

การศึกษาสภาวะที่เหมาะสมในการดูดซับไอออนเหล็กโดยใช้ซิลิกาที่เตรียมจากแกลบข้าวเหนียวและแกลบข้าวเจ้า

Optimal Conditions for Adsorption of Fe(II) ions Using Silica Prepared from Glutinous and Jasmine Rice Husks

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บทคัดย่อ

งานวิจัยนี้ได้เตรียมซิลิกาจากแกลบข้าวเหนียว (พันธุ์ กข 6) และแกลบข้าวเจ้า (พันธุ์หอมมะลิ 105) และศึกษาสภาวะที่เหมาะสมในการดูดซับไอออนเหล็ก ในน้ำของซิลิกาที่เตรียมได้ โดยทำการสกัดซิลิกาจากแกลบโดยการรีฟลักซ์ด้วยกรดไฮโดรคลอริกเข้มข้น 1 โมลาร์ จากนั้นกรองและเผาที่อุณหภูมิ 600 องศาเซลเซียส เป็นเวลา 6 ชั่วโมง ได้ซิลิกามีลักษณะเป็นผง สีขาว ยืนยันโครงสร้างด้วยเทคนิคเอกซเรย์ดิฟแฟรกชัน ศึกษารูปร่างภายนอกและพื้นผิวด้วยกล้องจุลทรรศน์อิเล็กตรอนแบบส่องกราด และวิเคราะห์ความบริสุทธิ์ของซิลิกาด้วยเทคนิคเอกซเรย์ฟลูออเรสเซนซ์ ผลการศึกษา พบว่ารูปแบบการเลี้ยวเบนรังสีเอกซ์ของซิลิกาที่เตรียมได้จากแกลบทั้งสองปรากฏพิคกว้างอยู่ในช่วงประมาณ 22 องศาซึ่งเป็นพิคเอกลักษณ์ของซิลิกาอสัณฐาน พื้นผิวของซิลิกามีลักษณะขรุขระ มีรูพรุน และมีความบริสุทธิ์มากกว่า 99 เปอร์เซ็นต์ ซิลิกาที่เตรียมได้นำมาศึกษาสภาวะที่เหมาะสมในการดูดซับไอออนเหล็กโดยการทดลองแบบแบช ศึกษาพารามิเตอร์ต่างๆ ที่มีผลต่อการดูดซับ ได้แก่ พีเอช เวลาที่ใช้ในการดูดซับ ปริมาณตัวดูดซับ และความเข้มข้นของไอออนเหล็กเริ่มต้น โดยวิเคราะห์ปริมาณเหล็กด้วยเทคนิคอะตอมมิกแอบซอร์พชันสเปกโทรสโกปี จากการศึกษาพบว่าสภาวะที่เหมาะสมในการดูดซับเหล็กของซิลิกาจากแกลบข้าวเหนียว คือ พีเอช 6 เวลาที่ใช้ในการดูดซับ 30 นาที ปริมาณตัวดูดซับ 0.4 กรัม และความเข้มข้นของเหล็กเริ่มต้นที่ 30 มิลลิกรัมต่อลิตร สำหรับสภาวะที่เหมาะสมในการดูดซับเหล็กของซิลิกาจากแกลบข้าวเจ้าใกล้เคียงกับซิลิกาจากแกลบข้าวเหนียว คือ พีเอช 6 เวลาที่ใช้ในการดูดซับที่เหมาะสม คือ 20 นาที ปริมาณตัวดูดซับ 0.6 กรัม และความเข้มข้นของเหล็กเริ่มต้น คือ 20 มิลลิกรัมต่อลิตร ซิลิกาจากแกลบข้าวทั้งสองชนิด มีเปอร์เซ็นต์การดูดซับที่ดีใกล้เคียงกัน โดยดูดซับได้สูงสุดที่ 99 เปอร์เซ็นต์

