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Ampel Bamboo Leaves Silicon Dioxide (SiO₂) Extraction

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Abstract. The bamboo tree trunk was the most commonly used part of daily life. Bamboo leaves often were considered waste by the community, and bamboo leaves contain Silicon dioxide (SiO₂). We have developed and compare two silicon dioxide method, using combustion to washing method (A) and washing to combustion method (B). Atom purity of either method was 99.9 %, with the tetragonal crystal structure. Mg, Au, Ca, and K impurities were found in Method A sample, and not in Method B.

Keywords: Silicon dioxide, Ampel bamboo leaves, amorphous.

1. Introduction

Indonesia was a tropical country rich in flora and fauna with variations and varieties. One of them is bamboo. There were 143 species of bamboo in Indonesia, and 60 species are on the island of Java. Bamboo leaves could grow in wet climates to dry climates, plateau or low [1].

Bamboo rod was the most common part used by the community to make paper, handicrafts and medicines. Utilization of other bamboo parts such as the roots, leaves, and branches of bamboo plants have not been fully utilized [1]. Bamboo leaves were often considered garbage by the public and received less attention. It was very regrettable because in bamboo leaves there are still compounds that can be utilized, namely silicon dioxide or silica [2].

Silicon oxide (silica) was the basic material for the manufacture of glass, ceramics, and refractory industries. Silica has been used for essential raw materials for the production of soluble silicates and silicon carbide [3]. The price of mineral silica was currently quite expensive, so the use of silica from organic materials has become another alternative in silicon chip industry [4]. The average weight of silica in the earth's crust was 27.7% [5].

In 2015, Aminullah has extracted silica from bamboo leaves with 3% HCl done after burning. The EDX test has shown that the purity of silica obtained is 65.85-74.49% with the impurities of Al₂O₃, Rb₂O, and Fe₂O₃ [6]. In 2016, Sa'diyah has extracted the silica from bamboo leaves by changing the phase in the extraction method, i.e. by transferring the washing process to before the combustion with the concentration of HCl to wash the bamboo leaves was 3%. The purity has been gained by changing this phase is 67.62% with one impurity, that was carbon. Therefore, it was necessary to optimize HCl concentration to obtain silica with higher purity and free of impurities. The objective was to calculate atomic purity and analyze the effect of HCl concentration (1%, 3%, and 5%) with two different extraction phases, and calculates the elemental composition and crystalline silica of Ampel bamboo leaves.

Bamboo has been known to contain high enough silicon oxide [7]. Based on the results of the research, bamboo has a cellulose content ranging from 42.4% -53.6%, lignin content ranges from 19.8% -26.6%, powdered 1.24% -3.77%, ash content 1.24% -3.77%, silica 0.10% -1.28%, extractive levels of 0.9% -6.9% and bamboo containing holocellulose (cellulose and hemicellulose) ranged from 73.32% -83.80% [8]. Due to the silica content owned by bamboo, it was possible to utilize bamboo to



be more optimal by extracting the silica content from the bamboo. Silica was a chemical that utilization and its application is quite wide in various fields [2][9]. Table 1 has shown the Chemical Composition of Bamboo Leaf Ash, whereas Table 2 have shown the Chemical Composition of Abu Husam Rice [10].

Table 1. Chemical Composition of Bamboo Leaf Ash [12]

Element	Mass (%)
SiO ₂	75.90
Al ₂ O ₃	4.13
Fe ₂ O ₃	1.22
CaO	7.47
MgO	1.85
K ₂ O	5.62
Na ₂ O	0.21
TiO ₂	0.20
SO ₃	1.06

Table 2. Chemical composition from rice husk ash [12]

Element	Mass (%)
Fe ₂ O ₃	0.95
SiO ₂	67.30
CaO	1.36
Al ₂ O ₃	4.90
MgO	1.81
L.O.I	17.78

Silicon dioxide was a compound that can be found in everyday life and often has been used as raw materials of the electronics industry [4]. Silicon atoms could form four bonds simultaneously and arrange tetrahedrally. In silica, each Si atom was bonded to four O atoms and each O atom was bonded to two sides of Si atom (Figure 1). The oxygen atom was electronegative and the atomic density of silica has been transferred to the oxygen atom [6]. Table 3 has shown the characteristics of SiO₂ [11].

