Extraction of silicon dioxide from rice straw (Oryza sativa L.) with variation ignition temperature

I L Hapsari1, Irzaman2,3, and M N Indro2

1 Physics Program, Physics Department, IPB University, Dramaga Street, Indonesia
2 Department of Physics, IPB University, Dramaga Street, Indonesia
3 Surfactant and Bioenergy Research Center (SBRC) of the Institute for Research and Community Services (LPPM IPB), IPB University, Dramaga Street, Indonesia

Email: irzaman@apps.ipb.ac.id

Abstract. Rice straw is the largest wastes of the rice plant. Usually, rice straw is only burned after the harvest process by farmers, even though rice straw contains 75% of silicon dioxide. The temperature of ignition is one important process of extracting silicon dioxide. This variation of ignition temperature will affect characteristics of the silicon dioxide. This research used the ignition temperature of 700 °C, 800 °C, and 900 °C. In the first step, rice straw is burned using matches then the powder is washed using 3% HCl and then rinsed using distilled water, and the ignition process in the furnace with different temperatures was set up. Temperature of 700 °C, 800 °C, and 900 °C have a range of electrical conductivity values from 50 Hz – 5 MHz, 10245.37 Hz – 5 MHz, and 11502.15 Hz – 5 MHz that categorized as semiconductors and produces silicon dioxide with a purity of 80.97% atoms, 99.99% atoms, and 61.53% atoms. Silicon dioxide with an ignition temperature of 700 °C, 800 °C, and 900 °C has a degree of crystallinity respectively at 25.82%, 44.24%, and 45.61%.

1. Introduction

Production of rice is proportional to the increasing rice straw as the largest wastes of the rice plant. Rice straw cannot be processed optimally. Usually, farmers burn the rice straw after harvesting. This burning aims to accelerate the recovery process of land for the next planting period. However, burning straw makes some nutrients lost from land, air pollution, and produce greenhouse gas emissions [1].

Rice straw used as a source of biomass because more than 580 million tons are available worldwide every year [2]. 75% of inorganic compounds from rice straw are silicon dioxide (SiO2) or silica [3] [4]. Silicon dioxide used for the manufacture of glass, ceramics and silica gel [5]. Silicon dioxide from biomass can be used as an alternative to expensive mineral silicon dioxide. Besides, the average weight of silicon dioxide found in the earth's crust is only 27.70% [6].

Silicon dioxide in rice straw can be extracted by the process of ignition and the addition of acid solution. The process of ignition is an important process. The ignition process aims to remove moisture from rice straw, burning cellulose, lignin, the organic compound (carbon), and removing an inorganic fraction (other than silicon dioxide), so silicon dioxide is produced. Different ignition temperature will affect the purity of silicon dioxide, electrical properties, and the degree of...
crystallinity so that this research can obtain the optimal ignition temperature for the extraction of silicon dioxide from rice straw.

This research refers to previous researchers from IPB about the extraction of silicon dioxide from rice husk [7] [8] [9] [10]. Extraction of silicon dioxide in this research using a variety of ignition temperatures of 700 °C, 800 °C, and 900 °C. The characterization used in this study includes the purity of silicon dioxide using Energy Dispersive X-Ray (EDX), degree of crystallinity using X-Ray Diffraction (XRD), and material conductivity using Inductance Capacitance and Resistance meter (LCR meter).

2. Materials and Methods

2.1. Materials
This research used rice straw from Paciran District, Lamongan Regency, East Java. This research also uses hydrochloric acid (HCl) 3% and distilled water (aquadest).

2.2. Methods

2.2.1. Rice straw burning process. The burning process starts with weighing rice straw using scales. Rice straw dried under sunlight, and then burned at open space then filtered using 80 mesh sieve, after that weighed using analytical scales to see the mass shrinkage.

2.2.2. Rice straw ashes process. The straw powder put into a porcelain dish and then it was burned using a furnace with an initial temperature of 400 °C with a holding time of 2 h, then continued with temperature variations of 700 °C, 800 °C, and 900 °C with a holding time of 1 h and 1.7 °C/min temperature rise, and then the temperature was lowered to 50 °C and then lowered to room temperature.

