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Synthesis of silica particles from rice straw waste using a simple extraction method

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Abstract. The purpose of this study was to synthesize silica particles from rice straw waste using a simple extraction method. The experiment was conducted by heating and extracting rice straw waste into basic solution. To get silica particles, the extracted solution was then put into acid solution and heated to remove the remained solvent. The experimental results showed that the aggregated silica particles with sizes of about 200 nm were successfully produced. The XRD and FTIR analysis showed that the final product was silica and free of graphite. However, we found that some KCl component in the final product in which this was possibly from the use of KOH as the extraction agent. Therefore, further studies are still required to synthesize high purity silica particles from rice straw waste.

1. Introduction

Silica is one of the most important components and can be found in various applications such as biotechnology-related materials [1][2], environmental treatment components[3][4], raw materials for cement industry [5], composite materials [6][7], and medical-related materials [8][9][10]. The main reason for the use of silica is because of its excellent performances: chemically, physically, and thermally stable, compatible with various materials [6][7], largely available, and relatively inexpensive [1].

One of the potential silica raw materials is rice plant-related waste. This waste can be found in tropical countries, especially Indonesia. However, the use of rice straw waste as a silica raw material is still suboptimal. In fact, using rice straw as the silica raw material will be beneficial not only for economical aspect but also for environmental point of view. [11]

Many researches relating to extraction of silica from rice plant have been reported, including rice husk [12][13][14] and rice straw [10][15]. In general, to get silica, they used extraction agent (e.g. basic solution). Reports on the influence of mol ratios of extraction chemical and silica, extraction time, and process temperature on the successful production of silica have been reported [13][15][16]. Although their reports showed good for industrial applications, several disadvantages are found, including



complicated synthesis procedures, sophisticated apparatuses, high-temperature process, and time-consuming extraction process.

Here, the purpose of this study was to synthesize silica particles from rice straw waste using a simple extraction method. In short, our method was conducted by adding rice straw waste into basic solution. Then, the extracted solution was then put into acid solution to precipitate the silica. Different from other reports, our method is successful for extraction silica under atmospheric pressure. The synthesis method for silica particles is easily accomplished under relatively low-temperature process, while other methods must be done at a temperature of more than 80°C. Our extraction processing time is also relatively short (up to 2 hours), while other reports need more than 3 hours. To confirm our successful synthesis of silica materials, several characterizations have been conducted, including an X-Ray Diffraction (XRD), Scanning Electron Microscope (SEM), Thermo Gravity Differential Thermal Analysis (TG-DTA), Atomic Absorption Spectroscopy (AAS), and Fourier Transform Infra Red (FTIR).

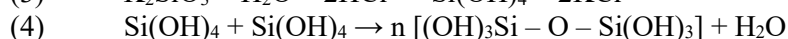
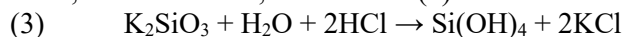
2. Hypothetical analysis for the extraction of silica from rice straw waste

Rice straw contains cellulose (32-47 wt%), hemicellulose (19-32 wt%), lignin (5-24 wt%), and other components (13-20 wt%) [15][17]. Other components in the rice straw contains silica with composition about 60-80 wt% [15][18]. The chemical composition inside the rice straw depends on type of rice, planting time, climate, and geographical-related condition.[12] Since silica contains in the rice straw ash with high concentration, the rice straw can be classified as one of the prospective silica raw materials.

To extract silica from rice straw, a simple extraction method can be used. The reaction happening during the extraction process can be written as [14][19]



Then, for some cases, the reaction (2) can be written as [21]



3. Experimental Method

3.1. Raw materials

We used rice straw waste (rice field in Subang, Indonesia), kalium hydroxide (KOH; technical grade; PT. Bratachem, Indonesia), and hydrochloric acid (HCl; technical grade; PT. Bratachem, Indonesia). Prior to using, rice straw waste was washed with de-ionized water, dried naturally for 3 days, and cut with sizes of about 1 cm. For KOH and HCl, these chemicals were used without further purification process.

3.2. Synthesis of silica particles

First, rice straw waste was heated with an electrical furnace at a specific temperature. Then, the heated rice straw waste (as ashes) was grinded, put into basic solution (KOH), and mixed with 600 rpm at temperature of 60°C for 2 hours. After the extraction process was finished, the extracted solution was filtered. The filtered ash is named as the residual ash, whereas the solution is the filtered solution. The filtered solution was then added to the acid solution (HCl). The process was conducted at pH = 2 to form clear gel. The gel was then aged for 1 night. Finally, the solution containing gel was filtered, washed with de-ionized water, and heated at 150°C for 30 minutes.

