

DEVELOPMENT AND UTILIZATION OF AMINO ACID MODIFIED BIOPOLYMER-BASED RESIN TO REMOVE Cr(VI) IONS FROM ITS AQUEOUS SOLUTION

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ABSTRACT

Industrial wastewater contaminated with heavy metals like chromium poses a significant environmental threat due to their toxicity. The current study investigates biosorption of Cr (VI) ions by Tamarind triazine-2-amino-3-mercaptopropanoic acid (TTAMP) resin. Tamarind kernel powder contains excellent water adsorption capacity and high viscosity. In this study, the batch adsorption technique has been applied for the removal of Cr (VI) using TTAMP resin. For examination of the pH, contact time, and metal ion concentration impact on biosorption, Langmuir and Freundlich adsorption have been used. At pH 2, TTAMP resin was found to have a 93.4% removal efficiency of Cr (VI). This resin improves green technology, makes the planet greener, fosters a healthy environment and way of life by removing harmful heavy metals from industrial effluent and subsequently can help in wastewater management (SDG-6).

Keywords: Heavy Metals, Cystine, Polysaccharide, Chelating Resin, Tamarind Kernel Powder, and Industrial Wastewater Treatment.

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INTRODUCTION

The leading challenges to the ecological community are environmental degradation caused by urbanization and industrialization.¹ Metal ions, in particular, are emitted as a result of numerous anthropogenic factors, resulting in a wide range of severe environmental repercussions.² The usage of harmful heavy metals in many industries has increased significantly in recent decades, with many of them entering the natural environment and harming the plant and animal kingdom.³ Due to the indestructible nature of heavy metals⁴, they persist in the environment long after discharge and pose severe risks to human health at elevated concentrations, including carcinogenicity. This environmental longevity has driven researchers and environmentalists to develop viable water purification strategies.⁵ While conventional filtration techniques relying on chemical precipitation are widely available, they are constrained by the need to strictly control parameters such as pH, temperature, and ion concentration.⁶ Also, a major drawback of these traditional methods is the generation of large volumes of toxic sludge and colloidal byproducts, which often exceed the volume of the target heavy metals themselves.⁷ To remove heavy metals from industrial effluent, several adsorbents have been analysed, such as fly ash⁸, zeolites⁹, biomass, polymers, activated charcoal, and graphene oxide.¹⁰ Other techniques, such as reverse osmosis, electrolysis, phytoextraction, and ultra-filtration, have been employed to lower the concentration of metal ions in water. A highly toxic, carcinogenic, and mutagenic pollutant, hexavalent chromium, is frequently created by industrial processes like steel production, leather tanning, and electroplating. Several jurisdictions set drinking water guidelines of 0.05 mg L⁻¹. Chromium exists in two forms: less mobile Cr (III) and the more mobile and toxic Cr (VI), and many standards have discharge limits of ≤ 0.1 mg L⁻¹ (Cr total) and ≤ 0.05 mg L⁻¹ (Cr (VI)).¹¹ Significant health issues, like lung cancer, liver and kidney damage, skin ulcers, and respiratory issues^{12,13}, are brought on by exposure. Conventional treatment methods involve physicochemical processes like coagulation and flocculation⁶ using diverse chemical reagents (ferric or aluminum chloride, polyelectrolytes, etc.)¹⁴ generate large amount of sludge. With strict changes in the regulations of wastewater disposal and rising demands to enhance water quality indicators, it is essential to develop and implement more effective and efficient processes such as ion exchange, ultrafiltration, reverse osmosis, chemical precipitation, and electrochemical technologies.¹³