คำสำคัญ : การดูดซับ เหล็ก ซิลิกา แกลบข้าวเหนียว แกลบข้าวเจ้า

Abstract

Silica (SiO₂) was prepared from two types of rice husks, glutinous rice (type RD6) and jasmine rice (type Hom Mali 105) the optimal conditions for adsorption of Fe(II) ions in synthetic water using the prepared silica were studied. Silica was extracted from rice husks by refluxing in 1 M hydrochloric acid, filtration and heating at 600 °C for 6 hours in an oven. Silica products were obtained as white powder. Amorphous silica phase was confirmed by X-ray diffraction, morphology and surface were revealed by scanning electron microscope and the silica content was analyzed by X-ray

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fluorescence spectroscopy. The XRD patterns of products from glutinous and jasmine rice husks were similar and showed a broad peak at about 22° which corresponded to the amorphous silica phase. The prepared silica had a rough surface with porous morphology and high purity (SiO_2 content about 99%). Batch experiments were carried out to determine the optimal conditions of Fe(II) adsorption. The factors affecting the adsorption percentage included initial pH, contact time, adsorbent dosage, and initial Fe(II) concentration. Atomic absorption spectroscopy was employed to analyze the Fe(II) adsorption percentages. The optimal conditions for Fe(II) adsorption by silica extracted from glutinous rice husks were pH 6, 30 minute contact time, 0.4 g adsorbent dosage and 30 mg/L initial Fe(II) concentration. The optimal conditions for silica extracted from jasmine rice husk were similar to those of silica from glutinous rice husk including pH 6, 20 minute contact time, 0.6 g adsorbent dosage and 20 mg/L initial Fe(II) concentration. Silica extracted from both rice husks showed good Fe(II) adsorption with 99 percentages.

Keywords : adsorption, Fe(II), silica, glutinous rice husk, jasmine rice husk

Introduction

Iron is a common metal found in the earth's crust. Groundwater which percolates through soil and rock can dissolve minerals containing iron and hold them in solution. When exposed to air, dissolved iron in groundwater becomes insoluble and leaves the water with brown-red colour and may cause taste, odour, or turbidity problems. Also household activities and industries such as electroplating, mining, and alloy manufacturing also cause metal contamination in water. Based on groundwater quality reported by Department of Groundwater Resources in Nakhon Ratchasima-Ubon Ratchathani basin, the most commonly found metal contaminant is iron with the highest amount of 600 mg/L found at Srakaeo, Kankong District, Buriram Province¹ This amount is 600 times higher than public drinking groundwater standards requirement iron ($\text{Fe} \leq 1.0 \text{ mg/L}$).²

There are many techniques for metal removal such as precipitation, ion-exchange, coagulation and electrolysis. Among these techniques adsorption techniques are widely used for metal removal in water treatment and the adsorption capacity depends on morphology, composition, and porosity of the adsorbents used. Activated carbon has been frequently used as an adsorbent in water and wastewater treatment industries, however activated carbon is expensive. The adsorption of heavy metal ions by low cost adsorbents or agricultural waste materials have been widely studied.^[3-7]

Rice production is a significant portion of the agriculture in Thailand and rice husk is an agricultural

waste material generated in the rice polishing process. The weight of rice husk is around one third of the total rice weight. Rice husk consists of cellulose, hemicellulose and lignin at about 74% and inorganic contents such as silica alumina and calcium at about 26%. Eighty percent of inorganic matter is silica.⁸ Due to the high percentage of silica in rice husks, therefore, extraction of silica from rice husks and application in various fields have been carried out by many research groups.⁹⁻¹⁰

Silica content in rice husk ash varies from 83–98% depending on preparation techniques and conditions. Sorbent materials derived from rice husks have been effectively used for removal of various heavy metals such as Fe, Mn, Zn, Cu, Cd and Pb.¹¹

Silica from rice husks is an alternative and value material for metal removal because silica is insoluble in water, has good chemical stability, high biosorption capacity, high cost effectiveness, and abundant availability.¹²

This work aims to prepare silica from glutinous and jasmine rice husks and find suitable conditions for Fe(II) adsorption of the prepared silica. If this study produced a favorable result it would support the use of silica as an alternative adsorbent for iron removal in groundwater.

