

# CHARACTERIZATION OF SILICA EXTRACTED FROM POST HARVEST RESIDUE

R. M. S. Virajini, V. P. S. Perera and J. C. N. Rajendra\*

*Department of Physics, The Open University of Sri Lanka, Nawala, Nugegoda*

## INTRODUCTION

Silicon is one of the most widely used resources in modern technology. Industrially speaking silicon is the basis of semiconductors, glasses, ceramics, insulators, moisture shields, fillers, solar cells, etc. These devices require pure silica, which is currently produced by smelting and high end techniques. Development of a simple low energy and cost effective production of pure silica is desirable and welcome by many industrial application.

In recent years, several researchers (Kalapathy *et al.*, 2000; Nittaya Thuadaij *et al.*, 2008; Dissanayake *et al.*, 2013) have reported that the production of pure nanocrystalline Si and SiO<sub>2</sub> from Rice Husk Ash (RHA) is possible and these RHA constitute 80 - 90 % of silica. This has led us to believe that a similar standard of silica may also be existing in other post-harvest residues such as coconut husk and rice straw. Very few investigations have been done in Sri Lanka to extract silica from post-harvest residues (Ismail and Lokuliyana, 1983; Lianage *et al.*, 1991 -1993).

The objective of this study is to conduct a preliminarily investigation of the physical properties of Sri Lankan based post-harvest residues such as Rice husk, Coconut husk and Rice straw, and to extract silica from them to compare and see whether these could be further synthesize for possible usage in industry.

## METHODOLOGY

### Moisture content

Standard oven dried method was used to determine the moisture content of each post-harvest residues used in this study. An empty aluminum pan was weighed using an electronic balance to the nearest 0.1 mg. The grounded residue was then placed on the pan and its wet weight was measured. The pan containing residue was heated in an aluminum pan at 125 °C until a constant weight was achieved. The pan containing the dried material was cooled to room temperature in a desiccator and then its dry weight was measured. The moisture content of the residue was determined using the equation,  $MC = \left( \frac{W_{wet} - W_{dry}}{W_{wet}} \right) \times 100$ , where,  $MC$  is the moisture content (%),  $W_{wet}$  is the wet weight of the residue and pan, and  $W_{dry}$  is the dry weight of the residue and pan.

### Bulk density

An empty container (7 ml) was weighed using an electronic balance to the nearest 0.1 mg. The container was filled with the post-harvest residue used in this study and was slightly compressed to ensure absence of large void spaces. The container and the residue were then weighed. The wet bulk density was determined using the equation,  $\rho_{bulk} = \frac{W_2 - W_1}{V}$ , where,  $\rho_{bulk}$  is the bulk density,  $W_2$  is the weight of the container and residue,  $W_1$  is the weight of the container and  $V$  is the volume of the container

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\* All correspondence should be addressed to Prof. J. C. N. Rajendra, Department of Physics, Open University of Sri Lanka (e-mail: jcr@ou.ac.lk)

## Porosity

Standard water pycnometer method was used to determine the porosity of each post-harvest residue used in this study. A sample residue of approximately 5 ml was taken in a 10 ml graduated cylinder. To avoid floating of residue, a wire mesh was used to cover the top. Distilled water (5 ml) was then poured gradually until the water level was above the residue. Cylinder was gently rocked several times to remove air bubbles trapped inside. The amount of added distilled water and the settled water level in the cylinder containing residue were recorded. The percentage of porosity of the residue was determined using the equation,

$$P(\%) = \left( \frac{V_i - V_f}{V_s} \right) \times 100, \text{ where, } P \text{ is the porosity of the residue (\%), } V_i \text{ is the initial total}$$

volume of the residue and the added water,  $V_f$  is the final total volume of the residue and the added water,  $V_s$  is the volume of the residue.

