THE EFFECT OF MILLING DURATION TO THE STRUCTURAL PROPERTIES OF SILICA FROM RICE HUSK

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Abstract. A thorough analysis of Rice Husk Ash (RHA) under calcinations is a common concern in order to gain results for producing amorphous silica. This research presented observations on the characteristics of ground and un-ground 450 °C RHA burning temperature obtained from Muar, Johor Malaysia. The procedure was continuing with RHA milling times for one, two, and three hours. All the samples was label as RHA (raw), RHA 1H, RHA 2H and RHA 3H respectively. X-ray diffraction (XRD) was used to recognize crystalline structures, Fourier Transform Infrared Spectroscopy (FTIR) was used to recognize chemical bonding of particles, Particle Size Analysis and Specific Area Surface were used to identify physical characteristics, and using of scanning electron microscope (SEM) to identify surface morphology and microstructures. The findings show that each of the specimens had a comparable XRD pattern with substantial quartz and cristobalite peaks, confirming the presence of amorphous silica. Apparently, the samples of RHA have a nearly identical FTIR spectrum graph but with a distinct peak value. This is due to the particle sizes of the sample have little effect on the spectrum graph. Other than that, RHA that has been milled for three hours has a lower particle size than other RHA. The SEM results show that RHA (3H) has a fine surface compared to RHA (2H), RHA (1H) and RHA (raw). RHA that undergoing 3 hours (3H) period of milling time is believed have a better characterization in term of physical, chemical, morphological and phase.

Keywords: RHA, rice husk ash silica, ball milling, milling time, amorphous silica

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Introduction

The outer layer of the rice grain, or rice husk, is a byproduct of the rice milling process. All nations that produce rice view it as agricultural trash [1]. The majority of rice husk is thrown or burnt in open locations, resulting land degradation and ecological contamination. Numerous initiatives have been launched to use rice husk as an alternative fuel for the formation of activated carbon energy and as a raw material in the synthesis of industrial chemicals focused on silica and silicon compounds [2,3].

Organic substances such as hemicellulose, cellulose, and lignin are the primary components of rice husk, which account for 75 – 90 % of the total weight, with the remainder of the ash content accounting approximately 17-20 % [4]. The ash is mostly silica with minor metallic particles. Under regulated settings, rice husk ash is produced during the burning of rice husks containing practically pure silica. Prior to burning, pre-treatments process may remove metallic contaminants such as iron (Fe), sodium (Na) and manganese (Mn), which affect the purity and colour of the silica [5].

It has been observed that at temperatures lower than 700°C and depends on the duration of the combustion, amorphous silica is formed, but at higher temperature, crystalline silica is obtained [5-7]. In this systematic investigation, rice husks were burned for 8 hours in a muffle furnace at 450 °C. Previous study state that after burning away the carbonaceous material at various durations and temperatures, the relative quantity of silica was raised. After 6 hours of heat treatment at 700 °C, a 95 % silica powder could be generated. After milling process, the specific surface area of the particles rose from 54 to 81 m^2/g. The current study's goal is to look into the RHA high specific surface area silica processing and characterization. Heat treatment enhanced the relative quantity of silica, and varying milling times of 1 h, 2 h, and 3 h improved the specific surface area.

Materials and Methods

Silica Production from Rice Husk

The RHA sample was taken in Muar, Johor, Malaysia, in a rice mill, and was subjected to direct combustion at 450 °C for eight hours and is greyish in colour. After that, the rice husk was milled with varying milling times of one hour, two hours, and three hours respectively.

Characterizations of Rice Husk Silica

X-ray powder diffractometer, (XRD, Shimadzu XRD-6000, D8-Advance, Bruker, German) with CuKα radiation was used to describe the crystalline phases of the materials. The XRD travels at a speed of 0.02 s1 with a continuous scanning angle of 2θ adjusted between 20° to 80°. The densities of powder were determined using the samples' mass and size. A Thermo Scientific FTIR Nicolet 6700 fitted with an attenuated total reflectance (ATR) accessory was used to identify the functional groups in the sample. The spectra were captured with 32 scans at a resolution of 4 cm^{-1} in the 4000 to 400 cm^{-1} range. Laser diffraction was used to determine particle size and specific surface area of the powder (Cilas, model 1064L, USA). The scanning electron microscope SEM was used to examine the cross-sectional morphology of RHA powder (Hitachi, SU1510, Japan). The element distribution in the composite was determined using energy dispersive X-ray (EDX).

