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SYNTHESIS AND CHARACTERIZATION OF SILICA USING RICE HUSK WASTE WITH MODIFIED REAGENTS

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ABSTRACT

Synthesize and characterize silica using the sol-gel method from rice husk waste by varying acid reagents, as an effort to utilize agricultural waste in a sustainable and environmentally friendly manner. Rice husks were combusted at 800°C to obtain silica-rich ash, then extracted with 1N NaOH to form sodium silicate. Silica precipitation was carried out using acetic acid, hydrochloric acid, and citric acid until a neutral pH was achieved. The synthesized silica was characterized by FTIR spectroscopy, moisture content, and humidity testing. Results indicated that acid variation significantly influenced silica properties. Silica produced with citric acid exhibited the lowest moisture content (0.0842%) and the most stable humidity (62%), indicating a compact structure and high purity. In contrast, silica obtained with HCl showed the highest moisture content (0.3212%) and humidity up to 95%, suggesting a porous and hygroscopic structure. FTIR spectra confirmed these findings through differences in –OH and Si–O–Si band intensities. Therefore, citric acid is the most effective reagent for producing high-quality and eco-friendly silica.

ABSTRAK

Penelitian ini berfokus pada sintesis dan karakterisasi silika dari limbah sekam padi melalui metode sol-gel dengan variasi pereaksi asam sebagai upaya pemanfaatan limbah pertanian berkelanjutan. Sekam padi dibakar pada suhu 800°C untuk menghasilkan abu silika, lalu diekstraksi menggunakan larutan NaOH 1N membentuk natrium silikat. Presipitasi dilakukan menggunakan asam asetat, asam klorida, dan asam sitrat hingga pH netral. Karakterisasi meliputi analisis FTIR, kadar air, dan kelembaban. Hasil menunjukkan variasi pereaksi asam memengaruhi sifat silika yang diperoleh. Silika dengan asam sitrat memiliki kadar air terendah (0,0842%) dan kelembaban paling stabil (62%), menandakan struktur lebih kompak dan murni. Sebaliknya, silika dengan HCl menunjukkan kadar air tertinggi (0,3212%) serta kelembaban hingga 95%, mengindikasikan sifat berpori dan higroskopis. Data FTIR memperkuat temuan ini melalui varias i intensitas pita –OH dan Si–O–Si. Dengan demikian, asam sitrat dinilai paling efektif menghasilkan silika berkualitas tinggi sekaligus ramah lingkungan.

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INTRODUCTION

Silica, also known as silicon dioxide (SiO_2), can be obtained from various sources, including plant-based silica, minerals, and synthetic crystals. Silica is available in various forms, including amorphous and crystalline. This material is widely used as a drying agent, adsorbent, filtration media, and catalyst component (Rosmiyani et al., 2023). In addition, 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 et al., 2004). This material is typically obtained from primary sources, such as quartz sand, via a synthetic process that requires high energy and specific chemicals. However, overexploitation of this natural resource can deplete quartz sand reserves and have significant environmental impacts (Yudhistira & Hidayarto., 2011). Therefore, alternative, more environmentally friendly and sustainable raw materials are needed to produce silica gel more efficiently. Silica gel has a large pore size distribution, diverse particle sizes, and a high surface area, which enhances its effectiveness in adsorption applications (Kristianingrum et al., 2020). The high silica content in rice husk waste makes it a potential raw material for silica production (Prameswara et al., 2023).

One potential alternative raw material is rice husk waste. Rice husk is a byproduct of rice processing and is abundant, particularly in agricultural countries such as Indonesia. It is estimated that every ton of rice produced will produce around 20% rice husk, containing 87-97% silica after complete combustion. Most of this remains underutilized and is often discarded or burned, creating environmental problems (Meliyana & Handayani, 2019). The abundance of rice husk waste and its slow natural disposal process impact the environment and human health. However, the current utilization of rice husk waste is still minimal (Pujotomo, 2017). Processing this waste not only reduces environmental pollution but also provides added value to the agricultural and industrial sectors.