There are specific benefits and some drawbacks to each of these therapeutic modalities. Therefore, the low cost, renewable nature, and adjustable surface chemistry of biosorbents derived from agro-industrial wastes make them appealing. Although it doesn't remove bacteria or viruses like RO does, ion exchange is a powerful complementary or primary tool for specific needs because it is highly versatile, selective, and efficient at removing specific dissolved ions (like hardness minerals, heavy metals, and nitrates) that other methods miss, producing pure water for homes and industries (boilers, electronics), cost-effective due to regenerable resins, promoting sustainability, and easily scalable.¹⁵ Tamarind kernel powder (TKP) has good surface functionality and hydrolytic stability following benign crosslinking. Bidentate carboxylates and a disulfide with redox activity and soft-metal contacts are contributed by cysteine. Multiple carbonyl and imide nitrogen atoms are provided by cyanuric acid (2,4,6-trione-1,3,5-triazine), which forms hydrogen-bond networks and chelate/ion-pair interactions. In order to target anionic Cr species, we propose that combining these three results in complementary binding domains. In accordance with TKP-based chelating resins and cysteine/cysteine-derived sorbents documented in the literature, mechanistic investigations imply mixed Chemisorption-S/N/O coordination and ion-exchange. The study validates a low-cost, scalable method of polishing Cr utilizing amino acid chemistry and agro-derived polymers.^{3,16}

EXPERIMENTAL

Materials and Methods

Tamarind kernel powder (TKP), cyanuric chloride, L-cysteine, conc. NaOH solution, sodium bicarbonate, and dioxane.

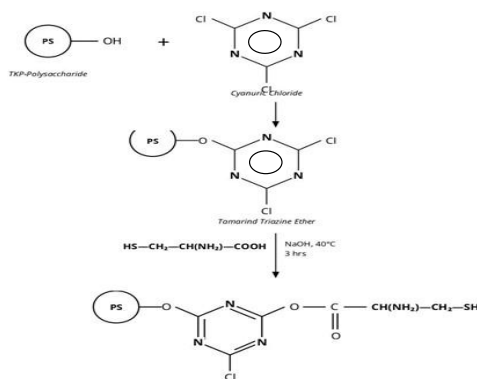
Preparation of Tamarind Derivative

The synthesis began by adding 0.1 mol of powdered tamarind kernels (TKP) in a round-bottom flask with 100mL dioxane, which was externally cooled to approximately 5°C. Following the addition of 7.2 g of cyanuric chloride, the mixture's pH was adjusted to 7–8 using sodium bicarbonate and stirred for 30 minutes. Next, 0.1 mol of cysteine was introduced, and the pH was raised to 9–10 using a concentrated NaOH solution. The reaction mixture was then magnetically stirred vigorously at 25–30°C for four hours. The resultant Tamarind triazine 2-amino-3-mercaptopropanoic acid (TTAMP) resin was isolated by filtration after derivatisation, washed thoroughly with distilled water, and then dried at a temperature of 110°C.⁸

Preparation of Cr (VI) Solution

The stock solution of chromium (VI) was prepared with a concentration of 1000 mg/L by disintegrating 2.828 g of potassium dichromate ($K_2Cr_2O_7$, with a purity of 99.00%) in 1 liter of double-distilled water.¹⁷ This stock solution was then utilized to create working solutions with concentrations ranging from 50 to 300 mg/L¹⁸. The pH of these solutions was meticulously monitored using a calibrated pH meter and adjusted to the necessary specifications with 0.1 N H_2SO_4 or 0.1 N NaOH.¹⁹

Mechanism of Resin Synthesis



Tamarind triazine 2-amino-3-mercaptopropanoic acid (TTAMP resin)

