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Purity, Morphological, and Electrical Characterization of Silicon Dioxide from Cogon Grass (Imperata cylindrica) Using Different Ashing Temperatures

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Abstract

Cogon grass (Imperata cylindrica) was a detrimental plant and often found in the tropics which contains silicon dioxide. The objective of this study was to extract silicon dioxide from cogon grass and to study the purity, crystal structural, and electrical properties of silicon dioxide from cogon grass. The silica of cogon grass was extracted by washing dry cogon grass using 3% HCl before the burning process, and the ashing temperatures were 500°C, 600°C, and 700°C with a temperature increasing rate of 1°C/minute. The SEM-EDX results showed that silica of cogon grass has an irregular shape and a purity of 99.99%. the XRD results showed that silica has an amorphous phase with peaks at $2\theta = 21\text{-}23°$. The electrical analysis showed that the higher the ashing temperature led to the higher the conductance, the conductivity, and the dielectric constant on silica of cogon grass. Besides, the higher the frequency, the higher the electrical conductance and conductivity, but the lower the dielectric constant of silica. Based on the electrical conductivity, the silica of cogon grass was in the range of semiconductor materials.

Keywords: cogon grass; bio-silica; purity; morphological; electrical; semiconductor

1. Introduction

Cogon grass (Imperata cylindrica) is one of the most detrimental and hardly eradicate plants. It hardly decomposes, allelopathic and unpalatable, and rhizomatous perennial and pyrogenic. It has infested around 500 million hectares in the world [1-3], where Indonesia has a cogon grass population of 8.5 million hectares [4]. Cogon grass is usually utilized by the community in making house roofs, briquettes, and livestock, however, its utilization is still limited [5]. In addition, its roots are utilized as medicine, and the leaves are discharged into agro-waste. Cogon grass has a low silica composition compared to bamboo leaves and rice husk, which is only about 2.9 - 3.67% with a high cellulose content of 37.1 - 48.12% [6,7]. Silica has colorless crystal properties, a specific gravity of 2.2 -2.6, not soluble in water and acids except HF, soluble in alkaline solution, and melting points between 1600°C - 1750°C [8]. The potential for silica development is based on the wide use of these silica-based materials in today's industry. Silica powder in the amorphous form has several potential applications in the production of composite materials such as absorbents, aerogel, zeolite, reinforcement of coatings, pigment carriers [6], production of cement [9,10], and silicon carbide [11]. Besides being processed, silica is also used directly for refining vegetable oil, as an additive in pharmaceutical and detergent products [12]. Silica in the biomass can be extracted by acid leaching [13], where Kow [6]

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specifically explained that low levels of silica from cogon grass can be purified by HCl treatment.

Several studies related to the extraction of silica from biomass such as rice husk using 3% HCl and various ashing temperatures have been conducted such as Masrur et al. [14], Rohaeti et al. [15], Fatoni [16], and Irzaman et al. [17] by producing silica purity more than 99%. Besides, several studies on silica from bamboo leaf also have been conducted by Aminullah et al. [18,19] and Sa'diyah [20] as well as Irzaman et al. [21] using HCl leaching. The silica of bagasse charcoal and rice straw, and rice husk also have been studied by Adli et al. [22], Nazopatul et al. [23], and Sintha et al. [24], respectively, using HCl of 3%. Irzaman et al. [17] also reported that 3% HCl can produce the highest purity and electrical conductance, conductivity, and dielectric constant on silica of rice husk. However, data on silica content from cogon grass and its properties such as electrical, surface morphological, and structural have not been much documented.

This study aims to study and analyze the ashing temperatures on the silica content of cogon grass and measure its purity, surface morphology, structure, and electrical properties with several frequency sources.

2. Experimental

Cogon grass washing [16,20]

Cogon grasses were chopped (about 1 cm x 1 cm) before being put in a beaker glass, then mixed it using 3% HCl (12 mL of 3% HCl for 1 g of cogon grass), then heated on the hotplate at a temperature of 200°C for 2 hours. After that, the samples were washed using distilled water repeatedly until free of acid.

