



Effective removal of Cr (VI) ions using low-cost biomass leaves (*Sambucus nigra* L.) in aqueous solution

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Abstract

The tannery industries have become an important part of societal growth; however, these processes have produced huge volumes of effluents containing heavy metals, particularly Cr(VI) oxyanions. The study is crucial and cost-effective for reducing the chromium (VI) from industrial wastewater. In order to meet the sustainable development goal (SDG) objective 6.3, the capacity of *Sambucus nigra* L. to adsorb heavy metal is established with the purpose of eradicating hazardous chemical contamination and reducing pollution. In this study, discontinuous tests were carried out to determine the efficiency of Cr(VI) sorption on leaves of *Sambucus nigra* L. Adsorption factors such as pH, temperature, adsorbent dosage, and contact time were evaluated. At a dosage of 3 g/L and pH 2, an efficiency of 98.22% was achieved under favorable conditions. The equilibrium and kinetic models that best fitted the experimental data are non-linear Freundlich and pseudo-second order, and intra-particle diffusion, respectively. The thermodynamic parameters of the adsorption process, including Gibbs free energy (ΔG^0), enthalpy (ΔH^0), and entropy (ΔS^0), were measured at 291, 303, 323, and 343 K, indicating that the phenomena was spontaneous and endothermic. The chemical analyses and surface morphology of the adsorbent were analyzed using SEM (scanning electron microscopy), EDS (energy dispersive spectroscopy), FTIR (Fourier transform infra-red), XRD (X-ray diffraction), and ICP-OES (inductively coupled plasma optical-emission spectroscopy) techniques. The results showed that *Sambucus nigra* L. has a significant removal efficiency of Cr(VI) in the contaminated solutions, establishing adsorbent as a low cost, readily available, and environmentally friendly and ensuring its potential for industrial usage.

Graphical abstract.

Keywords *Sambucus nigra* L. · Adsorption of chromium · Heavy metal

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Introduction

Currently, industrial wastewater management is unsustainable, which has a detrimental impact on achieving sustainable development by 2030. The presence of metal ions and organic compounds in water supplies is a result of economic expansion and development (Mughal et al. 2022). Biological molecules, unlike metal ions, are mostly susceptible to organic reduction (Beksissa et al. 2021). Because of their high toxicity and bioavailability, metal ions are a constant source of worry in various ecosystems. In this sense, chromium metal is found in many ecosystems because of a lack of strategic management in corporate social responsibility (CSR) (Peng et al. 2020). Metallurgical industries, mining, tanneries, paints, batteries, radiators, smelters, and mineral weathering all emit considerable quantities of chromium (Rouhaninezhad et al. 2020; Thabede et al. 2020; Tao et al. 2021). Chromium's most prevalent oxidation states in the environment are Cr (III) and Cr (VI). Among them, Cr (III) is essential for cellular metabolism (sugars, fats, and proteins) in humans, animals, and plants (Cherdchoo et al. 2019). Soil (humic acid, pH, redox potential) and groundwater oxidize Cr (III), transforming it to Cr (VI) metallic element. Even in trace concentration, this metallic state is more harmful and toxic to public health because of its teratogenic, carcinogenic, and mutagenic properties (Cherdchoo et al. 2019; Shooto 2020b; Khalil et al. 2021).

According to the EPA (Environmental Protection Agency), the maximum allowed contamination level of water standard for total chromium is 0.1 mg/L (US EPA 2022). However, the WHO (World Health Organization) standard recommends 0.05 mg/L (WHO 2020; WHO, n.d.). In view of these negative effects and to improve public health, Cr (VI) ions must be removed prior to discharge. To date, technologies such as coagulation, filtration, chemical precipitation, reverse osmosis, ion exchange, and membrane systems have been used (Ray et al. 2020; Rouhaninezhad et al. 2020; Tejada et al. 2021). However, these technologies have certain drawbacks, such as high operating costs, generation of secondary pollution, and limited efficiency. In this sense, adsorption technology is considered a more efficient and easier to monitor (Cherdchoo et al. 2019), low cost, and environmentally friendly way (Shooto 2020b). Many adsorbents have been investigated for managing the Cr (VI) from industrial effluents, including coffee and tea residues (Cherdchoo et al. 2019), tea residues (Nigam et al. 2019), black cumin seed (Thabede et al. 2020), *Harpagophytum* residues (Shooto 2020b), *Eichhornia crassipes* and *Lemna minor* leaves (Balasubramanian et al. 2020), mango bark (Pathania et al. 2020), *Mentha piperita* residues (Al et al. 2020), *Acacia*

sawdust (Khalid et al. 2018), rice husk (Khalil et al. 2021), and water hyacinth residues (Kumar and Chauhan 2019). In this line, it is always interesting to search for new adsorbents that are affordable, accessible, effective, and easy to apply.

Sambucus nigra L., sometimes referred to as “Sauco,” is a member of the Caprifoliaceae family and is indigenous to Asia, Europe, and America (Ağalar 2019; Domínguez et al. 2020; Ruíz and Mejía 2020). It is a plant well-known for its physical, chemical, and biological properties. It is a tree or shrub that ranges in height from 1 to 12 m which has longitudinal fractures, deep furrows, and aged shafts. It has leaves with seven to nine leaflets that are imparipinnate, oblong, and pointed at the apex and have serrated edges that are 4 to 6 cm long and 3 to 7 cm wide (National Institute for the Defense of Competition and the Protection of Intellectual Property 2018; Ağalar 2019; Quiñones et al. 2020; Navas et al. 2021; Boroduske et al. 2021). The tree has been demonstrated to have antiviral qualities, which have been used to treat COVID-19 (Boroduske et al. 2021; Huaccho et al. 2020), as antioxidants to treat malignant cells (Filip et al. 2021), and for the food sector (Ağalar 2019). These goodnesses are due to chemical components such as polyphenols, phenolic acids, flavonoids, and tannins (Filip et al. 2021). In addition to these chemical elements, *Sambucus nigra* L. contains 41.9% fiber, 21.2% protein, 18.3% dry matter, and 19.4% carbohydrates (Quiñones et al. 2020); likewise, chemical components such as polysaccharides (cellulose, hemicellulose, and lignin), these chemical groups characterize the biomaterial as a best option for the treatment of heavy metals from industrial effluents (Kumar and Chauhan 2019).

Sambucus nigra L. has not yet been studied for its potential to adsorb heavy metals. Therefore, based on the aforementioned criteria, the biomass leaves were employed as an adsorbent to control the chromium (VI) from synthetic solutions. FTIR, SEM, and X-ray diffraction analyses were performed to know the surface characteristics and determine the chromium (VI) adsorption, and the adsorption experiments were carried out batch-wise and it was studied based on evaluating the effect of contact time, adsorbent dosage, pH of the sample, and adsorption capacity in aqueous solution.