Materials and methods

1. Materials

Thai glutinous rice (type RD6) husk and Thai jasmine rice (type Hom Mali 105) husk were used as raw

materials. The husks were collected from a local rice mill at Phosai District, Ubon Ratchathani, Thailand.

2. Instruments

Powder X-ray diffraction (XRD) was used to characterize the phase of prepared silica. Chemical compositions of the husks and the silica were determined by using an X-ray fluorescence (XRF) spectrometer. Morphology and porosity of silica were characterized using a scanning electron microscope (SEM). Fe(II) concentrations were determined using an atomic absorption spectrometer (AAS). XRD and AAS instruments used in this work were provided by the Faculty of Science, Mahasarakham University. XRF and SEM instruments were provided by the center for scientific and technological equipment, Suranaree University of Technology.

3. Methods

3.1 Preparation of silica from rice husks

Silica from glutinous and jasmine rice husks was prepared using the procedure described by Leela-adisorn.¹³ Husks were first cleaned with water then dried in an oven at 105 °C for one night. After drying, 30 g of the husk sample was put in a round-bottom flask. 300 ml of 1 M HCl added and refluxed for 3 hours. After refluxing, the sample was filtered and washed several times with distilled water until the pH of the rinsed water reached 7, confirming that there was no HCl remaining in the sample, which was then dried in an oven at 105 °C to remove all remaining moisture. Finally, the dried husk sample was heated in an oven at 600 °C for 6 hours, resulting in a white powder sample ready for characterization.

3.2 Determination of optimal conditions for Fe(II) adsorption

Stock solution of 200 mg/L of Fe(II) aqueous solution was first prepared by dissolving 0.4965 g of ferrous sulfate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) in 500 ml distilled water. Fe(II) solutions with lower concentrations were prepared by diluting the required volume of stock solution with phthalate buffer solution at desired pH. The pH of the sample solution was adjusted by using HCl or NaOH solution.

The parameters used to determine suitable conditions for Fe(II) adsorption on prepared silica were

pH, contact time, adsorbent dose, and the initial metal concentration. The adsorption tests of both prepared silicas were carried out using batch procedures. General experimental set up used shaking speed of 130 rpm for 30 minutes and Fe(II) adsorption determined by AAS was reported as adsorption percentage calculated by the following equation:

$$\% \text{ Adsorption} = \frac{C_0 - C_e}{C_0} \times 100$$

where C_0 is the initial concentration of Fe(II) ions (mg/L) and C_e is the concentration of Fe(II) ions at adsorption equilibrium (mg/L)

The studied details of each parameters are as follows:

Effect of initial pH. Five 20 mg/L Fe(II) solutions with different pH, 2 4 6 8 and 10, were studied and prepared by diluting the stock solution with desired pH buffer and pH adjusted with HCl and NaOH solution. Then, 50 mL of each sample solution was transferred to a conical flask and 0.05 g of silica was added to each flask. The sample was shaken on a table shaker at 130 rpm for 30 minutes. Finally, the sample solution was filtered through a filter paper, and the filtered solution analyzed for the remaining Fe(II) ions using AAS measurement.

Effect of contact time. The solution of 20 mg/L Fe(II) ions was prepared from stock and adjust pH to 6. Solutions of 50 mL were transferred to conical flasks and 0.05 g of silica was added to each flask. The mixtures were shaken on a table shaker at 130 rpm with varying shaking time by 20 30 40 50 and 60 minutes. After the desired shaking time, the mixture were filtered through a filter paper and remaining Fe(II) ions was determined by AAS.