Results and Discussion

Phase Analysis of RHA Silica

Temperature and duration of burning out are crucial determinants in determining whether silica stays amorphous, as in RHA, or crystalline. Using 450 °C burning temperatures and 8 hours of heat treatment, the silica structure in the ash remained basically amorphous [8]. The amorphous nature of SiO_2 generated from rice husk was confirmed by the presence of intense and broad diffraction peaks in the XRD investigations, as illustrated in Figure 1. For all types of SiO_2, which were RHA raw, 1 H, 2 H and 3 H, there were a similar type of phase of silica formed. The type of silica phase of cristobalite (JCPDS No.: 00-039-1425) and quartz (JCPDS No.: 00-046-1045) was identified as a result for XRD. Other than that, the result of XRD pattern of rice husk ash milled for one hour, two hours, and three hours obtained was comparing to the XRD pattern of Muda et al. As the comparison, the pattern appears to be the same, with prominent cristobalite and quartz type of peak. This situation also confirms the existence of amorphous silica in all the sample [9-11].

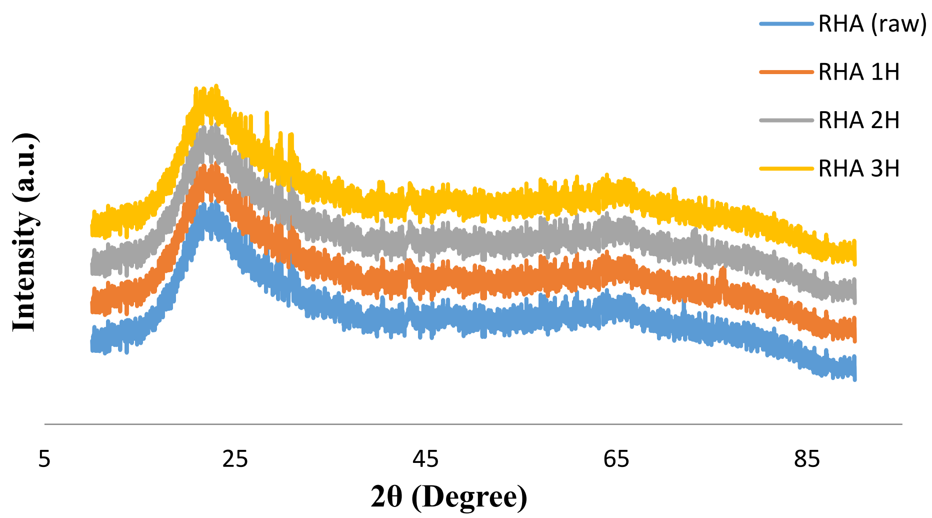

Figure 1: XRD diffractograms for rice husk ash for different milling duration

Chemical Bonding Analysis of RHA Silica

The spectrum graph of the RHA raw, 1 h, 2 h and 3 h samples were depicted in Figure 2. These data allow for the identification of the four distinct peaks of RHA. The FTIR instrument passes infrared light ranging from 10,000 to 100 cm^{-1} through a material, with some absorbed and some passing through. The sample molecules transform the absorbed radiation into rotational and/or vibrational energy. The resultant signal at the detector appears as a spectrum, generally ranging from 4000 cm^{-1} to 400 cm^{-1}, showing the sample's molecular fingerprint. The first bands for all the samples were 794.04 cm^{-1}, 795.46 cm^{-1}, 797.28 cm^{-1} and 795.26 cm^{-1}. This band correspond to symmetrical stretching vibrations of O−Si−O and are consistent with Javed's observations between 806 and 797 cm^{-1} [12], Muslim at 800 cm^{-1} [13] and Ferraro at 773 cm^{-1} [14] are included in the tables of silica group peak wave numbers around 805 cm^{-1}.

