EEET ECOLOGICAL ENGINEERING & ENVIRONMENTAL TECHNOLOGY

Ecological Engineering & Environmental Technology 2024, 25(10), 126–133
https://doi.org/10.12912/27197050/191416
ISSN 2299–8993, License CC-BY 4.0

Received: 2024.07.06
Accepted: 2024.08.15
Published: 2024.09.01

Removal of Copper, Cadmium, and Lead Ions by Adsorption Method using Chitosan from Fish Scales and its Application in Laboratory Wastewater Treatment

David Bahrin[1], Tuty Emilia Agustina[1,2*], Susila Arita[1], Elda Melwita[1], Lisa Apriani[1], Fitri Indriani Nurhasanah[1], Maria Siswi Wijayanti[3]

[1] Chemical Engineering Department, Faculty of Engineering, Universitas Sriwijaya, Jl. Raya Palembang-Prabumulih KM 32 Indralaya, Ogan Ilir 30662, South Sumatera, Indonesia

[2] Water and Wastewater Engineering Research Group, UPT Integrated Laboratory, Universitas Serasan, Jl. H. Pangeran Danal No. 142, Muara Enim 31312, South Sumatra, Indonesia

[3] Educational Laboratory Institution, UPT Integrated Laboratory, Universitas Sriwijaya, Jl. Raya Palembang-Prabumulih KM 32 Indralaya, Ogan Ilir 30662, South Sumatra, Indonesia

* Corresponding author's e-mail: tuty_agustina@unsri.ac.id

ABSTRACT

Higher education requires learning in the form of practice and research which is often carried out in the laboratories. The research completed in the laboratory usually produces wastewater which not only has a high organic content, but also contains heavy metal ions, such as copper (Cu), cadmium (Cd), and lead (Pb). These heavy metal ions will harm the environment when directly disposed of without being treated first. One of methods to reduce the level of heavy metal ions in laboratory wastewater is adsorption using chitosan. Therefore, this research was focused on removal heavy metal ions in laboratory wastewater by adsorption method using a chitosan derived from fish scales. The objective of this research was to investigate the effect of pH on the removal of Cu, Cd, and Pb ions in the treatment of synthetic wastewater using chitosan. The effect of chitosan dose on the treatment of laboratory wastewater was also examined. Initially, the chitosan was applied in reducing the metal ions in synthetic wastewater. The synthetic wastewater was prepared using single-component and multi-component samples of heavy metal ions. In this study, the pH was varied from 3–7 for application of chitosan on single and multicomponent synthetic wastewater experiments. Optimum pH was produced and then used to remove the metal ions included in the laboratory wastewater sample. The chitosan dose was varied 0.5–2 g/L. As a result, the highest removal percentage for the reduction of Cu, Cd, and Pb metal ions in single-component synthetic wastewater was at pH 7. However, the highest removal percentage for the reduction of Cu, Cd, and Pb metal ions in multicomponent synthetic wastewater were found in different pH, such as Cu ions at pH 5, Cd ions at pH 7, and Pb ions at pH 3. When applied to laboratory wastewater assessments with pH of 7 and chitosan doses of 0.5–2 g/L, the highest percentage removal of Pb ions (93.75%) was reached when using chitosan dosage of 1.5 g/L; the highest percentage removal of Cu ions (28.99%) was obtained when using chitosan dosage of 2 g/L. Alas, the chitosan dose of 0.5–2 g/L did not have a significant effect on reducing the Cd ions.

Keywords: heavy metals, adsorption, chitosan, laboratory wastewater.

