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The Extracted Pectin from Ambarella Fruit Peel (*Spondias dulcis*) as Biosorbent in Adsorption of Cu(II) Metal Ions

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Abstract

Heavy metals are known to seriously injure land plants and animals, including humans, as well as marine species when present in contaminated water. For the batch adsorption technique used in this study, Cu(II) ions were removed from an aqueous solution using an inexpensive environmentally friendly adsorbent prepared from the extracted pectin of ambarella fruit peel. The functional groups of the extracted pectin were studied using Fourier Transform Infrared Spectroscopy (FT-IR). Operational conditions like pH, contact time, and initial adsorbate concentration were investigated. FTIR characterization showed that pectin was successfully extracted from ambarella fruit peel with the appearance of the peaks at 3331.36 cm^{-1}, 2924.02 cm^{-1}, 1727.38 cm^{-1}, 1626.20 cm^{-1}, 1329.31cm^{-1}, 1232.43cm^{-1}, 994.18 cm^{-1} and these peaks have similar characteristic with a commercial pectin. The maximum uptake of Cu(II) ions was obtained at a pH of 4, a contact time of 90 min, an initial metal concentration of 150 ppm. The extracted pectin has an adsorption capacity of 6.5860 mg/g for the removal of Cu(II) ions from aqueous solution under the optimum adsorption conditions. In addition, the data obtained from contacting the extracted pectin of ambarella fruit peel in Batang Arau River water showed an adsorption capacity of 0.0376 mg/g. Therefore, the pectin that was extracted from the peel of the ambarella fruit could be used to filter out Cu(II) ions from aqueous solutions.

Keywords: *adsorption; Cu(II); pectin; ambarella fruit peel.*

Introduction

The human body needs copper (Cu) to support a number of biological functions, including cellular respiration, iron transfer, enzyme activity, and brain development. Nonetheless, excessive Cu(II) exposure can be harmful to human health[1]. Cu(II) can be obtained from both anthropogenic and natural sources. The main industrial operations that release copper into the environment include those in the fertilizer, mining, battery, electronics, and machinery sectors. As a result, Cu (II)-contaminated water is regularly found in industrial development zones[2].

Currently, there are many methods that can be used to remove heavy metals, such as precipitation[3], coagulation-flocculation[4], ion exchange[3], membrane processes[5], electrochemical techniques[6], adsorption[7], and others. However, there are some downsides to this method, including unpredictability in efficiency, high energy and reagent requirements, a demanding operation stage, a high cost, and other issues. Biosorption offers a low cost, simple, and environmentally friendly

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method. Biosorption is an adsorption process that uses biomass as an adsorbent. It is known as biosorbents. In previous studies, biosorbents have been widely used for the adsorption process of Cu(II) ions such as *Saccharomyces cerevisiae*[8], *Pseudomonas putida*[9], *Microalgae chlorella sp*[10], dragon fruit skin[11], coffee skin[12] as well as pectin from tongka langit banana's crust[13], and pectin from sweet orange peel[14].

Pectin is a polymer compound of D-galacturonic acid found in the cell wall plants connected by 1,4 glycosidic bonds and is abundant in the middle lamella wall cell. Pectin contains carboxylic groups which can bind heavy metal ions to form a complex compound that is insoluble in water and can be used as a heavy metal biosorbent[15]. Pectin from ambarella fruit peel has been used as a biosorbent for Cr(VI) ion[16]. But there hasn't been a single report on the use of ambarella fruit peel to adsorb Cu(II) ions among the studies that have been published (*Spondias dulcis*). Ambarella fruit peel have a high pectin content which has the potential to adsorb Cu(II) ions, making them useful as biosorbents. Another advantage from ambarella fruit peel which is easy to find in tropical countries such as Indonesia. Therefore, the aim of this study is to extract the pectin from ambarella fruit peel which is used as a biosorbent for the absorption of Cu(II) metal ions. Effects of pH, contact time, and initial adsorbate concentration (equilibrium isotherm) were investigated to determine the maximum adsorption capacity. The biosorbent was tested on samples of Batang Arau River water.

Experimental

Materials

The materials used were ambarella fruit peel. The biomass was obtained from Padang, Indonesia, all of the reagents were provide from Merck, $Cu(NO_3)_2$, citric acid,0.1 M NaOH, and acetone, aquadest, 96% ethanol.

