

ADSORPTION OF CU (II) HEAVY METAL FROM ACID MINE DRAINAGE USING MODIFIED BENTONITE WITH RISK HUSK ACTIVATED CARBON

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Abstract: Acid mine drainage is a waste product of the mining process that contains several heavy metals, including copper. The purpose of this study was to develop bentonite adsorbent-modified activated carbon from rice husks that will be used to adsorb Cu metal from acid mine drainage. Bentonite was chemically activated with HCl, whereas rice husk was physically and chemically activated in a furnace at 500°C for 2 hours and soaked in HCl. The modification was accomplished by combining activated bentonite and rice husk activated carbon in variations of ratio, which are 1:1, 1:2, 1:3, 1:4, and 1:5. Morphological analysis revealed a surface structure similar to that of coral (sponge). The functional groups identified include the OH, OH₂, Al, SiO, SiOAl, and SiOSi functions. The XRD analysis showed a diffractogram at $2\theta = 19000^\circ$ and 20000° that indicates the presence of montmorillonite minerals. The Cu metal adsorption capacity of rice husk-modified bentonite was 3.153 mg/g. The adsorption isotherm obtained in this study is the Freundlich isotherm, with an R² value of 0.9541. These findings indicate the potential usefulness of the modified adsorbent in mitigating the environmental impact of the industry dealing with acid mine drainage.

Keywords: Acid mine drainage; Adsorption; Bentonite

Abstrak: Air asam tambang merupakan limbah hasil proses penambangan yang mengandung beberapa logam berat, diantaranya tembaga. Tujuan penelitian ini adalah mengembangkan karbon aktif termodifikasi adsorben bentonit dari sekam padi yang akan digunakan untuk mengadsorpsi logam Cu dari air asam tambang. Bentonit diaktivasi secara kimia dengan HCl, sedangkan sekam padi diaktivasi secara fisik dan kimia dengan tanur pada suhu 500°C selama 2 jam dan direndam dalam HCl. Modifikasi dilakukan dengan menggabungkan bentonit aktif dan karbon aktif sekam padi dengan variasi perbandingan 1:1, 1:2, 1:3, 1:4, dan 1:5. Analisis morfologi mengungkapkan struktur permukaan yang mirip dengan karang (spons). Gugus fungsi yang teridentifikasi meliputi fungsi OH, OH₂, Al, SiO, SiOAl, dan SiOSi. Analisis XRD menunjukkan difraktogram pada $2\theta = 19000^\circ$ dan 20000° yang menunjukkan adanya mineral montmorillonit. Kapasitas adsorpsi logam Cu pada bentonit termodifikasi sekam padi adalah 3,153 mg/g. Isoterm adsorpsi yang diperoleh pada penelitian ini adalah isoterm Freundlich, dengan nilai R² sebesar 0,9541. Temuan-temuan ini menunjukkan potensi

kegunaan adsorben yang dimodifikasi dalam mengurangi dampak lingkungan bagi industri yang berurusan dengan limbah air tambang asam.

Kata kunci: Air asam tambang; Adsorpsi; Bentonit

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Introduction

Mining activities in Indonesia cause acid mine drainage (AMD), which is extremely hazardous to the environment. AMD occurs when sulfide-containing materials are exposed to oxygen and water, resulting in the formation of sulfuric acid and the release of metal ions. AMD contains zinc (Zn), copper (Cu), manganese (Mn), and iron (Fe) (Hidayat et al., 2021). High acidity and increased trace metal concentrations in AMD can cause changes in water characteristics that disrupt aquatic ecosystems, and these heavy metals will accumulate in living organisms, causing serious consequences for human health and environmental resources (Karami et al., 2019). Acid mine drainage is difficult to prevent in open pit mines, particularly those with a high percentage of potentially acid-forming (PAF) rock. AMD has the potential to pollute the environment, corrode mining equipment, and reduce water quality. Untreated AMD disposal into bodies of water can cause significant harm to aquatic flora and fauna. In order to reduce the negative impact, a pretreatment procedure is required to remove the heavy metals present in AMD (Zhan et al., 2019).