## Sample preparation to extract silica

The raw post-harvest residue was washed and burnt in air until it became black ash. This black ash residue was further put into a muffle furnace at 700 °C for 2 hours until it became white ash. This white ash sample was used to extract silica. This process was done separately for each raw post-harvest residue and the samples (Rice Husk Ash (RHA), Coconut Husk Ash (CHA) and Rice Straw Ash (RSA)) were obtained.

## Thermogravimetric analysis (TGA)

Thermogravimetric analysis was done for each post-harvest residue to observe their mass reduction in different stages of combustion. All the measurements were taken in the temperature range of 25°C to 700°C at a heating rate of 10 °C/min.

## Extraction of silica

Extraction of silica was done for RHA, CHA and RSA samples separately in the following method. Ten grams of each sample was stirred in 80 ml distilled 2.5 M sodium hydroxide solutions. These were then boiled in a covered 250 ml Erlenmeyer flask separately for 3 hours. The solutions were filtered using the Whatman No. 41 ashless filter paper and the respective residues were washed with 20 ml boiling water separately. Each filtrate was allowed to cool down to room temperature and added 5 M H<sub>2</sub>SO<sub>4</sub> until they reach pH 2. Then NH<sub>4</sub>OH was added to each filtrate until they reach pH 8.5 and allowed to be at room temperature for 3.5 hours. The silica as residue was separated from each suspension by suction filtration using a Buchner funnel and thoroughly washed. Each residue was then oven dried at 120 °C for 12 hours and cooled down to room temperature.

## XRD analysis

X-ray diffraction (XRD) patterns of extracted silica samples from RHA, CHA and RSA were obtained by a XRD system using an acceleration voltage of 40 kV and current 30 mA. The diffraction angle  $2\theta$  was scanned 10° to 80° at a rate of 4°/min.

## RESULTS AND DISCUSSION

The results of moisture content, bulk density and porosity of the raw post-harvest residues are presented in Table. 1. The moisture content of the rice husk obtained in this study is 10.4 % and this value is comparable to the value of 10.7% reported by Zhou *et al* (2009) for the rice husk from China, the value of 8.7 – 10.5 % reported by Ismail *et al* (1983) for various rice husk from Sri Lanka and the value of 9.38% reported by Olawale *et al* (2012) for the rice husk from Nigeria. However, it is lower than the values reported by Yaning Zhang *et al* (2012) for short grain 5.63% and long grain 4.72% rice husk. This lower value of moisture content may be mainly due to drying procedure. The sample used in this study was oven dried

at 125°C whereas the samples reported by Yaning Zhang *et al* (2012) were dried at 105°C until a constant weight is achieved. The moisture content of the rice straw is 14.8% and the coconut husk is 12.7%. These values are higher than the value obtained for the rice husk under the same condition and processing method.

Table 1: Moisture content, the bulk density and porosity of the post-harvest residues

Post-harvest residues	Moisture content (%)	Bulk density (kg/m <sup>3</sup> )	Porosity (%)
Rice husk	10.4	351.4	54
Coconut husk	12.7	198.4	60
Rice straw	14.8	220.8	56

The moisture content of 14.8 % estimated for the rice straw in this study is much higher than the values reported by Yaning Zhang *et al.*, (2012) for short grain 6.92% and long grain 6.58%. These high moisture content variations may be due to drying procedure, geographical condition of the rice production and the variety. The bulk density obtained in this study for rice husk and rice straw are 351.4 kg/m<sup>3</sup> and 220.8 kg/m<sup>3</sup> respectively. These values are comparable to the values of 377.24 kg/m<sup>3</sup> and 166.29 kg/m<sup>3</sup> for long grain rice husk and rice straw respectively obtained by Yaning Zhang *et al.*, (2012). Slight variation of these values may be due to different varieties of rice straw used in these studies and their different geographical location in the production of paddy. Figure 1 shows the Thermal Gravimetry Analysis (TGA) of rice husk, coconut husk and rice straw. In all three samples, two or more stages of decomposition regions were observed. First region may be due to the removal of water and the last region represents the burning of fixed carbon in the samples. Except rice husk other two samples (coconut husk and the straw) seems to have undergone multiple stage decomposition indicating the removal of other kinds of volatile components. Table 2 shows the onset and offset temperatures associated with the combustions and the mass loss.

