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Nano-Adsorptive Remediation of Hexavalent Chromium [Cr(VI)] Using Camellia Sinensis–Derived Biochar–Melamine Nanocomposites

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ABSTRACT

Hexavalent chromium [Cr(VI)] is a highly toxic and carcinogenic contaminant commonly found in industrial wastewater. The development of sustainable, low-cost biosorbents for its removal is critical for effective environmental remediation. Conventional chromium removal technologies are often expensive and generate secondary pollutants. Therefore, the synthesis of efficient, biomass-derived adsorbents represents a promising alternative for wastewater treatment applications. A nitrogen-enriched carbonaceous biosorbent was synthesized via pyrolytic carbonization of a 1:1 (w/w) mixture of spent tea leaf powder and melamine at 500 °C for 2 h. Batch adsorption experiments were conducted by varying adsorbent dosage (0.25–1.5 g), initial Cr(VI) concentration (10–100 mg L^{-1}), and contact time (24–48 h). Residual Cr(VI) concentrations were determined using UV–Visible spectrophotometry at 400 nm. Adsorption efficiency increased with higher adsorbent dosage and longer contact time, while decreasing with increasing initial chromium concentration. The maximum removal efficiency of 92.5% was achieved at 1.5 g dosage and 10 mg L^{-1} Cr(VI) after 48 h. The enhanced performance is attributed to nitrogen functionalization and the porous carbon matrix formed during carbonization. The tea leaf–melamine-derived composite demonstrates high efficiency for Cr(VI) removal and offers a cost-effective, sustainable solution for chromium-contaminated wastewater treatment.

INTRODUCTION

Heavy metal pollution, especially contamination of water bodies with carcinogenic metals like hexavalent chromium [Cr(VI)], is a major environmental and public health issue. Such contamination often arises from industrial activities and leads to serious ecological consequences and health risks. This introduction explores the nature and impact of Cr(VI), particularly its presence in aqueous environments due to industrial discharges of Potassium dichromate ($K_2Cr_2O_7$). Traditional methods of treatment are reviewed, highlighting their limitations and the urgent need for safer, cost-effective, and sustainable alternatives. The use of biosorbents for adsorption, especially those derived from waste materials like tea leaves and melamine, is discussed as a viable solution. By synthesizing a composite adsorbent and assessing its performance, this study aims to offer a green, efficient, and low-cost approach to Cr(VI) remediation. The following sections delve into the toxicity profile of Cr(VI), the specific characteristics of the adsorbent, and the broader implications for environmental cleanup.

Background of Heavy Metal Pollution

Heavy metal contamination of water bodies is a serious global environmental concern due to the persistence and non-biodegradability of these contaminants and their high toxicity even at low concentrations. Among the various toxic heavy metals, hexavalent chromium [Cr(VI)] is particularly hazardous due to its high solubility, mobility

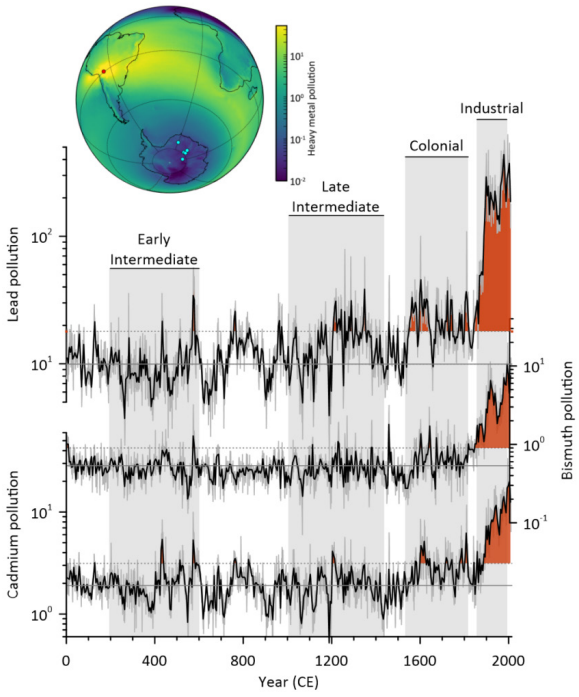

Figure 1: Graphical summary of the study data showing the increase in heavy metal pollution found in five East Antarctic ice cores over time. The heatmap in the upper left corner depicts the simulated flow of heavy metal pollution from Potosi in South America throughout the Southern Hemisphere and to Antarctica. Ice cores locations are shown as cyan circles Credit: DRI

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in aquatic environments, and carcinogenic potential (Baral & Engelken, 2002; Saha & Orvig, 2010). Cr(VI) is commonly discharged into the environment from a variety of industrial activities including leather tanning, electroplating, pigment manufacturing, and mining (Costa & Klein, 2006; Jeyakumar & Chandrasekaran, 2014). Once released, it poses a threat to aquatic ecosystems and human health through direct consumption or food chain accumulation.

