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Synthesis of Rice Husk–Based Silica Gel Using Chemical Precipitation Method as an Adsorbent for Cu^{2+} Ions

Anisa Puspita Wardani, Febriana Putri Sagita

Departement of Chemical Engineering, Faculty of Engineering, Universitas Negeri Semarang, Indonesia

*Corresponding Author: anisapuspita979@students.unnes.ac.id

Abstract

Rice husk, an abundant agricultural waste in Indonesia, is a promising source of silica. This study investigates the synthesis of silica gel from rice husks using chemical precipitation and its potential for removing copper ions (Cu^{2+}) from wastewater. The silica gel was synthesized by dissolving rice husk ash in a 2 N NaOH solution, followed by precipitation with 1 N HCl. Adsorption experiments were conducted using $CuSO_4$ solutions at concentrations of 3, 6, and 9 ppm, with varying adsorbent masses (0.3, 0.6, and 0.9 g). The results demonstrated that increasing the adsorbent mass improved Cu^{2+} removal efficiency, with the highest efficiency observed at 0.9 g for the 3 ppm solution, achieving 86.43%. Adsorption isotherms were analyzed using the Langmuir and Freundlich models. The Freundlich model provided a better fit for the data, indicating the heterogeneous nature of the silica gel surface. This study highlights the potential of rice husk-derived silica gel as an effective adsorbent for heavy metal removal, contributing to both waste valorization and environmental protection.

Keywords: silica gel; rice husk; adsorption; Cu^{2+} ions; Langmuir isotherm; Freundlich isotherm

INTRODUCTION

Rice husk, one of the most abundant agricultural biomass wastes in Indonesia, is a byproduct of the country's high rice production. Due to its vast availability, rice husk presents significant potential for more optimal utilization as a value-added raw material [1]. Chemically, rice husks are composed of cellulose, lignin, and silica, with amorphous silica content ranging from 15 to 20%. This composition makes rice husk a promising source of natural silica [2]. However, despite its potential, the utilization of rice husk remains limited. Most are either burned or discarded without proper management, leading to environmental concerns. Several studies have explored methods to enhance the value of rice husks, such as converting them into silica gel. Silica gel is a porous material with a high surface area, good chemical stability, and excellent adsorption capacity, making it widely used in various purification and wastewater treatment applications [3]. The synthesis of silica gel from biomass, including rice husk, has gained attention, particularly through chemical precipitation methods, where silica is precipitated from a silicate solution using reagents to form a gel structure [4]. This method offers several advantages: it is relatively simple and enables the production of high-quality silica gel from biomass-based waste, such as rice husk ash [5]. Previous research has focused on synthesizing silica gel from various waste materials, but limited attention has been given to the specific potential of rice husk as a silica gel precursor and its application in wastewater treatment.

Heavy metal contamination, particularly copper (Cu^{2+}), in aquatic environments remains a significant concern due to its harmful effects on human health and ecosystems. Copper is commonly found in industrial wastewater from processes such as electroplating, metal refining, and textile production [6]. At high concentrations, Cu^{2+} ions are toxic and can cause organ damage, metabolic disorders, and bioaccumulation in aquatic organisms, potentially disrupting the food chain [7]. The Regulation of the Minister of Environment and Forestry of the Republic of Indonesia Number 5 of 2014 on Industrial Wastewater Quality Standards sets the maximum allowable concentration of copper in industrial effluent at 3 mg/L, which underscores the need for effective treatment methods to reduce Cu^{2+} concentrations to meet environmental quality standards.

Adsorption is widely regarded as one of the most cost-effective and efficient methods for wastewater treatment. A variety of biomass-based adsorbents, such as cellulose, activated carbon, and silica, have been extensively studied for the removal of heavy metals [8]. Silica gel, in particular, has shown great promise for Cu^{2+} ion adsorption due to its highly porous structure and chemical stability [9]. However, previous studies focusing on the use of rice husk-based silica gel synthesized via chemical precipitation, and investigating the effect of adsorbent mass on Cu^{2+} adsorption efficiency and capacity, remain scarce. For instance, while studies have explored the synthesis of silica from rice husk, few have systematically examined its application in heavy metal ion removal, especially in relation to mass variations of the adsorbent [10]. This research aims to fill this gap by investigating the synthesis of silica gel from rice husks via chemical precipitation and evaluating the impact of adsorbent mass on the efficiency of Cu^{2+} ion removal. By addressing this gap, this study provides valuable insights into optimizing the use of rice husk-derived silica gel for heavy metal removal, contributing to both waste valorization and environmental protection.