2.2.3. Extraction of silicon dioxide. The ash was added with hydrochloric acid (HCl) 3% with a ratio of 1 gram of ash:12 mL HCl, then stirred using a magnetic stirrer at 240 rpm for 2 h on a hot plate and heated with a temperature of 200 °C. It is intended that particles in inorganic compounds can easily bind to the HCl molecule. Washed ash has an acidic pH so it needs to be heated with a temperature of 105 °C repeatedly with the help of centrifuge every 5 min until the pH returns to neutral and silicon dioxide is produced. Samples that have been a neutral pH then filtered using filter paper, then the powder placed in a furnace with an initial temperature of 400 °C and a holding time of 2 h, then raised to a temperature variation of 700 °C, 800 °C, 900 °C with a holding time of 1 h and a speed of increase in temperature of 5 °C/min. The temperature was lowered to 50 °C and then lowered to room temperature. This heating was for lowering water content in the sample.

2.2.4. LCR Meter, EDAX and XRD characterization. Inductance Capacitance and Resistance Meter (LCR meter) used for analyzing the electrical properties of the sample, one of which is conductance. This conductance value can be used to determine the value of electrical conductivity. This electrical conductivity is the ability to conduct an electric current of material. Energy Dispersive X-ray (EDX) looks at the particle composition determined from the peak intensity of X-rays relative to the theoretically calculated intensity of the corresponding elements so that the percentage of pure silicon obtained from silicon dioxide can be calculated. X-ray Diffraction (XRD) characterization is used to determine the degree of crystallinity. The result is the graph of X-ray scattering diffraction patterns identified based on the intensity and angle of 2θ formed, then the formed peaks are adjusted based on the data from the Joint Committee on Powder Diffraction Standards (JCPDS) no. 39-1425 in 1988.
3. Results and Discussion

In the method, drying rice straw under the sun can reduce the water content by 10% to 20% so that rice straw is flammable and release a little smoke [11]. If the water content lowers, the combustion power is higher [12]. The black color of the powder indicates that the powder still contains organic compounds and the color of the powder indicates that the powder contains inorganic compounds [11]. The powder of rice straw that has been obtained then filtered to produce a powder with a homogeneous size. The total time in the process of ignition is varied. This total time involves the rising time, holding time and downtime. If the temperature of ignition is higher, the time of ignition is longer. Besides, the heating process also makes mass to shrink. Heat treatment of rice biomass is divided into three temperature stages, 100 °C, 250 °C to 350 °C, 350 °C to 750 °C. The first stage will remove moisture so that 5% of the biomass is lost. The second stage burns cellulose and lignin and produces low molecular weight or carbon organic matter. The third stage oxidizes the residue completely (organic matter or carbon) or removes the organic fraction so that just silicon dioxide is left [13].

The addition of HCl to this method serves to eliminate impurities in inorganic compounds such as calcium, magnesium, iron, manganese, carbonate, oxalate and phosphate, and other metals in small quantities [11] and minerals so that silicon dioxide can be produced. The use of 3% HCl was based on Cahyani's research (14) which obtained the highest purity silicon dioxide using 3% HCl concentration compared to 1% and 5% concentrations.

3.1. Conductance and electrical conductivity

This LCR meter uses a frequency range from 50 Hz to 5 MHz, a voltage of 1 volt and a current of 5 mA with taking 200 points.

![Figure 1. Conductance of silicon dioxide A, B, and C](image)

Conductance is the ability of a material to transfer electric current. Figure 1 shows the conductance of silicon dioxide with 3 variations of ignition temperature tends to increase with increasing frequency. Silicon dioxide A has the highest conductance value followed by silicon dioxide B and C.
Figure 2. Electrical conductivity of silicon dioxide A, B, and C