3.3. Characterization

We used several characterization analysis methods: X-Ray Diffraction (XRD; PANalytical X'Pert PRO; Philips Corp., Netherland), Thermo Gravity Differential Thermal Analysis (TG-DTA, DTG 60A TA 60 WS, Shimadzu Corp., Japan; operated at 5°C/min with 200 mL/min of carrier gas (oxygen gas)), Atomic Absorption Spectroscopy (AAS, Varian Spectra 240 FS, Varian Inc., California), Fourier Transform Infrared (FTIR, FTIR-8400 dan Prestige 21, Shimadzu Corp., Japan), dan Scanning Electron Microscopy (SEM, JSM-6360LA; JEOL Ltd., Japan).

4. Results and Discussion

4.1. Physicochemical properties of rice straw waste

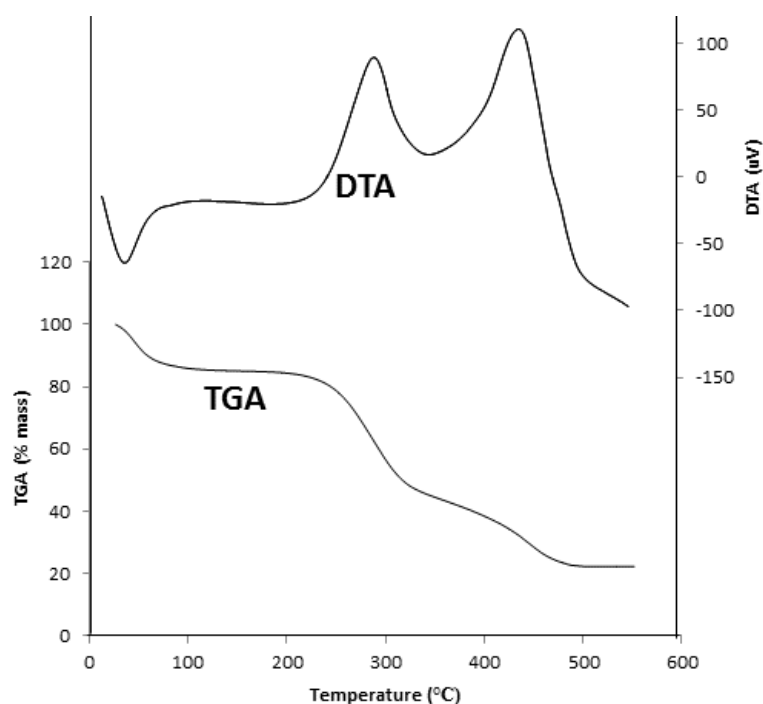


Figure 1. TG-DTA analysis result of rice straw waste

Figure 1 shows TG-DTA analysis results of rice straw waste. From this figure, we obtained four temperature zones, as discussed in the following:

- (1) 20-100°C. The mass reduction (about 5 wt%) occurred because of evaporation of physically adsorbed water [6][7]. The evaporation process was confirmed by the detection of endothermic peak at 50°C.
- (2) 250-320°C. The mass loss (48 wt%) happened due to the degradation of cellulose-related component to form carbon [11][16]. This is confirmed by the existence of exothermic peak at 299°C.
- (3) 330-470°C. The mass reduction (25 wt%) was identified, in which this was due to degradation of lignin [11][16]. This was verified by the detection of exothermic peak at 442°C in the DTA analysis.
- (4) More than 500°C. No mass loss was identified. The final mass was about 22 wt%. No change in mass was found because all organic compounds have been converted completely.

Based on the above TG-DTA results, the heat treatment is important to be added before the silica extraction process. The heat treatment can eliminate organic compound of up to 80 wt%, which is important to make the extraction of silica easily. [16]

To confirm the composition of silica in the heated sample, the AAS and the gravimetric analysis were conducted (**Table 1**). The result showed that the heated rice straw contained 85 wt% of silica. This result is in a good agreement with other reports [15][18]. This result also indicates that the present rice straw is one of the good candidates for silica raw materials.

Table 1. AAS and gravimetric analysis results of heated rice straw. Composition of silica was re-analysis using Gravimetric analysis method.

