EFFECT OF ULTRASONIC TREATMENT AND CALCINATION TEMPERATURE OF RICE HUSK ASH ON THE CHARACTERISTICS OF SILICA-CHITOSAN COMPOSITES

Ani Purwanti, Fajar Yulianto Prabowo

Faculty of Engineering, Universitas AKPRIND Indonesia
28 Kalisahak St., Gondokusuman, Yogyakarta, 55222
Indonesia, ani4wanti@akprind.ac.id (A.P.); fajaryulianto@akprind.ac.id (F.Y.P.).

Received 21 January 2026
Accepted 07 May 2026
DOI: 10.59957/jctm.v61.i5.2026.2

ABSTRACT

This study investigates the effect of calcination temperature and ultrasonic treatment during the extraction of silica from rice husk ash and its implications for the characteristics of silica-chitosan composites. Rice husk ash was calcined at temperatures of 500, 600, and 700°C, followed by extraction using 2M NaOH solution with and without ultrasonic treatment. The extracted silica was subsequently combined with chitosan to form silica-chitosan composites. Characterization was carried out using X-ray fluorescence (XRF) to determine SiO_2 purity, Fourier-transform infrared spectroscopy (FT-IR) for functional group identification, and X-ray diffraction (XRD) for structural analysis. The results showed that increasing the calcination temperature from 500°C to 700°C generally enhanced the silica yield (79.6 - 90.8 %) and purity (92.3 - 97.4 %). Ultrasonic treatment further improved the yield by approximately 2 - 5 % and increased the purity by about 0.5 - 2 % at each calcination temperature. FT-IR and XRD analyses indicated that the obtained silica was predominantly in the amorphous phase with strong Si-O-Si bonds, while the silica-chitosan composites exhibited intermolecular interactions involving -OH, $-NH_2$, and Si-OH functional groups. The combination of calcination temperatures of 600 - 700°C and ultrasonic treatment produced high-quality silica that has strong potential to enhance the performance of silica-chitosan composites for dye adsorption and heavy metal ion removal applications.

Keywords: *rice husk ash, silica, chitosan, ultrasonic treatment, calcination temperature.*

INTRODUCTION

Rice husk is an abundant agricultural by-product and represents a low cost and sustainable source of silica. However, its utilization is still largely limited to low value applications, leading to environmental concerns when improperly managed [1]. Due to its high silica content, rice husk ash has attracted considerable attention as an alternative raw material for silica production in various technological applications [2 - 6].

The characteristics of silica derived from rice husk ash are strongly influenced by thermal treatment, particularly calcination temperature, which governs the removal of organic components and affects the purity and reactivity of the final product [7]. Silica obtained from such processes has been widely applied in areas such as construction, agriculture, and adsorption systems due to its chemical stability and surface properties [8 - 10]. Compared to other agricultural residues, rice husk ash remains one of the most favorable sources because of its relatively high silica content and wide availability [11 - 13].

Silica extraction is commonly carried out through alkaline dissolution followed by acid precipitation via the sol-gel process. The use of alkaline solutions such as NaOH facilitates the dissolution of silica, while subsequent acidification leads to the formation of SiO_2 with properties dependent on the processing conditions and reagents used [5, 6, 14, 15]. In addition to chemical treatment, process intensification techniques

such as ultrasonic-assisted extraction have been increasingly applied to improve extraction efficiency. Ultrasonic cavitation enhances mass transfer and particle dispersion, resulting in improved silica recovery and material properties [16].

Chitosan is a biodegradable polymer widely used in adsorption applications due to the presence of amine functional groups capable of binding various pollutants [17 - 19]. To enhance its performance, silica is often incorporated into the chitosan matrix, forming silica-chitosan composites with improved stability and adsorption capacity. The performance of these composites is closely related to the properties of silica, which are affected by calcination temperature and extraction conditions. Calcination temperature determines whether silica remains in an amorphous or crystalline form, while ultrasonic-assisted extraction improves dissolution and dispersion, thereby influencing the final composite characteristics [20, 21].

Silica-chitosan composites have been extensively studied for dye and heavy metal adsorption, demonstrating improved adsorption performance due to the synergistic interaction between silica and chitosan functional groups [22]. However, studies that systematically investigate the combined influence of calcination temperature and ultrasonic-assisted extraction on the characteristics of silica and their subsequent effect on silica-chitosan composites are still limited.

Therefore, this study aims to evaluate the effect of calcination temperature and ultrasonic-assisted extraction on the yield and purity of silica derived from rice husk ash, as well as to investigate their impact on the structural characteristics and interactions within silica-chitosan composites. In particular, this work provides a more integrated understanding of how thermal and ultrasonic processes simultaneously influence silica quality and composite performance, which has not been clearly addressed in previous studies. The combined evaluation of these two key parameters offers a more systematic approach for optimizing silica-based composite materials derived from agricultural waste.

