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Extraction and Characterization of Silicon Dioxide from Rice Straw

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Abstract. Study of silicon dioxide (silica) extraction from biomass has been successfully done. The biomass used in this study is rice straw. That is one of the rice production waste. Rice straw burning method at initial temperature of 400\degree C to 900\degree C with heating rate of 1.7s for 2 hours. Then rice straw ash washed by hydrochloric acid (HCl) with a concentration of 3\% to remove the impurities. Last, that was burned at 900\degree C at a rate of 1.7 for 1 hour to obtain silica in crystalline form. The resulting silica samples were compared by commercial silica with purity of 99.95\%. Based on FTIR characterization data of rice straw silica has a functional group representing the formation of SiO\textsubscript{2} compound, that is at 483.39 cm\textsuperscript{-1}, 789.41 cm\textsuperscript{-1}, and 1095.65 cm\textsuperscript{-1} wavenumber. According to the XRD results also have shown the pattern of crystalline diffraction with crystallinity percentage of 80.40\%. The result of the characterization is similar to the pattern shown by the commercial silica used as the comparison. Although this method has succeeded in forming silicon dioxide, but the final result is very dense powder, so it difficult to crush. Therefore, further study is needed to find the most optimal temperature. The characterization also necessary to determine the purity of rice straw silicon dioxide.

1. Introduction

Rice straw is one of the waste from rice production in the form of stems after the seeds are separated. Usually, farmers (especially in Lombok, Indonesia) will burn rice straw after harvest. This happens because the lack knowledge about usefulness of rice straw. Rice straw has many potentials that can be utilized such as energy raw materials [1] [2] and as a chemicals [3] [4].

The rice straw ash has a chemicals content, one of that is silicon dioxide (SiO\textsubscript{2}) about 75\% [5]. silicon dioxide is a chemical that is widely used in the glass, building, and ceramic industry. Silicon dioxide can be produced from various biomass such as rice husk [6] [7] [8] [9] and bamboo leaves [10]. Silicon dioxide from rice straw can be extracted using chemical solutions such as KOH [4], HCl, H\textsubscript{2}SO\textsubscript{4} and HNO\textsubscript{3} [11] [12]. However, studies [11] [12] suggest that HCl solutions are more effective to remove metal impurities than H\textsubscript{2}SO\textsubscript{4} and HNO\textsubscript{3}.

This study aims to extract silicon dioxide from rice straw that referring to previous research methods [6] [7] [8] [9] [10]. The extraction agent used was HCl with a concentration of 3\%, then the heating
temperature was at 400°C then increased to 900°C at a rate of 1.7°C / min for two hours. Forming method of rice straw ash by heating process at initial temperature 400°C, then temperature increased to 900°C with rate of 1.7°C/min for 2 hours. The extraction agent used HCl with a concentration of 3%.
The extraction results obtained were characterized using FTIR (Fourier Transform Infra Red) and XRD (X-Ray Diffraction). FTIR is a tool used to determine functional groups formed from samples that have been extracted. XRD is a tool to determine the crystal phase of the sample. In this case, commercial silicon dioxide will characterized for compared by silicon dioxide from rice straw.

2. Material and Method
Material this study is rice straw, commercial silicon dioxide (SiO$_2$ 99.95% of purity), hydrochloric acid (HCl) 3%, and aquabides. Research methods consists of burning, ash, silica extraction, and characterization of rice straw.

2.1. Rice Straw Burning Process
This process begins from drying rice straw with the help of sunlight. The dried straw weighed as much as 1000 gr. Then the rice straw burned in the open space without additional fuel.

2.2. Rice Straw Ashes Process
The rice straw charcoal from the previous process weighed and placed in a porcelain dish. Then it heated using furnace with initial temperature 400°C with rate 1.7°C/min for 2 hours to obtain rice straw ashes. Then the temperature of heating increased to 900°C for 1 hour.

2.3. Silica Extraction
Rice straw ashes washed with 3% HCl as an extraction agent to reducing the impurity content other than SiO$_2$ in rice straw ash. Rice straw ashes mixed with 3% HCl solution with ratio of 12 ml 3% HCl for 1 gram of rice straw ashes. That sample heated on hotplate and stirred using a magnetic stirrer. Then the sample was filtered and the result was heated at 900°C for 1 hour.

2.4. FTIR and XRD Characterization
Fourier Transform Infra Red (FTIR) used to determine the pattern of SiO$_2$ functional groups from extracted silicon dioxide samples. The FTIR tool used is Perkin-Elmer Spectrum One. X-Ray Diffraction (XRD) used to analyze the crystal phase of the sample. The XRD tool used is the SHIMADZU XRD 7000 X-Ray MAXima Diffractometer. The initial angle took at 10° and the end angle at 80° with a reading rate of 0.02° per minute. The results of FTIR and XRD analysis of rice straw silicon dioxide extraction samples were compared with commercial silicon dioxide with purity of 99.95%.

