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Induced porosity of fired clay using melon husk to temperatures of 650, 700, 750, 800, 850, 900, and 1000 OC came under interest in this study.

Samples of two colors- brown and white, were tested for porosity and bulk density levels before and after firing. Samples were prepared into cores 20 mm radius 50 mm height and tested for pre-fired porosity; batch-mixed with 500 µm melon husk at 0%, 15 %, 20 %, and 25 %, prepared with 5 % grog into cubes 50 x 50 x 50 mm and fired in a gas kiln for post-fired porosity tests. Pre-fired porosities were 53.75 % and 50.34 %, and bulk densities were 1.225 g/cm3 and 1.315 g/cm3 for brown and white clay, and their 1000 OC post-fired porosity marginal values were 25.99 ± 10.05 %, 45.475 ± 9.705 %, 60.535 ± 1.165 % and 62.01 ± 1.36 % at 0 wt. %, 15 wt. %, 20 wt. % and 25 wt. % melon husk premix, respectively. It was concluded that premixing clay with melon husk biomass at minor increments to 20 % enhances porosity in porous ceramics fired at 1000 OC to a value twice that of homogenous fired clay.

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I. INTRODUCTION

Porosity is a natural phenomenon in clays. Clays are products of the weathering of primary igneous rocks such as granites, feldspars and pegmatites [1] which are soil-based derivatives of the earth's crust. The residue of pore space in soil, sediments, and rock is termed porosity [2]. [3] posits that pore spaces are an essential component to soil-based clays, since their volume, size, and distribution provide the attributes for industrial use. [4] defines pore space as a collection of pathways that enable the passage of fluid within the body of the soil. Thus porosity in clay can be the fraction of total clay volume taken up by pore space [4]. Clays naturally retain water through their pores as they turn plastic. On the application of heat, water is driven out of the pore spaces as the clay sets resulting in changes in porosity and water absorption levels [4] [5]. Porosity in ceramics can be classified as apparent, which is the ratio of the volume of open pores to the bulk volume of material; sealed porosity, which is the ratio of the volume of the sealed pores to the bulk volume, and true porosity, which is the total ratio of the volume of the open and sealed pores to the bulk volume [1]. Ceramics also has different sizes of pore spaces called macro-, meso-, and micropores [5]. Macro- and mesopores are associated with particle-to-particle interactions, whereas micropores are primarily localized in the interlayer spaces of clay minerals.

The science of application of pore spaces inherent to fired clay is not new in the industry. Porosity in ceramic materials has become one of the key features enabling wide range of applications which allow for the channels to retain and separate the different states of matter one from the other. The term porous ceramics is used to describe fired clays having high percentage porosity between 20 and 95 % [6], more levels than usual to leverage the unique characteristics.

Porous ceramics are used in applications requiring light-weightiness, strength and stability with accompanying surface characteristics such as open-to- closed pore ratio and abrasion resistance. Examples are waste-water purifiers and water filters [7] [8]; granular filters of dust particles in high-purity processes [9]; bioreactor membranes and environmental filters for diesel engine emissions [10] [11]; and support structures for catalysis applications [12]. In addition, various porous ceramics, such as lightweight cellular structures, can be used as thermal insulators in buildings [13].

One of the production methods applied to manufacture porous ceramics is by incorporating sacrificial phase (pore former), whereby combustible material is impregnated into the green ceramic body through mixing, and incinerated during firing procedure, thus leaving behind an aggregate of pore spaces greater than a homogenous fired clay. Several researchers have investigated diverse categories of biomass pore formers for different ceramic applications, and examples include sawdust [7] [8] [14] [15]; bagasse [16]; banana and plantain peels [9] [17]; and rice husk [15] [18]. A greater number of these biomasses sought out and applied have been used to replace commercial starch, the industrial pore former material which is reported to be expensive [17] and, in some locations, unavailable in large quantities. In this present study, melon husk, which is an agro waste, is considered as a replacement for commercial starch pore former due to its lightness, easy handling, and availability in Southern Nigeria in the production of porous ceramics using locally sourced clays.