EXPERIMENTAL

Materials and instruments

The materials used in this study included rice husk, chitosan (degree of deacetylation, DD > 75 %), 2M NaOH solution, 1M HCl solution, 1 % (volume per volume) acetic acid solution, and deionized water. The main instruments employed were a muffle furnace, ultrasonic bath, drying oven, magnetic stirrer with hot plate, and analytical instruments for material characterization, including Fourier-transform infrared spectroscopy (FT-IR), X-ray fluorescence (XRF), and X-ray diffraction (XRD).

Experimental procedure

Silica synthesis

The rice husk was first washed thoroughly with water to remove adhering impurities and then dried under direct sunlight for approximately 24 h to reduce its moisture content. The dried rice husk was subsequently calcined in a furnace at three different temperatures, namely 500, 600, and 700°C, for 2 h. The selection of the temperature range of 500 - 700°C was based on reported findings indicating that temperatures above 500°C are required to oxidize organic components and residual carbon, while temperatures up to 700°C are still able to preserve silica in an amorphous phase, which exhibits higher reactivity for gel formation and adsorption applications [20].

The calcined rice husk ash was then extracted using 2 M NaOH solution with a solid-to-liquid ratio of 1:10 (weight per volume). The extraction process was carried out using two different methods: (1) conventional stirring at 80°C for 1 h using a hot-plate magnetic stirrer, and (2) ultrasonic-assisted extraction for 30 min at a frequency of 20 kHz. After extraction, the resulting filtrate was separated from the solid residue by filtration and subsequently acidified using 2M HCl until a neutral pH (pH = 7) was reached to precipitate silica in the form of a gel. Neutral pH precipitation was selected to ensure a balance between silicate solubility and silica gel stability; at excessively low pH, silica tends to dissolve, while at high pH, silicate species remain dissolved [23].

The obtained silica gel was washed repeatedly with deionized water until it was free from chloride ions, as confirmed by the absence of a white AgCl precipitate upon the addition of silver nitrate solution. The wet silica was then dried in an oven at 80°C for 12 h to achieve minimal moisture content, following procedures commonly reported for silica synthesis from rice husk ash [7, 15].

Chitin and chitosan synthesis

Shrimp shells were washed, dried, ground, and sieved to obtain particles with a size of 100 mesh. A total of 100 g of the prepared material was placed in a beaker, followed by the addition of 900 mL of 3.5 % (weight per volume) NaOH solution. The mixture was heated at 65°C for 2 h under continuous stirring for the deproteinization process. Afterward, the suspension was neutralized with deionized water. The resulting solid residue was then treated with 1M HCl for demineralization and subsequently washed with deionized water until neutral pH was achieved. The obtained chitin was dried at 65°C. Subsequently, 500 mL of 50 % (weight per volume) NaOH solution was added to the dried chitin powder, and the mixture was heated at 100°C for 30 min to carry out the deacetylation process. These deacetylation conditions were selected to obtain chitosan with a degree of deacetylation (DD) higher than 75 %, which is required to ensure a sufficiently high number of free -NH_2 groups for adsorption applications and for effective interaction with silica [24]. The resulting mixture was filtered, and the solid product was neutralized with deionized water and dried at 65°C to obtain chitosan.

Synthesis of silica - chitosan composite

A chitosan solution was prepared by dissolving 2 g of chitosan powder (100 mesh) in 100 mL of 2 % (volume per volume) acetic acid solution at room temperature under continuous stirring for 24 h. The homogeneous solution was then cast onto a glass mold and evenly spread using a glass rod. The formed chitosan membrane was left in open air for 12 h to allow solvent evaporation. After drying, the membrane was immersed in 5 % (weight per volume) NaOH solution for 5 min to neutralize residual acetic acid, followed by thorough washing with deionized water to remove excess NaOH. The chitosan membrane was then stored in a desiccator until further use.

For the preparation of the chitosan-silica composite, silica particles were incorporated into the chitosan solution to enhance the affinity and functional performance of the composite. Specifically, 2 g of chitosan (100 mesh) was dissolved in 100 mL of 2 % (volume per volume) acetic acid, after which 24 g of silica was added to the solution, corresponding to a silica to chitosan mass ratio of 12 g g^{-1}. The mixture was stirred for 24 h to obtain a homogeneous dispersion. The resulting suspension was then cast onto a glass mold and allowed to dry in open air for 12 hours to form a composite membrane.

The dried composite membrane was subsequently immersed in 5 % (weight per volume) NaOH solution at 80°C for 5 min. To promote partial silica redistribution and to induce the formation of a macroporous silica-chitosan structure, the membrane was maintained at 80°C for 2 h. Afterward, the composite membrane was thoroughly washed with deionized water to remove residual chemicals and finally stored in a desiccator.

The high silica to chitosan mass ratio (12 g g^{-1}) was selected to produce a composite dominated by the inorganic phase, thereby enhancing mechanical strength and thermal stability, while chitosan remained as the polymeric matrix and the primary source of active amine functional groups. A similar approach has been reported for silica-chitosan composites used in dye adsorption, where increasing the silica fraction improves pore structure stability and resistance to deformation [22].

