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A NOVEL APPROACH OF USING BAMBOO ROOT CELLULOSE: AN ALTERNATIVE FOR IRON(II) REMOVAL FROM WASTEWATER

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ABSTRACT

A novel approach was taken using the bamboo root transformed into activated cellulose, implying dilute nitric acid as the agent activator. The activated bio-sorbent was applied for Iron(II) (Fe(II)) adsorption in batch mode. Then, bio-sorbent characterization was established using infrared spectroscopy and a scanning electron microscope (SEM). The effect of acid variation, iodine number, contact time, particle size, temperature, and concentration on the Fe(II) adsorption was determined in the batch method. The Freundlich and Langmuir isotherm were analyzed for determining the isotherm of adsorption. The Langmuir model of adsorption is the most suitable for illustrating Fe(II) adsorption. The optimum adsorption capacity occurred at the bio-sorbent particle size of 50 µm, contact time of 300 minutes, a temperature of 50˚C, a pH of 5, and 150 ppm. The bamboo roots have been applied to remove Fe(II) from wastewater, and the recovery% shows 100%. Comparative studies were analyzed with some bio-sorbents for further development. It is hoped that this novel alternative can be applied as bamboo is abundant in Indonesia and many countries in Asia regions.

Keywords: Adsorption, Bamboo Root, Bio-Sorbent, Cellulose, Iron(II).

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INTRODUCTION

Iron (Fe(II)) is the industry's most widely used metal among all metals.[1] Iron is needed in the body to form hemoglobin and it is necessary for plant metabolism.[2] Besides its benefit, iron also has disadvantages to the environment if the concentration is excess in water.[3] Iron (Fe(II)) causes a yellow color in drinking water, deposition in pipes, growth of bacteria, and turbidity. For several decades along with the development of industrial activities, Fe(II) contamination of the environment with wastewater, including heavy metals, has become a major problem. Odor in drinking water and block pipes because of the precipitation of Fe(II) hydroxide[4]. It can also accumulate in the alveoli and cause lung dysfunction.[5,6] So, Fe(II) contained in the wastewater must be removed before being disposed of into the river. Many efforts have been made to decrease metal concentration in industrial wastewater. Conventional methods include extraction, ion exchange[7], electrocoagulation[8], membrane filtration[9], adsorption, and electrochemical treatment.[10] The advantage of the adsorption method compared to other methods is that the process is simple, does not produce sludge, and is relatively inexpensive. One of the methods is adsorption using activated carbon, but this method is quite expensive. Various research has been conducted to remove Fe(II) using crab shell[11], wooden charcoal[12], and kapok banana peel[13] but their adsorption capacity was still low. The bamboo root that contains cellulose, has a high abundance in nature and is relatively cheap. Previous studies used cellulose as a bio-sorbent for heavy metals using lemongrass leaves[14] and coconut shells.[15] The cellulose of bamboo root content of fiber is about 62.67%.[16] Therefore, the bamboo root produced in Indonesia is chosen in this study. Cellulose, specially produced by plants and trees, is a natural polymer that is easy to biodegradation and is an eco-friendly resource. The cellulose molecules have many active groups of hydroxyls. Therefore, it can be chemically modified based on introducing various functional groups on hydroxyl. The absorption mechanism occurs due to the exchange of ions between the hydroxy groups of the cellulose molecules and the Fe^{2+} ions from the metal surface.[17] Cellulose has been proven to be an effective and simple method to remove Fe(II) ions using ion exchange techniques.[18] Previous studies have carried out adsorption from bamboo roots to absorb copper and zinc metals.[19] Based on the explanation above, bamboo roots have the potential to be applied as heavy metal bio-sorbents. Therefore, in this study, activated bamboo roots were developed to absorb Fe(II) metal ions in the batch process and then it was applied to the wastewater sample. The effects of different parameters were studied, including particle size, contact time, initial Fe(II) concentration, pH, and temperature. The results were applied for removing Fe(II) in wastewater.