3. Result and Discussions
Silicon dioxide extraction in this study is a preliminary study to producing alternative silicon dioxide from a biomass, which one of them is rice straw. The methods used consists of burning process to obtain charcoal and heating process to obtain ash at an initial temperature of 400°C, because at temperatures below 500°C the content such as lignin and cellulose in rice straw ash degraded [4].
While the temperature increased to 900°C, that intended to keep only the SiO$_2$ content remaining. The next step is washing rice straw as using 3% HCl, that process intended to remove the impurities that still contained in the ash. Then, the process is the neutralization with aquabides, that aims to eliminate the salt content of the previous process. Furthermore, heating at a temperature of 900°C ultimately yields a particle suspected to be a silica particle. The final heating temperature is 900°C,
because at temperatures above 500°C will form crystalline structural silicon dioxide, while heating at temperatures below 500°C will form amorphous silicon dioxide.

The results of silicon dioxide samples characterization from rice straw and commercial silicon dioxide using FTIR are shown in Figure 1 at the wavenumber range of 500 cm⁻¹-4000 cm⁻¹. At that figure seen the peaks of functional groups that represent the compounds were formed. Functional groups showing the formation of SiO₂ compounds represented by peaks at 483.39 cm⁻¹, 789.41 cm⁻¹, and 1095.65 cm⁻¹ wavenumbers of rice straw silicon dioxide sample. The peak at the 483.39 cm⁻¹ wave number indicates the bending vibration of the Si-O group [9]. The wave number of 789.41 cm⁻¹ shows the absorption peak for symmetric stretching of the Si-O-Si group [4]. While the wavenumber of 1095.65 cm⁻¹ shows the absorption peak for the Si-O-Si group with asymmetric stretching mode [4].

The result of absorption peak of commercial silicon dioxide has a pattern similar to the pattern of the rice straw silicon dioxide extraction sample. That can give the evidence that the silicon dioxide particle was formed from extraction sample. The absorption pattern has peaks representing SiO₂ functional groups, that is peak of 495.08 cm⁻¹, 800.15 cm⁻¹, and 1150.86 cm⁻¹ wavenumber.

At 495.08 cm⁻¹, absorption peak of the Si-O group with bending vibration mode formed. The absorption peak of Si-O-Si group with symmetric stretching modes also shows at 800.15 cm⁻¹ wavenumbers. While the Si-O-Si group with asymmetric stretching pattern represented by absorption pattern at wavenumber of 1150.86 cm⁻¹.

In addition, the absorption peak pattern from the extraction sample of rice straw silicon dioxide and commercial silicon dioxide powder saw the presence of a silanol OH (Si-OH) functional group. Silanol group is formed by the addition of hydrochloric acid which cause an increase of water content. The higher concentration of HCl will increase the protons in the solution, so that the content of silanol groups in silica extraction will increase [13]. That represented by the wavenumber between 3435.38 cm⁻¹ and 3854.09 cm⁻¹ in rice straw silicon dioxide sample, then the wavenumber between 3384.25 cm⁻¹ and 3904 cm⁻¹ in commercial silicon dioxide. The silanol OH group has an asymmetric stretching and bending vibration modes [14].
Characterization using the XRD of both samples shows in Figure 2 at the 2θ range of 10°-80°. The rice straw silicon dioxide samples resulted diffraction pattern of 7 peaks for SiO$_2$ with the highest intensity at 21.84° hkl (100). While the comparable commercial silicon dioxide also shows a similar pattern with rice straw silicon dioxide at angle of 20.89° hkl (100), 26.67° hkl (101), 36.57° hkl (011), 39.48° hkl (102), 40.31° hkl (111), 42.46° hkl (200), 45.84° hkl (201), 50.16° hkl (003), 54.89° hkl (202), 59.99° hkl (211), 67.77° hkl (212) and 68.19° hkl (203). Peak of the highest intensity of diffraction pattern occurs at angle of 26.66° hkl (101). Both samples have a crystal structure with 80.40% of crystallinity percentage for rice straw silicon dioxide and 98.64% for commercial silicon dioxide.

Table 1. Lattice parameters value of rice straw silica and commercial silica.

|          | Lattice parameters(Å) |
|----------|-----------------------|
|          | a=b | c         |
| Rice straw | 7.6 | 10.49    |
| Commercial | 6.9 | 9.37     |
| JCPDS     | 4.9 | 6.92     |

Based on XRD data, lattice parameters of rice straw silicon dioxide and commercial silicon dioxide can be calculated, then the values compared by JCPDS ICDD 1997 data. The value of each parameter has shown in table 1. According to data in the table, lattice parameter value of commercial silicon dioxide was closer to JCPDS data then lattice parameter value of rice straw silicon dioxide. That caused by commercial silicon dioxide has cristalinity value was higher than rice straw silicon dioxide.

4. Conclusions
We successfully extracted silicon oxide particles from rice straw using HCl as an extraction agent. Based on FTIR and XRD data from silicon oxide of rice straw has been shown to form SiO$_2$
compounds. The result of the characterization has a pattern similar to commercial silicon oxide that intended as a comparison. Although this method has succeeded in forming silicon oxide, but the final result is very dense powder, so it difficult to crush. This is probably because of the final heating temperature is not appropriate, because every kind of biomass has its own temperature in forming a good silica powder. As in previous studies with the same method, silicon oxide from rice husks has an optimal final heating temperature at 900°C, while for bamboo leaves at 1000°C. Therefore, further study is needed to find the most optimal temperature. The characterization also necessary to determine the high purity of rice straw silicon oxide.

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