Materials and methods

Materials and reagents

The chemical reagent used for the execution of the research was highly pure and analytical grade. Potassium dichromate ($K_2Cr_2O_7$) of 99.0% purity, purchased from Spectrum

Chemical Mfg. Corp. All the glass materials were rinsed with double distilled water and dried in an oven at 55 °C before use.

Preparation of the bioadsorbent

Four kilograms of *Sambucus nigra* L. leaves was collected from the university campus of the Universidad Nacional del Centro del Perú, washed with tap water to remove dust and other adhered particles, and then rinsed with double distilled water. They were then dried at 65 °C for 72 h until they reached a constant weight, ground, and sieved with mesh No. 60 (ASTM), labeled, and stored in drying conditions until further use.

Batch adsorption studies

A standard chromium stock solution of 1000 mg/L was made by dissolving the required amount of potassium dichromate in double distilled water. Working solutions at different concentrations were made by successive dilutions of the standard solution. Adsorption experiments were performed in 50 mL of 10 mg/L chromium (VI) solution at a dose of 3 g/L of adsorbent. The pH of the solution was adjusted by adding a few drops of 0.1 N HNO₃ or 0.1 N NaOH. After the contact process, they were filtered with Whatman N° 41 filter paper. The residual Cr (VI) concentration was analyzed using the ANALYTIK JENA NOVA PRO-400 atomic absorption spectrophotometer. Sorption tests were conducted at various Cr (VI) concentrations, pH levels, temperatures, reaction times, and adsorbent dosages. The adsorption capacity of *Sambucus nigra* L., q_e (mg/g), was determined using the mass balance equation as presented in Eq. 1.

$$q_e = \frac{(C_o - C_e)}{m} * V \quad (1)$$

where C_o (mg/L) and C_e (mg/L) represent the initial and equilibrium concentration of Cr (VI) respectively, V is the volume of the solution (L), and m is the weight of the dry biomass (g).

The percentage removal capacity of Cr (VI) was calculated by using the Eq. 2:

$$\%R = \frac{C_o - C_f}{C_o} * 100 \quad (2)$$

where C_o and C_f (mg/L) are the initial and final Cr (VI) concentration before and after adsorption, respectively. The maximum adsorption capacity and the efficiency of the sorbate-biosorbent phenomenon were determined in relation to the experimental adsorption data.

Equilibrium study

To determine the sorbent-sorbate ratio at different Cr (VI) concentrations and equilibrium data, adsorption isotherms were modeled. In this study, Langmuir and Freundlich equilibrium isotherms were used.

Langmuir isotherm model

The Langmuir isotherm (Langmuir 1918) suggests that the phenomenon occurs on a homogeneous surface forming a monolayer when the adsorbent has reached saturation (each active site was occupied by one element), with no interaction between the adsorbed metals, uniform energy, and no transmission of the adsorbate (Ighalo and Adeniyi 2020).

The linearized Langmuir equation is presented in Eq. 3.

$$\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{C_e}{q_m} \quad (3)$$

where q_m (mg/g) is the maximum adsorption capacity by the adsorbent on the monolayer and K_L (L/mg) is the energy constant of the adsorption process. The values of q_m and K_L (Langmuir constant) were determined from the slope and the interaction of the C_e/q_e vs C_e figure. Also, the feasibility of the adsorption process was determined by the dimensionless factor (R_L). The R_L values were determined based on the Eq. 4:

$$R_L = \frac{1}{1 + K_L C_o} \quad (4)$$

The R_L values represents whether the adsorption is unfavorable or favorable. Unfavorable when $R_L > 1$, linear when $R_L = 1$, favorable when it is between $0 < R_L < 1$ and irreversible when $R_L = 0$.

Non-linear Langmuir equation is shown in Eq. 5.

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

The Freundlich isotherm

This model proposes a multilayer sorption with interactions between the adsorbed particles and heterogeneous energy distribution at the active sites, i.e., the binding forces between the sorbent-sorbate are stronger at the beginning of the process and decreases as the level of accumulation increases. The linearized form of the Freundlich isotherm (Freundlich 1906) is shown in Eq. 6. The non-linear equation is presented in Eq. 7.

$$\text{Log}q_e = \text{Log}K_F + \frac{1}{n}\text{Log}C_e \tag{6}$$

$$q_e = K_F C_e^{\frac{1}{n}} \tag{7}$$

where q_e is the amount of contaminant removed (mg/g), K_F (mg/g) is the Freundlich’s constant, C_e is the equilibrium concentration (mg/L), and n is related to adsorption capacity and adsorption intensity. The values of K_F and n are determined from the slope and intercept of the linear figure $\text{Log } q_e$ vs $\text{Log } C_e$.

Study of the kinetics

The main factors in the adsorption process are mass transfer, chemical reaction, and reaction rate (Ighalo and Adeniyi 2020; Tejada et al. 2021). The mass transfer kinetics is described by the velocity between the aqueous and solid phase (Bazzazzadeh et al. 2020). To calculate the removal rate, the following times were considered: 2, 4, 15, 25, 35, 55, 100, 130, and 150 min with a concentration of 10 mg/L.

Pseudo-first-order model

The model refers that the mass transfer of the adsorbate ions is corresponding to the active sites on the adsorbate surface could due to carbonyl group, alcohol, carboxylic acid, or phenols, N–H bend of amine with an unsaturated hydrocarbon, alkyl halides. The linearized form of the pseudo-first-order equations is presented in Eq. 8.

$$\text{Log}(q_t - q_e) = -\frac{K_1}{2.303}t + \text{Log}q_e \tag{8}$$

where q_t and q_e (mg/g) are the amount of Cr (VI) adsorbed at time t (min) and equilibrium, respectively and first-order rate constant is represented by K_1 (min^{-1}). The value of q_e and K_1 was determined from the intercept and slope of the $\log (q_e - q_t)$ vs t plot. The non-linear form of pseudo-first-order equation is shown in Eq. 9.

$$q_t = q_e [1 - \exp(-k_1 t)] \tag{9}$$

Pseudo-second-order model

To determine the adsorbate contact time at the solid–liquid interface, adsorption mechanism depending on the physical and chemical characteristics of the adsorbent, crucial factors in the construction of wastewater treatment facilities. Furthermore, this model characterizes all the processes of adsorption, such as internal-exterior particle diffusion, and describes the chemical processes involving valence forces via electron

exchange between adsorbent and adsorbate. The linearized equation of the model is shown in Eq. 10.

$$\frac{t}{q_t} = \frac{t}{q_e} + \frac{1}{K_2 q_e^2} \tag{10}$$

where K_2 is the second-order rate constant (mg/g min). The value of K_2 and q_e were estimated from the intercept and slope of the t/q_t vs t figure. The non-linear form of pseudo-second-order equation is presented in Eq. 11.

$$q_t = \frac{q_e^2 K_2 t}{1 + q_e K_2 t} \tag{11}$$

The kinetic of adsorption process was also evaluated by the Webber’s pore-diffusion equation which is shown in Eq. 12.

$$q_t = K_{int} t^{\frac{1}{2}} + C \tag{12}$$

where K_{int} is the rate constant for the intraparticle diffusion rate.

