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Exploring the Chromium Removal from Landfill Leachate Using *Azadirachta indica* Leaf as a Natural Adsorbent

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Abstract

Background: The removal of Cr (VI) from residential and commercial wastewater via the biosorption method is expedient, due to its high efficiency, natural affinity, selectivity, ease of use, affordability, and eco-friendliness. **Aim:** The present study demonstrates the removal of hexavalent chromium ion, Cr (VI), from landfill leachate by the biosorbent *Azadirachta indica* leaf powder (AILP). **Methods:** In the present study, the experimental method was used to investigate the optimisation of operational parameters such as pH, AILP dosage, reaction time, and temperature. **Results:** The experimental results demonstrated that the removal percentage of Cr (VI) was highest (91%) below the following optimized conditions: a reaction time of 120 min, a dose of AILP 1.0 g/L, a pH of 8.8, and a temperature of 298 K. Adsorption was also studied by assessing various isotherm models, kinetic models and thermodynamic factors. The outcomes indicate that the adsorption system followed Langmuir and pseudo-second-order models. The thermodynamic factors determined from the test demonstrated that the process is exothermic and spontaneous. **Conclusion:** AILP has a significant removal efficiency of Cr (VI) from landfill leachate. The result significantly shows that AILP can be utilized as a bio-adsorbent for toxic metal removal, such as Cr (VI), from polluted water and soil in geographic locations where neem trees grow in abundance.

Major Findings: Maximum Cr (VI) removal efficiency of 91% was achieved at 120 min reaction time, 1.0 g/L AILP dose, pH 8.8, and 298 K. Thermodynamic analysis confirmed that the adsorption process was spontaneous and exothermic.

Keywords: *Azadirachta indica* (Neem) Leaf Powder, Cr (VI) Removal, Isotherm, Kinetic Models, Landfill Leachate

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1. Introduction

Urban and industrial waste disposal in landfills generates leachate. This contaminated liquid poses serious environmental risks when released untreated into surface and groundwater[1]. Leachate contains toxic heavy metals, including chromium (Cr), nickel (Ni), arsenic (As), lead (Pb), and cadmium (Cd). These metals originate from industrial activities such as metallurgy, mining, tanneries, electroplating, and battery manufacturing[2]. They present significant ecological threats due to their mutagenic and carcinogenic effects, endangering human health and ecosystems[3,4]. Among these, hexavalent chromium (Cr (VI)) is particularly hazardous, causing kidney dysfunction, bone and liver cancers, and respiratory and neurological disorders[5]. Various treatment methods like hyperfiltration, chemical precipitation, and ion exchange have been developed to mitigate Cr (VI) pollution[6,7]. However, these approaches often produce chemical sludge, are expensive, and require high energy inputs while offering limited removal efficiency[4,8]. In contrast, adsorption offers a cost-effective, eco-friendly, and efficient alternative[9,10]. Natural adsorbents like jackfruit leaves[1],

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orange peel[11], waste tea and coffee[12], jackfruit seed powder[13], and guava leaves[14] have been studied for Cr (VI) removal. From the above materials, *Azadirachta indica* is a highly effective natural absorbent to remove chromium and have a potent to eliminate the water-based solutions of heavy metals[15]. *Azadirachta indica* (neem), rich in phytochemicals[16], effectively removes heavy metals including Cr (VI), Cu (II), Zn (II), Pb (II), and Ni (II) from wastewaters, especially in southern India[17,18]. However, the effectiveness of AILP in removing Cr (VI) from landfill leachate has not been well demonstrated by relevant research. Therefore, the current study evaluates AILP for removing Cr (VI) from landfill leachate. The specific aims of this study include (i) assessing parameters such as reaction time, pH, AILP dosage, and temperature affecting removal efficiency. Additionally, it estimates thermal and adsorption kinetics under optimised conditions.

2. Materials and Methods

2.1 Materials

Analytical grade chemicals, viz. Hydrochloric acid, acetone, nitric acid, sodium hydroxide, sulfuric acid, diphenyl carbazide, and phosphoric acid were utilised in the present investigation.