EXPERIMENTAL

Material and Methods

The apparatus used in this study include glassware commonly used in chemistry laboratories. The materials used are Indonesian bamboo root, $(NH_4)_2FeSO_4.6H_2O$ (Merck), HCl (Merck), HNO_3 (Merck), H_3PO_4 (Merck), H_2SO_4 (Merck), $HClO_4$, $(NH_4)_2FeSO_4.6H_2O$ (Merck), I_2 (Merck), KI (Merck), $Na_2S_2O_3.5H_2O$ (Merck), $K_2Cr_2O_7$ (Merck) and starch (Merck) and water samples. The instrumentations used in this study were UV-Vis Spectrophotometer (Agilent 8453), a Prestige-21-Shimadzu Fourier Transform Infra-Red, and a Scanning Electron Microscope (SU3500). Atomic Absorption Spectrometer (GBC Scientific–Avanta).

General Procedure and Detection Method

Bio-sorbent Activation

The bamboo roots were washed and brittle in the oven at 70°C overnight in the first stage. After that, the seeds were grounded. After that, these materials were activated in each of the following various acids in the same 0.3 M concentration: HNO_3, HCl, H_3PO_4, H_2SO_4, and $HClO_4$ for 24 hours. After the activation, the distilled water was used to remove the ash until pH 7; the activated bio-sorbent was brittle in the oven for 24 hours and 60^0C. The size of particles (50, 75, 150, and 355 µm) was obtained by sieving and put in a desiccator for the subsequent study.

Adsorption Studies

The adsorption was studied by batch method. A specific volume of Fe(II) solutions with different initial concentrations was immersed into a certain amount of bamboo roots. The parameter studied includes contact time and temperatures. After equilibration, the mixture was centrifuged and analyzed using Atomic Absorption Spectrometer (AAS). The obtained data from the batch method was used to study the equilibrium Fe(II) number with equation (1).

$$q_t = \frac{V}{m}(C_o - C_t) \quad (1)$$

Where qt (mg/g) is the Fe(II) adsorbed per unit weight of bamboo root, V (L) is the solution volume, Ct (mg/L) is the concentration of Fe(II), Co (mg/L) is the initial Fe(II) concentration and m is the mass of bamboo root used.

Determination of Iodine Number

Several bamboo root biomass was immersed in 0.1 N iodine solution with a stirring time of 15 minutes. After the filtered, 10 mL filtrate was pipetted into an Erlenmeyer then it was titrated with sodium thiosulfate, which had been standardized first with potassium dichromate.

$$\text{Iodine Number} = \frac{V\,x\,N\,x\,12.69}{W} \quad (2)$$

Where,
V: volume of titrant (mL)
N: concentration of sodium thiosulphate (N)
W: activated cellulose mass (g)

Isotherm of Adsorption Studies

10 mg of bio-sorbent stirred with five mg/L of Fe(II) solutions 5–50 mg/L. The calculation of adsorption isotherms was then applied.

Activated Bio-sorbent Characterization

The activated cellulose of bamboo root (activated bio-sorbent) was heated at 60 °C for 24 hours and then was added sufficiently KBr and put under pressure to form pellets. Samples are ready to be analyzed using Fourier Transform Infra-Red (FTIR) with a transmittance between 400 and 4000 cm^{-1} wavelengths. The peaks were achieved in a scanning time of fewer than 30 seconds. While the surface morphological analysis was performed using SEM. Samples do not need to be pre-prepared, and the magnification was about 2500x.

Application To the Environmental Samples

The first step to measure the recovery% is the measurement of the calibration curve and then plotting the linearity. The solution of Fe(II) with various concentrations of 1, 2, 3, 4, and 5 ppm was measured using AAS. A calibration curve is made as concentration versus absorbance. For the sample, 0.5 mL of a standard solution of Fe(II) 150 ppm was put in a volumetric flask of 25 mL and then diluted in a wastewater sample. Three samples were then analyzed using AAS at 248 nm. The absorbance resulting from the sample measurement was too high, so the dilution step was applied. The calculation of the percent recovery is obtained by using equation 6:

