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Synthesis of Silica from Rice Husk Waste Using NaOH as a Reagent

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Abstrak

Dalam penelitian ini, silika disintesis dari limbah sekam padi menggunakan NaOH sebagai reagen. Limbah sekam padi dipilih sebagai sumber silika karena ketersediaannya yang melimpah dan kandungan silika yang tinggi. Metode sintesis melibatkan ekstraksi silika dengan larutan alkali diikuti dengan presipitasi menggunakan berbagai jenis asam untuk memperoleh silika yang optimal. Proses ekstraksi dilakukan dengan dua variasi waktu, yaitu 60 menit dan 90 menit. Karakteristik fisikokimia yang dianalisis meliputi kadar air, kelembaban, dan kapasitas adsorpsi. Hasil penelitian menunjukkan bahwa silika yang diekstraksi dengan variasi waktu 90 menit lebih optimal, yang dibuktikan dengan tingkat kelembaban sebesar 48% dan kadar air sebesar 0,14%.

Kata Kunci: *Sekam Padi, Ekstraksi Silika, Reagen*

Abstract

In this study, silica was synthesized from rice husk waste using NaOH as a reagent. Rice husk waste was selected as the silica source due to its abundant availability and high silica content. The synthesis method involved silica extraction with an alkaline solution followed by precipitation using various types of acids to obtain optimal silica. The extraction process was conducted with two time variations: 60 minutes and 90 minutes. The physicochemical characteristics analyzed included moisture content, humidity, and adsorption capacity. The results indicated that silica extracted with a 90-minute variation was more optimal, as evidenced by a humidity level of 48% and a moisture content of 0.14%.
Keywords: *Rice Husk, Silica Extraction, Reagent*

INTRODUCTION

The increasing demand for rice in Indonesia, particularly in Bantaeng Regency, continues to rise annually. According to the Central Bureau of Statistics (2024), the estimated harvested rice area is 10.05 million hectares, with a total rice production of approximately 52.66 million tons of dry milled grain. Based on this production, the amount of rice husk generated as waste is approximately 15-20%, equivalent to 7,899-10,532 kg. Rice husk is composed of a highly rigid cellulose fiber network that contains significant amounts of silica (Rosmiyani et al., 2023). According to research by Yusrin et al., (2014), rice husk contains the highest silica content compared to other materials, such as coconut husk ash, which contains only 42.98% silica. The abundant rice husk waste and its slow natural degradation process have environmental and health implications (Pramudji et al., 2021).. However, the utilization of rice husk waste remains minimal (Pujotomo, 2017).

It is estimated that rice husk waste contains 87-97% silica after complete combustion, with most of it still underutilized (Meliyana et al.,2019). Silica, also known as silicon, silicate, or silicon dioxide (SiO_2), is derived from various sources, including plant-based silica, minerals, and synthetic crystals. This material is available in both amorphous and crystalline forms and has diverse applications, including use as a desiccant, adsorbent, filtration medium, and catalyst component. Additionally, silica is a primary raw material in the glass, ceramics, and refractory industries and plays a crucial role in the production of silicate solutions, silicon, and metal alloys (Kirk and Othmer, 2003). Silica gel is one of the inorganic solids that function as an adsorbent due to the presence of silanol (Si-OH) and siloxane (Si-O-Si) groups, which serve as active sites on its surface. Furthermore, silica gel has a large pore structure, a variety of particle sizes, and a distinct surface area, enhancing its effectiveness in adsorption applications (Kristianingrum *et al.,* 2016). The high silica content in rice husk waste makes it a potential raw material for silica production (Prameswara *et al.,* 2023). Processing this waste not only reduces environmental pollution but also adds value to the agricultural and industrial sectors. In addition to its abundance, silica from rice husk waste can be obtained easily and at a relatively low cost.