2.2 Collection of Samples

The mature leaves of *Azadirachta indica* were collected from Periyar Maniammai Institute of Science and Technology premises, Thanjavur, Tamil Nadu, India (Latitude-10° 47' 13.1964" N, Longitude - 79° 8' 16.1700" E). The leachate was collected from the stabilised landfill of the municipal solid waste disposal site, Thanjavur. It was filtered to remove suspended impurities and kept at 4°C to prevent biological oxidation before being characterised.

2.3 Adsorbent Preparation

The dust and other insoluble contaminants present in the leaves of *A. indica* were removed by washing with normal water. Then, double-distilled water was used to wash the leaves to remove hydrophilic pollutants. Then, the washed leaves were dried under sunlight and placed in a hot air oven (353°K) for several hours. After that, the dried leaves were crushed and passed through a sieve analyser (74 µm). The crushed powder was washed again and allowed to dry for 30 min to remove moisture. Then, the dried powder was mixed with H_3PO_4 in a crucible, kept in a furnace for 20 min, and maintained at 260°C. After that, the sample was removed from the furnace and allowed to cool down at room temperature. After cooling, the sample was cleaned using hot water to eliminate the remaining moisture and free acid and then dried for 25 min in an electric oven. The processed AILP adsorbent, as shown in Figure 1, was stored in an airtight cover for experimental investigations.

2.4 Batch Adsorbent Trials

To examine the sorption capability of AILP for removing Cr (VI) from landfill leachate, a batch adsorption test was performed at room temperatures (25 ± 3°C or 298°K ± 3°C). A total of 100 mL of landfilled leachate was filled in a capacity of 250ml conical flask. A glass electrode (Labman-LMPH-10) was used to adjust the desired pH with 1N H_2SO_4 and NaOH. The influence of variables such as dosage of AILP, reaction time, pH, and temperature is as follows: AILP dose, 0.1 g/L-5 g/L; reaction time, 15-180 min; pH,(3-11); and temperature,

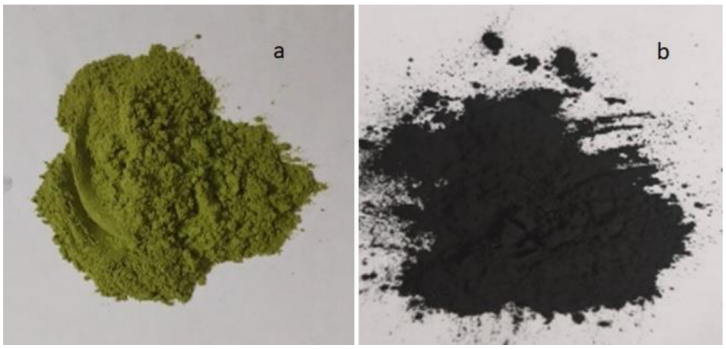


Figure 1. *Azadirachta indica* leaf powder (AILP) (a. Before acid washing; b. After acid washing).

298–328 K. The AILP and leachate were taken to conical flasks and mixed with the help of an orbital shaker at 150 rpm, and every 15 min, the sample was collected. Then, the collected sample was centrifuged at 6000 rpm for 5 min, and the supernatant (10 ml) was filtered. The concentration of Cr (VI) in the filtrate was measured at 540 nm (UV-visible spectrophotometer Shimadzu-UV 19001).

In accordance with the provisions of the American Public Health Association (APHA) method, the leachate characteristics were studied[19]. Three trials were performed in all experiments. The q_e and removal % of Cr (VI) were determined using equations (1) and (2)[20].

$$\% \text{ removal of chromium (VI)} = \frac{C_1 - C_2}{C_3} \times 100 \quad (1)$$

$$\text{Adsorption capacity } q_e \text{ (mg/g)} = \frac{C_1 - C_2}{Q} \times V \quad (2)$$

Where, $C_1$and $C_2$are the initial and final Cr (VI) concentration (mg/L), Q - mass of the AILP dose (grams), and V - Solution volume (L).

2.5 Kinetic Models

The models are utilised to evaluate the efficiency of the adsorption process. It was assessed using pseudo-first and second-order models[21,22], which helps to predict the concepts of adsorption. Wang *et al.*, reported that the mechanisms of solid-liquid systems and liquid-liquid systems are represented by pseudo-first and second-order models, respectively[23].

