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Biosorption of Cu^{2+} Metal Ions Using Immobilized Green Algae (Spirogyra Setiformis) Biomass with Sodium Silicate

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A B S T R A C T

This study aims to evaluate the performance of Spirogyra setiformis biomass immobilized with sodium silicate as a biosorbent for the removal of Cu^{2+} ions from aqueous solutions. The research investigates the effects of pH, initial ion concentration, and flow rate as independent variables, while adsorption capacity serves as the dependent variable. A column system was employed, with biosorbent characterization conducted using Fourier Transform Infrared Spectroscopy (FTIR) and adsorption capacity analyzed through Atomic Absorption Spectrophotometry (AAS) during the observation period. The results showed that the immobilized Spirogyra exhibited good stability, significant Cu^{2+} adsorption capacity, and reusability over several cycles. Application to real wastewater samples also demonstrated high effectiveness, indicating its potential as a sustainable and low-cost biosorbent for heavy metal remediation in aquatic environments.

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INTRODUCTION

The rapid advancement of science and technology has had a significant impact on industrial growth. Although this progress provides many benefits in meeting the increasing needs of human life, it has also led to adverse effects in the form of environmental pollution, particularly within aquatic ecosystems. One of the most serious environmental problems commonly encountered is heavy metal contamination, such as Pb^{2+} and Cu^{2+}, which are non-biodegradable, toxic, and bioaccumulative, posing serious threats to aquatic organisms as well as to human health. Conventional methods such as chemical precipitation, ion exchange, and membrane technologies have several limitations in terms of cost, efficiency, and sustainability. A promising alternative is biosorption, which utilizes biomass containing active functional groups to bind metal ions through mechanisms such as ion exchange, complex formation, and adsorption.

Green algae (Chlorophyta) have emerged as potential candidates for biosorption due to their abundance, environmental friendliness, and high metal uptake capacity, attributed to cell wall structures rich in functional groups. Among them, Spirogyra setiformis offers great potential as a biosorbent; however, its direct application still faces limitations such as poor mechanical stability and difficulties in separation after the biosorption process. To overcome these limitations, an immobilization method using sodium silicate was applied as a supporting matrix to enhance the stability of the biomass and enable its reuse. This study aims to evaluate the performance of immobilized Spirogyra setiformis in the biosorption of Cu^{2+} ions by examining the effects of pH, initial concentration, and flow rate, as well as assessing its stability through reusability tests and application to real wastewater samples. The results of this study are expected to contribute to the development of more sustainable and effective wastewater treatment technologies for the remediation of heavy metals in aquatic environments.

LITERATURE REVIEW

Green Algae

Green algae represent one of the most promising biosorbents due to their high metal ion adsorption capacity and natural abundance in aquatic environments. The cell wall structure of green algae is composed of cellulose, polysaccharides, glycoproteins, and pectin, which are rich in functional groups such as amine, carboxyl, hydroxyl, imidazole, and sulfate. These functional groups play a crucial role in the interaction with metal ions through mechanisms including ion exchange, complex formation, and physical adsorption. One of the most extensively studied species is Spirogyra setiformis, a green alga characterized by its spiral chloroplasts and commonly found in freshwater environments. The cell wall of S. setiformis is rich in polysaccharides and proteins, providing numerous active sites for the biosorption of heavy metal ions, including copper (Cu^{2+}) ions.

Biosorption

Biosorption is a process in which pollutants, particularly heavy metals, are removed from aqueous solutions using biomass as a biosorbent. This process occurs through various physicochemical interactions such as adsorption, ion exchange, complexation, and micro-precipitation. Functional groups present on the biomass surface, such as carboxyl (-COOH), hydroxyl (-OH), carbonyl (C=O), and amine ($-NH_2$) play a dominant role in binding metal ions. The biosorption method can be performed using two main approaches: the batch (static) system and the column (dynamic) system. In the column system, the flow rate plays a crucial role in determining the adsorption efficiency.

Metal Ion Cu^{2+}

Heavy metals are elements with a density greater than 5 g/cm^3 and are toxic even at low concentrations. One of the most widely used heavy metals in industrial applications is copper (Cu). Copper is extensively utilized in electrical and electronic industries, piping systems, and as an alloying material. However, the release of copper into the environment can lead to contamination of water and soil, posing serious risks to aquatic organisms and human health. The high concentration of Cu^{2+} in industrial wastewater necessitates the development of effective and environmentally friendly treatment technologies, one of which is biosorption using green alga.