Charcoal process of cogon grass

The charcoal process of cogon grass was conducted by referencing the studies on silica of bamboo leaf [28-31] and rice husk [14,15,17]. The washed cogon grasses were dried using sunlight, then weighed as much as 1000 grams. Furthermore, dried cogon grasses were burned in an open chamber without additional fuel. Charcoal of cogon grass was then weighed, and the mass losses were calculated.

Ashing process of cogon grass charcoal and silica extraction [14,15,17-21]

Cogon grass charcoal was inserted into a porcelain dish and then burned in the furnace using the initial temperature of room temperature. After that, it raised to 400°C with a holding time of 2 hours at a temperature increasing rate of 1°C/minute. Subsequent heating was continued to the temperatures of 500°C, 600°C, and 700°C, respectively, with a holding time of 1 hour. Then, the temperature was decreased into room temperature. The resulted ash was weighed. To extract silica, the ash was heated in a furnace at 800°C and held for 1 hour using a temperature increasing rate of 1°C/minute, and then it was decreased into room temperature.

Analysis on silica of cogon grass

Silica powder of cogon grass was characterized by SEM with the magnification of 1000 and 3000 for surface morphological and EDX (JSM-6510LA Analytical Scanning Electron Microscope) for a component of silicon dioxide. The structure of silica was characterized by X-ray diffractometer (SHIMADZU XRD 7000 X-Ray Diffractometer mAXima) with CuKα λ=1.5406 Å, and the diffraction angle was scanned from 10° to 80° at a scanning rate of 0.02° per minute. LCR meter (HIOKI 3532-50 LCR HITESTER) was used for characterizing electrical properties such as electrical conductance, conductivity, and dielectric constant at frequencies were scanned from 0.05 – 5000 kHz with 200 points of observed frequencies.

3. Results and Discussion

The mass loss on cogon grass charcoal amounted to 51.33%, while the mass losses in the ashing process using temperatures of 500°C, 600°C, 700°C are 40.89%, 50.88%, and 51.47%, respectively. These show that the higher the ashing temperature leads to the higher the mass loss, which indicates that the higher the ashing temperature, the more effective the ashing process of cogon grass. All organic matters from cogon grass charcoal evaporate so that only the ashes are left.

Surface morphology and purity of silica from cogon grass using SEM-EDX

Figure 1 shows that the grain size of silica at the ashing temperatures of 500°C, 600°C, and 700°C are in the range of 2.647-6.765 µm, 2.5-6.,389 µm, and 2.33-6 µm, respectively. This indicates that the higher the ashing temperature leads to the smaller the size of silica. The temperature has an influence on surface morphology where higher temperatures cause silica to become denser and fused that make lower the number of pores and larger the surface area [25]. Besides, these figures also show the uniformity of silica's surface morphology, and there is a tendency that the higher the ashing temperature, the lower the grain formation. This morphology is consistent with Azmi et al. [26] and Fernandes et al. [27], which reported that the silicon dioxide particles were irregular geometry and jagged. These formations reveal that the silicon dioxide from cogon grass has an amorphous phase.

