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Synthesis of Bio Adsorbent from Parthenium Weed (*Parthenium hysterophorus***) Plant Biomass and Its Application in Removal of Hexavalent Chromium Ion from Aqueous Solution**

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Abstract

Rapid industrialization leads to serious environmental hazards due to increasing the release of toxic metal into the environment. The study focuses on utilizing *Parthenium hysterophorus* weed biomass as a novel bio adsorbent for removing hexavalent chromium (Cr (VI)) ions from aqueous solutions. The bio sorbent was characterized using FT-IR provides valuable insights into the chemical interactions between the functional groups of the bio sorbent and the Cr (VI) ions, shedding light on the mechanism of adsorption and the role of specific functional groups in the removal process. Batch adsorption experiments determined optimal conditions: pH 2, 90 minutes contact time, 1g adsorbent dosage, and 150 mg/L initial metal ion concentration. The study concludes that Parthenium bio sorbents are effective, low-cost alternatives for Cr (VI) ion removal, showcasing high bio sorption capacity and potential for further exploration with other heavy metals. Suggesting further exploration of its scalability and practical application for environmental remediation. Recommendations include optimizing the bio sorption process and investigating bio sorbent regeneration for sustainable water treatment solutions.

Keywords: Bio Sorption; Hexavalent Chromium; *Parthenium hysterophorus*

Introduction

Heavy metal pollution has been one of the most challenging environmental problems due to their toxicity, persistence and bioaccumulation tendencies [1]. With the rapid development of many industries such as, mining, surface finishing, textile, fertilizer, pesticide, metallurgy, iron and steel, electroplating, leather, photography, and metal surface treating, wastes containing metals are directly or indirectly being discharged in to the environment causing serious environmental pollution and even threating human life [2]. In Ethiopia, leather industry is one of the most pollutants of heavy metals that use chromium to convert animal skins to leather. Currently, there are 20 operational tanneries that turn out wastes directly into the nearby water bodies like the industrial activities. This makes industrial and chemical pollution to become the major problems in the country [3].

Worldwide, it is estimated that the industry is responsible for dumping 300- 400 million tons of heavy metals, solvents, toxic sludge, and other wastes into water in each year [4]. Among the different heavy metals, chromium, specifically

the hexavalent species, is very toxic to biological system, which commonly associated with cancer and hemorrhage in the digestive tract [5]. In the chromium tanning process, the leather takes up only 60–80% of the applied chromium, and the rest is usually discharged into the sewage system causing serious environmental impact. Chromium ions in liquid tanning waste occurs mainly in trivalent form, which gets further oxidized to hexavalent chromium form, due to the presence of organics [6]. The latter form is of particular concern due to its greater toxicity. Unlike organic pollutants, heavy metals are non-biodegradable, they only changed from one oxidation state to another, accumulate in living organisms via the food chain, and some of these metals are extremely toxic in relatively low concentrations [7].

Therefore, it becomes importance to develop technologies that result in effective removal of heavy metals from industrial effluents. Several techniques have been designed for heavy metal removal from aqueous solutions such as, ion exchange, chemical precipitation, filtration, coagulation, membrane technologies, reverse osmosis and commercial activated carbon. However, these methods have many disadvantages such as, incomplete metal removal, high reagent and energy requirements, generation of toxic sludge, or other waste products that require disposal or treatment. These shortcomings have made researchers seek alternative techniques for heavy metal remediation, which can be costeffective and eco-friendly.

According to a recent study by Kim, et al. [8] biological treatment methods for heavy metal removal involve the utilization of living organisms such as algae, fungi, bacteria, and plants to absorb, accumulate, or transform heavy metal contaminants in the environment. These organisms possess specific cell wall constituents and functional groups that enable them to effectively bind and sequester heavy metals. For instance, fungi serve as efficient biosorbents due to their high cell wall material content, while bacteria utilize anionic functional groups in their cell walls for metal binding. Phytoremediation, another biological method, employs plants to eliminate contaminants from soil or water by either absorbing and storing the pollutants or facilitating their degradation. These biological approaches provide a cost-effective and environmentally friendly alternative to traditional methods of heavy metal remediation.