Characterization of Resin

FTIR

The major functional groups on the adsorbent's surface were identified using Fourier Transform Infrared (FTIR) spectroscopy data.²⁰ This analysis focused on Tamarind Kernel Powder (TKP) derivatives to ascertain the best characteristics for effective adsorption. In the hydroxyl and amine regions (3500–3000 cm^{-1}), a broad, shallow peak at 3246.5 cm^{-1} is observed; this is typical of the O-H stretching vibrations of the polysaccharide backbone.²¹ Since the focus here is on L-cysteine functionalization, it would seem that the broad absorption in this region is due to -OH groups in TKP and/or N-H stretching vibrations.²² Within the aliphatic C-H stretching range of 3000–2800 cm^{-1} , a significant absorption peak is observed at 2922.2 cm^{-1} .¹⁹ This peak is linked to the stretching vibrations of CH₂ groups, which reflect the organic backbone structure present in polysaccharides and amino acids.²³ In the carbonyl and amide area (1800–1500 cm^{-1}), there is a significant absorption peak at 1736.9 cm^{-1} , which is assigned to C=O stretching vibrations²⁴, indicative of the presence of carbonyl functionality like esters or carboxylic groups introduced as part of the modification of the resin. Another peak of importance in this range is the one observed at 1591.8 cm^{-1} due to N-H bending (Amide II) vibrations and/or asymmetrical stretching of COO⁻ groups. For the L-cysteine-modified resin, this peak proves that the amino acids have been incorporated into the polymer structure. The fingerprint area (1400–600 cm^{-1}) has a variety of complex absorption peaks that are connected to the molecular structure of the substance. One of them, at 1384.2 cm^{-1} , may be attributed to C-H bending vibrations and/or symmetrical stretching of carboxylate groups, indicating the introduction of functional groups via the modification method. The prominent “saccharide band” at 995.2 cm^{-1} is mainly caused by the stretching of the C-O-C glycosidic bond and the C-O stretching in the polysaccharide component.²⁵ Moreover, in the lower wavenumber range, the peaks at 663.5 cm^{-1} and 618.7 cm^{-1} belong to the C-S stretching or O-C-O bending vibrations, which are indicative of the thiol/disulfide character of the modified material containing cysteine. Overall, the peaks at 1591.8 cm^{-1} and 618 cm^{-1} indicate the availability of nitrogen and sulphur functional groups in the resin.^{7,15,21} These amines and thiols are critical for the elimination of metals, because they provide the active lone pairs of electrons necessary to coordinate with and “trap” metal ions such as Chromium (VI).

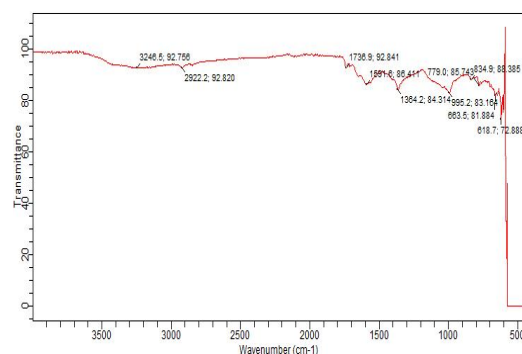


Fig.-1: FTIR Spectra of TTAMP Resin

Batch Experiment Studies

The batch studies were carried out systematically by varying parameters such as pH (ranging from 2.0 to 8.0), contact time (from 0 to 3 hrs), adsorbent dosage (between 0.1 and 1.0 grams), and initial Cr(VI) concentrations (50, 100, 150, 200, 250, and 300 mg/L). Following digestion, the metal ions were isolated using a G-3 sintered glass crucible. A volume of 10 millilitres of the solution was reserved for chromium VI ion analysis, while the remaining solution was stored for future use. The analysis of chromium VI was performed using a twin-beam UV-Vis spectrophotometer. The percentage of chromium removal by the resin was calculated using the following equation:²⁶

$$\% \text{ Chromium(VI) removal} = [(C_0 - C_e) / C_0] \times 100 \quad (1)$$

Where C_0 and C_e are the initial and equilibrium Cr (VI) ion concentrations, respectively.

Table-1: Experimental Conditions

Experimental parameters	Ms (mgL ⁻¹)	pH	Ps (μm)	Time (min)	C _o (mg/L)
Adsorbent dosage Ms (g/L)	0-20	8	120	50	50
pH	10	1-12	120	50	50
Contact time T (min)	10	8	120	0-180	50
Concentration of Cr (VI) ion C _o (mg/L)	10	8	120	50	50-500

RESULT AND DISCUSSION

Impact of Concentration

The effect of initial hexavalent chromium concentration (50–500 mg/L) on removal efficiency was studied in this experiment. It can be observed from Fig.-2(a) that there is clearly an inverse correlation between concentration and removal efficiency – the higher the chromium concentration, the lower the efficiency is. When the concentration of chromium is at its minimum value at 50 mg/L, the clearance efficiency reaches the value of approximately 94%. On the other hand, when the concentration is 500 mg/L, then the efficiency becomes 71%. This might be accounted for by the fact that there is only Limited number of available adsorption sites of the resin. Under low concentrations, the number of binding sites is sufficient to capture the majority of Cr (VI) ions present in the solution; however, when the concentration is high, there are more ions competing for the same number of sites, leading to rapid saturation. In this way, the drop in efficiency demonstrates the limited adsorption capacity of the resin.^{27,28}