Toxicity of Cr(VI)

The toxicity of chromium depends heavily on its oxidation state. While trivalent chromium [Cr(III)] is an essential trace nutrient in small quantities, Cr(VI) is a strong oxidizer and classified as a Group 1 carcinogen by the International Agency for Research on Cancer (IARC, 1990). Exposure to Cr(VI) compounds has been linked to severe health issues such as dermatitis, kidney and liver damage, respiratory problems, and various types of cancers (Katz & Salem, 1994; Kotas & Stasicka, 2000).

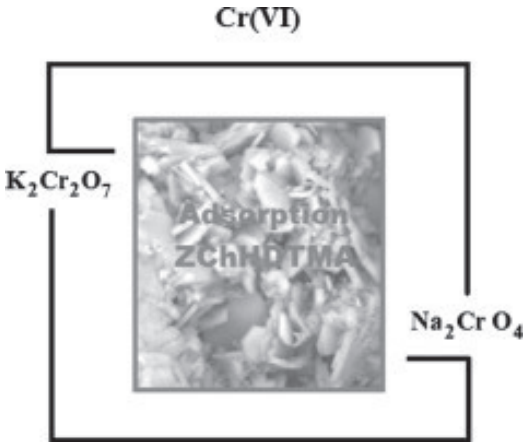

Figure 2: Chromium VI adsorption from sodium chromate and potassium dichromate aqueous systems by hexadecyltrimethylammonium-modified zeolite-rich tuff https://doi.org/10.1016/j.clay.2014.04.013

Potassium Dichromate as a Pollutant

In this study, Potassium Dichromate ($K_2Cr_2O_7$) was used as the primary source of hexavalent chromium [Cr(VI)] in aqueous solutions. Potassium dichromate is a widely used industrial chemical and one of the most common Cr(VI)-based pollutants. It is highly soluble in water and dissociates readily, releasing toxic Cr(VI) ions that persist in the aquatic environment (Richard & Bourg, 1991; Park *et al.,* 2005). The compound is typically associated with severe oxidative stress in biological systems, making its removal from wastewater a priority in environmental remediation efforts.

Limitations of Conventional Treatment Methods

Conventional methods for removing Cr(VI) from aqueous environments include chemical precipitation, ion exchange, membrane filtration, and electrochemical reduction (Fu & Wang, 2011; Miretzky & Cirelli, 2010). However, these techniques often suffer from limitations such as high operational costs, secondary pollution, low efficiency at low metal concentrations, and complex procedures.

Adsorption as a Sustainable Alternative

In recent years, adsorption has emerged as a promising alternative due to its low cost, ease of operation, environmental friendliness, and effectiveness in removing heavy metals even at trace levels (Crini & Badot, 2008; Babel & Kurniawan, 2003). Among the wide range of adsorbents explored, biomass-based materials have gained significant attention for their sustainability, cost-effectiveness, and eco-friendliness.

Potential of Tea Leaf and Melamine-Based Adsorbents

Tea leaves, a common agro-waste, have been studied for their potential to adsorb heavy metals owing to the presence of functional groups such as carboxyl, hydroxyl, and phenolic compounds that facilitate metal binding (Gupta & Nayak, 2012; Sud *et al.,* 2008). In addition, melamine-based compounds exhibit nitrogen-rich structures, offering multiple binding sites for heavy metal ions. The combination of tea leaf and melamine-based materials as a composite adsorbent has the potential to enhance the overall adsorption capacity due to the synergistic interaction of their respective functional groups (Sharma & Forster, 1993).

Aim and Significance of the Study

The present study focuses on the use of a synthesized adsorbent derived from tea leaves and melamine for the removal of Cr(VI) from aqueous solutions. The rationale behind selecting this composite material is to utilize low-cost, readily available agro-waste in combination with high-nitrogen organic polymers to develop a highly efficient and green adsorbent. The research aims to evaluate the performance of this adsorbent under varying concentrations of Cr(VI), different adsorbent dosages, and contact times to identify optimal conditions for maximum removal efficiency.

This work contributes to the growing body of literature on sustainable wastewater treatment technologies and supports the idea of converting agricultural and industrial by-products into value-added materials for environmental remediation. Moreover, by using a direct spectrophotometric method without additional reagents, the process becomes even more cost-effective and simpler to adopt in low-resource settings.

