

Separation and Characterization of Silica from Rice Husk with Varying Heating Times

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ABSTRACT

Rice husk is a by-product of rice processing that has many uses but is rarely used by most people. Rice husk has abundant silica content which can be used as an adsorbent. This research was conducted to separate silica from rice husk by using the alkali fusion method with varying heating times (30, 60, 120, and 180 minutes) and to characterize silica using FTIR, XRD, and SEM-EDS. the maximum silica yield achieved was 50.75% after heating 60 minutes. The results of XRD characterization showed the presence of a spectrum in the 22° region at an angle of 2θ with a non-sharp spectral shape which is an amorphous nature of silica. In the FTIR test, there is a typical absorption of the silanol group in the absorption area of 1099.48 cm^{-1} , while the results of the SEM-EDS analysis show the percentage of silica mass is 34.39%.

Introduction

Rice is a plant that is widely available in Indonesia and is a staple food source for the majority of the Indonesian population. Processing rice as a food ingredient through the rice milling process will produce rice as the main ingredient and rice husks as a by-product. Rice husks are usually left piled up in the milling area or burned, which can cause environmental problems such as the spread of rice husk material in the environment around the rice milling location. A significant increase in rice production will affect the increase in rice husk waste as a by-product of rice plants. Accumulation of rice husks can be minimized because rice husks have many benefits by processing the rice husks. One potential that can be developed is rice husk as a source of silica, the content of which can reach 94% of rice husk ash [1]. Some of the benefits that can be obtained from processing rice husks include biocharcoal briquettes, a cement substitute, and a composite material [2] [3] [4]. Apart from being able to be used directly, the contents of rice husks also have many uses.

Silica is one of the supporting materials contained in rice husks which makes rice husks widely used for various purposes Silica can be used as an adsorbent, as a supporting material

for photocatalysts in degrading textile industry waste, as a stationary phase in chromatography columns, and as a synthetic material for Zeolite ZSM-5 [5] [6] [7] [8].

Silica is a compound resulting from the polymerization of silicic acid which is composed of chains of tetrahedral SiO_4 units with the general formula SiO_2 [9]. The process of separating silica from rice husks can be carried out using an extraction method by burning at high temperatures and then carrying out an alkalization process, namely dissolving it with an alkaline solution to bind sodium silicate and then dissolving it again with a strong acid solution [10]. The abundant presence of silica in rice husks and various resulting uses, it necessary to analyze the silica content contained in rice husks. In this research, silica was separated using the alkafusion method and analyzed for functional groups using FTIR, crystal phase using XRD, and surface structure using SEM.

Method

Material

The materials used were rice husks, NaOH (Merck), HCl (Merck), and distilled water. The instruments used were Shimadzu Brand Fourier Transform Infra-Red (FTIR), Bruker D8 Advance X-Ray Diffraction (XRD), and JSM-6510 Scanning Electron Microscopy (SEM).

Sample Preparation

A total of 50 grams of rice husks are soaked in hot water for 1 hour to clean the rice husks from dirt such as sand and gravel. After that, the rice husks are dried in the open air. Then the dried rice husks are put into porcelain crucibles and then heated in a furnace for 4 hours at a temperature of 600-700°C. The resulting ash was sieved with a 45-mesh sieve.

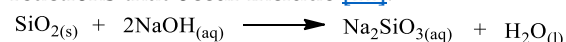
Silica Extraction

The Four 10-gram portions of rice husk ash samples were combined with 60 mL of 5% NaOH. This mixture was then heated with stirring at 85°C for different durations, specifically 30, 60, 120, and 180 minutes. The resulting filtrate underwent further treatment with 1 N HCl. The precipitate obtained from this process was washed with distilled water until reaching a neutral pH. Subsequently, the precipitate was subjected to heating in an oven and then weighed. The sample exhibiting the highest yield, obtained through silica extraction with varying optimal heating times, was selected for further analysis to determine silica characterization.

Result and Discussion

Silica Extraction

The extraction process uses the alkali fusion method, which is the process of dissolving silica in an alkaline solution and precipitating it again using concentrated acid. The addition of NaOH solution functions to form sodium silicate. The reactions that occur include [11]:



When adding a base solution, the silica compound formed is still in the form of a sodium silicate solution. Then 1N HCl is added whose function is to bind the sodium so that it separates from the silicate. HCl functions as a

precipitation agent. The reaction mechanism that occurs is as follows:

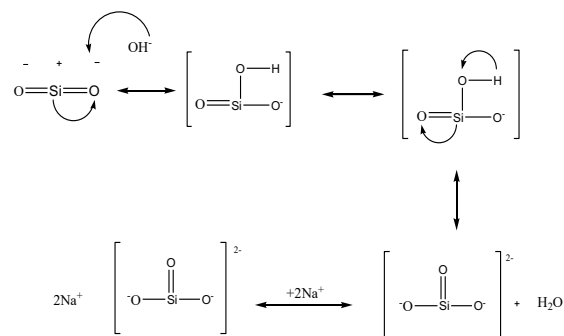


Figure 1. Reaction Mechanism of Silica and Base

In SiO_2 , the high electronegativity of the O atom causes Si to be more electropositive, and an unstable intermediate $[\text{SiO}_2\text{OH}]^-$ is formed, resulting in dehydrogenation. The second OH-ion will bond with hydrogen to form a water molecule and two Na^+ ions will balance the negative charge of the SiO_3^{2-} ion, thus forming sodium silicate (Na_2SiO_3) [12].