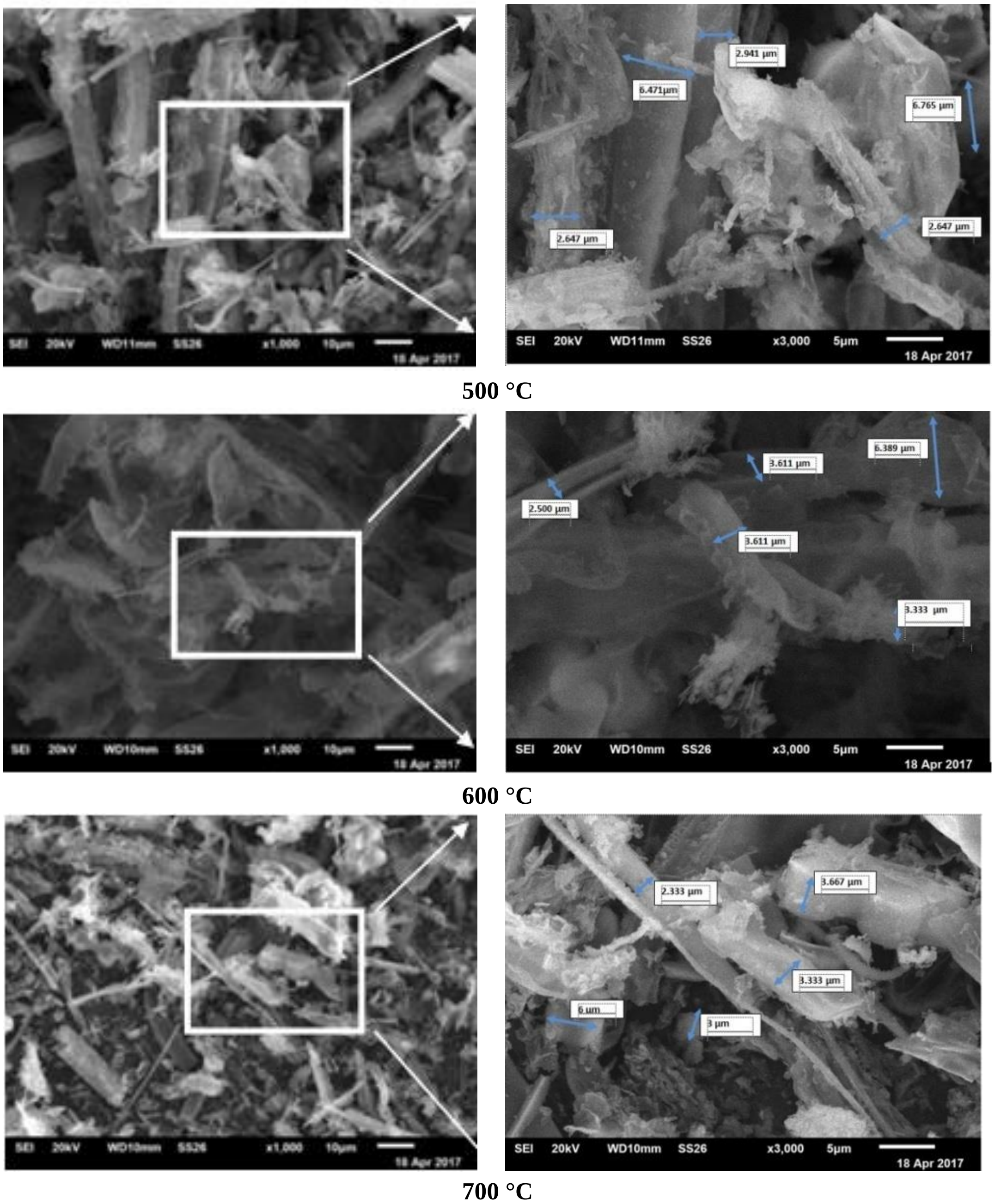

Fig. 1. Surface morphology of silica at ashing temperature of 500°C, 600°C, and 700°C using magnification of (a) 1000 x and (b) 3000 x

Liou [28] stated that a combination of acid or HCl addition and thermal decomposition can reduce impurities in a substance. Furthermore, Kow [6] explained that organic compounds in cogon grass will evaporate at temperatures of 200°C to 420°C. Silica purity can be measured using EDX, which is integrated into SEM. Table 1 shows that the purity of silica at all ashing temperatures reach 99.99%. The EDX of silica at 600°C shows that it has no impurities, while silicas at 500°C and 700°C have a gold (Au) element from the coating process. This high purity value is influenced by the washing process using HCl in the initial stage of silica extraction. According to Fatoni [16] and Sa'diyah [20] that the washing process using HCL before raw material combustion was more effective and easier to eliminate the impurities than washing after the combustion.