Instruments

The equipment used was a set of glassware, an analytical balance, a 150 μm sieve, an oven, a shaker, pH meter, filter paper. The instruments used were FTIR (Fourier Transform InfraRed) Spectrometer Frontier Version 10.6.1 and AA-6300 Shimadzu Atomic Absorption Spectroscopy.

Methods

Preparation of Biosorbent

Ambarella fruit peel were cleaned of dirt and separated from the flesh, then cut into small pieces and dried at room temperature. The dried ambarella fruit peel was then mashed and sieved using a 150 μm sieve. The addition of 5% citric acid solvent was carried out in 20 grams of dry powder from ambarella fruit peel, with a weight ratio of dry powder to solvent of 1:50. Then it was extracted by boiling at 85^{o}C for 120 minutes and stirring speed 600 rpm, after which it was filtered. Furthermore, the filtrate obtained was evaporated at 100^{o}C in a heating furnace until the volume reached half, then cooled. The cooled filtrate was then added to 96% ethanol in a 1:1 ratio with extracted pectin and allowed to stand for 24 hours before being filtered. The pectin precipitate obtained was then washed in 96% ethanol until the washing water became clear. The pectin was dried in an oven for 24 hours at 40^{o}C. Then the extracted pectin was characterized using an FTIR instrument and the methoxyl content was determined.

Methoxyl Content Of Pectin

A number of 0.25 grams of pectin extract was added in 50 mL of distilled water in Erlenmeyer and It was added by 6 drops of pp indicator. Then, it was titrated by using 0.1 N NaOH until it reached the end point of the titration which was marked by a change in color from clear brownish to pink. Methoxyl pectin content can be calculated using the equation:[16],[17]

$$\text{methoxyl content} = \frac{\text{V NaOH x 31 x NaOH}}{\text{Initial sample weight (mg)}} x100\%$$

Effect of pH on adsorption process

0.2 g of the extracted pectin was dissolved in 10 mL of a pH range of 2–7, 100 mg/L Cu(II) ion solution. Prior to adding the adsorbent, the pH was adjusted to the required value by adding 0.1 M $C_6H_8O_7$ and/or 0.1 M NaOH. The mixture-containing flask was shaken at 100 rpm for 90 minutes to equilibrate it. The solution was then filtered, and the concentration of Cu(II) that was still in the solution but not yet absorbed was examined using an Atomic Absorption Spectrophotometer.

Effect of contact time

The experiment was carried out at optimal pH 4 with contact times of 15, 30, 60, 90, and 120 minutes. In 10 mL of each of the metal solutions with 100 mg/L concentrations, 0.2 g of the extracted pectin was added. In a water bath shaker with a speed limit of 100 rpm, the samples were stirred. After the contact period had passed, the mixture's suspension was filtered, and an Atomic Absorption Spectrophotometer was used to check for Cu(II) ions in the liquid medium.

Effect of initial concentration of the Cu(II) ions on adsorption

0.1 g of extracted pectin was treated with 10 mL of changing concentration (50-250 mg/L) Cu(II) ions solution at ideal pH 4 for 90 minutes. The pH of the solution was adjusted with 0.1 M $C_6H_8O_7$ and 0.1 M NaOH. An orbital shaker operating at 100 rpm was used to continuously shake the mixes. The solution was then filtered, and the amount of Cu(II) ion in the filtrate was determined using an Atomic Absorption Spectrophotometer.

The value of adsorption capacity can be calculated using the equation[8]:

$$\text{Adsorption Capacity (mg/g)} = \frac{(\text{Co-Ce}) \cdot \text{V}}{\text{m}}$$

With C_o is an initial concentration of Cu^{2+} ions (mg/L) ; C_e is concentration of Cu^{2+} ions in equilibrium in the liquid phase (mg/L) ; m is mass of adsorbent (gram); and V is volume metal solution (L).

Adsorption isotherm

Adsorption isotherm was used to describe the interaction between the adsorbent and the metal ions. Langmuir and Freundlich model were adopted to fit the equilibrium isotherm[15].