INTRODUCTION

Activities carried out in the laboratory are closely related to the use of chemicals that are acidic, corrosive and toxic. This certainly produces wastewater containing hazardous materials, such as heavy metals. While laboratory wastewater may not reach the same volume as industrial wastewater, improper discharge without treatment can still compromise water and environmental quality (Agustina et al., 2022). The wastewater generated is dominated by heavy metals, such as copper, cadmium, and lead. Heavy metals, with a spgr exceeding 5 g/cm^3, are major contaminants in the environment (Irianti et al., 2017). Heavy metals are a category of metals that has a high toxicity and high density to living organisms, particularly human beings. Microorganisms are unable to break down this type of waste, leading to its accumulation in the environment and subsequently can cause further pollution (Arita et al., 2022). The wastewater produced from implementation of research might contain heavy metals, which must be managed before release into the environment to avoid environmental pollution. The inherent difficulty in degrading metals renders heavy metals potentially toxic (Novia et al., 2023).

The efforts to prevent environmental pollution due to laboratory wastewater can include the adsorption method. The technique of adsorption is effective in removing the heavy metals out of the wastewater as a result of its accessibility, affordability, and environmentally friendly practices (Mokif et al., 2024). One adsorbent that can be used and easily obtained is chitosan. Chitosan is a natural polymer compound made from chitin and is found in many marine animals; for instance, it occurrs in fish scales. Chitosan is a natural polymer that is biodegradable, biocompatible, non-toxic, and possesses exceptional adsorption capabilities (Kurita, 2006; Phuong et al., 2023). The main feature of chitosan is the content of many amino and hydroxyl groups, and the amino groups are easily protonated in acidic media (Keshvardoostchokami et al., 2021; Jamka et al., 2023). The presence of the protonated ($-NH^{3+}$), makes chitosan a potential ligand capable of binding heavy metal ions (Amalraj et al., 2020). With the adsorption process using chitosan, it can reduce or remove the heavy metal ions in laboratory wastewater; therefore, this study used an adsorbent in the form of fish scale chitosan. Fish scales can be found abundantly in maritime countries like Indonesia. Therefore, the chitosan derived from marine creatures can be easily obtained at a low price.

On the basis of the data from research conducted by (Marzuki et al., 2018), it was stated that the chitosan from shrimp shells influences reducing metal ions of Cd and Pb. The adsorption performance of heavy metal tests was determined by varying pH interactions of 2–8. Interaction media conditions with a volume of 100 mL, chitosan weight used of 1 g, contact time 60 minutes, heavy metal concentrations of 200 μg/g cadmium ions and 100 μg/g lead ions. The results showed that the best condition of Cd adsorption occurred in the optimum pH range of 2–5 and the maximum adsorption value of Cd is 198.21 μg/g (99.05%) while Pb has an optimum pH at 4 with Pb adsorption value of 59.33 μg/g (59.33%). The results show that the adsorption of Cd is greater than that of Pb.

A prototype for treating laboratory wastewater was developed and built based on prior research. This prototype utilized a combined process involving coagulation, adsorption, and photo-Fenton processes. The pollutants were characterized by such parameters as pH, TSS, COD, BOD, and heavy metal ions, including Cd, Zn, Cu, Cr, Pb, and Fe. The adsorption process was carried out using activated carbon. After the treatment, the parameters of TSS, and Zn heavy metal ions, as well as total Cr, and Fe, have met the environmental quality standards of regulations (Novia et al., 2023), however the ions of Cu, Cd, and Pb have not fulfilled the regulations. Therefore, in this investigation, the adsorption process of these metal ions was studied by using chitosan as adsorbent.

The objective of this research was to investigate the effect of pH on the removal of Cu, Cd, and Pb ions in the treatment of synthetic wastewater using chitosan. The effect of chitosan dose on the treatment of laboratory wastewater was also examined. In this study, single-component and multicomponent removal of Cu, Cd, and Pb heavy metal ions in the synthetic wastewater were experimented. The pH condition and optimum chitosan dosage obtained from the synthetic wastewater treatment will be applied to the laboratory wastewater. The adsorption process is affected by the adsorbent's capacity to take in a solution, which is also impacted by the solution pH due to the protonation and deprotonation of the adsorbent's active surface sites (Nurhayati et al., 2018; Agustina et al., 2024). The findings obtained from this examine were expected to help in the efforts to reduce heavy metals so they can comply with environmental quality standards.