Methods for reducing metal levels in acid mine drainage typically involve ion exchange (Maslova et al., 2020), metal deposition, remediation (Luo et al., 2020), bioremediation (Singh & Chakraborty, 2021), polymer membranes (Zawierucha et al., 2020), sulfur reduction (Sun et al., 2020), and adsorption (El-Nagar et al., 2020). The adsorption method is one of the most commonly used. This method is also low-cost, making it suitable for a broader range of applications. Another advantage of this technique is that it is quick and simple, efficient, readily available, simple to operate, and requires little maintenance and operation, saving money. This method is also popular because the adsorbent used in the process of adsorption can be regenerated and reused (Ntwampe & Moothi, 2018).

Natural materials that are easily obtained in Indonesia, such as bentonite, can be used as adsorbents in a variety of ways. Bentonite is a layered clay or pseudo-clay mineral with strong adsorptive properties (Irdhawati et al., 2021). Bentonite has a high specific surface area of 800 m²/g, allowing it an effective adsorbent for the removal of heavy metals (Chang et al., 2020). Pretreatments such as calcination, chemical and physical activation, or modification with activated carbon can

increase bentonite's adsorption capacity (Ouakouak et al., 2020). This modification is intended to improve bentonite's performance in detecting samples at very low levels and to increase its adsorption in adsorbing waste. Furthermore, this modification was performed to eliminate bentonite's weakness, which makes it difficult to recover clay particles from aqueous media after adsorption.

Due to its large surface area, microporous structure, and high adsorption capacity, activated carbon is the most extensively used adsorbent on a large scale, but its use is limited due to its high cost. The usage of activated carbon obtained from low-cost raw materials is intended to minimize the total cost of achieving optimal outcomes. Adsorbents made from abundant natural biomass materials, such as agricultural waste, specifically rice husk, have the potential to be cost-effective. When activated carbon has reached the end of its lifespan, it can be profitably regenerated (Suhendrayatna et al., 2019).

The aim of the study, as stated above, was to identify the properties of modified adsorbents between bentonite and rice husk. A scanning electron microscope (SEM) was used to examine the adsorbent's surface structure, FTIR was used to examine functional groups and X-ray diffraction was used to examine the diffusion pattern (XRD). The effect of the modification ratio of rice husk activated carbon and activated bentonite on the efficiency and adsorption capacity of Cu heavy metal was also investigated. Cu metal was chosen due to its high concentration in AMD waste. This paper also develops an isotherm model for the adsorption of Cu metal using a modified adsorbent.

Materials and Methods

Materials

The primary materials used in this study were bentonite from the Aceh Utara District and rice husks from a rice mill in Bireuen (Figure 1). Hydrochloric acid (HCl, 37%, Merck, Germany) was used to activate activated carbon and copper (II) nitrate ($\text{Cu}(\text{NO}_3)_2$, Merck, Germany) was used as an artificial solution for Cu metal.