METHOD

Materials

The main material used in this study was rice husk, which served as the silica source. A 2 N NaOH solution and a 1 N HCl solution were used as reagents for the silica dissolution and precipitation processes, respectively. Additionally, $CuSO_4$ solutions with concentrations of 3, 6, and 9 ppm were prepared as the adsorption test solutions. Distilled water was used as a solvent and for washing all materials and reagents involved in the preparation process.

Equipment

The equipment used in this study included a furnace at 700°C, a hot plate magnetic stirrer, an analytical balance, a ball-filler, glass bottles, burettes, porcelain dishes, glass funnels, 250 mL Erlenmeyer flasks, watch glasses, 100 mL and 200 mL beakers, a digital pH meter, filter paper, porcelain crucibles, 100 mL volumetric flasks with stoppers, a magnetic stirrer, trays, crucible tongs, 5 mL and 10 mL measuring pipettes, dropper pipettes, and spatulas. The Cu^{2+} ion content in the solutions was analyzed using an Atomic Absorption Spectrophotometer (AAS).

Procedure

The study began with the preparation of rice husks, which were cleaned from impurities and then dried in an oven at 105 °C. The dried husks were subsequently burned in a furnace at 700 °C for 3 hours to produce grayish ash. The ash was cooled to room temperature and manually ground using a mortar and pestle. This grinding process aimed to obtain a finer and more uniform texture while preserving the initial particle characteristics, as no sieving was performed. The next step was silica extraction by dissolving the rice husk ash in a 2 N NaOH solution. A volume of 200 mL of 2 N NaOH solution was used for this process. The mixture was heated to 80°C while stirring continuously on a hot plate stirrer for 2 hours until a sodium silicate solution was formed. The resulting solution was then filtered using filter paper to separate insoluble residues. The sodium silicate filtrate underwent precipitation by the slow addition of 1 N HCl until a neutral pH was reached. This process yielded a silica gel precipitate, which was allowed to settle completely before washing. The precipitate was repeatedly washed with distilled water until free of ionic impurities. The purified silica gel was then dried in an oven at 120 °C to remove residual moisture. After drying, the silica gel was re-ground using a mortar and pestle to obtain a powdered form. The resulting powder was used as the adsorbent for the adsorption experiments. Manual homogenization of particle size was performed to maintain consistent particle characteristics, ensuring the adsorbent was ready for use [11].

Adsorption experiments were conducted using $CuSO_4$ solutions with concentrations of 3, 6, and 9 ppm, prepared separately. Each solution was mixed with silica gel adsorbent masses of 0.3, 0.6, and 0.9 g according to the experimental variations. To assess the relative effectiveness of the silica gel adsorbent, a control experiment was also conducted using a commercial silica adsorbent and a blank solution with no adsorbent. The mixtures were stirred for a predetermined contact time to ensure optimal interaction between the metal ions and the adsorbent surface. After adsorption, the mixtures were filtered to obtain the filtrates, which were analyzed using Atomic Absorption Spectrophotometry (AAS) to determine the remaining Cu^{2+} ion concentrations [12]. The results from the silica gel adsorbent were compared to those from the commercial silica adsorbent and the blank to evaluate the relative effectiveness of the silica gel. The Cu^{2+} concentration data from AAS analysis were used to calculate the adsorption capacity of silica gel for copper ions. Adsorption capacity was determined based on the difference between initial and final ion concentrations in the solution. These values served as the basis for evaluating the effectiveness of the adsorbent under different concentration and adsorbent mass conditions. The results were then integrated with operational parameters to assess the adsorbent performance, providing quantitative information on the potential of rice husk silica gel as a Cu^{2+} ion adsorbent [13].

