Berhane *et al. BMC Chemistry* (2025) 19:105
https://doi.org/10.1186/s13065-025-01479-1

BMC Chemistry

RESEARCH Open Access

Cabbage waste derived activated carbon for removing hexavalent chromium from synthetic and tannery wastewater

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Abstract

Industrial wastewater discharge without proper treatment causes severe ecological and public health problems. Heavy metals like chromium are pollutants found in untreated or partially treated industrial wastewater. This study investigates the removal of hexavalent chromium ion (Cr (VI)) from synthetic and tannery wastewater using activated carbon derived from cabbage waste. The cabbage waste was activated with H_3PO_4 and carbonized at 450 °C. Characterization was performed using proximate analysis, XRD, and FTIR spectroscopy. Batch adsorption studies were conducted under various conditions, including initial chromium concentrations (10–100 mg/L), adsorbent dose (1–20 g/L), contact time (30–300 min), pH (2–11), shaker speed (50–250 rpm), and temperature (20–70 °C). The optimum conditions for maximum chromium removal (99.87%) from synthetic wastewater were pH 3, initial chromium concentration 10 mg/L, shaking speed 150 rpm, contact time 150 min, adsorbent dose 2 g/L, and temperature 25 °C. For tannery wastewater, 83.81% chromium removal was achieved under the same conditions. The Freundlich isotherm model best described the adsorption process, with an adsorption capacity of 4.9 mg/g. The adsorption followed pseudo second order kinetics. The thermodynamics study indicates that the adsorption of Cr (VI) is spontaneous, exothermic and driven by an increase in entropy. A negative value of Gibbs Free Energy change ($\Delta G°$) indicates that the adsorption process is thermodynamically spontaneous and feasible.

Keywords Activated carbon, Adsorption, Cabbage waste, Hexavalent chromium, Tannery wastewater

Introduction

Wastewater treatment plays a crucial role in achieving Sustainable Development Goal 6:"Clean water and sanitation for all."However, data shows that 80–95% of all wastewater produced in the world is released into the environment without treatment [25]. Heavy metals in untreated wastewater are especially concerning because they remain in the environment for a long time and are highly toxic [45]. According to the World Health Organization, exposure to toxic substances, including heavy metals, causes about 1.7 million deaths every year, showing significant risks to human health [43].

Chromium is a polyvalent element that commonly exists in various oxidation states, with chromium (III) and chromium (VI) being the most commonly found in nature [49]. However, Cr (VI) is of particular concern because of its high toxicity to many plants, animals, and aquatic environments [40] and has carcinogenic potential to humans [44]. The WHO and US EPA limit and recommend Cr (VI) release into drinking and inland

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surface water to be 0.05 mg/L and 0.1 mg/L, respectively due to its toxicity potential [8, 33].

However, Cr (VI) containing pollutants are released into the water bodies higher than the permitted for surface water discharge [29]. The tannery industries in Ethiopia are one of the main sources of chromium pollution. According to a study conducted by Zewdu & Amare [46] in Bahir Dar tannery, total Cr, Cr(III), and Cr(VI) were found to be 2.957 mg/L, 2.88 mg/L, and 0.08 mg/L, respectively after the treatment. In another study in Kombolcha, South Wollo, Ethiopia, the concentrations of total Cr, Cr (III), and Cr (VI) ranged from 1.240 to 823.87 mg/L, 1.207 to 821.86 mg/L, and 0.033 to 2.014 mg/L, respectively, in the effluent of the tannery, exceeding the WHO permissible limit [4]. So, for countries like Ethiopia removing Cr (VI) from wastewater remains as significant challenge.

The most common conventional methods of Cr (VI) removal from aqueous media are membrane technology, ion exchange, chemical precipitation, solvent extraction, filtering, electrochemical treatment, and adsorption [16, 22]. In comparison to other wastewater treatment techniques, the adsorption approach is a very effective process that has been utilized extensively in recent years. In addition to its removal efficiency, adsorption is inexpensive, simple to use, and relatively environmentally friendly [14]. Specifically, bio-adsorbents made of cellulose exhibit excellent adsorption properties in aqueous solutions due to their high surface area, ion exchange capacity and high affinity towards Cr (VI) [3, 37].