According to a recent study by Patel, et al. [9] plant biomass has emerged as a promising option for the development of cost-effective and environmentally friendly treatment of industrial effluents through a process known as biosorption. Biosorption involves the passive binding of heavy metals to non-living biomass from an aqueous solution, where the removal mechanism is not metabolically controlled. The process of metal ion bio sorption may involve electrostatic

interactions between metal cations and negatively charged sites, such as phosphoryl, phosphate, carboxyl, sulfate, amino, and hydroxyl groups, on the surface of the bio sorbent. Bio sorption of heavy metals has been proven very promising in the removal of contaminants from aqueous solutions and effluents. The adsorption technique has been proved as one of the excellent method to treat industrial waste effluents, offering significant advantages like low-cost, availability, profitability, easy operation and efficiency and regeneration of biosorbents with possibility of metal recovery [10].

In our study, we examined the adsorption efficiency of *Parthenium hysterophorus* plant using their leaves as a dried-biomass for adsorption process. This biosorbent is not yet explored for any adsorption of Hexavalent Chromium locally. In the present study, an attempt was made to use the leaves of *Parthenium hysterophorus* plant as a potential, novel, environmentally friendly and low-cost adsorbent for the remediation of heavy metal ion from aqueous solution. The kinetics models and equilibrium interactions sorption were applied in describing sorbate-sorbent interactions and kinetics of biosorption process. Therefore, the objective of this study is synthesis of bio adsorbent from parthenium weed (*Parthenium hysterophorus*) plant biomass and its application in removal of hexavalent chromium ion from aqueous solution.

Statement of the problem

Due to modernization, the industrial use of metals especially, heavy metals have been alarmingly, thus becoming of prior concern because of their toxicity to all life forms. Due to the limitation of conventional treatment methods like incomplete metal removal, high energy and nutrient requirements, expensive (high cost), and generation of toxic sludge, there is a need for searching an alternative methods for the effective removal of heavy metals by using novel dead-biomass especially, from plant origin, which is cost effective, relatively efficient and possibility of biomass regeneration and metal recovery [11].

Material and Methods

Materials and Chemicals

Parthenium hysterophorus weed biomass: Purity: Ideally, the biomass should be free from contaminants and impurities. Specification: The biomass should be dried thoroughly to ensure consistent adsorption properties.

Potassium dichromate (K2Cr2O7):

Purity: Reagent grade or higher (typically \geq 99% purity). Specification: Ensure the compound is dissolved completely in distilled water to prepare the stock solution.

Hydrochloric acid (HCl) and Sodium hydroxide (NaOH): Purity: Analytical grade or higher (typically $\geq 95\%$ purity). **3**

Specification: Use precise volumes to adjust the pH of solutions accurately.

Nitric acid $(HNO₃)$: Purity: Reagent grade or higher (typically ≥ 65% purity). Specification: Thoroughly rinse glassware with the acid solution to ensure cleanliness.

Diphenyl carbazide: Purity: Analytical grade or higher (typically $\geq 98\%$ purity).

Specification: Follow the recommended analytical method for analyzing residual chromium ion concentration.

Analytical siever (100 μm): Specification: Ensure the sieve is made of stainless steel or suitable material for accurate particle size separation.

Polyethylene bags: Specification: Use airtight and moistureresistant bags to preserve the powdered biosorbents effectively.

Distilled water: Purity: Distilled water should be of analytical grade or higher to prevent contamination. Specification: Use freshly distilled water to avoid any impurities affecting the experimental results.