Thermodynamic studies

Thermodynamic parameters determine the nature of the adsorption procedure (Nigam et al. 2019). For the thermodynamic analysis of the sorption process, the Gibbs free energy change (ΔG°), entropy change (ΔS°), and enthalpy change (ΔH°) were determined in relation to temperatures 291, 303, 323, and 343 K. The calculation of the thermodynamic parameters using the adsorption equilibrium constant and K_d is presented in Van’t Hoff Eqs. 13 and 14. K_d dimensionless equation explained by Lima et al. (2019) is considered as shown in Eq. 15.

$$\Delta G^\circ = \Delta H^\circ - T \Delta S^\circ \tag{13}$$

$$\text{Ln}K_d = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \tag{14}$$

$$K_d = \frac{q_e}{C_e} \times \frac{m}{V} \tag{15}$$

where ΔG° is the Gibbs free energy (kJ/mol), ΔH° is the sorption enthalpy variation (kJ/mol), T is the temperature (K), ΔS° is the entropy (kJ/mol K), R is the gas constant (8.314 J/mol K), m is the mass of the adsorbent, and V is the volume of the adsorbate solution.

Results and discussions

The effectiveness of adsorption processes of experimental batch technique is determined by using the several factors (pH, adsorbent dosage, contact duration).

Effect of pH

The pH point of zero charge (pHpzc) of the adsorbent is a very important factor in the sorption process, which is defined as the pH value of the aqueous solution at which the net surface charge of the sorbent is neutral or null (Labied et al. 2018) and was determined following the experimental protocol defined by Lavado-Meza et al. (2021), for which a series of solutions was prepared with 0.05 g of adsorbent in 50 ml of deionized water, initial pH levels from 2.0 to 12.0, pH were adjusted using 0.1 M solutions of HCl and NaOH, and samples were shaken at 150 rpm at 20 °C for 24 h. The final pH levels were then measured and the final pH values (pH_f) were plotted against the initial pH values (pH_i). The pHpzc of the adsorbent was 5.86, which is shown in Fig. 1. This implies that at pH values lower than pHpzc the adsorbent acquired positive charges determining a higher performance in the removal of Cr (VI) anions, likewise, at pH > pHpzc, the adsorbent acquired negative charges significantly reducing the removal of Cr (VI) ions. This is due to strong electrostatic attraction between the positively charged surface of the adsorbent and the dichromate (Cr₂O₇²⁻) and chromate (HCrO₄⁻) anion species (in acidic medium, anion HCrO₄⁻ is the more dominant) of the adsorbate, resulting in increased adsorption efficiency. These results are similar to those obtained by Baruffi et al. (2019) and Haroon et al. (2020).

Figure 2 shows the adsorption capabilities of *Sambucus nigra* L. adsorbent to adsorb Cr (VI) at various pH levels (range of 2 to 12) with chromium species distribution as described by Shen and Ke (1986) and Spessato et al. (2021). During the removal process, pH becomes a crucial parameter for determining adsorption effectiveness. The maximum

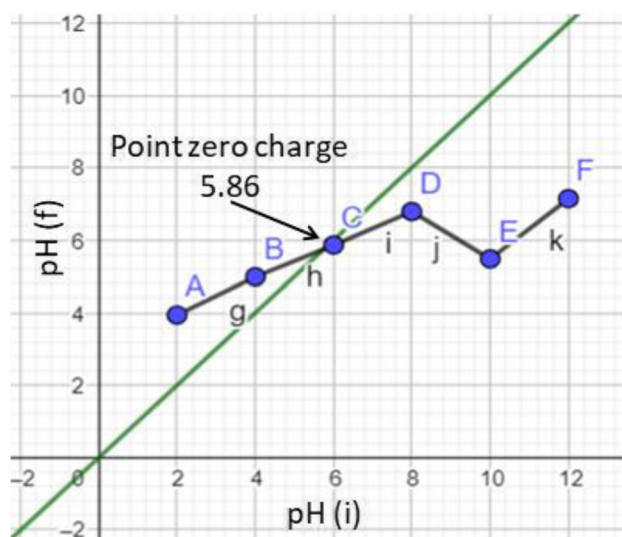


Fig. 1 pH point of zero charge (pHpzc) graph

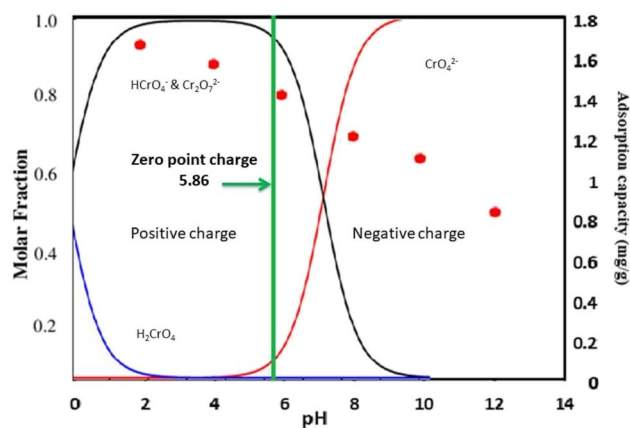


Fig. 2 Effect of pH on Cr (VI) adsorption with Cr species distribution

removal efficiency was achieved at pH = 2.0 (98.2%) with a q_e of 1.64 mg/g, while the minimum efficiency at pH = 12 was 49.8% with a q_e of 0.83 mg/g.

At different pH levels, Cr (VI) appears to take on distinct ionic forms. Cr (VI) occurs mostly in the form of HCrO₄⁻, and Cr₂O₇²⁻ in the pH range 2.0 to 6.0, with HCrO₄⁻ dominating (Ghorbani et al. 2020; Pathania et al. 2020). The maximum removal was determined due to electrostatic attraction at acidic pH between HCrO₄⁻ ions and the adsorbent surface (protonated H+) with positive charge (below the zero charge point 5.86), which leads to an increase in the amount adsorbed; however, a decrease in removal was observed at pH 6.0 to 12, which could be due to negative charge on adsorbent surface, resulting in a reduction of the adsorbed amount. According to Saha and Orvig (2010), there are four mechanisms for removing chromium from aqueous solution: (1) anionic adsorption, (2) reduction coupled to adsorption, (3) cationic and anionic adsorption, and (4) reduction and anionic adsorption. Consequently, the Cr (VI) adsorption ions could be referred to as mechanism 4 (reduction and anionic adsorption). A fraction of Cr (VI) would be reduced to Cr (III) by the action of the functional groups on the surface of the biomaterial and dissolving in the solution, while the majority of Cr (VI) ions would be adsorbed by the adsorbent via electrostatic attraction (Ghorbani et al. 2020; Pathania et al. 2020; Shooto 2020b).