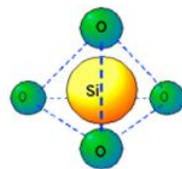


Figure 1. Crystal Structure Silica²

Tabel 3. SiO₂ Characteristics¹³

Crystal Structure	Amorf
Atomic Weight	60.08 g/mol
Density	2.27/2.18 g/cm ³
Molecule	$2.3 \times 10^{22}/\text{cm}^3$
Specific heat	1.0 J/g-K
Melting Point	1700 °C
Thermal expansion coefficient	$5.6 \times 10^{-7}/\text{K}$
Young Modulus	$6.6 \times 10^{10}\text{N/m}^2$
Poisson Ratio	0.17
Thermal conductivity	1.1 W/m-K – 1.4 W/m-K
Relative dielectric constant	3.7-3.9
Dielectric strength	10^7V/cm
Energybandgap	8.9 eV
DC resistivity	$10^{17}\Omega\text{cm}$

2. Materials and Methods

This research consists of two methods. Method A has started from the burning of bamboo leaves, bamboo charcoal staking, silica extraction, and analysis of the composition of crystal structure elements, and electrical properties. The result of extraction from method A was called silica A. Method B has been started from leaching (extraction process), bamboo leaves burning, bamboo charcoal leaching and analysis of elemental composition, crystal structure. The result of extraction from method B was called Silica B.

Bamboo Leaf Burning

Bamboo leaves were dried with the help of sunlight and weighed as much as 2 kg. Dried bamboo leaves have been burned in open space without additional fuel. The resulting bamboo charcoal then was weighed with an analytical balance [12].

Bamboo Coal Billing

The milling process was carried out by adopting several studies on the process of rice husk fertilization [4][7][12]. The bamboo charcoal was weighed 10 grams, then put in a porcelain dish and arranged so that it has the same thickness and burned in a furnace with an initial temperature of room temperature, then raised to 400 °C with 2 hours hold time. The next heating was continued with a temperature of 950 °C with a holding time of 1 hour. The temperature rise rate was set at 1 °C / min. Furthermore, the temperature was lowered to room temperature.

Silica Extraction Process A

After the probe process, bamboo ash leaves were weighed and washed using 1%, 3%, and 5% HCl. The washing process was aimed to reduce the impurities present in the ash of bamboo leaves other than silica. The washing process was carried out as follows: first ash was inserted into a cup glass, then mixed with 1% HCl, 3%, and 5% technical (12 mL 1% HCl, 3%, and 5% technical for 1 gram of bamboo ash), then was heated over hotplate, temperature control knob on the hot plate temperature to show the temperature scale of 200 °C and stirred with a magnetic stirrer at 240 rpm for 2 hours. Afterwards, the sample was washed with repeatedly acid-free double distilled water (aquabides) (tested using indicator pH), then filtered with ash-free filter paper. The result of the filtration was heated in a kiln at a temperature of 1000 °C for 1 hour.

Silica B Extraction Process (Bamboo Leaching)

The washing process was aimed to remove the existing impurities in the ash of bamboo leaves other than silica. Dry bamboo leaves have been soaked in 1%, 3%, and 5% technical HCl (i.e. 12 mL HCl 1%, 3%, and 5% technical for 1 gram of bamboo leaves). The sample was heated on top of the hotplate, the temperature setting button on the hotplate is set so that it shows a 200 °C temperature scale for 2 hours. Afterwards, the sample was washed using aquabides repeatedly until it was acid-free (tested using indicator pH). Furthermore, the sample was filtered and dried. After the washing process, then bamboo burning was done then the process of making bamboo charcoal briquette continues. The bamboo leaves have been heated in a kiln with a temperature of 1000 °C for 1 hour.

SEM/EDX

The resulting silica of all treatments was analyzed using SEM / EDX. The EDXini test is performed to identify the composition of the ingredients contained in the sample so as to determine the purity of the silica [13]. The morphology of the silica surface was analyzed using Scanning Electron Microscopy (SEM).

X-Ray Diffraction

X-ray diffraction X-ray spectroscopy (XRD) was used to identify the crystalline phase in the material by determining the lattice structure parameters. XRD has been used the principle of X-ray diffraction that undergoes scattering. The resulting diffraction pattern was representing the crystal structure. From the analysis of diffraction patterns, we could be determined lattice parameters, crystal size, crystalline phase identification. The type of material could be determined by comparing the XRD results with the catalogue of results diffused by various materials. The XRD randomized detector used was the SHIMADZU XRD 7000 X-RAY MAXima Diffractometer, the target source using CuK α 1 (λ = 1.54060 Å). Samples were prepared as much as 2 grams, then inserted into a holder of 2 × 2 cm in the diffractometer. The initial angle was taken at 10 ° and the end angle at 80 ° with a reading rate of 0.02 ° per minute. This analysis was conducted to find out the degree of sample crystallinity and calculate the lattice parameter value by using Cohan method. The results have been compared with the Joint Committee on Powder Diffraction Standards database (JCPDS).