Effect of adsorbent dose. Prepared solutions of 20 mg/L Fe(II) at pH 6 were transferred to conical flasks in 50 mL for each flask. Different amounts of silica were added to the solution in each flask (0.2, 0.4, 0.6, 0.8, and 1.0 g). Each mixture was shaken on a table shaker at 130 rpm for 30 minutes. Finally, the sample solution was filtered through a filter paper, and the filtered solution was

analyzed for the remaining Fe(II).

Effect of initial Fe(II) concentration. A series of concentrations of Fe(II) solution, comprising 10, 20, 30, 40, and 50 mg/L were prepared from the stock solution. The pH of each solution was adjusted to 6. 50 mL solutions of each concentration were transferred to conical flask and 0.05 g of prepared silica were added and shaken on a table shaker at 130 rpm for 30 minutes. After filtration, the solution was analyzed for the remaining Fe(II) ions.

Results and Discussion

1. Silica extraction from rice husks

Glutinous rice husk showed more brown color and was thicker than jasmine rice husk. After refluxing in acid and heating the filtrated solid at 600 °C in oven, white silica products were obtained at percentage yield of 13.67 and 12.76% for glutinous and jasmine rice husks respectively. Yield of silica powder from glutinous rice husk was slightly higher than that obtained from jasmine rice husk which agrees with the percent of silica presented in rice husk raw materials as shown in Table 1. By naked eyes observation, silica powder extracted from jasmine rice husk is finer than those from glutinous rice husk as shown in Figure 1.

Table 1 Composition analysis of rice husk raw materials and extracted silica before and after Fe(II) adsorption (%w/w).

Composition (%)	glutinous rice husk raw materials	jasmine rice husk raw materials	Silica from glutinous rice husk		Silica from jasmine rice husk	
			Before adsorption	After adsorption	Before adsorption	After adsorption
SiO ₂	89.28	85.87	99.75	99.68	99.45	99.52
P ₂ O ₅	4.57	5.76				
SO ₃	1.28	1.44	0.10	N.D.	0.13	N.D.
K ₂ O	2.01	3.08				
CaO	1.00	0.87	0.11	0.04	0.28	0.06
TiO ₂	0.60	0.73				
MnO ₂	0.80	1.35				
Fe ₂ O ₃	0.42	0.80	N.D.	0.26	0.11	0.41
others	0.06	0.10	0.03	0.02	0.02	0.01

2. Characterization of extracted silica

2.1 Elemental composition analysis by

XRF

XRF is a powerful quantitative and qualitative analytical technique for elemental analysis of materials. When elements in a sample are exposed to high intensity X-rays, fluorescent X-rays will be emitted from the sample at unique energy levels related to those elements. The quantity of elements are reported as oxide based on weight percentages. Elemental compositions of rice husk raw materials, extracted silica before and after Fe(II) adsorption shown in Table 1. The main inorganic composition of both rice husks is silica (SiO₂) of which

glutinous and jasmine rice husks contain 89.28 and 85.87% respectively. Extracted silica from both rice husks showed similar and high percentage silica purity, about 99% because the refluxing in acid and heating in an oven at high temperature can remove the organic contents and some of inorganic impurity. These results agree with the data presented by Tanachai Jongsuwanpaisan.¹⁴ After Fe adsorption, the composition of silica adsorbent were analyzed to confirm the presence of Fe. The percentage of Fe in silica after adsorption increased to 0.26 and 0.41% for glutinous and jasmine rice husk silica, respectively. This result shows that Fe content was adsorbed into both samples of silica.