Immobilization

Immobilization is a technique used to entrap or bind biomass onto a supporting matrix, such as sodium silicate, to enhance mechanical stability, resistance to degradation, and enable the regeneration of the biosorbent for repeated use. Immobilized biomass has been shown to improve metal ion uptake capacity, as the supporting matrix provides a more stable structure for functional groups to interact effectively with metal ions. Previous studies have shown that immobilized biomass exhibits higher biosorption efficiency compared to free biomass. When biomass such as algae, bacteria, or fungi is immobilized, typically using supporting materials like sodium silicate, calcium alginate, or natural/synthetic polymers, the process encapsulates the cells or biomass particles within a solid matrix. The matrix functions as a mechanical protective barrier, preventing the biomass from damage, disintegration, or dissolution during repeated use in wastewater systems, and enhancing its durability under continuous operation. This makes immobilization an important approach to improving the effectiveness of heavy metal biosorption.

HSAB (Hard and Soft Acids Bases)

The Hard and Soft Acids and Bases (HSAB) theory explains the tendency of interactions between metal ions and functional groups on the adsorbent surface. According to this theory, the most stable interactions are formed when hard acids bind to hard bases, or soft acids to soft bases. The Cu^{2+} ion is classified as an intermediate soft acid, which tends to interact with hard base groups such as OH or COOH that are abundantly present on the cell wall of algae. This supports the potential of Spirogyra setiformis as a biosorbent, since its functional groups can strongly interact with Cu^{2+} ions based on the HSAB principle.