Sample Collection and Preparation for Biosorbents

The leaves of *Parthenium hysterophorus* weeds was collected from Addis Ababa Science and Technology University Campus, Kilinto, Addis Ababa, latitude of 1 N and longitude 38.810 E. The plants was authenticated at Ethiopian Biodiversity Institute, Kebena, Addis Ababa. It was washed with tap water followed by deionized water to remove dirt and dust. The plants was chopped in to small pieces and dried in shadow areas for six days. Then, the dried plant leaves (biosorbents) were grinded in to fine powder by using grinder. The powder was then sieved through analytical siever (100 µm) to obtain biosorbents with different size fractions and was preserved in polyethylene bags until use for biosorption process.

Preparation of Chromium (Vi) Stock Solution

All glassware and containers were washed in detergent and tap water and rinsed with distilled water and thoroughly soaked in 2N HNO₃ and then rinsed with distilled water and dried. Analytical grade reagents were used. A stock solution of 1000 mg/L of Cr (VI) was prepared by dissolving 2.827g of potassium dichromate in one liters of distilled water, shaking it for 30 minute at 150 rpm to dissolve completely (APHA, 1986). The solution was filtered using filter paper. The normal pH of the solution was 5.45 and adjusted to the desired value using HCl and NaOH. The stock solution was diluted to desired concentration.

Batch Adsorption Studies

According to the study by Chen, et al. [11] the study conducted by a standard experimental design for batch sorption studies. They utilized triplicate samples for each condition tested, ensuring that three samples were taken for the removal of Cr (VI) ions using the bio sorbent under various experimental conditions. This triplicate sampling approach was implemented to enhance the reproducibility and reliability of the results obtained during the removal process.

By conducting triplicate experiments for each condition, a total of nine samples were analyzed in the study for the removal of Cr (VI) ions using the bio sorbent. This comprehensive approach allowed the study to gather robust data and draw meaningful conclusions regarding the effectiveness of the bio sorbent in removing Cr (VI) ions from aqueous solutions.

Overall, the use of triplicate samples and conducting multiple experiments for each condition ensured the accuracy and consistency of the experimental results, contributing to the reliability of the findings presented in the study by Chen, et al. [11].

The experimental design used in the study is a batch sorption experiment, where the bio sorbent material is exposed to a known concentration of Cr (VI) solution under controlled conditions such as pH, contact time and adsorbent dosage. By varying these parameters systematically and analyzing the adsorption capacity of the bio sorbent, the study were able to determine the optimal conditions for effective removal of Cr (VI) ions.

Batch adsorption experiments was carried out using 1g of powdered leaves of *Parthenium hysterophorus* for the adsorption of Cr (VI) (100 mg/L) ion in 250 ml conical flasks separately. Then, the mixtures were shakedusing incubator shaker at a speed of 150 rpm at 25oC for 90 minutes. After the required time interval 10 ml from the mixture was withdrawn and centrifuged and then, filtered using whatmann no. 1 filter paper. Then, the filtrates were analyzed for the presence hexavalent chromium ion that residues in the solution. The residual concentration of chromium ion left in solution was analyzed using Diphenyl carbazide at 540 nm in a UV- spectrophotometer (Biochrom model). The effects of various parameters such as pH, adsorbent dosage, contact time and initial ion concentration were investigated by varying them at a certain range and by keeping the remaining parameters constant [12]. The pH, adsorbent dosage, initial metal ion concentration, and contact time were investigated by varying the range from 1-9, 0.1 - 1g, 10 -150 mg/L, 15- 90 minutes respectively. According to Patel, et al. [13] the removal percentage was calculated by using the equations;

Percentage Removal **of Cr (VI) = (C0- Ce) /C0 (100)** Where,

C0 and Ce are initial and equilibrium concentration of metal ions

 The specific metal uptake after adsorption was calculated by the equation;

Qe= (C0- Ce) v/W

Where

Qe is the adsorption capacity in (mg g−1),

 \mathbf{C}_0 and \mathbf{C}_e are initial and equilibrium concentrations (mg L−1) of Cr (VI) in the solution,

V is the volume (L) ofCr (VI) solution taken for the experiment and

M is the mass (g) of adsorbent used.

