Sintering Temperature Dependent Variation of Compressive Strength with Porosity of Rice Husk Ash Ceramics

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Abstract

This paper present compressive strength variation with porosity of ceramic made with Rice Husk Ash as the major raw material. The ash powder used in this work was initially produced at a controlled temperature of 650^{0}C and the green strength ceramic disks formed by pressing method were then subsequently sintered at the temperature range of 1000^{o}C – 1400^{o}C. The chemical composition of the rice husk ash was determined by XRF and the crystalline phases by XRD. The rice husk ceramics formed at this temperature range retained the same $Si0_2$ purity of 82.80% and major Impurities such as P_2O_5, K_2O, and CaO. The result of X-ray diffraction shows the presence of two major forms of crystallographic phases; cristobalite and tridymite. Compressive strength increased with sintering temperature as porosity decreases. This result suggests a trade off on porosity for typical application like water filter design that requires combined high strength and porosity of the ceramic composite.

Keywords: Compressive strength, porosity, ceramics, rice husk ash.

1. Introduction

Rice is an important crop produced by many countries globally. Rice husk is a product of the beneficiation process in milling industries. Countries that produce rice today on large scale face disposal challenges [3]. This is a serious threat to the environment and it causes damage on the land [8]. Rice husk ash is produced by burning the husk. The ashes produced have low nutritious value, have chemical, mineralogical and morphological characteristics that depend on the equipment and parameters of the process during burning of the husk [4]. Therefore, the affected countries have been trying in different ways to save their environments by sponsoring research on how the husk and its ash can be put into beneficial uses. This is then paving ways towards minimizing the environmental problems. Amorphous silica is produced when rice husk ash is burnt at a controlled temperature of less or equal 700℃ . When rice husk ash is burnt at a temperature above 700℃, crystalline silica is produced. The major compound in rice husk ash is silica about 80% to 95% [1]. It is reported that the melting point of rice husk ash is 1440℃ [7]

Rice husk ash is abundant and cheap thus finds applications where high siliceous compound are needed. Examples of such applications include cement pozzolan [2], refractory bricks, thermal insulation and water purification. Most of the applications stems from the porous nature of high silica products. Domestic water filters contain silica exceeding 70% with porosity above 30% and still reasonably durable. This paper thus investigates compressive strength variation with porosity of rice husk ash ceramic at sintering temperature range of 1000^{0}C to 1400^{0}C.

2. Materials and Methods

2.1 Rice Husk Ash (RHA) production

Samples of rice husk were collected from the Middle Belt region of Nigeria. The husk was first burnt in an open environment. The ash produced at this stage was black. Cooling was also done in an open environment for 24hours, carbolite furnace model GPC12/81+103 with temperature range 0-1200 ℃ was used firing the ashes at a controlled temperature of 650℃. At this stage, white amorphous rice husk ash was produced. The chemical composition of the ashes was determined using energy dispersive X-ray fluorescence spectrometry minipal 4 model© 2005,pw 4025/45B panalytical B.V.

2.2 Formulation of Rectangular Blocks

Rice husk ash ceramics were formulated using rice husk ash as the major raw material, starch, sodium silicate and Bentonite as binders and plasticizer and water. The components were mixed manually for 15 minutes. The amount of each constituent of the ceramics paste was determined in terms of weight (mass). The water ratio was calculated based on the weight of the solid mass. The maximum amount of wood saw dust added was 15g to every 500g of RHA to avoid laminating effect during forming and firing (sintering). The ceramics were formed by the pressing method. The specimens were molded in rectangular form.

The RHA-binder composites produced were dried in an oven. The ceramics were sintered in a carbolite furnace model RHF 16/16 for a maximum period of six hours at the rate of 250℃/hour. At each particular sintering temperature the soaking time was two hours. The sintering temperatures were $1000℃, 1100℃, 1200℃, 1250℃, 1300℃ \; and \; 1400℃$

2.3 X-ray Diffraction Analysis

X-ray diffraction analysis was done on samples sintered in the temperature range of $1000\ ℃ - 1400℃$. X-ray mini diffractometer model MD-10 with $Cuk\alpha$ radiation of wavelength 1.5406nm was used for the analysis. The samples were exposed to X-ray generator running at 25kv. The 2θ angle for the machine ranges from $16° - 72$ The unknown samples were search matched with the known samples from the database available at International Center for Diffraction data (ICDD). Crystallite size was calculated using XRD result by means of scherrer equation.