MATERIALS AND METHODS

Materials

The substances and chemicals used were the chitosan from fish scales, $CuSO_4 \cdot 5H_2O$, $CdSO_4 \cdot 2H_2O$, $Pb(NO_3)_2$, NaOH, HCl, CH_3COOH, and aquadest.

Methods

Adsorption method on single component synthetic wastewater

When preparing single component synthetic wastewater metal ions of Cu, Cd, and Pb with an initial content of 15 ppm, as much as 100 mL were placed into a beaker. The adsorption process was carried out with a chitosan dose of 0.5 g/L and pH variations of 3, 4, 5, 6, and 7; the stirring process was carried out with a magnetic stirrer for 60 minutes at 300 rpm (Nucifera et al., 2016). The synthetic wastewater sample was then filtered, and the filtrate was taken, then tested for heavy metal ions content using the atomic absorption spectrophotometry (AAS) method to determine the removal percentage of ions.

Adsorption method on multicomponent synthetic wastewater

Multicomponent synthetic wastewater containing ions of Cu, Cd, and Pb, each with an initial level of 15 ppm was prepared by placing 100 mL into a beaker. The adsorption process was carried out with a chitosan dose of 0.5 g/L and pH variations of 3, 4, 5, 6, and 7; the stirring process was carried out with a magnetic stirrer for 60 minutes at 300 rpm (Nucifera et al., 2016). The synthetic wastewater sample was then filtered, and the filtrate was taken, and tested for heavy metal content using the Atomic Absorption Spectrophotometry (AAS) method to determine the removal percentage of ions.

Adsorption method on laboratory wastewater

The laboratory wastewater that has been tested initially was poured into a beaker in an amount of 100 mL. The experiment was carried out by mixing doses of chitosan with weight variations (0.5; 1; 1.5; 2 g/L) in laboratory wastewater with pH 7 as the optimum pH obtained from single component and multi-component experiments (Iriana et al., 2018). Laboratory wastewater and chitosan solution were mixed in a glass beaker and stirred using a magnetic stirrer for 60 minutes with a stirring speed of 300 rpm. Then, it was filtered and the heavy metal content was tested using AAS method to determine the removal percentage of ions.

Analysis of metal removal percentage

Analysis of Cu, Cd and Pb ion content was accomplished by determining the final content of each ions using the AAS analytical method. The percentage of removal value of the ions were then calculated by the following Equation:

$$\eta = \frac{Co-C}{Co} \times 100\% \quad (1)$$

Equation 1 illustrates the formulation to determine the removal percentage of ions, while Co is the initial content, C is the content after adsorption treatment with chitosan, and η is the percentage of removal (Sharma, 2020).

RESULTS AND DISCUSSION

FTIR characterisation of chitosan

Figure 1 shows the value of the degree of deacetylation of chitosan is 76.61%. According to (Ifa et al., 2019) chitosan which has a deacetylation degree of more than 60% can be used as an adsorbent. The characterisation results also showed the presence of absorption at wave number 3414.58 cm^{-1} which is the stretch of -OH and overlaps with the absorption of the -NH group of