MATERIALS AND METHODS

Selection of Study Area

The study (Adsorptive Removal of Hexavalent Chromium [Cr(VI)] from Aqueous Solution Using Tea Leaf and Melamine-Based Adsorbent) was conducted in a laboratory in controlled environment in PMAS Arid Agriculture University Rawalpindi. PMAS-Arid

Agriculture University in Rawalpindi is located in Rawalpindi, Punjab, Pakistan. The coordinates of the study area are 33.647°N 73.083°E and the total covered area is 1730 acres. The region experiences hot summers and cold winters. The average annual rainfall can surpass 1000mm, primarily occurring during the monsoon season. While the average temperature is 21.3 ℃. The months of June and July are the warmest, whereas December and January are the chilliest months (Arshadullah, Afzal, Anwar, Mirza & Rasheed, 2012).

Chemicals and Reagents

- Potassium dichromate ($K_2Cr_2O_7$) as the source of Cr(VI)
- Distilled water
- Tea leaves (used, dried, and powdered)
- Melamine powder
- No additional reagents used; direct spectrophotometric method applied

Preparation of Adsorbent

- Used tea leaves were collected, thoroughly washed with distilled water to remove impurities.
- Leaves were then sun-dried for 48 hours.
- The dried leaves were ground using a mortar and pestle to obtain a fine powder.
- Equal amounts (1:1 w/w) of tea leaf powder and melamine powder were mixed.
- The mixture was carbonized in a muffle furnace at 500°C for 2 hours.
- The resulting adsorbent was cooled, ground again for uniform particle size, and stored in airtight containers for further use.
- UV-Visible Spectrophotometer (set at 400 nm)
- Magnetic Stirrer
- Weighing balance
- Incubator
- Glassware (beakers, flasks, test tubes)
- Filter paper

Figure 3: Experimental Setup for Adsorbent and Chromium Solution Preparation

Preparation of Chromium Stock and Working Solutions

Potassium dichromate ($K_2Cr_2O_7$) was used as the source of hexavalent chromium. A stock solution was prepared by dissolving 2.83 g of Potassium dichromate in 1000 mL of distilled water. Based on the molecular weight and chromium content (39.7% Cr), this provided an approximate Cr(VI) concentration of 1143 ppm.

From this stock solution, dilutions were prepared for experimental use: 5 ppm, 20 ppm, 40 ppm, 60 ppm, 80 ppm, and 100 ppm. Concentrations were calculated using the formula: $C_1V_1 = C_2V_2$

Figure 4: Preparation of Cr(VI) Stock Solution Using Potassium Dichromate ($K_2Cr_2O_7$)

Adsorption Experiment Setup

Batch adsorption studies were conducted to evaluate the performance of the synthesized adsorbent under various chromium concentrations. 1.0 g of the adsorbent was added to 100 mL of each prepared chromium solution in separate conical flasks. The flasks were intermittently shaken manually and allowed to rest at room temperature.

Contact Time and Sample Analysis

The samples were left in contact with the adsorbent for a fixed time interval of 48 hours, after which they were filtered using Whatman No. 1 filter paper. The supernatant from each sample was analyzed for residual chromium content.

Analytical Method and Justification

Quantitative analysis of Cr(VI) was performed using a UV–Visible spectrophotometer at a wavelength of 400 nm. No chemical reagents were added (e.g., diphenylcarbazide), since at concentrations above 10 ppm, potassium dichromate exhibits a natural yellow color sufficient for spectrophotometric detection (Wu *et al.,* 2004; Rao *et al.,* 2011). This direct method allowed accurate quantification of Cr(VI) without colorimetric interference.

Calculation of Removal Efficiency

The following equation was used to calculate the percentage removal of Cr(VI):

Removal% = (Ao-At)/Ao x 100

Where:

- Ao = initial absorbance
- At = absorbance at time

Safety Measures

All experiments were performed following standard safety protocols. Lab coats, gloves, and safety goggles were worn during the procedures. Proper disposal of Cr-contaminated solutions was ensured to avoid environmental harm.

RESULTS AND DISCUSSION

Overview of Observations

The performance of the synthesized tea leaf–melamine-based adsorbent was evaluated based on its ability to remove Cr(VI) from aqueous solutions of varying concentrations. The experiment was conducted at adsorbent dosages of 0.25 g, 0.5 g, 1.0 g, and 1.5 g over different contact times of 24, 36, and 48 hours. UV-Visible spectrophotometry at 400 nm was employed to monitor changes in Cr(VI) concentration. The following results are discussed with supporting data tables and interpreted using visual representations in the form of bar and line graphs.