A common occurrence in clay transformation into the ceramic body is the thermal action of the sintering mechanism, which drives the movement of clay particles across grain boundaries leading to particle consolidation, under sustained elevated temperatures from 700 degree centigrade and upwards. The driving force of sintering is the reduction in surface free energy of the consolidated mass of particles [19]. The sintering process converts the green microstructure to the microstructure of the dense ceramic component [20]. However, despite widely held practices of attaining enhanced mechanical properties at sintering temperatures exceeding 1000 OC, where densification has advanced into total consolidation, it has been proven that low-temperature sintering can also improve the porosity and strength of ceramic materials. [10] reported that ceramics sintered at 850 OC and mixed with 5 % wt pore former of powdery high-density polyethylene gave the optimum properties in terms of the porosity, and mechanical strength of the samples. Summarily, the consolidation of clay particles and grain growth, although altogether increasing the mass, cohesiveness and strength of the produced ceramic body, has detrimental effects on the functionality of retained porosity of the green body. It is therefore of interest to experimentally study the effects of melon husk pore former addition at different weight loads, to the interactions between porosity and the counter forces of sintering with rising temperature. In addition, the application of melon husk biomass in manufacturing porous ceramics is novel. This research attempts to apply and examine the porosity performance of ceramics using this biomass as a pore former.

II. MATERIALS AND METHODS


Figure 2.1: Melon husk


Figure 2.2: Brown clay


Figure 2.3: White clay

Clays used for the study were sourced from Ikot Ebom Itam quarry in Itu local government area, Akwa Ibom State, South Eastern Nigeria. At the quarry, the clays existed in two distinct colorations, which are brown and white clay, Figures 2.2 and 2.3, respectively. 2.5 kg of each sample was excavated and taken to the laboratory for analysis. Melon (Citrullus colocynthis lanatus) seeds, Figure 2.1, harvested from farms in Uyo, Akwa Ibom State were de-milled with the empty husks collected to fill a 10-liter bucket. These were then sun-dried for five days and blended to powder using a Faberware model PC550 blending machine. The residue powder weighing 475g was collected.

2.1 Porosity and Bulk Density Tests on Unfired Clay

The as-mined chunks of brown and white clay weighing 2.5 kg each were crushed into powder and sieved through 500 µm mesh to remove extraneous organic contaminants. Furthermore, the clay powder was charged into an electric oven set at 110 OC to preheat the powder samples for 30 minutes.

2.1.1 Porosity Test

The equipment used to determine the porosity of clay, called OFITE Core Nitrogen Gas Porosimeter model 350, in Figure 2.5, operates on Boyles Law.

Clay powders were further shaped into cylindrical cores of diameter 40 mm and 50 mm length using procedures for core specimen preparation method adopted from [21]. Nitrogen gas was filled into the reference chamber with predetermined injection pressure P1. The reference chamber and sample chamber were connected to force gas molecules into sample pores. Pressure P2 was recorded in the sample chamber, and core grain volume VG, was calculated from the formula:

$$V_G = V_2 - V_3 \qquad \text{Eq. (2.1)}$$

Where,

$$V_3 = \frac{P_1 V_1}{P_2}$$

V2 is the porosimeter constant given as 155.14 cm3

Measurements of specimen bulk volume and calculated grain volume were used to calculate porosity (ϕ).

$$\text{Porosity, } \phi = \frac{V_B - V_G}{V_B} \times 100\% \qquad (2.2)$$

Where,

V_B - Bulk volume and V_G - Grain volume.

Figure 2.5: OFITE Core Nitrogen Gas Porosimeter

2.1.2 Bulk density test

Bulk density of clay powder was determined directly by measuring its mass in a 100 ml flask using an analytical weighing balance (Camry model no ACS-30-ZC41) and dividing with a known volume.

$$\text{Bulk Density} = \frac{Mass\ (g)}{Volume\ (cm^3)} \qquad \text{Eq. (2.3)}$$

2.2 Formulation of Clay and Melon Husk Mixture

Brown Ikot Ebom Itam (BIE) and White Ikot Ebom Itam (WIE) clay samples were separately dry-mixed with 500 µm sieved melon husk and grog using the proportions shown in Table 2.1. 100% clay was set as the control.