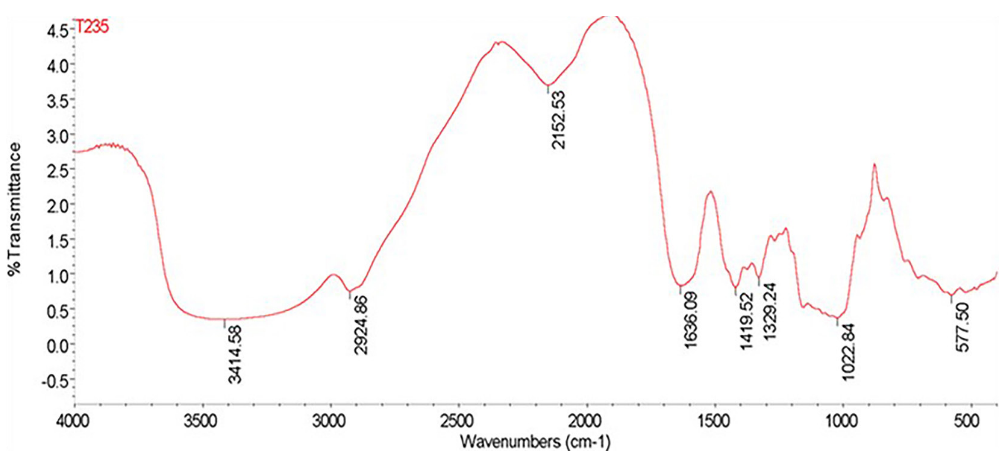

Figure 1. FTIR of chitosan from fish scales

amines at the same wave number. The -NH group indicates the presence of amine which is a characteristic of chitosan. There is an alkane C-H bond detected at wave number of 2924.86 cm^{-1}. The C-C bond of alkenes was also detected at wave number of 1636.09 cm^{-1}. On the basis of the analysis of the FTIR spectrophotometric characterisation of chitosan, it can be concluded that there are functional groups which are characteristic of chitosan.

Effect of pH on the adsorption of single component Cu, Cd, and Pb metal ions

The pH effect on the removal percentage of single component synthetic wastewater of Cu, Cd and Pb ions with initial levels of 15 ppm each using a chitosan dose of 0.5 g/L with stirring using a magnetic stirrer for 60 minutes can be seen in Figures 2, 3, and 4.

Figure 2 shows the graph of the removal percentage of Cu metal from pH 3–7 has increased but not significantly. The largest removal percentage of Cu metal using chitosan adsorbent is at pH 7, which is 99.78%. The high removal percentage value indicates that chitosan adsorbent is effective in the Cu metal adsorption process. This is supported by another study that found a high removal percentage of Cu^{2+} metal absorption of 99.79% at pH 7. The presence of OH^- ions resulted from the NaOH solution which was added to increase the pH of the solution to make it alkaline. The more the pH is raised, the greater the concentration of OH^- ions and the weaker the influence of protons so that OH^- ions interact more easily with Cu ions to form ionic bonds (Ahmad, et al 2020). The more unprotonated amino groups in chitosan, the greater the adsorption of Cu.

Figure 3 presents the graph of the removal percentage of Pb showing that the absorption of Pb has increased significantly. At pH 3, the removal percentage was 92.87%, pH 4 was 93.47%, pH 5 was 96.53%, pH 6 was 97.53%, and the highest removal percentage at pH 7 was 98.60%. This significant increase is due to the small competition between hydrogen ions and Pb ions so that there is a large absorption of Pb. The increase in

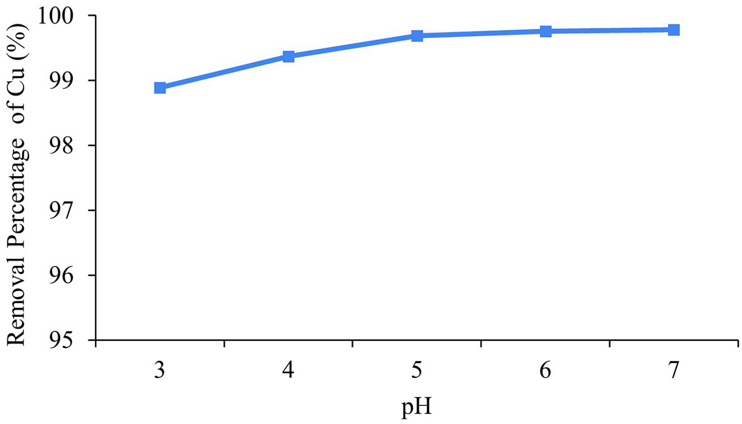

Figure 2. Effect of pH on the removal percentage of Cu ions in synthetic wastewater

