Reduction of silicon dioxide from bamboo leaves and its analysis using energy dispersive x-ray and fourier transform-infrared

To cite this article: Aminullah et al 2018 IOP Conf. Ser.: Earth Environ. Sci. 209 012048

View the article online for updates and enhancements.
Reduction of silicon dioxide from bamboo leaves and its analysis using energy dispersive x-ray and fourier transform-infrared

Aminullah¹, E Rohaeti², B Yuliarto³ and Irzaman⁴,⁵*

¹ Department of Food Technology and Nutrition, Djuanda University, Bogor 16720, Indonesia
² Department of Chemistry, Bogor Agricultural University, Bogor 16680, Indonesia
³ Department of Engineering Physics, Bandung Institute of Technology, Bandung 40132, Indonesia
⁴ Departemen of Physics, Bogor Agricultural University, Bogor 16680, Indonesia
⁵ Surfactant and Bioenergy Research Center, Bogor Agricultural University, Bogor 16143, Indonesia
irzaman@apps.ipb.ac.id

Abstract. Silicon dioxide or silica from the organic materials such as bamboo leaf ash (BLA) was interesting because of its large content of 76-80% and it was the main ingredient in glass and solar cell industries. The objective was to study the effect of temperature rate and the acid leaching to silica content in bamboo leaves. This study consists of two main stages, namely 1) ashing the leaves which was conducted through burning the dry leaves and ashing the bamboo leaves charcoal with temperature rates of 0.3, 0.4, and 0.5 °C/min, and 2) reducing the silica which was conducted through leaching the ash with HCl 3% and then heating in a furnace. The results showed that BLA has a yield of 18.10 - 18.64% to dried bamboo leaves. Silica at rates of 0.3, 0.4, and 0.5 °C/min resulted in purity levels of 65.85, 74.49, and 72.69%, respectively. Silica still contained a low percentage impurities, namely rubidium oxide, aluminum oxide, and iron oxide. In addition, FTIR analysis showed that the silica rates of 0.3, 0.4, and 0.5 °C/min have absorption band at wave numbers of 1095 cm⁻¹, 802 cm⁻¹ and 455-463 cm⁻¹ which indicated the asymmetric stretching vibration and bending vibration Si-O of siloxane groups (Si-O-Si) with force constant of 1030.531 N.m⁻¹.

1. Introduction
Bamboo is one of the grasses which belongs to the Graminae family and part of a non-wood forest product commodity besides rattan, tengkawang, gondorukem and extractive substances. Bamboo is a composite material composed of cellulose fibers, which is embedded in lignin matrix [1]. It is widely distributed in most tropical countries where the plant can grow in the lowlands to the mountain slopes at an altitude of 3000 m above sea level. This plant has more spread on the Java island which reaches 29.14 million clumps or about 76.83% of the total population of Indonesia's bamboo, while the remaining approximately 8.79 million clumps (23.17%) are outside Java. Bamboo crops in Java are concentrated in three consecutive provinces in, respectively, West Java (28.09%), Central Java (21.59%), and East Java (19.38%) [2].

Bamboo leaves, which can be seen in Figure 1, are agro-waste which have potential to produce silicon dioxide or silica that has the chemical formula SiO₂. The silica of this organic material is then referred
to as bio-silica [3]. The ash content of bamboo leaf (BLA) was 20\%4 with silica content of 75.90 - 82.86\% [4,5] where the silica content of BLA is the second largest after ash of rice husk that is equal to 93.2\% [6]. However, as shown in Table 1, the percentage of impurities in BLA (compounds other than SiO\textsubscript{2}) is quite high when compared to the impurities in rice husk ash.