Table 2.1: Batch formulation of clay, melon husk powder, grog and the fired temperature

S/No	Code	Clay wt%	Melon Husk (MH) wt%	Grog wt%	Temp °C
1	MH25-650	70	25	5	650
2	MH25-700	70	25	5	700
3	MH25-750	70	25	5	750
4	MH25-800	70	25	5	800
5	MH25-850	70	25	5	850
6	MH25-900	70	25	5	900
7	MH25-1000	70	25	5	1000
8	MH20-650	75	20	5	650
9	MH20-700	75	20	5	700
10	MH20-750	75	20	5	750
11	MH20-800	75	20	5	800
12	MH20-850	75	20	5	850
13	MH20-900	75	20	5	900
14	MH20-1000	75	20	5	1000
15	MH15-650	80	15	5	650
16	MH15-700	80	15	5	700
17	MH15-750	80	15	5	750
18	MH15-800	80	15	5	800
19	MH15-850	80	15	5	850
20	MH15-900	80	15	5	900

21	MH15-1000	80	15	5	1000
22	Control-650	100	0	0	650
23	Control-700	100	0	0	700
24	Control-750	100	0	0	750
25	Control-800	100	0	0	800
26	Control-850	100	0	0	850
27	Control-900	100	0	0	900
28	Control-1000	100	0	0	1000

600 grams dry weight of 500 µm clay, melon husk, and grog in their predetermined ratios, as reflected above were mixed to leather hard consistency with the addition of 90 cm3 of water.

The mixture was wedged and rolled to achieve homogeneity. The plastic clays were shaped into cube dimensions 50 mm x 50 mm x 50 mm by filling into prefabricated aluminum pans.

Afterward, the pans were undressed, leaving behind the clay cubes, which were left to dry in air for 72 hours, which they were charged into a kiln and fired, Figure 2.4.

The firing of clays was achieved using a gas-fired brick kiln with a digital thermostat probe (model no TM 902C) to monitor the temperature. The firing consisted of different stages, namely, dehydration 100-300 OC (first 90 minutes), biomass burn off 400-500 OC (between 150 -180 min), and vitrification 600-1000 OC (between 240 – 300 min). Post-heat treatment included a soak time of 4 hours.

2.3 Apparent porosity and bulk density tests on fired porous ceramic body

The apparent porosity and bulk density tests were performed using the liquid displacement method based on Archimedes' principle (ASTM C20-87). The fired specimen's weight was weighed and recorded. The specimens were allowed to cool and then immersed in a beaker of water. Their soaked (saturated) weights were measured and recorded.

They were then suspended in a beaker one after the other using a sling, and their respective suspended weights were recorded. Dry weight was noted as D, Suspended weight S, while saturated weight was W.

The apparent porosity was determined using the formula:

$$\text{\% Apparent Porosity} = \frac{W-D}{W-S} \times 100\% \qquad \text{Eq.(2.4)}$$

While bulk density was determined using:

