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Characteristics of silica rice husk ash from Mojogedang Karanganyar Indonesia

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Abstract. Indonesia is one of the countries in the world as the most abundant rice producer. Many researchers have demonstrated that the highest composition in the rice husk ash (RHA) is silica. Some of the advantages in utilizing silica as the raw material is the manufacture of ceramics, zeolite synthesis, fabrication of glass, electronic insulator materials, and as a catalyst. The amount of silica from rice husk ash is different for each region. Therefore, the study of silica from RHA is still promising, especially rice organic fertilizers. In this study, the rice came from Mojogedang Karanganyar Indonesia. Rice husk was dried under the solar radiation. Then the rice husk was heated in two steps: the first step at a temperature of 300°C and the second step at a temperature of 1200°C with a holding time at 2 h and 1 h, respectively. Furthermore, the temperature of the second step was varied at 1400 °C and 1600 °C. This heating process produced RHA. The content of RHA was observed on the EDAX spectrums while the morphology was observed from SEM images. The crystal structure of RHA was determined from XRD spectrums. The EDAX spectrums showed that RHA composition was dominated by elements Si and O for all the heating temperature. SEM images showed an agglomeration towards larger domains as heating temperatures increase. Analysis of XRD spectra is polycrystalline silica formed with the significant crystal orientation at 101, 102 and 200. The intensity of 101 increases significantly with increasing temperature. It is concluded that the crystal growth in the direction of 101 is preferred.

1. Introduction

Indonesia is one of the biggest rice-producer countries in the world. Thus, there is a huge of rice husk (RH) waste. It still widely used as a fuel by burning it. Approximately 20% of ash can be obtained from burning RH [1-3]. Many researchers report that the rice husk ash (RHA) contains about 87%-98% of silica (SiO_2) [4-6]. The rest is metallic impurities such as K_2O, Al_2O_3, CaO, MgO, Na_2O, Fe_2O_3 with the percentages of each compound are less than 1% [3, 7, 8].

In industrial application, silica is the raw material in the manufacture of ceramics, zeolite synthesis, glass, electronic insulator materials, and as a catalyst [4, 8]. Recently, silica from RHA because of the high silica content is found to be the source of silicon (Si) through common fabrication methods such as submerged electric arc method and magnesium (Mg) reduction [3, 5, 9, 10]. It is applied in many electronic devices. One is semiconductor grade silicon (SGS) which is the material for a solar cell that it becomes one of the most attractive studies to overcome the energy crisis issues nowadays [3]. Silica must have high purity and crystallinity to produce semiconductor grade silicon [11]. Therefore, we have been conducting a study to gain a high purity (>99 %~100%) and crystallinity of silica in the RHA.

Table 1. The results of RHA fabrication in the previous research [12]

Sintering temperatures (°C)	Percentage of silica in RHA (%)	Products and crystallinity (%)
1000	95.11	Crystalline and 97.43
900	94.72	Amorphous
800	94.96	Amorphous

a b c

Figure 1. Images of (a) RH (b) RH charcoal and (c) RHA

The silica content in RHA is affected by the crop variety, soil chemistry, climate condition, fertilizer condition, and even the geographic location [8, 9]. Additionally, other factors such as the temperature, holding time, the rate and heating technique also have their impact [8]. Several studies have reported that silica is an amorphous phase when it is heated up to low temperature (500°C-700°C). It forms high purity and crystalline at a high sintering temperature (>800°C) depended on the time combustion [3, 6]. Thus, the study to produce high purity silica in RHA (>99 %~100%) is still challenging.

In our previous study [12], RHA has been synthesized by two steps heating; the first heating called carbonization at a temperature of 300°C and the second heating called sintering at different temperatures of 800°C, 900°C, and 1000°C. The results in Table 1 showed that silica RHA was still amorphous at the temperatures of 800°C and 900°C and changed to the crystalline phase at the temperature of 1000°C. It yielded silica with more than 90% for each sintering temperature. However, the metallic impurities such as CaO, P_2O_5, Al_2O_3, K_2O, Cl, SO_3, Fe_2O_3, MnO, Cr_2O_3, NiO, SnO_2, and ZnO remained. Consequently, the heating process has been done 3 times and the sintering temperatures have been increased in this study.