Impact of pH

Adsorption behaviour of Cr (VI) was studied at varied pH conditions in the pH range of 1 to 12 at a constant dose of adsorbent 10 mg/L and starting concentration of chromium 60 mg L⁻¹. It is seen from Fig.-2 (b) that adsorption effectiveness depends largely on the solution pH. The removal efficiency increases linearly from pH 1 to pH 8, with the highest efficiency of 93.5% at pH 8.0 being achieved. In comparison, the removal efficiency at pH 1.0 is approximately 65.2%. The increase in adsorption effectiveness at higher pH is due to decreased concentration of H⁺ ions, which reduces competition for active adsorption sites and thus increases absorption of Cr (VI) ions.^{29,30} The removal effectiveness decreases marginally for pH values higher than the optimal value of 8. The efficiency goes down to 91.6% at pH 9, 91.2% at pH 10, and 90.8% at pH 11 and 12. This slight decrease could be linked to alterations in the adsorbent's surface charge, as well as the speciation of chromium at alkaline pH, which diminishes the adsorbate's attraction to the adsorbent. However, the adsorption system maintains relatively high removal efficiency at alkaline pH, which shows the strong adsorption efficacy and stability at diverse pH values.

Impact of Resin Dosage

Experiments were conducted with the dosage of the resin from 2 to 20 mg/L. From Fig.-2(c), it can be observed that removal efficiency shows a clear dependency on the amount of resin used. The maximum efficiency is obtained at the dosage of 10 mg/L-approximately 94%. The dependence of removal efficiency on the amount of resin used can be split into two different stages: Active Adsorption Stage (2–10 mg/L). The clearance efficiency improves steadily with the increase in the dose from 2 mg L⁻¹ to 10 mg/L. The increase in the quantity of resin provides an increased number of active adsorption sites and surface area,^{27,28} enhancing the interaction with the pollutant. Saturation/Stability Stage (12-20 mg/L). Beyond the optimum dose of 10 mg/L, the rate of elimination continues to decrease and finally reaches 83% at a dosage of 20 mg/L. This indicates that if the quantity of the resin used increases above the ideal value, no more advantages will be achieved. This may be caused by aggregation of resin particles, resulting in a reduction of the overall surface area or creation of a concentration gradient, which slows down the adsorption kinetics.^{31,32,33} Therefore, the best removal efficiency is achieved with the use of 10 mg/L resin.

Impact of Contact Time

The influence of duration of contact was studied at different doses of resin (2-20 mg/L) to examine the influence of resin doses on Cr⁺⁶ removal effectiveness. Figure-2(d) shows that the adsorption performance was substantially dependent on the quantity of resin used.

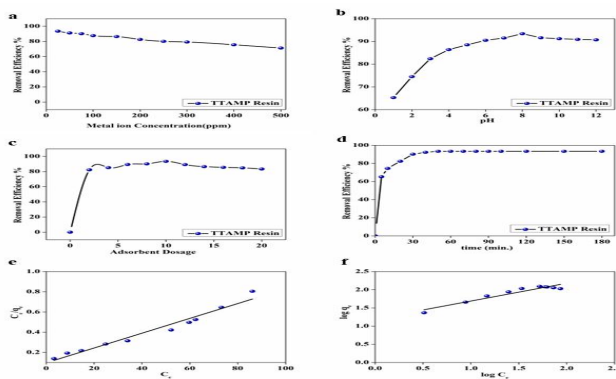


Fig.-2: The effect of (a) Metal Ion Concentration, (b) pH, (c) Resin Dose, (d) Contact Time on Removal Efficiency, (e) Langmuir Adsorption Isotherm for TTAMP Resin, and (f) Freundlich Adsorption Isotherm for TTAMP Resin