Silica can be extracted using an alkaline extraction method, which involves dissolving amorphous silica in an alkaline solution such as NaOH, followed by precipitation using an acid like acetic acid (Meliyana et al.,2019). The synthesis of silica gel requires specific treatments to achieve optimal results, which can be accomplished through various methods, including the sol-gel process, gas-phase method, co-precipitation, plasma spraying & forging, and emulsion techniques (Yusuf *et al.*, 2014). Consequently, the final characteristics of the synthesized silica depend significantly on the extraction method and the type of reagent used.

According to research by Utari *et al.*,(2020) the synthesis of silica gel involves modifying silica gel with diphenylcarbazone. Additionally, a study conducted by Thahir *et al.*,(2021) examined the util ization of rice husk as a silica source using a 10% NaOH extraction method, which yielded silica with a purity of up to 90.92%. Meanwhile, the study by Sholikha *et al.*,(2010) indicated that hydrochloric acid concentration affects the silica gel produced; higher acid concentrations result in increased yield. The optimal silica gel yield was obtained using a 5 N hydrochloric acid solution, achieving 8.2610%. Research conducted by Sarah et al., (2022) synthesized silica using acetic acid as a solvent, resulting in mesoporous silica with optimal physical and chemical characteristics. The use of acidic solutions facilitates the formation of silanol groups, which can bind water molecules through hydrogen bonds. The greater the number of silanol groups, the more hydrogen bonds are formed (Fathurrahman et al., 2020). Therefore, further research is needed on silica synthesis with acid modification as a gel-forming agent derived from rice husk.

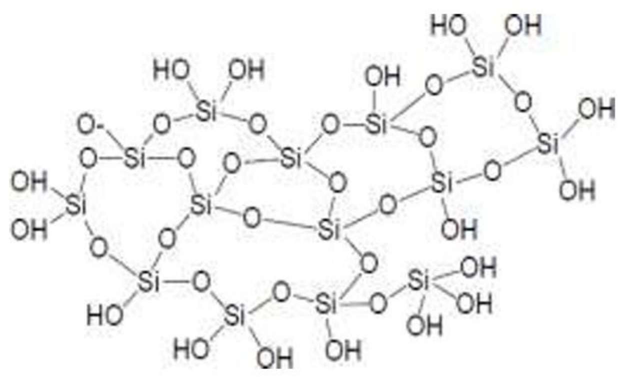

Figure 1. Silica Structure (SiO_4) (Kaim dan Schwederski, 2013).

RESEARCH METHOD

Preparation of Sodium Silicate Solution

The cleaned rice husk was oven-dried and then subjected to furnace heating at 800°C for 2 hours. According to research by Hadi and Sudiarta (2013) at temperatures ranging from 700-900°C, the resulting ash content remains constant at 21.00%. The combustion process converts organic material into silica-rich ash through the following reaction:

$$\text{Rice Husk} + O_2 \rightarrow SiO_2 + CO_2 + H_2O + \text{Byproducts}$$

After cooling, the rice husk ash was ground to increase the surface area. The ash was then extracted using 1 N NaOH at 80°C on a hotplate with a magnetic stirrer (with time variations of 60 and 90 minutes and a stirring speed of 200 rpm), producing sodium silicate via the reaction:

$$SiO_2 + 2NaOH \rightarrow Na_2SiO_3 + H_2O$$ (Kamath and Proctor, 1998)

The solution was then filtered and washed with heated distilled water to remove residues and obtain pure sodium silicate, followed by pH measurement.

Silica Synthesis

The obtained residue was treated with 1 N CH_3COOH (Widyastuti et al., 2022) until the solution reached a neutral pH of 7(Kalapathy et al., 2002), initiating the following reactions:

$$Na_2SiO_3 + 2CH_3COOH \rightarrow H_2SiO_3 + 2CH_3COONa$$

This was followed by condensation of silicic acid:

$$H_2SiO_3 \rightarrow SiO_2 + H_2O$$

And polymerization forming a silica network:

$$Si\text{-}OH + HO\text{-}Si \rightarrow Si\text{-}O\text{-}Si + H_2O$$

The resulting residue was allowed to stand for 22 hours to mature the gel, ensuring the formation of a stable silica gel network. The silica gel was then dried in an oven at 80°C to remove water content through evaporation, forming xerogel. The final silica gel appeared pink and gray (Nazriati *et al.,* 2018).