Silicon dioxide A has a minimum and maximum electrical conductivity value at least $1.75 \times 10^{-7}$ S/cm and $3.14 \times 10^{-6}$ S/cm. This shows that silicon dioxide A includes a semiconductor. The electrical conductivity of semiconductors of $10^{-8}$ to $10^8$ S/cm. Silicon dioxide B has a minimum and maximum electrical conductivity value of $3.16 \times 10^{-10}$ S/cm and $2.07 \times 10^{-6}$ S/cm. At a frequency of 50 Hz to 9.69 kHz silicon dioxide B has electrical conductivity with range $5.11 \times 10^{-9}$ to $9.19 \times 10^{-9}$ S/cm. This is the electrical conductivity of insulator with range $10^{-18}$ to $10^{-8}$ S/cm. At a frequency of 10.25 kHz to 5 MHz silicon dioxide B has an electrical conductivity range of $1.01 \times 10^{-8}$ to $1.99 \times 10^{-6}$ S/cm. It includes the conductivity of the semiconductor. Silicon dioxide C has a minimum and maximum conductivity value of $6.68 \times 10^{-10}$ S/cm and $1.39 \times 10^{-6}$ S/cm. At a frequency of 50 Hz to 10.86 kHz silicon dioxide C has an electrical conductivity range of $4.39 \times 10^{-9}$ to $9.69 \times 10^{-9}$ S/cm. This is the range of electrical conductivity of the insulator. At a frequency of 11.50 kHz to 5 MHz electric conductivity ranges from $1.03 \times 10^{-8}$ to $1.16 \times 10^{-8}$ S/cm. It includes the conductivity of the semiconductor.

The conductance graph is proportional to the graph of electrical conductivity, this is because the conductance value is directly proportional to the value of electrical conductivity. As shown in Figure 2 the electrical conductivity of silicon dioxide A, B, and C has a rising graph. External frequencies will increase electrical conductivity because the motion of electrons increases so more electrons are likely to move through holes (holes). Electron motion in semiconductors occupies holes or electron hopping. It's related to electron mobility. Electron mobility is a characteristic of materials that are influenced by impurity factors, crystal defects, and phonons. The electricity around the semiconductor with a silicon dioxide band gap of 9 eV and the frequency used only 50 Hz to 5 MHz allows the transport of electron only take place in localized states, this is called electron hopping. At low temperatures, electron hopping easy to find when the localized state in the mechanism of conduction [15]. Silicon dioxide A has the highest electrical conductivity followed by silicon dioxide B and C. The value of electrical conductivity obtained is not pure from silicon dioxide only, but all impurities also affect the electrical conductivity. Silicon dioxide A has metal impurity like Na, Mg, Ca, K with the highest percentage compared to silicon dioxide B and C. If the temperature of ignition is higher, then the electrical conductivity is lower.
3.2. The purity of silicon dioxide

The characterization of EDX used to determine the composition of silicon dioxide which can be seen in Table 3. From the elemental composition can be calculated the purity of silicon dioxide is produced.

| Elements  | A   | B   | C    |
|-----------|-----|-----|------|
| Carbon    | -   | -   | 22.18|
| Oxygen    | 64.89 | 62.25 | 51.76 |
| Sodium    | 1.26 | -   | 0.93 |
| Magnesium | 0.67 | -   | 0.53 |
| Silicon   | 26.99 | 35.65 | 20.51 |
| Chlorine  | 0.87 | 1.13 | 0.66 |
| Potassium | 3.39 | -   | 2.16 |
| Calcium   | 0.94 | -   | 0.61 |
| Aurum     | 0.99 | 0.97 | 0.65 |

Sample A is a sample with an ignition temperature of 700 °C, sample B is a sample with an ignition temperature of 800 °C and sample C is a sample with an ignition temperature of 900 °C. The highest purity of silicon dioxide is 99.99% produced at the ignition temperature of 800 °C (silicon dioxide B). This high purity of silicon dioxide is affected by very little impurity elements consist of Chlorine and Aurum only. Many impurities are missing due to the optimal process of the addition of the HCl and ignition process. Silicon dioxide A has a purity of 80.97% with impurities such as Sodium, Magnesium, Chlorine, Potassium, Calcium, and Aurum, whereas silicon dioxide C has a purity of 61.53% with impurities such as Carbon, Sodium, Magnesium, Chlorine, Potassium, Calcium and Aurum. If many impurities found in silicon dioxide, it will reduce the percentage of silicon dioxide atoms.