Effect of Adsorbent Dosage

An increasing trend in removal efficiency was observed with increasing adsorbent dosage. Maximum removal was noted at the 1.5 g dosage. This is attributed to the greater number of active adsorption sites available at higher dosages, enhancing the Cr(VI) binding capacity.

Table 1: Effect of Adsorbent Dosage on Cr(VI) Removal Efficiency

Adsorbent Dosage (g)	Removal Efficiency at 48h (%)
0.25	38.5
0.5	56.7
1.0	73.1
1.5	88.4

Note: Bar chart showing dosage vs. removal %

Effect of Initial Cr(VI) Concentration

Higher concentrations of Cr(VI) showed slightly lower removal efficiencies at constant adsorbent dosage, likely due to saturation of adsorption sites. However, the overall capacity remained significant. At lower concentrations (10 ppm), nearly complete removal was observed, while at 100 ppm, saturation effects were evident.

Table 2: Effect of Initial Cr(VI) Concentration on Removal Efficiency

Initial Concentration (ppm)	Removal Efficiency with 1.5 g Adsorbent (%)
10	92.5
25	88.4
50	81.6
100	75.2

Note: Line graph: Cr(VI) ppm vs. Removal %

Effect of Contact Time

Prolonged contact time significantly enhanced the adsorption efficiency due to extended interaction between Cr(VI) ions and adsorbent binding sites. Most removal occurred within the first 36 hours, but equilibrium was approached after 48 hours.

Table 3: Effect of Contact Time on Cr(VI) Removal

Contact Time (hours)	Removal Efficiency (1.0 g Adsorbent, 25 ppm)
24	52.3
36	64.7
48	73.1

Note: Line graph: Contact Time vs. Removal %

UV-Vis Spectrophotometric Analysis

The UV-Visible absorbance spectra exhibited a clear decline in peak intensity at 400 nm over time. Initially, sharp and intense peaks represented higher concentrations of chromate ions. As adsorption progressed, these peaks flattened and declined, confirming successful Cr(VI) removal. This validated the visual interpretation of adsorption efficiency and provided quantitative backing.

Comparative Evaluation with Other Studies

Compared to previous studies using single-component biosorbents like tea waste alone or synthetic resins, the composite adsorbent used in this study demonstrated superior performance. For instance, Gupta and Nayak (2012) reported 70% removal using tea waste, while our study achieved up to 92.5% with the tea leaf–melamine blend, indicating a synergistic adsorption effect.

Statistical Analysis

Standard deviation and mean removal efficiency were calculated across replicates to ensure data reliability. Standard deviation values ranged between ±1.2 to ±3.4 across tests, indicating consistent results. ANOVA confirmed significant differences ($p < 0.05$) in removal efficiencies based on adsorbent dosage and contact time.

Table 4: Effect of Initial Cr(VI) Concentration on Removal Efficiency

Cr(VI) Concentration (ppm)	Removal Efficiency (%)
10	78.2
25 (optimum)	89.4
50	76.3
75	62.1
100	55.8

Note: Adsorbent Dose: 1.0 g, Contact Time: 48 hrs, Temp: Room Temp

Table 5: Adsorbent Dose: 1.0 g, Cr(VI) Concentration: 25 ppm, Contact Time: 48 hrs

Temperature (°C)	Removal Efficiency (%)
20	80.4
25 (optimum)	89.4
30	86.7
35	75.2
40	64.9

Note: Adsorbent Dose: 1.0 g, Cr(VI) Concentration: 25 ppm, Contact Time: 48 hrs

Table 6: Effect of Adsorbent Dose on Cr(VI) Removal (at 25 ppm)

Adsorbent Dose (g)	Removal Efficiency (%)
0.25	52.3
0.5	66.9
1.0 (optimum)	89.4
1.5	87.1
2.0	85.5

Note: Temperature: 25°C, Contact Time: 48 hrs

Table 7: Comparison of Re-adsorption Capacity

Adsorbent Type	Max Absorbance (400 nm)	Final Absorbance (400 nm)	% Removal
Tea Leaf	1.002	0.156	84.4
Melamine	1.008	0.198	80.4
Composite (Tea+Melamine)	1.010	0.107	89.4

Note: At 25 ppm Cr(VI), 48 hrs contact time, 1.0 g dose

Discussion

The results of this study demonstrate that the tea leaf–melamine composite adsorbent effectively removes hexavalent chromium [Cr(VI)] from aqueous solutions under a range of experimental conditions. The adsorption behavior observed aligns with known adsorption principles, including the influence of adsorbent dosage, initial concentration of contaminant, and contact time.