Plant leaves, vegetables, and fruit peel are highly resourceful chemical species with high adsorption capacity, containing cellulose, hemicellulose, lignin, chlorophyll, carotene, anthocyanin, and tannin contributing to metal biosorption [9]. Similarly, other agricultural waste biomasses such as *Helianthus annuus* (Sunflower) stem waste [17, 18] and banana peel [5] are efficiently used to adsorb hexavalent chromium from aqueous system and industrial wastewater. The surface functional groups of bio-adsorbents are the primary component that contributes to their adsorption property because the chemical adsorption reaction was induced by the presence of oxygen, carbon, and mineral fractions [27].

Cabbage (*Brassica oleracea*) is a regularly utilized vegetable. However, waste cabbage is the byproduct of sorting the outer shells of the cabbage; according to Zhang et al. [48], 30% of the cabbage is discarded as waste. This byproduct is abundantly available, low-cost, and has good adsorption for different heavy metals due to its high cellulose and lignin content. Carboxyl, hydroxyl, and amino groups present in cabbage waste were found to be the most important functional groups for metal adsorption [15].

Cabbage waste, a byproduct of food processing, contains approximately 37% cellulose on a dry-weight basis [23] which is important for biosorption because cellulose and hemicellulose contain many active hydroxyls (OH) that can bind metal ions and possess a large surface area and porosity [21]. For this reason numerous researchers use cabbage waste as an adsorbent for the removal of some heavy metal ions including Pb (II), Cd (II), Cu (II), and Zn (II) [19]. However, based on our best searching strategies, no prior reports were found on Cr (VI) biosorption using cabbage waste derived activated carbon.

A phosphoric acid solution of 45% was used to activate the carbon derived from cabbage waste [42]. Phosphoric acid plays a great role in increasing the porosity and surface area of the carbon structure, which is very important in improving removal efficiency of Cr (VI) ions. Thus, this modification can facilitate the adsorption mechanisms like ion exchange and electrostatic attraction, enhancing adsorption capacity for Cr (VI) removal.

Therefore, the aim of this study is to evaluate the eco-efficiency of activated carbon derived from cabbage waste for the removal of hexavalent chromium (Cr (VI)) from both synthetic and tannery wastewater. The objectives include characterizing the physicochemical properties of the activated carbon, assessing its removal efficiency for Cr (VI) ions, optimizing key adsorption parameters, and determining the adsorption isotherm and kinetic models associated with chromium removal. In this study, the novelty lies in the use of waste cabbage, an abundantly available and low-cost byproduct, as an effective adsorbent for Cr (VI) removal, with no prior reports on its biosorption capabilities. Additionally, the use of phosphoric acid activation to enhance the adsorbent's porosity and surface area is innovative. The scope of the study encompasses utilizing cabbage waste-derived activated carbon for Cr (VI) adsorption, characterizing its properties, assessing removal efficiency, optimizing adsorption conditions, and evaluating adsorption mechanisms through isotherm and kinetic models.

Materials and methods

Wastewater sample collection and preservation

Real wastewater sample was taken from untreated tannery wastewater at Batu Tannery PLC in Addis Ababa, Ethiopia. The sample was taken into an acid-washed plastic bottle to prevent the complexation of the organic waste in the wastewater with the Cr (VI) ions and immediately treated with a solution of sulfuric acid at pH 2. The sample was stored at a temperature

4 °C to stabilize Cr (VI) ions and transported to Jimma University Environmental Health Science and Technology Laboratory for the adsorption study.

Preparation of stock solution

Stock solutions of Cr (VI) with a concentration of 1000 mg/L were prepared by dissolving 2.829 g of potassium dichromate ($K_2Cr_2O_7$, 99.5%, FINKEM, England) in 1000 mL of distilled water. Then standard solution containing 100 mg/L of Cr (VI) was prepared in a 500 mL volumetric flask by dissolving 50 mL of stock solution with distilled water and filled up to the mark. The working solution was prepared by diluting 50 mL from 100 mg/L standard solution with distilled water in a 500 mL volumetric flask to give the appropriate working solution containing 10 mg/L of Cr (VI). A series of five working standard solutions containing 0.01, 0.1, 0.5, 1, and 2 mg/L of Cr (VI), were prepared from the stock solution.