2.6 Pseudo-First and Second-Order Model

The pseudo-first and second-order equations stated by Qiu *et al.*,[24] and Xiang *et al.*,[25] are mathematically expressed as

$$\log((q_e - q_t) = \log(q_e)) - \frac{k_t}{2.303} t \quad (3)$$

$$\frac{t}{q_e} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (4)$$

Where, q_t and q_e are (mg/g), the amounts adsorbed at time and equilibrium, respectively,

k_1 and k_2 indicate the rate constant of first and second-order equations (min).

2.7 Equilibrium Study

In this proposed study, the equilibrium isotherm models given by the Freundlich and Langmuir models were formulated to evaluate the ratio of sorbent-sorbate at different equilibrium data and concentrations of Cr (VI).

2.8 Langmuir and Freundlich Isotherms

According to Mancilla *et al.*,[8] the mechanism for the formation of a homogeneous monolayer at adsorbent saturation, with uniform energy, no transmission, and no interaction between the adsorbed metals and adsorbate, is determined by the Langmuir isotherm. In this investigation, Langmuir isotherm variables were used to determine the correlation between the active sites on the sorbent surface and the concentrations of sorbate present in the solution under equilibrium conditions[26,27]. The Freundlich isotherm is used to characterize both multilayer (physisorption) and single-layer (chemisorption) adsorption. The non-homogeneous distribution of energy at the active site during adsorption on the surface of an adsorbent was studied using this model. During adsorption, the binding forces between the absorbent and adsorbate are initially stronger. When the level of sorbate accumulation increases, a reduction in the binding force between the AILP and Cr (VI) is observed. The Langmuir and the Freundlich isotherms can be expressed as shown in equations (5), (6), and (7)[28,29]. By plotting the graphs of Ce/qe vs Ce and Log qe vs Log Ce, the values of qm, n, and K_L, K_F were estimated from the slope and intercept of the respective plots.

$$\frac{C_0}{q_e} = \frac{C_0}{q_m} + \frac{1}{K_L q_m} \quad (5)$$

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (6)$$

$$R_L = \frac{1}{1 + K_L C_0} \quad (7)$$

Where,

- K_F (mg/g) and K_L (L/mg) are the Freundlich and Langmuir isotherm constants (adsorption capacity)
- q_e and q_m(mg/g) are respectively the equilibrium and maximum adsorption capacities of the adsorbent on the monolayer.
- n- Freundlich adsorption intensity

- C_e- Solution concentration of Cr (VI) at equilibrium (mg/L)
- R_L -Constant of equilibrium, C_o-Initial Cr (VI) conc.

The R_L indicates the isotherm type. The conditions of R_L and 1/n are considered to predict whether the isotherm is unfavourable or favourable, optimum or non-optimum, or linear or irreversible adsorption[12,30,1].

2.9 Reusability of AILP

After the adsorption process, the AILP (AA-AILP) was collected in a conical flask with deionized water and mixed well using an orbital shaker for 24 hours. Then, the filtrate was separated and dried using a hot air oven overnight at 105°C. Reusability tests using AA-AILP were conducted under optimum conditions.

3. Results and Discussion

3.1 Characteristics of Leachate

The characteristics of the leachate were evaluated as per the APHA manual[19], and the results obtained were as follows: pH -8.8, COD -3489 mg/L, fixed solids -5800 mg/L, chlorides -2345 mg/L, dissolved solids -8569 mg/L, suspended solids – 760 mg/L, volatile solids -2890 mg/L, $BOD_{5,\ 20}$ -78 mg/L, and Cr (VI) -5 mg/L.

3.2 Adsorption Studies of the AILP Adsorbent

3.2.1 Reaction Time

The influence of reaction time on the adsorption characteristics of Cr (VI) using AILP at variable time intervals ranging between 15 and 180 minutes was determined, and the results are presented in Figure 2. At 120 min, the Cr (VI) removal percentage rapidly increased to 93%. A minimal removal percentage was observed upon further increasing the reaction time. The equilibrium time to remove these Cr (VI) ions was 120 min, which is primarily due to the initial availability of large vacant adsorption sites on the AILP. During the 120-minute contact period, the adsorbent (AILP) surface sites are exhausted. A similar finding was reported by Balasubramanian *et al.*,[5] and Patel[31], for the removal of Cr (VI) in synthetic wastewater using *Eichhornia crassipes* and *Lemna minor*.