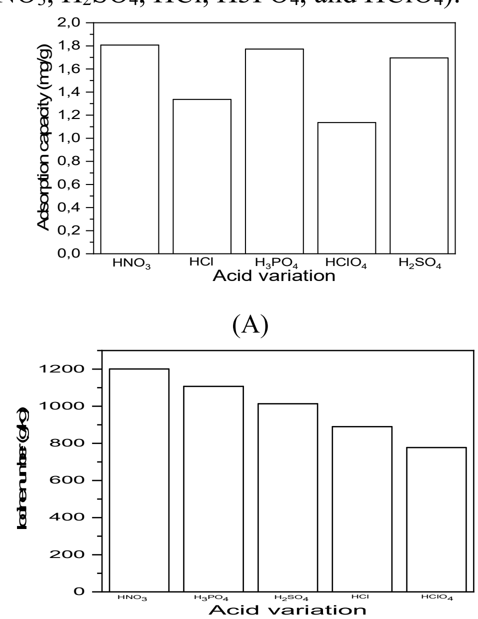
$$\text{Recovery } \% = \frac{[Fe^{2+}]_{\text{final}}}{[Fe^{2+}]_{\text{initial}}} \quad (3)$$

RESULTS AND DISCUSSION

The application of bamboo root to adsorb Fe metal has been carried out. Its effectiveness and absorption mechanism have been studied with several parameters, which will be explained in this section.

Acid Selection for Bamboo Root Cellulose

Bamboo root as a bio-sorbent was used in this research because it contains more cellulose than other biosorbents (mentioned in the introduction chapter). The higher amount of cellulose is expected to increase its adsorption capacity. The adsorption capacity of metal ions by activated bio-sorbents is greater than that of non-activated biosorbents. In this study, the activation was carried out by soaking the biosorbent of bamboo root using several acids (HNO_3, H_2SO_4, HCl, H3PO4, and $HClO_4$).



(B)

Fig.-1: The Acid Selection (A) and Determination of Iodine Number (B)

Acid can react with elements in bamboo to produce different reactions and active sites. As described in Fig.-1A, the highest absorption is obtained from the biosorbent activated using HNO_3, shown by the adsorption capacity of 1.8407 mg/g. Based on the level of acidity, the acids used can be sorted as follows: $HClO_4$ < HCl < H_2SO_4 < H_3PO_4 < HNO_3. The acids mentioned above are oxidizing acids, except for HCl.

Oxidizing acids can form oxides on the surface of roots which can increase their hydrophilic.[20] A previous study stated that HNO_3 increased the concentration of oxygen groups on the surface which caused it to easily react with Fe(II). Determination of the iodine number is studied to drive the level of activity of the biosorbent. An iodine number is a number that shows the amount of iodine adsorbed on the biosorbent. The higher the activity level, the higher the iodine number.[21] It was done by an analysis of the iodine value of the biosorbent with particle size < 75 was carried out with different acids, namely: HNO_3, H_2SO_4, HCl, H_3PO_4, and $HClO_4$. The analysis carried out obtained data that is in Fig.-1B. From Fig.-1B, it was found that cellulose that was activated with HNO_3 has the highest iodine number among the others, with the amount of 1200.66 g/kg. This shows that HNO_3 has the best acid for biosorbents. In the previous study, such results were obtained[22] on activated bamboo, with HNO_3 having the highest iodine number among H_2SO_4, HCl, $ZnCl_2$, H_3PO_4, and NaOH, which was 1198 g/kg. It can be concluded that HNO_3 was the best activator to activate the carbon from bamboo because it produced many active sites, and a chemisorption reaction occurred in the carbon pores during activation.

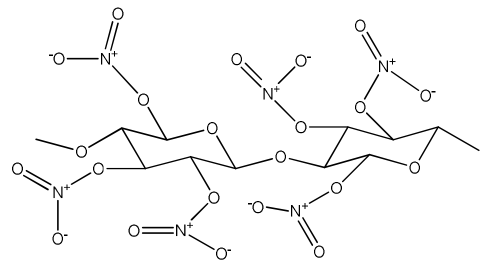


Fig.-2: The Reaction of Cellulose with Nitric Acid

The reaction of HNO_3 with cellulose produces cellulose nitrate (nitrocellulose), which is formed during the activation of cellulose with nitric acid to produce an active reaction site for the metal ion adsorption process. The reaction mechanism can be seen in Fig.-2. Each subsequent reaction of the –OH group with the nitrating agent yields nitrocellulose. The interaction between O- bound and nitrogen is probably forming a coordination complex in the existence of a lone pair on the oxygen atom of the group of NO. Complex metal compounds will be formed when a lone pair of electrons occupy the empty orbital of the Fe^{2+} metal ion.