Characterization Study

FT-IR characterizations were carried out before and after the adsorption in the range of 400 to 4000 cm-1 in 65-FT-IR (PerkinElmer) using KBr techniques to study the functional group present in the natural biomass and to examine which functional group binds to the Cr (VI) ion. Functional group binds to Cr (VI) ion was determined based on the change in peak intensity. The responsible functional groups present on the 0biosorbents were determined and interpreted based on the following table.

Table 1: Bioactive component and functional groups with corresponding wave numbers cm^{-1} [14].

Results and Discussion

Ft-Ir Analysis

The FT-IR analysis conducted on the biosorbent derived from Parthenium weed biomass before and after the adsorption of Cr (VI) aimed to identify the functional groups involved

in the adsorption process. The analysis revealed specific peaks corresponding to various functional groups. Before adsorption, peaks at 515 cm^-1 indicated alkyl halide (C-Br, S-S), 750 cm^{λ}-1 corresponded to C-H, 1043 cm λ -1 to C=0, 1429 cm^{\land}-1 to C-C stretch, 1660 cm \land -1 to N-H, and 3400 cm^-1 to O-H groups. After adsorption, changes were observed in the peak intensity of the C-C functional group, while the -OH functional group peak intensity remained unchanged. The FT-IR spectra of the natural biosorbent and the metal-loaded form displayed signals for C-H, O-H, C=O, N-H, and C-C functional groups, with alterations in peak intensity observed in the metal-loaded biosorbent.

Figure 1: Image of FT-IR analysis of the *Parthenium hysterophorus* adsorbent before and after adsorption.

The FT-IR analysis of the *Parthenium hysterophorus* weed biomass before and after the adsorption of Cr (VI) ions, the following functional groups can be associated with specific bands or wave numbers:

• Alkyl halide (C-Br, S-S): Peak at 515 cm^-1 $C-H$: Peak at 750 cm^-1 $C=O$: Peak at 1043 cm^-1 • C-C stretch: Peak at 1429 cm^-1 $N-H$: Peak at 1660 cm^-1 $O-H:$

Peak at 3400 cm^-1

It is important to note that the change in peak intensity or position after the adsorption of Cr (VI) ions can provide insights into the interaction of these functional groups

with the metal ions. By associating the peaks with specific functional groups, the study can accurately identify the chemical entities involved in the adsorption process, contributing to a better understanding of the mechanism of Cr (VI) ion removal by the *Parthenium hysterophorus* weed biomass.

This analysis provides valuable insights into the chemical interactions between the functional groups of the biosorbent and the Cr (VI) ions, shedding light on the mechanism of adsorption and the role of specific functional groups in the removal process.

Xrd Analysis Of *Parthenium hysterophorus* **Adsorbent**

The X-ray diffraction (XRD) technique is utilized as a powerful analytical tool to characterize the biosorbent's properties. The XRD spectrum of the Cr (VI)-unloaded biosorbent reveals minerals such as Calcium Sulfate Hydrate ($CaSO_4 \cdot 0.15H_2O$), Aluminum Oxide Hydrate (AlOOH), and Calcium Aluminum Oxide ($Ca₃Al₁₀O₁₈$), with diffraction peaks at 2 θ angles of 22.7756, 21.8977, and 20.3618, respectively (Figure 3). For the Cr (VI)-loaded biosorbent, minerals including Calcium Iron Oxide ($Ca₂Fe₉O₁₃$) and Calcium Silicate Hydrate (Ca₂SiO₄·H₂O) appear, showing diffraction peaks at 2θ angles of 29.4189, 22.7756, and 23.0350, respectively **(Figure 2)**. The broad peaks in the spectrum of the Cr (VI)-unloaded biosorbent suggest a predominant crystalline structure, indicating its effectiveness for Cr (VI) biosorption onto the surface of *Parthenium hysterophorus* weed biomass.