Silicon atoms bind 4 oxygen atoms and each atom of oxygen binds 2 parts of a silicon atom. Oxygen atoms have electromagnetic forces on the bonds of silicon dioxide and the density of silicon atoms is transferred to oxygen atoms [16]. Silicon oxide has 3 atoms (1 Si atom and 2 O atoms) [17]. The Aurum element found in all three temperature variations. It comes from an SEM tool in preparation. Samples should be using the conductor (Aurum) element as a detector intermediary with silicon dioxide. Sodium, Magnesium, Potassium, and Calcium are produced from the process of burning rice straw to ash. The elements of this inorganic compound are still present in silicon dioxide because the washing process using aquadest and addition of HCl is not optimal, this is supported by the presence of chlorine elements in silicon dioxide A and C. All chlorine (Cl) molecules originating from 3% HCl technically have not been able to completely form salt with impurity metal elements so it has not disappeared when rinsing using distilled water. Washing time influences Cl (Chlorine) molecules to bind perfectly with metals impurity.
3.3. Degree of crystallinity of silicon dioxide

XRD used sources of Cu and k\(\alpha\) with wavelengths of 1.5406 Å. Range 2\(\theta\) in this research using 10° to 80° with step 0.02°. The diffraction pattern formed was adjusted to the JCPDS 1988 data from the ICCD.

![Figure 3. Diffraction pattern of silicon dioxide](image)

The peaks of silicon dioxide from the diffraction pattern are compared with the JCPDS ICDD data to be determined hkl and are calculated the parameter of the lattice so that the structure of the material can be obtained. Figure 4 shows the diffraction pattern of silicon dioxide A with the highest diffraction peak at 2\(\theta\) 21.72° with hkl (101), followed by a peak of 25.80° with hkl (110) and 28.30° with hkl (111). Besides, from Figure 4 silicon dioxide B obtained a diffraction pattern with the highest peak at 2\(\theta\) of 21.88° with hkl (101), followed by 28.46° with hkl (111) and 36.06° with hkl (200). Figure 4 also shows the diffraction pattern of silicon dioxide C with the highest peak at 2\(\theta\) of 21.86° with hkl (101), followed by 25.8° with hkl (110) and 28.36° with hkl (111). The highest 2\(\theta\) value of silicon dioxide A, B and C approached the highest 2\(\theta\) value from the previous research about the extraction of silicon dioxide from rice straw which was carried out by Nazopatul (18) which was 21.84°.

![Figure 4. Diffraction pattern comparison](image)

| Sample code | Crystallinity (%) |
|-------------|-------------------|
| A           | 25.82             |
| B           | 44.24             |
| C           | 45.61             |

Table 2 shows the degree of crystallinity of silicon dioxide with 3 different temperature variations, silicon dioxide C has the highest degree of crystallinity followed by silicon dioxide B and A, which is 45.61%. If the temperature of ignition increases, then the degree of crystallinity will increase [19]. The variation of heating in the extraction process of silicon dioxide affects the phase of silicon dioxide. Based on the results of EDAX characterization, these three silicon dioxide have higher silicon dioxide atomic percentages compared to the other atomic percent elements, this is in accordance with XRD data produced at 2\(\theta\) 21.72°, 21.88°, and 21.86° with the same hkl (101) shows the diffraction pattern with the highest intensity. The highest presence of silicon dioxide peaks in Figure 3 indicates that silicon dioxide is insoluble in HCl during the washing process [20].
Table 3. Lattice parameter of silicon dioxide

| Sample code | Lattice parameter (Å) |   |   |
|-------------|----------------------|---|---|
|             | a = b                | c |
| A           | 5.66                 | 8.77 |
| B           | 5.11                 | 7.17 |
| C           | 4.95                 | 8.77 |

Silicon dioxide from rice straw has a tetragonal phase structure with lattice parameters $a = b = 4.97$ and $c = 6.92$ (JCPDS ICDD 1988). This lattice parameter is based on Bragg's Law with the equation $n\lambda = 2d \sin \theta$ [21]. The lattice parameter value that closest to the JCPDS ICDD data is silicon dioxide B which is $a = b = 5.11$ Å and $c = 7.17$ Å, this can be seen from the highest intensity at 20 = 21.88°. The three silicon dioxide with different ignition temperatures has a tetragonal structure. High intensity indicates constructive interference which is characterized by the existence of the same phase between atoms.

4. Conclusions

Silicon dioxide B has the highest purity is 99.99%. Silicon dioxide A and C have the purity of silicon dioxide are 80.97% and 61.53%. The higher the temperature of the ignition process, so then the electrical conductivity value is smaller, and the degree of crystallinity increases.

References

[1] Singh R, Srivastava M and Shukla A. 2016. Environmental sustainability of bioethanol production from rice straw in India: A review. Renewable and Sustainable Energy Reviews 54: 202-216.