With increasing dose of resin, the removal efficiency rose and reached a maximum value of around 94% at 10 mg L⁻¹, indicating the optimal working condition. The observed conduct may be split into two different phases. In the first stage (2–10 mg /L), the removal efficiency rose gradually with the increase in resin dose. This improvement is due to the extra resin providing more active adsorption sites and a higher effective surface area³⁴, which allows for more effective interaction between the adsorbent and Cr (VI) ions.^{27,28} In the second stage (12–20 mg/L), there was a progressive drop in removal efficiency. Above the optimal dose of 10 mg/L, the effectiveness declined gradually to reach ca. 83% at 20 mg/L. The decrease in performance shows that the extra resin does not always increase the adsorption performance. The decrease might be due to particle aggregation at larger doses, which may result in the reduction of effective surface area for adsorption. Furthermore, the mass transfer and adsorption kinetics may be hindered by the overcrowding of adsorbent particles, leading to a decreased overall removal efficiency.^{31,32,33} Thus, the optimum condition was found to be a resin dose of 10 mg/L which resulted in the best removal efficiency while being cost-effective by efficient utilisation of the adsorbent material.

Adsorption Isotherms

The Freundlich and Langmuir adsorption isotherm models were utilized to analyze the static data related to the removal of Cr (VI).³⁵ These models provide valuable insights into the distribution of metal ions between the aqueous phase and TTAMP resin. According to the Langmuir model, adsorption takes place on a uniform surface with a fixed number of similar sites, resulting in the formation of a monolayer. The linear form of the equation is expressed as follows³⁶:

$$\frac{C_e}{q_e} = \frac{1}{q_{max}} K_L + \frac{C_e}{q_{max}} \quad (2)$$

In this context, C_e (mg/l) represents the equilibrium concentration of the metal ion, while q_e (mg/g) indicates the amount of solute that is absorbed per unit weight of the adsorbent. Additionally, q_{max} (mg/g) refers to the maximum adsorption capacity, which is the peak level of the surface's ability to adsorb. Lastly, K_L is the Langmuir adsorption constant, which is bound up with the energy of the adsorption process.^{37,38} The Slope ($1/q_m$) is the inverse of the slope and is a measure of the maximum capacity of the material. A flatter line suggests a greater adsorption capacity and The Intercept ($1/q_m K_L$). This value helps assess the affinity between the adsorbent and the adsorbate. The linearity (R^2) indicates that the points in your image are closely aligned along a straight trajectory, it suggests that the Langmuir model is an excellent fit for this specific experiment, confirming that monolayer adsorption is likely occurring. A good correlation value ($R^2 > 0.98$) for this model suggests that the functional groups are evenly distributed in the TKP matrix.

Freundlich Isotherms

The Freundlich model is used to describe adsorption on uneven surfaces.³⁹ It's linear form is:

$$\log q_e = \log K_f + 1/n(\log C_e) \quad (3)$$

The chemisorptive capacity of biomass for various metal ions may be compared using an adsorption isotherm described by K_f , the Freundlich constant for the adsorption capacity, a constant whose value describes the affinity and surface properties of the sorbent. In this study, two of the many isotherm equations have been used, namely Freundlich and Langmuir isotherms.^{37,38} Table-2 presents the estimated values of Langmuir and Freundlich model parameters obtained from fitting experimental data of Cr(VI).

Table-2: Langmuir and Freundlich Isotherm

Langmuir Isotherm			Freundlich isotherm		
R^2	$Q_{\max}$ mg/g	b , L/mg	R^2	K_f mg/g	n
0.989	149.3	0.0756	0.982	15.85	2.04

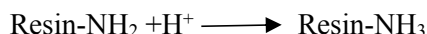
The Langmuir model shows in Fig.-2 (e) and (f) a slightly higher R^2 (0.989 vs 0.982), this implies that the adsorption happens probably on a monolayer with comparable sites. Adsorption Intensity (n): Since the calculated n is 2.04 (which is between 1 and 10), it confirms that the adsorption process is highly favorable for this material.^{40,41}

Adsorption Mechanism

Surface phenomenon- Adsorption vs. Absorption as Surface Phenomena: Cr(VI) elimination is only a surface phenomenon (adsorption). Physical Adsorption: First, metal ions move from the bulk liquid to the resin surface by film diffusion. Chemical Adsorption (Chemisorption): On reaching the surface, ions form strong chemical bonds with the cysteine functional groups. The wide “landing strip” for these ions is provided by the significant surface area and high porosity of the cross-linked Tamarind Kernel Powder.