![Figure 1](image1.png) (a) Fresh and (b) dry bamboo leaves

**Table 1.** Percentage comparison of compounds between rice husk ash and BLA

| Materials              | SiO\textsubscript{2} (%) | Al\textsubscript{2}O\textsubscript{3} | Fe\textsubscript{2}O\textsubscript{3} | CaO  | MgO  | Na\textsubscript{2}O | K\textsubscript{2}O | Others |
|------------------------|--------------------------|--------------------------------------|-------------------------------------|------|------|----------------------|---------------|--------|
| Rice husk ash          | 93.2                     | 0.4                                  | 0.1                                 | 1.1  | 0.1  | 0.1                  | 1.3           | 3.7     |
| Bamboo leaf ash        | 75.90                    | 4.13                                 | 1.22                                | 7.47 | 1.85 | 0.21                 | 5.2           | 4.02    |

Silica is naturally present in quartz sand, opal rock, flint, and gemstone. This compound has several properties such as a colorless clear crystal, water and acid-insoluble except fluoride acid (HF), and melting point between 1600\degree C - 1750\degree C [7] with a density of 2.6 g.cm\textsuperscript{-3}. In industry, silica is utilized for the glass manufacture, ceramics, silica gel and silicic acid8 and as the most important raw material in the production of soluble silicate and silicon carbide [9]. In addition, the silicon chip industry utilizes silica from organic materials as an alternative source of silicon [10] besides silica sand as the primary source [11]. Silica in plants is found only in the form of amorphous silica [12]. It has a density of 2.21 g.cm\textsuperscript{-3} with an elasticity modulus of 10 x 10\textsuperscript{6} psi. The silicon and oxygen contents in this silica are 46.7\% and 53.3\%, respectively [13]. The schematic comparison of crystalline and amorphous silica can be seen in Figure 2.

![Figure 2](image2.png) Schematic comparison of (a) crystalline and (b) amorphous SiO\textsubscript{2}

The purity level measurement of silica from biomass has been carried out extensively in paddy [14-17]. The silica purity level of the rice husk are in the range of 60 - 99\% with increasing temperature rates of 1 - 5 \textdegree C.min\textsuperscript{-1}. This purity level is measured using an EDX (Energy Dispersive X-Ray) instrument commonly integrated with SEM (Scanning Electron Microscopy) instrument. In addition to the purity level measurements, on rice husk also carried out functional group measurements using Fourier Transform Infrared (FTIR) spectroscopy. From this measurement can be known the vibration pattern of a molecule and also the force constant of a molecular bond in which a molecule can be
viewed as a mass system connected by a bond with a springlike property, characterized by a constant, \( k \) (Hooke’s law). In this study, silica purity level of BLA is measured using lower increasing temperature rate than 1 °C.min\(^{-1}\) to obtain higher purity levels and eliminate impurities on BLA which are higher than silica in rice husk ash and to study the functional group pattern on BLA’s silica.

2. Experimental
The materials were bamboo leaves, 3% hydrochloric acid (HCl), and aquadest. While the equipments were furnace type 3-130 NDI Vulcan, hot plate, glass cup, magnetic stirrer, digital balance, spindle, dropper drops, universal indicator pH, filter paper, porselein filter funnel, and erlenmeyer tube.

The ashing process of bamboo leaf, which can be seen in Figure 3, through two stages namely burning dried bamboo leaves and ashing it using a furnace. The burning of bamboo leaves was conducted with refers to Dwivedi [4]. Dry bamboo leaves were weighed and burned in open space. The resulting bamboo charcoal was then weighed with a digital balance and the next process is done. The charcoal to be weighed is weighed by a digital balance sheet. This process refers to several studies of rice husk ash [14-17], where the weighted charcoal was introduced into the porcelain cup and then burned in a furnace using increasing rates of 0.3, 0.4, and 0.5 °C.min\(^{-1}\) then raised to 400°C with a holding time of 2 hours then raised again to 950 °C with a holding time of 1 hour and finally lowered to room temperature.