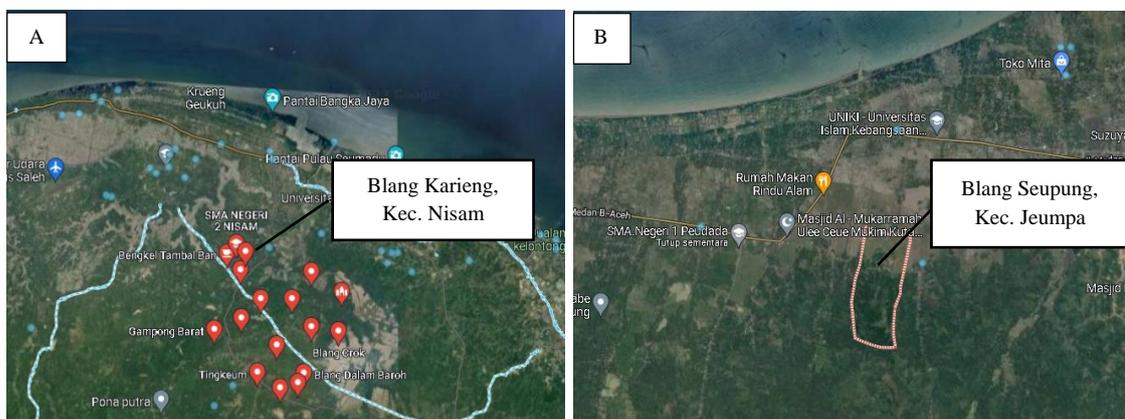


Figure 1. Maps of where to find (a) rice husk and (b) bentonite

Methods

Activation of Bentonite

Firstly, bentonite was ground into a 100-mesh size. Furthermore, bentonite was mixed with 0.5 M HCl and heated at 70°C for 2 hours while being stirred with a magnetic stirrer. After that, the bentonite was filtered and washed with water to achieve a neutral pH. Activated bentonite was dried for 4 hours at 105°C.

Rice Husk Activated Carbon

The rice husks were washed in a container filled with 90°C distilled water. The washed rice husks were then dried in an oven set to 100°C. The rice husk was physically activated in the furnace for 2 hours at 500°C via pyrolysis. The activated carbon that resulted was milled with a mortar and alu, then sieved to a mesh size of 100. The chemical activation process was carried out for 24 hours in 0.3 M HCl.

Modified Bentonite with Rice Husk Activated Carbon

Bentonite and rice husk activated carbon were modified by mixing according to the mass ratio (bentonite: rice husk activated carbon) 1:1, 1:2, 1:3, 1:4, 1:5, and then grinding thoroughly with mortar and Alu.

Characterization of Adsorbents

SEM analysis (JEOL JSM-IT 200) is used to determine the adsorbent's surface morphology. Three samples (bentonite, modified bentonite: rice husk 1:1, and bentonite) were dried in an oven. The sample is then prepared to form a conductive layer that can be read by an electron beam. The SEM was performed at a magnification of 20,000X. On the same sample, FTIR analysis (PerkinElmer spectrum 100 FT-IR) was performed; the sample was placed above the objective, and a reading was taken at a wavelength of 400-4000 cm⁻¹. In addition, XRD was used to analyze diffraction patterns (SHIMADZU XRD-7000). The data was collected at an angle of 2θ between 100,000-800,000°.

Adsorption efficiency and capacity

Cu solutions with initial concentrations of 11.758, 20.149, 33.091, 49.309, and 71.721 mg/l were prepared. In the adsorption process, 1 gram of adsorbent mass was taken for each sample, which was then contacted with 50 ml of Cu solution that had been prepared. Adsorption time was carried out for 60 minutes under magnetic stirring at 180 rpm at room temperature. The concentration of Cu after adsorption was then determined using atomic adsorption spectrometry (AAS). Equations 1 and 2 were used to calculate efficiency (E, %) and adsorption capacity (q_e, mmol/g).

$$E = \frac{C_i - C_e}{C_i} 100\% \dots\dots\dots(1)$$

$$q_e = \frac{C_i - C_e}{gr} V \dots\dots\dots(2)$$

Where C_i and C_e (mmol/L) represent the initial and filtrate concentrations, V (L) is the volume of the adsorbed solution, and gr (g) is the mass of the adsorbent used.

Isotherms Model

An enhanced understanding of the adsorption mechanism, as well as evidence of the surface characteristics, compatibility, and adsorption capacity of the adsorbent, can be obtained by the use of adsorption isotherms (de Castro et al., 2018). The results of the experiment on adsorption were put through a series of experiments to identify the adsorption isotherm model. This was done by constructing a graph using the Langmuir and Freundlich isotherm models, respectively. In order to carry out the Langmuir isotherm, a logarithmic curve is formed that extends from C_e/Q_e to C_e (Equation 3).

$$\frac{C_e}{q_e} = \frac{1}{k_l q_m} + \left(\frac{1}{q_m}\right) C_e \dots \dots \dots (3)$$

$$\log q = N \log C_e + \log K_F \dots \dots \dots (4)$$

The Freundlich isotherm pattern was evaluated by constructing a $\log Q_e$ curve against a $\log C_e$ curve, and the adsorption pattern was determined by comparing the degree of linearity of the curve, as indicated by the value of R^2 . The Freundlich isotherm's linearization form is written mathematically in Equation 4.