Effect of adsorbent dose

The adsorption capacity of the adsorbent at a defined initial concentration depends on the dose of the biomaterial. The present study employed an initial concentration of 5 mg/L Cr (VI), a contact time of 25 min, and a pH of 2 to investigate the effect of adsorbent dose ranging from 1 to 5 g/L.

As demonstrated in Fig. 3, increasing the adsorbent dosage from 1 to 3 g/L increased the adsorption percentage from

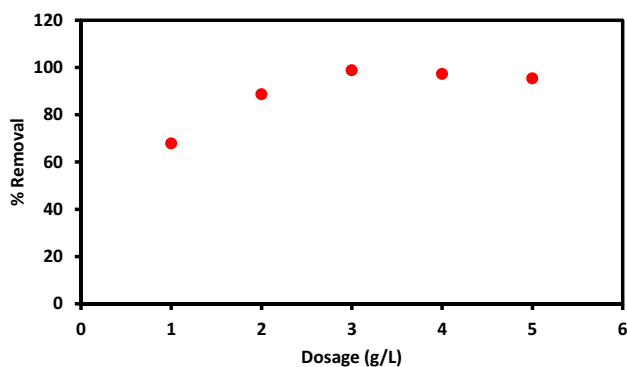


Fig. 3 Effect of adsorbent dosage (g/L) on adsorption of Cr (VI)

67.86 to 98.78. Indeed, this rise reflects an increase in the number of active groups (carbonyl group, alcohol, carboxylic acid, or phenols, N–H bend of amine with an unsaturated hydrocarbon, alkyl halides) as well as an expansion of contact surface areas. In addition, at doses ranging from 4 to 5 g/L, chromium (VI) adsorption was reduced from 97.24 to 95.34%. This occurrence was presumably caused by adsorption site overlapping, an imbalance in sorbate-adsorbent concentration, and overcrowding of biosorbent particles in the solution (Al et al. 2020; Bazzazzadeh et al. 2020). According to Khalil et al. (2020), the low removal rate of Cr (VI) with increasing adsorbent dose could be due to unsaturated binding sites, which shows that the adsorption sites increase with increasing adsorbent dose, but the ratio between the doses determined with the initial ratio of Chromium (VI) ions adsorption is reduced.

Effect of contact time

The percentages of Cr (VI) removal capacity as a function of time are shown in Fig. 4. In an aqueous solution at a concentration of 15 mg/L, pH=2, 3 g/L adsorbent, and 18 °C, the impact of contact time (2–150 min) was studied.

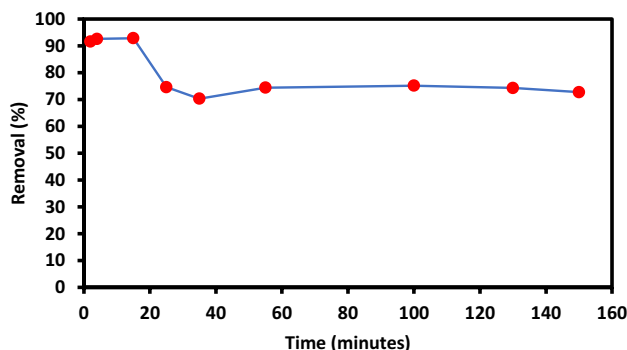


Fig. 4 Effect of contact time on adsorption of Cr (VI) via *Sambucus nigra* L.

The removal capacity of Cr (VI) by the adsorbent *Sambucus nigra* L. was very fast in the initial phase, achieving a removal of 92.83% in the first 15 min, but this effect gradually decreased with time, most likely due to competition between the reduction of surface active sites and Cr (VI) ions, so that after 35 min, 70.31% was removed. At the beginning of the sorption process, the percentage of chromium removal was higher due to a higher availability of the surface area and the porosity of the adsorbent (Haroon et al. 2020; Nnadozie and Ajibade 2020). The slight increase was observed from 55 min; this could be explained by the longer agitation time in the sorption process (Khalil et al. 2021). After attaining the maximal adsorption capacity, equilibrium was attained, indicating that all the adsorbent’s active sites were completely saturated with Cr (VI) and that the adsorbent had no more accessible functional groups to react the metal. These results are consistent with findings reported by Achary et al. (2020), Khalil et al. (2020), and Al et al. (2020).

Adsorption isotherm

To understand the Cr (VI) removal process in solution on the adsorbent *Sambucus nigra* L., it was necessary to analyze the equilibrium isotherm. The linear Langmuir equilibrium figure is presented in Fig. 5a, and the calculated parameters are summarized in Table 1. At 18 °C, the maximum adsorption

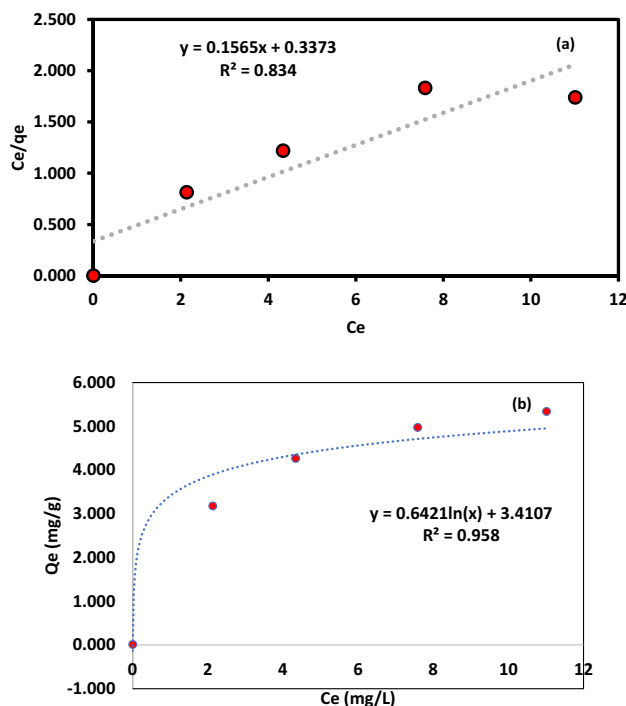


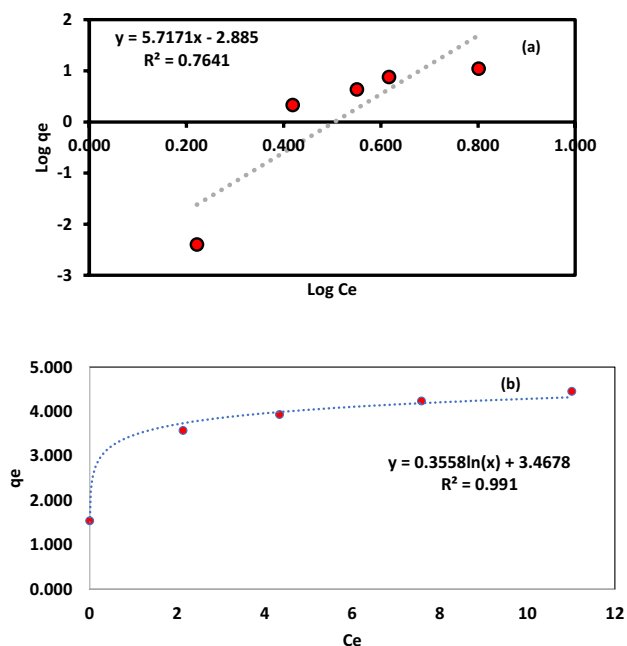
Fig. 5 Langmuir models for Cr (VI) adsorption: (a) Linear, (b) non-linear regression