Table 1. Purity analysis of silica from cogon grass

Element	Ashing Temperature		
	500°C	600°C	700°C
Oxygen	36.62	47.9	53.17
Silicon	61.56	52.1	44.3
Gold	1.82	-	2.53
Silica Purity	99.99	99.99	99.99

Structure analysis of silica from cogon grass using XRD

The XRD analysis of silica as shown in Figure 2 shows that the absorption peaks at ashing temperatures of 500°C, 600°C, and 700°C are at 2θ = 21.8008°, 24.8641°, and 21,6604°, respectively. These temperature differences in producing ash of cogon grass affect the silica phase, where the amorphous phase is shown at all treatments. This is indicated by a sharp absorption peak in the range of 2θ = 22°, where the highest absorption peak in the range 2θ = 22° - 23° is a characteristic of amorphous silica [29]. Several studies [30-33] also suggested that ash of cogon grass has a peak centered at approximately 2θ = 23°. This data also supports the surface morphology of silica from cogon grass, which has an irregular shape of the amorphous phase.

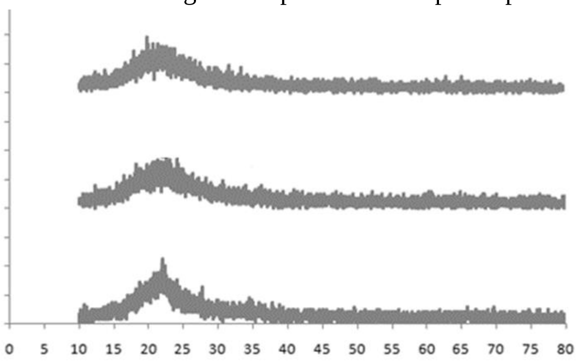

Fig. 2. XRD graph of silica from cogon grass at ashing temperatures of 500°C, 600°C, and 700°C

Electrical properties of silica from cogon grass using LCR

The electrical analysis on silica of cogon grass is conductance, conductivity, and dielectric constant analysis. Figure 3 shows a relationship of conductance and frequency from 50 Hz to 1 MHz, which indicates that the higher the frequency leads to the higher the conductance value of silica. An increase in frequency (energy) causes a faster and more movement of charges that make greater conductance value. This is in accordance with Irzaman et al. [17], which reported the higher the frequency, the higher the conductance of bio-silica from rice husks. In addition, the higher the ashing temperature, the more free charge can move.

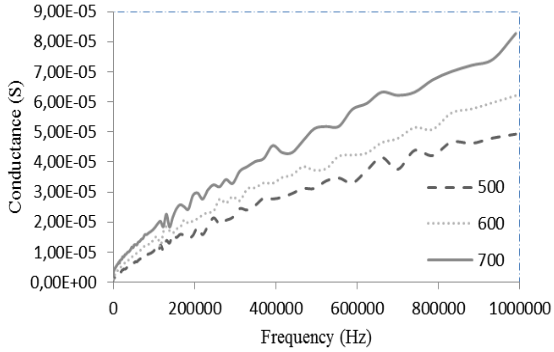

Fig. 3. Silica conductance at ashing temperatures of 500°C, 600°C, and 700°C with frequencies of 50 Hz - 1 MHz

Figure 4 shows that the higher the frequency is given to the sample and the ashing temperature leads to the higher the electrical conductivity of silica from cogon grass. This is in accordance with Irzaman et al. [17], who reported an increase in the conductivity of silica from rice husks by increasing the frequency. Increasing temperature and frequency make the electrons to get excited in the valence band and jump into the conduction band and make more the number of holes created. This movement of electrons and formation of holes increase the electrical conductivity. Conductivity values in the range of 1.2 x 10^{-6} - 0 indicate that the silica of cogon grass belongs to the semiconductor material. Kwok et al. [34] stated that semiconductor materials have conductivity values of 10^{-8} - 10^{3} $S.cm^{-1}$.