Preparation of activated carbon (adsorbent) from cabbage waste

Cabbage waste was collected from fruit and vegetable markets in Jimma town. The collected cabbage waste was washed several times with distilled water to remove dust and fines until the color of the washed cabbage waste became colorless. It was then chopped into small pieces (< 5 mm) to facilitate drying at 105 °C in an electric oven for 24 h to eliminate moisture and other volatile impurities [36]. The dried cabbage waste was impregnated by thoroughly mixing it with an activating agent of 45% (w/w) Phosphoric acid (H_3PO_4) (Riedel-de Haën, Germany) in a 2:1 ratio relative to the cabbage waste and allowing it to soak overnight.

The impregnated cabbage waste was carbonized by heating it for 90 min at 450 °C in an adjustable electric furnace. After carbonization, the product was cooled to room temperature in a desiccator and sealed with aluminum foil. Afterward, the activated carbon was crushed and sieved using 0.425 mm stainless steel mesh to get a fine and uniform powder. The entire preparation process is illustrated in Fig. 1. The excess activating agent, H_3PO_4, was removed from the carbonized products by washing it with distilled water. Afterward, the powder was dried at 105 °C for 24 h. Then the fine powder was stored in tightly sealed plastic bottles, labeled and preserved for subsequent use.

Fig. 1 Preparation of activated carbon derived from cabbage waste

Characterization of the adsorbent

Physicochemical characterization of the adsorbent was performed using proximate analysis to determine its moisture content, ash content, fixed carbon content and volatile matter. The Sear approach was used to determine the adsorbent's specific surface area [1]. Using a 250-mL conical flask, approximately 1.5 g of the produced adsorbent was mixed with 30 g of NaCl (99.9%) and dissolved in 100 mL of distilled water. The mixtures were agitated for five min. After that, the pH of the solution was adjusted to 4, and it was titrated with 0.1 M NaOH until the pH reached 9. The volume of NaOH needed to raise the pH from 4 to 9 was recorded. The specific surface area of the sample was calculated using the Eq. (1) [1]:

$$Specific\ surface\ area\ \left({}^{m^2}\!/_{g}\right) = 32V - 25 \quad (1)$$

where: V is the volume (mL) of NaOH required to raise the pH of the sample. The numbers 32 and 25 are physical constants.

The points of zero charge (pzc) of the adsorbent were determined using the solid addition method [44]. A 50 mL solution of 0.01 M NaCl was prepared in conical flasks, and the pH was adjusted from 2 to 11 using 0.1 M HCl (37%, Riedel-de Haën, Germany) or NaOH (90%, BDH, England) [39]. Then, 2 g/L of adsorbent was added to each flask, and the mixtures were shaken at 150 rpm for 24 h. The equilibrium pH values were measured, and the pHpzc was calculated from the variations in observed pH as a function of the initial pH [11]. The intersection point of the ΔpH against initial pH curves indicated the adsorbent's pzc [9].

To identify the functional groups present, the adsorbent was characterized by FTIR (Perkin Elmer Spectrum two, USA) both before and after the adsorption process, using an average wavelength range of 400 to 4000 cm^{-1} to observe any changes in the functional groups [39]. The prepared activated carbon sample powder was mixed with KBr particles to make it suitable for infrared analysis. The mixture was then pressed to a small thickness to create a pellet required for the FTIR study.

To study the amorphous and crystal structure of the substance, the activated carbon powder was analyzed using an XRD (Drawell XRD-7000, China). The powder was packed into the holder, then pressed down and flattened in order to present a smooth diffraction surface. Then, the diffractometer was run over a 2θ range of 10° to 80°. Both FTIR and XRD analysis were conducted at Jimma University, Institute of Technology (JIT) Faculty of Material Science and Engineering.

Batch adsorption studies

All batch adsorption studies were conducted in 50 mL of chromium water solutions at the the range of pH 2–11, contact time 30–300 min, initial concentration of Cr (VI) 10–100 mg/L, shaking speed 50–250 rpm, temperature 20–70 °C, and dose of adsorbent 1–20 g/L to know the optimum conditions. The optimum value of each parameter was determined by the highest adsorption efficiency and capacity recorded in the range of testing variables [31]. A 50-mL synthetic wastewater was added to the plastic bottle, which contains adsorbent. After shaking, the sample separation of the sorbent and solutions was carried out using Whatman filter paper of pore size 0.45 µm. Then, the adsorption process was monitored using a UV–visible spectrophotometer (HACH DR 5000, USA) by analyzing the remaining chromium in the solution. Equation (2) was utilized to calculate adsorption efficiency (%), while Eq. (3) was used to determine the adsorption capacity of the activated carbon [47].