Table 1. The Effect of Extraction Time on the Silica Yield Produced

Time (minute)	Yield (%)
30	23.16
60	50.75
120	4.12
180	-

Silica extraction in this study used varying heating times, namely 30; 60; 120; and 180 minutes. The optimum heating time is 60 minutes. The yield affects the optimum time for the silica extraction stage. Time variations can be seen in Table 1.

The percentage yield obtained at 30 minutes and 60 minutes of heating is indicated that the silica extraction process takes place in stages until the optimum amount that can be extracted is reached. Meanwhile, an extraction time that is too long turns out to produce very little yield because the extraction time is too long resulting in most of the previously dissolved silica being reabsorbed by the husk so that it does not become a filtrate. Data obtained from variations in silica extraction time are as follows:

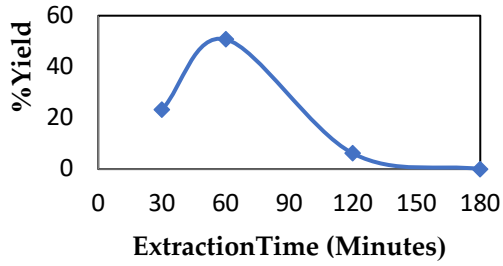


Figure 2. The Effect Time of Extraction on Silica Yield

FTIR Test

The FTIR test was used to analyze the silanol groups. The comparison of the IR spectrum between the standard silica spectrum and the sample silica spectrum shown figure 3. The standard silica spectrum for the OH functional group is at a wave number of 3437 cm^{-1} , while the spectrum for a silica sample is at a wave number of 3452.58 cm^{-1} . It can be seen that the wave number area around $3400\text{--}3500\text{ cm}^{-1}$ is the specification for OH stretching in silanol. Then the absorption band 2927.94 cm^{-1} shows that the silanol and H_2O -OH groups are absorbed and in the standard spectrum it is in the absorption band 3246 cm^{-1} . For the Si-O functional group, the sample spectrum is at a wavelength of 964.41 cm^{-1} , while the standard Si-O spectrum is at 1111 cm^{-1} , and for the Si-O-Si functional group, the sample spectrum is at a wavelength of 474 cm^{-1} . In the sample spectrum, it is at a wavelength of 468.70 cm^{-1} .

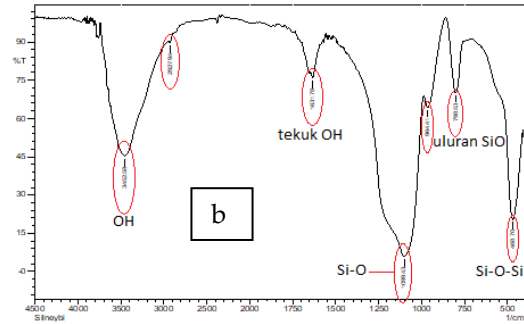
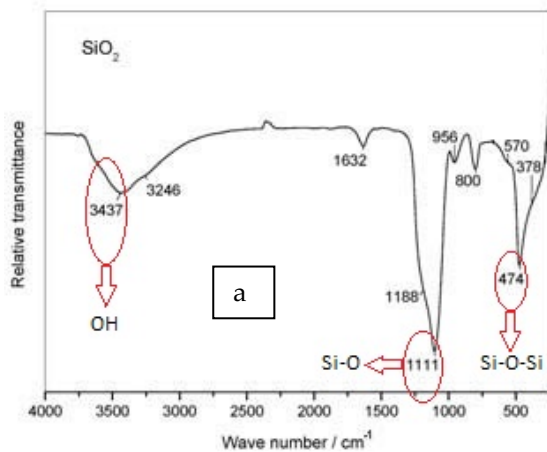


Figure 3. SiO_2 IR Spectrum (a) Standard [13] (b) Sample

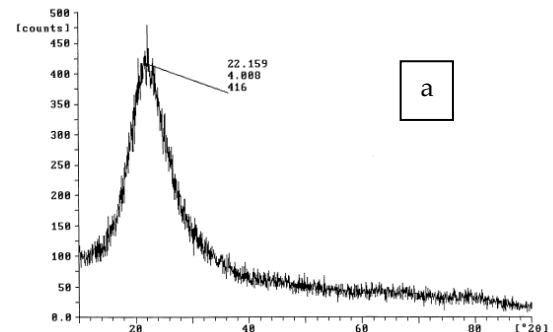
The tabulation of IR data can be seen in Table 2. From the comparison of the spectra of silica and standard silica samples, it can be seen that the resulting absorption is not much different.