**Figure 3. The ashing process of bamboo leaf**

After heating, shown in Figure 4, the ash was weighed and washed using hydrochloric acid (HCl) 3%. The washing process was conducted as follows: first, the ash was inserted in a cup glass, then mixed with technical hydrochloric acid (HCl) 3% (12 mL of technical HCl to 1 gram of BLA), then heated on the hotplate with temperature of 200 °C and stirred at a speed of 240 rpm for 2 hours [15-17]. After that, the sample was washed using hot water repeatedly until it was acid-free (tested using litmus paper), then filtered with paper. The filtering result was heated in a furnace at 1000 °C. Then the sample was cooled in the furnace until equal to the ambient temperature. Finally, all the samples were tested using EDX and FTIR.

**Figure 4. Reduction process of silicon dioxide**
3. Result and Discussion

Dry yellowish bamboo leaves of 2000 grams are burned manually (regular combustion) [4] to form a perfectly blackish charcoal. The formed charcoal has a mass weight of about 500 grams or 25% of the total weight of dry bamboo leaves. The resulting charcoal was then taken as much as 120 grams, which is divided into three samples, to be ashed with the increasing rates of 0.3, 0.4, and 0.5 °C.min⁻¹ generate mass losses of 27.60%, 25.42%, and 26.62%, respectively. These mean that about 72.40 - 74.58% of the bamboo charcoal is ash or 18.10 - 18.64% of dry bamboo leaves. According to Dwivendi et al. [4], the ash content in bamboo leaf (BLA) was about 20%.

3.1. Silica characterization using EDX measurement

The elements contained in the silica samples are determined using EDX instrument type IVO Zeiss detector Bruker 133eV Germany which further determined the purity of silicon dioxide. The results are summarized in Table 2.

| Element      | Silica_1 % atom | Silica_2 % atom | Silica_3 % atom |
|--------------|-----------------|-----------------|-----------------|
| Oxygen       | 74.34           | 71.65           | 72.56           |
| Silicon      | 21.95           | 24.83           | 24.24           |
| Rubidium     | 2.45            | 3.07            | 2.94            |
| Aluminum     | 0.69            | 0.23            | -               |
| Iron         | 0.56            | 0.22            | 0.28            |
| Purity of silica | 65.85   | 74.49           | 72.69           |

Table 2 shows that the purity levels of silica at increasing rates of 0.3, 0.4, and 0.5 °C.min⁻¹ were 65.85, 74.49, and 72.69%, respectively. The higher the increasing temperature rate lead to the higher the purity level. The presence of impurities or compounds other than SiO₂ in the three samples of BLA tested is the cause of these purity levels. High percentage of impurities in BLA, which is about 10-12 times higher than that of rice husk ash [4] introduce lower purity levels than 99.99%. BLA’s silica in this study has a level of purity similar to that of rice husk ash husk in previous studies which were in the range of 76.17 – 99.15% [17,19,20]. Sa’diyah et al. [18] reported that purity level of silica in BLA was 67.62% with carbon as an impurity where the washing process of HCl was conducted before the combustion. This indicates that bamboo leaf is a potential source of silica after rice husk. In addition, this modified method can remove high impurities on BLA.

3.2. Silica characterization using FTIR spectroscopy

In addition to characterize BLA’s silica using EDX to know the elements contained in BLA samples, characterization was performed using FTIR to identify the functional groups in a compound in which each absorption of a specific wavelength describes the presence of a specific functional group. The resulting infrared pattern of BLA’s silica as shown in Figures 5-7.
Figure 5. FTIR spectra of silica_1

Figure 6. FTIR spectra of silica_2
The silica samples in Figures 5-7 demonstrate several peaks that indicate the presence of several functional groups in the samples, which are 1095 cm\(^{-1}\), 802 cm\(^{-1}\), and 463 cm\(^{-1}\) for silica_1; 1095 cm\(^{-1}\), 802 cm\(^{-1}\), and 455 cm\(^{-1}\) for silica_2; and 1095 cm\(^{-1}\), 802 cm\(^{-1}\), and 463 cm\(^{-1}\) for silica_3. The absorption band at 1095 cm\(^{-1}\) shows the presence of asymmetric stretching vibration of Si-O from Si-O-Si \[19\]. In addition, according to Launer \[20\], the absorption band at 794.6 - 806.2 cm\(^{-1}\) also shows the asymmetric stretching vibration of Si-O from Si-O-Si. While the absorption band at 455 - 470 cm\(^{-1}\) shows the bending vibration of Si-O from Si-O-Si. In addition, the absorption band at 620 cm\(^{-1}\) shows the stretching vibration of Si-O from Si-O-Si \[20\], however in this study there is no peak at this wave number. This is assumed at wave number of 620 cm\(^{-1}\) enclosed by molecular groups of silica impurities or more complex silica molecular groups.