Research conducted by Utari et al. (2020) synthesized modified silica using diphenylcarbazone. Furthermore, the synthesis of silica from rice husks typically involves silica extraction via the sol-gel method. This method exploits the high solubility of amorphous silica in alkaline solutions such as KOH, Na_2CO_3, or NaOH, and the precipitation of dissolved silica using acids such as hydrochloric acid, citric acid, acetic acid, and oxalic acid (Meliyana & Handayani, 2019). Therefore, the final characteristics of the resulting silica are highly dependent on the extraction method and the type of reagents used in the process. Therefore, the modifying reagents in this synthesis process are key factors in enhancing extraction efficiency and the quality of the resulting silica, including porosity, surface area, and purity.

Several previous studies have examined the use of rice husks as a silica source. Thahir et al. (2021) reported that extraction using 10% NaOH produced silica with a purity of up to 90.92%. Meanwhile, KOH, as an alternative solvent, demonstrated strong potential, with a yield of 82%. Furthermore, research by Sholikha et al. (2010) showed that the use of hydrochloric acid affected the properties of the silica gel produced. Higher concentrations of hydrochloric acid significantly improved the yield. The optimal silica gel yield obtained using a 5 N hydrochloric acid solution was 8.2610%. Therefore, further research is needed to investigate the effect of reagent modification on the characteristics of silica obtained from rice husks.

Building on this background, this study aims to synthesize and characterize silica from rice husk waste via a reagent-modification approach. This research is expected to provide a more efficient and environmentally friendly method for silica production, and to support the principles of a circular economy and more sustainable waste management.

METHODS

Materials

The materials used in this study included rice husk waste collected from farms in the Lamalaka area, Bantaeng Regency, distilled water, sodium hydroxide (NaOH), acetic acid (CH_3COOH), hydrochloric acid (HCl), citric acid ($C_6H_8O_7$), and filter paper.

Tools

The tools used in this study included an analytical balance, blender, hotplate, tripod and gauze, sample tray, beaker, plastic beaker, volumetric flask, dropper pipette, graduated pipette, graduated cylinder, glass funnel, stirrer, mortar and pestle, crucible, oven, furnace, magnetic stirrer, ziplock bags, hygrometer, pH meter, and supporting characterization equipment, namely FTIR (Fourier Transform Infrared).

Procedure

Preparation of Sodium Silicate Solution

The sodium silicate preparation process is based on the research of Arief et al. (2025), but uses different stirring times and a furnace hold at 800°C for 1 hour, which converts organic material into silica-rich ash. The cooled ash is then ground to increase the surface area for contact. The refined ash is then extracted using 1 N NaOH at 80°C for 90 minutes. The solution was then filtered and washed with hot water, and the initial pH was measured.

Rice Husk Silica Synthesis

Weigh 10g of the washed solution and add each modified reagent or acid solution separately, referring to the research of Riza et al. (2022), namely acetic acid (CH_3COOH), hydrochloric acid (HCl), and citric acid ($C_6H_8O_7$), 1 N until the solution pH is 7 (neutral), because under neutral conditions, silica gel will produce the largest yield and surface area (Yusrin et al., 2014). Subsequently, following the study by Meliyana & Handayani. (2019), the solution was left for 18 hours and then dried at 80 °C until constant weight.

Silica Characterization

FTIR (Fourier Transform Infrared)

The resulting silica was analyzed by Fourier transform infrared (FTIR) spectroscopy to identify the functional groups present (Putri et al., 2022). This allowed for the successful synthesis of silica from rice husks.

Humidity Test

The resulting silica was then tested for moisture content by placing it in a plastic zip-lock bag and measuring the moisture content with a calibrated hygrometer. The moisture content was then determined for 1 hour.

Moisture Content Determination

In the study by Fathurrahman et al. (2020), the method involved heating in an oven at 110°C. However, the heating time and amount of silica used differed: 1 g was heated for 2 hours, then cooled in a desiccator for 10 minutes, and then weighed until constant weight.