Characterization and testing

The synthesized silica and silica-chitosan composites were characterized using X-ray fluorescence (XRF), Fourier-transform infrared spectroscopy (FT-IR), and X-ray diffraction (XRD). XRF analysis was employed to determine the major oxide composition (SiO_2, Al_2O_3, Fe_2O_3, K_2O, MgO) before and after the extraction process, allowing the calculation of SiO_2 purity enhancement and the reduction of impurity levels.

FT-IR spectroscopy was used to identify the characteristic functional groups of silica (SiOSi, SiOH) and to analyze their interactions with chitosan functional groups (OH, NH_2, and amide groups). XRD analysis was conducted to distinguish between amorphous and crystalline phases of silica based on diffraction patterns within the chitosan matrix [25 - 27].

Experimental design

The experimental design was established to evaluate the effects of calcination temperature and ultrasonic treatment on the characteristics of silica and silica-chitosan composites. Three different calcination temperatures (500, 600, and 700°C) were applied, each with and without ultrasonic assisted extraction. The sample codes and experimental conditions are summarized in Table 1.

RESULTS AND DISCUSSION

Effect of calcination temperature and ultrasonic treatment on silica yield and purity

The results of silica extraction from rice husk ash presented in Table 1 indicate that both calcination temperature and ultrasonic treatment exert a significant influence on the yield and purity of the obtained silica. An increase in calcination temperature leads to a reduction in residual carbon content, resulting in the formation of silica with higher purity. Meanwhile, ultrasonic treatment accelerates the dissolution of silica in the NaOH solution and enhances particle dispersion, thereby increasing the effective contact area between the solid and liquid phases.

As a result, both the extraction efficiency and the quality of silica are improved. The combined effects of calcination temperature and ultrasonic-assisted extraction on silica yield and purity are summarized in Table 2.

The data presented in Table 2 show that increasing the calcination temperature from 500 to 600 and 700°C generally enhances the silica yield under both non-ultrasonic and ultrasonic-assisted conditions. Specifically, raising the calcination temperature from 500 to 700°C increased the silica yield from 79.6 % (S01) to 86.1 % (S05) for the process without ultrasonic treatment, and from 83.8 % (S02) to 90.8 % (S06) for the ultrasonic-assisted process. This improvement in yield is associated with the more complete decomposition of organic and volatile compounds in rice husk at higher temperatures, which increases the relative fraction of the inorganic phase, particularly SiO_2, and facilitates its extraction by the NaOH solution.

For the experiments conducted without ultrasonic treatment, an increase in calcination temperature of 100°C resulted in an increase in silica yield in the range of 1.70 - 4.80 %. In contrast, the application of ultrasonic treatment led to a higher average increase in yield of approximately 4.47 % compared with the conventional process. This observation is consistent with previous studies reporting that the enhancement of silica dissolution under ultrasonic conditions is attributed to the cavitation effect, which improves the penetration of the NaOH solution into the rice husk ash matrix and thus promotes more efficient silica dissolution [16]. Furthermore, higher calcination temperatures contribute to a more complete decomposition of organic components, producing purer silica that is more readily extracted. Elevated temperatures also improve the pore structure of rice husk ash, thereby increasing the contact between NaOH and silica during the extraction process.

A similar trend was observed for silica purity. Under non-ultrasonic conditions, the purity increased from 92.3 % at 500°C to 96.9 % at 700°C, whereas under ultrasonic-assisted conditions, the purity increased from 94.2 % to 97.4 % over the same temperature range. This increase in purity indicates that, in addition to carbon, impurity metal oxides such as Al_2O_3, Fe_2O_3, and K_2O are more effectively decomposed or dissolved at higher calcination temperatures, leading to an increase in the

Table 1. Experimental design and sample coding.

Sample code	Calcination temperature, °C	Ultrasonic treatment
S1	500	No
S2	500	Yes
S3	600	No
S4	600	Yes
S5	700	No
S6	700	Yes

Table 2. Silica yield and SiO_2 purity obtained from the extraction process.

Sample code	Calcination temperature, °C	Ultrasonic treatment	Silica yield, %	SiO_2 purity, %
S01	500	No	79.6	92.3
S02	500	Yes	83.8	94.2
S03	600	No	84.4	94.7
S04	600	Yes	88.9	96.5
S05	700	No	86.1	96.9
S06	700	Yes	90.8	97.4

relative SiO_2 content. This trend is consistent with several reports indicating that controlled calcination in the range of 600 - 700°C produces rice husk ash with high SiO_2 content and relatively low impurity levels [28].