$$R\ (\%) = \frac{C_o - C_t}{C_o} \times 100 \tag{2}$$

$$\mathrm{q} = \frac{(\mathrm{C_o} - \mathrm{C_e})\mathrm{V}}{\mathrm{W}} \tag{3}$$

where: R = Adsorption efficiency, C_o = initial Cr (VI) concentration (mg/L) and C_t = final Cr (VI) concentration (mg/L) after a certain period of time (t); q = Adsorption capacity (mg/g), C_e is the equilibrium concentration (mg/L), V is the volume of the solution (L), and W is the weight of the adsorbent (g).The equilibrium of this experiment was studied using Langmuir and Freundlich isotherm models. The kinetics of adsorption of Cr (VI) was also investigated by testing for pseudo first order and pseudo second order models. The effects of operation parameters on adsorption efficiency were studied by varying the conditions.

Desorption study

The desorption studies were carried out to restore the used adsorbent, an essential step for making the treatment process economically viable [9]. If not disposed of properly, the exhausted material can cause environmental damage. In the case of desorption studies, 2 g/L of adsorbent was added to 100 mL Erlenmeyer flasks containing 10 mg/L of Cr (VI) ions. After being left to reach equilibrium for 150 min at 25 °C, the adsorbent was retrieved, and the adsorption capacity was determined. It was assumed that desorption equilibrium coincided with adsorption equilibrium [9]. The residues on the filter paper were used for the desorption experiments. The residual metal ions on the used adsorbent surface were removed by washing with desorption agents and then dried in an oven at 105 °C until a constant mass was achieved. The metal ions were desorbed from the adsorbent using five different desorption agents: 1 M solutions of NaOH, KOH (90%, BDH, England), H_2SO_4 (98%, UNI CHEM, Germany), HNO_3 (69%, LOBA, Chem, India), and HCl [30]. Each desorption agent (50 mL) was placed in 100 mL Erlenmeyer flasks with the recovered adsorbent and agitated for 150 min at 150 rpm and a temperature of 25 °C. After equilibration, the adsorbate-adsorbent mixtures were filtered using Whatman filter paper of pore size 0.45 µm, and the concentration of the Cr (VI) ion in the filtrate was determined using a UV–vis spectrophotometer. The Eq. (4) was used to calculate the efficiency of the desorption [30].

$$\text{Desorption}\ (\%) = \frac{Amount\ of\ Cr\ (VI)\ desorbed}{Amount\ of\ Cr\ (VI)\ adsorbed} \times 100 \tag{4}$$

Results and discussions

Characteristics of the adsorbent

Proximate analysis

The study result showed that the activated carbon derived from cabbage waste possessed a high fixed carbon content (60.414%), low moisture content (6.191%), low ash content (17.477%), and low volatile matter (15.918%). The carbonization and activation process involves heating the organic materials, leading to the breakdown of chemical bonds and releasing vapor through gases and liquids. As a result, a material with high carbon content was created. The high fixed carbon content in the composition leads to higher surface area (87 m^2/g) of the adsorbent and enhancing its adsorption capabilities [3].

The present study is supported by the studies of Tibebu et al. [39], who stated that activated carbons with a high fixed carbon content provided excellent adsorption capacity because of the increased surface area and limited inorganic material. This finding is further supported by Dagnew and Bantie [9], who specified that low moisture and ash in the adsorbent resulted in higher chromium adsorption capacity.

Point of zero charge

The point of zero charge (pzc) is the pH at which the net surface charge of adsorbents is zero [1]. If the pH of the solution is below the point of zero charge (pH < pHpzc), the adsorbent surface attracts anions (negatively charged ions) because of its positive charge. The highly protonated adsorbent surface electrostatically attracts oxyanions ($HCrO_4^-$ and $Cr_2O_7^{2-}$). If the pH exceeds the pzc, the adsorbent surface is negatively charged, attracting cations (positively charged ions) [1]. Therefore, anion adsorption on any adsorbent is favorable at a pH lower than pzc. In this study, the pzc (ΔpH vs. initial pH) of activated carbon (adsorbent) from cabbage waste was obtained at pH 4 (Fig. 2).