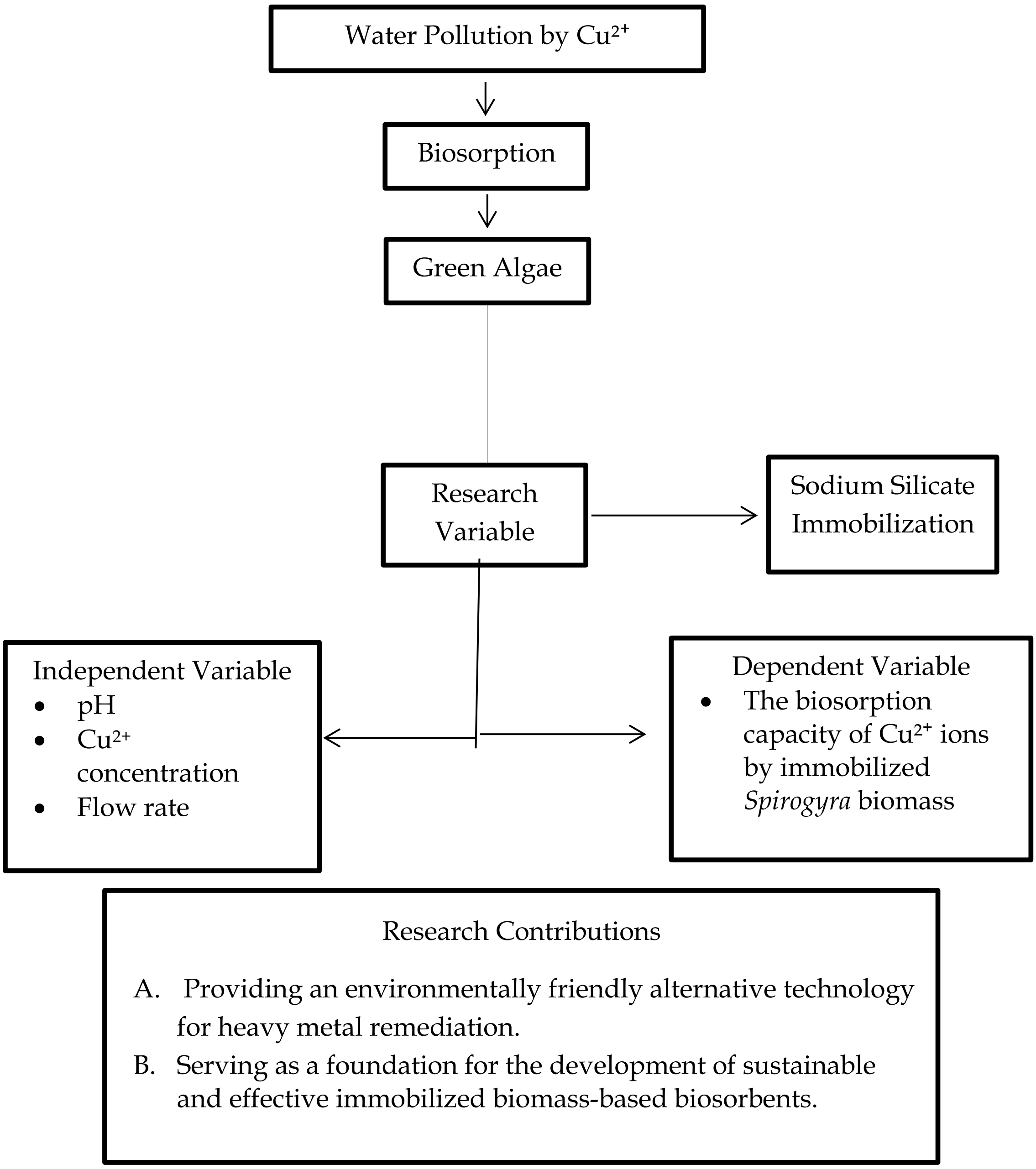

Figure 1. Conceptual Framework

METHODOLOGY

This study was conducted at the Research Laboratory of the Faculty of Mathematics and Natural Sciences, Universitas Negeri Padang, and at the LLDIKTI Region X Laboratory in Padang during the period of May–August 2025. The research focused on the biosorption process of Cu^{2+} ions using Spirogyra setiformis biomass immobilized with sodium silicate. The study population consisted of liquid waste containing heavy metal ions, while the samples used included a standard Cu^{2+} solution prepared in the laboratory and actual wastewater obtained from the LLDIKTI Region X Laboratory in Padang.

The research variables consisted of independent variables (pH, initial concentration, and flow rate), a dependent variable (Cu^{2+} biosorption capacity), and a control variable represented by the Spirogyra setiformis biosorbent. The experiment was conducted using a column method, in which the biosorbent was packed into a glass column and passed through metal ion solutions with variations in pH, concentration, and flow rate.

The research stages consisted of: (1) preparation of Spirogyra setiformis biomass through washing, drying, grinding, and acid treatment; (2) immobilization of the biomass using sodium silicate to enhance its mechanical stability; (3) biosorption testing of Cu^{2+} ions under variations of pH, initial concentration, and flow rate; (4) evaluation of biosorbent reusability through adsorption desorption cycles; and (5) application of the biosorbent to real wastewater samples to assess its effectiveness.

The concentration of metal ions after the biosorption process was measured using an Atomic Absorption Spectrophotometer (AAS), while the functional groups of the biosorbent were characterized using Fourier Transform Infrared Spectroscopy (FTIR). The obtained data were analyzed to determine the maximum adsorption capacity, the optimum biosorption conditions, and the stability of the biosorbent during repeated use.

RESEARCH RESULT

Identification of Green Algae Sample

The green algae sample collected from the Lintau Buo River was successfully identified as Spirogyra setiformis (Figure 2).

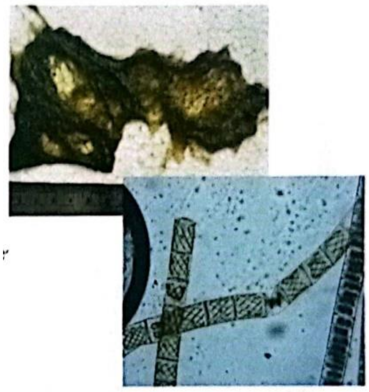

Figure 2. Identification results from the Biology Laboratory, Andalas University (UNAND)

Functional Group Characteristics of Biomass

FTIR analysis showed a shift in the OH and Si O bands, indicating the successful immobilization of biomass with sodium silicate and the involvement of functional groups in the binding of Cu^{2+} ions (Figure 3).