No.	Composition	Quantity (%)
1	SiO ₂	84.60
2	K ₂ O	6.39
3	CaO	0.88
4	MgO	0.44
5	Fe ₂ O ₃	0.16
6	Other	7.53

4.2. Synthesis of silica particles from rice straw waste

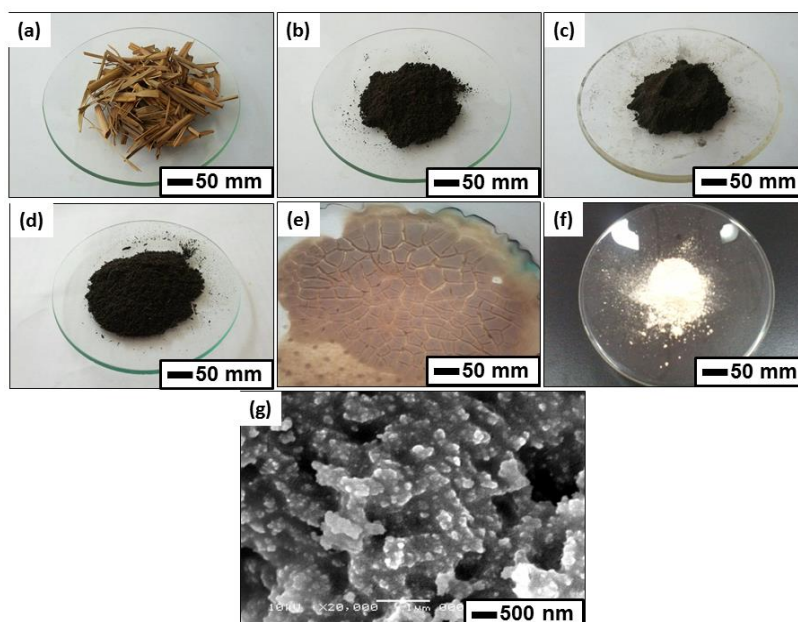


Figure 2. The photograph images (a-f) and the SEM analysis result of samples (g). Figure (a) is the initial rice straw waste. Figures (b)-(d) are the grinded samples after heat treatment process for 10 minutes with various temperatures: (a) 200, (b) 300, (c) 400, and (d) 500°C. Figure (d) is the sample after putting in the basic and acid treatment. Figure (f) is the product after final heating process. Figure (g) is the SEM analysis image of product shown in Figure (f). To make easy for observing visual observation, Figure (f) was put in the dark background, whereas Figures (a)-(e) was in the white background.

Figure 2 is the photograph and the SEM images of sample heated with various temperatures. **Figure 2a** shows rice straw sample after washed and cut into sizes of about 1 cm. After rice straw was heated

and grinded, black powders were obtained (**Figures 2b-d**). The color of powders was getting brighter with increasing process temperatures. These figures confirmed that the transformation of rice straw into carbon from temperature of 200°C [20]. The phenomena of the brighter dark powder were due to the increases in silica composition in the ash [12]. This result is in a good agreement with TG-DTA analysis results in the above that increasing the temperature causes the decreases mass of organic compound. From these results, we conclude that the best heat treatment process is 500°C.

After sample was put into the basic solution, black solution (coffee-like solution) was obtained (not displayed). Then, filtering the solution, we got clear, transparent, and black solution. Adding this filtered solution into acid solution, the mild gel solution was obtained, in which the purified gel is shown in **Figure 2e**. To get silica nanopowder, the purified gel was heated (**Figure 2f**). As shown in **Figure 2e**, the crack in the sample was found. Silica starts to form smaller particles solitary [11]. This result was confirmed by the observation of the SEM image in **Figure 2g**, in which the product contained aggregated particles with sizes of about 200 nm. The white powder was obtained, indicating the potential material consisted in the product was silica [19].