3. Results and Discussions

This research has resulted in silica derived from Ampel bamboo leaves waste. Silica extraction in this study was obtained by using two methods, namely method A and method B. Method A began with the burning of bamboo leaves until the ampere of bamboo leaves, the bamboo leaves have been heated using a blast furnace to produce ash. The bamboo leaves of this ampel were then reheated in the furnace to obtain silica. The extraction silica using method A was called silica A. The bamboo charcoal of 40 amps of bamboo leaves was yielding 22.63 g of silica A. Method B has been started with the washing of bamboo leaves of Ampel. Bamboo leaves that have been washed and neutralized then burned to charcoal. Charcoal bamboo charcoal leaves were heated into the furnace until silica is obtained.

The extraction silica using method B was called silica B. The leaves of bamboo ampel was 80 g, which gives silica 8.15 g. Mass shrinkage for silica B extraction was more than silica A. The purity of Silica A was higher but has many impurities. This Impurity has been allowing for inaccuracies of the

data being analyzed because when testing the sample, the probable probability was the material other on impurities, so only one silica A sample could be tested SEM / EDX and XRD, while Silica B should be tested SEM / EDX and XDR for each washing variation.

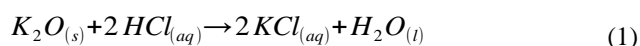
Silica Purity based on EDX Test

EDX test has been aimed to determine the chemical content contained in the sample. The EDX test results for method A and method B has been shown in Table 4.

Tabel 4. Percentage (%) of sample atoms of EDX test results

Elemental Name	Atom (%)			
	SilicaA		SilicaB	
	1%	1%	3%	5%
Oxygen	38.01	47.6	65.19	37.71
Silicon	54.89	52.29	34.81	60.19
Magnesium	0.98	-	-	-
Aurum	3.97	1.1	-	2.1
Calsium	1.13	-	-	-
Kalium	1.01	-	-	-
Silica Purity	99.99	99.99	99.99	99.99
Silicon Purity	39.43	28.04	2.215	42.435

Silica A was the silica obtained by washing process after combustion. Previous research has shown that silica A still contains some impurities, but the high purity of silica. So for silica A only sample by washing using 1% technical HCl hastested its chemical compositions because it has the best conductivity value. While Silica B was performed washing before burning, based on the calculation of the purity of silica atoms. Silica A has an atomic purity value of 99.99% with four impurities, namely magnesium, aurum, calcium, and potassium. When burning, the composition of the ampere bamboo leaves has undergone a chemical reaction so that there was a breaking of the bond. This breaking of the bonds has caused reactions that occur when leaching of bamboo charcoal using HCl cannot bind to the compound and the impurities found as shown in Table 4. The HCl concentration during washing was affecting the compound successfully attached by the HCl. Reactions that may occur when washing the bamboo leaves of the ample:



The higher the technical HCl concentration, the reaction will run toward the product. In the reaction was written in equation (1), the resulting product was KCl and H₂O. KCl was lost when washed with aquabides. While H₂O or water evaporates during heat treatment. The purity of silica B atoms based on the EDX test result was 99.99%. Silica B washed using 1% and 5% HCl concentrations have one impurity, ie aurum. While silica B washed using 3% HCl concentration did not have any impurities. Aurum was a layer used during testing to make silica more conductive. Based on table 4, there was no aurum content in bamboo leaves. Thus the aurum present in the sample was a deliberately coated impurity on the sample at the time of testing. Silica B with high purity present in Silica B by washing using 3% HCl was technically preferable because the percentage of atoms was close to the required value for obtaining pure SiO₂. In addition to silica, there was pure silicon in the sample. So if the silicon extraction could be done from the sample, the process will be easier.