Analysis

The equilibrium concentration of Cu^{2+} ions after the adsorption process (C_e) for each initial concentration was determined using Atomic Absorption Spectrophotometry (AAS). The adsorption capacity at equilibrium (Q) and removal efficiency (%E) were calculated using the following equations: Adsorption capacity [14]:

$$Q = \left(\frac{C_0 - C_e}{m}\right) \times V \tag{1}$$

Removal efficiency:

$$\%\,E = \left(\frac{C_0 - C_e}{C_0}\right) \times 100\% \tag{2}$$

Where:

Q : Adsorption capacity (mg/g)
C_0 : Initial concentration (mg/L)
C_e : Equilibrium concentration (mg/L)
V : Solution volume (L)
m : Adsorbent mass (g)
$\%\,E$: Removal efficiency

The Langmuir and Freundlich isotherm models were linearized to evaluate adsorption equilibrium and determine isotherm constants. The Langmuir model is expressed as follows:

$$qe = qm.K_L \frac{C_e}{1 + K_L \cdot C_e} \tag{3}$$

The linear form of the Langmuir equation is:

$$\frac{Ce}{qe} = \frac{1}{qm.K_L} + \frac{C_e}{qm} \tag{4}$$

Where:

qm : Langmuir adsorption capacity (mg/g)
KL : Langmuir adsorption constant (L/mg)

The Freundlich isotherm model is given by:

$$qe = K_F \,.\, C_e^{1/2} \tag{5}$$

Its linearized form is:

$$\log qe = \log K_F + {}^{1}\!/_{2}\, log\, C_e \tag{6}$$

Where :

qe : Freundlich adsorption capacity (mg/g)
C_e : Equilibrium solute concentration (mg/L)
K_F : Freundlich adsorption capacity constant
n : Adsorption intensity

RESULTS AND DISCUSSION

Silica Gel Yield

The yield of silica gel was determined based on the ratio between the initial mass of rice husks and the mass of silica gel obtained. Yield determination plays an important role in evaluating the efficiency of the silica extraction process. The calculated silica gel yield data obtained in this study are presented in the following tables and figures to illustrate the trend of increase for each variation in rice husk mass.

Table 1. Silica Gel Yield

Rice Husk Mass (g)	Silica Gel Mass (g)	Yield
3,02	1,98	0,66
6,03	4,64	0,77
9,02	7,15	0,79

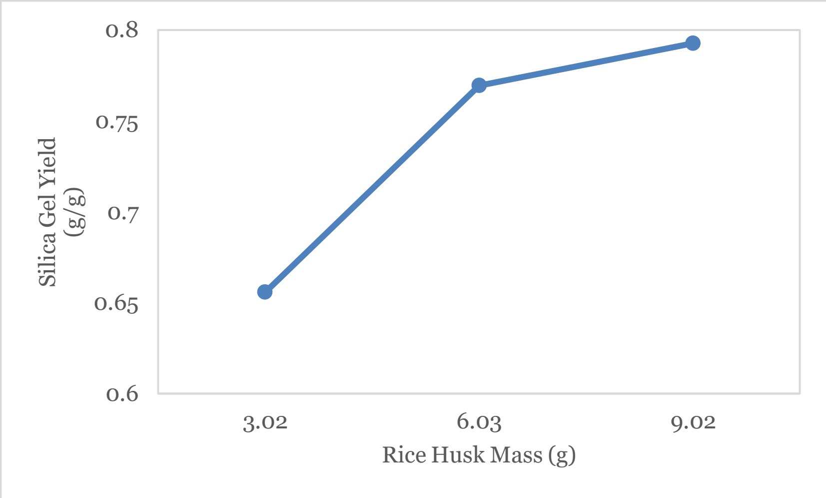

Figure 1. Silica Gel Yield Graph

Based on Table and Figure 1, the mass of rice husks plays a crucial role in the formation of silica gel. Variations in rice husk mass provide an insight into the capability of the extraction method to produce silica gel from different amounts of raw material. As the mass of rice husks increases, the resulting silica gel yield tends to rise. The use of 3.02 g of rice husks produced 1.98 g of silica gel, corresponding to a yield of 0.66 g/g. When the rice husk mass was increased to 6.03 g, the yield rose to 0.77 g/g. At 9.02 g of rice husks, the yield reached the highest value of 0.79 g/g. This increase in yield indicates that a larger amount of raw material provides more amorphous silica available for extraction during the dissolution and precipitation processes [14]. The phenomenon aligns with the findings of Carneiro et al. (2025), which reported that rice husks contain 15–20% silica; therefore, using a greater biomass amount increases the potential amorphous silica extractable in the dissolution and precipitation stages. Furthermore, the efficiency of the chemical precipitation process also affects the yield. A higher rice husk mass produces a larger volume of sodium silicate solution, enabling the gelation process to form a greater quantity of silica gel precipitate [1].