Results and Discussion

Morphology

SEM uses a high-energy electron beam to observe the morphology of sample surfaces at high magnification. Figure 2 shows SEM test results for rice husk activated carbon, bentonite, and a 1:1 ratio (rice husk activated carbon to bentonite). The results of the SEM test on rice husk-activated carbon are shown in Fig. 2a, the structure is sponge-like. Point A represents the sample pores, whereas point B represents silica, which still covers the sample pores. In Figure 2a, rice husk activated carbon has open pores and some silica that has not been released, whereas, in Figure 2b, bentonite has an open pore surface structure with a larger size and shape than in Figure 2a; this illustrates that each of these samples underwent physical activation in which the nanocarbon material evaporated, which was quite effective at forming and widening pores (Febryanti et al., 2020). Figure 2c is comparable to Figure 2b due to the similarity in the shape of the pores. This occurs because bentonite has a greater activation capacity than rice husk activated carbon. In addition, sample activation has an effect on breaking hydrocarbon bonds or oxidizing surface molecules, resulting in a change in the carbon surface's size and an increase in its adsorption capacity. The activator also functions to open the pores

of the adsorbent and release the matrix that clogs the pores of the adsorbent, allowing for a more efficient adsorption process (Mentari et al., 2018).

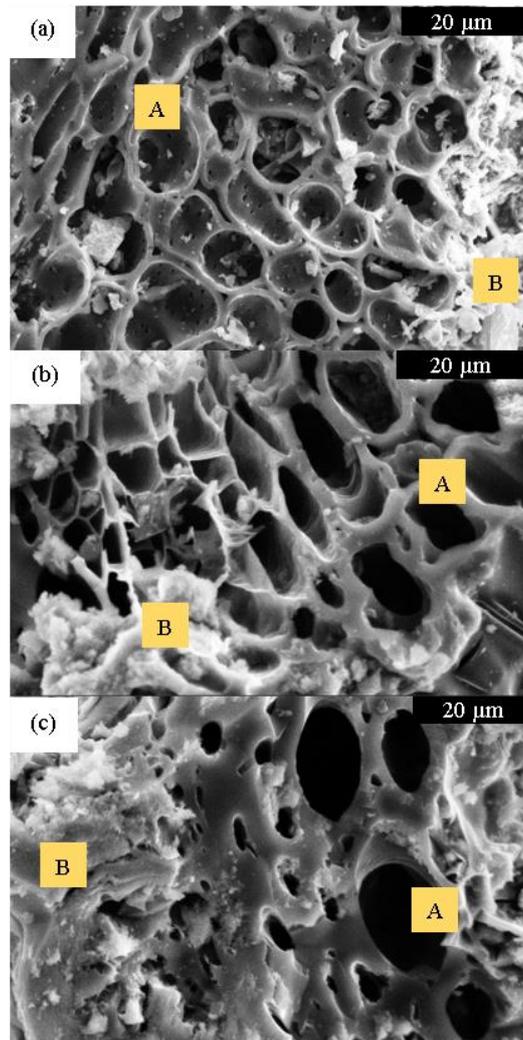


Figure 2. The pore structure of the adsorbent (a) rice husk activated carbon, (b) activated bentonite, (c) modified bentonite with a ratio of 1:1

Functional Group

FTIR analysis is to determine the differences in the functional group's characteristics of the three samples. Figure 3 shows that the peak rice husk sample has an O-H stretching vibration at a wavelength of 3408 cm^{-1} , an aliphatic C-H stretching vibration at 2930 cm^{-1} , a SiO_4 tetrahedral vibration at 1091 cm^{-1} , and a SiO_4 tetrahedral vibration at 815 cm^{-1} . The spectrum then shows a clear difference, with a sharp peak disappearing at wave numbers around 1600 cm^{-1} and a sharp peak with low intensity appearing at wave numbers around 800 cm^{-1} . This demonstrates that the carbonyl group has vanished and Si-O-Si groups have formed as a result of

the condensation of Si-OH, which contains high levels of fixed carbon but low levels of silica (Solihudin et al., 2015). The FTIR spectrum of the bentonite samples and the 1:1 ratio are the same. This is due to the fact that bentonite is more dominant than rice husk, as evidenced by the SEM test results (Figure 2), which have the same morphological structure.