3.2.2 Solution pH

The impact of variations in pH (3–11) was examined on the adsorption of Cr (VI), as shown in Figure 3. The highest Cr (VI) removal efficiency was 96% at a pH of 8.8, with an adsorbent dose of 1.5 mg/L, and an initial Cr (VI) concentration of 5 mg/L. When the pH increased from 10 to 11, the removal of chromium ions decreased. pH is influenced by the type of adsorbate, the adsorbent surface charge, and the ionisation level of the particle surface[32,33]. Electrostatic interactions are the primary mechanism that induces the surface of the AILP adsorbent to adsorb metal ions. Based on the solution concentration and pH, Cr (VI) has different anionic structures, such as CrO_4^{2-}, $Cr_2O_7^{2-}$, $HCrO^{4-}$, $H_2Cr_2O_7$, and HCr_2O_7. At low pH, chromium ions and hydrogen ions compete for suitable sites of the adsorbent on the surface due to the repulsion between sorbent and sorbate. This also results in a decline in the surface charge[17].

3.2.3 AILP Dosage

By utilising different amounts of adsorbent in the adsorbate solution, the adsorption process was performed, and the outcomes are presented in Figure 4. The highest Cr (VI) removal efficiency was observed as 92%. The removal percentage of Cr (VI) increased by 73% to 93% as the dosage of AILP increased from 0.1 to 1 g/L. This was primarily due to the availability of sufficient surface area

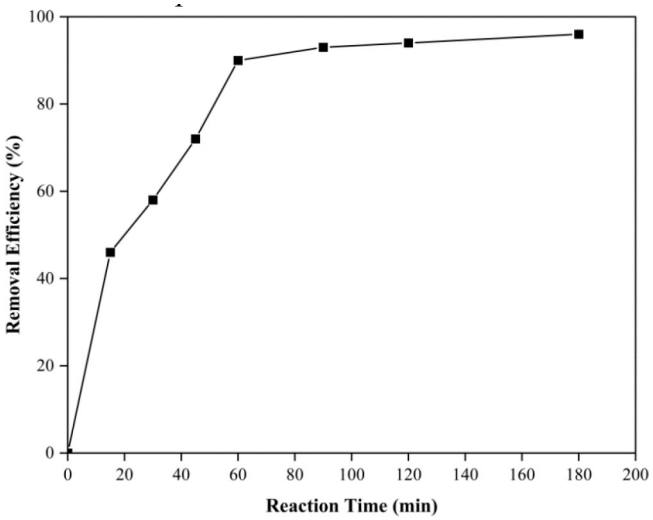


Figure 2. Effect of reaction time on percentage removal of Cr (VI) on AILP.

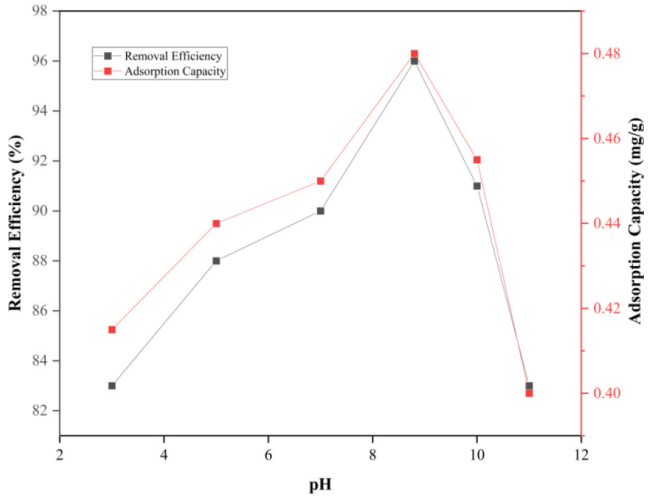


Figure 3. Effect of pH on the Cr (VI) removal percentage on AILP.