Table 2. Sample IR Spectrum Data

Wave Number	Band	Intensity	Group
3452.58	Broad	Medium	O-H
2927.94	Sharp	Weak	O-H
1631.78	Sharp/ broad	Medium	O-H
1099.48	Broad	Strong	Si-O
964.41	Sharp	Medium	Si-O
798.53	Sharp	Medium	Si-O
468.70	Sharp	Strong	Si-O-Si

XRD Test

The specific peak produced usually ranges between $21\text{--}23^\circ$ at an angle of 2θ for silica material. The wavelength used in this research is $1.54060\text{ \AA}$.



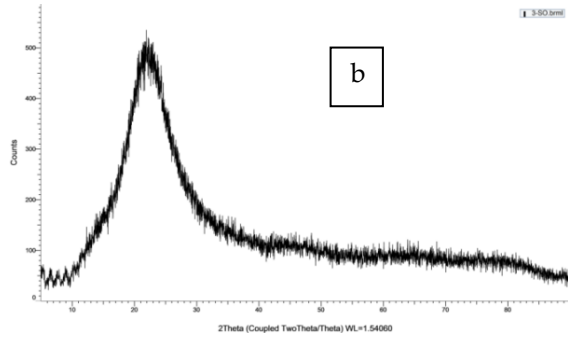


Figure 4. SiO₂ XRD Spectrum (a) Standard [13] (b) Sample

Figure 4 is a comparison between the XRD spectrum of the research results and the XRD spectrum of the silica sample. The similarity between the two spectra above is that the spectrum peak is in the area around 22-23°.

The characteristic crystalline nature of the spectrum will show a sharp peak with high intensity, whereas in the two spectra above the peak is slightly broadened. A spectrum with broadened peaks is characteristic of amorphous properties [14].

From the XRD data, the crystallinity percentage is 26.9% while the amorphous percentage is 73.1%, so it can be concluded that the silica sample phase from rice husks has an amorphous phase.

SEM/EDS analysis

In Figure 5 is obtained with an uneven surface, aggerated in several places with light and dark colors. It is suspected that the light-colored surface image in the aggerates is the element silica, while the dark-colored surface is other elements.

Metal elements with higher atomic numbers will produce brighter colors than metal elements with lower atomic numbers [15]. It can be seen that the dominant elements are Si and O. There is a correlation between the SEM and FTIR test results which is confirmed by IR data which shows that there are silanol groups in the silica samples.

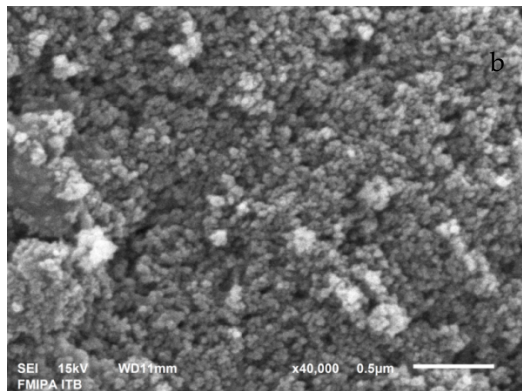
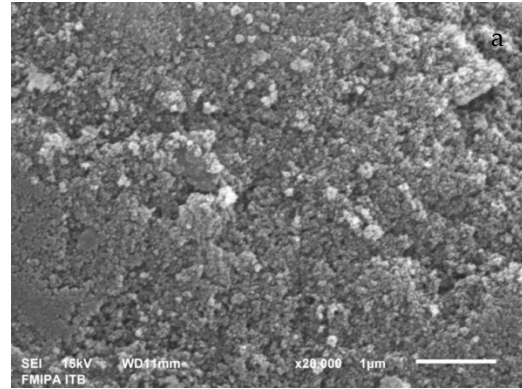


Figure 5. Sample Analysis Result (a) 20,000x (b) 40,000x

EDS data shown the chemical composition detected included C, O, Na, and Si. The results of the data above show that the most abundant elements are Si and O, while there are only a few Na and C elements. Apart from showing the possible phases contained in the sample, the EDS analysis results also show that the mass percentage of silica is 34.39%.

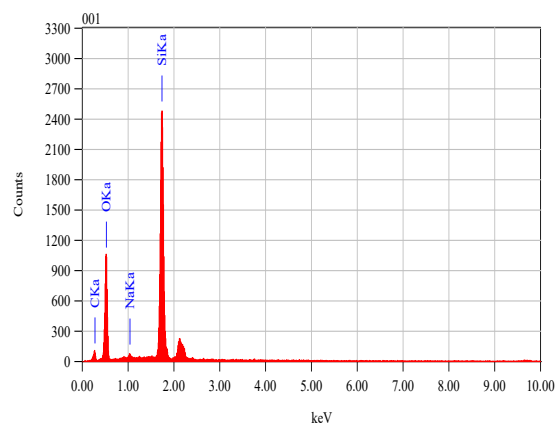


Figure 6. EDS Spectrum

Conclusions

The separation and characterization of silica from rice husks has been successfully carried out. The results of silica extraction with the greatest yield were at the optimum extraction time, namely 60 minutes. The FTIR test results show that there is a typical absorption of the silanol group in the absorption area of 1099.48 cm⁻¹. Silica characterization using XRD and SEM shows that the silica sample is in the amorphous phase and the mass percentage of silica is 34.39%.

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