Sem Studies of *Parthenium hysterophorus* **Adsorbent**

The surface morphology of the *Parthenium hysterophorus* adsorbent was examined using scanning electron microscopy (SEM) (INSPECT F50, Japan). The SEM micrograph of the

biosorbent before and after biosorption of Cr (VI) showed morphological changes in the surface structure. The morphology of the biosorbent before adsorption was rough with numerous heterogeneous pores through which the Cr (VI) can be trapped and adsorbed. While after Cr (VI) sorption, the surface of the biosorbent reveals a smooth texture indicating the entrapment of Cr (VI) (Figure). Similar changes in the surface porosity were observed in Gelidium due to methylene blue adsorption and tartrazine and allura red adsorption on Spirulina platensis.

Figure 3: SEM electron micrograph of hysterophorus adsorbent with (A) Cr (VI)- unloaded and (B) Cr (VI) loaded (INSPECT F50, Japan).

Effect of pH

Maximum adsorption capacity was obtained at pH= 2 for *Parthenium hysterophorus* with the removal percentage of 95.1%. Maximum adsorption was occurred in acidic conditions.

Figure 4: Effect of pH on adsorption of chromium (VI) using *Parthenium hysterophorus* biosorbent. (Initial metal ion concentration = 100 mg/L , Contact time = 90 minute, biosorbent= 0.5g, Temperature = 250C, Agitation speed= 150rpm).

It has been reported by earlier researchers [15], that biosorption of heavy metal is strongly pH sensitive. Our

result revealed that the optimum pH for the adsorption of Cr (VI) ion was 2 for Parthenium weed, which was highly acidic. This is because, when the pH was lowered, the numbers of H+ ions were present which in turn increased their coordination with the functional group present in the biosorbent. This resulted in an increase adsorption of negatively charged Cr species. When the pH was increased, the numbers of -OH ions were increased and the net charge on the biosorbent surface became negative. This negatively charged species caused hindrance for the sorption of Cr species through repulsion, which was the reason for the sharp decrease in Cr (VI) adsorption at higher pH values [16].

Effect of Adsorbent Dosage

The maximum adsorption capacity was attained at 1g for *Parthenium hysterophorus* biosorbent.

chromium (VI) using *Parthenium hysterophorus* biosorbent (pH=2, Cr (VI) = 100 mg/L, T= 250C, Agitation speed= 150rpm, Agitated time= 90 minutes).

An increase in biomass dosage generally increases the biosorption of metal ions due to an increase the surface area of the biosorbent. As it can be seen in figure 2 an increase the biosorbent dosage of Parthenium biosorbent from 0.2 to 1g, the removal percentage increased to 75.77% to 84.39% and became decreased when increase the dosage. The initial rapid increase observed could be due to the increased availability of binding sites and surface area which makes the adsorption of ions easy until equilibrium was reached [17]. The subsequent decrease in the removal efficiency could be due to the metal ions to be sorbed could not cover the available sites or due to aggregation or overlapping of the adsorption sites.

Effect Of Contact Time

 The effect of contact time was investigated from 15 to 90 min and maximum adsorption was obtained at 90 min with the removal percentage of 83.1% for *Parthenium hysterophorus* biosorbent respectively. After increasing the contact time the adsorption capacity became decreased.

Figure 6: Effect of contact time on adsorption of Cr (VI) ion using *Parthenium hysterophorus* biosorbent (pH=2 and dosage= 1g, Cr (VI) = 100 mg/L, Agitation= 150rpm, Agitation time = 90 minutes, Temperature = 25° C).

The extent of adsorption efficiency increases sharply with time and attains equilibrium at about 90 minutes Parthenium biosorbent with the removal percentage of 83.1%. As the contact time increased from 15-90 minutes, the removal efficiency increases from 68.75% to 83.1% for Parthenium weed. The subsequent reduction in the removal efficiency could be due to the saturation of the active site on the biosorbent. Findings in this study are similar to others reported in literature [18].