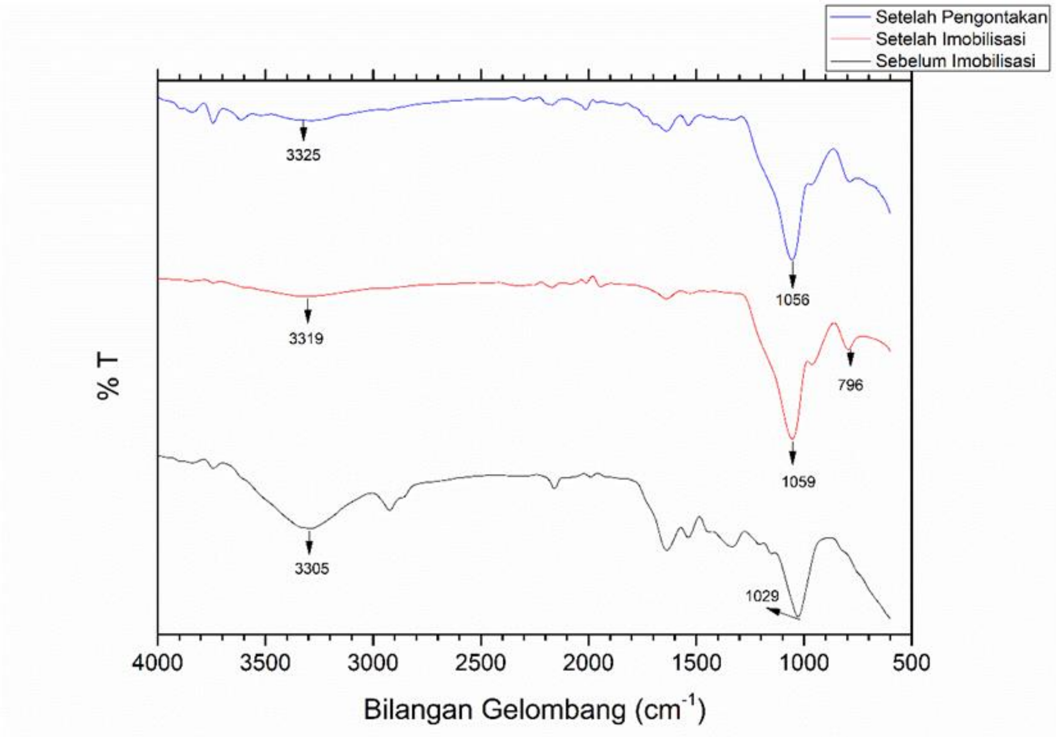

Figure 3. FTIR Spectra of Green Algae Before Immobilization, After Immobilization, and After Metal Ion Contact

Effect of pH

The optimum biosorption was achieved at pH 4 with an adsorption capacity of 3.47 mg/g (Figure 4). At very low pH values, the active sites become protonated, while at pH levels above 6, Cu^{2+} ions begin to precipitate.

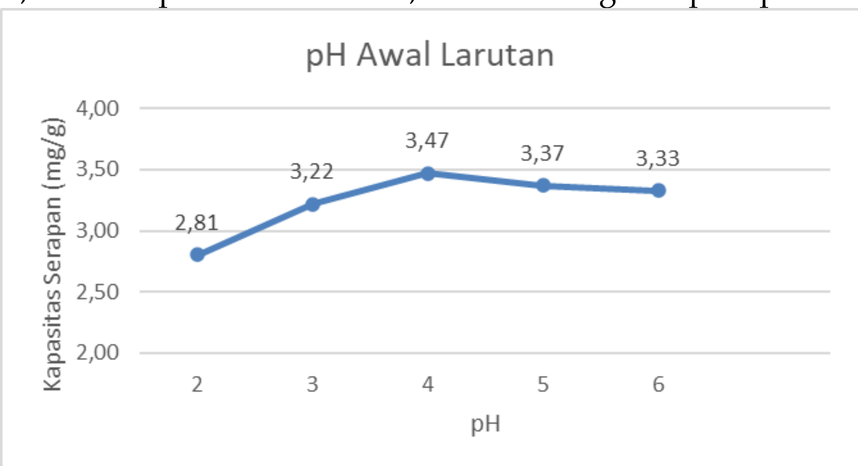

Figure 4. Effect of Initial pH of Cu^{2+} Ions on the Biosorption by Green Algae Spirogyra Setiformis Biosorbent

Effect of Initial Concentration

The adsorption capacity increased with the initial concentration up to 200 ppm (4.42 mg/g) and subsequently decreased at 250 ppm (4.08 mg/g), which was attributed to the saturation of active binding sites. The experimental data were best fitted to the Langmuir isotherm model, with a correlation coefficient (R^2) of 0.9977 (Figures 5–7).