RESULT AND DISCUSSION

Rice Husk Silica Synthesis

Chemically, the main reaction in the synthesis of silica from sodium silicate involves neutralization by acid as follows:

$$Na_2SiO_3\ (aq) + 2H^+ \rightarrow H_2SiO_3\ (s) + 2Na^+$$

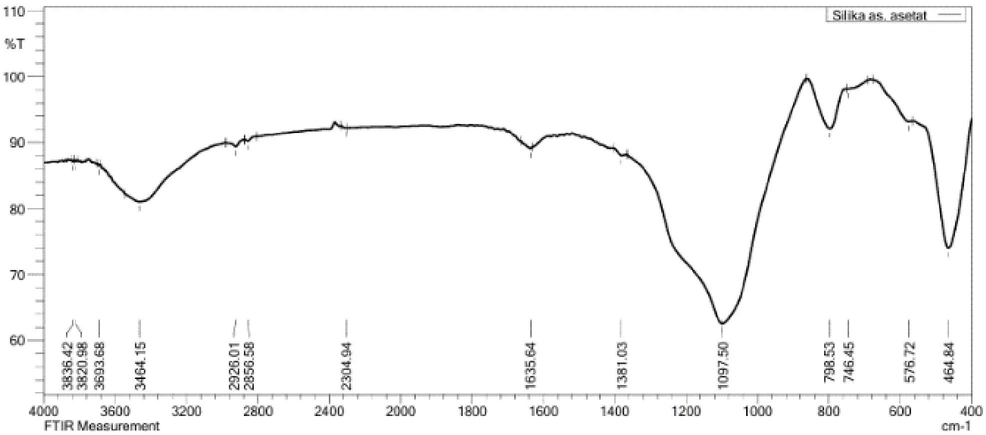
H_2SiO_3 (silicic acid) will undergo polymerization through the condensation of silanol groups (Si–OH) into a siloxane network (Si–O–Si), which then forms a silica gel. The type of acid used affects both the precipitation rate and the final gel structure. Strong acids such as HCl tend to form gels more quickly and with larger pores, while weak acids such as citric acid form gels more slowly, resulting in a denser and more uniform structure.

The silica synthesis process is carried out via the sol-gel method using rice husk ash calcined at 800°C. Silica extraction using 1N NaOH solution produces sodium silicate, which is then precipitated with acidic reagents (acetic acid, HCl, and citric acid) to form silica gel. After aging and heating, dry silica is obtained for analysis.

Silica Characterization

The FTIR characterization aims to identify the main functional groups in the synthesized silica to determine the success of the synthesis from rice husk silica. The characterization results for the three reagents used are as follows:

Acetic acid silica



(a) (b)

Figure 1. FTIR of Acetic Acid Silica

Absorption band (a) 1097.50 cm⁻¹: Asymmetric stretching vibration of the Si–O–Si group, indicating the primary structure of silica. (b) 798.53 cm⁻¹ and 746.45 cm⁻¹: Bending and symmetric vibration of Si–O–Si. (c) 464.84 cm⁻¹: Bending vibration of Si–O. (d) 3464.15 cm⁻¹ and 1635.64 cm⁻¹: Absorption of the –OH group (silanol and bound water). (e) 2926.01 cm⁻¹ and 2856.58 cm⁻¹: The appearance of small C–H absorptions, likely organic residues from acetic acid.

Citric acid silica.

