

Study the Silica Content of Paddy Husks from Different Varieties of Paddy Cultivated in North Western Province, Sri Lanka

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Abstract – Rice husk is one of the main agricultural waste available in Sri Lanka. They are the outer covering of rice grains. Once the paddy is separated from rice grain, the husk is removed from rest of the grain. This study show depending on rice husk variety moisture content, average bulk density, amount of precipitated silica varies. Further EDX analysis show depending on variety percentage of Oxygen, Silicon and other contaminants are varied. Eight different rice husk varieties available in North Western Province is studied in this research. Moisture Content (MC), average bulk density and silica content in each variety were initially calculated. Moisture content varies between 10.42% to 12.84%. Average bulk density varies between 214.42 kg m^{-3} to 268.57 kg m^{-3} . Highest amount of silica was obtained as 98.2% from Murugakanayan rice variety. Lowest amount of silica was obtained as 74% from BG 360 rice variety. A typical XRD, SEM patterns and EDX was done to silica samples obtained from rice husk ash. Amorphous nature of the obtained silica was confirmed from XRD spectra. There is a broad peak with maximum intensity at $2\theta = 22^\circ$ in XRD spectra. SEM images have shown that these silica particles were aggregated as irregular rod shape clusters. EDX images confirm high amount of sample weight contain silicon and oxygen. Yet very less amount of sodium is available in the sample.

Keywords - Rice Husk, Rice Husk Ash, Silica, Extraction

1. INTRODUCTION

Rice is one of the main crops in the world (Sun, 2001). Each year 600 million tons of rice is produced in the world, while in Sri Lanka 2-3 million tons of rice is produced (Ajward, 2017). Rice is the seed of the grass species *oryzaglaberrima* (African rice) or *oryza sativa* (Asian rice). As a cereal grain, it is the most widely consumed staple food for a large part of the world's human population, especially in Asia and Africa.

According to the variety and geographical location, the percentage of Rice Husk (RH) varies between 14% to 27%. As a fair average it can be taken as 20% (Perera, *et al.*, 2014). RH is outer covering sheaths that cover the rice grains which are removed during the refining process (JinHyung Lee, 2017). RH is one of the most widely available by product in rice producing countries throughout the world (Batteggazzore, *et al.*). Major constituent of RH is lignin, cellulose, hemicellulose and hydrated silica. These are

varying with variety, climate and geographical location of growth (Batteggazzore, *et al.*).

Usually a large amount of RH is burnt creating ash which causes damage to both land and environment. Burning RH provides about 25% of rice husk Ash (Bakar, *et al.*, 2016). When the burning temperature of RH is not uniform, it may yield two types of RHA. Those are Black Rice Husk Ash (BRHA), White Rice Husk Ash (WRHA) (Rathnayake, 2018). The upper layer of RH is exposed to open air and BRHA is formed in the carbonized layer. Inner layer is exposed to high temperature and the carbonized layer is oxidized and WRHA is yield which is predominant in silica. Ash which has undergone maximum extent of combustion contains a higher amount of silica. It appears in white-gray color. Black color ash is obtained from incomplete combustion. Carbon present in this black ash hinders silica digestion reaction process and product characteristics may change. It is important to select white-gray ash for this process (Mittal, 1997).

Kalapathy has proposed a new route for producing silica, suspending RH in sodium hydroxide and then precipitating it with an acid for extracting silica (Kalapathy, 2000). Silica exists as a gel, crystalline or amorphous form (Todkar, 2016). It has been reported that at 600°C to 1000°C amorphous silica is formed. At high temperature crystalline silica is formed (Bakar, *et al.*, 2016).

Once silica produced from plant origins such as RH have been noted to have some significant advantages over those from mineral and synthetic origins. It has been observed that final silica powder produced from plant sources contain a narrow range of metal impurities, which provide high purity silica at modest cost (Zemnukhova, 2006).

Liou (2004) states that silica is widely used in electronics, ceramic, and polymer material industries. Because of their small-diameter particles it has many technological applications. By processing silica colloidal silica, highly pure silica and silica gel can be obtained. Among these silica types amorphous silica has large industrial applications. Those are coatings, plastics, rubbers, electronics, optics, composite fillers and fire retardant materials. Also Sharifnasab (2017) indicates it's used to synthesize chemicals like sodium silicate, zeolite catalysts, aerogel, silicon nitride, silicon carbide. High tech industries (computer, biotechnology and pharmaceuticals) require Nano silica particles due to their unique properties. Further Todkar (2016) shows that silica is use for reinforcement of silicon rubber, reinforcement of elastomeric products like shoe soles, covering material in tires, sheathing compounds for cables, use in toothpaste to control rheological properties and as a cleansing agent, improve mechanical properties of PVC, use as absorbent and cosmetics, hydrophobic precipitated silica is used in mineral oil and silicon oil, use in purification and stabilization of beer, specially prepared silica are used for making thermal insulators, in food industry as anti-caking agent.

