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### CONVENTIONAL SOLID STATE TECHNIQUE FOR THE PREPARATION OF WHITE SILICA FROM WASTE RICE HUSK

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#### ABSTRACT

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In this work, the effects of acid treatment on waste rice husk (WRH) for the removal of metallic constituents associated to it was studied. The WRH contains showed the weight percentages of silica and other metallic oxides substances. WRH Leached with hydrochloric acid shown to be effective significantly in removing most of the metallic constituents. The WRH heated to 700 °C for six consecutive hours, for a complete combustion of WRH, which formed a white rice husk ash (WRHA). The ash residues acquired from acid treatment were completely white colour in nature. Hence, the acid treatment did not affect the amorphicity of the silica.

#### INTRODUCTION

Waste rice husk (WRH) is an agricultural deposit in large quantities existing in rice producing countries such as Nigeria. The annual WRH produced in Nigeria amounts to more than 3 million tons. As an agricultural waste material WRH is a tough material with excessive silica content. It is the most under-utilized biomass resources, exists as an ideal fuel for electricity generation which produces heat energy through direct combustion and gasification. The main ingredients of WRH are lignin, cellulose and ash which have higher content compared to other biomass fuels. The definite composition is flexible, normally: pentosans, 18%; ash, 20%; cellulose, 38%; lignin, 22%; and other organics, 2% (Matori et al. 2009; Noushad et al., 2012). WRH comprises silica

ash in the range of 20–25 wt% (El Mir et al. 2015; El Ghouli et al., 2013). WRH contains silica (SiO<sub>2</sub>) that occurs in the hydrated amorphous form like silica gel. Pyrolysis and thermal degradation of WRH, trailed by combustion of the burnt, results in extremely porous and amorphous silica with a changing percentage of unburnt carbon (Eraiah & Bhat, 2007). Combusted at reasonable temperature, the white ash acquired from WRH covers approximately 92–97% of amorphous silica (Uegaito et al., 2011) and some quantity of metallic impurities that can be removed by acid-leaching treatment. It reports that acid leaching of the WRH helps to achieve quite pure silica with great specific surface area (Cui et al., 2015).

Leaching is a process of removing impurities from the materials by dissolving them away from the solids or an extraction of certain materials from a carrier into a liquid. The chemical industries use organic solvents for leaching processes. Leaching has numerous applications, which include separation of metal from ore by using acid and separation of sugar from beets by using hot water. The major theory of leaching deserts mechanisms for retaining the material on the solid. Even though ion exchange and adsorption can fix materials tightly to solids. The analysis is simplified by considering only the dissolution of soluble ingredients away from an insoluble solid such as removing salt from sand by extraction with water. One of the most significant techniques of instrumental elemental analysis is inductively coupled plasma (ICP). This technique is to determine over 70 elements in a range of matrices. In the current research, the ICP and Optical Emission Spectrometry (OES) machine are going to be used in order to trace the elements that contain in WRH. Again, the ICP-OES is a form of emission spectrometry (same with ICP-AES) that uses plasma to yield excited atom that discharges electromagnetic radiation at a wavelength specific to particular element (Philipp & Ehrenreich, 1963). The strength of this emission is revealing the concentration of the element within the sample. The silica in the (WRHA) ash experiences structural transformations depending on the temperature establishment it goes through during combustion. The analyses show the highest quantity of amorphous silica presents

in WRH burn combusted at 700 °C, and at higher temperature, crystalline silica is formed (Chen et al., 2012). In regard to thermal treatment, the amorphous silica changes to crystalline phase of  $\beta$ -tridymite at 870-1470°C and  $\beta$ -cristobalite at 1470-1710°C (Weidenkaff et al., 2001). However, under controlled burning conditions of WRH, large surface area, amorphous silica with high reactivity and ultra-fine size is produced. The micro silica is a source for making advanced materials like elemental Si,  $Mg_2Si$ , SiC and  $Si_3N_4$ . Because of the high pozzolanic activity, the WRHA silica also has an application in high strength concrete as a substitute for silica fume. Chandrasekhar and co has mentioned the possibility of using this silica as filler in polymers- workers (Ergül, 2010).