Adsorption Characterization

Infra-red characterization was carried out on the biosorbent before activation, after activation, and after adsorption. The results of the characterization as described in Table-1. From Fig.-3, it can be seen that there was a shift in the wavenumber. At the time before activation and after activation, the -OH group showed a shift in wavenumber 3435 cm^{-1} to 3374 cm^{-1} and there were no big changes in the wavenumber for bio-sorbent after Fe(II) adsorption.

Table-1: Fourier Transform Infra-Red Data

Wavenumber (cm^{-1})			Functional group
A	B	C	
3435	3374	3365	-OH
2868	2870	2879	C-H
1654	1653	1654	$C-NO_2$
1378	1378	1378	C-O

Note: A = before cellulose activation, B = after cellulose activation dan C = after adsorption of Fe(II)

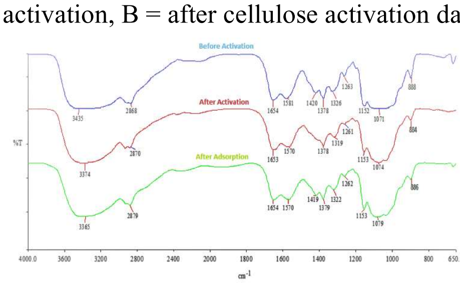
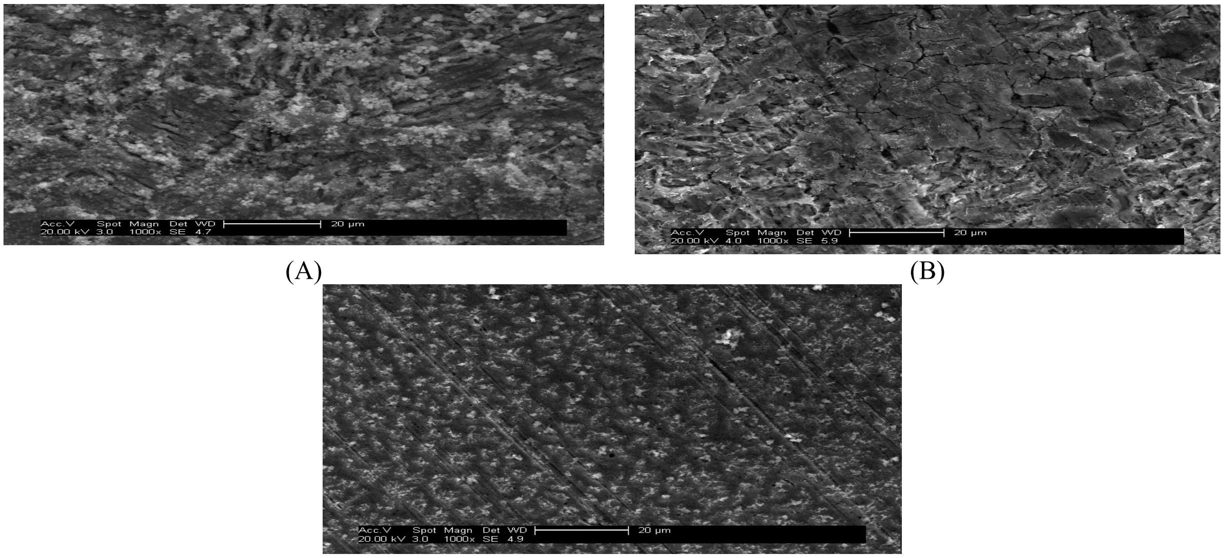


Fig.-3: Infra-Red Spectra of Bamboo Root Cellulose



(A) (B)

(C)

Fig.-4: Scanning Electron Microscope Image of Cellulose (A) Activated Cellulose (B) Adsorption Fe(II) into Activated Cellulose (C)

SEM was studied to see the different surfaces of bio-sorbent and study the surface's morphology. Figure-4 describes pore differences from bio-sorbent before activation, after activation, and after adsorption. It can be seen in the picture of cellulose before surface activation that the surface looked like fiber, and after activation with HNO_3, pores were formed. The pores in the cellulose were then closed again because of the possibility that Fe(II) was being adsorbed.