2.4 Compressive Strength and Porosity Measurement

This test was carried out to measure the maximum of compressive load the ceramics can bear before fracturing. The test pieces which were rectangular in shape were compressed between the platens of a compressive testing machine.

The ceramics were first weighed and recorded as W_O. They were then immersed in water at room temperature and removed; the weight recorded as w_1. The rice husk ash ceramics were then allowed to absorb water to saturation; the weight recorded as w_2 .

Volume of pore spaces available= $W_2 - \mathbf{W_0}$

Volume of test pieces= $W_2 - W_1$

$$porosity = \left[\frac{w_2 - w_0}{(w_2 - w_1)\rho_w} \right] \times 100$$

Where ρ_w is the density of water (1gcm^{-3}). The test was done in accordance with ASTM C20-00

3. Results

3.1 Chemical Composition by XRF

Chemical composition of rice husk ash by XRF shows the presence of twenty-one compounds with silica having the highest percentage of 82.8%. The result is shown in table 1 below;

Table 1 Chemical Composition of Rice Husk Ash by XRF

Compound	Concentration (%)	Compound	Concent -ration (%)	Compound	Concen -tration (%)
$Si0_2$	82.800	$RU0_2$	0.275	$Zr0_2$	0.020
P_20_5	5.400	$S0_3$	0.200	Re_20_7	0.020
K_20	2.570	$Ti0_2$	0.110	Y_20_3	0.012
Ca0	1.660	Zn0	0.090	EU_20_3	0.010
Fe_20_3	0.836	Cu0	0.066	Cr_20_3	0.014
Mg0	0.800	Rb_20	0.038	Ni0	0.008
Mn0	0.321	Ba0	0.030		

3.2. Result of Compressive Strength and Porosity

The result of compressive strength and porosity at high temperatures is shown in the table below. As seen in the table, the compressive strength of the ceramics increase as porosity decreases with increase in sintering temperature

Table 2. Compressive Strength and Porosity of Rice Husk Ash Ceramics.

Sintering Temperature (^{o}C)	Apparent Porosity (%)	Compressive Strength (Mpa)
1000	60	2.75
1100	51	7.50
1200	40	8.60
1300	5	19.90
1400	4	27.50

The figure below is a graph of compressive strength against sintering temperature. The graph shows how compressive strength increases with increased sintering temperature of the rice husk ash ceramics.

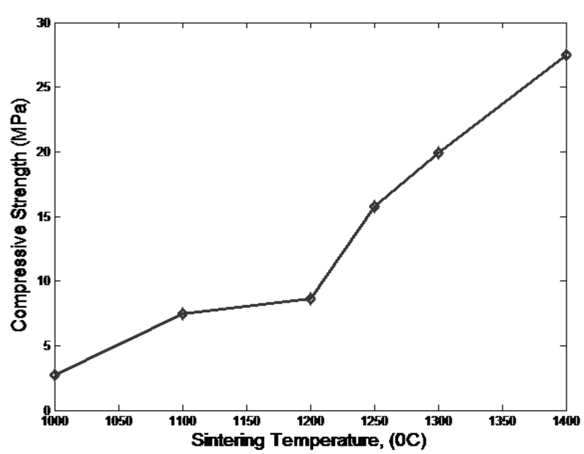
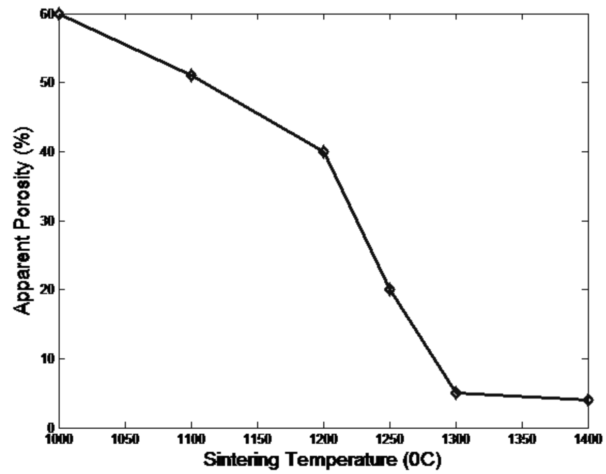


Fig 1: Graph of Compressive Strength against sintering temperature

The graph shown in the figure below shows how apparent porosity decreases with increase in sintering temperature of the rice husk ash ceramics.