Silica surface morphology based on SEM Test

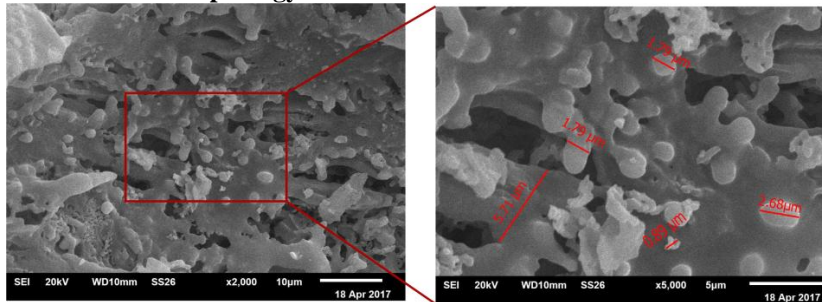


Figure 2. Morphology of Silica A by washing using HCl 1% technical magnification 5000x

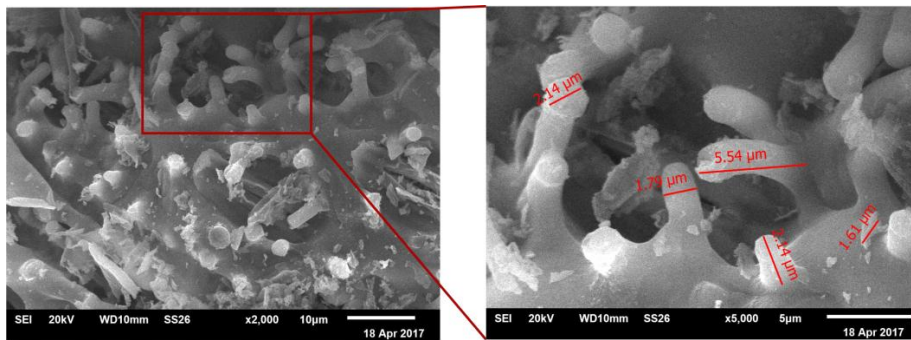


Figure 3. Silica B morphology with washing using HCl 1% technical 5000x magnification

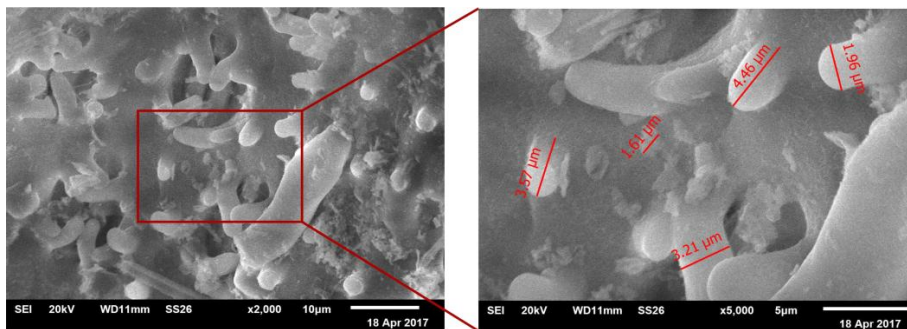


Figure 4. Morphology of Silica B by washing using HCl 3% technical magnification 5000x

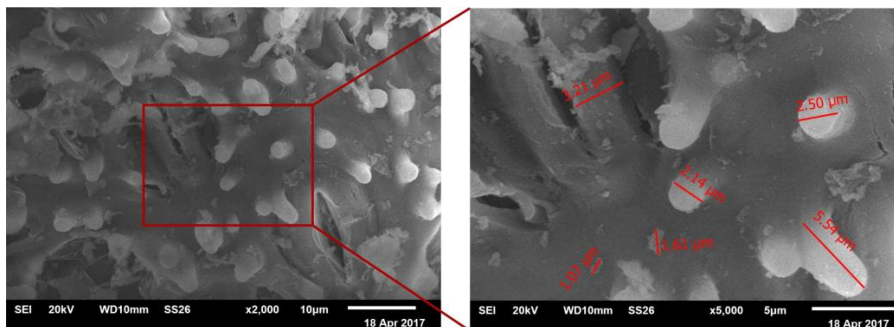


Figure 5. Silica B morphology with washing using 5% HCl technical 5000x magnification

The magnification performed during SEM testing was 5000x for each sample. The morphology of silica A and silica B surfaces was not homogeneous and there are elliptical clumps that look like colonies. In silica A, there are crystalline grains that have been dispersed although not homogeneous (Fig. 2). There are very few crystals was present in silica B (Fig. 3, 4, 5). The distribution of crystal grains that was visible on the surface morphology may affect the X-ray diffraction pattern on the XRD test. Silica B has produced images with more pores. This because silica B was more dominated by amorphous structures.

Grid Parameter Value and Degree of Silica Crystal based on XRD Test

The XRD characterization test yields the diffraction pattern shown in Figure 6. From the diffraction pattern shows prominent peaks. The peak then produces a lattice parameter of silica.