$$\text{Bulk Density} = \frac{D}{W-S} \ (g/cm^3) \qquad \text{Eq. (2.5)}$$

III. RESULTS

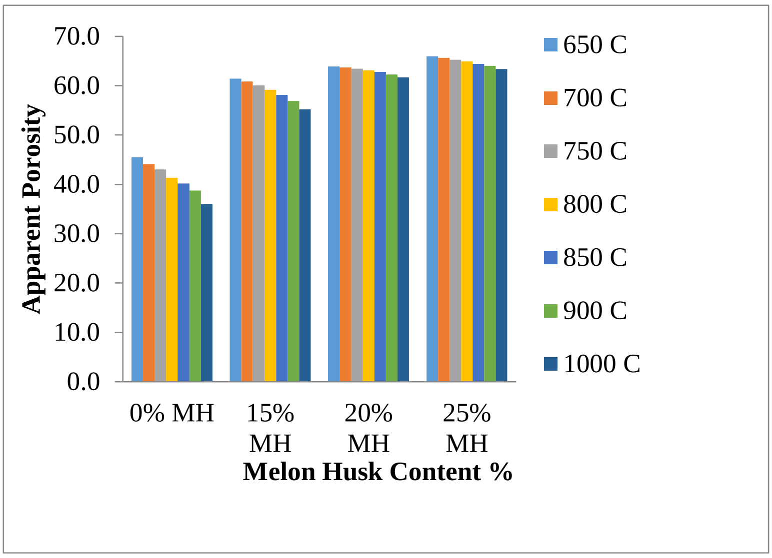

Figure 3.1: Plot of BIE Apparent Porosity as a Function of Melon Husk Composition