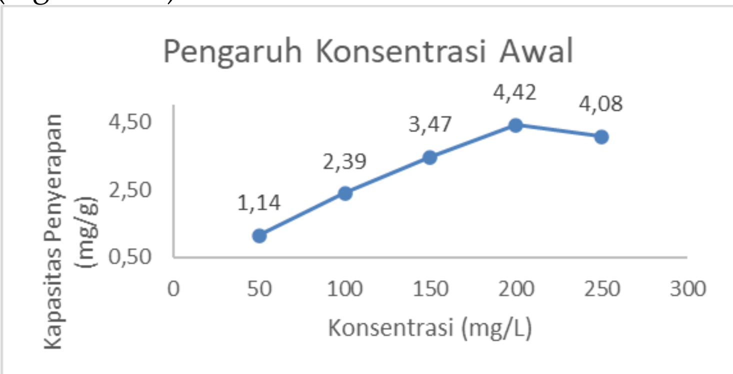

Figure 5. Effect of Initial Cu^{2+} Ion Concentration on the Biosorption Performance of Spirogyra Setiformis Green Algae Biosorbent

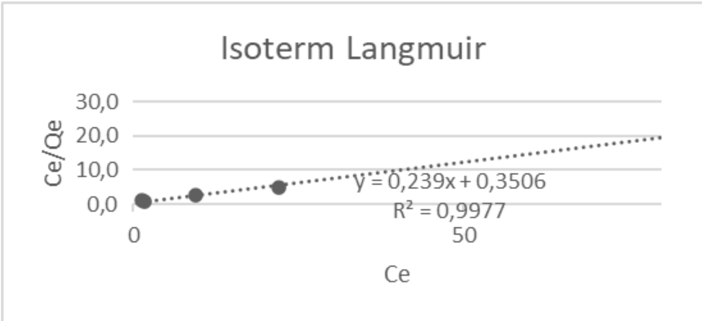

Figure 6. Langmuir Isotherm Curve for Cu^{2+} Ion Adsorption

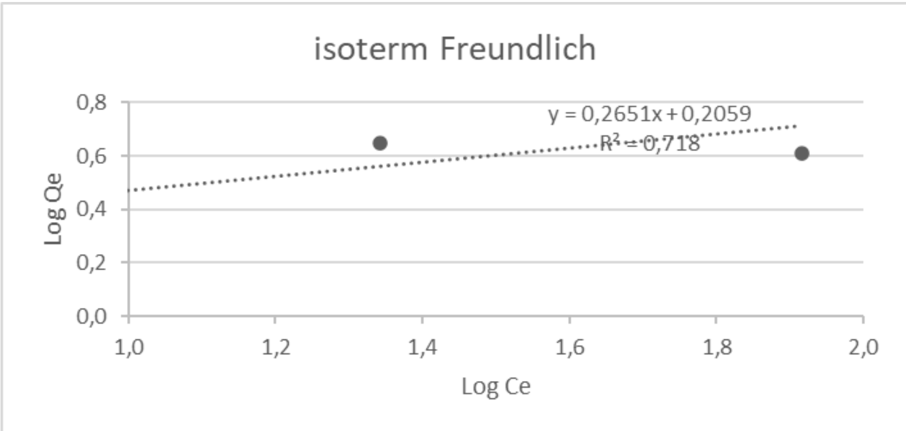

Figure 7. Freundlich Isotherm Curve for Cu^{2+} Ion Adsorption

Effect of Flow Rate

The maximum adsorption capacity (4.44 mg/g) was achieved at a flow rate of 20 drops/min. Higher flow rates reduced adsorption efficiency due to shorter contact time and the occurrence of channeling effects (Figure 8).