Copper Adsorption Capacity

The adsorption capacity of copper ions was tested using adsorbent masses of 0.3, 0.6, and 0.9 g. Based on the analysis of copper adsorption capacity at various concentrations, the results are shown as follows:

Table 1. Copper Adsorption Capacity

Adsorbent Mass (g)	Copper Adsorption Capacity at Different Concentrations (mg/g)		
	3	6	9
0,3	0,16	0,38	1,16
0,6	0,13	0,24	0,58
0,9	0,15	0,20	0,42

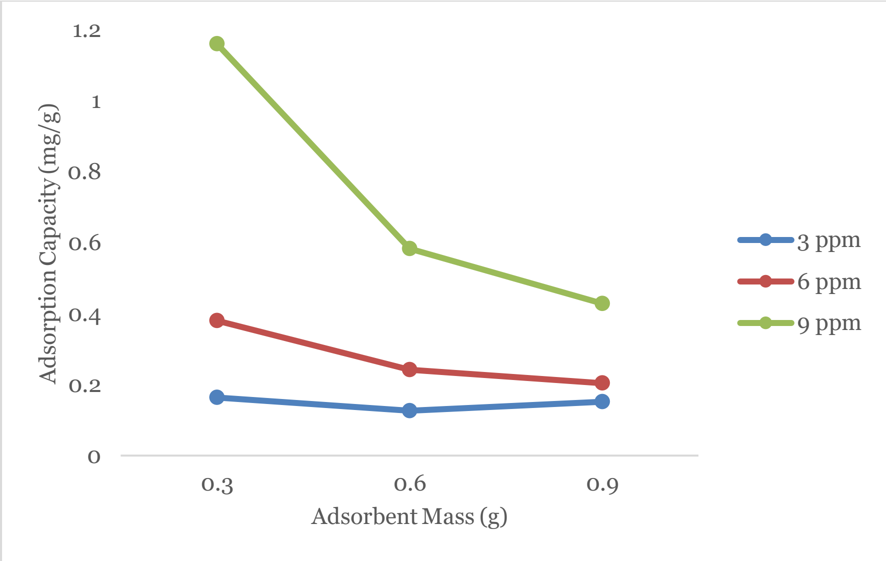

Figure 2. Copper Adsorption Capacity

Based on the analysis of Table and Figure 2, it can be observed that the adsorbent mass affects the adsorption capacity. As the adsorbent mass increases, the adsorption capacity per unit mass tends to decrease. This is due to the reduction in the number of ions adsorbed per unit mass of adsorbent [15]. Bourliva et al. (2025) stated that this phenomenon results from the adsorbent overdose effect, where the number of available active sites exceeds the number of metal ions in the solution, as well as possible agglomeration of adsorbent particles, which reduces the effective surface area and accessibility of active sites [16].

Silica Removal Efficiency for Copper Ions

The variation in adsorbent mass was used to determine the mass that achieved the highest Cu removal. The highest percentage of Cu removal indicates the most suitable adsorbent mass for the adsorption process in this study using rice husk silica gel. The adsorbent masses tested were 0.3, 0.6, and 0.9 g with $CuSO_4$ solution concentrations of 3, 6, and 9 ppm. The results of the silica removal efficiency for Cu ions are presented in the following table:

Table 2. Silica Removal Efficiency for Copper Ions

Adsorbent Mass (g)	Cu Removal Efficiency (%) at Different Concentrations		
	3	**6**	**9**
0,3	30,98	41,31	61,18
0,6	47,88	52,61	61,52
0,9	86,43	66,51	67,81