The langmuir isotherm can be determined in the linear form of:

$$\frac{C_e}{q_e} = \frac{1}{q_{\max} k} + \frac{1}{q_{\max}} C_e$$

Where Ce (mg L^{-1}) is the equilibrium concentration, qe (mg g^{-1}) refers to amount of heavy metal adsorbed by pectin at equilibrium, qmax is the maximum uptake at a monolayer, and k refers to the Langmuir adsorption constant (L mg^{-1}). Separation factor (RL) is one of the most important parameters of Langmuir isotherm. From this value, we can determine whether an adsorption is favorable (0<RL<1), unfavorable (RL>1), or irreversible (RL=0).

$$R_L = \frac{1}{1 + kCo}$$

The empirical Freundlich model can be represented as :

$$\ln q_e = \ln K_f + \frac{1}{n} C_e$$

Where K_f is the Freundlich constant [$L^{1/n}mg^{1-1/n}g^{-1}$], n refers to the heterogeneity factor. The value of 1/n is demostrated if the adsorption is irreversible (1/n = 1), favorable (0<1/n<1) or unfavorable (1/n>1).

Contact with water sample from the Batang Arau River

Samples were collected in the Batang Arau River, West Sumatra, Indonesia. The sample was filtered and placed up to 25 mL into an Erlenmeyer containing extracted pectin, where Cu(II) adsorption was carried out under optimum conditions.

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Results and Discussion

Characterization of the extracted pectin

FTIR spectroscopy analysis was utilized to determine the nature of potential extracted pectin-metal ion interactions. Using an FTIR spectrometer at a wavenumber of 400–4,000 cm^{-1}, the spectra of samples of extracted pectin were recorded both before and after the biosorption procedure. FT-IR was used to examine the various functional groups present on the extracted pectin surface before and after adsorption to Cu(II) ion, as shown in Fig. 1. Figure 1(a) shows the spectra of the extracted pectin, with the peak at 3331.36 cm^{-1} assigned to O-H vibrations, the peak at 2924.02 cm^{-1} to -CH stretching of the alkanes group, the peak at 1727.38 cm^{-1} to C=O stretching of carbonyl groups, and the peak at 1626.20 cm^{-1} to a band of stretching carboxylic ions. The peak noted at 1329.31cm^{-1} was allotted to the -CH bending vibration. In the fingerprint region of pectin absorption band at range 1300-800 is assigned as C-O absorption band. The band of the methoxyl group -OCH_3 was observed at 1232.43cm^{-1}. The bands observed at 994.18 cm^{-1} was the characteristic peaks of galacturonic acid in peptic polysaccharides. These peaks are the characteristic absorption of pectin[18]–[20].