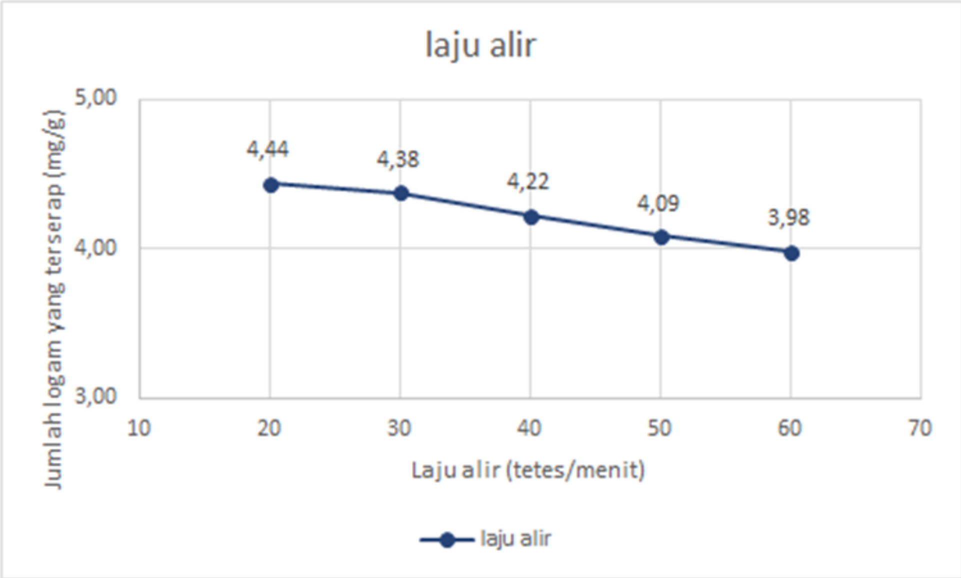

Figure 8. Effect Of Cu^{2+} Ion Flow Rate on the Biosorption Performance of Spirogyra Setiformis Green Algae Biosorbent

Effect of Pb^{2+} Coexistence

The presence of Pb^{2+} reduced Cu^{2+} uptake from 4.44 mg/g to 2.13 mg/g at a 1:2 ratio, indicating competition and selective affinity of the biosorbent toward Pb^{2+} (Figure 9).

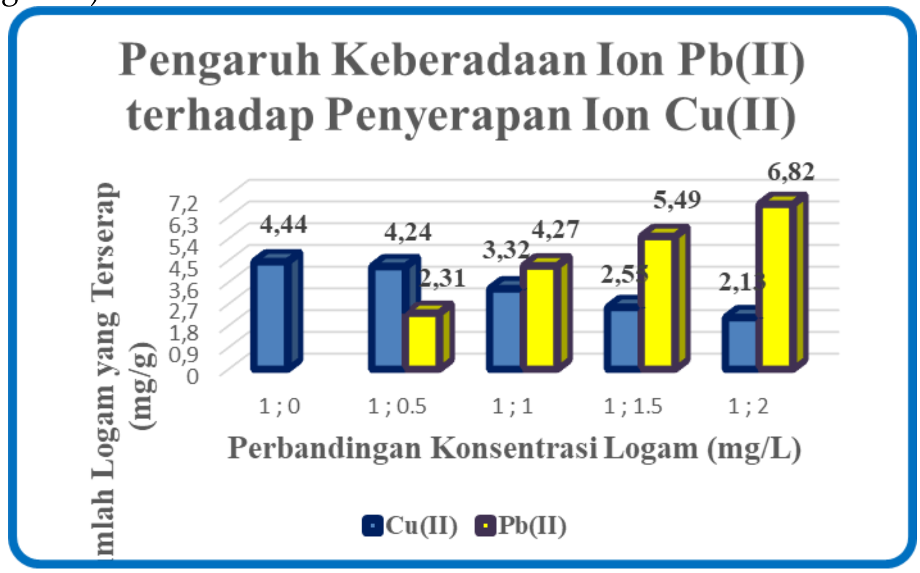

Figure 9. Effect of Pb(II) Ion Presence on the Biosorption Capacity of Cu(II) Ions

Reusability Test

The immobilized biosorbent exhibited greater stability, with adsorption capacity (Qe) decreasing slightly from 4.23 to 3.76 mg/g over cycles 1–3, whereas the non-immobilized biosorbent showed a sharp decline to 0.17 mg/g by the third cycle.

