TURNITIN Homogenization of Green SiO2

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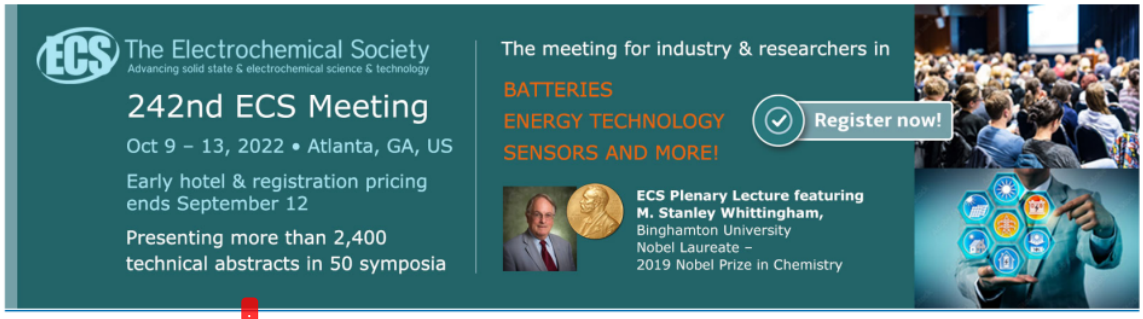

Homogenization of Green SiO_2 from Rice Husk Burn through Potassium Hydroxide Solid-Liquid Extraction

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Abstract. In the agricultural countries, rice husk is an abundant waste, especially as one of the largest sources of silica (SiO_2) production that can be produced. By complete combustion, to about 87% - 97% SiO_2 content can be produced from rice husks. Alkaline solution is used as a solvent in the solid-liquid extraction process of rice husk ash. The mass of 10 grams of rice husk ash was weighed for the extraction process added with 80 ml of potassium hydroxide (KOH) solution with 10%, 15% and 20% various concentration for 60 minutes to extract the SiO_2 content. The solution was added with 1 N hydrochloric acid (HCl) solution to precipitate the SiO_2, after the extraction process was complete. The SiO_2 formed is then separated from the rest of the solution by filtration. Next step is the drying process which aims to remove the moisture content of the resulting SiO_2. In a systematic study, for 60 minutes the rice husks were soaked and washed using HCl and then heated in a muffle furnace. The results of this study showed that all samples are succeeded in homogenizing SiO_2 with a purity close to 90%. Furthermore, through X-Ray Fluorescence (XRF) analysis was proven these results obtained through solid-liquid extraction of KOH from rice husks. Green SiO_2, known as biosilica, is useful and has potential in reinforcing compounds, including applications as filler in tires and natural rubber compounds.

1. Introduction

In developing countries, there are more than 75 countries that grow rice as a staple food source, and at least more than 97% of rice husks are produced [1, 10–17]. The main staple crop in Indonesia is rice, where the impact in an agrarian country is an increase in staple food production. The phenomena that occurred in 2006 was the expansion of the planted area from 11,786,400 to 14,116,600 ha, where the increase in the expansion rate reached 19.77% if accumulated since 2015. This was due to the encouragement of increased rice production. Meanwhile in 2006, 54,459,900 tons of rice were produced and up to 75,397,800 tons were produced in 2015. This means, there is an increase in production per year up to 3.84% [2]. From 2016 to 2019, on average the top 10 rice producers came from Indonesia, which is on the third ranks in the world as a rice production area, after China and India. China ranks first with an average rice production exceeding 211,378,225 tons, followed by India with 171,140,232.5 tons. Indonesia is in third place in increasing annual rice production exceeding 58,156,641.75 tons in 2019 [3].

In addition to producing rice, it also produces rice husks as a by-product. As many 80 million tonnes of dry milled rice were produced in Indonesia in 2017, and at least 16 million tonnes of rice

husks are produced per year. Factors that affect the composition of rice husks is geographical situation, in addition to the type of fertilizer and rice varieties also play a role. Organic components can be converted into 20 percent of carbon dioxide, air, and ash by burning rice husks. About 90 – 98% silica (SiO_2) is contains in the main component present in the rice husk, and Indonesia will produce 3.2 million tons of SiO_2 from rice husk ash per year [4].

Waste derived from rice husks is generated in most of all agricultural countries that able in producing rice. Utilization of rice husks is usually only disposed of even in open land for burning, which will result in environmental pollution and disturbance to the soil. The intention efforts is required to produce SiO_2 from the utilization of rice husk waste [5]. After complete combustion, in SiO_2 production, one of the important sources is rice husk, as many 87 – 97%. By controlling the combustion of rice husk ash at 500 – 600^oC elevated temperatures, many chemical processes are also can be used by SiO_2 ash formed [5]. SiO_2 is a compound of silicon dioxide (SiO_2) that can be used in various ways, it is the main standard material in the glass, ceramics, refractory industries and a play role for the silicate solutions manufacturing, include silicon alloys [6].