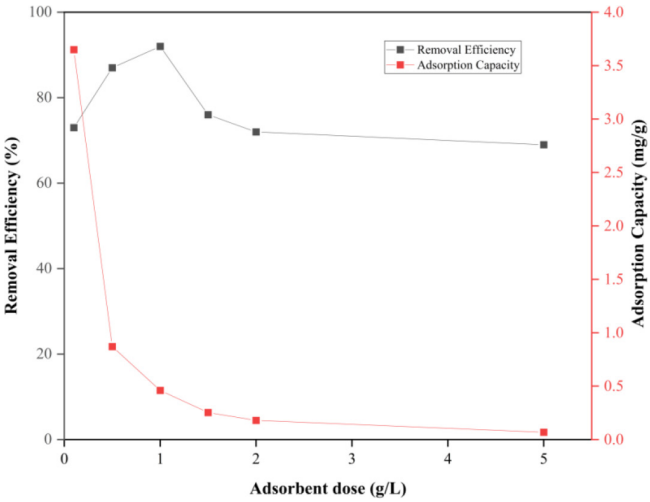


Figure 4. Effect of the AILP dose on the Cr (VI) removal percentage and capacity of the AILP.

and exchangeable active sites or functional groups present in AILPs[5,8,17]. However, increasing the AILP dose beyond 1.0 g/L decreases the Cr(VI) removal efficiency. This is because of the blockage of the active site or overlapping of adsorption sites or unsaturated binding sites, overcrowding of adsorbent particles, and the concentration of Cr (VI) ions reaching equilibrium in between the solution and solid phases, as well as an imbalance in the sorbate-adsorbent concentration[20,34,35]. When the AILP dose was increased from 1.5 to 5 g/L, the q_e decreased from 0.25 to 0.07 due to reduced usage of active sites[30].

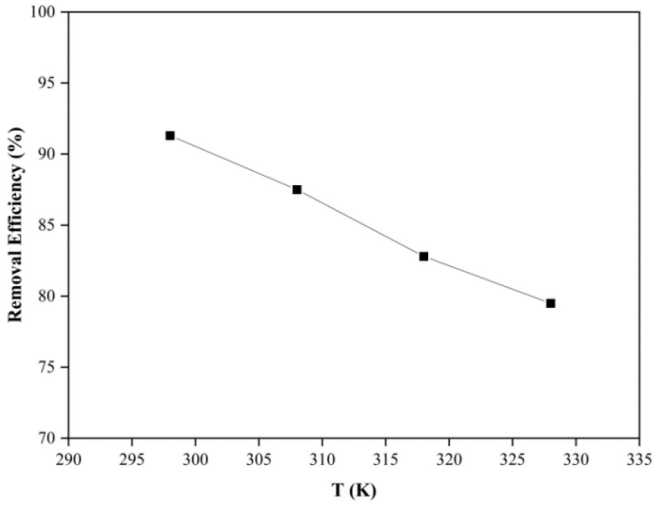


Figure 5. Impact of temperature on Cr (VI) removal efficiency.

3.2.4 *Impact of Temperature*

The effect of the temperature of AILP on the adsorption of Cr (VI) is presented in Figure 5. When the temperature increased from 298°K to 328°K, the adsorption efficiency significantly decreased from 91.3% to 79.5%, respectively. This is because, at high temperatures, the active adsorbent sites for binding with the adsorbate sites undergo reduction[16]. At lower temperatures, removal was high and is partially attributed to the strong attractive force between the sorbate and sorbent, which results in an exothermic adsorption process.

3.2.5 *Adsorption Kinetics*

The contact period plays a vital role because adsorption efficiency is significantly affected by the adsorption kinetics of the adsorption process. The pseudo-first and second-order models were applied to study the adsorption kinetics of the Cr (VI) on the AILP surface under optimal experimental conditions: pH of 8.8, AILP dose of 1g/L, time of 120 min, and temperature of 298°K. The results of fitting the adsorption kinetics by the models pseudo-first order and second order are shown in Figures 6(a) and 6(b). It is observed that the R^2 value (0.9866) of the pseudo-second order was higher than that of the pseudo-first order. However, the value of q_e was determined by curve fitting using the pseudo-second-order model as presented in Table 1. The

experimental results closely match the theoretical results. The electrons transfer between the sorbent and sorbate during the chemisorption process depends upon the functional adsorbent groups[36,37].

