ADSORBENT SYNTHESIS FROM *Penaeus monodon* CHITOSAN AS COPPER ABSORPTION IN ELECTROPLATING LIQUID WASTE

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ABSTRACT

Chitosan in Penaeus monodon is a chitin compound removed from the acetyl group leaving a polysaccharide amine group and is polycationic through the chitin deoxidation process. The purpose of this study was to identify the potential use of chitosan adsorbent from Penaeus monodon (Chi-ShSW) on decreasing levels of copper (Cu) in electroplating industrial wastewater and to analyze the effect of variations in flow rate using chitosan adsorbents on reducing levels of copper (Cu) in electroplating industrial sewage. Chi-ShSW can be an adsorbent to reduce Cu (II) metal ions in the Metal Coating Industry wastewater. The adsorption test was carried out with a continuous flow system with different debits. The highest reduction in Cu waste in the study occurred at a flow rate of 215 ml/minute with a time of 84 minutes with a Cu content in the Metal Coating Industry wastewater of 1.227 mg/l and a reduction efficiency of 75.86%. The utilization of chitosan from Penaeus monodon was successfully synthesized as an adsorbent for the wastewater treatment of the metal coating industry. It can be concluded that Penaeus monodon can be used as an adsorbent to degrade Cu(II) metal ions contained in electroplating wastewater.

Keywords: *Adsorption, Chitosan, Copper, Electroplating, Waste*

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Introduction

The development of export-oriented Indonesian fishery commodities is increasing, including in shrimp. The structure of shrimp consists of the body, head, and legs (Supriyantini, 2018). As a result, waste originating from the shrimp export industry also varies, ranging from 65 - 85% of the weight of shrimp. In general, solid shrimp waste is used partly as a mixture of animal feed, and some only accumulate into waste in the environment that emits unpleasant odors (Natalia, 2017).

The tiger shrimp species, often known as *Penaeus monodon* or giant tiger prawn, is a crustacean widely cultivated for consumption (Begum, 2021; Iwan, 2018). Crustacean have problems related to solid waste from shrimp skins, heads, and legs (ShSW) that are not sent for human consumption. So far, the waste is dried and only used as feed and fertilizer (Victor, 2016). Even though ShSW contains many chitosan substances that can be used, they have higher economic value (Rahaman, 2021). Research conducted by Mello (2022) has utilized chitosan from shrimp shells as an adsorbent.

The nature of chitin biopolymers that are not toxic and easily degradable in the environment encourages chitin modification intending to optimize the usefulness and expand the field of application of chitin (Elystia, 2021). This non-toxic natural biopolymer is produced commercially from shrimp shell waste. One of the derivative compounds of chitin that is widely developed because of its wide application is chitosan (Omobayo, 2022).

Chitosan is a chitin compound removed from the acetyl group leaving a polysaccharide amine group and is polycationic through the chitin deoxidation process (Coura, 2020). According to S. Elystia et al. (2022), shrimp shells contain protein (25-40%), calcium carbonate (45%-50%), and chitin (15%-20%). The presence of hydroxyl and amine groups along the polymer chain makes chitosan very effective in binding heavy metal ion cations and cations of organic substances. Chitosan can be an adsorbent/absorbent that can absorb heavy metals, such as Zn, Cd, Cu, Pb, Mg, and Fe (Pourmotazayi, 2019). The active site of chitosan, either in NH_2 or in protonated state NH_3^+, can adsorb heavy metals through chelate formation mechanisms or ion exchangers (Trung, 2022). Chitosan has the sound absorbent and coagulating properties. This compound can be used as a porous material for heavy metals produced by electroplating industry waste (metal plating) (Darmawan, 2019).