FTIR analysis

The FTIR spectra of activated carbon derived from cabbage waste before and after adsorption of Cr (VI) were illustrated in (Fig. 3). A broad peak around the region 3500–3200 cm^{-1} is assigned as O–H (hydroxyl) and N–H (amine) functional groups stretching vibration [34]. A peak around 2923 cm^{-1} was attributed to aliphatic hydrocarbon C-H stretching; the peak at 1633 cm^{-1} was assigned to the C= O group of the carboxyl group; the C-N stretch for aliphatic amines was at 1035 cm^{-1}, and the bands around 848 cm^{-1} regions were C-H out-of-plane (OOP) bending vibrations of aromatic compounds [32, 34]. The very weak absorption observed at 500 cm^{-1} was attributed to the long chain band of aliphatic alkane.

After the adsorption of Cr (VI) on the adsorbent, there was a small shift in frequency values, and some frequency regions were absent. This indicated the involvement of Cr (VI) with activated carbon derived from cabbage waste. Similar findings have been reported by Kebede et al. [22] on "Tella" residue and Pea (*Pisum sativum*) seed shell carbon which showed that after adsorption the spectral peaks shifted, indicating Cr(VI) ions were effectively bound to the adsorbent. Further evidence shows that Cr (VI) interacts with functional groups on carbon-based materials. Research involving Manjistha (*Rubia cordifolia*) as an adsorbent also reported considerable changes in FTIR spectra post-adsorption of Cr (VI) [44].

XRD analysis

The XRD spectra of the adsorbent, before and after adsorption were illustrated in Fig. 4. XRD analysis does not show any observable change in the patterns of activated carbon derived from cabbage waste before and after loading with Cr (VI) ions. This stipulates that Cr (VI) adsorption on the materials does not change its structural nature. Besides, an amorphous phase within the bio-adsorbent was contributed to a broad, irregular baseline in its XRD spectrum. This is because amorphous materials do not have the long-range order necessary to give rise to sharp diffraction peaks but instead yield a diffused or noisy signal. The minor changes observed in the diffraction patterns of the adsorbents may suggest the