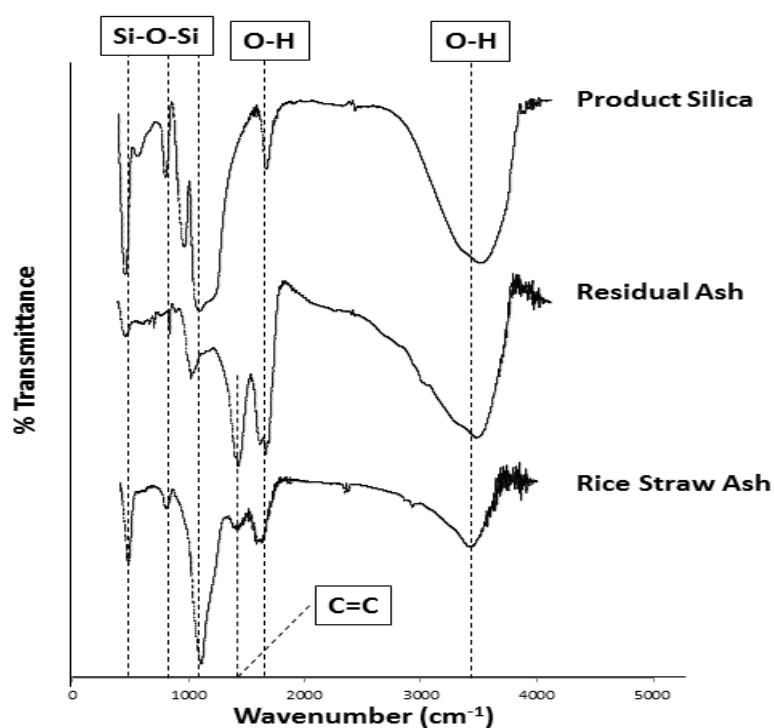


Figure 3. FTIR analysis result of rice straw ash, residual ash, and product samples

Figure 3 shows the FTIR analysis result of rice straw ash, residual ash, and product samples. All samples have some peaks at 460, 700, and 1000 cm^{-1} , in which these peaks are peaks for Si-O-Si bending [11], Si-O-Si symmetric stretching [12], and Si-O-Si asymmetric stretching [19], respectively. These results confirmed that all samples containing silica component. However, for rice straw ash and residual ash, we found a peak that was not found in the product sample. Specifically, we detected peak at 1400 cm^{-1} that indicated C=C aromatic from graphite. This result verified that the product was silica and free of graphite component. In the product sample, we also found a peak at 952 cm^{-1} , indicating that some water molecule (OH) was trapped inside the silica matrix [11]. Detailed analysis of peaks detected by FTIR is shown in **Table 2**.

Table 2. Peaks detected in the FTIR analysis results of rice straw ash, residual ash, and product samples

No.	Functional Group	Wavenumber (cm ⁻¹)		
		Rice Straw Ash	Residual Ash	Product
1	Si-O-Si bending	468	466	464
2	Si-O-Si symmetric stretching	796	756	798
3	Air dalam matrik silika	-	-	952
4	Si-O-Si assymmetric stretching	1099	1026	1083
5	C=C dari grafit	1421	1404	-
6	OH bending	1627	1651	1641
7	OH stretching	3423	3382	3427

Figure 4 shows the XRD analysis results of rice straw ash, residual ash, and product samples. As references, we also added several JCPDS result. The XRD of rice straw ash sample detects peaks of silica, CaMgSi₂O₅ (diopside), dan graphite. The appearance of diopside was different with AAS result in **Table 1**. AAS detected SiO₂, CaO and MgO, instead of diopside. AAS analysis uses flame that possibly changes the structure of diopside into their stable oxides, i.e. SiO₂, CaO, and MgO. The stability of diopside was also confirmed by the disappearance of diopside peaks in both residual ash and product samples.

Based on the XRD results of the product sample, the peak at 27° was not detected, confirming the product is free of graphite. The intensity of peaks of silica (JCPDS 27-1402) verified that the main product containing silica. However, in product, we found KCl peak, in which this was possibly from the KOH as the extraction agent. Therefore, further purification process is required for increasing the purity of silica.

Based on the above results, we conclude the hypothetical analysis for the synthesis of silica particles in **Figure 5**. Rice straw is prospective for silica raw source since the silica is contained in the rice straw (verified by TG-DTA, AAS, and gravimetric analyses). To get excellent process for isolating silica component from rice straw, additional heat treatment is the best choice. The heat treatment is effective to eliminate most of the organic components in the rice straw via the formation of ash. Indeed, by isolating silica from the rice straw ash, carbon porous materials can be produced; however, this will be performed in our future work. Then, to get silica component, combination of co-precipitation method (via acid etching process) and heat treatment can be used. Finally, the formation of silica particles in the final product can be observed directly by visual observation (white powder). Indeed, confirmation of silica particles can be achieved by conducting further physicochemical analysis such as SEM, XRD, and FTIR analysis.