Table 1 Estimated constants for the linear Langmuir and Freundlich models

Isotherm models	Parameters	Values
Langmuir	q_m (mg/g)	6.389
	K_L (L/mg)	0.464
	R_L	0.301
	R^2	0.834
Freundlich	K_F (mg/g)	0.001
	N	0.175
	R^2	0.764

capacity (q_m) for complete coverage on the monolayer was 6.389 mg/g. This result shows that the nature of the adsorbent surface was homogeneous. Also, the R_L value is in the range of 0 to 1, which indicates that the adsorption process is favorable. These results agree with Nigam et al. (2019), Pathania et al. (2020), and Thabede et al. (2020).

The Freundlich isotherm is shown in Fig. 6a, and the equilibrium parameters are presented in Table 1. The value of $1/n$ comprised between 0 and 1 indicates a stronger binding to the adsorbent, i.e., a chemical process, whereas a value of $1/n$ more than 1.0 evidences an unfavorable removal process (Cherdchoo et al. 2019; Al et al. 2020). The result of $1/n = 5.72$ being more than 1. Consequently, the adsorption process was not optimal with respect to the value of $K_F = 0.001$ mg/g, concluding that the laboratory analyses did not accommodate the linear form of Freundlich equation.

**Fig. 6** Freundlich models for Cr (VI) adsorption: (a) Linear, (b) non-linear regression

The non-linear isotherms of Langmuir and Freundlich models are shown in Figs. 5b and 6b, respectively. The summarized data of non-linear models of Langmuir and Freundlich isotherms are shown in Table 2. The adsorption capacity of 1.557 (mg/g) can be attributed to a low initial solute concentration and a high adsorbent dosage which provides more adsorption active sites that even after the removal process remain unsaturated (Kumar and Chauhan 2019; Wang et al. 2020). In this regard, Cr (VI) ion forms a homogeneous monolayer with the active sites, so that when the adsorbent surface has formed a single layer, this can make the adsorption process not to continue forming adjacent layers (Neolaka et al. 2020). The non-linear models show that the R^2 values of adsorption process are 0.958 and 0.991 for Langmuir and Freundlich isotherms, respectively. These results are higher compared to linear model; hence, non-linear isotherm is better fit for the study.

Kinetic study

The modelling of heavy metal removal rate equations in liquid systems is to be analyzed using non-linear regression (Tan and Hameed 2017). The adsorption rate consists of three steps: the transfer of adsorbate to the adsorbent surface (film diffusion), the transfer of adsorbate from the surface to the active sites (intraparticle diffusion), and the binding of adsorbate to the active sites (Chandana et al. 2018). To determine the mechanisms involved in the sorption rate process, non-linear, pseudo-first order, pseudo-second order, and intra-particle diffusion kinetic models were analyzed, as shown in Table 3. To determine which of the models best fit the experimental data, the correlation value (R^2) closest to unity (1) was used. The pseudo-first order, pseudo-second order, and intra-particle diffusion values were compared, noting that the pseudo-second order (0.974) and intra-particle diffusion (0.928) equations showed higher correlation (R^2) values closer to unity. The good fit the pseudo-second order and intraparticle diffusion models which show that the removal of Cr (VI) ions was based on electrostatic interactions (Khalil et al. 2020). The models determined that

Table 2 Estimated constants for the non-linear Langmuir and Freundlich models

Isotherm models	Parameters	Values
Langmuir	q_m (mg/g)	1.557
	K_L (L/mg)	0.188
	R_L	0.301
	R^2	0.958
Freundlich	K_F (L/mg)	2936.3
	N	2.811
	R^2	0.991

Table 3 Calculated constants of the kinetic models for Cr (VI) adsorption on *Sambucus nigra* L. based on linear and non-linear models

Kinetic models	Parameters	Values
Linear model		
Pseudo-first order	K_1 (1/min)	0.003
	q_e (mg/g)	4.869
	R^2	0.848
Pseudo-second order	K_2 (g/mg min)	0.029
	q_e (mg/g)	2.412
	q_e (projected) (mg/g)	1.353
	R^2	0.996
Non-linear model		
Pseudo-first order	K_1 (1/min)	3.135
	q_e (mg/g)	4.650
	R^2	0.864
Pseudo-second order	K_2 (g/mg min)	2.847
	q_e (mg/g)	2.350
	q_e (projected) (mg/g)	1.353
	R^2	0.974
Intraparticle diffusion model	K_1 (1/min)	0.745
	R^2	0.928

adsorption occurred on the surface or through the pores of the adsorbent, characterizing it as chemical sorption or chemisorption.

Study of thermodynamic properties

Temperature variation affects the adsorption procedure by modifying the surface activity of the adsorbent along with the kinetic energy of the metal ions (Lima et al. 2019; Bazzazzadeh et al. 2020). The increase in Cr (VI) removal was observed by varying the temperature in the range 291–343 K. This effect conditioned the mobility of Cr (VI) ions and as well as the activity of the adsorbent functional groups (Shooto 2020a). The calculated values of the thermodynamic equilibrium parameters are presented in Table 4. Figure 7 shows that the sorption capacity of Cr (VI) increased as the solution temperature was higher. This effect demonstrates that high temperatures provide the metal ions with sufficient kinetic energy to overcome all the forces that hinder the removal processes (Shooto 2020a).

The positive value of ΔH° defines the endothermic condition in Cr (VI) removal. The result of entropy variation

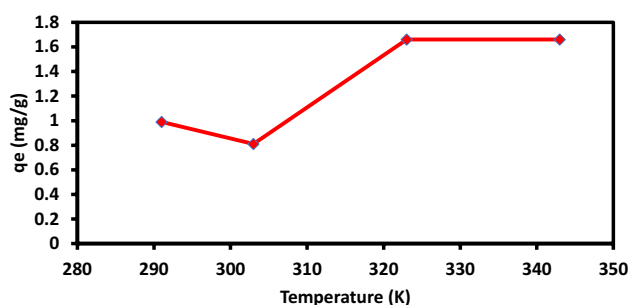


Fig. 7 Effect of Temperature on adsorption of Cr (VI) via *Sambucus nigra* L

$\Delta S^\circ = 0.40$ points to higher randomness at the solid–liquid interface, demonstrating higher sorption capacity of Cr (VI) ions (Mathai et al. 2022). It has been observed that the free energy changes (at temperatures of 303–343 K) are negative, while at 291 K, it is positive. Therefore, the adsorption of Cr (VI) at room temperature is not spontaneous, while at higher temperatures, it is spontaneous and feasible; these comparable results are reported by (Thabede et al. 2020; Al et al. 2020).