2. Materials and methods

The synthesis of silica included washing the RH, drying, carbonization, and sintering. The RH was prepared from Mojogedang Karangayar, Indonesia displayed by Fig. 1a. It was cleaned to remove the dust and sand attached to the rice husk. Drying was directly exposed under the sun to eliminate water content in rice husk after cleaning process. The carbonization was carried out using furnace at a temperature of 300 °C for 1 h in the air ambient. It was to decompose the volatile organic substances (cellulose, hemicellulose, and lignin) contained in the RH. It produced RH in the black color shown in Fig. 1b. This black RH composed much of carbon evaporated on heating > 400 °C [3]. It was then sintered in two sintering processes; first sintered at a temperature of 900 °C for 2 h producing RHA shown by Figure 1c and second sintered at varied temperatures of 1200 °C, 1400 °C, and 1600 °C with the holding time of 6 h for each variation. The second sintering was performed to remove the metallic impurities left after the first sintering. The RHA was then characterized by X-ray Diffraction (XRD) and Scanning Electron Microscope-Electron Dispersive X-Ray (SEM-EDX) equipment.

3. Results and discussion

Figure 2 presents XRD patterns of the samples formed at different temperatures. All the diffraction peaks are in good agreement with literature data (ICDD #82121410). They are defined as SiO_2 with a tetragonal structure and space group of $P4_12_12$. Additionally, the highest peaks were investigated as the increased temperature displayed by the (101) plane in Fig. 2. Based on the Scherrer formula as performed in ref. [13], it implies that the crystallite size of silica grew with increasing temperature presented in Table 2. It also indicates improving crystallization of the material which means high crystallinity achieved [6]. The most substantial peak showing by Bragg's plane of (101) is one of the fingerprints of silica cristobalite phase. It leads to being the key of the fabricating single crystal SGS for a solar cell in our next study. The single crystal is more easily grown on a substrate than the poly-crystal.

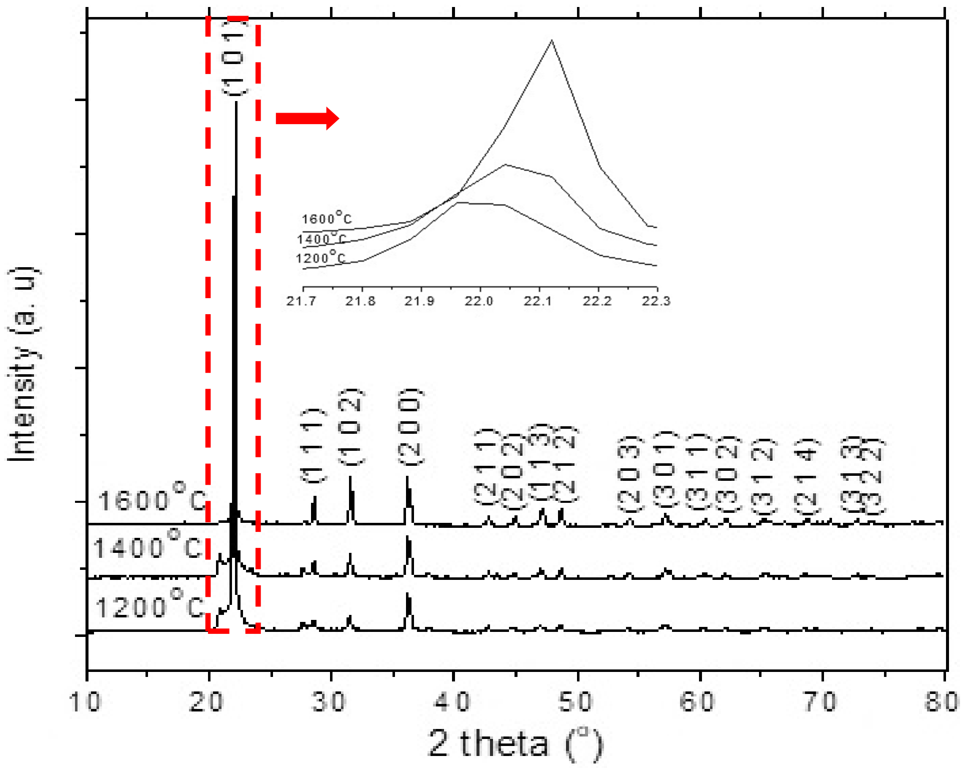
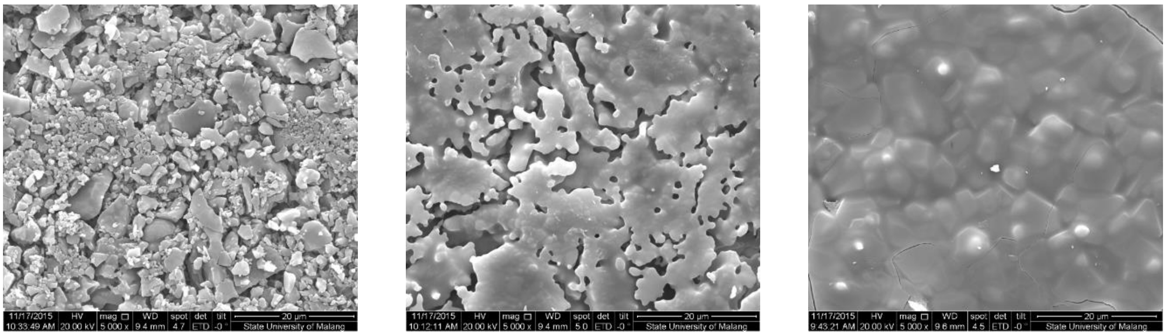