The O−Si−O vibrations caused by uneven stretching bands were also recorded in bands which were at 1050 cm^{-1} for RHA (raw), 1058.52 cm^{-1} for RHA (1 h), 1062.57 cm^{-1} for RHA (2 h) and 1059 cm^{-1} for RHA (3 h). These results were also compatible with Javed findings where the bands located at 1097 to 1091 cm^{-1} and Ferraro's measurements at 1080 cm^{-1}. The entire bands remain within the 1200 cm^{-1} to 1000 cm^{-1}. Other than that, a free water band was discovered at 3683 cm^{-1} for RHA (raw), 3672 cm^{-1} for RHA (1 h), 3660 cm^{-1} for RHA (2 h) and 3657 cm^{-1} for RHA (3 h) due to the stretching vibration of H_2O molecules. This also agrees with the findings by Javed (3636 cm^{-1} to 3427 cm^{-1}) and Ferraro (3436 cm^{-1} in the range of extremely broad hydroxyl groups at 3500 cm^{-1} to 2500 cm^{-1}) in the peak of wavenumber tables [12-14]. Apparently, all the samples of RHA has a nearly identical spectrum graph but with a distinct peak value. This situation is due to the different particle sizes of the sample that affect the result obtained from the spectrum graph.

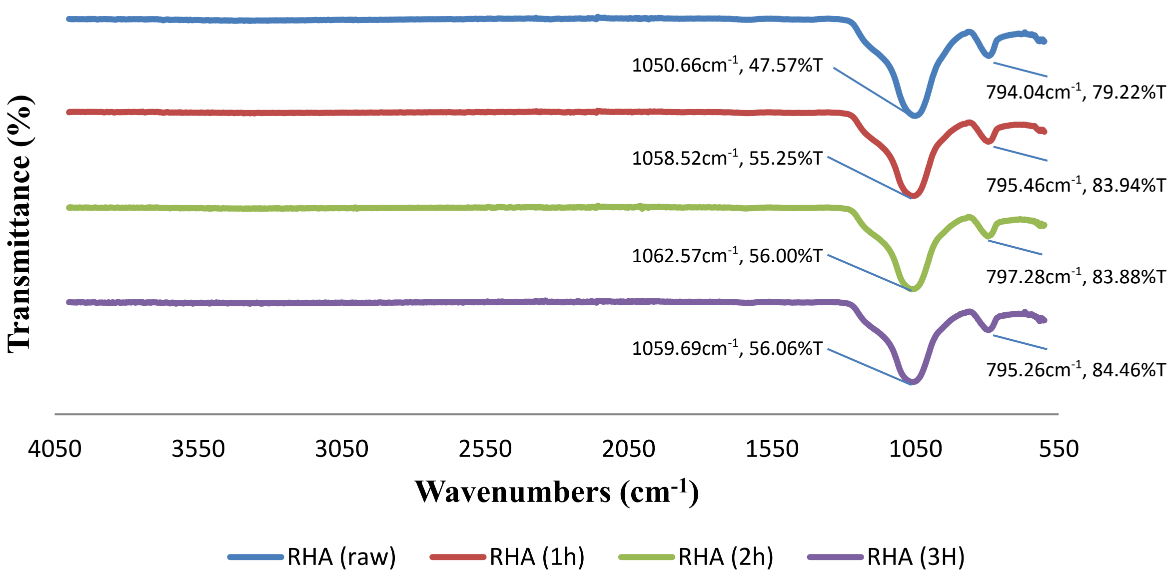

Figure 2: FTIR analysis for all samples

Particle Size Analysis and Specific Surface Area of RHA Silica

Figure 3 shows the particle distribution of RHA for diameter of cumulative values at 10 %, 30 %, and 60 % and its specific surface. The data clearly shows that increasing the time for milling rice husk ash increases its specific surface approximately thrice from the initial size which is increasing from 1316.36 cm^2/g to 3977.73 cm^2/g after undergoing 3 hours of milling process as shown in Table 1. Other than that, the ground processes by using jar mill decreasing the diameter size of RHA particles as the time for milling is increasing in the term of diameter of cumulative value at 10 %, 30 % and 60 %. The fact that the RHA colour is greyish indicates that the amount of unburned carbon is considerable, implying that the physical characteristics of rice husk ash are highly influenced as a rice mill's burning process result. Graph of particle size distribution revealed that the diameter was 60 % of the unground rice husk which mean 60 % of particles has size about 37.67 µm, but ground process of three hours could decrease the diameter tremendously around 12.98 µm.