A direct comparison between samples with and without ultrasonic treatment at the same calcination temperature further indicates that ultrasonic-assisted extraction provides an additional increase in silica yield of approximately 4.2 % at 500°C, 4.5 % at 600°C, and 4.7 % at 700°C. This enhancement can be attributed to the acoustic cavitation effect generated by ultrasonic waves in the NaOH solution. The repeated formation and collapse of microbubbles induce micro-turbulence and local increases in pressure and temperature, which accelerate the diffusion of OH^- ions into the ash matrix and promote the breakdown of particle agglomerates. As a result, silica dissolution becomes more efficient, and silica phases that were previously trapped within pores or bound to inorganic impurities are more readily extracted [16].

Analysis of SiO_2 purity enhancement based on XRF

A detailed evaluation of the X-ray fluorescence (XRF) results was conducted to compare the SiO_2 purity of rice husk ash before and after the extraction process. The variations in oxide composition and the corresponding enhancement in SiO_2 purity are presented in Table 3 and Table 4. XRF analysis enables quantitative identification of the major oxide components, including SiO_2, Al_2O_3, Fe_2O_3, K_2O, MgO, and other minor oxides. By comparing the oxide composition at different calcination temperatures and extraction conditions, the extent of impurity removal and the effectiveness of ultrasonic-assisted extraction in improving silica purity can be systematically assessed.

The compositional changes reflected in Table 3 and Table 4 provide direct evidence of the progressive increase in SiO_2 content and the concurrent decrease in impurity oxides after alkaline extraction and acid precipitation, particularly at higher calcination temperatures and under ultrasonic-assisted conditions. The SiO_2 content in rice husk ash prior to extraction was in the range of 78.5 - 83.0 %, depending on the calcination temperature. After the extraction process using 2 M NaOH with and without ultrasonic treatment, the SiO_2 purity increased significantly with increasing calcination temperature, reaching values of 92.3 - 97.4 %. This enhancement in purity is attributed to the dissolution of impurity oxides such as Al_2O_3, Fe_2O_3, K_2O, and MgO into the liquid phase during alkaline extraction. In addition, more complete combustion of carbonaceous compounds and residual impurities at higher calcination temperatures further increases the relative SiO_2 content in the ash.

The percentage increase in SiO_2 purity was calculated using Eq. (1), which relates the difference between the SiO_2 content after and before extraction to the initial SiO_2 content.

Table 3. XRF composition of rice husk ash before extraction.

Calcination temperature, °C	SiO_2, wt. %	Al_2O_3, wt. %	Fe_2O_3, wt. %	K_2O, wt. %	CaO, wt. %
500	92.3	1.80	0.95	2.10	1.20
600	94.7	1.50	0.85	1.85	0.90
700	96.9	1.20	0.60	1.25	0.60

Table 4. XRF composition of silica after extraction and precipitation.

Calcination temperature, °C	Ultrasonic treatment	SiO_2, wt. %	Al_2O_3, wt. %	Fe_2O_3, wt. %	K_2O, wt. %
500	No	92.3	1.80	0.95	2.10
500	Yes	94.2	1.50	0.85	1.85
600	No	94.7	1.50	0.85	1.85
600	Yes	96.5	1.25	0.65	1.45
700	No	96.9	1.20	0.60	1.25
700	Yes	97.4	1.00	0.50	1.05

$$\%\ \text{increase in } SiO_2 = \frac{SiO_{2,\ after} - SiO_{2,\ before}}{SiO_{2,\ before}} x100 \quad (1)$$

The calculated results of the percentage increase in SiO_2 purity based on Eq. (1) are presented in Table 5.

The data presented in Table 4 clearly indicate that SiO_2 purity increases with increasing calcination temperature. At 500°C, the SiO_2 purity ranges from 92.3 to 94.2 %, while at 600°C it increases to 94.7 - 96.5 %, and at 700°C it reaches the highest values of 96.9 - 97.4 %. This trend can be attributed to the more effective combustion of organic components and the decomposition of mineral impurities at elevated temperatures, resulting in a more dominant silica fraction. In addition, higher calcination temperatures reduce the residual carbon content, which contributes to improved accuracy in SiO_2 purity determination by XRF analysis.

Under non-ultrasonic conditions, the SiO_2 purity increased from 92.3 % to 96.9 % as the calcination temperature was raised from 500°C to 700°C. Under the same temperature range, ultrasonic-assisted extraction produced higher purity values of 94.2 - 97.4 %. In absolute terms, the highest SiO_2 purity was obtained for sample S06 (700°C with ultrasonic treatment), reaching 97.4 %. However, when the relative percentage increase in purity with respect to the initial ash is considered, the greatest improvement was observed at the intermediate temperature of 600°C. This suggests that a balance between sufficiently high calcination temperature and the predominance of the amorphous silica phase provides optimal conditions for impurity removal by NaOH [28].