Heavy metals are a source of pollution that is very harmful to the environment. Some examples of heavy metals that are toxic to humans are arsenic (As), cadmium (Cd), copper (Cu), lead (Pb), mercury (Hg), nickel (Ni), and zinc (Zn) (Permanasari, 2010). Heavy metals are dangerous because they can interfere with the life of organisms in the environment if their existence exceeds the threshold (Bisirivu, 2020). In addition, it will also be toxic if it crosses the threshold and accumulates in the human body. Cu heavy metal contamination from the electroplating, paint and dye, petroleum refining, fertilizer, mining and metallurgy, explosives, pesticides, and iron and steel industries is hazardous when the liquid waste is released in an environment. It is even carcinogenic to humans and aquatic ecosystems (Nurfatihayati, 2022; Iwan, 2021; Sukma, 2018).

Various efforts were made to overcome this heavy metal problem, such as photoreduction methods, ion exchange (resin), precipitation, electrolysis, and adsorption, and developing all these methods within an environmentally friendly framework (Iriana, 2018). Several methods exist for treating liquid waste containing heavy metals, such as precipitation, electrolysis, and solidification (Mallik, 2022; Iwan, 2020). These methods, such as electrolysis, still need improvement, which requires immense energy (Atangana, 2020; Iwan, 2020). One easy and environmentally friendly waste treatment method is the absorption method with natural adsorbents such as chitosan (Jaya, 2017).

In this study, we reported the utilization of chitosan from tiger shrimp shell waste as an adsorbent. The results of tiger shrimp shell chitosan adsorbent synthesis can be used as a decrease in Cu (II) metal ion levels in electroplating industry liquid waste. The purpose of this study was to identify the potential use of chitosan adsorbent from Chi-ShSW on decreasing levels of copper (Cu) in electroplating industrial wastewater and to analyze the effect of variations in flow rate using chitosan adsorbents on reducing levels of copper (Cu) in electroplating industrial sewage.

Research Methods
Tools and Materials

The materials used in this study were samples of tiger shrimp ShSW, NaOH, HCl, NaOCl (all materials obtained from Merck 99%),

acetone (Merck 98%), and metal coating industry liquid waste (MCI-Liquid Waste). The tools used include a blender laboratory, analytical laboratory (Analytical Balance 220 g x 0.0001 g Lucky), pH meter (Metrohm, 827 pH Lab), oven (Memmert Oven Laboratory UN 55 53 L), magnetic stirrer, spatula, shaker (Stuart reciprocating shaker SSL2), centrifugation (Centrifuge Gemmy, Plc 05 12 Hole), and centrifugation tube.

The analysis of the data obtained is in the form of the amount of initial and final copper metal concentration after contracting from each treatment. Calculating the concentration of copper metal absorbed by chitosan is used with equation (1).

$$C_{absorbed} = initial\ C - C_{sampling} \quad ....(1)$$

As for the absorption percentage, it is calculated using equation (2).

$$\%\ of\ absorption = x\ 100\% \quad .... (2)—$$

Explanation of equation 2. i.e., absorbed C is the absorbed metal concentration (mg/l), then initial C is the metal concentration before contracting (mg/L), and final C is the metal concentration after contracting (mg/L). The hypothesis for this research is that H_o as Chitosan from tiger shrimp shells cannot be used as an adsorbent in reducing Copper (Cu) levels in Metal Plating Industry liquid waste. Furthermore, H_a is a problem from tiger shrimp shells that can be used as an adsorbent in reducing Copper (Cu) levels in the liquid waste industry.

Procedures

The research is conducted through several steps as follows. Making adsorbents from tiger shrimp ShSW consists of a) Preliminary Stage: Tiger shrimp shells are washed until the fishy smell disappears and then drained. Dry in an oven at 50°C for 2 hours. The tiger shrimp shell is blended, and a smooth tiger shrimp shell is obtained. b) The deproteination stage: this process is carried out at 60-70 °C using 1 M NaOH solution to create an alkaline atmosphere in the mixture. Then the ratio of shrimp powder with NaOH = 1:10 (gr powder/ml NaOH) while stirring for 60 minutes.