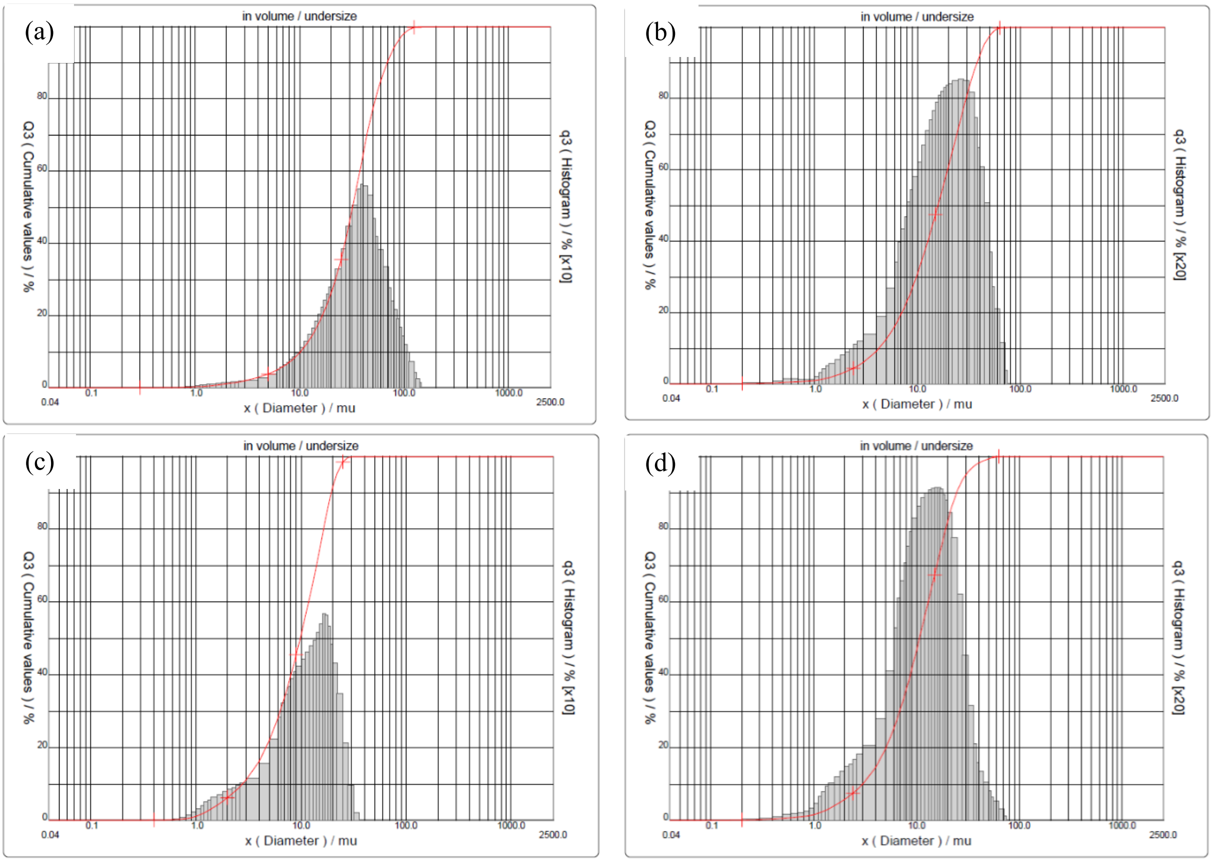

Figure 3: Particle size distribution graph of RHA, (a) RHA raw, (b) RHA (1H), (c) RHA (2H) and (d) RHA (3H).