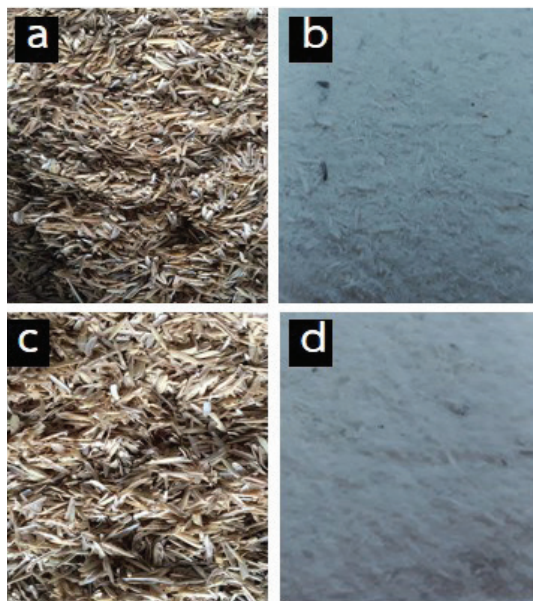


Figure 1 Glutinous and jasmine rice husk raw materials (a, c) and extracted silica powder from glutinous and jasmine rice husk (b, d).

2.2 Phase analysis by XRD

XRD is an analytical technique primarily used for phase identification of a crystalline material and also measurement of sample purity. The analyzed material should be finely ground, homogenized, and average bulk composition determined.

The XRD patterns of silica from both rice husks showed a broad peak at 22° which is a characteristic of amorphous silica¹⁵⁻¹⁶ as shown in Figure 2. There was no impurity peak present in the patterns indicating that the extracted silica was high purity.

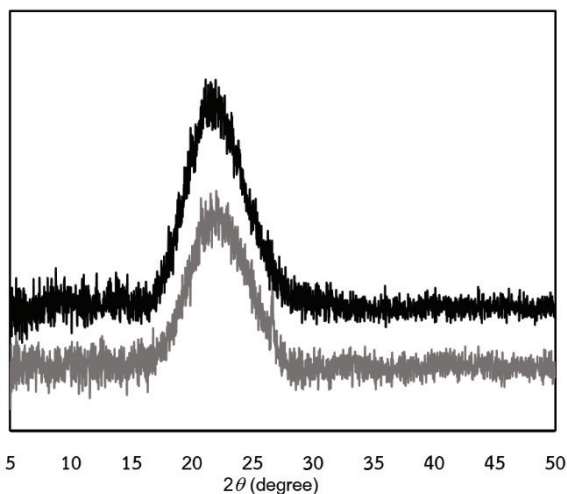


Figure 2 XRD patterns of silica from glutinous rice husk (top) and jasmine rice husk (bottom).

This result agrees with the composition analysis by XRF. The characteristic peak of elements in trace amount may be overcome by background signal, therefore no major impurity peaks were present.

2.3 Morphological analysis by SEM

SEM uses a finely focused electron beam to produce a high resolution image of a sample and shows a three dimensional appearance, which is very useful when examining the morphology, surface structure and porosity of a sample. SEM images of extracted silica from glutinous and jasmine rice husks show a polygonal shape with rough surface and porosity. After Fe(II) adsorption, the morphology of silica is similar. This result suggest that extracted silica from rice husks can be used as adsorbent without losing the surface structure as shown in Figure 3.

3. Study of optimal conditions for Fe(II) ion adsorption

The adsorption experiments were performed using aqueous solutions of ferrous sulphate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$), optimized conditions including initial pH, contact time, adsorbent dose and initial Fe(II) concentration were studied by batch experiment.

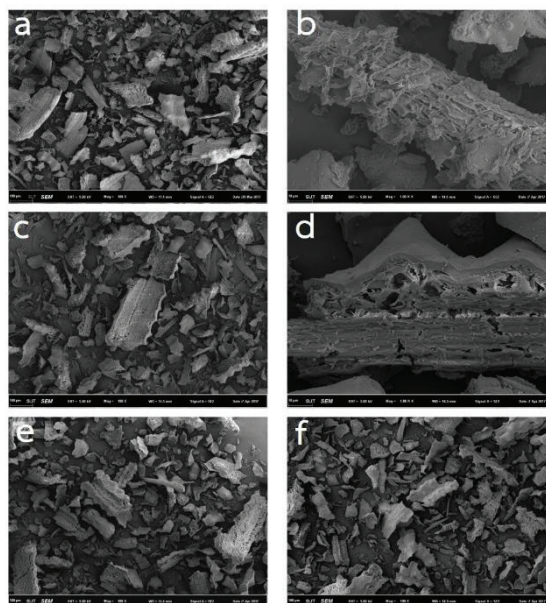


Figure 3 SEM magnifications of silica from glutinous rice husk (a, b), silica from jasmine rice husk (c, d), silica from glutinous rice husk after adsorption (e), silica from jasmine rice husk after adsorption (f).