2. METHODOLOGY

2.1 Sample collection

Rice varieties BG360, BG310, BG450, BG352, Murugakanayan, Suduheenati, BG250, BG370 which are abundant in North Western Province were collected from bathalagoda rice research and development institute, bathalagoda. Samples were washed from water in order to remove dirty and water soluble impurities. Water rinsed rice husk samples and HCL solution was mixed for 1h and acid leaching process was carried out for all rice husk samples separately. The acid solution was filtered and the rice husk was washed several times with distilled water until filtrate was free from acid.

2.2 Determination of moisture content

Moisture content of the RH samples were determined by using the standard oven dried method. 200g of Each RH sample were washed and air dried for 48 hours. Then samples were dried in the oven at the temperature of 105°C to 110°C for 24 hours. Dry weight of the rice husk samples was measured. Using the below equation the moisture content of the RH sample was determined.

$$MC = \left\{ \frac{W-D}{W} \right\} \times 100\% \dots\dots\dots(1)$$

MC - moisture content (%)

W - wet weight of the RH sample

D - dry weight (oven dried weight) of the RH sample.

2.3 Determination of average bulk density

Bulk density of RH sample was determined by standard method. RH sample was filled to the container till it filled into the total volume of the container. Then weight of the container and RH sample was measured (W2), after that weight of the empty container (W1) and volume of the container (V) was measured. By using below equation average bulk density was determined.

$$\rho_{Bulk} = \frac{w_2-w_1}{v} \dots\dots\dots(2)$$

2.4 Determination of silica content in RH samples

Oven dried RH samples were put into the muffle furnace at a temperature of 700°C for 4 hours until it became white-gray color. This process was done to all the eight different varieties of RH samples separately. 10g of each RHA sample was separately put into an Erlenmeyer flask and boiled 100 ml of 2.5M sodium hydroxide solutions for 3 hours while stirring. The flask was covered using watch glass in order to avoid evaporation. The solutions were filtered using Whatman No. 41 filter paper and remaining residues were washed with a small amount of water. Each filtrate was allowed to cool down into room temperature. 5 M H₂SO₄ was added to each filtrate until PH became 2. Then NH₄OH was added until pH reached to 8 and allowed to be at room temperature for 3 hours.

Each silica sample was separated by using suction filtration and the filtrate was washed from distilled water in order to remove water soluble impurities. Then each sample was oven dried at 120°C temperature for 12 hours and allowed to cool down to room temperature. After that obtained silica samples were ground well.

2.5 XRD, EDX and SEM analysis

X-ray diffraction (XRD) patterns, SEM images and EDX patterns were obtained for Silica samples. XRD operated under an acceleration voltage of 40 kV and current of 7.5 mA. The diffraction angle 2 θ was in between 10° to 80°.

3. RESULTS AND DISCUSSION

The moisture content varies in between 10.42% to 12.84% among these eight different varieties of RH. The average bulk density varies in between 214.42 kgm⁻³ to 268.57 kgm⁻³ for these selected RH varieties. When comparing these RH varieties, BG352 has the lowest amount of moisture content and sudu heenati RH variety has the highest amount of moisture content. RH

variety BG250 has lowest average bulk density while BG352 has highest bulk density.

Table 1 : Moisture content and average bulk density of different varieties of RH found in North Western Province

Rice variety	Moisture content %	Average bulk density kgm ⁻³
BG360	10.5	235.7
BG370	11.5	252.85
BG250	11.00	214.42
Murugakanayan	11.12	264.2
BG352	10.42	268.57
BG450	10.9	240
BG310	11.02	265.7
Suduheenati	12.84	262.85

Extracted silica samples were white color powder. Highest amount of silica was extracted from murugakanayan variety (98.2%), lowest amount of silica was extracted from BG360 variety (74%). Percentage of extracted silica was shown in table 2.

Table 2 : Extracted silica content (%) from different varieties of RHA

RH Variety	% of silica percentage
BG360	74
BG370	85.2
BG250	79.8
Murugakanayan	98.2
BG352	86
BG450	83.1
BG310	82.3
Suduheenati	76.2

A typical XRD spectra and SEM image of silica obtained from RHA of BG360 is presented in Fig. 1(a) and Fig. 2(b) respectively. The SEM image shows large particles of silica with a carbon background. The XRD spectra of silica shows broader peak centered at 2 θ angle of 22°. It confirms the amorphous nature of the silica. SEM images confirm that these Silica are fine nano particles.