Effect of Initial Metal Ion Concentration

The effect of initial metal ion concentration on removal percentage and specific metal uptake is represented in fig.4. Initially when the metal ion concentration increases the percentage removal and the specific uptake increase up to 150 mg/L and become decreased with subsequent increase in metal ion concentration using parthenium bio sorbents.

Figure 7: Effect of initial Cr (VI) ion concentration for biosorption of Cr (VI) using Parthenium biosorbent (pH = 2, dosage= 1g, contact time= 90 minutes, Agitation speed= 150rpm, Temperature = 25°C).

The effect of initial metal ion concentration was investigated from 10- 150 mg/L and maximum adsorption was attained at 150 mg/L for Parthenium biosorbent. When the metal ion concentration increased from 10-150 mg/L, the removal percentage also increased from 91% to 98% for Parthenium weed and becomes decreased on further increasing the metal ion concentration. The percentage removal of Cr (VI) decreased with increasing initial Cr (VI) concentrations due to the saturation of adsorption sites on the biosorbent and hence adsorption sites became the limiting factor [19]. When we compared the two biosorbents they have more or less similar efficiencies. Parthenium biosorbents has high removal efficiency at pH 2.

Biosorption Isotherms

To understand the mechanism of biosorption of Cr (VI) by Parthenium weed biosorbent the equilibrium data was evaluated by Langmuir and Freundlich isotherm models.

Figure 9: Graphical representation of Freundlich Isotherm model using *parthenium hysterophorus* biosorbents.

Kinetics Models

The results of pseudo first order and pseudo second order kinetics models are shown below. Both pseudo first order and pseudo second order was fitted for this adsorption.

Figure 10: Graphical representation of pseudo first order adsorption kinetics of Cr (VI) using Parthenium biosorbent.

Figure 11: Graphical representation of Pseudo second order adsorption kinetics of Cr (VI) using Parthenium biosorbent.

For the adsorption isotherm model, the values of regression coefficients obtained from this model were used as the fitting criteria for these isotherms. The equilibrium data was best fitted to the Freundlich isotherm model ($R2 =$ 0.929 for *Parthenium hysterophorus* biosorbents), indicating heterogeneous surface energies of multilayer adsorption. For the reaction kinetics model pseudo second order best describes the reaction kinetics for parthenium biosorbents.

Conclusion and Recommendation

Conclusion

The study focused on the development of low-cost biosorbents from the waste biomass of Parthenium weed and applied for the removal of Cr (VI) ion from aqueous solution. The biomass was characterized by FT-IR and it was established that carboxyl, amine, carbonyl were involved in the adsorption. The various kinetics parameters were investigated and the optimum pH, contact time, adsorbent dosage and initial metal ion concentration were 2, 90 minute, 1g and150 mg/L for Parthenium biosorbent. At these conditions the maximum percentage removal Cr (VI) ion was found 98.7%. The data was fitted well to the Freundlich isotherm model and the reaction kinetics followed pseudo second order kinetics. Based on these results the study can be concluded that Parthenium biosorbents are an effective and alternative biomass for the removal of Cr (VI) ion from aqueous solution because of its biosorption capacity, easily available and low-cost nature.

Recommendation

In future studies, explore using the plant biomasses in continuous column systems to test their efficiency in removing hexavalent chromium and other toxic heavy metals. BET and proximate & ultimate analysis of the biomass will be characterized. Additionally, address the regeneration of the adsorbent material for sustainability.

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Appendix

Table 2: Langmuir isotherm model for *Parthenium hysterophorus* biosorbent.

Table 3: Freundlich isotherm model using *Parthenium*

hysterophorus biosorbent

Table 4: Pseudo first order kinetics constants for Cr (VI) adsorption using *Parthenium hysterophorus* biosorbent.

Table 5: Pseudo Second order kinetics constants for Cr (VI) adsorption using *Parthenium hysterophorus* biosorbent.

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