Effect of initial pH.

The pH of solution media affects the adsorption capacity because pH can induces charge on the surface of the adsorbent, and alter the degree of ionization and speciation of the adsorbate.[17] The effect of initial pH on Fe(II) adsorption was studied by varying the pH as 2, 4, 6, 8 and 10.

The effects of pH on the adsorption of Fe(II) ions from the aqueous solution in terms of adsorption percentage is presented in Fig. 4(a). It is clear that Fe(II) ions were effectively adsorbed at pH 6 and the maximum adsorption

percentages by silica from glutinous and jasmine rice husks are 48.2 and 69.3%, respectively. Thus, pH 6 was chosen in further studies of other parameters. These results agree with the data obtained by Rodda and Samala [18-19] for heavy metal ions sorption onto agricultural waste sorbents. In the basic solution (pH 8 and 10), the hydrolysis of Fe(II) ions takes place and forms poorly soluble iron(II) hydroxide solid precipitate in solution.²⁰ In highly acidic condition (pH 2 and 4), the surface of adsorbents are protonated by H⁺ which reduces the Fe(II) adsorption capacity.²¹

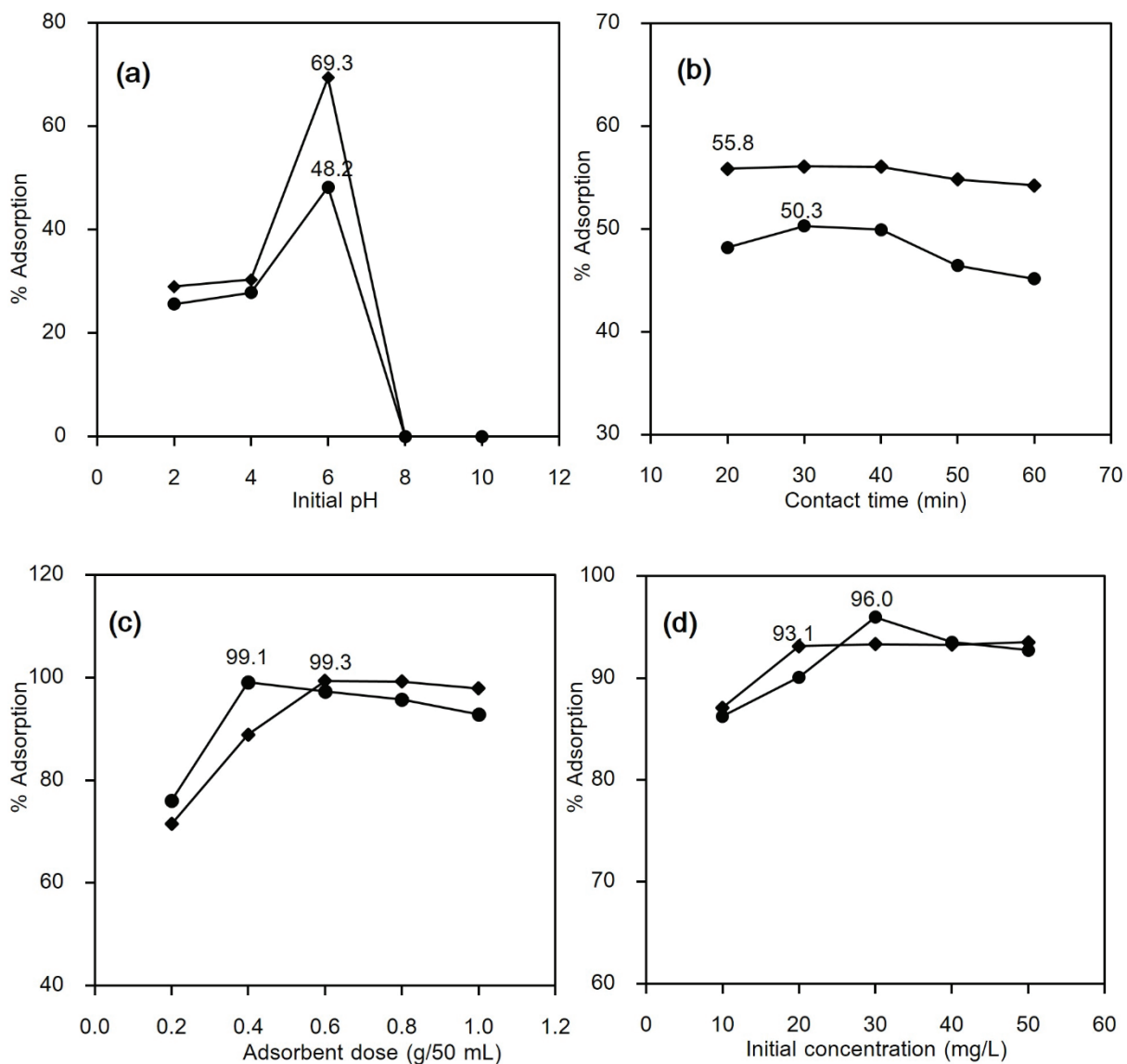


Figure 4 Effect of adsorption parameter on Fe(II) ionsuptake of prepared silica, (a) initial pH (b) contact time (c) adsorbent dosage (d) initial Fe(II) concentration (● = silica from glutinous rice husk ◆ = silica from jasmine rice husk).

Effect of contact time.

The effects of contact time on the adsorption were assessed by varying of shaking time to 20 30 40 50 and 60 minutes with other parameters remaining fixed. It was observed that the highest uptake of Fe(II) ions was at 20 and 30 minutes for silica extracted from glutinous and jasmine rice husks, respectively. After this time. the adsorption equilibrium and adsorption percentage slightly decreased as shown in Figure 4(b). Silica extracted from jasmine rice husk reached equilibrium within 20 minutes