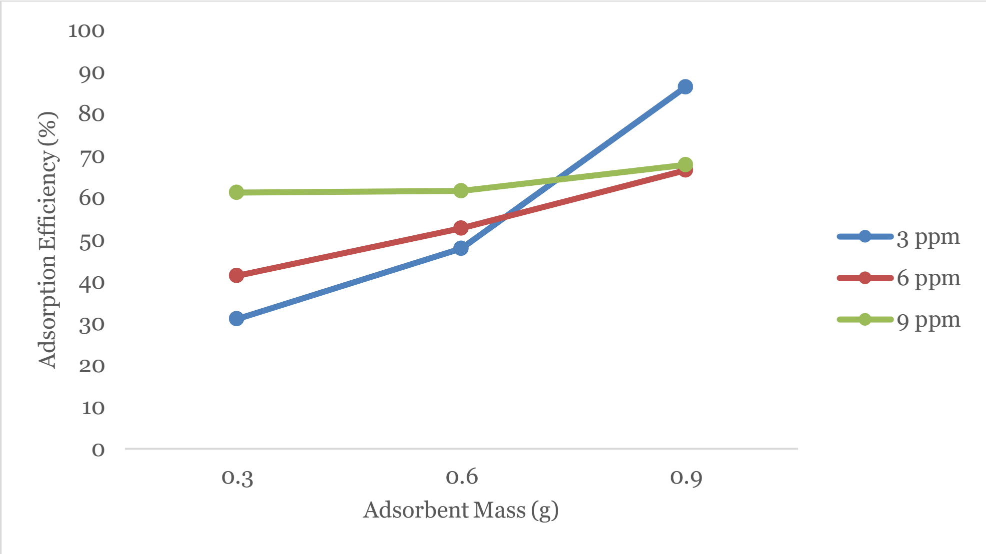

Figure 3. Adsorption Efficiency of Silica Gel toward Cu^{2+} Ions

Based on the data presented in Table and Figure 3, it is evident that increasing the adsorbent mass results in a gradual increase in the percentage of Cu removal. However, an anomaly occurs at an adsorbent mass of 0.9 g, where the efficiency at 3 ppm exceeds the expected trend and reaches 86.43%, which is higher than the 67.81% observed at 9 ppm. This increase in efficiency at lower concentrations can be attributed to the higher available surface area of the adsorbent at a higher mass, which facilitates more interaction sites for Cu^{2+} ions [16]. As the adsorbent mass increases, more active sites become available for adsorption. At higher concentrations of Cu^{2+}, such as 9 ppm, the efficiency does not increase linearly, which suggests the potential limitation of active sites. At higher adsorbate concentrations, the active sites on the adsorbent may become saturated, leading to a reduction in adsorption efficiency. This phenomenon occurs due to the competitive nature of the adsorption process, where Cu^{2+} ions at high concentrations may compete for limited active sites on the adsorbent surface [17].Thus, while the adsorbent mass has a significant effect on Cu removal efficiency, the adsorbent/adsorbate ratio plays a critical role in determining the effectiveness of the adsorption process. At higher concentrations, the limited number of active sites can hinder further increases in adsorption efficiency, highlighting the need for optimized adsorbent mass in relation to the concentration of the contaminant. The increase in adsorbent mass results in a larger surface area, allowing more adsorbate to be adsorbed [18]. Thus, as the adsorbent mass increases, the silica removal efficiency for Cu ions also rises. This occurs because a higher adsorbent mass corresponds to an increased number of particles and a larger surface area, thereby increasing the number of active sites available for ion binding and enhancing removal efficiency [19].

Adsorption Isotherm Models

Adsorption isotherm models are used to determine the adsorption capacity of an adsorbent toward an adsorbate. Adsorption isotherms provide equilibrium equations that describe the ability of the adsorbent to bind the adsorbate. The equilibrium data are generally presented in isotherm curves, which show the relationship between the amount of adsorbate adsorbed on the adsorbent (q_e) and the adsorbate concentration in the solution at equilibrium (C_e).

Langmuir Method

The Langmuir isotherm equation was determined by calculating the copper concentration after the adsorption process (C), the amount of copper adsorbed (X), and the mass of silica adsorbent (M). The relationship between the equilibrium concentration (C_e) and the adsorption capacity at equilibrium (Q_e), derived from $\frac{x}{m}$ was then used as the basis for constructing the isotherm model, as illustrated in the following figure:

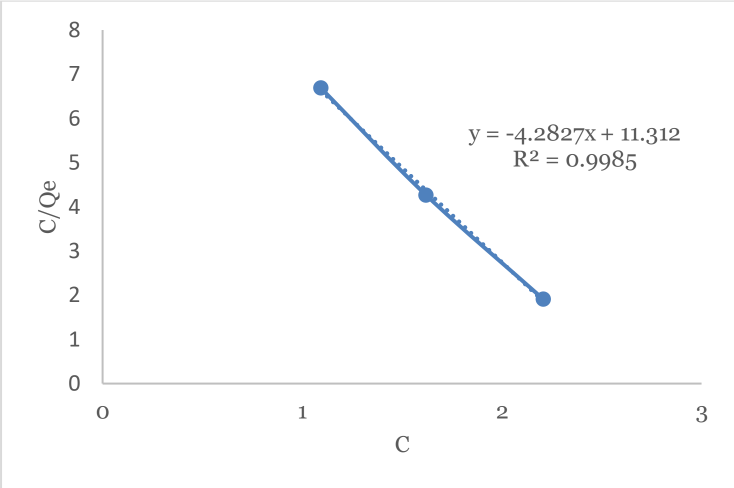

Figure 4. Langmuir Isotherm Plot at an Adsorbent Mass of 0.3 g

Based on Figure 3, the R^2 value obtained using the Langmuir method was 0.9985. The coefficient of determination (R^2) is used as an indicator of the isotherm model fit. An R^2 value close to 1 indicates that the model can explain the majority of the variability in the adsorption data [20].