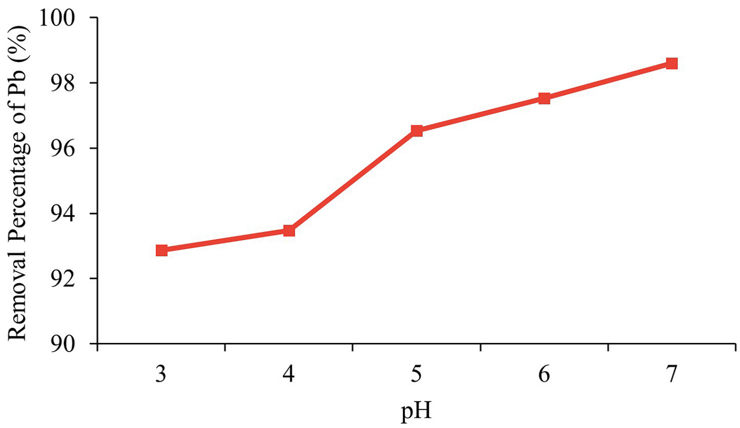

Figure 3. Effect of pH on the removal percentage of Pb ions in synthetic wastewater

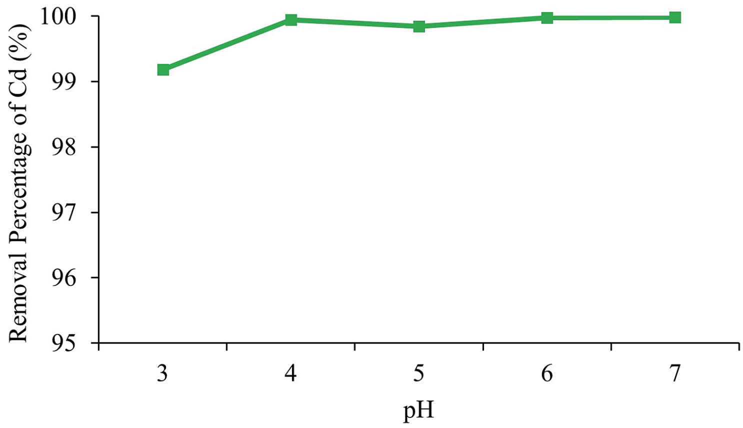

Figure 4. Effect of pH on the removal percentage of Cd ions in synthetic wastewater

OH^- groups present in the chitosan adsorbent enables to absorb more Pb ions. The absorption of Pb metal per cent increased significantly along with the increase in pH and was the most stable.

The graph in Figure 4, shows the removal percentage of Cd. The results of this experiment also show that Cd has the highest removal percentage, compared to Cu and Pb metals, namely at pH 5 of 99.85%, at pH 6 of 99.97% and at pH 7 99.99%. The increase in removal percentage did not increase significantly. This happens, because at pH 7 the presence of H^+ ions can be stabilised by OH^- ions and the NH_2 active site will bind more optimally with Cd. This condition causes equilibrium during the adsorption process.

Effect of pH on the adsorption of multicomponent Cu, Cd, and Pb ions

Figure 5 shows the test of the effect of pH on Cu, Cd and Pb ions removal with initial levels of 15 ppm in multicomponent synthetic wastewater, while each using a dose of 0.5 g/L of chitosan with stirring using a magnetic stirrer for 60 minutes. From the figure, the highest Cu, Cd and Pb ions removal in multicomponent synthetic wastewater is at different pH for each metal. The highest removal percentage of Cu ions were at pH 5 with a removal percentage of 89.40%, Cd ions were at pH 7 with a removal percentage of 74.80%, and Pb ions were at pH 3 with a removal percentage of 96.87%. The highest removal percentage for the metal ions in multicomponent synthetic wastewater was found in Pb ions. This shows that for multicomponent synthetic wastewater with Cu, Cd, and Pb ions content, the adsorption process has a high effectiveness on Pb metal ions removal, compared to Cu and Cd metal ions. The difference in percentage of metal removal in the multicomponent synthetic wastewater occurs due to the competition of metal ions to bond with the adsorbent.

