# RICE HUSK ASH REFRACTORY: THE TEMPERATURE DEPENDENT CRYSTALLINE PHASE ASPECTS

**[1]A.D. Onojah, [2]N.A. Agbendeh & [3]C. Mbakaan**
[1,2]Department of Physics, Federal University of Agriculture, Makurdi, Benue State, Nigeria
[3]Department of science Laboratory Technology, Benue State Polytechnic, Ugbokolo, Nigeria

**ABSTRACT**
Cylindrical blocks of Rice Husk Ash thermal insulators were made using die pressing method. The husks were burnt in an open environment and the resulting black ash heated in a carbolite furnace at a controlled temperature of 650$^{O}$C. The initial impurities of the resulting milky white RHA were determined by XRF technique. The RHA were subsequently mixed with binders and sintered at a temperature range of 1000 to 1400$^{O}$C. X-ray diffraction (XRD) Spectroscopy was carried out to determine the major crystallographic phases. Two crystalline silica phases, tridymite and cristobalite were dominant. Crystallite size of the insulators at this range of temperature varies from 6nm to 10.02nm depending on the dominant phase.

**Keywords**: *Rice Husk Ash, Thermal insulators, X-ray diffraction, Silicon (IV) oxide ($SiO_2$).*

## 1. INTRODUCTION
A challenging problem faced by many rice producing countries of the world today is how a large quantity of husk available as a waste from rice milling stations can be properly disposed off. These countries have been trying to use this biomass in an economical manner [3]. Rice husk when burnt at low temperature below 700 $^{O}$C produces amorphous silica, while crystalline silica is produced at temperatures above 700 $^{O}$C [1]. Rice husk ash is a major source of silicon dioxide [2]. The ashes contain about 80% to 95% silicon dioxide and have low thermal conductivity and low mechanical properties. The ashes are also resistant to chemical etching [4] and have high melting point of 1440$^{0}$C [5].

Crystallization in rice husk ash begins at temperature above 500 $^{\circ}$C, below this temperature rice husk ash is purely amorphous. The phase transformation in rice husk ash is shown below.

$$\text{Amorphous Silica} \xrightarrow{600\,^{O}C} \text{Quartz} \xrightarrow{800\,^{O}C} \text{Tridymite} \xrightarrow{1470\,^{O}C} \text{Cristobalite}$$

The chemical properties of rice husk ash vary from one region to another. This variation is due to the condition under which rice is grown. These conditions include climate, soil, paddy (rice) variety and use of fertilizers [6]. Due to various properties of rice husk ash; the ashes are used as high temperature insulators and have found applications in industrial processes such as an active pozzolan in the cement and concrete industry, kiln and steel furnace refractory lining .

The main focus of this work is on the crystallographic phases of RHA refractory in the temperature range of 1000 to 1400$^{o}$C. Most silica refractory used in kilns, steel furnace and copula are operated in these temperature ranges.

## 2. MATERIALS AND METHODS

### 2.1. Rice Husk Ash 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 Cylindrical Insulator Blocks
Rice husk ash thermal insulators 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 insulator 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 insulators were formed by the pressing method. The specimens were molded in cylindrical form.
The RHA-binder composites produced were dried in an oven. The thermal insulators were sintered in a carbolite furnace model RHF 16/16 for a maximum period of six hours at the rate of 250°C/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.

## 3. RESULTS

The chemical composition of rice husk ash burnt at 650C measured by XRF is shown in the table 1. The XRD spectra for the five samples are in figure 1. The computed crystallite sizes in table 2 and corresponding plot on figure 2

Table 1: Chemical composition of Rice Husk Ash by XRF

| Compound | Concentration (%) | Compound | Concentration (%) | Compound | Concentration (%) |
|---|---|---|---|---|---|
| $Si0_2$ | 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 | | |