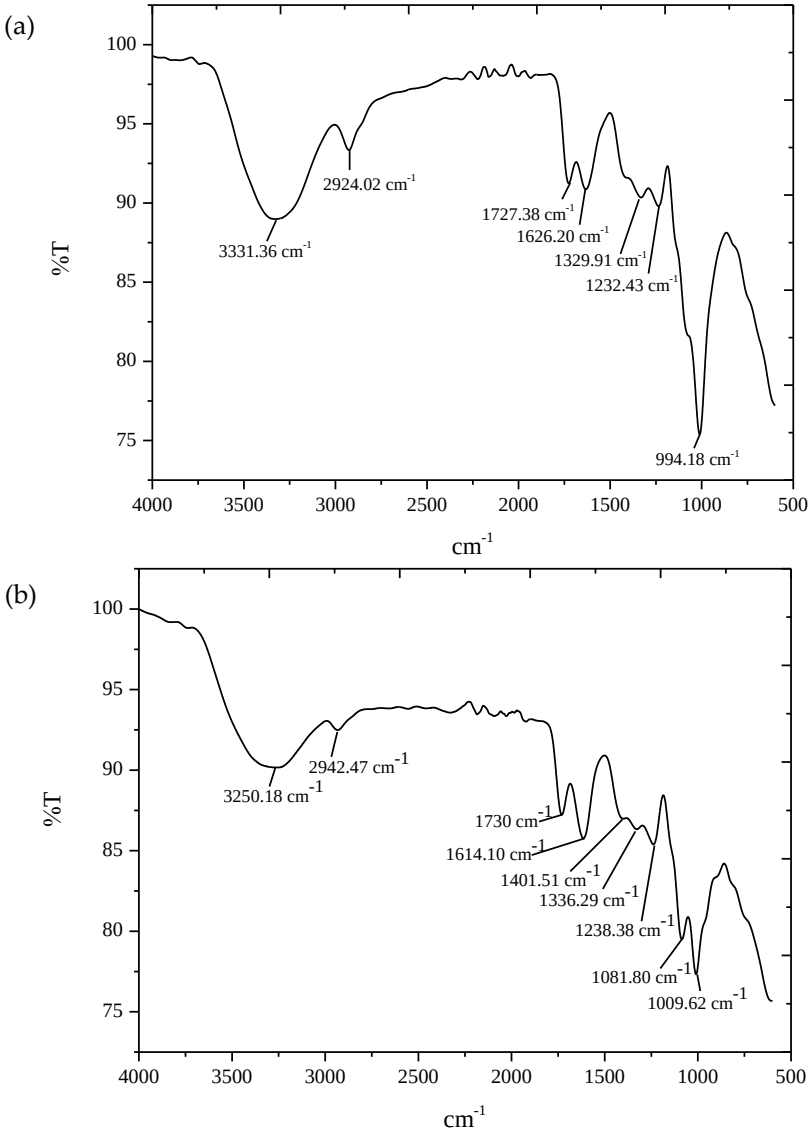

Figure 1. Functional groups of pectin compounds before (a) and after contact with Cu(II) (b)

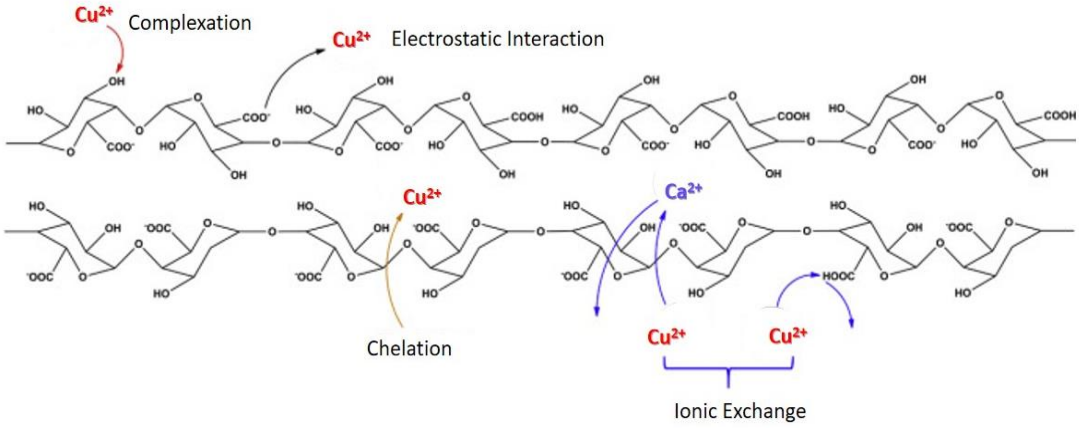

Figure 2. Illustration of interaction between Cu(II) ions and active functional group of the extracted pectin[15]

Figure 1(b) depicts the deformation and shifting of several peaks in the Cu (II) ion-loaded biosorbent material. There was a shift in the stretching vibration band of -OH from 3331.36 to 3250.18 cm^{-1}. The shift in the OH adsorption peak demonstrated an interaction between the hydroxyl group of pectin and the Cu(II) ion. In addition to shifting the C=O vibration band from 1727.38 to 1730 cm^{-1}, the C-O group's vibration band was also moved from 994.18 to 1009.6 cm^{-1} and overlapped with the peak of 1081.30 cm^{-1} following Cu(II) sorption[15], [21]. There are several possible mechanisms reported for the interaction between Cu(II) metal ions and the active sites of pectin and it is is illustrated in Figure 2.

The extracted pectin was also determined for its methoxyl content using the titration method[16]. Based on the methoxyl content, pectin is devide into, low methoxyl (<7%) and high methoxyl (>7%). The lower of methoxyl content, the more hydrocarbon groups there will be in substance. The hydrocarbon group in the pectin will bind to Cu(II) ions so the Cu(II) ions will absorbed by the pectin extract. The results of the analysis, the methoxyl content of pectin was 1.92%. This is classified as low-methoxyl level, which is less than 7% which can be used as an adsorbent.