3.2.6 Isotherm Study

It is essential to examine the isotherm equilibrium of the removal mechanism of Cr (VI) in the AILP solution. The linear isotherms proposed by Freundlich and Langmuir are presented in Figures 7(a) and 7(b), and the mathematical model parameters are presented in Table 2. These results show the homogeneous and monolayer coverage of Cr (VI) on the AILP sorbent surface. The values R^2 of the adsorption process are determined as 0.9879 and 0.8905 for the isotherms proposed respectively by Langmuir and Freundlich. The Langmuir isotherm had a higher R^2 value compared with the Freundlich isotherm. Zhang *et al.*,[38] stated the same pattern of results. Thangagiri *et al.*,[21] reported that corn straw biochar and neem biochar (activated carbon) remove Cr (VI) by combining a chemical process with monolayer adsorption. The results show that the R_L value is 0.07617and the value of 1/n is 0.369. Stronger binding to the adsorbent is observed by the R_L in the range of 0 to 1 and a 1/n value between 0 and 1, which in turn indicates a favourable adsorption process at the optimum adsorption state.

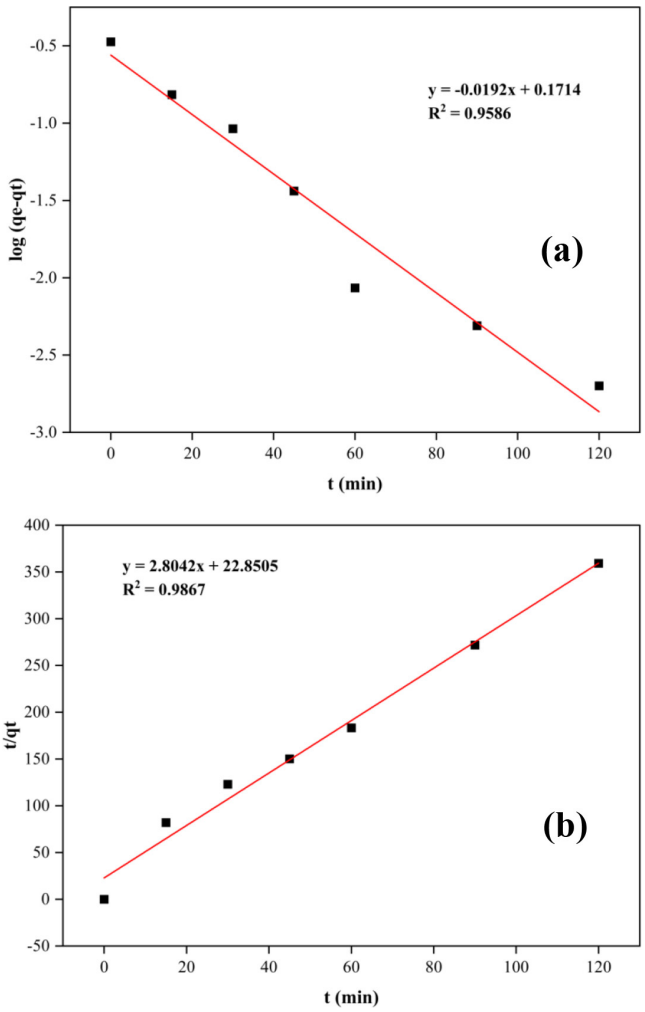


Figure 6. Kinetic models: a) first order and b) second order.