Table 1. Reusability Performance of Sodium Silicate Immobilized and Non-Immobilized Green Algae in the Biosorption Process of Cu^{2+} Ions

Cycle	Qe Cu2+(mg/g) Immobilized	Efficiency (%)	Qe Cu2+ (mg/g) Non - Immobilized	Efficiency (%) Non - Immobilized
1	4,23	85,16	1,94	38,97
2	3,90	78,40	0,96	19,31
3	3,76	75,62	0,17	3,51

Application to Real Wastewater

The biosorbent was able to reduce the Cu^{2+} concentration from 50.07 mg/L to 1.69–2.13 mg/L, with an average adsorption capacity of 1.21 mg/g. These results demonstrate the consistent performance of the biosorbent when applied to laboratory wastewater from LLDIKTI.

Table 2. Effect of Real Wastewater Samples on the Adsorption Performance of Immobilized Green Algae

Real Sample	C0 (mg/L)	C1 (mg/L)	Cu Adsorbed	qe
Limbah 1	50,07	1,69	48,38	1,21
Limbah 2	50,07	1,86	48,20	1,21
Limbah 3	50,07	2,13	47,94	1,20

DISCUSSION

The effectiveness of Spirogyra as a Biosorbent for Cu(II) and Pb(II) Ions

This study demonstrates that green algae Spirogyra setiformis immobilized with sodium silicate possesses an effective biosorption capacity for Cu^{2+} ions. The optimum conditions obtained namely specific pH, ion concentration, and flow rate align with sorption chemistry principles, where the interaction between the functional groups of the green algae and metal ions is influenced by environmental conditions. The optimal pH affects the surface charge of the biosorbent and the metal ions, thereby enhancing binding at the active sites.

Numerous studies have reported that Spirogyra biomass, whether dried or pretreated, exhibits good adsorption capacity for heavy metal ions such as Cu(II) and Pb(II). These findings are consistent with the results of Gupta et al., which demonstrated that Spirogyra efficiently adsorbs Cu(II) and Pb(II) under laboratory batch conditions. A commonly reported mechanistic explanation is the presence of functional groups on the cell wall (COOH, OH, NH, etc.) that bind metal ions through ion-exchange, surface complexation, and physical adsorption mechanisms. Characterization studies using FTIR in several investigations support the role of these groups in metal biomass interactions.

Effect of Solution pH

Solution pH is the most critical parameter affecting biosorption capacity. Most studies indicate that the optimum pH for Cu(II) and Pb(II) ranges from approximately 4 to 6. At low pH values (≤2), protonation of surface functional groups reduces binding affinity, leading to decreased adsorption, whereas at high pH, metal hydroxide precipitation may occur, potentially confounding the interpretation of pure adsorption mechanisms. Therefore, the literature recommends careful analysis of pH effects and verification of whether metal removal occurs via adsorption or precipitation.

The optimum pH was found to be 4, which aligns with reports indicating that pH is a dominant parameter, as it determines the degree of protonation of surface functional groups and the speciation of metal ions in solution. At very low pH values (≤2), the active sites become protonated, resulting in reduced adsorption capacity, while at higher pH, there is a risk of $Cu(OH)_2$ precipitation, which can obscure or be misinterpreted as adsorption.

Solution pH strongly affects the surface charge of the biosorbent and the speciation of metal ions. At low pH values (≤2), carboxyl and hydroxyl groups become protonated, inhibiting metal ion binding. Conversely, at the optimum pH range of 4–6, protonation decreases and electrostatic interactions are strengthened, enhancing biosorption efficiency. However, at pH values above 6, metals such as Cu(II) can form $Cu(OH)_2$ precipitates, leading to an apparent reduction in actual adsorption.

The presence of Pb^{2+} ions in the system demonstrated competitive behavior at the active sites of the biosorbent. Pb^{2+} ions preferentially bind due to their smaller hydrated radius, lower hydration energy, and chemical properties that are more compatible with the functional groups of the algae. This indicates the selectivity of immobilized green algae toward specific metal ions, which can be exploited for the separation or treatment of mixed-metal wastewater. These findings are consistent with the hard soft acid base (HSAB) principle in coordination chemistry, which explains the affinity of metal ions for particular functional groups. Changes in pH also affect the surface charge and the availability of active sites, thereby influencing the adsorption capacity.