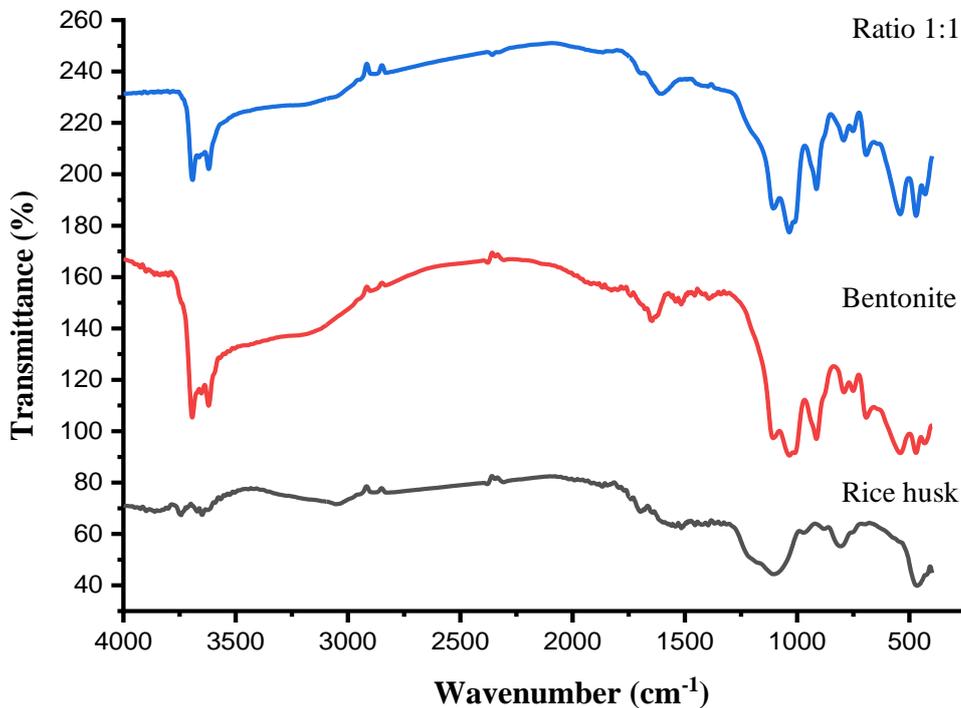


Figure 3. FTIR Spectra of Various Adsorbent

Diffraction Pattern

X-ray diffraction (XRD) is an analytical method for determining the pattern of crystalline materials and obtaining information on their monolayers. The XRD test was used in this study to obtain the diffraction pattern of each sample tested, which was then analyzed to determine what types of compounds were present in the sample. According to Figure 4, the three samples have different diffraction patterns, with rice husk activated carbon having an amorphous diffraction pattern. This is demonstrated by the highest peak in the diffractogram in the range $2\theta = 22000^\circ$, which is typical of amorphous silica. This is consistent with previous findings that rice husk silica has an amorphous structure (Sapei et al., 2012). The bentonite and the 1:1 ratio have an identical diffraction pattern, but the 1:1 ratio tends to form the bentonite diffraction pattern. The peak range of the bentonite diffractogram is $2\theta = 19000^\circ$, while the 1:1 is 20000° . The presence of montmorillonite minerals is indicated by this peak (Fauziyati, 2019). The bentonite and the ratio 1:1 diffractogram at $2\theta = 28000^\circ$, then identified compounds containing aluminium, silica, and oxygen. This corresponds to the properties of

bentonite, which is composed of montmorillonite compounds as well as compounds containing aluminium, silica, and oxygen.