The mixture is allowed to stand first until it looks separate between the precipitate and the filtrate. After that, it is separated by filtering and then centrifuged so that the separation process is maximally taken for the precipitate. Washing of the precipitate is carried out using aquades until neutral pH. Next, it is filtered for precipitation and

dried in an oven at <50^{0}C for 2 hours. The demineralizing stage is the removal of minerals at a temperature of 25-30°C using a 1 M HCl solution with a ratio of samples with HCl = 1:10 solution (gr powder/ml HCl) while stirring with a magnetic stir for 120 minutes. Then it is filtered for precipitation.

Depigmentation stage, which is a demineralized precipitate extracted with acetone and bleached with 0.315% NaOCl (w/v) for 5 minutes at room temperature. Solid and solvent ratio 1:10 (w/v). The deacetylation stage is carried out with chitin produced in the above process inserted in a NaOH solution with a concentration of 50% (by weight) at a temperature of 90-100°C while stirring at a constant speed for 60 minutes. Then it is filtered for precipitation. The precipitate is washed using aquadest until neutral pH and then dried in an oven <50^{0}C for 2 hours. Thus, chitosan adsorbent is formed from ShSW (Chi-ShSW).

The test stages and scheme of continuous absorption equipment include absorption test steps by inserting wastewater samples into the reservoir (effluent tub). Then a sample of wastewater in a feeder bath through a pump. Air in the feeder bath is allowed to stand until it is calm. After the water condition is mild, it is ready to put water on the reactor tube so that water can flow from bottom to top, including the Up-flow method. Figure 1 explains the parts of the tool scheme, namely: a) First part: The main container for collecting wastewater from (MCI-Liquid Waste) (Container A), which is used to hold liquid waste from the metal plating industry, which is then pumped and flowed to the feeder basin.

In this study, flow rate variations were carried out at 215 ml/minute, 250 ml/minute, and 280 ml/minute to get optimal results in the adsorption process. b). The second part is a feeder container so that wastewater will flow into the column (reactor tube). c) The third part is an adsorption column with a synthesized adsorbent mass (Chi-ShSW) of 10 grams. Next, d) the fourth part: Container for storing treated water (effluent). The sampling time at an affluent time is 0-7 minutes (T0, T1, T2, T3, T4, T5, T6, and T7). The research stage includes the absorption test, namely inserting wastewater samples (MCI-Liquid Waste) into the effluent reservoir.
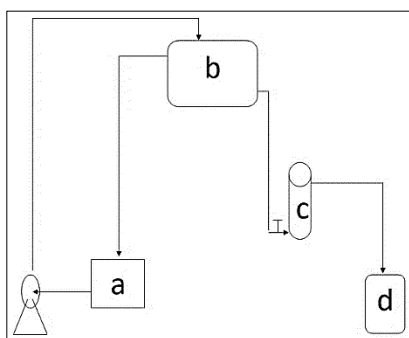
Figure 1. Scheme of absorption apparatus

Furthermore, flow the wastewater sample in the feeder bath through the pump and leave the water in the tub until mild. The final step is to open the tap on the reactor tube so water can flow from bottom to top (Up-flow). Then the sampling process can be completed.

Result and Discussion

MCI-Liquid Waste Sample Quality. To reduce water pollution from industrial wastes, samples used in research directly from copper wastewater were taken from one of the metal plating industries (MCI-Liquid Waste) as an application. Decrease in Copper Content (Cu) discharges 215 ml/min. The adsorbed processing of this study is carried out consistently. The mass of adsorbents used is 10 grams of chitosan. Debit entry is set at 215 ml/min. The time of retrieval of the sample on outflow is minute 0, 4, 24, 44, 64, 84, 104, and 124. The results of the study are presented in Table 1.