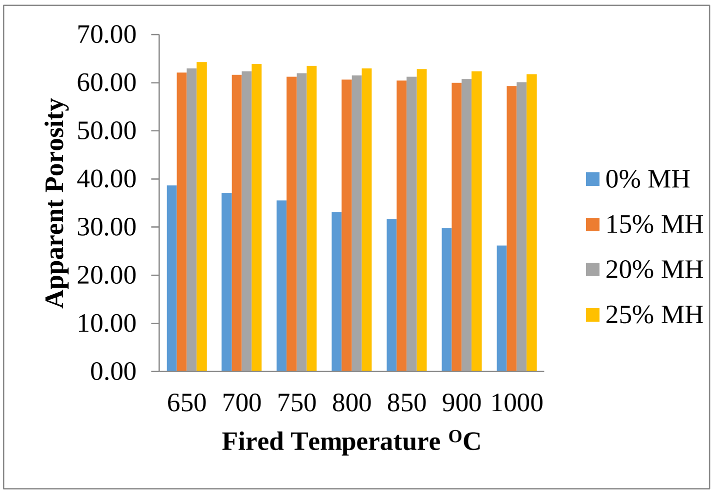

Figure 3.2: Plot of BIE Apparent Porosity as a Function of Temperature

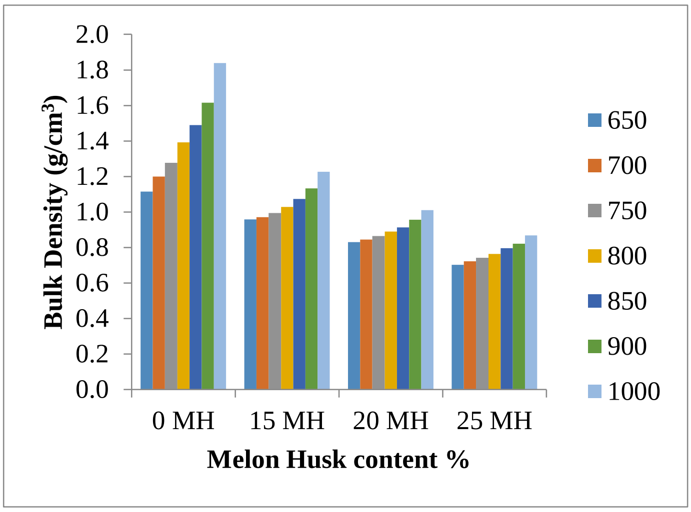

Figure 3.3: Plot of BIE Bulk Density as a Function of Melon Husk Composition

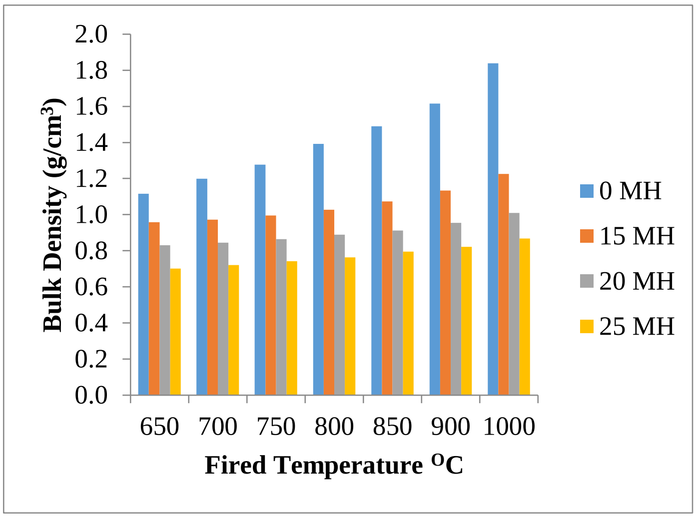

Figure 3.4: Plot of BIE Bulk Density as a Function of Temperature

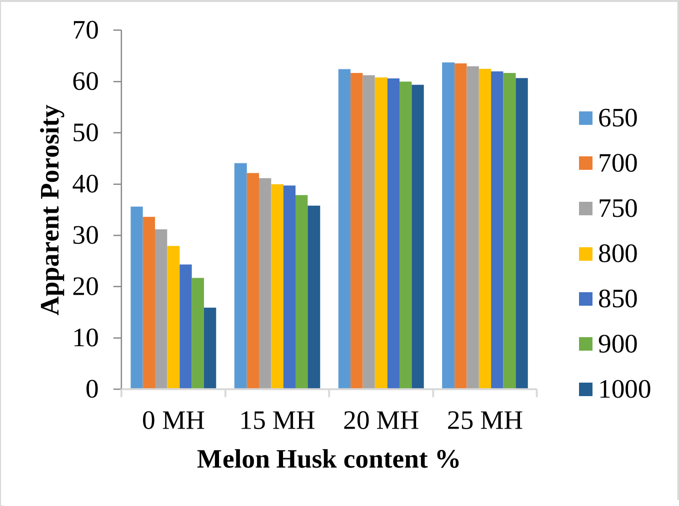

Figure 3.5: Plot of WIE Apparent Porosity as a Function of Melon Husk Composition