5. Conclusions

We successfully synthesized silica particles from rice straw waste using simple extraction method. The experiment was conducted by heating and extracting rice straw waste into basic solution. To get silica particles, the extracted solution was then put into acid solution and heated to remove the remained solvent. The experimental results showed that the aggregated silica particles with sizes of about 200 nm were successfully produced. Although this method succeeded to form silica particles, the KCl in the product is still problems as impurities. The existence of KCl was possibly from the use of KOH as the extraction agent. Therefore, further studies on how to produce high purity silica from rice straw waste and how purify the product are still needed.

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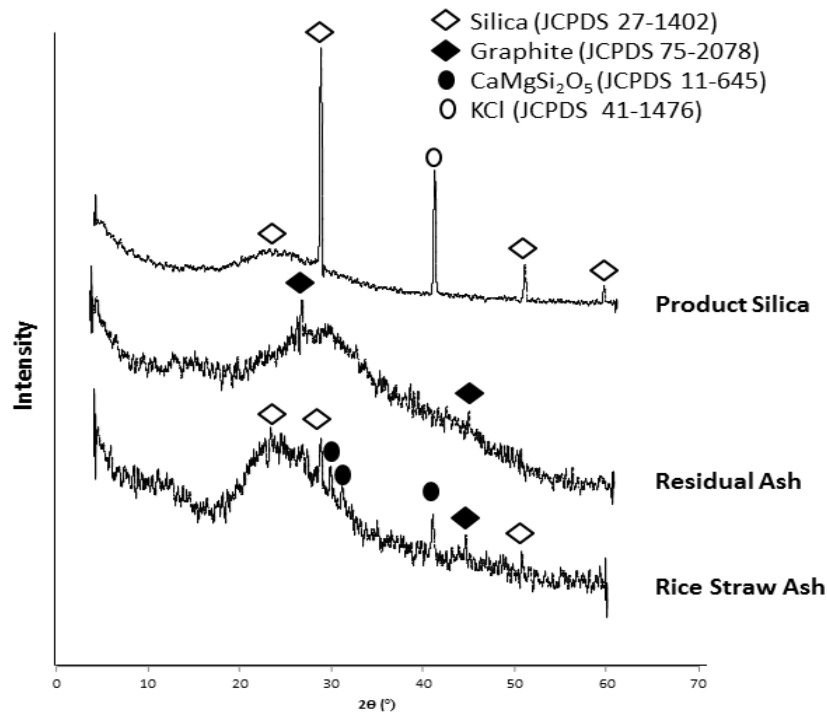


Figure 4. XRD analysis results of rice straw ash, residual ash, and product samples

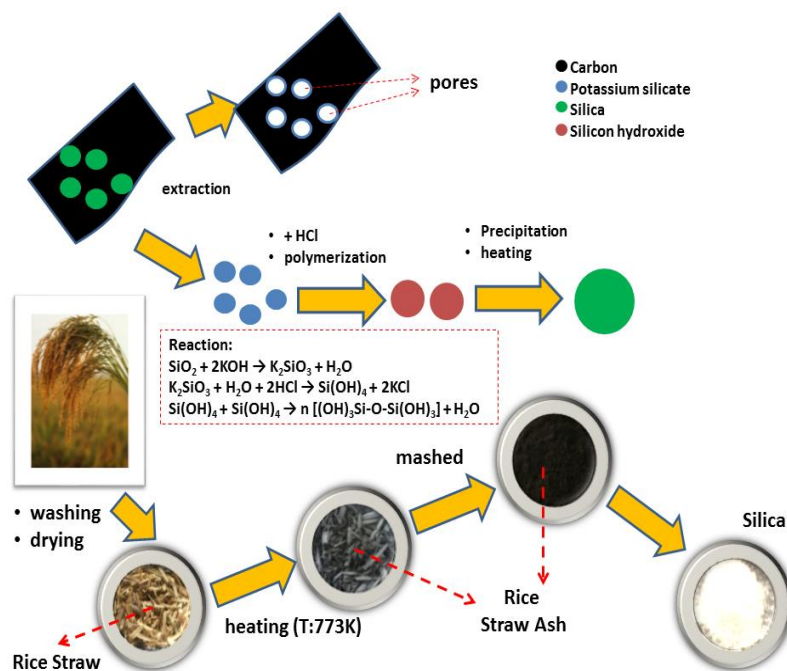


Figure 5. Hypothetical analysis for the synthesis of silica particles from rice straw waste

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