Table 1. Percentage of Decrease in Cu (II) Metal Ion (discharge 215 ml/min)

No	Time (Minutes)	Initial Cu concentration (mg/L)	Average concentration of Cu (mg/L)	Percentage Decrease (%)
1	4	5,084	4,466	12,15
2	24	5,084	3,646	28,28
3	44	5,084	3,109	38.84
4	64	5,084	2,277	55,21
5	84	5,084	1,227	75,86
6	104	5,084	2,584	49,17
7	124	5,084	4,454	12,39

Source: Primary data, 2015

Based on Figure 2, it can be seen from the 0 minute to the 84th minute at a flow discharge of 215 ml/minute, the highest adsorption process occurs. The equilibrium of adsorbs happened around the 84th minute; this was proven in the 84th minute at a discharge of 215 ml/minute, able to reduce Cu levels up to 1,227 mg/l with a percentage of 75.86% where the maximum Cu

content was 3 mg / l. From the 84th minute to the 124th minute, the adsorbent ability began to decline slowly and began to experience saturation. This phenomenon is because the slight flow discharge causes the contact time of wastewater in the reactor to be longer so that absorption is higher.
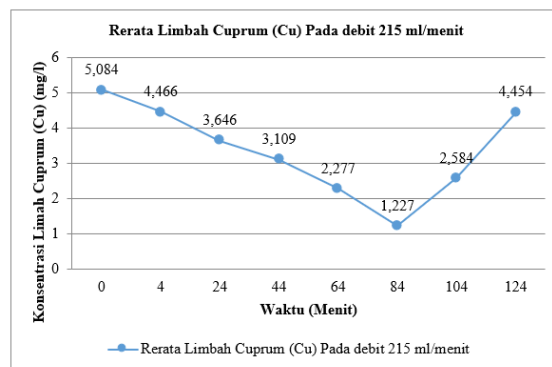

Figure 2. The relationship between the length of time of adsorbs with Cu concentration at discharge 215 ml/min.

Decrease in Copper Content (Cu) discharge 250 ml/min. Based on laboratory results, the 250 ml/minute discharge (testing) study showed that the effectiveness of reducing Copper (Cu) levels was less effective than the study (testing) of Copper (Cu) levels at 215 ml/minute flow discharge. The results of research discharge of 250 ml/min are presented in Table 2.

Table 2. Percentage of Cu (II) Metal Ion Reduction (discharge 250 ml/min)

No	Time (Minutes)	Initial Cu conc (mg/L)	The average conc of Cu (mg/L)	Percentage Decrease (%)
1	4	5,084	4,500	11,48
2	24	5,084	4,313	15,16
3	44	5,084	3,661	27,98
4	64	5,084	2,642	48,03
5	84	5,084	1,956	61,52
6	104	5,084	3,118	38,67
7	124	5,084	3,516	30,84

Source: Primary data, 2015.

Based on Figure 3, it showed that at a flow discharge of 250 ml/minute, from the 0 minute to the 84th minute, chitosan adsorbent was still able to reduce copper (Cu) levels by 1,956 mg/l (by 61.52%) in metal coating wastewater, although not more significant than from research (testing) copper (Cu) levels in flow discharges of 215 ml/minute. This is thought to be caused because the release of 250 ml/minute is greater than the discharge of 215 ml/minute, so the contact time between the solution and the solid (residence time) in the reactor is shorter. So that, the Cu from the solution is not greater than the study (testing) of

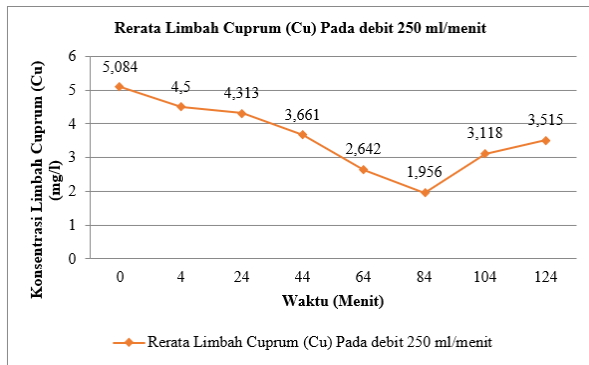Copper (Cu) content at a discharge of 215 ml/minute.