The influence of ultrasonic treatment on SiO_2 purity can be clearly observed from the differences between paired samples S01 - S02, S03 - S04, and S05 - S06. At each calcination temperature, ultrasonic treatment consistently resulted in higher SiO_2 purity compared with the corresponding conventional process. At 500°C, ultrasonic treatment increased the purity from 92.3 % to 94.2 % (an increase of 1.9 %); at 600°C, the purity increased by 1.8 %, from 94.7 % (without ultrasonics) to 96.5 % (with ultrasonics); and at 700°C, the purity increased from 96.9 % to 97.4 % (an increase of 0.5 %). Although the absolute increases in purity appear moderate, the consistent enhancement across all temperatures indicates that ultrasonic treatment promotes more efficient dissolution of impurities, particularly metal oxides and residual carbon.

This conclusion is further supported by the significant decrease in the contents of Al_2O_3, Fe_2O_3, and K_2O after ultrasonic-assisted extraction, demonstrating that this method is effective for the removal of inorganic impurities. Ultrasonic treatment accelerates impurity release by breaking down particle agglomerates and increasing the effective contact surface area between the alkaline solution (NaOH) and the ash particles, thereby facilitating impurity dissolution.

Ultrasonic treatment consistently provided higher relative increases in SiO_2 purity compared with the non-ultrasonic process. The highest relative enhancement was observed for sample S02 (500°C with ultrasonic treatment), reaching 20.0 %. Calcination at 600°C combined with ultrasonic treatment was also highly effective, yielding a 20.37 % increase in purity. Overall, ultrasonic treatment contributed to an additional 2 - 3 % increase in SiO_2 purity compared with the conventional process. The combined application of high calcination temperature (700°C) and ultrasonic treatment produced

Table 5. Percentage increase in SiO_2 purity before and after extraction.

Sample code	Temperature, °C	Ultrasonic treatment	SiO_2 before, %	SiO_2 after, %	Increase, %
S01	500	No	78.5	92.3	17.56
S02	500	Yes	78.5	94.2	20.00
S03	600	No	80.2	94.7	18.06
S04	600	Yes	80.2	96.5	20.37
S05	700	No	83.0	96.9	16.75
S06	700	Yes	83.0	97.4	17.35

a silica yield of 90.8 % with a purity of 97.4 %, representing the highest values obtained in this study. Compared with the initial condition (500°C without ultrasonic treatment), this corresponds to an increase in yield of 11.2% and an increase in purity of 5.1 %.

These results demonstrate that the synergistic optimization of calcination temperature and ultrasonic-assisted extraction significantly enhances both the quality and quantity of silica recovered from rice husk ash. The extraction process successfully increased the SiO_2 purity from an initial range of 78 - 83 % to 92 - 97 %. The optimal calcination temperature range was found to be 600 - 700°C, as this range is sufficient to remove most carbonaceous components without inducing sintering that could hinder impurity release and silica dissolution.

Implication of calcination temperature on silica structure (amorphous vs. crystalline)

The XRD results reveal that the silica obtained at calcination temperatures in the range of 500°C - 700°C is predominantly characterized by a broad diffraction peak centered at approximately $2\theta \approx 22°$, which is a typical fingerprint of amorphous silica. This observation is in good agreement with previously reported studies indicating that, within this temperature range, the crystallinity of SiO_2 derived from rice husk ash remains very low, while significant transformation into more ordered crystalline phases (such as quartz and cristobalite) generally occurs only at temperatures above ~700°C - 800°C.

Amorphous silica is highly desirable for composite and adsorbent applications due to its higher specific surface area, abundance of reactive silanol (Si-OH) groups, and greater ease of chemical modification. Therefore, the selection of a maximum calcination temperature of 700°C in this study can be considered an appropriate compromise between achieving higher silica purity through effective removal of organic impurities and residual carbon and preserving the amorphous nature of silica that is essential for optimal interfacial interaction and performance of the silica-chitosan composite.

FT-IR analysis of silica and silica-chitosan composite

FT-IR analysis was carried out to identify the functional groups present in the synthesized silica and silica-chitosan composite. The FT-IR spectra exhibit strong absorption bands at approximately 1080 cm^{-1} and 800 cm^{-1}, which are respectively attributed to the asymmetric and symmetric stretching vibrations of the Si-O-Si bonding network. The absorption band observed at around 460 cm^{-1} corresponds to the bending vibration of the Si-O bond, confirming the typical structural features of silica.

In the silica-chitosan composite, an additional broad absorption band appears at approximately 3440 cm^{-1}, which is assigned to the overlapping stretching vibrations of O-H and N-H groups. Furthermore, a characteristic absorption band at around 1650 cm^{-1} is observed, corresponding to the C=O stretching vibration of the amide group in chitosan. The interaction between silanol (Si-OH) groups on the silica surface and the amine groups of chitosan results in slight shifts in several FT-IR absorption bands, indicating the formation of hydrogen bonding within the composite structure. Fig. 1 presents the simulated FT-IR spectra of pure silica and the silica-chitosan composite, illustrating the successful incorporation of chitosan into the silica matrix through intermolecular interactions.