Adsorption Study

The analysis was carried out with several parameters, including bio-sorbent particle size, acid activation, contact time, concentration, temperature, and pH. These parameters were studied to determine the best conditions for the bio-sorbent adsorption process. The optimum conditions were determined based on the highest adsorption capacity (Qe). These optimum conditions will be applied to the environmental samples later. In the first stage of this research, the particle size effect on the adsorption capacity was studied. Variations in particle sizes of 355, 150, 75, and 50 µm were used. Figure-5A shows a significant decrease in the capacity of adsorption from 1.8477 mg/g to 0.5349 mg/g for bio-sorbent particle sizes of 50 µm to 355 µm. The cellulose of bamboo root can optimally absorb Fe(II) at a particle size of 50 µm. The Fe(II) adsorption increased when particle size was reduced. The smaller particle size will allow a larger bio-sorbent surface area per unit volume so that more Fe(II) will be adsorbed. Based on previous research[22], the particle size of 150 µm showed the highest % adsorption. A particle size of 50 µm will be used for the next experiment.

Adsorption capacity measurement was carried out based on variations in concentration (50, 100, 150, and 250 ppm) which aims to determine the optimum saturation of the bio-sorbent in absorbing Fe(II) ions. The measurement was studied at the optimum particle size. The optimum absorption vs. concentration of Fe(II) can be seen in Fig.-5B. Based on the data obtained, the optimum cellulose bamboo root adsorption capacity was at 150 ppm of Fe(II) concentration with Qe = 1.5634 mg/g. After passing the optimum concentration, the absorption capacity decreases considerably. It could be explained that the bio-sorbent absorption capacity has been saturated. If there was another Fe(II), the capability of the bio-sorbent to absorb was slight. In the end, a desorption process causes the Qe value to decrease.In the end, a desorption process causes the Qe value to decrease. Next, the contact time effect to the adsorption capacity were carried out to obtain the optimum absorption capacity. Variations were made between 60, 180, 300, and 420 minutes. From Fig.-5C, it can be seen that the optimum absorption at 300 minutes was with an absorption capacity value of 2.3768 mg/g, and there was very slight decrease at 420 minutes to 2.3764 mg/g, but this can be assumed as a constant. It was due to the saturated state of the bio-sorbent adsorbing Fe(II) so that when the optimum time passes, desorption occurs, which results in a slight decrease in Fe(II) in the bio-sorbent. Therefore, the optimum time was preferred in optimizing the time variation in a state of 300 minutes.