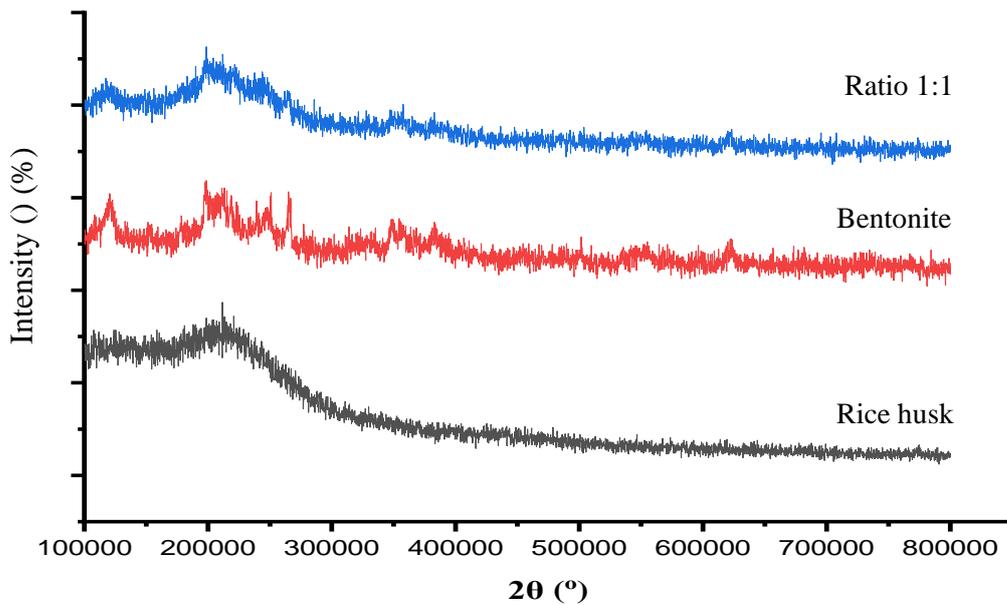


Figure 4. X-RD Pattern

The Effect of Ratio (bentonite: risk husk) on Adsorption Capacity

The effect of optimum adsorbent mass ratio on adsorption capacity will then be determined at a Cu concentration of 71.721 mg/L. According to Figure 5, the variation in mass ratio has a significant impact on the increase in the adsorption capacity of Cu metal, with the highest adsorption capacity obtained at a mass ratio of 1:5, which is equal to 3.261 mg/g. At mass ratios of adsorbents of 1:1, 1:2, 1:3, and 1:4, the adsorption capacities are 3.153, 3.161, 3.194, and 3.255 mg/g, respectively. It can also be seen that the increase in adsorption capacity is not stable in the 1:1 and 1:2 ratios, or that the distances are still close together. This can occur because the mass ratio of the adsorbent used is not optimal, or it can occur due to a less homogeneous mixing process between bentonite and rice husks. However, at adsorption mass ratios of 1:3, 1:4, and 1:5, it appears that a significant increase in adsorption capacity has occurred. This occurs because the adsorption process achieves the optimal conditions for maximum Cu metal adsorption (Astari et al., 2018).