Comparative study

Adsorption is considered the most convenient, cost-effective, and user-friendly method for the removal of metal ions from an aqueous solution. Researchers have studied many adsorbents for the removal of pollutants from wastewater, such as agricultural wastes, forestry wastes, hydrogels, activated carbon, and nanoadsorbents (Mathai et al. 2022; Saha and Orvig 2010; Wang et al. 2020; Yadav et al. 2022; Yusuff et al. 2022). In this regard, the efficiency of metal ion removal depends on many factors, such as the composition of the adsorbents and the types of metals. This is one of the main reasons why adsorption studies have become one of the most active and frequent works of scientists.

Biosorbents with high adsorption capacity, low economic cost, high availability, less sludge generation, reusability, non-toxic, and environmentally friendly nature are characteristics that open a line of research to study new biomaterials specific to each region. In particular, biosorbents obtained from agricultural, animal and industrial waste are of great interest. However, the choice of a biosorbent for a given metal is not easy and requires many experiments and in-depth research (Singha et al.

Table 4 Thermodynamic analysis for Cr (VI)

ΔG° (KJ/mol)				ΔH° (KJ/mol K)	ΔS° (KJ/mol K)	Ea (KJ/mol)
Temperature K						
291	303	323	343	117.21	0.40	– 105.92
0.52	– 4.29	– 12.31	– 20.33			

2011). Forest residues such as elderberry leaves generated from industrial activity can be a promising material for adsorption research. This biomass is composed of dietary pigments; anthocyanins; flavonoids; lectins; lupeol; β -sitosterol; holocalin; prunasin; zierin; rutin; sambunigrin; tannin; choline; beltulin; vitamins A, B1, B2, B3, B5, B6, B9, C, and P; and minerals (Boroduske et al. 2021; Kalak et al. 2020). These substances contain polar functional groups, such as carboxyl, phenolic, hydroxyl, sulpho, and amino groups that are able to bind metal ions. The binding mechanism may involve reactions such as electrostatic interactions, complexation, microprecipitation, and chelation (Khalil et al. 2021; Saha and Orvig 2010). Table 5 shows the comparative data of the results achieved by the leaves of *Sambucus nigra* L. in the treatment of wastewater having Cr (VI) with other biomasses reported in the literature. The comparison revealed that the adsorbent generated by the leaves of *Sambucus nigra* L., as leftover biomass from agroforestry and industrial activity, is a low-cost adsorbent with superior performance in the removal at low concentrations and without chemical activation. Consequently, it is worthwhile to study the removal capacity of Cr(VI) ions with native forest residues. The *Sambucus nigra* L. (elderberry) tree grows rapidly and is widely available in many regions of Peru, producing large quantities of leaves and bark that are discarded as solid waste.

Elderberry leaves have never been explored as a adsorbent for Cr(VI) removal in Peru, and this is the first study which show the adsorption capacity of native *Sambucus*

nigra L. These results demonstrate the significance of this biomass in industrial effluent remediation applications.

Adsorption mechanism of Cr(VI) via FTIR spectrum

The characterization of Cr(VI) adsorption mechanisms through *Sambucus nigra* L. (SNL) leaves using Fourier transform infrared (FTIR) spectra is presented in Fig. 8. The spectrum of Cr(VI) before and after adsorption was examined between 4000 and 400 cm^{-1} wavenumbers, which is shown in the figure as SNL and SNLCr(VI) for before and after adsorption, respectively. The peaks at 2921 and 2851 cm^{-1} correspond to alkane C-H stretching, whereas the peaks at 1730 and 1713 cm^{-1} indicate C=O stretching (carbonyl group), as shown by both spectra SNL and SNLCr (VI) (Moldovan et al. 2016). The band 1651 cm^{-1} corresponds to C=C vibrations, while the band 1683 to 1685 cm^{-1} for SNL to SNLCr(VI) spectra shows a minor shift. After Cr adsorption, the spectrum exhibits peaks at 1643, 1632, 1623, 1614, 1577, 1572, 1567, 1540, 1530, 1508, 1498, 1488, 1427, 1386, 1269, 806, 757, 520, and 464 cm^{-1} which are absent from SNL spectra. The FTIR spectra range from 4000 to 400 cm^{-1} is shown in Fig. 8I, and two range graphs are shown in Fig. 8II and III to better comprehend the peaks changes before and after adsorption. Spectrum peaks on the surface of SNLCr(VI) obtained at 1643, 1632, 1623, and 1614 cm^{-1} (in Fig. 8 zone a) are ascribed to the C=C stretching, indicating the presence of alkene groups. Chromium may attach to unsaturated alkene molecules. Peak 1594 cm^{-1} is not present in

Table 5 Comparison of Cr (VI) adsorption capacity with other adsorbents

Adsorbent	Maximum capacity (mg/g)	Dose g/L	T° C	pH	Best fitting isotherm and kinetics	References
Coffee and tea residue	87.72 94.34	2	30	2	Freundlich and pseudo-second order	(Cherdchoo et al. 2019)
Tea residues	79.08	6	30	3.9	Langmuir and pseudo-second order	(Nigam et al. 2019)
Black cumin seeds	9.98	1	40	1	Langmuir and pseudo-second order	(Thabede et al. 2020)
Harpagophytum residues	77.24	0.19	35	1	Freundlich and pseudo-second order	(Shooto 2020b)
Hyacinth and Lemnaminor Leaves	79.24 61.0	1 1	25 ± 1 25 ± 1	2 2	Langmuir and pseudo-second order; Langmuir and pseudo-second order	(Balasubramanian et al. 2020)
Mango bark	78.96	0.12	30	2	Langmuir and pseudo-second order	(Pathania et al. 2020)
Mentha piperita	29.23	0.45	25	2	Freundlich and pseudo-second order	(Al et al. 2020)
Acacia sawdust	6.34	4.9	30	2	Langmuir and pseudo-second order	(Khalil et al. 2020)
Rice husk	379.63	0.6	20 ± 2	5.2	Langmuir and pseudo-second order	(Khalil et al. 2021)
Hyacinth root	1.28	14	25 ± 5	3	Freundlich and pseudo second order	(Kumar & Chauhan 2019)
Acorus calamus	14.64	0.2	25	1	Langmuir and pseudo-second order	(Shooto 2020a)
<i>Sambucus nigra</i> L	6.39	3	18	2	Non-linear Freundlich, pseudo-second order, and intra-particle diffusion	Present study