3.2.7 Thermodynamic Studies

To study the viability of the process of adsorption, Some of the thermodynamic variables such as change free energy (ΔG= kJ/mol), enthalpy change (ΔH= kJ/mol), entropy change (ΔS= kJ/mol.K), R- gas constant (8.314J/mol.K), and temperature in K were estimated using equations (8)-(10) and the K_d dimensionless equation calculated by Eq.11[8, 27,39,40].

$$\Delta G^0 = RT \ln k_\alpha \tag{8}$$

$$\Delta G^0 = \Delta H^0 - T\Delta S^0 \tag{9}$$

$$\ln k_\alpha = \frac{-\Delta H^0}{RT} + \frac{\Delta S^0}{R} \tag{10}$$

$$\ln k_\alpha = \frac{q_e}{lk_\alpha} \times \frac{m}{v} \tag{11}$$

The ΔS° and ΔH° values were determined at various temperatures by plotting a graph between ln K_d and 1/T. As illustrated in Figure 8, the plot displays a linear change with a slope and an intercept of ΔH°/R and ΔS°/R. The values of all thermodynamic parameters are calculated and presented in Table 3. The process of adsorption is exothermic due to the negative value ΔH°.

The outcome of the (ΔS) was calculated as positive values, which indicates an increase in the randomness at the sorbent/sorbate interface. The negative value of ΔG indicates the spontaneous adsorption process. This shows that less adsorption occurs at higher temperatures[16,41].

Table 1. Kinetic models of adsorption for Cr (VI) removal using the AILP adsorbent

Model study	Slope	Intercept	Constant parameters	R^2
Pseudo first order	-0.0192	-0.5598	q_e = 0.2755 mg/g (calculated). q_e=0.336mg/g (experimental). k_1 = 0.04422 min^{-1}.	0.9586
Pseudo-second order	2.8042	22.851	q_e = 0.3566 mg/g; (calculated). q_e=0.336mg/g; (experimental). k_2= 0.3441 g/mg. min	0.9867

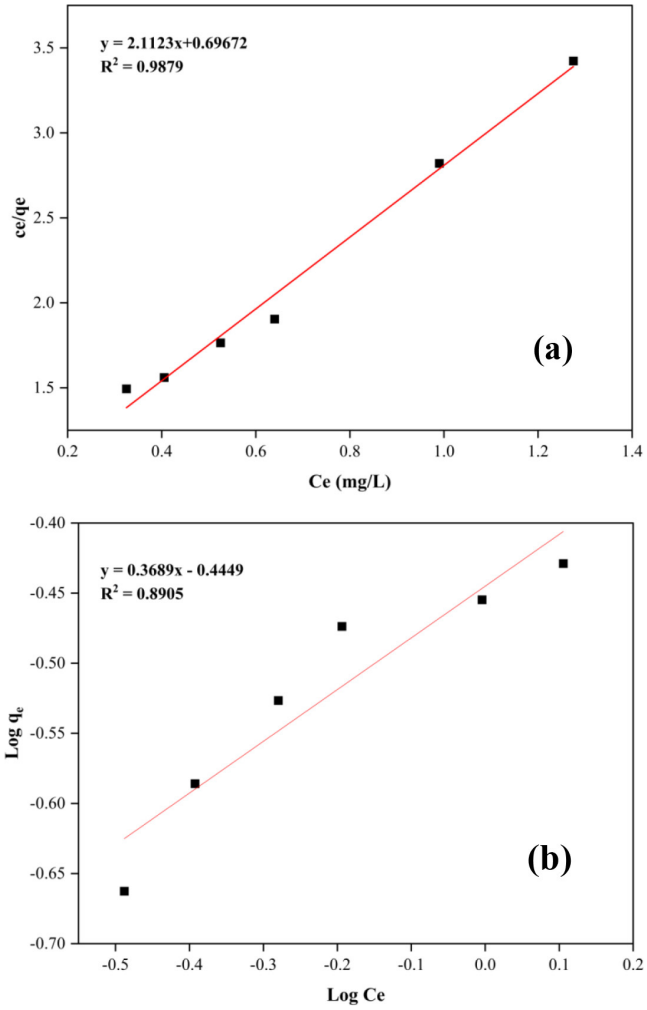


Figure 7. Isotherm models: a) Langmuir and b) Freundlich.

3.2.8 Reusability Study

The reusability test results of AA-AILP are displayed in Figure 9. The results showed that a similar adsorbent performance capability was obtained until the fifth cycle, after which the performance started to decrease with further increases in the recycling of AA-AILP. This is due to repeated use of the sorbent, and Cr (VI) may have blocked an adsorption site. Elkhaleefa *et al.*,[16] and Sadare *et al.*,[42] showed similar results when using *Azadirachta indica* leaves for lead and dibenzothiophene (DBT) removal from an aqueous solution.