Biomass Immobilization

Biomass immobilization (e.g., in matrices such as sodium silicate/sol gel or alginate) enhances handling convenience, mechanical stability, and the reusability of the biosorbent. Reviews and technical studies indicate that immobilized biomass systems reduce the loss of fine particles and facilitate solid liquid separation while retaining many of the original biosorption properties of the biomass. Different immobilization methods (silica sol gel, alginate, or other polymers) involve trade-offs between initial adsorption capacity and ease of recovery. In the case of immobilization with sodium silicate, both historical literature and recent experimental studies demonstrate that this approach is feasible and often improves practical performance in batch or column treatment systems.

Reusability test data clearly demonstrate that immobilization enhances mechanical stability and better preserves adsorption capacity over repeated cycles compared to free biomass. Related literature on immobilized microalgae, including the use of silica/sol gel matrices, reports similar operational advantages: ease of solid liquid separation, reusability, and reduced loss of fine particles.

Furthermore, the immobilization of green algae with sodium silicate has been shown to enhance biosorbent stability. Reusability tests indicate that the immobilized biosorbent can maintain its adsorption efficiency over multiple cycles, whereas non-immobilized green algae experience a significant decline. This confirms that immobilization not only preserves the physical integrity of the biosorbent but also protects the active sites from degradation or loss of adsorption capacity during repeated use. Zhang et al. (2023) reported that, although the total adsorption capacity may slightly decrease due to diffusion limitations, the advantages of immobilization in terms of biosorbent stability and reusability are far greater in industrial scale applications.

Competition Between Cu(II) and Pb(II) in a Binary Solution

In a binary system, competition between ions alters the adsorption capacity of each ion compared to single-ion experiments. Several studies have shown that Pb(II) often exhibits higher affinity toward many biosorbents than Cu(II), so in the presence of Pb(II), the adsorption of Cu(II) is typically "disrupted," resulting in a reduced Qe for Cu^{2+}. However, this effect depends on factors such as pH, initial concentration, concentration ratio, and the surface properties of the biosorbent. Isotherm models for binary systems (e.g., extended Langmuir, Sips, or predictions based on single-component isotherms) are commonly employed to model competition and predict adsorption capacities under mixed conditions.

According to Kajeiou et al. (2020), the biosorption of Cu^{2+} and Pb^{2+} by microalgal biomass is a spontaneous process (negative $\Delta G°$) and endothermic (positive $\Delta H°$), indicating that higher temperatures enhance the diffusion of metal ions toward the active sites of the biosorbent.

Biosorption tests on real wastewater samples confirm that the immobilized green algae biosorbent exhibits consistent performance in complex environments. Effectiveness on real wastewater is important as it provides a realistic assessment of the biosorbent's application in industrial or laboratory wastewater treatment, compared to tests conducted in simpler standard solutions. Overall, this study demonstrates that green algae biosorbents immobilized with sodium silicate have high applicative potential for metal wastewater treatment, exhibiting adequate stability, selectivity, and effectiveness.

Microalgae have demonstrated considerable efficiency in reducing heavy metal concentrations through biosorption and bioaccumulation mechanisms. The main challenges include the complexity of real wastewater (e.g., presence of competing ions, suboptimal pH conditions, high organic content), biomass stability, and the need for integration into existing treatment system.

CONCLUSIONS AND RECOMMENDATIONS

1. The optimum conditions for Cu^{2+} biosorption using Spirogyra setiformis immobilized with sodium silicate were pH 4, 200 ppm concentration, and a flow rate of 20 drops/min.
2. The presence of Pb^{2+} reduced Cu^{2+} biosorption capacity due to competition at active sites, with Pb^{2+} showing stronger affinity for immobilized green algae. This indicates that the biosorbent has higher selectivity toward Pb^{2+} compared to Cu^{2+}.
3. Immobilization with sodium silicate significantly improved biosorbent stability, as confirmed by reusability tests over three cycles (efficiency 86–76%), whereas non-immobilized algae exhibited a drastic decline (38–3%).
4. Biosorption tests on real wastewater demonstrated relatively consistent Cu^{2+} uptake (~1.21 mg/g), indicating stable and effective biosorbent performance in complex environments.

ADVANCED RESEARCH

Future studies should explore other species of green algae with different immobilization methods to compare biosorption effectiveness. It is recommended to test biosorption on other metal ions and different types of wastewater to obtain more comprehensive and applicable results for practical implementation.

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