Using equations (1) and (2) and compare them:

(i) \(n=0\rightarrow n=1, \Delta n=+1\),

\[\Delta \epsilon = \overline{\omega}_e (1-2x_e) \text{ cm}^{-1} \quad (1)\]

(ii) \(n=0\rightarrow n=2, \Delta n=+2\),

\[\Delta \epsilon = 2\overline{\omega}_e (1-3x_e) \text{ cm}^{-1} \quad (2)\]

where \(\Delta \epsilon_1\) and \(\Delta \epsilon_2\) are 802 cm\(^{-1}\) and 1095 cm\(^{-1}\), the \(x_e\) value is 0.1941 and by substituting it to equation (1), the \(\overline{\omega}_e\) value is 1311 cm\(^{-1}\) so that the frequency value, \(f\), can be obtained by substituting it to equations 3 and 4.

\[f = \frac{c}{A} = c\overline{\omega}_e \quad (3)\]

\[f_c = \frac{1}{2\pi} \left(\frac{k}{\mu}\right)^{-1} \quad (4)\]

where the \(\mu\) value of Si and O is \(1.68925 \times 10^{-26}\) kg, so that the force constant, \(k\), of Si-O is 1030.531 N.m\(^{-1}\) which can be seen in Table 3.
Table 3. The anharmonic force constant (N.m\(^{-1}\)) of silica with asymmetry stretching vibration of Si-O from Si-O-Si

| Sample   | \(\Delta \varepsilon_1\) (cm\(^{-1}\)) | \(\Delta \varepsilon_2\) (cm\(^{-1}\)) | \(\chi_e\)  | \(\tilde{\omega}_e\) (cm\(^{-1}\)) | \(f\) (Hz) | \(k\) (N.m\(^{-1}\)) |
|----------|-----------------------------------|-----------------------------------|-----------|-----------------------------------|---------|---------------|
| Silica_1 | 802                               | 1095                              | 0.1941    | 802                               | 3.933 \times 10\(^{13}\) | 1030.531 |
| Silica_2 | 802                               | 1095                              | 0.1941    | 802                               | 3.933 \times 10\(^{13}\) | 1030.531 |
| Silica_3 | 802                               | 1095                              | 0.1941    | 802                               | 3.933 \times 10\(^{13}\) | 1030.531 |

The Si-O-Si asymmetric stretching vibration present in the silica sample using FTIR can be determined by its force constant by assuming anharmonic oscillation is shown in Table 3. The force constant is identified as the bonding strength of the molecule. Anharmonic constant \((\chi_e)\) of Si-O from Si-O-Si of all samples has the same value of 0.1941 so that the force constant is 1030.531 N.m\(^{-1}\). This value shows that the temperature range of 0.3, 0.4, and 0.5 °C.min\(^{-1}\) produces molecules with the same strong molecular bond strength.

Conclusion
The ashing process of bamboo leaf consisting of two stages to produce about 500 grams of charcoal or 25% of dry bamboo leaves which were tested as much as 2000 grams. Whereas, the ash content produced from this process was about 18.10 - 18.64% of dry bamboo leaves. The reduction of silica from bamboo leaf ash produces purity level using EDX test were 65.85% - 74.49% with impurities of Al\(_2\)O\(_3\), Rb\(_2\)O, and Fe\(_2\)O\(_3\) due to high impurities in bamboo leaf ash and absorption band at 1095 cm\(^{-1}\), 802 cm\(^{-1}\), and 455 - 463 cm\(^{-1}\) showed the asymmetric stretching vibration of Si-O from Si-O-Si using FTIR spectroscopy with force constant of 1030.531 N.m\(^{-1}\).