Table 5. Crystal and Silica Crystal Lattice Parameters based on XRD data

Silica Samples		Grid Parameters (Å)		Structure
		a=b	c	
Silica A	1 %	5.2798	7.5730	Tetragonal
	1 %	2.4471	8.4543	Tetragonal
Silica B	3 %	2.8103	4.2831	Tetragonal
	5 %	2.2946	8.5860	Tetragonal
JCPDS		4.9730	6.9230	Tetragonal

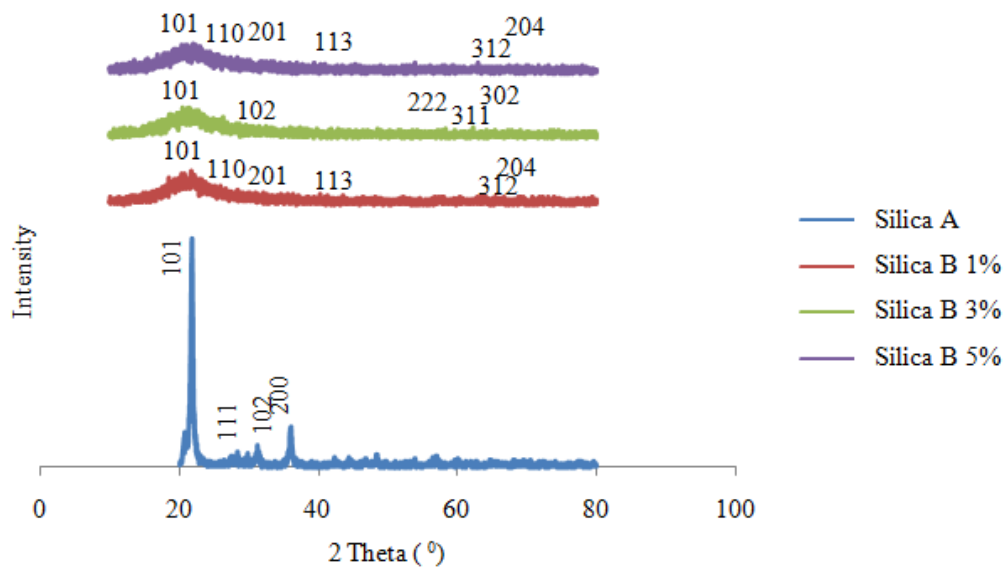


Figure 6. Pattern of X-Ray Diffraction on Silica A and Silica B

Table 5 has shown the values of the silica lattice parameters A and silica B compared with the lattice parameter values in JCPDS ICDD 1997. The values of the silica lattice parameter A was closer to JCPDS data than the values of the silica B lattice parameters. According to the XRD test, silica A has a higher crystallinity value than Silica B. The resultant peak based on the XRD test has shown that the intensity of silica A was higher and was found in four angles. The heat treatment has caused the amorphous crystal structure changes to either the crystal or the tridymite. In the combustion phase prior to washing it has caused the crystalline structure of Silica A to turn into a crystalline. This cristobalite structure has caused an increase in intensity when fired X-ray. High-intensity values have shown constructive interference. Constructive interference has occurred because of the similarity of phase, the same atomic orientation. Silica B was a silica with an amorphous phase so that the resulting peak was more gentle and steep with high intensity hard to find. The intensity was produced when X-

ray shooting on silica B was not high so that the peaks do not stand out. Washing on Silica B does not change the amorphous structure. The peaks formed on silica A and silica B did not correspond to the 2nd angle of the JCPDS database.

4. Conclusions

High purity silica has been obtained with two different leaching phases. Silica A has a purity of 99.99% but has many impurities, while Silica B has a purity of 99.99% with one impurity for silica washed using 1% HCl and 5% technical. The impurity was the gold obtained by the coating process during the SEM test. Silica B with 3% leaching was 99.99% purity without impurities. Silica A with washing using 1% HCl and silica B with a 1%, 3%, and 5% HCl washers was containing pure silicone content other than silica. With the existence of pure silicon content other than silica, then the silicon extraction process will be easier to do.

The effect of HCl concentration during washing was the binding of the compound performed by the HCl so that the purity and impurities of silica depend on the concentration of HCl. Silica has a tetragonal crystal structure. Silica A has a crystalline phase with a high degree of crystallinity. While silica B has an amorphous phase with a low degree of crystallinity. Silica A was a semiconductor for high frequency and isolator at low freq. This shows that silica A was not a good electrical conductor. While silica B was a semiconductor both at high frequency and low frequency.

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