Effect of pH on adsorption process

Adsorption capacity between adsorbent and adsorbate is influenced by the pH of the solution. As shown in Figure 3, the adsorption capacity of extracted pectin increased from 0.968 mg/g to 2.554 mg/g with a rise in pH from 2 to 4. At a pH of 4, the greatest adsorption was achieved. This result is supported by research conducted by Putu et al (2015) which produced an optimum pH of 4[22]. The web of positive charges in the extracted pectin and the competition between Cu(II) ions and H^+ in solution prevents Cu(II) ion removal at lower pH values[11], [12]. Low pH will make it easier for the carboxyl and hydroxyl groups in pectin to protonate, which will cause a reversal of charge and lessen the activity of binding metal ions. The negative charge network on extracted pectin goes up at higher pH levels as a result of the binding sites' deprotonation, and Cu(II) ion adsorption consequently increases. On the other hand, at higher pH the decreased adsorption capacity is associated with unstable pectin due to β-elimination and hydro-complexes of metal ions often formed with the increase of pH[15].

Effect of contact time

The effect of contact time on the adsorption of Cu(II) by extracted pectin is shown in Figure 4. With longer contact times, the biosorption increased, and for the first 15 minutes, only a little amount of Cu(II) was removed. After that, the adsorption capacity gradually increased until, after around 90 minutes, equilibrium was reached.

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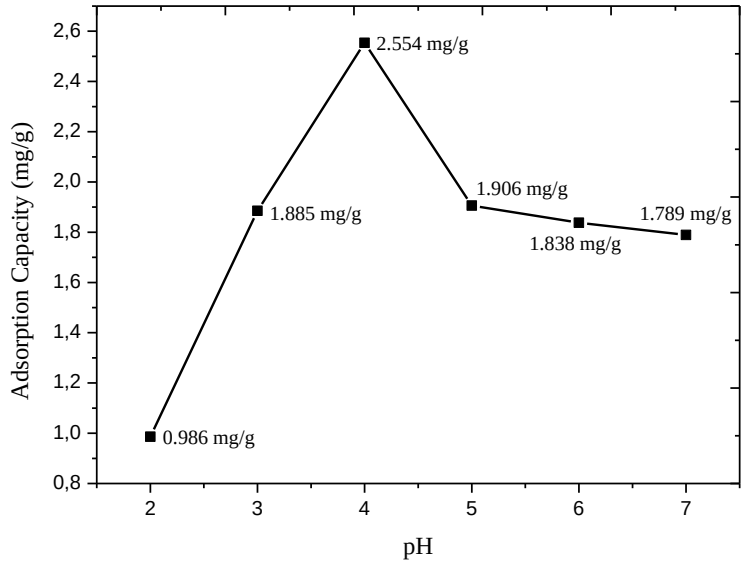

Figure 3. Effect of pH on biosorption of Cu (II) by extracted pectin from ambarella fruit peel

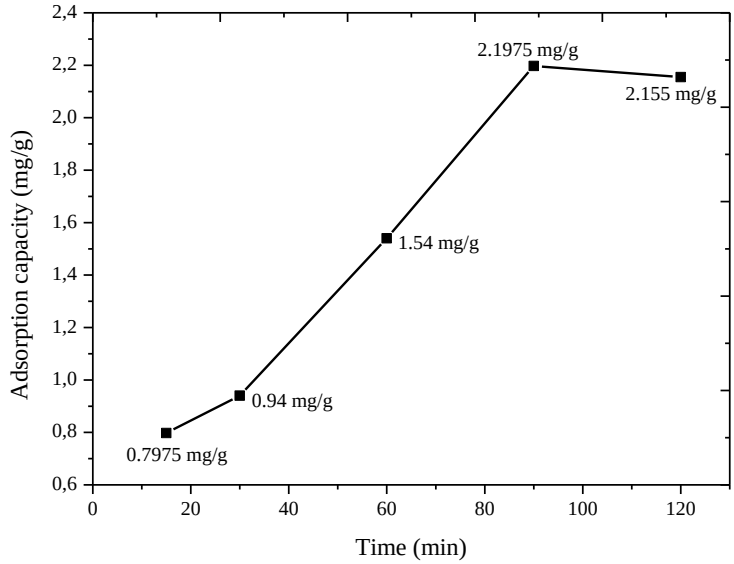

Figure 4. Effect of contact time on biosorption Cu(II) by extracted pectin of ambarella fruit peel

After that, the adsorption capacity gradually increased until, after around 90 minutes, equilibrium was reached. The best contact time was determined to be 90 minutes for subsequent studies because increasing the contact time further did not improve the biosorption yield. The quantity of available adsorption sites on the surface of the adsorbent defines the adsorption capacity in typical surface adsorption behavior, which is how this adsorption technique works. The extent of adsorption approaches a limit when Cu(II) has fully encapsulated the surface active sites, and this limit can be characterized by the maximal biosorption capacity. The amount of Cu(II) adsorbed, however, decreases once more after the equilibrium time because of the desorption of the Cu(II) adsorbed on the extracted pectin[12].