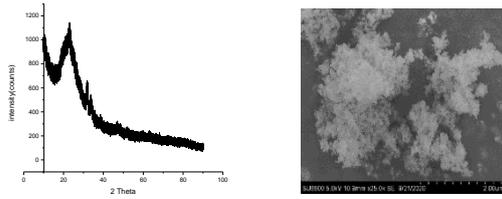


Figure 1: XRD spectra (left) and SEM image (right) of Silica sample obtained from BG360 RH

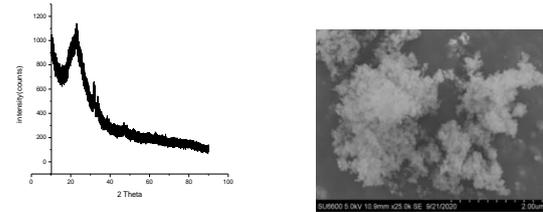


Figure 4: XRD spectra (left) and SEM image (right) of Silica sample obtained from murugakanayan

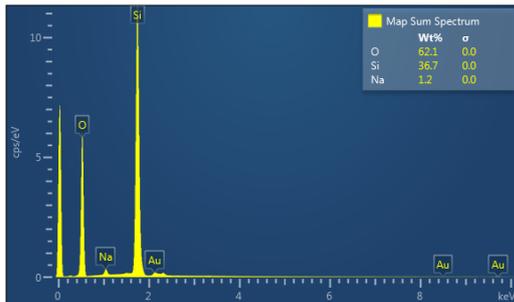


Figure 2: separate distribution of elements in silica sample obtained from BG360 RH variety

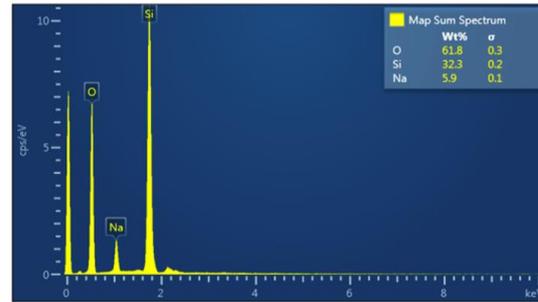


Figure 5: EDX analysis of silica obtained from murugakanayan RH variety

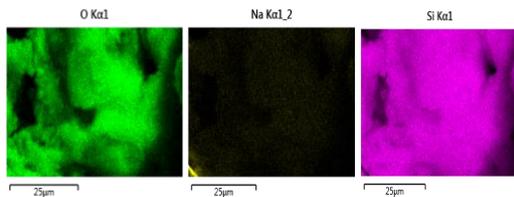


Figure 3: separate distribution of elements in silica sample obtained from BG360 RH variety

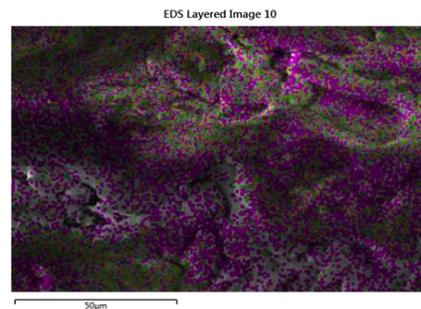


Figure 6: extracted silica from murugakanayan variety captured by EDX

The EDX patterns of Silica obtained from RHA of BG360 are presented in Fig. 2. Elements present in the extracted silica sample could be detectable from the EDX analysis. EDX is useful to identify elements present in an unknown sample. EDX is not suitable to calculate percentages of elements available in low levels. Because it fails to provide results when the percentage of elements is less. fig.3 shows a separate distribution of elements in the silica sample obtained from BG360 RH variety. These samples were placed on a carbon plate so that black area represents carbon.

A typical XRD spectra, SEM image and EDX patterns of silica obtained from RHA of

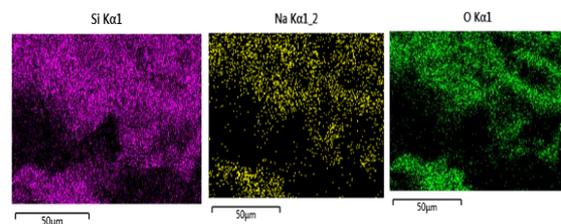


Figure 7: Separate distribution of elements in silica sample extracted from murugakanayan RH variety

murugakanayan rice variety is presented in Fig.4(a), Fig.4(b) and Fig.5 respectively. Further Fig.6 shows captured EDX patterns of extracted silica from murugakanayan rice variety. Fig 7

