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Extraction Silicon Dioxide (SiO$_2$) from Charcoal of Bagasse (Saccharum officinarum L)

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Abstract. Silicone dioxide (SiO2) was extracted from bagasse charcoal obtained from sugar factory. The charcoal was firstly dried at 105 °C for 2 hours followed by ashing at 800 °C and 900 °C. Treated bagasse was then washed with 3% HCl under stirring at 240 rpm at 200 °C for 2 hours. Subsequently, to neutralise the pH, the acid-washed bagasse was soaked in aquades repeteadly. Neutralized treated bagasse was then filtered using free ash filter paper. To obtain high purity silicone dioxide, the filtrate was heat treated at 800 °C for 1 hour. Physical properties of the obtained silicone dioxide were investigated using Spektrofotometer Fourier Transform Infra Red (FTIR), Scanin g Electron Microscope (SEM) and Energy Dispersive X-Ray (EDX).

Keywords: Bagasse charcoal, EDX, Extraction, SEM, Silicon dioxide.

1. Introduction
According to the Center for Agricultural Data and Information System of the Secretariat General of the Ministry of Agriculture 2014 in the "Outlook Komoditi Tebu," harvested area and production of Indonesian sugarcane increased consecutively to 466,119 ha and 2,550,992 tons in 2013[1]. Based on data from the Indonesian Sugar Plantation Research Center (P3GI), bagasse produced was 32% of each processed cane [2]. This bagasse is rich in silica content [3-4].

Silica or silica oxide is a chemical compound with the molecule formula SiO$_2$ (silicon dioxide) which was from mineral silica, vegetable, and crystal synthesis. Silica is the results of reacting silicon with oxygen or air at high temperatures [5]. Pure silicon oxide is present in two forms: quartz and cristobalte. Silicon is always tetrahedral bond to four oxygen atoms, but the bonds have ionic properties, while the structure silicon crystal is the cube.

Silicone oxide is as the base material of glass manufacture, while silicone with high purity level (> 95%) is useful for solar-cell, microchip computer, electronics, semiconductor and others [6-7]. This study is aimed to extract silica from bagasse. The analysis methods used in this study include: Fourier Transform Infra-Red (FTIR) Spectrophotometer analysis, XRD (X-ray Diffraction), Energy Dispersive X-Ray (EDX), Scanning Electron Microscope (SEM).
2. Materials
Charcoal of bagasse, as the silicon oxide resources, was obtained from PT. PG. Rajawali II Unit PG. Sindanglaut, Cirebon, Indonesia. The reagent used in this study was 3% HCl. The tools used for the analysis were Spektrofotometer Fourier Transform Infra-Red (FTIR), Energy Dispersive X-Ray (EDX), Scanning Electron Microscope (SEM). In addition the tools used were blast furnace, hotplate, magnetic stirrer, centrifugation, indicator pH, filter paper, gloves, mask, trash bag, analytical balance, husk furnace, matches, sifter, crucible furnace, mortar, porcelain cup, goblet, spoon, measuring cup, valve plastic, label, tissue, and stationery.

3. Methods
3.1 Preparation of Bagasse Ash
First, charcoal of bagasse was exposed to two step drying, sun and oven drying. Sun drying was conducted for 6 days. Afterwards, charcoal of bagasse was subject to oven drying at 105 °C for 2 h. Second, the charcoal of bagasse was fed into a porcelain dish and then was burned in a furnace until 400 °C. The burning rate was set as 1 °C per minute with 2 h holding time. Subsequent heating was continued with temperature variations of 800 °C and 900 °C with a holding period of 1 h. These two temperatures were selected according to the previous study indicated that amorphous silica occurs at a temperature of 550-800 °C and crystalline silica occurs at temperatures higher than 800 °C [8].

3.2 Silicon Oxide Extraction
The ash formed was washed with 3% reagent grade hydrochloride (HCl) acid (12 mL HCl 3% technical for 1 g bagasse ash). The washed ash was stirred by using hotplate at 240 rpm for 2 h and heated at 200 °C. Afterwards, the sample was washed using aquabides repeatedly until it was neutral (tested using indicator pH). The sample was filtered with ash-free filter paper. The filtrate result was heated to 400 °C with heating rate of 1 °C per minute with 2 h holding time. The sample was further cooled in the furnace trying until it reached temperature. Then the results were analyzed using Fourier Transform Infrared Spectrophotometer (FTIR, IRprestige-21, Shimadzu Corp., Jepang), and Energy-dispersive X-ray spectroscopy (EDX, JEOL JSM-6360LA) which was integrated to Scanning Electron Microscopy (SEM) to obtain the level of purity of silicon dioxide.