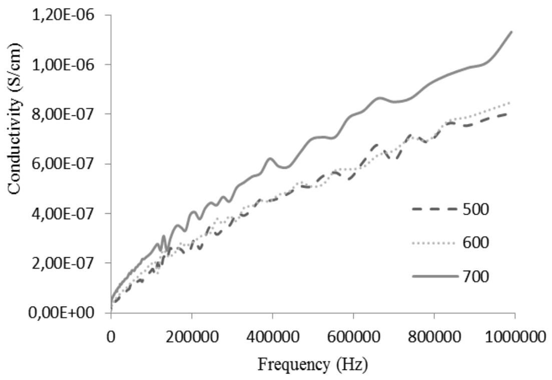

Fig. 4. Electrical conductivity of silica at ashing temperatures of 500°C, 600°C, 700°C with frequencies of 50 Hz - 1 MHz

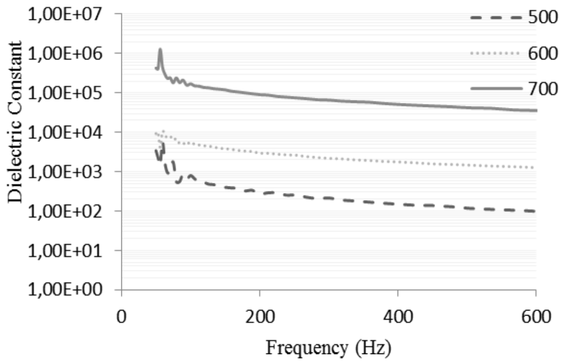

Fig. 5. Dielectric constant of silica at ashing temperatures of 500°C , 600°C, and 700°C with frequencies of 0.05-0.61 KHz

The measurement of the dielectric constant is conducted at low (0.05-0.61 kHz) and high (0.63-330 kHz) frequencies, where the results can be seen in Figures 5 and 6. Figure 5 indicates that minor element of gold (Au) also vibrate at ashing temperatures of 500°C and 700°C, which causes a peak at a frequency of 0.05-0.61 kHz. While at high frequencies of 0.63-330 kHz, the minor element does not vibrate so that the ideal curves are formed, as discussed by Sze [35]. This is also reported by Irzaman et al. [17] that gold (Au) can form a peak in the curve of the dielectric constant of silica from rice husk at low frequency. Those figures also show that the higher the frequency leads to the lower the dielectric constant of silica from cogon grass, which is in accordance with Irzaman et al. [17] on silica of rice husk. This is due to their dipole orientation, which correlates to unbalance the electrical field, causes a drop in the net polarization of the material, and then decreases its dielectric constant. In addition, as the frequency increases, the charge carriers move through the dielectric and get trapped at the defect site, so the speed of the charge carrier will be slow down and impact the lower dielectric constant [36]. This pattern also reported by Yadav et al. [37] where the higher frequency is given, the lower the dielectric constant and this follows equation 1 [38],

$$\varepsilon^* = \varepsilon_\infty + [(\varepsilon_0 - \varepsilon_\infty)/(1+i\omega\tau)] \qquad 1$$

where ε_0 is dielectric constant at low frequency, ε_∞ is dielectric constant at high frequency, ω is angular frequency, and τ is relaxation time.

4. Conclusion

Silicon dioxide of cogon grass at ashing temperatures of 500°C, 600°C, and 700°C has a purity of 99.99%. The surface morphology of silica from cogon grass showed irregular and non-uniform shapes, which indicate the amorphous phase of silica. This was supported by the XRD results, which showed peaks around 2θ = 21-23° of the peak of the amorphous phase. In addition, the higher the ashing temperature, the higher the conductance, conductivity, and dielectric constant of the silica. Increasing frequencies led to an increase in conductance and electrical conductivity, while the dielectric constant decreases. Based on the electrical analysis, the silica from cogon grass was in the range of semiconductor materials.

5. Conflicts of interest

There are no conflicts to declare.

6. Acknowledgments

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