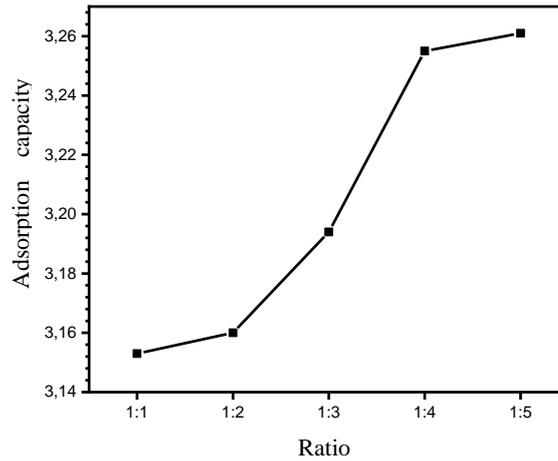


Figure 5. Correlation between adsorbent mass ratio with adsorption capacity

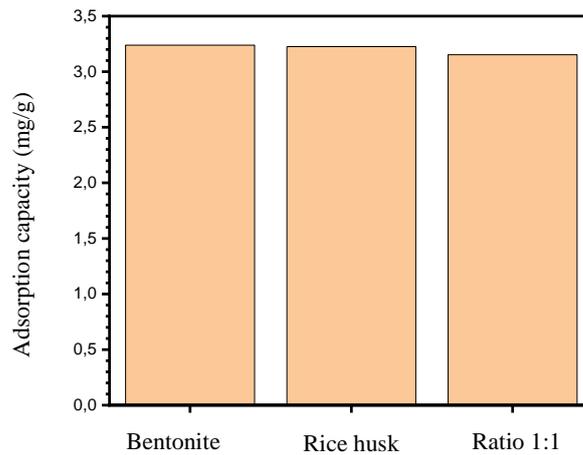


Figure 6. The adsorption capacity of each adsorbent

Figure 6 shows the adsorption capacity of each adsorbent. Bentonite's ability to bind Cu(II) ions results in the greatest capacity gain. The activation procedure has cleared the bentonite pores of water molecules and metal oxides, both of which are regarded as contaminants, hence increasing bentonite's adsorption capacity. The empty voids created on the surface of bentonite can extend the active surface, and significantly improve its adsorption capacity (Fauziyati, 2019). The results of the bentonite surface area analysis in Figure 1 support this. However, when compared to activated bentonite and rice husk activated carbon, the adsorption capacity of the modified adsorbent had no significant effect.

The Effect of Cu(II) Concentration on Adsorption Efficiency

Figure 7 shows the correlation between Cu Concentration and adsorption Efficiency in a 1:1 (bentonite: risk husk) adsorbent mass ratio with a 60-minute

adsorption time. Furthermore, the adsorption efficiency of each adsorbent is shown in Figure 8. Concentration is a composition that clearly demonstrates the ratio of solute. To determine the optimum Cu concentration that could be absorbed by modified bentonite and rice husk adsorbents at a 1:1 ratio, the effect of Cu solution concentration on adsorption efficiency was optimized. As can be seen in Figure 7, the adsorption efficiency increases as the Cu concentration increases. The highest adsorption efficiency was obtained at a concentration of 71.721 mg/L, which was 87.935%, while successive adsorption efficiencies of 38.288; 65,963; 81,318; and 87.572% were obtained at concentrations of 11.758; 20.149; 33,091; and 49.309 mg/L. This increase in adsorption efficiency is due to the fact that the adsorbent absorbs more Cu ions when there are more Cu ions in the solution.

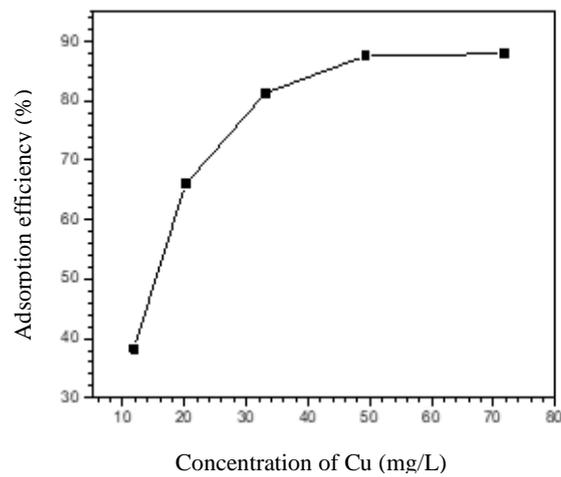


Figure 7. Correlation between Cu concentration and adsorption efficiency

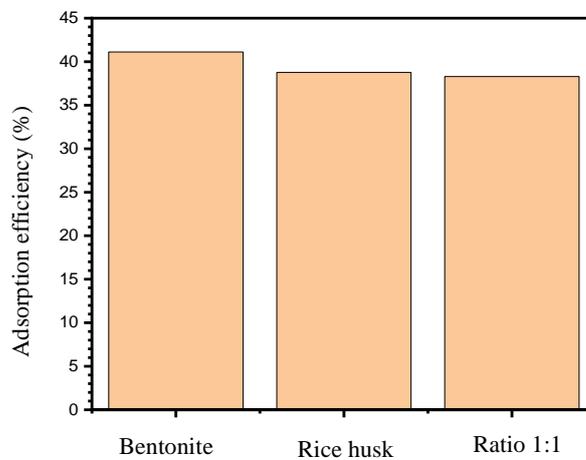


Figure 8. Adsorption efficiency of each adsorbent

As a result in Figure 8, the highest adsorption efficiency was obtained in the bentonite sample, which was 41.12%; it was 38.75% in the activated carbon sample; and 38.288% at a 1:1 ratio. Because of the large number of Cu ions that can be absorbed, bentonite has the highest efficiency gain. This is supported by the results of the SEM test (Figure 2), which provide information about the structure of the cell surface, which has larger cell pores where the silica content in bentonite is decomposed, resulting in higher efficiency than activated carbon and a 1:1 ratio.