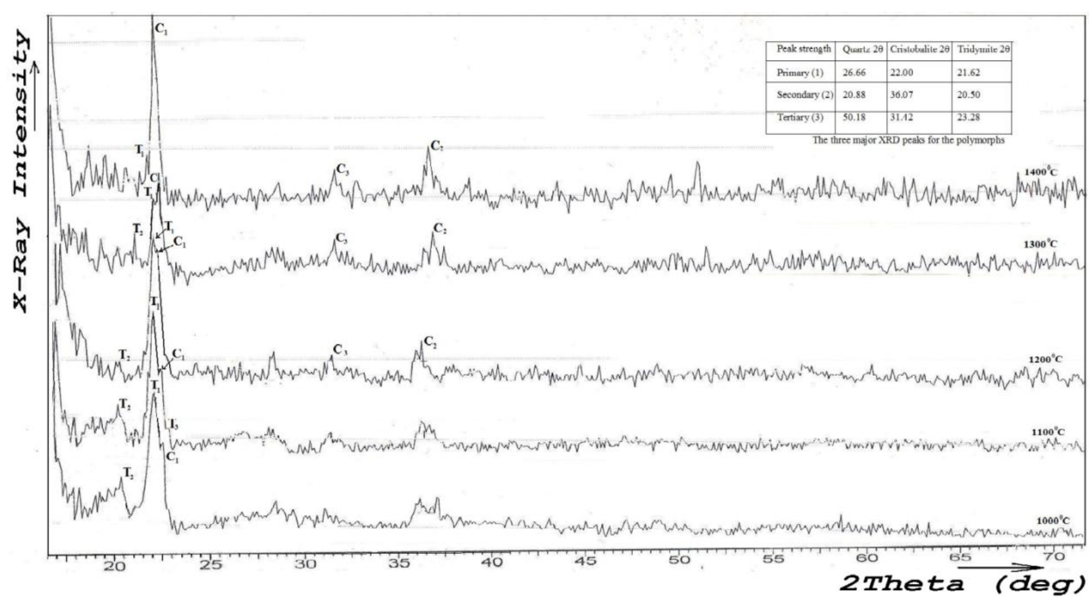


Fig. 1 X-ray Diffractograms for RHA Refractory in Temperature range 1000 °C to1400 °C (The symbols C and T are peak positions for Cristobalite and Tridymite respectively)

Table 2. Crystallite size of Rice Husk Ash Thermal Insulators Sintered at high temperature Calculated using XRD Result by means of scherrer equation

| Sintered Temperature (℃) | 1000 | 1100 | 1200 | 1250 | 1300 | 1400 |
|---|---|---|---|---|---|---|
| Crystallite size (nm) | 10.07 | 10.06 | 6.33 | 10.12 | 10.11 | 10.00 |

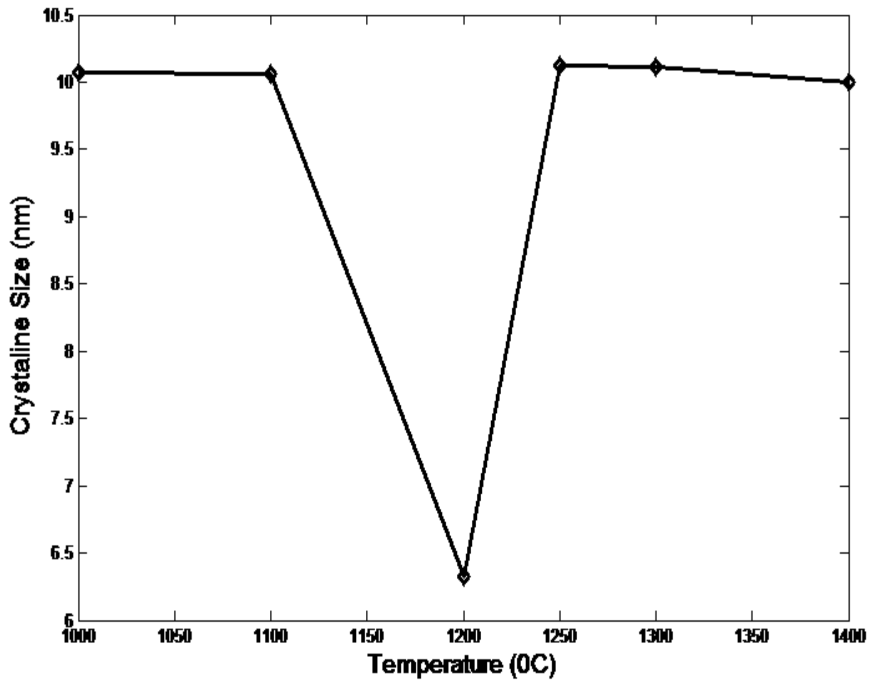


Fig2. Plot of Crystallite Size against Sintering Temperature

## 4. DISCUSSIONS

XRD result shows the presence of crystalline silica ($Si0_2$) as the major compound in the rice husk ash Refractory. The polymorphs of silica present include cristobalite and tridymite. In the temperature range of $1000℃ - 1200℃$ cristobalite dominates the diffractogram while from the temperature of 1250℃ to 1400℃, tridymite peaks became prominent. Result of crystallite size of the rice husk ash thermal insulators obtained from XRD result by means of scherrer equation shows a decrease in particle size from $1000℃\ (10.07nm)\ to\ 1100℃\ (10.06nm)$ and a sharp decrease at $1200℃$ (6.33nm). However, there is an increase in crystallite size at 1300℃ (10.11nm) and a decrease at 1400℃ (10.00nm). The decrease may be attributed to phase transformation from cristobalite to tridymite or due to glass formation about to occur or the approach to melting point. The melting point of rice husk ash is 1440℃ .

## 5. CONCLUSIONS

From XRF result, it can be concluded that the major compound in Rice Husk Ash Thermal Insulators is Silicon (IV) oxide ($Si0_2$). The polymorphs of silica present in the $Si0_2$ are cristobalite and tridymite at all the sintering temperatures ($1000℃ - 1400℃$ ). Cristobalite is the major phase from $1000℃$ to 1200℃ while tridymite phase become prominent from $1200℃ - 1400℃$.

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