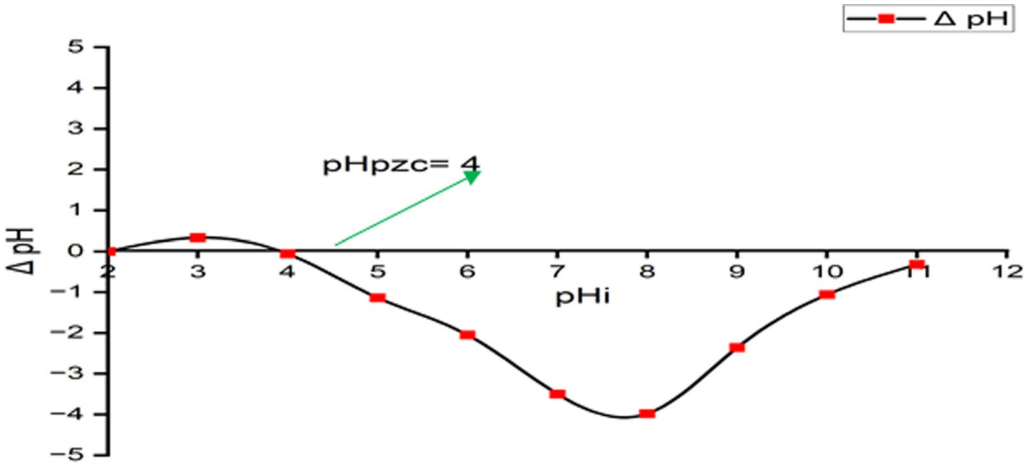

Fig. 2 Point of zero charge of adsorbent derived from cabbage waste

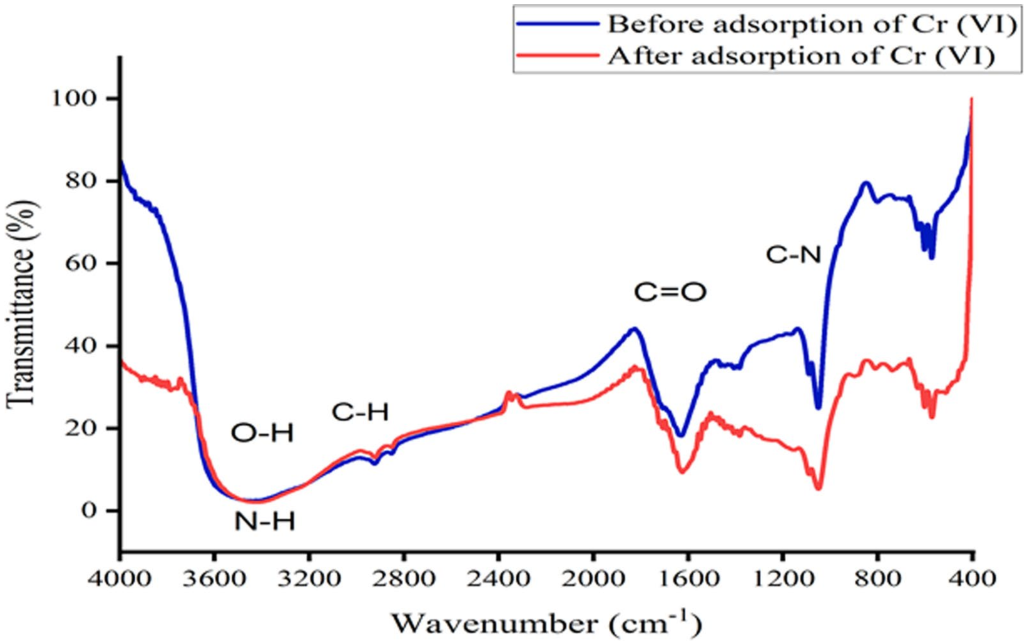

Fig. 3 FTIR spectra of activated carbon (adsorbent) before and after adsorption of Cr (VI)

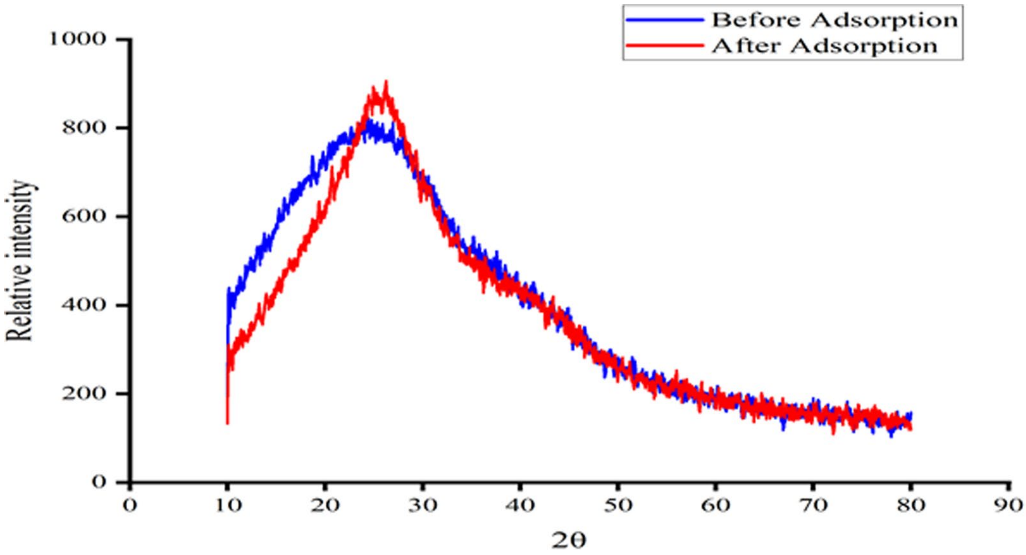

Fig. 4 XRD spectra of activated carbon before and after adsorption of Cr (VI)

adsorption of Cr (VI). Therefore, the majority distorted form indicates the adsorbents lack crystalline structure and are predominantly amorphous [40].