Effect of initial concentration of the Cu(II) ions on adsorption

Figure 5 demonstrates how an increase in Cu(II) ion solution concentration leads to an

increase in the adsorption capacity of Cu(II) ions. Based on the % efficiency, the optimum condition is obtained at 150 ppm, with an adsorption capacity of 6.586 mg/g and an efficiency percentage of 43.9%. So, the optimum condition of initial concentration of Cu(II) ion on adsorption was selected at 150 ppm.

The active sites of the biosorbent influences the value of the adsorption capacity. By creating linkages between the active sites on the surface of the biosorbent, chemical adsorption takes place. If the active sites are not saturated, the biosorbent will continue to adsorb more Cu(II) ions. This adsorption process will continue until the active sites approach saturation, at which point there will be no more increases in the biosorbent's adsorption of Cu(II) metal ions, so that the rate of adsorption is no longer impacted by the concentration increase[11], [15].

Figure 6 shows the results of the Langmuir and Freundlich isotherm techniques from the initial concentration data. The Langmuir and Freundlich isotherm method was utilized to create an equilibrium equation that could be used to calculate how much biosorbate mass extracted pectin from ambarella fruit peel might adsorb.

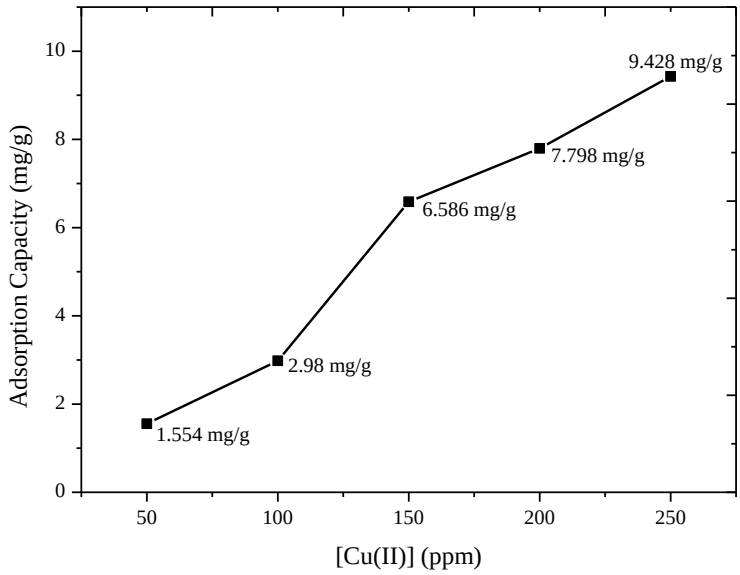

Figure 5. Effect of concentration on biosorption Cu(II) by extracted pectin of ambarella fruit peel