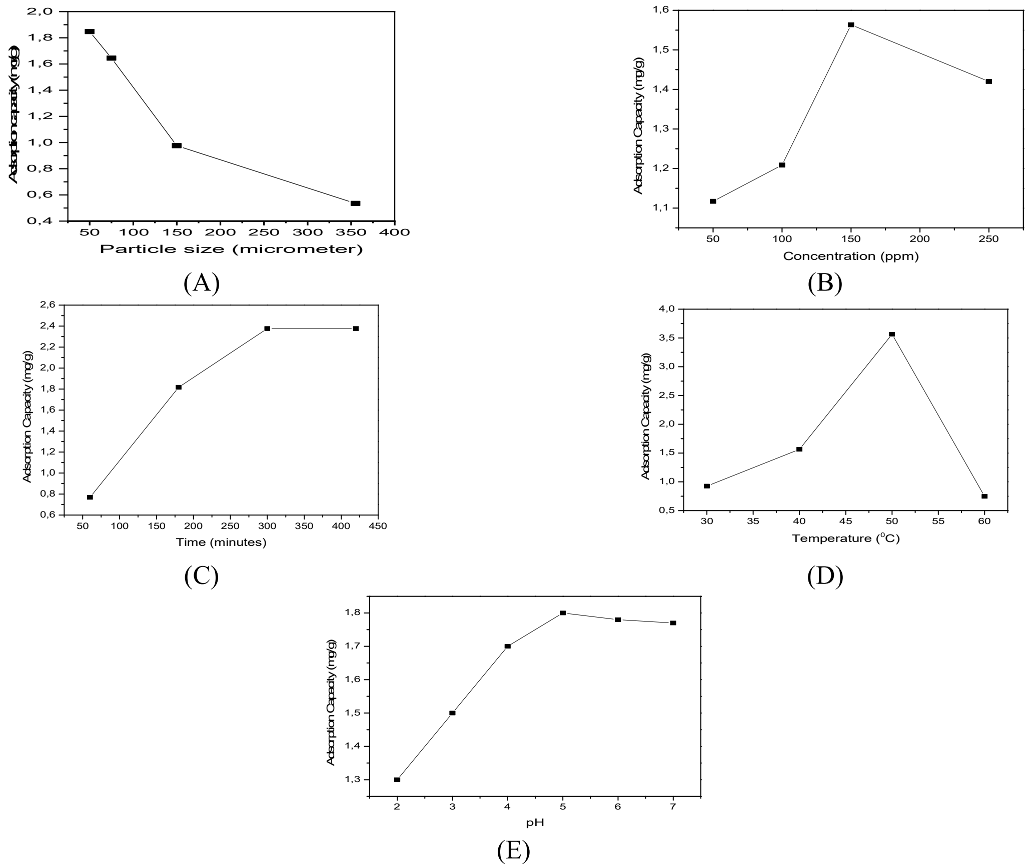


Fig.-5: Adsorption Study Based on Various Parameters: Particle Size (A), Concentration of Fe(II) (B), Contact Time (C), Temperature (D), pH (E)

The previous study[23] showed that the capacity of adsorption was precisely equivalent to the time up to a certain point, then decreased after passing that point. Then, optimization determined the adsorption capacity with the temperature variations. The observed temperatures ranged from 30, 40, 50, to 60°C. In addition to contact time and particle size, the temperature can also affect the work of the bio-sorbent in absorption. From the research that has been done, the data are presented in Fig.-5D. The optimum absorption capacity is at 3.56 mg/g at a temperature of 50°C. Supported research[24], states that the absorption capacity increases with increasing temperature. This was probably related to the more active movement of metal ions due to increasing temperature so transferring the mass of metal ions from the solution to the biosorbent surface becomes more accessible. However, after passing the optimum temperature, the absorption capacity decreases. An increase in the adsorption temperature, the energy for desorption increases so that more metal ions were released from the bio-sorbent compared to the number of metal ions adsorbed in the bio-sorbent. The pH response was investigated in the Fe(II) absorption mechanism towards bamboo root bio-sorbent. Figure-5E shows the pH response to the capacity of adsorption. The pH of Fe(II) was reviewed from 2 until 7. The Fig. shows an increase in adsorption capacity from pH 2 to 5 and the optimum at pH 5. Adsorption seems to decrease starting from pH 6. It can be explained as follows: at acidic pH, Fe(II) will remain in the form of Fe(II), while at alkaline pH will form a precipitate of $Fe(OH)_2$.

Adsorption Isotherm Study

The isotherm of adsorption describes the number of adsorbates adsorbed at a certain temperature. The adsorption isotherm is crucial to elevating and knowing the use of adsorbent as well; the isotherms of adsorption analysis can help to interpreted the bio-sorbent adsorption mechanism (activated cellulose of bamboo root) and adsorbate (Fe(II)). The data of equilibrium was evaluated with a model from Freundlich

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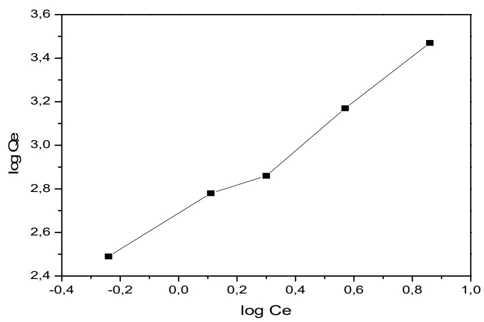
and Langmuir.[25] Based on Langmuir, adsorption happens at homogeneous adsorbent sites, a heterogeneous adsorbent surface attained by Freundlich with the non-uniform spread of the adsorption warmth over the surface.[26] Equation 4 marks out the Freundlich logarithmic form, while Equation 5 shows the Langmuir equation logarithmic form.[27]