Table 3. Langmuir Isotherm Parameters for Cu^{2+} Adsorption

Mass (g)	1/KQm	1/Qm	Qm	K
0,3	11,312	-4,282	-0,233	-0,378
0,6	8,1357	-2,016	-0,496	-0247
0,9	1,7456	1,675	0,596	0,959

Based on Table 4, it can be observed that as the adsorbent mass increases, the Q_m value also increases. This indicates that the monolayer adsorption capacity of the adsorbent tends to be higher when the adsorbent mass is smaller [21]. According to Hussein et al. (2025), an increase in adsorbent mass corresponds to a higher number of particles and a larger adsorbent surface area, which increases the number of available ion binding sites. The greater the adsorbent mass, the higher the adsorption capacity, because during the initial stage of the adsorption process, Cu^{2+} ions rapidly bind to the silica surface while all active sites and pore cavities of the adsorbent remain fully accessible. This condition allows Cu^{2+} ions to interact intensively and occupy the silica surface through ion exchange and coordination bonding mechanisms. As the adsorbent mass increases, the number of pores and available surface area of the silica also rises, providing more binding sites for Cu^{2+} ions. The addition of surface area is responsible for the increased adsorption capacity, as more Cu^{2+} ions can be accumulated on the rice husk silica surface [23].

Freundlich Method

The Freundlich isotherm equation was determined by calculating the mass of Cu after the adsorption process (C), the amount of Cu adsorbed (X), and the mass of the silica adsorbent (M). The relationship between log C and log Q_e, derived from X/M, was then used as the basis for constructing the isotherm model, as illustrated in the following figure:

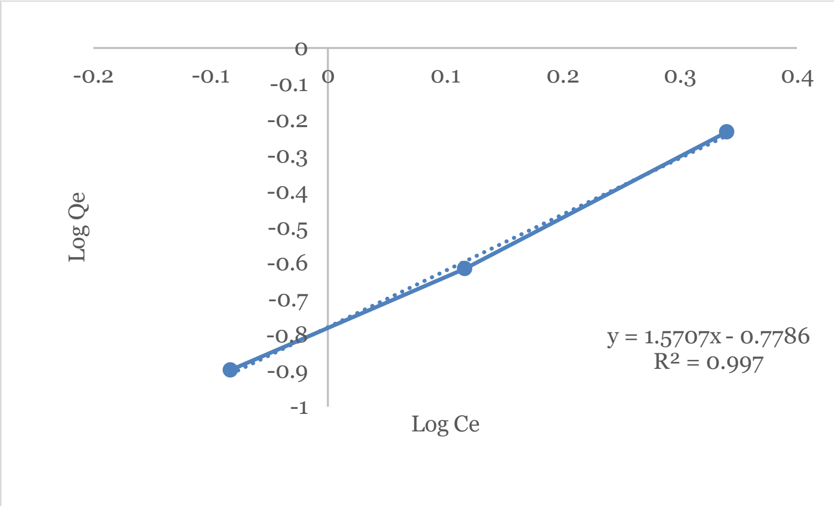

Figure 5. Freundlich Isotherm Model for Cu^{2+} Adsorption at an Adsorbent Mass of 0.6 g

Based on the Freundlich isotherm analysis, an adsorbent mass of 0.6 g exhibited the best model fit, with an R^2 value of 0.997. The logarithmic regression line from the graph was used to determine the adsorption intensity parameter (n) and the adsorption capacity constant (K_F). The values of these two parameters are presented in the following table as a basis for evaluating the adsorption characteristics of silica gel toward Cu^{2+} ions:

Table 4. Freundlich Isotherm Adsorption Capacity Parameters

Mass (g)	Log K	1/n	K	n
0,3	-0,925	2,759	0,118	0,362
0,6	-0,778	1,570	0,166	0636
0,9	-0,561	0,438	0,274	2,278