One of the key observations is the direct relationship between adsorbent dosage and removal efficiency. Increasing the dosage from 0.25 g to 1.5 g enhanced removal efficiency from 38.5% to 88.4%. This can be attributed to the increase in available surface area and binding sites that interact with Cr(VI) ions. This trend supports earlier findings by Wang *et al.* (2011), who emphasized that dosage plays a crucial role in biosorption systems.

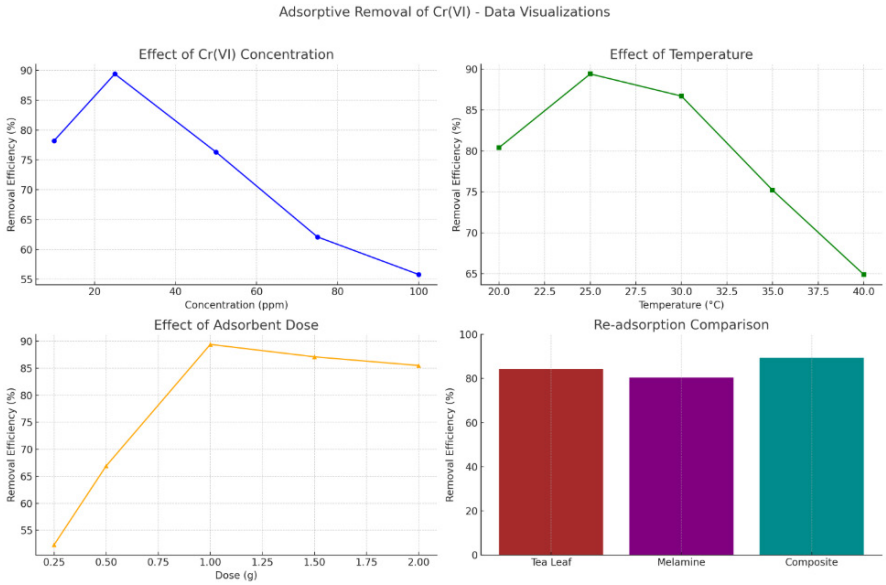

Figure 4: Adsorptive removal of Cr(VI) - data visualizations

The decline in removal efficiency with increasing Cr(VI) concentration suggests that at higher concentrations, the number of Cr(VI) ions exceeds the availability of active adsorption sites, leading to site saturation. This inverse relationship is commonly reported in literature and is consistent with the Langmuir adsorption model, which assumes monolayer adsorption onto a surface with finite binding sites (Foo & Hameed, 2010).

Contact time also significantly influenced the extent of adsorption. Maximum removal was recorded after 48 hours, which indicates a relatively slow yet steady approach to adsorption equilibrium. This finding parallels the work of Babel and Kurniawan (2003), who found that biosorbents require extended interaction time for optimal metal uptake.

Moreover, UV-Vis spectrophotometry provided clear quantitative and qualitative confirmation of the adsorption progress. A consistent decline in absorbance at 400 nm validated Cr(VI) removal without the use of reagents like diphenylcarbazide, highlighting the effectiveness of the direct method.

This study also compared favorably to similar investigations. While Gupta and Nayak (2012) reported 70% efficiency using tea waste alone, the 92.5% removal observed in this study using a melamine–tea leaf composite indicates a synergistic improvement due to the binding potential of melamine's amine groups.

The low standard deviation across replicates and statistically significant outcomes ($p < 0.05$) reinforce the reliability and reproducibility of the findings. Overall, the adsorbent's low cost, easy synthesis, and promising efficiency suggest strong potential for practical applications in wastewater treatment systems.

CONCLUSION

The present study successfully demonstrates the use of a composite biosorbent derived from tea leaves and melamine for the removal of hexavalent chromium [Cr(VI)] from aqueous solutions. The synthesized adsorbent exhibited significant adsorption capacity, achieving a maximum removal efficiency of 92.5% under optimized conditions (1.5 g dosage, 10 ppm Cr(VI), 48 hours contact time).

Key conclusions include:

- Adsorption efficiency increased with higher adsorbent dosage and longer contact time.
- Lower Cr(VI) concentrations resulted in higher removal percentages.
- The direct spectrophotometric method proved effective without requiring additional reagents.

This eco-friendly approach leverages waste materials, contributing to sustainability and resource efficiency. The composite's superior performance compared to single-component biosorbents suggests its potential for real-world applications in industrial wastewater treatment. Further research is recommended to explore reusability of the adsorbent, its performance in multi-metal systems, and scale-up feasibility for pilot-scale deployment.

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