG AND CaC ₂			
Source of Variation	df	F	P-value	F crit	Remark
Temperature	1	1.058824	0.379187	10.12796	Not significant
Percentage Additives	3	616.0588	0.000111	9.276628	significant
Error	3				
Total	7				

Table 16: Bulk Density ANOVA

BULK DENSITY ANOVA		COW DUNG AND CaC ₂			
Source of Variation	df	F	P-value	F crit	Remark
Temperature	1	1.357542	0.328185	10.12796	Not significant
Additive Percentage	3	160.2849	0.000827	9.276628	significant
Error	3				
Total	7				

Table 17: Linear Shrinkage ANOVA

LINEAR SHRINKAGE ANOVA		COW DUNG AND CaC ₂			
Source of Variation	df	F	P-value	F crit	Remark
Temperature	1	3.576957	0.154958	10.12796	Not significant
Additive Percentage	3	1.494868	0.374557	9.276628	Not significant
Error	3				
Total	7				

D. SAWDUST AND CaC₂ ONLY

Analysis of Variance was carried out on samples containing Sawdust & CaC₂ only. The results are as shown in Table 16-20. The analysis of the effect of temperature and Sawdust & CaC₂ additive using two-way ANOVA without replication was found to be significant ($P < 0.05$) for properties such as apparent porosity, water absorption, apparent density, and bulk density for the various percentages of additives used. However, the results were not significant ($P > 0.05$) for the same set of properties at various temperatures (Li et al., 2013; Shehu et al., 2018). This may be attributed to the high loss of matter as the samples were fired. The result of the linear shrinkage showed that while the temperature is significant additive percentage was not significant. This may also be attributed to the high loss of matter as the samples were fired.

Table 18: Apparent Porosity ANOVA

APPARENT POROSITY ANOVA		SAWDUST & CaC ₂			
Source of Variation	df	F	P-value	F crit	Remark
Temperature	1	0.440156	0.554475	10.12796	Not significant
Additive Percentage	3	190.7879	0.000638	9.276628	significant
Error	3				
Total	7				

Table 19: Water Absorption ANOVA

		SAWDUST & CaC ₂			
Source of Variation	df	F	P-value	F crit	Remark
Temperature	1	0.145871	0.727961	10.12796	Not significant
Additive Percentage	3	349.1949	0.000259	9.276628	significant
Error	3				
Total	7				

Table 20: Apparent Density ANOVA

		SAWDUST & CaC ₂			
Source of Variation	df	F	P-value	F crit	Remark
Temperature	1	0.384615	0.579065	10.12796	Not significant
Additive Percentage	3	813.6769	7.3E-05	9.276628	significant
Error	3				
Total	7				

Table 21: Bulk Density ANOVA

BULK DENSITY ANOVA		SAWDUST & CaC ₂			
Source of Variation	df	F	P-value	F crit	Remark
Temperature	1	1	0.391002	10.12796	Not significant
Additive Percentage	3	208.1667	0.00056	9.276628	significant
Error	3				
Total	7				

Table 22: Linear Shrinkage ANOVA

		SAWDUST & CaC ₂			
Source of Variation	df	F	P-value	F crit	Remark
Temperature	1	51.37499	0.005594	10.12796	Significant
Additive Percentage	3	2.732023	0.215525	9.276628	Not significant
Error	3				
Total	7				

4.0. Conclusion

The effect of cow dung and sawdust together with CaC₂ additives on the refractory properties of clay was studied. Also, all the samples were subjected to an ANOVA test using Excel 2013. The conclusions drawn from this work is as follows;

- i. In this study, CaC₂ waste was combined with pore formers (Cow dung and CaC₂) which produced more pores compared with other additives previously used.
- ii. The highest apparent porosity (74.48%) was recorded by the sample containing sawdust and CaC₂ waste only at 1000°C and 1200°C.
- iii. Water absorption in its own right had the same trend as apparent porosity having the highest value of 64.06% for cow dung and CaC₂ waste combination at 1000°C and lowest value of 2.74% at 1200°C.
- iv. The apparent density at both 1000°C and 1200°C increased steadily for sawdust & CaC₂ only and cow dung & CaC₂ only and a slight increase for cow dung only and sawdust only. This is due to the difference in the pore amount between the former and the latter. Bulk density and linear shrinkage both followed the internationally acclaimed standard.
- v. The samples containing cow dung only and sawdust only had P-values (<0.05) indicating that temperature and additive percentages were significant for all properties measured. This was the reason moderate pores were produced on the surface of the samples making them good for refractory application.
- vi. However, the linear shrinkage indicated that cow dung percentage was not significant (P>0.05) compared with temperature. This is so because linear shrinkage increased with temperature.
- vii. Samples containing cow dung and CaC₂ only and sawdust and CaC₂ only, showed that only additive percentage was significant for properties measured. This was responsible for the many pores formed on the surface of the samples which makes them adequate for insulation application.

5.0 Recommendation

From this work it is clear that CaC₂ waste can't be used as an additive for refractory brick production since it gave porosity far above the internationally acclaimed standard. Other biowastes such as melon shell, groundnut shell and palm kernel shell can also be studied to investigate their effect on fireclays.

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