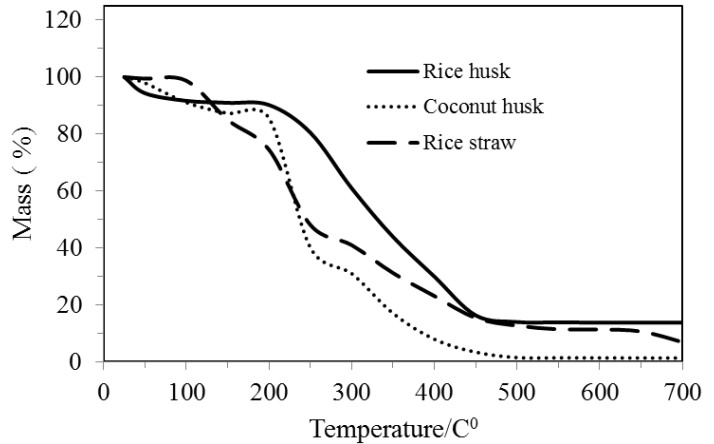


Figure 1: TGA curves for Rice husk, Coconut husk and Rice straw

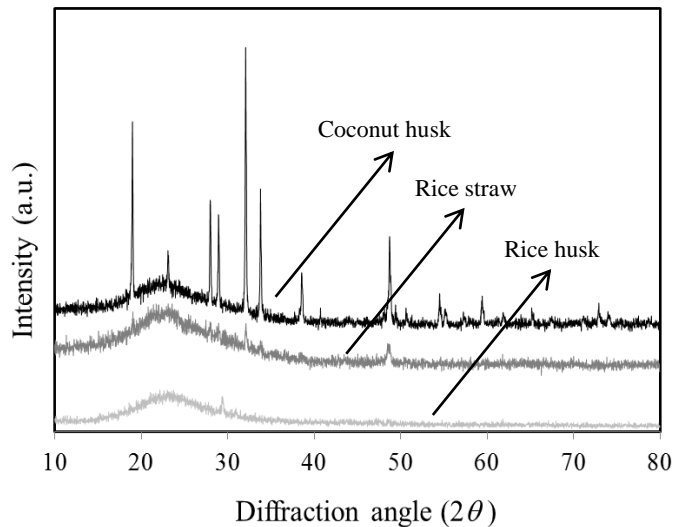


Figure 2: XRD spectra of silica from RHA, CHA and RSA

Table 2: Onset and offset temperature associated with the combustion and the mass loss

Post-harvest residues	Onset combustion Temperature	Off set combustion Temperature	Mass loss (%)
Rice husk	200°C	450°C	85.0
Coconut husk	75°C	425°C	98.5
Rice straw	100°C	450°C	90.0

TG graph reveals (Figure1) that the water content in the rice husk released in the temperature interval 25<sup>o</sup>C - 120<sup>o</sup>C is around 10%. It also shows clearly that the final residues obtained in the form of ash from rice husk, coconut husk and rice straw are 15%, 1.5% and 10% respectively. XRD spectra of silica obtained from these ashes (Figure2) shows broader peaks centred at 2 $\theta$  angle of 22<sup>o</sup> confirmed the amorphous nature of the silica (Kalapathy *et al.*, 2000 and Dissanayake *et al.*, 2013). The peak associated with rice husk is much broader than the other two. The initial analysis of XRD spectra using the Scherrer's formula reveals that the rice husk has particles in the range of nano scale, however further investigation is needed for conformation. Further the XRD spectra indicates that the quantity of silica in percentage obtained in rice straw and coconut husk are much lower than obtained in rice husk.

## CONCLUSIONS/RECOMMENDATIONS

The data obtained in this study shows that rice husk is a more suitable post-harvest waste material than coconut husk and rice straw for further investigation to obtain industrial standard silica having particle size possibly in the range of nano scale.

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