Acknowledgement
We gratefully acknowledge the funding from USAID SHERA program through Centre for Development of Sustainable Region (CDSR).

Reference
[1] Xiaobo L 2004 Physical, Chemical, and Mechanical Properties of Bamboo and Its Utilization Potential For Fiberboard Manufacturing Theses (Chinese Academy of Forestry)
[2] Departemen Kehutanan 2004 Potensi Hutan Rakyat Indonesia Downloaded from http://www.dephut.go.id/Halaman/pranalogi_kehutanan/PHRI_03/PHRI_03.htm. In Indonesia.
[3] Hosseini M M, Shao Y and Whalen J K 2011 Biosystems Engineering 110 351
[4] Dwivedi V N, Singh N P, Das S S, and Singh N B 2006 International Journal of Physical Sciences 1 106
[5] Mohapatra S, Sakthivel R, Roy G S, Varma S, Singh S K and Mishra D K 2011 Materials and Manufacturing Processes 26 1362
[6] Chindapasirt P, Ruksan S and Sirivivatnanon V 2008 Construction and Building Materials 22 932
[7] Kurniati E 2009 Ekstraksi Silica White Powder dari Limbah Padat Pembangkit Listrik Tenaga Panas Dieng (Surabaya: UPN Press). In Indonesia
[8] Turner F M 1956 The Condensed Chemical Dictionary (New York: Reinhold Publishing Corporation)
[9] Kirk O 1982 Encyclopedia of Chemical Technology vol 20 (Taiwan: John Wiley and Sons inc.) p 749
[10] Irzaman, Darmasiatiwan H, Alatas H, Irmansyah, Husin A D, Indro M N, Hardienata H, Abdullah K, Mandang T and Tojo S 2009 Symposium Advanced Technological Development
of Biomass Utilization in Southeast Asia in Tokyo University of Agriculture and Technology

32-35

[11] Bates S P 2000 Silicon Wafer Processing (IISME-ETP: Applied Materials)
[12] Sangster A G and Parry D W 1981 Ultrastructure of Silica Deposits in Higher Plants In ed T L Simpson and B E Vocani Silicon and Silaceous Structure in Biological Systems p 385
[13] Mantell C L 1958 Engineering Material Handbook (New York: McGraw-Hill Book Company)
[14] Ahmad L 2011 Uji Struktur dan Sifat Listrik Silikon Dioksida dan Silikon dari Sekam Padi Theses (Bogor: Institut Pertanian Bogor)
[15] Hikmawati 2010 Produksi Bahan Semikonduktor Silikon dari Silika Limbah Arang Sekam Padi Sebagai Alternatif Sumber Silikon Theses (Bogor: Institut Pertanian Bogor)
[16] Muzikarno O 2013 Penambahan Magnesium Berlebih Dalam Menghasilkan Silikon Murni Dari Sekam Padi Sebagai Bahan Semikonduktor Theses (Bogor: Institut Pertanian Bogor)
[17] Masrur 2014 Optimasi Penambahan Magnesium Berlebih dan Kelajuan Pemanasan pada Reduksi Silikon Dioksida dan Silikon Berbahan Dasar Sekam Padi Theses (Bogor: Institut Pertanian Bogor)
[18] Sa’diyah H, Nurhimawan S, Fatoni S A, Irmansyah, Irzaman 2016 Prosiding Seminar Nasional Fisika 5. In Indonesia
[19] Silverstein R M, Bassler G C and Morril T C 1991 Spectrometric Identification of Organic Compound 5th ed. (New York: John Wiley & Sons. Inc.)
[20] Launer P J 1987 Infrared Analysis of Organosilicon Compounds: Spectra-Structure Correlations (New York: Brunt Hill)