Figure 2. XRD patterns of RHA at different temperatures

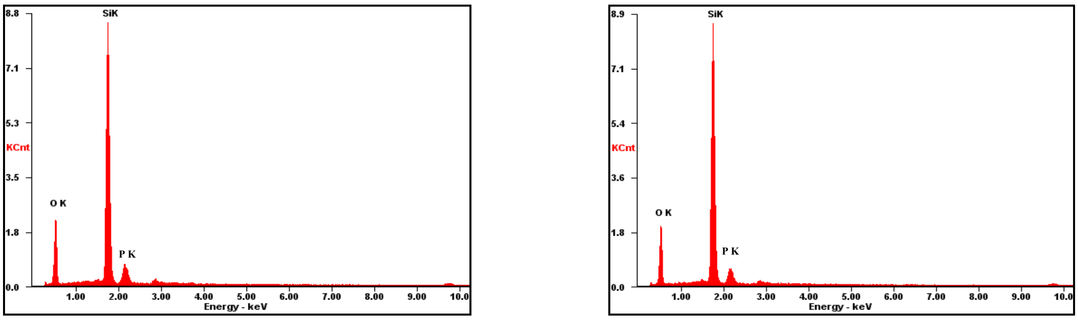
Table 2. Crystallite size of RHA at different temperatures

Temperatures (°C)	Crystallite size (nm)		
	(101)	(102)	(200)
1200	25.39	26.25	24.46
1400	31.18	26.42	32.59
1600	56.20	42.16	37.49



a b c

Figure 3. SEM images of RHA at temperatures of (a) 1200 °C (b) 1400 °C and (c) 1600 °C



a b

Figure 4. EDX spectrum of RHA at temperatures of (a) 1200 °C and (b) 1400 °C

Figure 3 shows the morphologies of the RHA at different temperatures. It is clear that the samples agglomerated with increasing temperature, which represents the crystal growth in the samples. Therefore, the SEM results are corresponding to the XRD patterns of the samples. This phenomenon excludes whose silica powder was melted at the sintering temperature of 1600 °C. It is due to the melting point of silica is around 1600 °C [14].

In order confirm the compounds in RHA, the samples with sintering temperatures of 1200 °C and 1400 °C were further examined by EDX. This equipment can be used to identify the chemical compositions of material. As revealed in Fig. 4, Si and O are the main components in RHA. The others elements with low intensity are assigned to phosphor (P). The elements determination of EDX result in RHA refers to the study reported by Hadipramana et al [2] and Yavakkumar et al [4].

The percentage of silica in RHA has not been reported in this study. However, lost in many type metallic contaminants and only leaving the little amount of phosphor indicates the higher content of silica in RHA obtained in this study compared to the previous study [12]. As a result, it can be considered as the raw material for producing silicon.

4. Conclusions

High content and crystallinity of silica from RHA can be extracted by two steps of sintering. Thus, it can be considered as the source of silicon. The crystal growth of silica at (101) orientation is indicating a silica crystobalite phase which able to be a basis for fabricating single crystal silicon which is easily grown on a substrate.

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