4. Results And Discussion
Sugar cane (Saccharum officinarum L.) is a green grass that thrives in hot and humid locations. Sugarcane is included in the family of poaceae and well-known as grass clumps commonly used to make sugar. Sugarcane grows in the lowlands of the tropics some subtropical regions. The main benefit of sugarcane is as a raw material for making sugar [9]. In addition to produce sugar, the waste derivation of sugarcane (bagasse) have the potential which is converted as a raw material of energy and useful chemicals. In addition to reduce waste, the utilization of bagasse to silica has the potential to substitute commercial silica [8].

The obtained sample is determined by the elements that is contained with the EDX characterization. Results of EDX test of silicon oxide of bagasse with 2 temperature variations of 800 °C and 900 °C with Oxygen value > 66.67% and Silicon < 33.33% is in Table 1. According to Aminullah et al. (2015), the purity of SiO₂ is calculated from the presentation (%) of atomic data that is the presentation of oxygen (O) and silica (Si) atoms. Thus, SiO₂ has three atoms (one of Si atom and two of O atoms) [9].
Table 1. Results of EDX Analysis

| Element Name | Percentage (%) of atoms | 800°C | 900°C |
|--------------|--------------------------|-------|-------|
| Oxygen       | 76.27                    | 77.04 |
| Silica       | 22.17                    | 22.96 |
| Magnesium    | 0.60                     | -     |
| Kalium       | 0.96                     | -     |
| Purity of SiO₂ | 66.51                 | 68.88 |

Figure 1. EDX Characterization of silicon dioxide, (a) 800 °C and (b) 900 °C

Figure 2. Morphology of silicon dioxide sample 800 °C (a) 20.000x magnification (b) 30.000x magnification

The washing process aims to reduce the content of impurities in bagasse other than silica. The EDX results indicated that the sample has no impurities at when it was treated at 900 °C while there is magnesium 0.60% and potassium 0.96% when it was treated 800 °C. In addition, the higher the heating temperature of SiO₂ purity level, the higher and the more perfect the proliferation process. All organic elements vaporize, and inorganic elements dissolve in HCl during washing [9]. Table 1 shows the SiO₂ obtained at heating at temperature of 900 °C and a temperature of 800 °C which is 68.88% and 66.51%, respectively. Cordeiro et al. (2011) burned bagasse produced silica percentage and ignition loss respectively 78.3% and 0.4% at 700-900 °C [10].
EDX combined Scanning Electron Microscope (SEM) was conducted to determine the morphology of a material [11]. The SEM-EDX result of the obtained samples are provided in Figures 2 and 3. SEM images were obtained at a voltage of 20.00 kV with 20,000x and 30,000x magnification. SEM images showed that the surface structure of silica bagasse is constructed of heterogeneous pores. The pore size is about 0.58 μm to 0.96 μm at a temperature of 900 °C and 0.19 μm to 0.5 μm at 900 °C at 30,000x magnification.

The results of FTIR SiO$_2$ spectrum of bagasse with a temperature of 800 °C and 900 °C are presented respectively in Figs. 8 and 9. According to Mohd et al. (2017), the peak vibration of Si-O-Si represents the silicon oxide characteristic in the sample [12]. The transmittance peaks show 1,076.28 cm$^{-1}$ (at 800 °C) and 1,411.86, 1,064.71 and 991.41 cm$^{-1}$ (at 900 °C). That matter indicates the presence of Si-O-Si bonds. The resulting Si-O-Si peaks is same with the previous study in the range 969 cm$^{-1}$ [13], 1,091 cm$^{-1}$ [14], 1,100 cm$^{-1}$ [15]. Amorphous silicon oxide show a peak of about 800 cm$^{-1}$ [16].

![Figure 3. Morphology of silicone dioxide solder 900 °C (a) 20,000x magnification (b) 30,000x magnification](image)

![Figure 4. FTIR spectrum of extracted SiO$_2$ from bagasse](image)
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