\ Fig 2: Graph of Apparent Porosity against Sintering Temperature

Fig 3 below is a graph showing how apparent porosity and compressive strength are related at different sintering temperature. It can be seen from the graph that compressive strength of the rice husk ash ceramics increases as porosity of the ceramics decreases with increased sintering temperature. The intersection of porosity and compressive strength at the temperature of $1260^{0}C$ indicate that, at this temperature, porosity has been reduced to the minimum (below 20%) and the compressive strength rises above 15.75MPa. The RHA ceramics at this temperature are strong and hard. However, at temperatures below $1250^{0}C$, porosity is high above 20% and compressive strength is low below 9.0MPa. The ceramics at temperatures below $1250^{0}C$ are not strong and hard compared to those sintered at temperatures above.

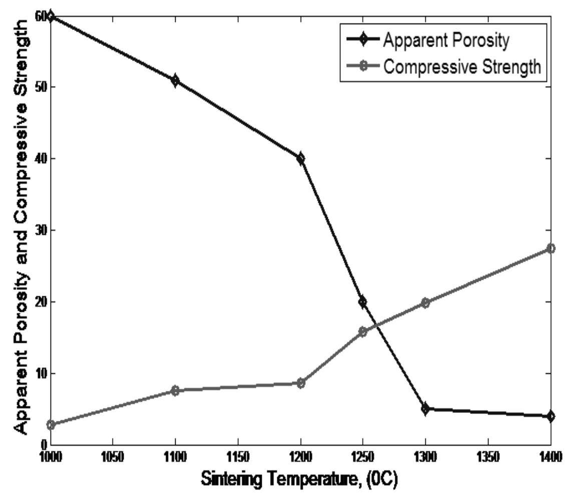


Fig 3. Correlation between Apparent Porosity, Compressive Strength and Sintering Temperature

3.3. X-ray Diffraction Result

The result of X-ray diffraction shown below shows the presence of two major crystallographic phases of silica cristobalite and tridymite. The amount of quartz is low and exists mainly at 1000^0C.

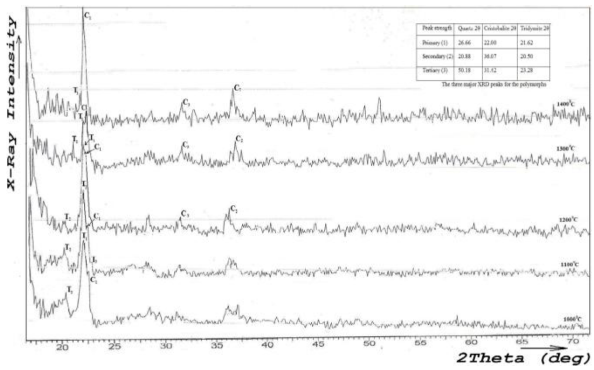


Fig. 1 X-ray Diffractograms for RHA Refractory in Temperature range 1000 ⁰C to1400 ⁰C (The symbols C and T are peak positions for Cristobalite and Tridymite respectively

4. Discussions

The rice husk ash used for the production of the ceramics was milky white in color with high percentage content of $Si0_2$ (82.80%). However major impurities such as $P_2 0_5, K_2 0, Ca0\ etc$ were present. Figure1, 2 and 3, shows an increase in compressive strength as porosity is reduced at different increased higher sintering temperatures. There is no significant change in the strength of the rice husk ash ceramics at sintering temperature $1000\text{-}1200^0$C. This is due to high porous structure (about 20%) of the ceramics which is capable of accommodating the volume change due to β- to - α cristobalite transformation [5]. The high strength seen at 1300^0C and 1400^0C shows that the strength of the ceramics is optimal at high elevated temperatures. The result obtained by XRD shows that cristobalite and tridymite are the major crystalline forms of silica at such elevated temperatures.

The decrease in porosity with increasing sintering temperature may be due to densification of low melting compounds of some of the impurities or decomposition and removal of some constituent which cause a reduction in volume

5. Conclusion

From the result, it can be concluded that the mechanism responsible for improved compressive strength of rice husk ash ceramics simultaneously reduced the porosity. At higher temperatures, porosity reduced to the minimum thereby increasing the strength of the ceramics. The ceramics at high temperature is mainly crystalline containing cristobalite and tridymite in high proportion.

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