with the maximum uptake of 56.8% while the silica extracted from glutinous rice husk reaches equilibrium at 30 minutes with the maximum uptake of 50.3%. The adsorption reaches equilibrium at beginning state due to the high concentrations of Fe(II) ions compared to amount of available adsorption site of adsorbent. The result agrees with the metal adsorption by zeolite studied by Mehdizadeh et al, in which the adsorption reached equilibrium at 10-30 minutes depending on metal ions.²⁰

Effect of adsorbent dosage

Adsorbent dosage or the amount of adsorbent is one of the important parameters on adsorption because it implies the adsorption capability when the concentration of Fe(II) ions is constant.

The experiment limited the amount of silica as adsorbent at 0.2 0.4 0.6 0.8 and 1.0 g in 20 mg/L Fe(II) solution 50 mL and pH to 6. Silica from glutinous rice showed maximum uptake at 0.4 g dosage while silica from jasmine rice showed maximum uptake at 0.6 g dosage with nearly 100% adsorption as shown in Figure 4(c). The adsorption of Fe(II) ions increased rapidly with increasing dose of silica due to the greater availability of the exchange sites or surface area.²⁰

Effect of initial concentration of Fe(II) ions

The effect of initial concentration of Fe(II) ions on the adsorption percentage of Fe(II) ions by extracted silica was studied by batch adsorption experiment using different initial Fe(II) concentrations at 10, 20, 30, 40 and 50 mg/L at optimal pH. The result is shown in Figure 4(d). It can be seen that the maximum adsorption of silica from glutinous and jasmine rice husks occurred at the initial concentration 30 and 20 mg/L, respectively, with the

adsorption percentage being more than 90%. At higher concentrations, the numbers of Fe(II) ion are relatively higher compared to availability of adsorption sites. Therefore, the adsorption is constant or decreases with increasing in initial concentration.²²