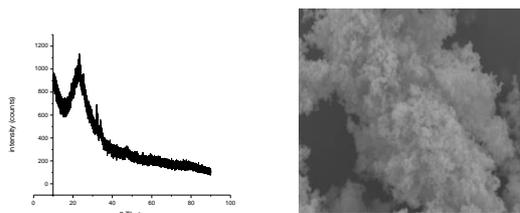


Figure 8: XRD spectra (left) and SEM patterns (right) of Silica sample obtained from sudu heenati

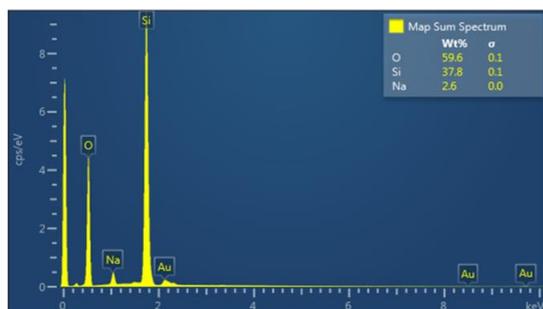


Figure 9: EDX analysis of silica obtained from sudu heenati RH variety

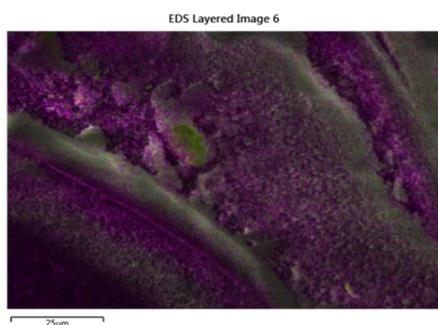


Figure 10: separate distribution of elements in silica sample obtained from sudu heenati Rice variety

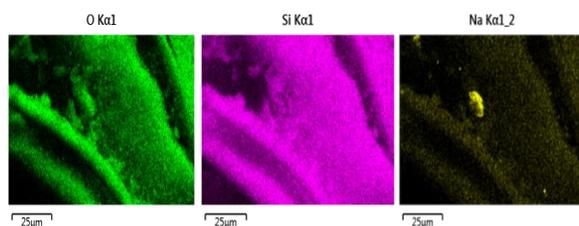


Figure 11: extracted silica from sudu heenati RH variety captured to EDX

shows separate distribution of Oxygen, Silicon and Sodium in the extracted silica sample from murugakanayan rice variety 7.

A typical XRD spectra, SEM image and EDX patterns of silica obtained from RHA of the sudu heenati rice variety is presented in Fig. 8(a), Fig. 8(b) and Fig. 9 respectively. Separate distribution of Oxygen, Silicon and Sodium in the extracted silica sample from sudu heenati rice variety is shown in the Fig. 10. Further Fig. 11 shows captured EDX images of extracted silica from the sudu heenati rice variety.

The EDX separate distribution of elements in the silica sample shows that silicon and Oxygen particles are distributed in the whole area so that less amount of black carbon area is visible. Figure 2 shows that the BG 360 rice variety contains 98.8% of silicon and oxygen. Fig. 5 shows that silica obtained from murugakanayan rice variety contains 94.1% of total silicon and oxygen. Fig. 9 shows that silica obtained from the sudu heenati rice variety contains 97.4% of total silicon and oxygen. It's observed that the highest amount of silicon and oxygen and lowest amount of impurities are available in the silica sample which is synthesized and extracted from the BG 360 rice variety.

4. CONCLUSION

Moisture content of the rice husk varies from 10.42% to 12.84%. BG352 has lowest moisture content and sudu heenati has highest moisture content from selected eight different varieties. Average bulk density varies from 214.42 kgm^{-3} to 268.57 kgm^{-3} . BG250 has lowest bulk density and BG352 has highest bulk density from selected RH varieties. Silica percentage varies from 74.0% to 98.2%. The silica extracted from murugakanayan RH variety has highest silica content and silica extracted from BG360 has lowest silica content from selected other varieties. XRD patterns confirm the amorphous nature of silica. There is a broad peak with maximum intensity at $2\theta = 22^\circ$. SEM images have shown that these silica particles were aggregated as irregular rod shape clusters and also it's observed in SEM images that extracted particles are nano size fine particles. EDX patterns confirm that extracted samples contain

high amounts of silicon and oxygen elements, further percentages vary depending on the rice variety. Moreover, silica obtained from the BG 360 rice variety contains less amount of impurities and high amount of silicon and oxygen. Production of silica from RH is not only a solution for waste management but also it provides valuable commercial product silica.

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