The results demonstrate that increasing the calcination temperature from 500 to 700°C produces silica with higher purity as a consequence of the more complete decomposition of organic matter in rice husk. Ultrasonic treatment was proven to enhance extraction efficiency by breaking down silica particle agglomerates and accelerating the diffusion of OH^- ions into the rice husk ash matrix. FT-IR analysis confirms the presence of characteristic silica functional groups in all samples and verifies the successful formation of the composite through the identification of amide functional groups originating from chitosan.

The combination of silica and chitosan in the composite provides strong potential for applications in dye and heavy metal adsorption, as the presence of amine and hydroxyl functional groups enriches the number of active adsorption sites. This finding is consistent with previous studies [15], which reported an enhanced adsorption capacity of silica-chitosan composites compared with pristine chitosan. Furthermore, silica-chitosan nanocomposites have been reported to be highly effective for the adsorption of organic pollutants from water, which can be used to further strengthen the discussion on the application potential of the silica-chitosan composite in environmental remediation [29].

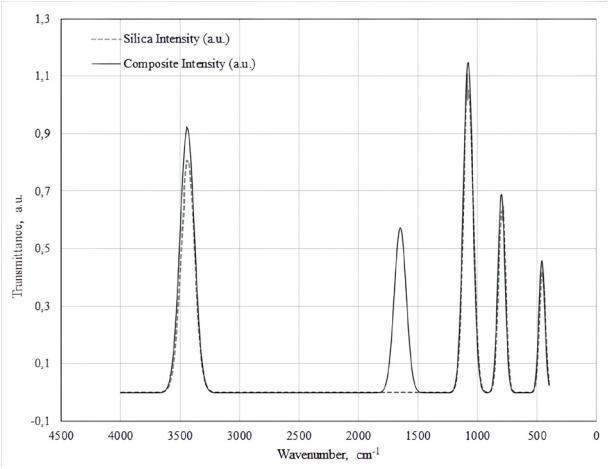

Fig. 1. FT-IR spectra of silica and silica-chitosan composite.

The FT-IR spectrum of silica exhibits strong absorption bands at approximately 1080 and 800 cm^{-1}, which are attributed to the asymmetric and symmetric stretching vibrations of the Si-O-Si network, respectively, along with a band at around 460 cm^{-1} corresponding to the Si-O bending vibration. A broad band near 3400 cm^{-1} and a band at approximately 1630 cm^{-1} are associated with O-H stretching vibrations and adsorbed H_2O molecules, respectively. These spectral features are consistent with those reported for amorphous silica in the literature [25].

In the silica-chitosan composite, a broad absorption band appears at around 3440 cm^{-1}, corresponding to the overlapping stretching vibrations of -OH and -NH groups from chitosan, while an absorption band at approximately 1650 cm^{-1} is assigned to the C=O stretching vibration of the amide group (amide I). In addition, bands observed at around 1550 cm^{-1} (amide II) and 1380 - 1420 cm^{-1} are attributed to -CH and -NH deformation vibrations. Compared with pristine chitosan, the composite spectrum shows slight shifts in the -OH/-NH band positions and changes in the intensity of the Si-O-Si bands, indicating the formation of hydrogen bonding interactions between silanol (Si-OH) groups on the silica surface and the amine/hydroxyl groups of chitosan.

The presence of these interactions is crucial, as they strongly influence the stability of the composite in aqueous media and the number of active sites that participate in the adsorption process. Several studies have reported that the strength of silica-chitosan interactions and the distribution of silica within the chitosan matrix govern pore morphology, adsorption capacity, and the mechanical stability of the composite material [22].

Implications for adsorption application potential

The combination of silica extracted from rice husk ash, which is rich in silanol functional groups, with chitosan containing amine and hydroxyl groups results in a composite material with a high density of active sites. The amine groups of chitosan act as chelating ligands for heavy metal ions, while silica serves as an inorganic support that enhances the specific surface area and structural stability of the composite.

The synthesis conditions that yielded the highest silica recovery and purity (700°C with ultrasonic treatment) are expected to contribute to improved adsorption performance, as the low content of impurities (e.g., alkali and alkaline earth metal ions) is likely to reduce competitive adsorption toward target ions. However, if the calcination temperature becomes excessively high and induces the formation of crystalline silica phases, this advantage may diminish due to a decrease in surface area and the number of available silanol groups. Therefore, based on the present results, the temperature range of 600 - 700°C combined with ultrasonic-assisted extraction can be regarded as an optimal compromise between high purity, predominantly amorphous character, and high silica yield.

Conceptually, the composites synthesized in this study are highly relevant for applications in the adsorption of textile dyes and heavy metal ions, as reported in numerous studies on silica-chitosan and silica-chitosan aerogel systems, which consistently demonstrate higher adsorption capacities than pristine chitosan [22, 30, 31]. Further investigations, including adsorption isotherms, kinetic studies, and regeneration tests, will be required to quantitatively evaluate the adsorption capacity and long-term stability of the adsorbent. Nevertheless, the present characterization results already indicate a promising potential for environmental remediation applications.