Application of Silica Gel for Air Humidity

Silica gel was placed in a ziplock bag/container, and a calibrated hygrometer was inserted. After one hour, the initial and final humidity levels were recorded. The interaction between silica gel and water vapor occurs through the adsorption process:

$$SiO_2 + nH_2O \rightleftharpoons SiO_2\cdot(H_2O)n$$

Determination of Moisture Content in Silica

A 1-gram sample of the produced silica was weighed in a pre-weighed crucible and dried in an oven at 110°C for 2 hours. After cooling in a desiccator for 30 minutes, the crucible with silica was reweighed until a constant weight was achieved.

RESULT AND DISCUSSION

The combination of NaOH and CH_3COOH (Retwan et al., 2024) offers several advantages: NaOH, as a strong base, effectively extracts silica, while CH_3COOH, as a weak acid, provides better control over gel formation. The resulting CH_3COONa salt is easily removed through washing, and the pore structure obtained is more uniform.

Moisture Content

Moisture content is a crucial parameter in assessing the quality of synthesized silica. Studies indicate that solvent combination, neutralizing agents, and extraction duration significantly impact the moisture content of silica gel. According to Ramadani (2018), low moisture content in silica gel reflects good dryness and purity, with an acceptable standard of less than 5%.

Experimental results showed that using NaOH combined with CH_3COOH as a neutralizing agent during 90-minute extraction produced the lowest moisture content of 0.14%. This finding aligns with the study by Putri et al.,(2022), which stated that NaOH is more effective in extracting silica than KOH. Additionally, CH_3COOH demonstrated superior neutralization compared to HCl, allowing for better-controlled neutralization (Riza.,2022).

Extending the extraction time from 60 to 90 minutes with NaOH tended to decrease moisture content, supporting Ramadani (2018), study, which suggested that longer extraction time facilitates further removal of water from the silica structure.

Table 1. Silica Content

No.	Solvent /Reagent	Neutralizing agent	Extraction time (m)	Content (%)
1.	NaOH	CH_3COOH	60	0,64
			90	0,14

Humidity Stability

Humidity is a crucial parameter in assessing the quality and effectiveness of silica gel as an adsorbent. Humidity testing is conducted by measuring the percentage of humidity before and after the sample is left for one hour, which serves to evaluate stability and water vapor absorption capacity. According to Alfiana *et al.*,(2018), good humidity stability can be identified by minimal changes in humidity values over a certain period.

The test results indicate that the combination of NaOH as a solvent and CH_3COOH as a neutralizing agent provides the most optimal humidity stability. The observed humidity change was relatively small, decreasing from 45% to 43% during the 60-minute extraction time and from 55% to 48% after 90 minutes. These findings align with the study by Riza.,(2022), which states that using weak acids such as CH_3COOH as a neutralizing agent can create a more uniform pore structure, thereby improving humidity control.

Furthermore, the effect of extraction time on humidity stability shows that a duration of 90 minutes generally results in silica gel with better performance. According to Putri et al., (2022), a longer extraction time allows for the formation of a more structured and stable silica network. The data indicate that samples extracted for 90 minutes exhibit more controlled humidity changes compared to those extracted for 60 minutes. These results are supported by Putri et al., (2022), who emphasize that optimizing extraction time is essential for producing silica gel with optimal adsorption characteristics.

Table 2. Humidity Stability

No.	Solvent /Reagent	Neutralizing agent	Extraction time (m)	Before (%)	After 1 hour (%)
1.	NaOH	CH_3COOH	60	45	43
			90	55	48

CONCLUSION

The extraction time and reagents used are crucial variables in silica synthesis from rice husk waste. The combination of NaOH and CH_3COOH, with longer extraction time, resulted in optimal moisture removal and improved humidity stability of the silica produced.

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