Figure 3. The relationship of the length of adsorption time with a Cu discharge concentration of 250 ml/min.

Decrease in Copper Content (Cu) discharges 280 ml/min. Based on laboratory results, the decline in copper (Cu) content at 90^0 (flow discharge 280 ml/min) the concentration of copper content (Cu) still decreased the same as at 30^0 (flow discharge 215 ml/minute) and 60^0 (flow discharge 250 ml/minute). The results of the study at 90^0 (flow discharge 280 ml/min) are presented in Table 3.

Table 3. Percentage of Cu (II) Metal Ion Reduction (discharge 280 ml/min)

No	Time (Minutes)	Initial Cu con (mg/L)	The average con of Cu (mg/L)	Percentage Decrease (%)
1	4	5,084	4,447	12,52
2	24	5,084	4,315	15,12
3	44	5,084	4,221	16,97
4	64	5,084	4,003	21,26
5	84	5,084	2,901	42,93
6	104	5,084	3,391	33,30
7	124	5,084	3,778	25,68

Source: Primary data, 2015.

Based on the graph of Figure 4, it showed that at a discharge of 280 ml/minute, from the 0th minute to the 84th minute, chitosan adsorbent was still able to reduce copper (Cu) levels by 2.901 mg/l (by 42.93%) in metal plating wastewater, although it was not more significant than the study (testing) of Copper (Cu) levels at a flow discharge of 215 ml/minute and at a flow discharge of 250 ml/min. This is thought to be caused because the flow discharge at a release of 280 ml/minute is greater than the discharge of 215 ml/minute.
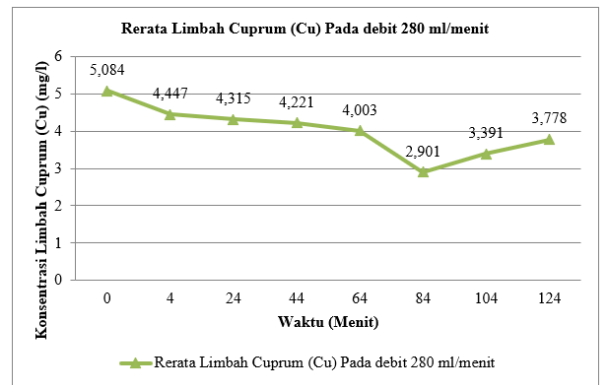

Figure 4. The relationship between the length of time of adsorbs with Cu concentration at discharge 280 ml/min.

A flow discharge of 250 ml/minute causes the contact time between the solution and solids (residence time) in the reactor to be very short so that the Cu absorption is not greater than research of Copper (Cu) content at a discharge of 215 ml/minute and a flow discharge of 250 ml/minute. At the 124th minute, the adsorbent has not experienced saturation compared to the release of 215 ml/minute and the dismissal of 250 ml/minute; this is because the flow discharge is faster than the two comparisons causing wastewater in the reactor column to stay very fast so that the adsorption process is lower.

Determination of conditions to be used in real-scale applications by comparing the research results (testing) conducted with flow discharges of 215 ml/min, 250 ml/min, and 280 ml/min. The basis for determining the conditions to be used is the best concentration of effluent. A comparison of research results (testing) using discharge 215 ml/min, 250 ml/min, and 280 ml/minute are presented in Figure 5.
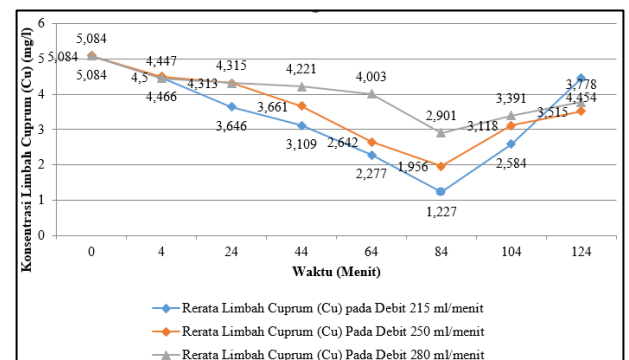

Figure 5. Time relationship of adsorption with discharge 215 ml/min, 250 ml/min, 280 ml/min.