Table 1: Particle size distribution and specific surface of RHA

Type of RHA	Specific surface (cm^2/g)	Diameter at 10% (µm)	Diameter at 30% (µm)	Diameter at 60% (µm)
RHA (raw)	1316.36	10.18	22.08	37.67
RHA 1H	2904.42	4.22	9.79	19.65
RHA 2H	3388.85	3.57	8.27	16.05
RHA 3H	3977.73	3.00	6.87	12.98

Morphology Analysis of Rice Husk

RHA microstructure was analyzed using SEM, and the test was run on a JEOL SEM with model number JSM-6380LA. The RHA is a powder, thus the sample should be coated with platinum using a JEOL JFC-1600 Auto Fine Coater to stop powder particles from contaminating the SEM apparatus and causing defect and tarnish images. A quantitative chemical study of RHA at a specific location was also performed using energy dispersive X-ray spectroscopy (EDX). Figure 4 reveals the different between ungrounded and grounded of RHA microstructure with 350X magnification respectively.

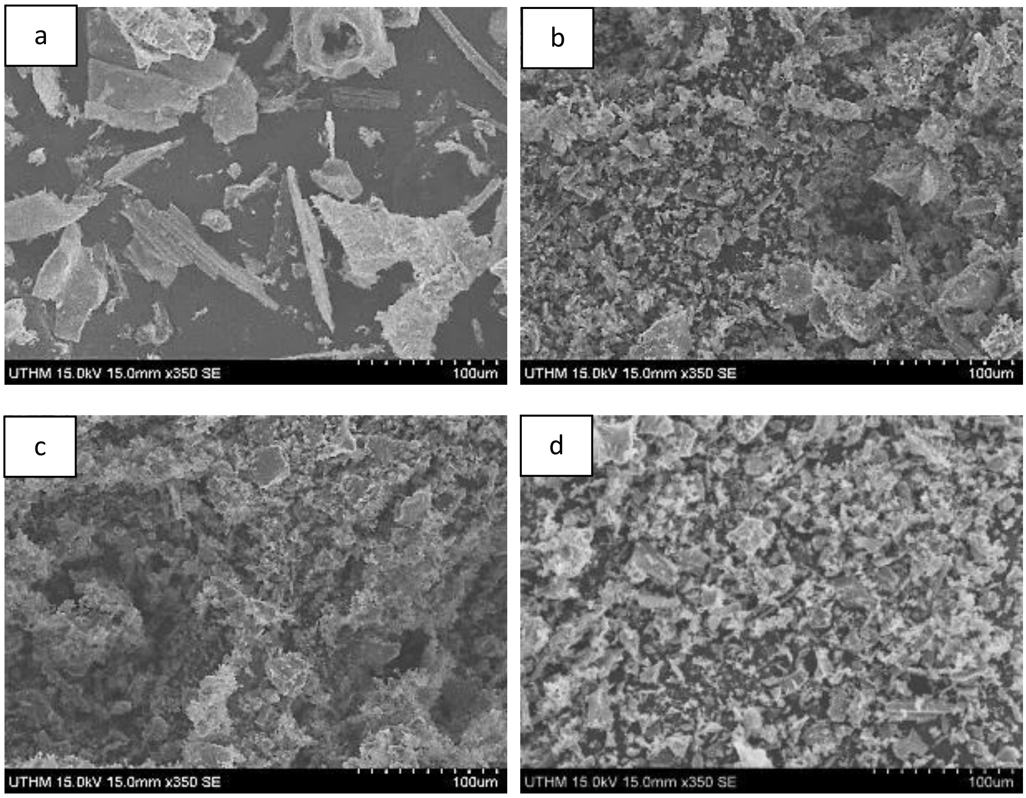

Figure 4: SEM images of surface morphology for RHA with different milling time (a) RHA raw (b) RHA 1H (c) RHA 2H and (d) RHA 3H