The removal percentage of Cu ions adsorption continued to rise, but at pH 6 the percentage removal of Cu ions decreased. The highest percentage of Cu metal removal is at pH 5, under acidic conditions with aquadest solvent Cu will

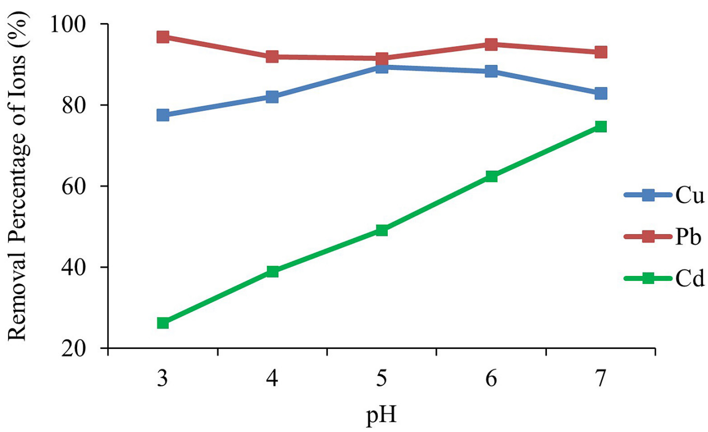

Figure 5. Effect of pH on percentage removal of Cu, Cd, and Pb ions in multicomponent synthetic wastewater

tend to form cations. Thus, under low pH conditions there tends to be competition between H^+ ions to bind to the active groups of chitosan $-NH_2$. It is this competition that causes Cu ions at acidic pH to be adsorbed slightly (Hudayni, 2018). At pH above 5, there is a decrease in the percentage removal of Cu metal absorption, this decrease can be caused by above pH 5 Cu metal species decreases and forms $Cu(OH)^+$ and $Cu(OH)_2$ precipitates.

The percentage removal of Cd ions continues to increase until the highest removal percentage is achieved at pH 7. It can be seen in Figure 5 that the percentage removal of Cd is likely to continue to increase along with the pH. This happens because the higher the pH, the concentration of H^+ ions will be smaller, so absorption will increase up to a certain pH.

The percentage of Pb removal increases along with pH and optimum absorption occurs at pH 5. Furthermore, the percentage of removal decreases with increasing pH due to the decrease in metal solubility in solution, because Pb ions experience the addition of OH so that they become PbOH and are still in the form of stable $Pb(NO_3)_2$. At pH below 5 the ability of adsorbents to adsorb metal ions is still not optimal (Lianasari et al., 2023).

Effect of chitosan dose on adsorption treatment in laboratory wastewater

To study the effect of chitosan dose on laboratory wastewater adsorption treatment, the sample of Oceanography and Marine Instrumentation Laboratory of Universitas Sriwijaya wastewater was used. This experiment was carried out by mixing chitosan with a weight variation of 0.5–2 g with 1 L of the laboratory wastewater and adjust the pH to 7, as the optimum pH which has the highest removal percentage obtained from single component and multicomponent synthetic wastewater experiments. The results are illustrated in Figure 6.

The presence of multicomponent in wastewater causes metal ion to interact each other, this is influenced by several factors, such as the number of co-cations competing on active sites, metal ions concentration, pH, nature and amount of adsorbent. The results showed that experiments on laboratory wastewater with chitosan dose variation showed that the results were not optimal in increasing the percentage removal Cu ions. Figure 6 show that the highest percentage removal of 93.75% and 28.99% was obtained for Pb and Cu ions by using chitosan dosage of 1.5 g/L and 2 g/L, respectively.