CONCLUSIONS

Based on the experimental results and comprehensive analysis, the following conclusions can be drawn:

- Calcination temperature and ultrasonic treatment

exert a significant effect on the yield and purity of silica extracted from rice husk ash. Increasing the calcination temperature from 500 to 700°C generally enhances the silica yield from 79.6 % to 90.8 % and the SiO_2 purity from 92.3 % to 97.4 %.

- Ultrasonic-assisted extraction during the NaOH leaching stage increases the silica yield by approximately 2 - 5 % and the SiO_2 purity by about 0.5 - 2 % at each calcination temperature. This enhancement is mainly attributed to the cavitation effect, which accelerates silica dissolution and promotes more efficient removal of impurities.
- The optimal condition in this study was achieved at a calcination temperature of 700°C combined with ultrasonic treatment. Under these conditions, the highest silica yield of 90.8 % and SiO_2 purity of 97.4 % were obtained, with a predominantly amorphous structure favorable for functional material applications. Compared with the initial condition (500°C without ultrasonic treatment), this corresponds to an increase in yield of 11.2 % points and an increase in purity of 5.1 % points.
- The NaOH-HCl extraction process, particularly when assisted by ultrasonics, successfully increased the SiO_2 purity from an initial range of 78 - 83 % in the raw ash to 92 - 97 % in the extracted silica. This process also led to a significant reduction in impurity oxides such as Al_2O_3, Fe_2O_3, and K_2O.
- FT-IR and XRD analyses confirmed the presence of predominantly amorphous silica with strong Si-O-Si bonding and verified the formation of interactions between silica and chitosan in the composite through -OH, $-NH_2$, and Si-OH functional groups. These results indicate that the synthesized silica-chitosan composite is highly promising for dye and heavy metal adsorption applications.
- Overall, the valorization of rice husk ash as a silica source, combined with chitosan as a biodegradable polymer matrix, provides a low-cost, waste-based composite material with strong potential for environmental and industrial applications.

Acknowledgments

The authors gratefully acknowledge the Directorate of Research and Community Service, Universitas AKPRIND Indonesia.

Authors' contributions

A.P.: Conceptualization, Design of the research, Project management and funding acquisition, Experimental work, Writing and editing the manuscript, were performed by the first author; F.Y.P.: Experimental work, Editing the manuscript, was fulfilled by the second author.

REFERENCES

1. R. Rachmat, Suismono, Model penggilingan padi terpadu untuk meningkatkan nilai tambah, Bul. Teknol. Pascapanen Pertanian, 8, 2, 2012, 99-111.
2. M.T. Rhaman, M.A. Haque, M.A. Rouf, M.A.B. Siddique, M.S. Islam, Preparation and characterization of activated carbon and amorphous silica from rice husk, Bangladesh J. Sci. Ind. Res., 50, 4, 2015, 263-270.
3. S. Ismail, A. Wali, Removal of heavy metals using rice husk ash: A review, Environ. Technol. Rev., 30, 3, 2011, 237-244.
4. J. Chun, J.H. Lee, Recent progress on the development of engineered silica particles derived from rice husk, Sustainability, 12, 24, 2020, 10683.
5. R. Dungani, R. Sari, D. Setyawati, S. Aprilia, E. N. Dewi, Potensi sekam padi sebagai sumber silika, J. Teknol., 7, 1, 2015, 10-17.
6. P.U. Nzereogu, A.D. Omah, F.I. Ezema, E.I. Iwuoha, A.C. Nwanya, Silica extraction from rice husk: Comprehensive review and applications, Hybrid Adv., 4, 6, 2023, 100111.
7. F. Kurniawati, M. Yusuf, Pengaruh suhu kalsinasi terhadap kemurnian silika dari abu sekam padi, J. Tek. Kimia, 24, 1, 2018, 15-22.
8. Y. García-Díaz, R. Torres-Ortega, M. Saba, E. Quiñones-Bolaños, J. Torres-Sanchez, Combined effect of nano-silica and silica fume to improve concrete workability and compressive strength: a case study, Ing. Competitividad, 25, 1, 2023, e-21612201.
9. P.A. Handayani, E. Nurjanah, W.D.P. Rengga, Pemanfaatan limbah sekam padi menjadi silika gel, J. Bahan Alam Terbarukan, 4, 2, 2015, 55-59.
10. A. Azhari, M. Aziz, Sintesis dan karakterisasi material berpori berbasis mineral silika Pulau Belitung, J. Teknol. Miner. Batubara, 12, 3, 2016,