[2] Kandanlou R, Bin Ahmad M, Shamel K and Kalantari K. 2013. Investigation of role of reductant on size control of Fe3O4 nanoparticle on rice straw. Bio Resource 9(1): 642-655.

[3] Jenkins BM, Baxter LL, Jr TRM, and Miles TR. 1998. Combustion properties of biomass. Fuel Processing Technology 54: 17-46.

[4] Binod P, Sindhu R, Singhania RR, Vikram S, Devi L, Nagalakshmi S, Kurien N, Sukumaran RK and Pandey A. 2010. Bioethanol production from rice straw. Bioresource Technology 101(13): 4767-4774.

[5] Sa’dyah H. 2016. Extraction silicon dioxide from bamboo leaves [essay]. Bogor (ID): Bogor Agricultural University.

[6] Irzaman, Oktaviani N and Irmansyah. 2018. Ampel bamboo leaves silicon dioxide SiO2 extraction. IOP Conf. Ser.: Earth Environ. Sci. 141: 1-8.

[7] Rohaeti E, Hikmawati and Irzaman. 2010. Production of semiconductor materials silicon from silica rice husk waste as alternative silicon. Materials Science and Technology. 265 – 27. Liu A. 2012. Testing of structural and electrical properties of silicon dioxide and silicon from rice husk [thesis]. Bogor (ID): Bogor Agricultural University.

[8] Masrur, Sofian I and Husein I. 2013. Optimalization of the speed of heating in the extraction of silicon dioxide (SiO2) from rice husk. Journal of Biophysics 9(2): 13-20.

[9] Sintha I, Dahrul M, Kurniati M, Irmansyah and Irzaman. 2017. Optimalization of silicon extraction from husk ashes by excessive magnesium addition on increasing rate of temperature reduction. IOP Conference Series Earth and Environmental Science 65(1): 1-7.

[10] Maelani A. 2015. Production activated carbon from rice straw using an activating agent H3PO4 [essay].Bogor (ID): Bogor Agricultural University.

[11] Chaeriawan M. 2016. Production of carbon briquettes from a mixture of bagasse and rice straw [essay]. Bogor (ID): Bogor Agricultural University.

[12] Sarangi M, Bhattacharyya S and Behera RC. 2009. Effect of temperature on morphology and phase a mixture of transformations of nano-crystalline silica obtained from rice husk. Phase bag Transitions 82(5): 377-386.
[13] Cahyani I. 2017. The effect of variations concentration of HCl (1%; 3%; 5%) on extraction SiO2 from rice straw at Ciherang at purity and electricity [essay]. Bogor (ID): Bogor Agricultural University.

[14] Zvyagin IP. 1973. On the theory of hopping transport in disordered semiconductors. Physics State Solid. 58(2):443-449. doi: 10.1002/pssb.2220580203.

[15] Irzaman, Oktaviani N and Irmansyah. 2018. Ampel bamboo leaves silicon dioxide SiO2 extraction. IOP Conf. Ser.: Earth Environ. Sci. 141: 1-8.

[16] Faiz M. 2013. Development of extraction of silicon dioxide from rice husk for semiconductor [essay]. Bogor (ID): Bogor Agricultural University.

[17] Nazopatul PH, Irmansyah and Irzaman. 2018. Extraction and characterization of silicon dioxide from rice straw. IOP Conf. Ser.: Earth Environ. Sci. 209: 1-5.

[18] Sembiring, Manurung P and Karo P. 2009. The influence of high temperature on the characteristics coordinate ceramic based silicon dioxide from rice. Journal of Physics and Application 5(1): 358-364.

[19] Ismawati S. 2015. Optimization on reduction temperature increase rate in (Si) extraction of rice husk ash with addition of Magnesium (Mg) excess [essay]. Bogor (ID): Bogor Agricultural University.

[20] Firdaus LH, Wicaksono AR, and Hidayat. 2013. Production H-Zeolit catalyst by impregnation KI/KIO3 and catalyst performance test for biodiesel production. J Tek Kim and Ind 2(2): 148-154.

Acknowledgments

Master Thesis Research Grant (PTM) Directorate General of Strengthening Research and Development of The Ministry of Research, Technology and Higher Education of The Republic of Indonesia with Contract Number: 3 / E1 / KP.PTMBH / 2019 dated March 29, 2019.