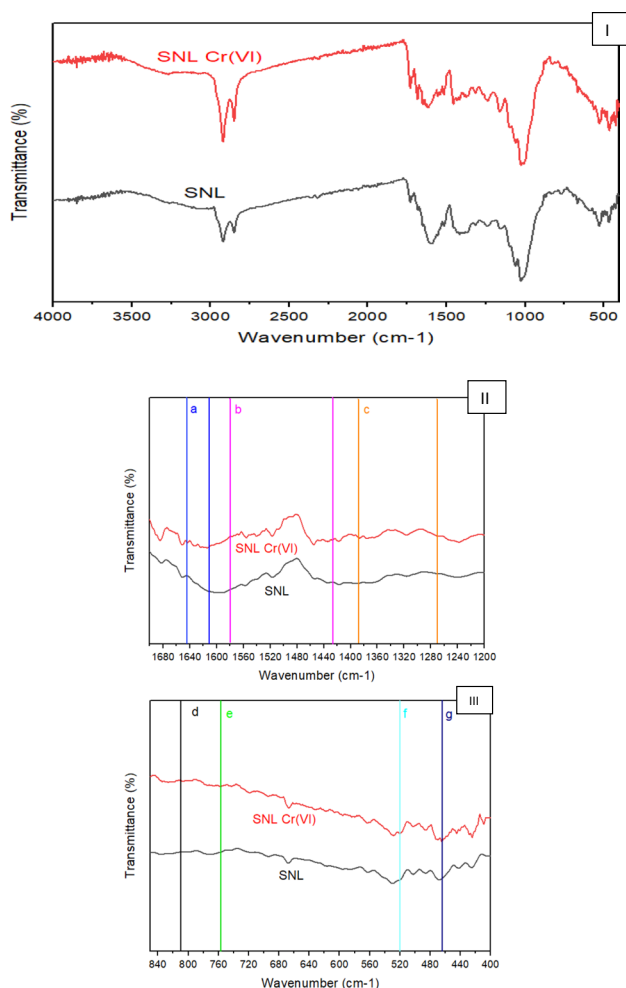


Fig. 8 FTIR spectrum graph of SNL and SNL Cr(VI) (before and after adsorption process). (I), Complete spectra range from 4000 to 400 cm^{-1} . (II), Spectra range from 1700 to 1200 cm^{-1} . (III), Spectra range from 860 to 400 cm^{-1} .

SNLCr(VI); however, a few additional peaks (1577, 1572, 1567, 1540, 1530, 1508, 1498, 1488, and 1427 cm^{-1}) (in Fig. 8 zone b) exist, indicating peak displacement owing to chromium loading on the surface of SNL. These peaks correspond to the N–H bend of amine with an unsaturated hydrocarbon functional group. The existence of chromium in the form of $(\text{NH}_4)_2\text{Cr}_2\text{O}_7$ is strongly supported by the N–H bending vibrations at these peak ranges (Schutte and Heyns 1970; Monico et al. 2016). Peaks 806 and 757 cm^{-1} are in the chromate (CrO_4^{2-}) region, which are the source of asymmetric bridge (Cr–O–Cr) vibrations in $\text{Cr}_2\text{O}_7^{2-}$ and Cr–O stretching vibrations (Thangagiri et al. 2022). The peaks at 1386 and 1269 cm^{-1} (in Fig. 8 zone c) are caused by C–H bending and C–O stretching, indicating the presence of alcohol, carboxylic acid, or phenols (Shahadat et al. 2015). Peak at 585 cm^{-1} , which corresponds to alkyl halides, is present in SNL but not in SNLCr(VI), due to peak

shifting, and additional lower frequency peaks are present in SNLCr(VI). The vibrational of O–Cr–O or Cl–Cr–Cl is seen at lower frequencies (520, 464, and 409 cm^{-1}) (Soptrajanov et al. 1999).

Other peaks are practically the same in both compounds SNL and SNLCr(VI) precise few which are slightly (1–10 cm^{-1}) shifted that are also due to chromium loading (Singha et al. 2011). The FTIR analysis indicates chromium loading on compounds during the adsorption process.

XRD analysis

The chromium adsorption phenomenon by *Sambucus nigra* L. was also studied using XRD. The XRD pattern of the produced *Sambucus nigra* L. (SNL) is shown in Fig. 9, revealing the biomaterial's amorphous nature. However, the few prominent peaks seen in the XRD at 24.34° and 31.15° can be attributed to the carbon diffraction planes in the biomaterial (Chellappan et al. 2018). X'Pert High-Score software was used for the analysis. The results established the presence of polyphenolic compounds in the SNL. Peak matches the reference number 01–076–1367, indicating the presence of potassium compounds with hydrocarbon groups.

The XRD pattern of the utilized SNL for the chromium adsorption is shown in Fig. 10. The halo of

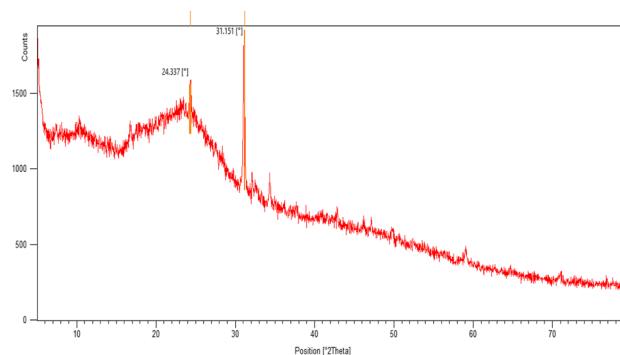


Fig. 9 XRD of SNL (before adsorption)

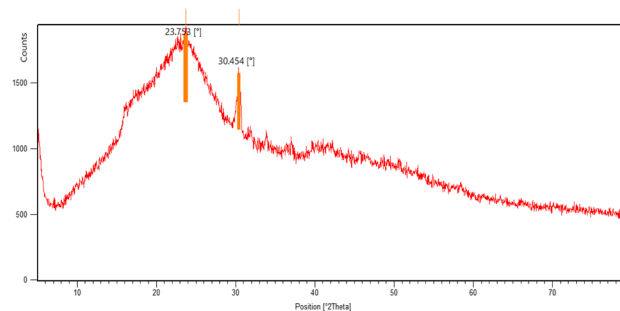


Fig. 10 XRD of Cr-SNL (after adsorption)

diffraction at 23.75° and sharp peak at 30.45° are the indicative of presence of chromium ions on the plant biomaterial (Thangagiri et al. 2022). The results indicated that chromium adsorption on this biomaterial is achievable, which was validated by XRD data. The XRD analysis also supports the presence of ammonium chromium oxide as mentioned in the FTIR analysis section. The peak seen in Cr-SNL corresponds to the reference code 01–075-1578. Two other reference card codes 01–084-1201 and 00–028-1188, coincide with the identified XRD peaks of the Cr-SNL sample, indicating the potential of chromium phosphate and sodium chromium phosphate compounds. Furthermore, the XRD pattern of the used biomaterial is observed the same as those of fresh material (Figs. 9 and 10)

representing that there is no change in the amorphous structure of biomaterial being used (Gu et al. 2022).