161-172.
11. L. Trivana, S. Sugiarti, E. Rohaeti, Sintesis dan karakterisasi natrium silikat (Na_2SiO_3) dari sekam padi, Indones. J. Chem. Sci., 4, 2, 2015, 86-93.
12. A. Hanafi, A.R. Nandang, Studi pengaruh bentuk silika dari abu ampas tebu terhadap kekuatan produk keramik, J. Kimia Indones., 5, 1, 2010, 35-38.
13. B.O. Adigun, I.O. Adeosun, A.A. Nwachukwu, Advanced materials development from corncob ash for glass-ceramic applications, Ceramics International, 46, 9, 2020, 15390-15398.
14. U. Kalapathy, A. Proctor, J. Schultz, A simple method for production of pure silica from rice hull ash, Bioresour. Technol., 73, 2000, 257-262.
15. T. Hidayat, R. Fajri, Sintesis silika dari abu sekam padi menggunakan variasi jenis asam pada proses sol-gel, Pros. ReTII, 15, 1, 2021, 201-207.
16. M.T. Rahman, M.A. Haque, M.A. Rouf, M.A.B. Siddique, M.S. Islam, Effect of ultrasonic treatment on moisture content and adsorption capacity of silica derived from rice husk ash, Bangladesh J. Sci. Ind. Res., 52, 2, 2017, 101-108.
17. S. Woranuch, R. Yoksan, Eugenol-loaded chitosan nanoparticles: Characterization and release study, Carbohydr. Polym., 96, 2, 2013, 586-591.
18. S. Agustina, I.M.D. Swantara, I.N. Suartha, Isolasi kitin, karakterisasi, dan sintesis kitosan dari kulit udang, J. Kimia, 9, 2, 2015, 271-278.
19. R.K.M. Raturoma, B. Mangallo, M.H. Langsa, Komposit kitosan-abu sekam padi sebagai pupuk lepas lambat Fe^{2+} dan Mn^{2+}, J. Natural, 17, 2, 2021, 97-103.
20. J. Ji, L.Li, L. Quan, B. Tian, P. Zhang, S. Li, The effects of calcination process parameters on RHA reactivity and mortar mechanical properties, Materials, 18, 3129, 2025, 1-12.
21. A.B.D. Nandiyanto, R. Oktiani, R. Ragadhita, How to read and interpret FT-IR spectra, Indones. J. Sci. Technol., 5, 2, 2020, 97-118.
22. M. Blachnio, M. Zienkiewicz-Strzalka, A. Derylo-Marczewska, L.V. Nosach, E.F. Voronin, Chitosan-silica composites for adsorption application in the treatment of water and wastewater from anionic dyes, International Journal of Molecular Sciences, 24, 14, 2023, 11818.
23. S. Mutrofin, L. Krisna, D. Mardiana, R.T. Tjahjanto, Acid isolation techniques for silica isolation from rice husk and determination of its physicochemical properties, J. Pure Appl. Chem. Res., 11, 1, 2022, 72-82.
24. A. Tetuko, R. Murhayanti, W. Sugiyo, Sintesis komposit kitosan–silika dan aplikasinya sebagai adsorben zat warna tekstil, Indones. J. Farmasi, 1, 1, 2016, 11-17.
25. Y. Farkhod, Y. Yulduzkhon, Y. Sukhrob, G. Temirov, Physicochemical characterization of anion-exchange resins based on polyvinyl chloride (PVC) and polyvinyl amine (PVA), Eur. J. Tech. Nat. Sci., 3, 2025, 24-28.
26. D. Dhaneswara, J.F. Fatriansyah, F.W. Situmorang, A.N. Haqoh, Synthesis of amorphous silica from rice husk ash: Comparing HCl and CH_3COOH acidification methods and various alkaline concentrations, Int. J. Technol., 11, 1, 2020, 200-208.
27. I.J. Fernandes, D.F. Calheiro, F.A.L. Sánchez, A.L.D. Camacho, T.L.A. de Campos Rocha, C.A.M. Moraes, V.C. de Sousa, Characterization of silica produced from rice husk ash: Comparison of purification and processing methods, Mater. Res., 20, Suppl. 2, 2017, 512-518.
28. R. Putri, R. Mulyawan, Z.A. Nasrul, Suryati, R. Nurlaila, Karakteristik silika dari sekam padi berdasarkan variasi waktu dan suhu pembakaran, Prosiding Seminar Nasional Fakultas Teknik, Universitas Malikussaleh, 2022, 906-911.
29. A. Salama, R.E. Abou-Zeid, Ionic chitosan/silica nanocomposite as efficient adsorbent for organic pollutants from aqueous systems, Colloids and Surfaces A: Physicochemical and Engineering Aspects, 626, 2021, 127042.
30. F.R. Scuto, C. Ciarlantini, V. Chiappini, L. Pietrelli, A. Piozzi, A.M. Girelli, Design of a 3D amino-functionalized rice husk ash nano-silica/chitosan/alginate composite as support for laccase immobilization, Polymers, 15, 14, 2023, 3127.
31. D. Rahmadhani, K.D. Yuliani, E. Frida, A. Taufiq, Hydrophobic and antibacterial properties of textiles using nanocomposite chitosan and SiO_2 from rice husk ash as coating, S. Afr. J. Chem. Eng., 48, 2024, 366-374.