On the other hand, previous researchers (Kalapathy et al.; Pandiangan et al.; and Suka et al.) have conducted research on SiO_2 extraction by extraction processes with alkaline solvents and precipitation of SiO_2 with acids [7–9]. Kalapathy group on 2001, extracted SiO_2 using 1 N NaOH from the rice husks with the extraction method of two-cycle and result 91% yield [7]. In addition, Pandiangan group on 2008, conducted a study to extract SiO_2 using various concentrations potassium hydroxide (KOH) solid gel solution initiated by rice husks and 10% HNO_3 liquid additive solution as a trigger, and resulted the 1.8690 grams highest yield mass from rice husk ash mass of 50 grams at a concentration of KOH solution of 1.5% for 30 minutes. [8]. Another study conducted by Suka on 2008 was obtain the highest yield of 40.8% with the use of 5% KOH solvent with a reaction time of one hour [9].

This study is purpose to define the extraction process condition on the rice husk ash made in Indonesia into SiO_2 with different extraction times and KOH concentrations and to identify the formation of SiO_2 gel. The synthesis of SiO_2 gel by the extraction method is influenced by the concentration of the solvent, both acid and base. Generally, the acid solvents used are inorganic acids such as hydrochloric acid, sulfuric acid and nitric acid. In addition, the temperature and reaction time also affect the microstructure and purity of the SiO_2 gel produced. The effect of KOH concentration and considerations on the formation of SiO_2 gel is discussed in this study.

2. Experimental Methods

Rice Husk Ash (RHA) is used in this research with 10 gr mass. Aquadest (H_2O) is used for mixtures in the manufacture of 1N HCL and KOH solvents, also acid neutral from SiO_2 gel. The solid Potassium Hydroxide (KOH) is used is solid KOH dissolved using Aquadest with a certain ratio becomes KOH with various concentration of 10%, 15%, and 20%. The Hydrochloric Acid (HCl) was used of 37% concentration to 1N HCL. Table 1 shows the rice husk ash main component.

Table 1. The rice husks main component.

Component	Content (% weight)
Water content	9,0
Crude protein	3,0
Fat	1,1
Crude fiber	32,6
Ash	20,3
Crude Carbohydrates	13,7
Carbon (Charcoal)	20,3

The laboratory glassware which functions as a container for liquid chemicals i.e. erlenmeyer tube is used in this research. This glass is also often used for the titration process to accommodate the

solution to be used. A measuring cup is also used as a one tool to measure the volume of a solution or liquid that looks like a funnel or glass that has varying millilitres volume sizes. Hot plate magnetic stirrer is laboratory equipment used to heat and stir the solution with one another which aims to make a homogeneous solution with the help of a magnetic rod stirrer or stir bar. As metal extraction from ore is used a furnace or also often called a device used for heating such a smelting. A furnace is often used also in refineries of oil and in other chemical plants, for example as a heat source for fractional distillation pipes. Furthermore, as equipment that serves to heat or dry is also using the oven. Usually it used for drying laboratory glassware, chemicals and organic solvents. The oven is used to dry utensils before use and to dry wet ingredients. In addition, the oven is also used for sterilization using dry air. This sterilizer is used to sterilize glassware such as Erlenmeyer, Petri-disk or petri dish, test tubes and other glasses. Mortar and Pestle are tools used to crush a material or sample. The mortar is the part of the container, while the pestle is the part of the stem you are holding. The duration of grinding depends on the type of material, the strength of the grinder, and the expertise in using the tool. A digital scale is used for precise measurements of materials in laboratories. Spatula glass is to take solid chemicals or powders at the time to be weighed. Apart from picking up chemicals, another function of the spatula is to stir the mixture in the form of a solution.

Various ratio of alkaline solvent KOH is used for 10%, 15%, and 30% with the extraction time of 60 minutes. The particle size is 100/80 mesh, the weight of ash is 10 g, the extraction temperature is 90°C, the acid solvent type of HCL 1 N is choosen with the mixing speed of 100 rpm. The step by step of research procedure is as follows, first 5 kg of rice husk charcoal is used, followed by pound with a pestle until smooth. The husk charcoal that has been pounded is followed by filter using an iron sieve measuring 60 meshes. 500 g mass of rice husk charcoal is taken and then put into the furnace. The heating process of the rice husk charcoal is carried out inside the conventional furnace at temperature of 700°C and hold for 4 hours, until the ash generated. A pestle is used to pounded the result of ash, followed by filter using a 100-mesh sieve.

In addition, the SiO_2 Extraction Process is started by 10 g mass of dry rice husk ash to be dissolved in 80 ml of KOH with concentrations (10%, 15%, and 20%). The heating process is carried out using a hot plate on the temperature of 85°C while stirred with a magnetic stirrer for a 60 min time. After cooling, the solution is filtered with filter paper to separate the filtrate and residue. The resulting filtrate is taken.

The process making SiO_2 Gel are as follows, first is 30 ml of potassium silicate solution is prepared into a breaker glass. Second is gently dropping the potassium SiO_2 solution with 1N HCl liquid gel with a magnetic stirrer which used to stir the solution until a white gel with a neutral pH is formed. Let the gel that has formed at room temperature for 18 hours. Third is filter and rinse the SiO_2 gel with distilled water to remove excess acid. Fourth, the SiO_2 gel is heated inside an oven at temperature of 120°C until the weight is constant, then crushed with a mortar. Last is filtering with a size of 40 mesh and SiO_2 gel were obtained. The composition of the chemical in the dry rice husks and ash of husk are shown in Table 2 and 3, respectively.