Conclusions

This work prepared silica from husks of glutinous and jasmine rice by refluxing in hydrochloric acid and heating at 600 °C for 6 hours with characterization by XRD XRF and SEM. The XRD pattern confirmed the amorphous silica phase for both extracted silica with high purity of nearly 100%. SEM images of prepared silica showed a polygonal shape with rough surface and porosity. After Fe(II) adsorption, the morphology of extracted silica were not changed. The adsorption batch method was employed to determine the effect of each adsorption parameter including pH, contact time, adsorbent dosage and Fe(II) ions concentration. The experiment was carried out at an ambient temperature. The optimum parameters corresponding to the maximum adsorption of Fe(II) ions for silica extracted from glutinous rice husk were pH 6, 30 minute contact time, 0.4 g adsorbent dosage and 30 mg/L Fe(II) concentrations. The silica extracted from jasmine rice husk showed maximum adsorption at pH 6, 20 minute contact time, 0.6 g adsorbent dosage and 20 mg/L Fe(II) concentrations.

Acknowledgements

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การกระเจิงของศักย์ยูกาวาว่าโดยการใช้หลักการควอนตัมเชิงพลวัต

Yukawa scattering treated by the Quantum dynamical principle

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บทคัดย่อ

การกระเจิงโดยศักย์ยูกาวาว่า ถูกยกเป็นกรณีการศึกษา ผ่านการใช้เทคนิคการคำนวณโดยหลักการเชิงพลวัตควอนตัมที่เสนอโดยชวิงเงอร์ ซึ่งเป็นวิธีการที่ขึ้นกับฟังก์ชันกำเนิดที่ถูกแทนที่ด้วยตัวกระทำการแบบฟังก์ชันเชิงอนุพันธ์ จากผลลัพธ์เราได้ลักษณะอสมโทติกของกรีนฟังก์ชันอิสระ ที่สามารถอธิบายลักษณะของการกระเจิงของอนุภาคต่อศักย์ยูกาวา และทำการแปรค่าพารามิเตอร์ของมวลให้มีค่าต่างๆ กัน นอกจากนี้ผลลัพธ์ที่ได้ยังนำไปสู่ค่าแอมพลิจูดของการกระเจิงและค่าภาคตัดขวางของการกระเจิงเชิงอนุพันธ์อันเนื่องมาจากศักย์ยูกาวาว่าอีกด้วย

คำสำคัญ: หลักการควอนตัมเชิงพลวัต การกระเจิงโดยศักย์ยูกาวาว่า ศักย์ระยะสั้น กรีนฟังก์ชัน

Abstract

Yukawa scattering is pedagogically interpreted, by the Schwinger's quantum dynamical principle involving the generating function, which is replaced by a functional differential operation. As for the results, we get the asymptotically free Green function that explains the behavior of the Yukawa potential when the mass parameter is increasing and it can also lead to scattering amplitude and differential cross section respectively.

Keywords: quantum dynamical principle, Yukawa scattering, short range potentials, Green functions.

Introduction

In quantum scattering, we are interested in an interaction between the incident particles and the potential of the target e.g., coulomb potential¹ $V(x)=1/x$ which describes the behavior of particle scattering. Yukawa² presented his study by considering the meson interaction, particle with mass, which eventually was called the Yukawa potential. Experimentally, researchers studied the scattering amplitude to determine these scattered particles. R. Feynman presented a diagram of particle

scattering with the path integral that uses the time-slicing derivation^{3,4}.

Accordingly, in this report, we use the quantum dynamical principle proposed by J. Schwinger⁵⁻⁹ to describe this situation. This method is very useful because it gives us the interested transformation function, also called the propagator. In particular, the Hamiltonian equation of this system involves external sources which generate degrees of freedom^{10,11,12}. The equation is precisely derived from the variation of the transformation

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