Based on the graph in Figure 5, it indicates that there are three stages of adsorption. Stage 1 occurs when the curve decreases. This condition suggests that the speed of adsorbate (Cu) transfer from solution to solid (tiger shrimp chitosan) is greater than that of Cu transfer from concrete to solution. Stage 2 occurs when the curve experiences a turning point from a descending to an upward condition. This condition indicates that the transfer of Cu from solution to solid equals the transfer rate of Cu from stable to solution. At this turning point, the adsorbent experiences saturation in adsorbs Cu. Stage 3 occurs when the curve increases. This condition indicates that the transfer of Cu from the solution to the solid is less than the transfer of Cu from the concrete to the solution.

Stage 1 occurs in minutes 0 to 84, during which time adsorption takes place higher (can be seen in Figure 5. where in minutes 0 to 84, the curve moves down). Stage 2 occurred in the 84th minute, where there was an adsorption equilibrium and, at the same time, became the turning point of adsorbent absorption (can be seen in Figure 5. In the 84th minute, the curve is at the highest adsorption point). Stage 3 occurs in the 84th minute to the 124th minute; the adsorbent begins to experience saturation (can be seen in Figure 5. In the 84th minute to the 124th minute, the curve slowly moves up). Several factors influence the absorption process, such as the contact time between the adsorbate and the adsorbent from tiger shrimp solid waste chitosan. Chitosan produced in this study, as in Table 4 which reached on international standard values to be used for various applications.

Table 4. Chitosan characterization

Parameter	The value of the chitosan obtained	Values from International Standards
Texture	Powder	Powder
Color	White	White to Pale yellow
Construction	Odorless	Odorless

A discharge of 215 ml/min, 250 ml/minute, and 280 ml/minute can reduce Copper (Cu) levels in metal plating wastewater. However, chitosan can reduce copper levels even more at a discharge of 215 ml/minute. The high effectiveness of reducing Copper (Cu) classes at a shot of 215 ml/minute, caused by a release of 215 ml/minute wastewater flows more slowly so that the residence time in the reactor column is longer; this causes the highest adsorption process.

Conclusion

The purpose of this study was to identify the potential use of chitosan adsorbent from Chi-ShSW on decreasing levels of copper (Cu) in electroplating industrial wastewater and to analyze the effect of variations in flow rate using chitosan adsorbents on reducing levels of copper (Cu) in electroplating industrial sewage.

The utilization of chitosan from *Penaeus monodon* was successfully synthesized as an adsorbent for the wastewater treatment of the metal coating industry. It can be concluded that Chi-ShSW can be used as an adsorbent to degrade Cu(II) metal ions contained in Metal Plating Industry wastewater. Furthermore, there was a flow discharge of 215 ml/minute, the highest decrease in minute 84, with Cu content in Metal Plating Industry wastewater of 1,227 mg / l and a decreased efficiency of 75.86%.

At a flow discharge of 250 ml/minute, the highest decrease was at minute 84, with Cu content in the Metal Plating Industry wastewater of 1,956 mg / l and a decreased efficiency of 61.52%. At a 280 ml/minute flow discharge, the highest decrease in minute 84 with Cu content in Metal Plating Industry wastewater of 2.901 mg / l and efficiency decrease of 42.93%. Then the difference in the variation of discharge used affects the speed of the water flow rate; the smaller the blast, the longer the contact with chitosan and the higher the ability of chitosan to degrade Cu metal. The highest reduction in Cu Waste in the study (testing) occurred at a flow discharge of 215 ml/minute with a time of 84 minutes with Cu levels in the Metal Plating Industry wastewater of 1,227 mg / l and a reduction efficiency of 75.86%.

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