#### METHODOLOGY

##### Sample Preparation

The WRH was washed thoroughly with distilled water in order to remove the adhering soil and dust. The WRH was then leached with 0.5M hydrochloric acid for an hour. The leached WRH was allowed to dry in open air for 24 h, and then transferred in to an oven at a temperature of 100 °C for another 24 h. The dried leached and unleached WRH were transferred in to an electric furnace with different crucibles. The samples were heated to 700 °C with heating rate of 10 °C per minute for six consecutive hours. The unleached or untreated WRH sample was acting as a control experiment. The leached and unleached WRH samples were sintered at 900 °C as shown in Figure 1.0.



(1) Unleached WRH



(2) Leached WRH



(3) Oven at 100 °C/24h



(6) Unleached WRHA powder (5) Leached WRHA powder (4) 700 °C/6h (10°C/min)  
**Figure 1.0: Preparations of white rice husk ash (WRHA)**

**Sample Characterization and Analysis**

On behalf of XRD, powdered form of the sintered samples were scanned through 2θ from 20 to 80°, at a scanning rate of 1°/s, by using an X-ray Diffractometer (PANalytical (Philips) X’Pert Pro PW3050/60) (with Cu Kα radiation, λ = 0.154 nm) at 40 kV and 30 mA. Hence, the apparent porosity was found out conferring to ASTM C 134/95, apparent density according to ASTM C 773/88.

**RESULTS AND DISCUSSION**

Table 1 illustrated the percentage elements for leached and unleached WRH. Comparing the leached samples and untreated WRH proved effective in removing some metals to a lower level. The percentage of the silicon dioxide (SiO<sub>2</sub>) in untreated WRH was 93.55 wt.%. Within the metallic elements in WRHA, the percentage of K<sub>2</sub>O was highest with 1.77 wt.%, followed by 1.64 wt.% SO<sub>3</sub> and 1.44 wt.% CaO.

The percentage of SiO<sub>2</sub> obtained from RH treated with (Babu & Buddhudu, 2013) is the highest (98.202 wt.%) compared to untreated WRH. This outcome was coherent with the work of Condradt and co-workers , who established the fact that WRH leached with HCl possessed the highest percentage of SiO<sub>2</sub> compared to untreated WRH samples. This result was also coherent to the work found by Chakraverty and co- workers (El Ghoul et al., 2010; Dembski et al., 2011) who accomplished that HCl was greater reagent for removing metallic elements in WRH. This occurred due to the anion (Cl<sup>-</sup>) from HCl which sheared the electrons with silicon to form silicon chloride (SiCl<sub>4</sub>) during the leaching process. Tetraoxosilicon found insoluble as such during leaching process silicon element was not removed. The minute percentage of Na 0.10 wt.% was added to react with insoluble SiCl<sub>4</sub> to form aqueous.

**Table 5.1: Elemental weightcompositions of WRHA**

Oxide Elements	Leached WRHA (wt.%)	Unleached WRHA (wt.%)
SiO <sub>2</sub>	98.202	93.550
SO <sub>3</sub>	00.495	01.640
CaO	00.473	01.118
ZnO	00.278	00.357
K <sub>2</sub> O	00.253	01.770
Fe <sub>2</sub> O <sub>3</sub>	00.202	00.450
MnO	00.054	00.570
CuO	00.043	00.545
TOTAL Weight Percentage (%)	100.00	100.00

**CONCLUSION**

In this investigation, the chemical contents of WRHA were established by using ICP. Hydrochloric acid was used in the leaching process of the WRHA samples. ICP data

displays that, samples that have experienced acid leaching contained a lower percentage of metallic elements compared to untreated sample. HCl leaching of waste rice husk takes a lead compared to H<sub>2</sub>SO<sub>4</sub> and HNO<sub>3</sub> from other

research for removing the metallic elements. Hence, it contains more percentage of SiO<sub>2</sub> compared to untreated WRHA sample. Concerning the heat treatment, the required combustion temperature is 700 °C, which produced white ash within a reasonable time (~6hrs in this research) and sintered at 900 °C

for 1hr. The XRD microgram show sthat the acid does not affect the amorphicity of the silica. Moreover, the peaks (cristobalite) gained from untreated husk is produced by the metallic elements that confined in the sample which might affect the structure of SiO<sub>2</sub>.

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