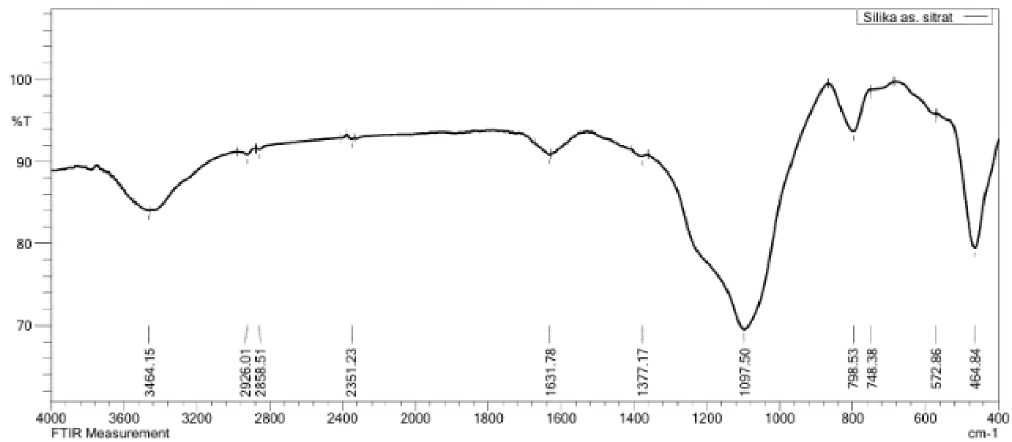


Figure 2. FTIR of Citric Acid Silica

Absorption band (a) 1097.50 cm⁻¹: Main absorption of Si–O–Si, good intensity indicates a well-organized structure. (b) 798.53 cm⁻¹ and 748.38 cm⁻¹: Bending of Si–O–Si. (c) 3464.15 cm⁻¹ and 1631.78 cm⁻¹: –OH groups, but the intensity is lower than that of acetic acid, indicating less bound water. (d) The spectrum shows sharp and narrow peaks, indicating the purity and good structure of the silica.

Silica Hydrochloric Acid

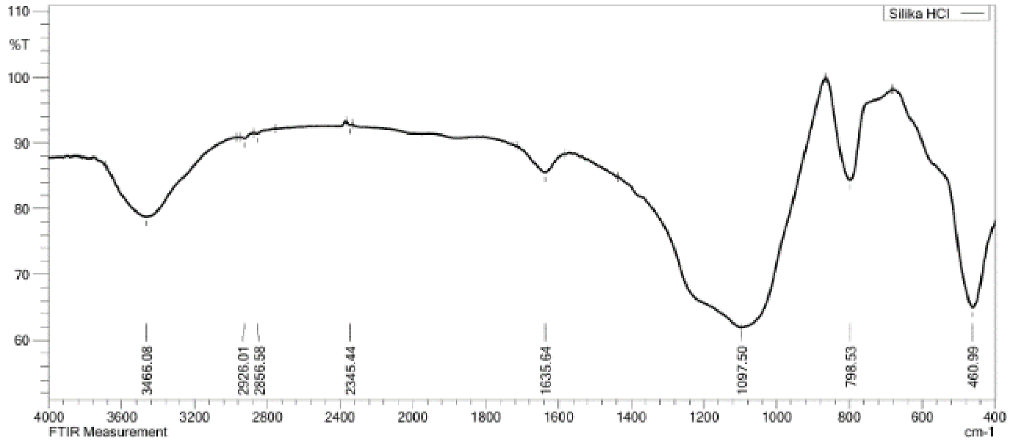


Figure 3. FTIR of Silica Acid Chloride

Absorption band (a) 1097.50 cm^{-1}: Strong absorption of Si–O–Si, with the largest area of the three samples, indicating the formation of an intense siloxane network structure. (b) 798.53 cm^{-1}: Bending of Si–O. (c) 3466.08 cm^{-1} and 1635.64 cm^{-1}: The –OH group is larger than the other two, consistent with high water content and high hygroscopicity. (d) Several broad peaks in the –OH region indicate the presence of large amounts of bound water.

FTIR spectral analysis indicates the presence of the main functional groups that make up the silica structure. The absorption band at approximately 1097 cm^{-1} in all three samples is characteristic of the asymmetric Si–O–Si stretching, indicating the formation of a silica base network. This is a key marker of the three-dimensional amorphous silica structure commonly found in biomass-combusted silica.

Another consistent absorption band at 798–746 cm^{-1} represents the symmetric and bending stretching of Si–O–Si, confirming the presence of a silica framework structure. Furthermore, the band at approximately 460 cm^{-1} indicates the bending vibrations of the Si–O groups, also commonly found in non-crystalline silica structures.