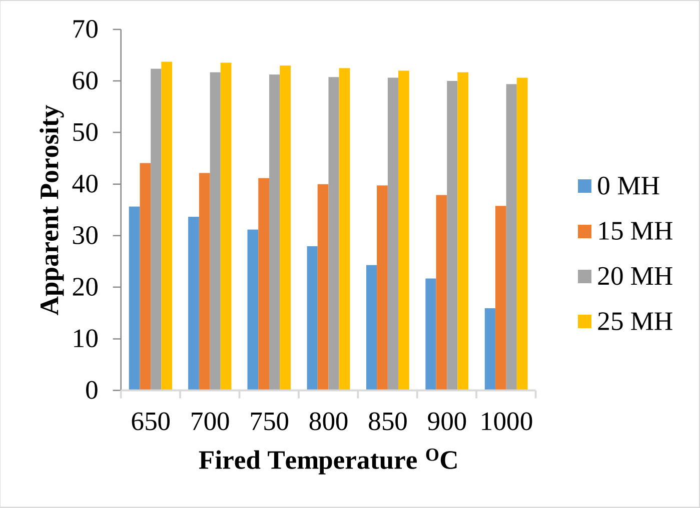

Figure 3.6: Plot of WIE Apparent Porosity as a Function of Temperature

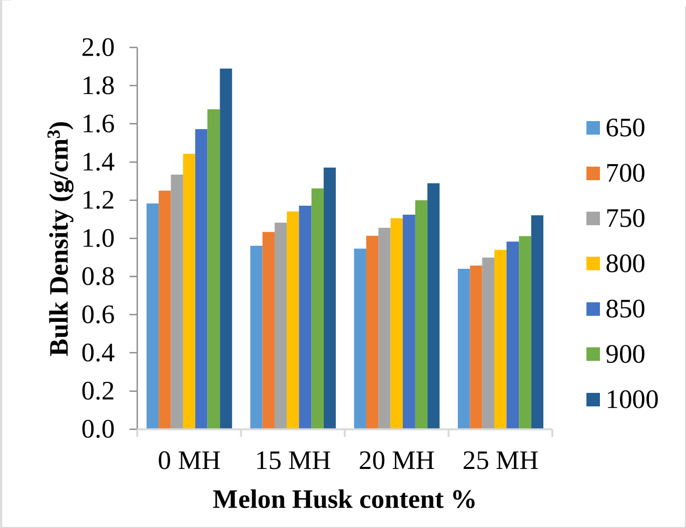

Figure 3.7: Plot of WIE Bulk Density as a Function of Melon Husk Composition

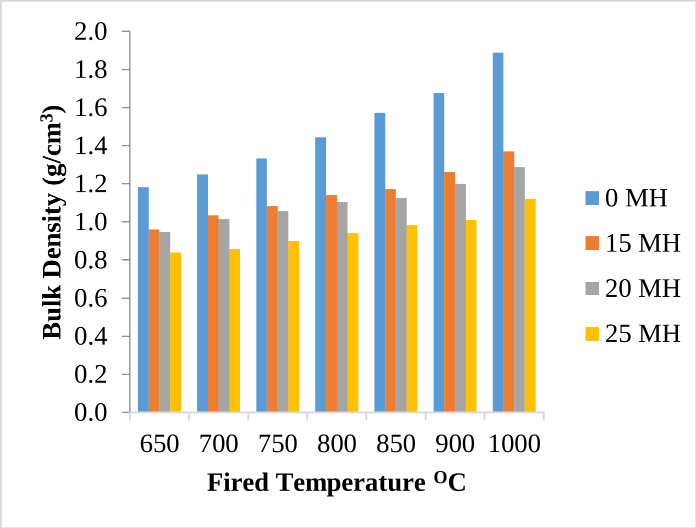

Figure 3.8: Plot of WIE bulk density as a function of temperature

The plots in Figures 3.1 to 3.8 represent the results of conducted tests on post-fired porosity and bulk density on the two test samples, BIE and WIE clay. It can be observed that the porosity values increased with increasing melon husk (MH) load, while bulk density values increased with increasing temperature. Changes in values of porosity and bulk density vary inversely by the increments of fired-off MH between 15, 20 and 25 weight percent content in the premixed clay. In contrast, the homogenous clay values constitute the lower limit boundary for porosity readings and the upper limit boundary for bulk density readings.

As MH loading increased, there was a significant impact upon rising porosity levels, particularly at the onset temperature of sintering (650 OC). At this temperature, incineration of the MH phase was completed, thus leaving behind additional pore spaces, which were largely unaffected by thermal-induced grain growth. Thus, the highest values of porosity recorded were obtained at 25 % MH preload, which corresponded to 65.99 % for BIE and 63.07 % for WIE porous ceramics.

As the kiln temperature rose beyond 650 OC, it was observed that the density of the ceramic body increased, which can be explained by the sintering mechanism of ceramic particles as clay grains diffuse across grain boundaries, leading to denser and closer packing of grains with resulting reduction in net porosity. Hence porosity loss during clay vitrification is defined by temperature rise. The mitigating effect of MH in predetermined loads premixed into the clay on the resultant porosity loss is seen upon the comparison of clay with MH to homogenous clay fired under identical conditions, Figures 3.1, 3.2, 3.5, and 3.6.

As the firing of the porous ceramic body progressed to 1000 OC, advanced stages of sintering set in. It was observed however, that the resultant effect of net pore spaces inherited from high preloads of incinerated MH lowered substantial densification in the premixed structure. This was apparent in BIE and WIE bodies with 20 % MH mixture. At this MH preload, the porosity levels of BIE was greater by a value of 2.33 % compared to WIE porous ceramic, with both samples attaining a marginal value of 60.535 ± 1.165 %, which was roughly twice the porosity value of the 1000 OC fired homogenous ceramic body with value 25.99 ± 10.05 %. Correspondingly, bulk density values recorded for produced porous ceramics at 1000 OC was observed to be higher in WIE with recorded results of 1.29 g/cm3 against 1.01 g/cm3 for the BIE body. Increases in bulk density imply strength and durability in ceramic wares production [21] [1], so it is predicted that the WIE porous ceramic will perform better than BIE during service applications irrespective of its lower porosity. It can be observed that the high preloads of MH pore former biomass mixed into the clay body do not affect the bulk density of fired clay; thus, this validates the combustible nature and fitness of its use as a pore former.