Electrostatic Interaction

Generally, in wastewater, chromium exists as chromate (HCrO_4^-) or chromate anion. At pH 2.0 the amino groups on the cysteine molecule undergo protonation.



Interaction

The negatively charged HCrO_4^- ions are strongly drawn to the newly positively charged resin surface by electrostatic forces. Therefore, very acidic settings are best for chromium removal.

Feature	Low pH<4.0
Surface charge	Positive
Active groups	Protonated amine
Target ion	Cr(VI) anionic
Interaction type	Electrostatic attraction

Ion Exchange Mechanism

Carboxylic acid groups ($-\text{COOH}$) are present in the cysteine molecule. The H^+ ions on these groups are responsible for replacing chromium Cr(VI); the exchange takes place at the protonated amino sites since Cr(VI) is an anion (HCrO_4^-). In this case, the resin may swap the chromate for either a hydroxyl ion (OH^-) or a chloride ion (Cl^-).

Chelation or Coordination Mechanism

Compared to simple ion exchange, chelation is a more complex and powerful type of binding. A single metal ion and two or more "donor atoms" (oxygen, nitrogen, or sulfur) from the cysteine molecule make multiple coordinate covalent bonds in this process.

Common Ion Effect

In real industrial wastewater, Cr (VI) ions rarely exist as the sole dissolved species. A diverse range of ions existing together, such as Cl^- , NO_3^- , SO_4^{2-} , Na^+ , and Ca^{2+} are often observed, and their presence may affect the adsorption capacity of the resin.^{8,9,10} Thus, the evaluation of the impact of the aforementioned ions becomes crucial in the assessment of the practical potential of the adsorbent. From all of the coexisting components, the anions like chloride (Cl^-), nitrate (NO_3^-), and sulphate (SO_4^{2-}) can considerably affect the elimination of Cr (VI). The presence of such anions leads to an increase in the ionic strength of the solution; thus, the screening effect is observed, and the electrostatic connection

between Cr (VI) species and positive adsorption sites is weakened.^{7,21,27} Thus, the chromium ions have more difficulty in reaching the active adsorption sites, and the adsorption process is decreased. Among the anions, sulphate ions demonstrate the biggest interfering ability. This is because sulphate ions have a divalent negative charge and possess electrostatic properties comparable with those of chromate ions (CrO_4^{2-}). Thus, the competition for the positive functional groups of the resin, like protonated amino groups (Resin-NH_3^+) takes place. As a consequence, the high content of sulphate ions may significantly decrease the adsorption of chromium due to the occupation of its adsorption sites.

Table: Interference Impact

Species	Type	Targeted metal	Impact on adsorption	mechanism
SO_4^{2-}	anion	Cr(VI)	High decrease	Competition for NH_3^+ Sites
$\text{Cl}^-/\text{NO}_3^-$	anion	Cr(VI)	Low decrease	General ionic strength\screening

CONCLUSION

The present study demonstrated that TTAMP resin can serve as a highly efficient adsorbent for the extraction of Cr (VI) ions from water solutions. It was found that the adsorption process is influenced by several operational factors, including the initial concentration of Cr (VI) ions, the pH level of the solution, the duration of contact, and the amount of resin used. Among all the conditions tested, the most effective adsorption occurred at the optimal pH, achieving a maximum removal efficiency of 93.5%. The data obtained from the adsorption experiments indicated that the adsorption isotherms are best described by the Langmuir isotherm equation, which implies the formation of a monolayer on a uniform surface with a finite number of adsorption sites. Therefore, the results presented affirm the significant adsorptive capacity of TTAMP resin and highlight its potential for the efficient removal of Cr (VI) ions. In summary, the investigation has revealed the remarkable effectiveness of TTAMP resin as an adsorbent for Cr (VI) ion removal, making it a promising candidate for wastewater treatment processes and the remediation of chromium-contaminated water and heavy metal pollution (SDG-6).

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CONFLICT OF INTERESTS

The authors declare that there are no conflicts of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-6: Clean Water and Sanitation*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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