The broad absorption band at approximately 3464–3466 cm^{-1} arises from the stretching vibrations of the hydroxyl (–OH) groups, both from silanol (Si–OH) groups and physically bound water. The presence of a band around 1630–1640 cm^{-1} represents the bending vibration of water, indicating that moisture is still trapped in the silica pores.

The silica samples from HCl had the highest intensity in the –OH band, which is consistent with the high humidity data (up to 95%). In contrast, the silica from citric acid had a lower –OH intensity, indicating less bound water and a more thermally and chemically stable structure.

Small peaks around 2856 and 2926 cm^{-1} in some samples suggest the possible presence of C–H bonds from residual organic compounds, such as acetic acid or citric acid. However, the low intensity indicates that these organic impurities are insignificant and do not compromise the overall purity of the silica.

Thus, the FTIR results indicate that all silica samples formed the typical Si–O–Si structure, and the type of reagent used influences the content of –OH groups and the likelihood of organic residues.

Chemical Interpretation

The FTIR characterization results confirm the previous results regarding water content and moisture content: (a) Silica from citric acid has a low –OH absorption and a dominant Si–O–Si structure, indicating optimal condensation, corresponding to the lowest water and humidity content. (b) Silica from HCl exhibits strong –OH absorption and a large Si–O–Si area, but still aggressively absorbs water due to its open porosity and incomplete gel structure. (c) Silica from acetic acid falls somewhere in between, with a relatively stable structure but retaining light organic groups and a moderate water content.

Silica Moisture Test Results

The moisture test was conducted to determine the silica's ability to absorb water vapor from the air over a specified period. The results are shown in the Table 1 as follows,

Table 1. Silica moisture content

Acid Types	Humidity (%) and Time
Acetic Acid	72% after 2.5 hours
Citric Acid	Stable at 62% after 2.5 hours
Cloric Acid	65% (5 minutes) → 75% (5 minutes) → 84% → 95% (2 hours)

From the results in Table 1, silica from HCl exhibits a sharp increase in moisture content over time, indicating a highly hygroscopic structure and high porosity. These properties are suitable for use as a desiccant, but are less stable for long-term storage. Conversely, silica from citric acid exhibits the most stable moisture content, indicating a denser microporous structure and greater resistance to water absorption. Silica from acetic acid falls in the middle, with relatively high absorption but relatively stable.

Silica Moisture Content Results

Moisture content determination aims to determine the amount of water still trapped within the dried silica structure. Moisture content test results show significant differences depending on the type of acid reagent used, as follows:.

Table 2. Silica water content

Acid Type	Water content (%)
Acetic acid (CH_3COOH)	0.22182
Cloric Acid (HCl)	0.3212
Citric Acid ($C_6H_8O_7$)	0.0842

Silica produced with citric acid had the lowest water content, indicating a denser structure and silanol (Si–OH) bonds that tend to polymerize completely into siloxane (Si–O–Si). Conversely, HCl produced silica with the highest water content, indicating the presence of free water or silanol groups that have not yet been optimally condensed.

The lowest water content was observed for silica synthesized with citric acid, indicating that the resulting silica has a more stable porous structure and stronger surface bonds to moisture. Conversely, the use of HCl produced silica with the highest water content, indicating either weaker water bonds or larger pores that trap water more readily.

CONCLUSION

Based on research on the synthesis of silica from rice husk waste using variations in acid reagents, it can be concluded that reagent modification significantly affects the characteristics of the resulting silica, particularly water content and moisture stability. Silica synthesized using citric acid exhibits the lowest water content (0.0842%) and the highest moisture stability (62%), making it the most suitable for applications requiring moisture resistance. The use of hydrochloric acid (HCl) yields silica with the highest moisture content (up to 95%), indicating a more open pore structure and a higher water absorption capacity. Silica from acetic acid has properties between the two, with a water content of 0.22182% and a moisture content of 72%.

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