Table 2. Isotherm study: a) Langmuir and b) Freundlich

Equilibrium Study	Constant parameters	R^2
a) Langmuir	K_L = 3.0320 Lmg^{-1} q_m = 0.47339 mgg^{-1} R_L = 0.07617	0.9879
b) Freundlich	K_F = 0.3589 mg/g; n = 2.7100; 1/n = 0.369	0.8905

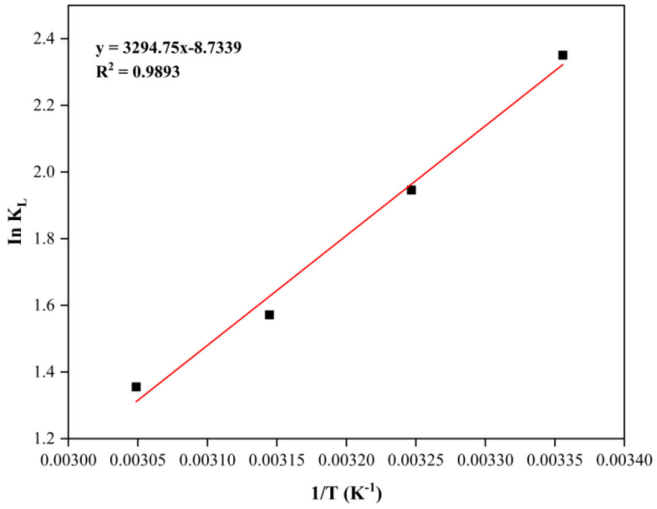


Figure 8. Plot of ln KD versus 1/T.

Table 3. Thermo kinetic parameters of Cr (VI) removal on ALIP

T, (K)	k_D	ΔG, kJ/mol	ΔS, J/mol K	ΔH, kJ/mol
298	10.49	- 5.82	72.61	-27.4
308	7	- 4.98		
318	4.81	- 4.15		
328	3.88	- 3.69		

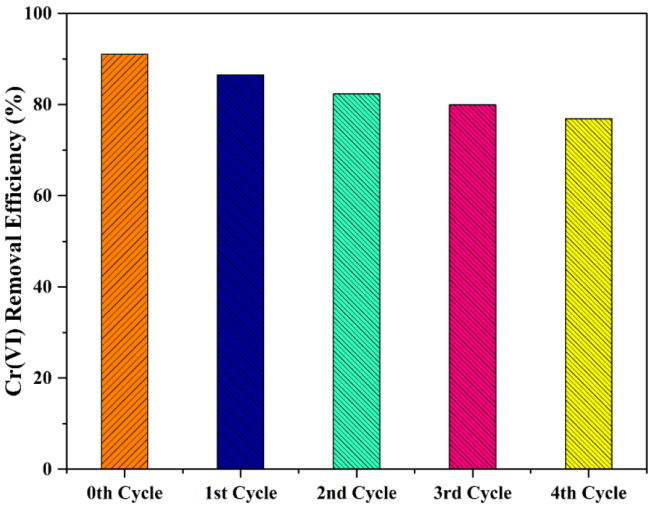


Figure 9. Reusability study of the AILP.

4. Conclusion

In the present investigation, the capacity of AILP to eliminate Cr (VI) from landfill leachate was studied using a batch technique. This study examined the impact of various experimental parameters on Cr (VI) removal from landfill leachate, namely, reaction time, pH, AILP dose, temperature, and concentration of Cr (VI). The maximum % removal of Cr (VI) in landfill leachate was 91% at the following optimized conditions: a reaction time of 120 min, an AILP dose of 1.0 g/L at pH 8.8, and a temperature of 298K. The isotherm models revealed that AILP had maximum monolayer and multilayer adsorption. The pseudo-second-order kinetic model was followed by adsorption kinetics and showed no significant resistance to the diffusion of the sorbent. The outcome of the current study strongly suggest that AILP may be utilized as a bio-adsorbent for toxic metal removal, such as Cr (VI), from polluted water and soil in geographic locations where neem trees grow in abundance.

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Author Contribution

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