SEM and EDS analysis

The SEM image of the utilized *Sambucus nigra* L. in the chromium removal is shown in Fig. 11, which confirmed the amorphous structure of the biomaterial, already revealed in XRD. The adsorbed chromium is accumulated the *Sambucus nigra* L. biomaterial and it is shown in the figure at the spectrum 17 as white spots. The EDS of spectrum 17 confirms the presence of chromium ions on the biomaterial and patches of white pattern on the SEM image are the evidence of the presence of the adsorbed chromium ions. Similar observation was reported earlier by Amaku et al.

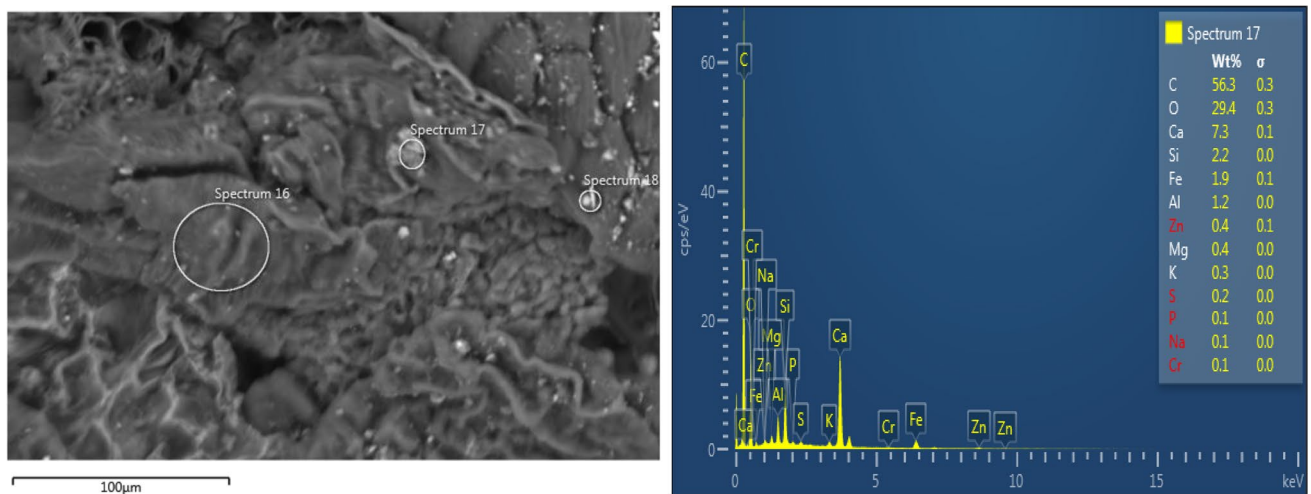


Fig. 11 SEM and EDS spectrum of Cr-SNL (spectrum-17)

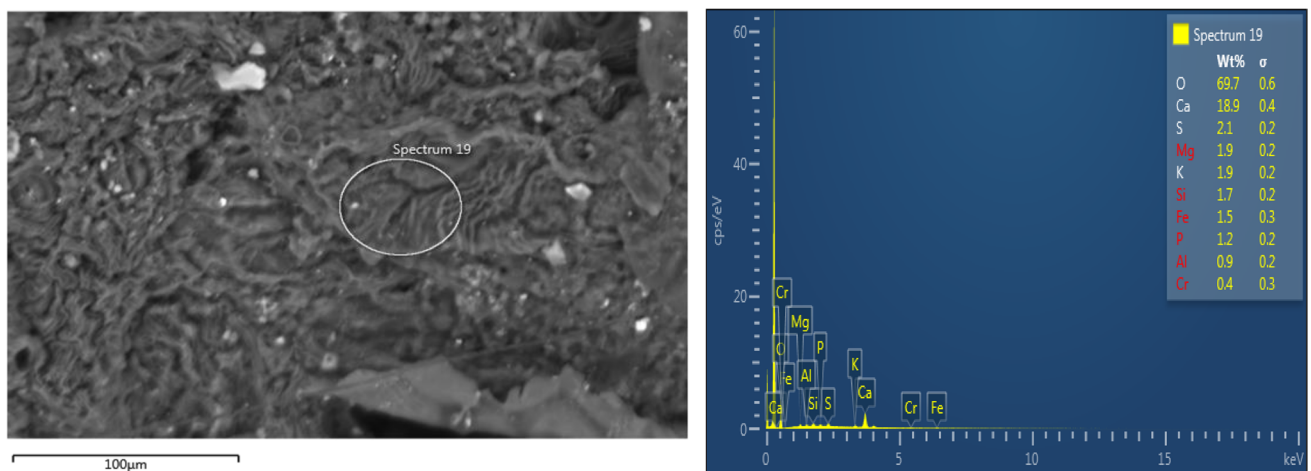


Fig. 12 SEM and EDS spectrum of Cr-SNL (spectrum-19)

(2021) for the chromium removal using extract of *Dacryodes edulis* leaves.

The another SEM image of the utilized SNL is presented in the Fig. 12, which is also confirmed by the presence of adsorbed chromium in the material and it is confirmed in the EDS of spectrum 19. In addition to chromium ions, silicon, iron, and aluminum ions are observed.

ICP-OES analysis

Furthermore, chromium adsorption was validated using the ICP-OES. ICP-OES equipment was used to evaluate the SNL and SNL-Cr samples. The acquired findings clearly demonstrated the chromium adsorption via *Sambucus nigra* L. Before adsorption, ICP-OES measurement revealed a chromium value of $< 0.0003 \mu\text{g/L}$, but after batch adsorption, the result was $1720.37 \mu\text{g/L}$.

Conclusion

This study describes the utilization of agroforestry and industrial residue *Sambucus nigra* L. as a novel physical adsorbent for the removal of Cr (VI), as an environmentally friendly and low-cost solution. The adsorption process experimental results best fitted the non-linear Freundlich equilibrium model. The sorption of Cr (VI) onto the adsorbent was rapid, reaching equilibrium and stabilizing within 35 min. The pseudo-second-order model defining the phenomena with a chemical ion exchange mechanism best depicted the elimination process. The thermodynamic parameters revealed that the occurrence was both spontaneous and favorable. The result of adsorption is strongly supported by XRD, FTIR, SEM–EDS, and ICP-OES analyses. The results of these analyses provide significant evidence for chromium adsorption via *Sambucus nigra* L. As a conclusion, it has been demonstrated that this novel adsorbent can be useful to manage chromium in industrial wastewater.

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Author contribution All authors contributed to the study conception and design. Material preparation and data collection were performed by HBM, MRC, PCA, JPP, and CTT, and analysis part contributed by MKJ and GG. The first draft of the manuscript was written by HBM, and all authors commented on previous versions of the manuscript and modified. All authors read and approved the final manuscript.

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Data availability Not applicable.

Declarations

Ethics approval and consent to participate Not applicable.

Consent for publication Not applicable.

Conflict of interest The authors declare no competing interests.

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