$$\text{Log Qe} = 1/n \log \text{Ce} + \log K_F \quad (4)$$

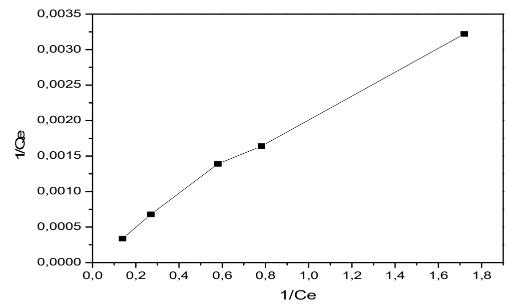
Where Qe (mg/g) is the number of adsorbates when reached equilibrium, Ce is the (Fe(II)) concentration of equilibrium, KF is the constant of Freundlich associated with capacity of adsorption, n is the constant associated with the intensity of adsorption associated with the heterogeneity factor. The plots of log Qe vs. log Ce present a linear graph, and n (slope) and KF (intercept) can be gained from the graph.

$$\frac{Ce}{Qe} = \frac{Ce}{qmax} + \frac{1}{K_{L.}qmax} \quad (5)$$

Where Ce (mg/g) is Fe(II) concentration at equilibrium. K_L is the Langmuir constant related to adsorption capacity (mg/g). KL is matched with the variation of the appropriate region and porosity of the adsorbent. Because of the large pore volume and surface area, adsorption capacity becomes high.



(A)



(B)

Fig.-6: Freundlich Adsorption Isotherm (log Ce vs log qe) and Adsorption Isotherm of Langmuir (1/Ce vs 1/Qe)

From the graph shown in Fig.-6A, B, and Table-2, the linear regression of the Langmuir and the Freundlich isotherm are 0.9919 and 0.9851, respectively. This linearity indicates the Langmuir is suitable for the Fe(II) ion adsorption activated by bamboo roots. The Qm value obtained from the calculation is 5 mg/g. This is also in accordance with the shape of the adsorption capacity curve in Fig.-6 with increasing the initial concentration of Fe(II) to a certain concentration, no longer increases the adsorption capacity because the active sites of NO_3^- are already saturated. The previous study also reported follow Freundlich isotherm adsorption.[28]

Table-2: Isotherms of Adsorption Parameter

Freundlich	Langmuir
R^2:0.9851	R^2:0.9919
K_F:468.27 (μg/g)(L/mg)$^{1/n}$	Q_{max}:5mg/g
1/n:0.8858	B:0.1 L/mg

Table-3: The Adsorption Capacities of Fe(II) with Different Bio-Sorbents

Bio-sorbent	Equilibrium Time (min)	Capacity of Adsorption (mg/g)
Crab shell[11]	840	1.08
Wooden charcoal[5]	180	1.88
Kepok banana peel[6]	30	0.64
Bamboo root (Our study)	300	3.56

While Table-3 shows the relation of maximum capacity and required time of equilibration of different bio-sorbents for removing Fe(II) from the solutions. It shows that the values were diverse for other materials. It depends on the composition of physical and chemical substances. Bamboo root has a relatively short equilibration time and good capacity compared to all bio-sorbents.

Removal Fe(II) from Wastewater

The recovery% was studied by correlating the final measurement of the concentration of the analyte with the introductory concentration (Eq. 3) by performing a spiked analysis of the sample whose concentration was known. A good % recovery value was in the range of 100 ± 5%. Based on the calculations, it described that the recovery% for removal Fe(II) from wastewater was 100%; these results indicate that the method used was suitable for wastewater. Based on all the parameters examined above, it can be understood that bamboo roots have the potential as bio-sorbents of heavy metals. This alternative method can reduce Fe(II) contamination in wastewater.

CONCLUSION

Bamboo roots have been successfully used as bio-sorbent for Fe(II). The optimum condition was achieved at the cellulose particle size of 50 µm. Bamboo root cellulose activated with HNO_3 showed maximum adsorption capacity compared to other acids indicated by the highest iodine number. Analysis using FTIR can prove that in the C-O group the activation and adsorption processes run well, analysis using SEM can provide an overview after the activation process and after the adsorption process. The optimum adsorption capacity occurred at 300 minutes, 50˚C, 150 ppm, and a pH of 5. The recovery% of removal Fe(II) from wastewater was 100%.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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