Table 2. Chemical compositions of dry rice husks.

Element	**C**	**H_2**	**O_2**	**N_2**	**Si**	**Ca**	**Fe**	**Mg**
Contents (%)	41,44	4,94	37,32	0,57	14,66	0,06	0,006	0,003

Table 3. Chemical compositions of husks ash.

Content	**SiO_2**	**K_2O**	**P_2O_5**	**CaO**	**SO_3**	**Al_2O_3**	**Cl**	**MnO**	**Fe_2O_3**	**TiO_2**	**ZnO**	**Rb_2O**
Levels (%)	90,38	3,18	1,61	1,24	1,02	0,88	0,76	0,40	0,40	0,05	0,02	0,01

3. Results and Discussions

Extraction is a process of separating a mixture using solvents. The type of extraction used in this research is solid-liquid extraction. Solid-liquid extraction is a process that involves mass transfer between phases, in this case what acts as the solid phase is rice husk ash and KOH as an extractant. In solid-liquid extraction, when the extracted material is mixed with the extractant, the extractant will react with the solid to form an extract. In the solid-liquid extraction process, a very long contact between the extractant and the solid is required. SiO_2 contained in rice husk ash is recovered by dissolving it in an alkaline liquid solution. Reaction (1) is occurs in between the SiO_2 solid contained which generated from rice husk ash and an alkaline liquid solution, with the KOH extract solution.

$$SiO_2 + 2KOH \rightarrow K_2SiO_3 + H_2O \quad (1)$$

The SiO_2 compound that is formed is still in the form of a potassium silicate solution. Then the solution is added with 1 N HCl which is used to bind potassium to produce SiO_2. The reactions that occur is described on the reaction (2). Figure 1 and 2 are shows the burning rice husk charcoal before and after entered into a furnace, respectively.

$$K_2SiO_3 + HCl \rightarrow SiO_2 + KCl + H_2O \quad (2)$$

Figure 1. Burning rice husk charcoal before furnace treatment

Figure 2. Burning rice husk charcoal after furnace treatment

The result of rice The hydrochloric acid solution here functions as a precipitation agent. SiO_2 compounds are easy to solve in alkaline conditions, and the precipitation process occur in an acidic environment [7]. This phenomena is to create an SiO_2 consolidation easily extracted by rice husks, an alkaline gel solvent, KOH is choosed, and after that an acid solution, 1 N HCl, is used to precipitate it again. After the SiO_2 compound re-settles, the H_2O levels which affect the moisture of the product can be removed by drying in an oven. SiO_2 deposits produced in this process are SiO_2 deposits which still contain a lot of coprecipitation. The following is a picture of the stages of forming the extracted SiO_2 gel.

Figure 3. Potassium silica extracted

Figure 4. SiO_2 deposits formed after acidification

Figure 5. Dried coarse SiO_2 precipitate

Density changes that occur due to the extraction of SiO_2 husk ash using various variations of the KOH and variations of the time for the extraction process. It can be seen in the above photo that the density of SiO_2 gel has a different diversity from each time variation and the KOH concentration variation. The factors that affect density due to extraction include the size of the material. The smaller the particle size, the greater the area of contact between the solid and the solvent. Extraction temperature also affects the density yield. The solubility of the extracted material and diffusivity will increase with increasing temperature. However, too high a temperature can damage the extracted material, so it is necessary to determine the optimum temperature.

In order to identify the composition of the chemical and the purity of the SiO_2 which generated from the rice husk is used XRF. Table 4 shows the contains of SiO_2 and other metallic impurities. The data on the Table 4 shows the other metallic impurities have low amount of contains compared with the SiO_2 is as the major components. Based on the data, mentioned in removing the metallic impurities is effective by using KOH extraction in rice husks.

Table 4. Element contains of SiO_2 in the KOH extraction result.

Elements (%)	**10% KOH**	**15% KOH**	**20% KOH**
SiO_2	85.663	88.487	90.078
MgO	1.729	1.863	1.596
Al_2O_3	1.908	2.693	1.968
P_2O_5	1.583	2.058	1.971
SO_3	0.047	NA	NA
K_2O	0.350	0.011	0.045
CaO	0.063	0.061	NA
MnO	0.437	NA	0.545
Fe_2O_3	2.395	0.751	0.412
ZnO	1.543	1.271	0.645
Cl	0.500	0.338	0.543
Na_2O	3.782	2.467	2.197

4. Conclusion

Green SiO_2 or biosilica with a purity close to 90% is produced from rice husks by extraction of solid liquid KOH. Through the conditions of the process carried out, SiO_2 is obtained which has the potential for application as a filler in rubber and tire compounds. The use of adsorbents can be obtained from high-purity biosilica, as well as in fine chemical synthesis as a support of catalyst.

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