A plot comparison of averages in apparent porosities of BIE with WIE-fired clay in Figure 3.9 indicates their hierarchy of differences in the measured property. Their pre-fired values in apparent porosity and bulk density, as seen in Table 3.1, also underscore these differences.

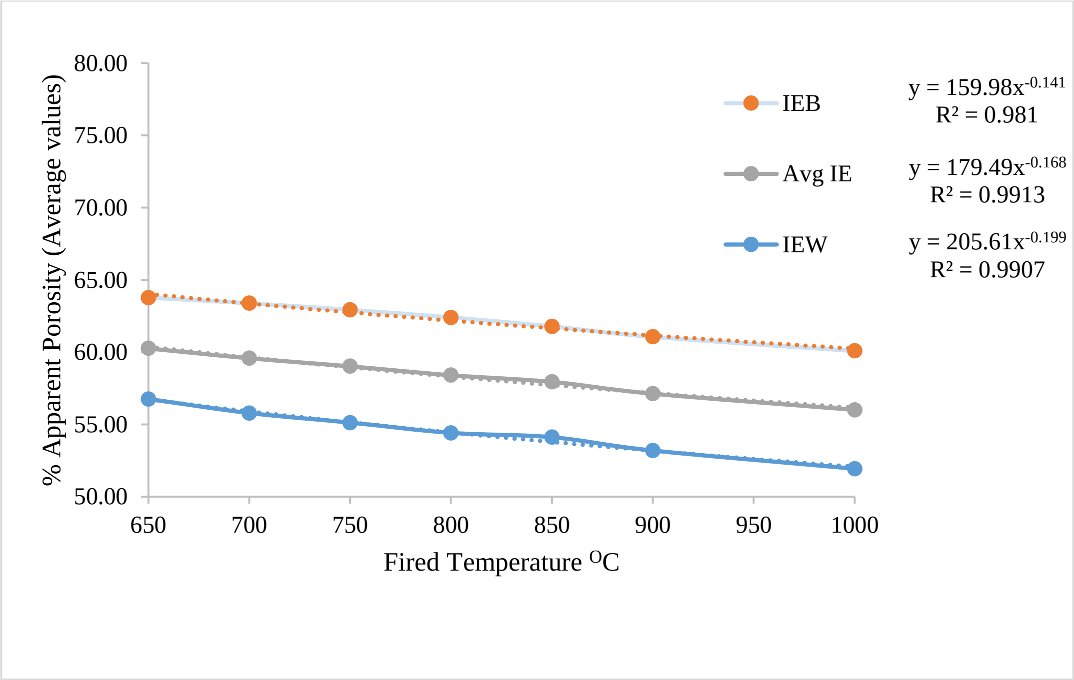

Figure 3.9: Plot of Apparent Porosity Mean Averages as a Function of Temperature for Ikot Ebom Porous Ceramics

Table 3.1: Summary of Porosity and Bulk Density Difference in Tested Clay Samples

	BIEclay				WIE clay			
Porosity at 110 ^{O}C	53.75%				50.34%			
Bulk Density at 110 ^{O}C	1.225 g/cm^3				1.315 g/cm^3			
Melon Husk content	0%	15%	20%	25%	0%	15%	20%	25%
Porosity at 1000 ^{O}C	36.04%	55.18%	61.7%	63.37%	15.94%	35.77%	59.37	60.65%
Bulk Density (g/cm^3) at 1000 ^{O}C	1.84	1.23	1.01	0.87	1.89	1.37	1.29	1.12