The initial concentration of Cu ions amounting to 4.45 ppm decreased to 3.16 ppm with a chitosan dose of 2 g/L which is almost close to the quality standard of environment of 3. However, if the chitosan dose was increased, the Cu ions level in laboratory wastewater may be further reduced. Furthermore, for Pb ions which have an initial of concentration 1.92 ppm, smaller decrease of 0.12 ppm was experienced that already met the environment quality standard of 1. In this study, the Cd ions in the laboratory wastewater did not experience a decrease in the concentration. This is influenced by several factors, namely strong competition between metal ions, the large atomic radius of Cd metal causes a low removal percentage, because it does not easily bind to the adsorbent in multicomponent systems like in the laboratory wastewater; and high COD levels in laboratory wastewater can cause the chitosan not only to act as an adsorbent but can also act as a coagulant. The addition of chitosan can act as a coagulant with a variety of different concentrations that can reduce COD values due to the removal of

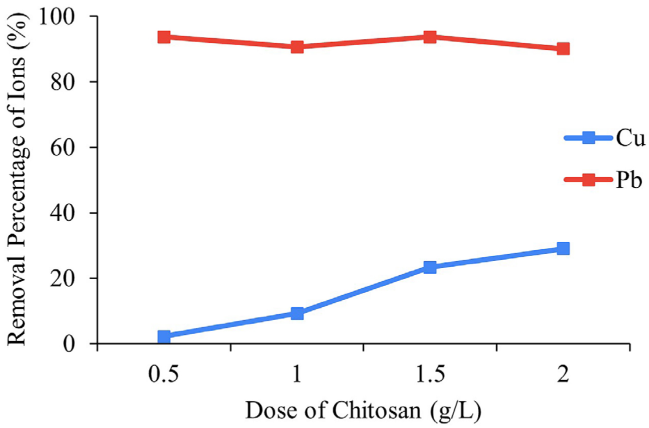

Figure 6. Effect of chitosan dosage on the removal percentage of Cu and Pb ions in laboratory wastewater

organic materials in the form of colloidal solids in wastewater (Bija et al., 2020). Other factors can be caused by the dose of chitosan that is too low, so it is necessary to increase the dose of chitosan.

In addition, the OCMC/CS-CH hydrogels exhibited significant practical adsorption efficiencies of 89.6% for Cu^{2+}, 80.5% for Cd^{2+}, and 91.8% for Pb^{2+} ions was reported by Zou in 2024, in his study about Cu, Cd, and Pb ions adsorption from wastewater using polysaccharide hydrogels made of oxidized carboxymethyl cellulose and chitosan grafted with catechol groups. Hydrogels, a three-dimensional hydrophilic polymer networks, display remarkable swelling and adsorption capabilities, making them superior to other adsorbents (Zou, 2024). Hence, it is possibe to enhance the adsorption process of chitosan in the form of hydrogels to increase the efficiency of adsorbtion which might be studied further.

CONCLUSIONS

The highest removal percentage of heavy metal ions of Cu, Cd, and Pb in single component of synthetic wastewater was achieved at pH 7. However, the highest removal percentages for the reduction of Cu, Cd, and Pb metal ions in multi-component synthetic wastewater were found at different pH for each type of metal ions, such as Cu ions at pH 5, Cd ions at pH 7, and Pb ions at pH 3; when using 0.5 g/L of chitosan, the highest removal percentage of Cu, Cd, and Pb ions of 89.40%, 74.80%, and 96.87% were found, respectively. The addition of chitosan dose from 0.5–2 g/L in the adsorption process of laboratory wastewater did not have a significant effect on the removal percentage for the reduction of Cd ions. The highest percentage removal of 28.99% was obtained for Cu ions by using chitosan dosage of 2 g/L. Moreover, the highest percentage removal of 93.75% was acquired for Pb ions by using chitosan dose of 1.5 g/L, which met the environmental quality standard. From this research, it was found that the Pb and Cu ions can be adsorbed with chitosan from fish scales. Therefore, the use of chitosan in treating laboratory wastewater, which often contains various kinds of heavy metal ions, is quite promising. The adsorption method using chitosan can be applied for the removal of other heavy metals by using the right dose of chitosan at the appropriate pH.

Acknowledgements

The author would thank to UPT Integrated Laboratory Universitas Sriwijaya for laboratory assistances. The publication of this article was funded by DIPA of Public Service Agency of Universitas Sriwijaya 2023. SP DIPA-023.17.2.677515/2023, on November 30, 2022. In accordance with Rector's Decree Number: 0187/UN9.3.1/SK/2023, on April 18, 2023.

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