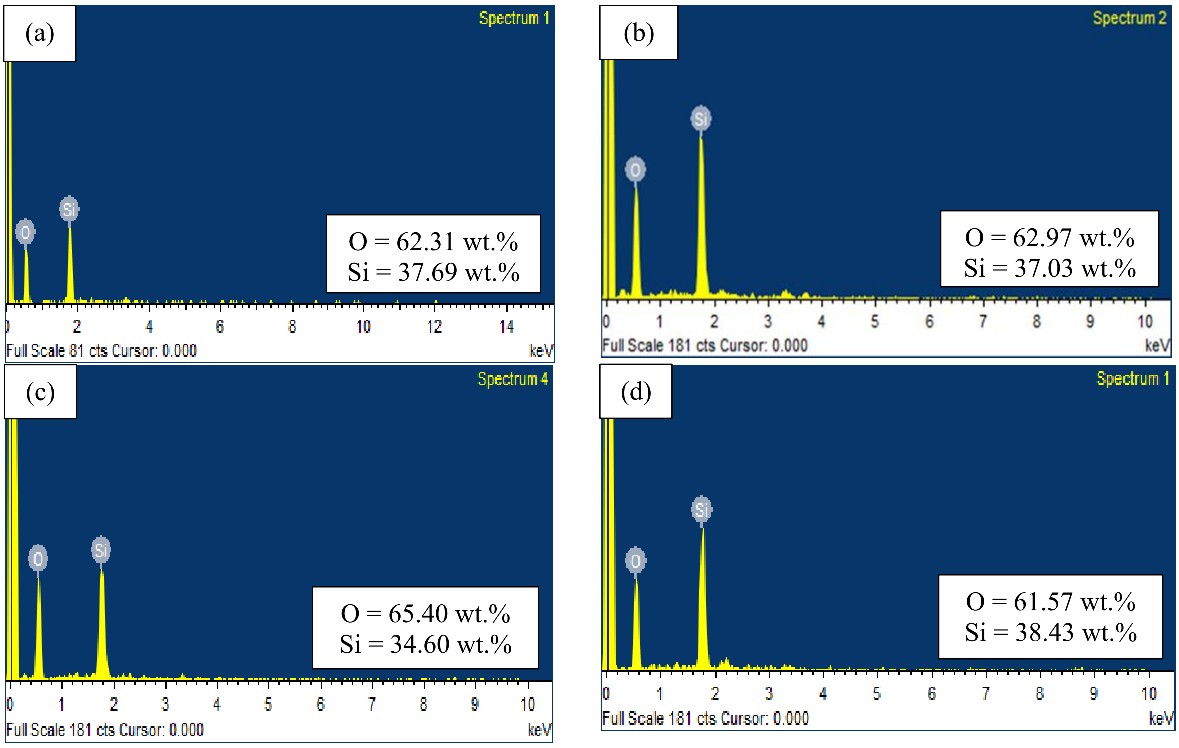

Figure 5: EDX spectrum of sample, (a) RHA raw, (b) RHA 1H, (c) RHA 2H and (d) RHA 3H

Figure 4(a) shows the exterior of the RHA, which is corrugated and has spiky, horn-like cuticles that are well structured. Figure 4(b), (c), and (d) depicts the microstructure of rice husk after milling with varied milling times of 1 h, 2 h, and 3 h, respectively. The results reveal that the milling process significantly reduces RHA size and causes the structure of RHA to become fine and smooth. The RHA chemical composition was validated by EDX spectrum analysis by using individual spot mapping toward all samples as shown in Figure 5. From the result, it is confirmed the existence of both elements which are Oxygen and Silica elements. Other than that, the weight percentage for both elements in all sample were quite similar and did not show a large different from each other.

Conclusions

The characterization of 450 °C and 8 hours of rice husk ash with different milling times which are 1 h, 2 h and 3 h, was conducted. The XRD result for all types of SiO_2, which were RHA raw, 1 H, 2 H and 3 H showed the similar cristobalite (JCPDS No.: 00-039-1425) and quartz (JCPDS No.: 00-046-1045) amorphous phase. Other than that, all samples of RHA have a nearly identical spectrum graph for FTIR but with a distinct peak value. This situation is due to the different particle sizes of the sample that affect the result obtained from the spectrum graph. In term of particles size, the results proved that increasing the time for milling rice husk ash increases its specific surface in the same time decreasing the diameter size of RHA particles. The SEM results reveal that the milling process significantly reduces RHA size and causes the structure of RHA to become fine and smooth. The EDX results proved the existence of Si and O elements in the samples. As a whole, the results show that the RHA that has been milled for three hours performs significantly better than the other samples in term of physical and morphology while show a quite similar property in term of chemical and phase.

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Author Contributions

All authors contributed toward data analysis, drafting and critically revising the paper and agree to be accountable for all aspects of the work.

Disclosure of Conflict of Interest

The authors have no disclosures to declare

Compliance with Ethical Standards

The work is compliant with ethical standards.

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