The selection of the appropriate adsorption isotherm model was carried out by comparing the coefficient of determination (R^2) and evaluating the physical validity of the obtained isotherm parameters. Although the Langmuir model produced a very high R^2 value of 0.9985 at an adsorbent mass of 0.3 g, this high linearity was not consistently observed at higher adsorbent masses. At 0.9 g, the R^2 value significantly decreased to 0.6156, indicating poor model reliability across different experimental conditions. Furthermore, several Langmuir parameters, including the maximum monolayer adsorption capacity (Qm) and the Langmuir constant (K), exhibited non-physical negative values at certain adsorbent masses. These results violate the fundamental assumptions of the Langmuir model, which presumes homogeneous surface sites, monolayer adsorption, and energetically equivalent binding sites [21]. Therefore, despite high R^2 values at specific conditions, the Langmuir model was deemed unsuitable for describing the adsorption behavior of Cu^{2+} ions onto rice husk–derived silica gel. In contrast, the Freundlich isotherm model demonstrated consistently high R^2 values across all adsorbent mass variations, with the best fit observed at an adsorbent mass of 0.6 g ($R^2 = 0.997$). The Freundlich model assumes a heterogeneous surface with non-uniform adsorption energies and allows for multilayer adsorption, which is more representative of the structural characteristics of silica gel derived from agricultural biomass [22].

The Freundlich parameters further support this interpretation. The adsorption intensity parameter (n) indicates favorable adsorption interactions, while the Freundlich constant (K_F) reflects adequate adsorption capacity at equilibrium. The relatively low values of 1/n suggest strong interactions between Cu^{2+} ions and the active sites of the silica gel surface. These findings are consistent with the observed adsorption capacity and efficiency trends, where optimal adsorption behavior was achieved at an adsorbent mass of 0.6 g. Based on the consistency of R^2 values, the physical validity of the isotherm parameters, and the agreement between experimental results and theoretical assumptions, the Freundlich isotherm model was identified as the most appropriate model for describing the adsorption behavior of Cu^{2+} ions onto rice husk based silica gel [23].

Adsorption Isotherm Model Selection

The selection of the adsorption isotherm model was performed by comparing the coefficient of determination (R^2) values of each linearized equation. According to Sajjadi (2024), the best model is determined based on the most linear regression line with the highest R^2 value. The correlation coefficient is used to assess the level of agreement between the isotherm model and the experimental data [24]. In this study, the Langmuir model produced the highest R^2 value of 0.9985 at an adsorbent mass of 0.3 g; however, this value was inconsistent across other adsorbent masses, as the R^2 decreased to only 0.6156 at 0.9 g. Moreover, some Langmuir parameters, such as Q_m and the adsorption constant K, were negative at certain adsorbent masses, which is physically invalid. Therefore, the Langmuir model cannot adequately describe the adsorption mechanism in this study.

On the other hand, the Freundlich model exhibited high and stable R^2 values across all adsorbent mass variations, with the best fit observed at 0.6 g, yielding an R^2 of 0.997. This pattern aligns with the characteristics of silica gel-based adsorbents, which have heterogeneous surfaces, causing the adsorption process to proceed in a multilayer manner with variable adsorption site energies. This finding is consistent with the study by Kagalkar et al. (2025), which stated that the Freundlich model is more representative for describing heavy metal adsorption on heterogeneous surfaces compared to the Langmuir model [25]. Based on the consistency of R^2 values, the validity of the obtained isotherm parameters, and the theoretical assumptions of the models relative to the surface characteristics of silica gel, it can be concluded that the Freundlich isotherm is the most appropriate model to describe Cu^{2+} ion adsorption in this study.

CONCLUSION

This study successfully synthesized silica gel from rice husks through chemical precipitation and evaluated its performance in removing Cu^{2+} ions from aqueous solutions. The adsorption efficiency increased with higher adsorbent masses, with 86.43% removal observed at 3 ppm and 0.9 g of silica gel. The Freundlich isotherm model was found to better describe the adsorption behavior, suggesting a heterogeneous surface with non-uniform adsorption sites. These findings demonstrate the potential of rice husk-derived silica gel as a low-cost and effective adsorbent for treating industrial wastewater contaminated with copper ions. Future studies could explore optimizing the silica gel synthesis process and investigating its application to other heavy metals and environmental pollutants.

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