Table 3.2: Statistics of Porosity Values for BIE and WIE Porous Ceramics at Fired Temperatures

Temperature	Minimum	Median	Maximum	Mean Apparent Porosity	SE Mean	StDev	Sum of Squares	Range
1000^{O}C	35.77	60.01	63.37	56.01	4.20	10.29	19349.15	27.60
900 ^{O}C	37.86	60.84	64.03	57.12	3.98	9.74	20052.80	26.16
850 ^{O}C	39.72	61.30	64.41	57.94	3.75	9.18	20565.48	24.70
800 ^{O}C	39.96	61.63	64.91	58.40	3.78	9.25	20892.55	24.95
750 ^{O}C	41.15	62.10	65.28	59.02	3.65	8.94	21299.05	24.13
700 ^{O}C	42.16	62.59	65.64	59.58	3.55	8.70	21676.03	23.47
650 ^{O}C	44.10	63.07	65.99	60.26	3.29	8.07	22110.54	21.89

From statistical analysis using Minitab and a plot of mean apparent porosities of total MH preloads for BIE and WIE clays given in Table 3.2, a power law regression equation was determined by applying the dependent variable apparent porosity as a function of temperature.

The determined relationship for BIE and WIE porous ceramics in comparison to the averages between the two clays of one origin is given below:

$$\text{Porosity of Ikot Ebom (IE) Porous Ceramics} = 179.49\ \text{Temperature}^{-0.168} \qquad (3.1)$$

$$\text{Porosity of BIE Porous Ceramics} = 159.98\ \text{Temperature}^{-0.141} \qquad (3.2)$$

$$\text{Porosity of WIE Porous Ceramics} = 205.61\ \text{Temperature}^{-0.199} \qquad (3.3)$$

Rearranging the indices in the equations above to have uniform indices, the equations become:

$$\text{Porosity of IE Porous Ceramics} = \frac{220.83}{T^{0.2}} \qquad \text{Eq. (3.4)}$$

$$\text{Porosity of BIE Porous Ceramics} = \frac{234.44}{T^{0.2}} \qquad \text{Eq. (3.5)}$$

$$\text{Porosity of WIE Porous Ceramics} = \frac{206.95}{T^{0.2}} \qquad \text{Eq. (3.6)}$$

Where T represents temperature.

The power relationships of recorded porosity results for BIE and WIE porous ceramics make clear the reciprocity between apparent porosity and temperature for ceramics.

IV. CONCLUSION AND RECOMMENDATION

The production of porous ceramic bodies from locally sourced Ikot Ebom clays using MH biomass as pore former was successfully carried out. From the result of the investigation, the following conclusions were drawn:

1. Produced porous ceramics from clays used in the present research fired with MH biomass for burnout material possess a post-fired porosity marginal value of 60.535 ± 1.165 % at 20 % MH premix and 1000 OC attained temperature, and a statistically derived porosity relationship to temperature given as
$$P = \frac{220.83}{T^{0.2}}.$$
2. MH biomass does not add to the density of the fired ceramic, which infers that it is wholly incinerated at 650^{O} C and, therefore, suitable as poreformer.
3. Premixing clay with MH at minor increments up to 20 % enhances porosity in porous ceramics fired at 1000^{O} C to a value twice the porosity of homogenous fired clay.
4. At this fired temperature and MH mixture, WIE porous clay has a higher recorded bulk density than BIE, with 1.29 g/cm3 against 1.01 g/cm3. However, it is much lower compared to the WIE homogenous fired ceramic at 1.89 g/cm3. The implication of this is that further increasing MH content beyond 20% will significantly reduce the bulk density, which in turn could undermine the strength and durability of the structure.

It is recommended upon these premises that MH agro waste is suitable as pore former materials in producing highly porous ceramics with the porosity to the range of 60%. The recommended starting mixture for optimum balance with bulk density is 20 weight percent. At this mixture, suggested firing temperature is 1000^{O} C.

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