Adsorption Isotherm Model

The Freundlich and Langmuir isotherms are the most common types of adsorption isotherms that can be used to study the adsorption mechanism of the liquid-solid phase. The goal of determining this isotherm is to determine whether the adsorption mechanism that occurs in modified bentonite and rice husk adsorbents for the adsorption of Cu metal is chemical or physical. Because the adsorption process is influenced by several factors, including the size of the adsorbate molecule, the polarity of the adsorbate substance, temperature, adsorbent size, adsorbent purity, and contact time, each adsorbent has a unique adsorption isotherm pattern (Cheremisinoff and Ellerbusch, 1978). Following that, the chemical and physical adsorption is determined by determining the Langmuir isotherm and the Freundlich isotherm.

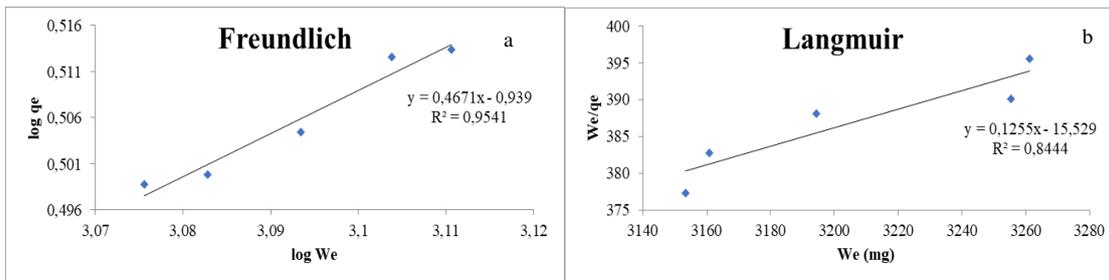


Figure 9. Adsorption isotherm model (a) Freundlich and (b) Langmuir

According to Figure 9(a) and (b), the Freundlich model yielded the highest value of the determinant coefficient (R^2), which is 0.9541, while the Langmuir model is 0.8444, indicating that the Freundlich adsorption isotherm model is used in this study. This indicates that there are active sites on the adsorbent's surface that can adsorb a single molecule. As a result, the modified bentonite and activated charcoal samples are diverse. The adsorbed substance will increase rapidly as the concentration of the solution increases in the Freundlich model isotherm adsorption and then slow down if the surface of the adsorbent, modified bentonite with activated carbon, is covered. The adsorbent-adsorbate bond can form both physically and chemically. The bond must be strong enough to keep the molecules that have been adsorbed along the surface in place. The active sites in the Langmuir

isotherm are found on the Langmuir adsorption surface. It is assumed that there are active sites on the adsorbent surface, the number of which is proportional to the adsorbent's surface area.

Conclusion

In this study, activated bentonite and rice husk activated carbon were successfully modified in a 1:1 ratio. SEM analysis reveals that the surface structure is similar to coral (sponge) and has a larger pore when compared to rice husks and modified bentonite (1:1). FTIR analysis of adsorbent functional groups revealed the presence of adsorption groups in rice husk adsorbents O-H, C-H, SiO₄, and Si-O-Si, while adsorption groups in bentonite and ratio (1:1) were OH, OH₂, Al, SiO, SiOAl, and SiOSi. The characteristics of the diffractogram on rice husk activated carbon revealed the presence of amorphous silica, whereas the diffractogram on bentonite and a ratio of 1:1 revealed the presence of montmorillonite minerals. The adsorption efficiency of modified bentonite in rice husk increases as Cu concentration increases. The adsorption capacity of the modified adsorbent did not differ significantly from that of activated bentonite and rice husk activated carbon. The adsorption isotherm obtained in this study is the Freundlich isotherm, with an R² value of 0.9541. The bentonite adsorbent produced the best results, with a removal efficiency of more than 40%, while the modified adsorbent had no effect. The adsorbent that was created can be used to remove Cu(II) metal from acid mine drainage.

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