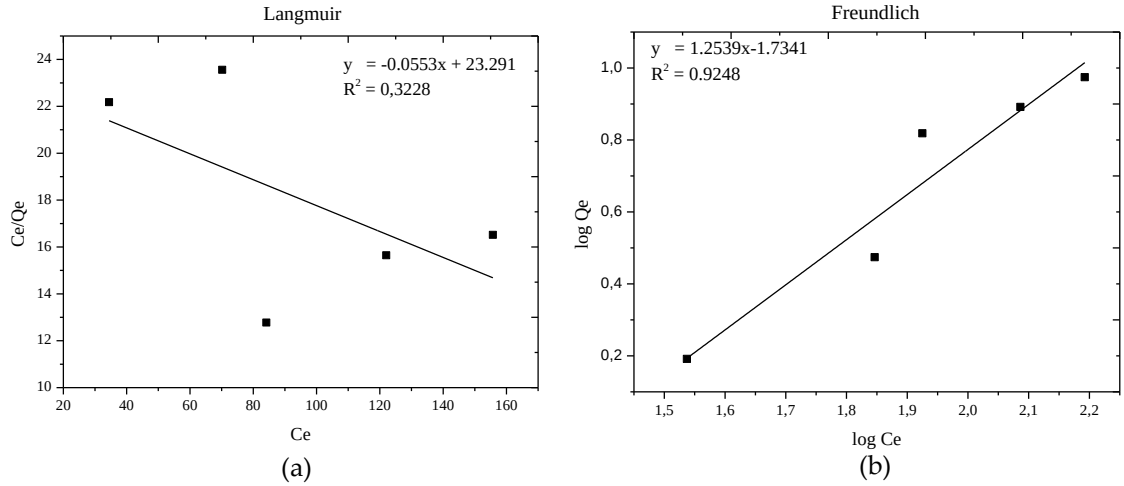

Figure 6. The plots of the experimental data compared to adsorption isotherm models: Langmuir (a) and Freundlich (b)

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Table 1. Langmuir and Freundlich constant of the adsorption of Cu(II) ions on to extracted pectin

Langmuir and Freundlich constant	Values
Adsorption coefficient, K ($mg^{-1}dm^3$)	0.0024
Adsorption capacity, q_e (mg g^{-1})	18.08
Separation factor (R_L)	0.73
Freundlich constant, K_f ($L^{1/n}mg^{1-1/n}g^{-1}$)	5.664
Heterogeneity factor, n	0.8

Table 2. Biosorption of Cu (II) from Batang Arau River using extracted pectin from ambarella fruit peel

Initial [Cu(II)]	Final [Cu(II)]	Biosorbed [Cu(II)]	Adsorption capacity
0.525 ppm	0.149 ppm	0.376 ppm	0.0376 mg/g

The Langmuir isotherm shows that there are a number of active sites proportional to the surface area on the biosorbent's surface, and each active site can only absorb one molecule. While the Freundlich equation describes a physical biosorption in which the biosorbent molecule forms many layers (multilayer)[13], [15].

A good linearization graph and a coefficient of determination R^2 ≥0.9 (close to 1) demonstrate the validity of Langmuir and Freundlich biosorption equations. Figure 5 shows that the Cu(II) ion biosorption equation for extracted pectin meets the Freundlich equation with R^2 = 0.9248 and the Langmuir biosorption equation with R^2 = 0.3228. This demonstrates that the Freundlich equation may be used to describe the process of Cu(II) ion biosorption by extracted pectins.

Table 1 shows a favorable adsorption of Cu(II) ions where R_L value is (0<RL<1) at langmuir model and at Freundlich model showed 1/n value (>1) indicating the occurrence of cooperative adsorption.

Contact with water samples from the Batang Arau River

The sample was collected from water on the surface of the Batang Arau River near the Siti Nurbaya Bridge which is a shipping area. According to Table 2, the amount of Cu(II) metal ions biosorbed is 0.376 ppm, whereas the biosorption capacity of Cu(II) ions in Batang Arau River water is 0.0376 mg/g. The initial concentration of Cu(II) metal ions in the water of the Batang Arau River determines how many metal ions are adsorbed by the biosorbent. Cu(II) metal ions have an initial concentration of 0.525 ppm, which is considered to be polluted water. The standardized Cu level is 0.02 mg/L, as stated in Government Regulation No. 22 of 2021 about river water quality regulations (PP No. 22, 2021). The low adsorption capacity of Cu(II) ions in the Batang Arau River could be due to the presence of other metals in the water, causing competition in adsorption.

Conclusions

The ambarella fruit peel has the potential to be utilized as an alternative and cost-effective biosorbent material for the removal of Cu (II) from aqueous solutions, according to data from batch adsorption tests. Using the pectin that was extracted from the peel of the ambarella fruit, Cu(II) was biosorbed using a variety of experimental parameters, including pH of the solution, contact time, and initial concentration

of Cu (II) ions. pH 4 with an adsorption capacity of 2.5540 mg/g, a contact time of 90 minutes with an adsorption capacity of 2.1975 mg/g, and a concentration of 150 ppm with an adsorption capacity of 6.5860 mg/g were the ideal conditions for the biosorption of Cu(II) ions using extracted pectin from ambarella fruit peel. The Freundlich models provided accurate descriptions of the equilibrium biosorption phenomena. Observations made after contacting the extracted pectin with water from the Batang Arau River show an adsorption capacity of 0.0376 mg/g.

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