Batch adsorption

Effect of pH

The pH of the solution is an important factor since it influences the adsorption of metal ions by influencing the surface properties of the adsorbents and ionic species of the chromium solutions [20]. The pH of the chromium solution was adjusted before and after adding adsorbents, and its optimization was carried out by studying the extent of removal of Cr (VI) by varied pH values from 2 to 11 while keeping the other parameters constant. As shown in Fig. 5, the maximum adsorption percentage and

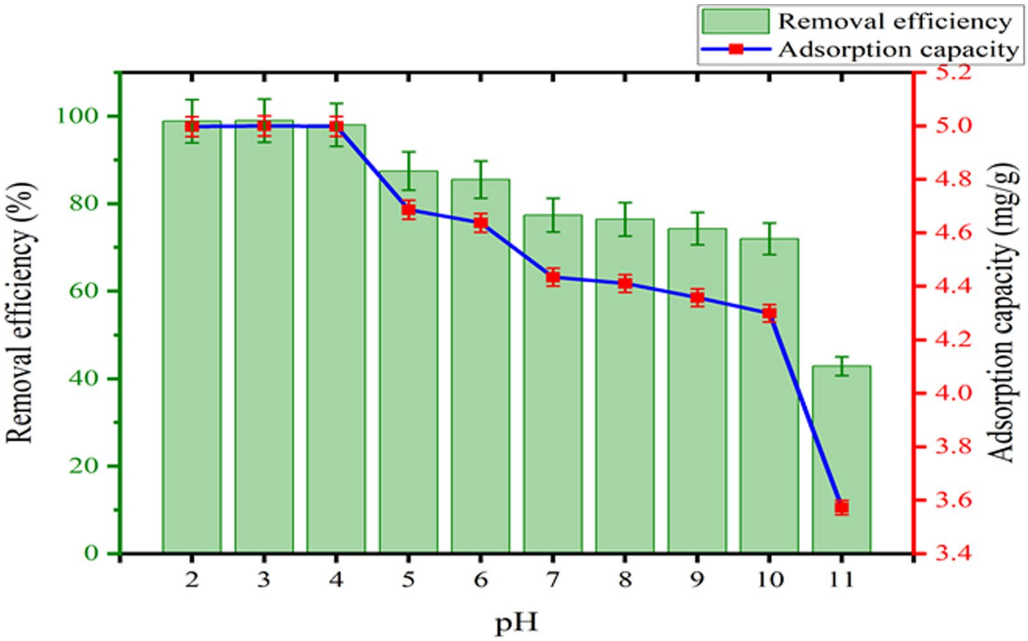

Fig. 5 Effects of pH on the removal of Cr (VI) by cabbage waste-derived activated carbon

adsorption capacity of chromium at pH = 3 were 98.99% and 4.9 mg/g, respectively.

This result indicates that the adsorption capacity and removal efficiency of Cr (VI) increases with a decrease in pH values. At low pH, the surface charge of the adsorbent may get positively charged as a result of being surrounded by H^+ ions neutralizing negatively charged hydroxyl groups (O–H) on the adsorbent surface, thereby reducing the hindrance to the diffusion of dichromate ions [40]. Different forms of Cr (VI) can be found across a pH spectrum. At pH 1, it exists as chromic acid (H_2CrO_4), but its concentration significantly decreases as pH increases.

Specific ions of chromium, such as $Cr_2O_7^{2-}$, $HCrO_4^-$, and $Cr_3O_{10}^{2-}$, are found in the pH range of 2–6 [26]. Among these ions, $HCrO_4^-$ is the dominant one in the pH range of 2–3. The smaller ionic size of $HCrO_4^-$ (44 cm^3 mol^{-1}) compared to $Cr_2O_7^{2-}$ (73 cm^3 mol^{-1}) enhances its ability to be adsorbed onto the active sites of adsorbents [13]. According to Fito et al. [11] study at a pH greater than point zero charge (pH > pHpzc), there was a decline in the percentage removal of Cr(VI) with an increase in pH due to the negatively charged adsorbent surface creating a repulsive effect with the chromium oxy-anions.

On the other hand, the reduction in the removal efficiency of Cr (VI) as the pH increases from 4 to 11 may be attributed to the competition between hydroxide (OH^-) ions and chromate (CrO_4^{2-}) ions. The hydroxide ions, being more dominant, reach the adsorbent surface more quickly than the chromium species [28]. In this study, therefore, the optimum pH value was 3 which provided maximum removal efficiency (98.99%) of Cr (VI).

Effect of initial concentration of adsorbate

The effect of initial concentration and isotherm model was studied by varying initial concentration in the range of 10–100 mg/L. The other variables were kept constant (pH 3, adsorbent dose 2 g/L, contact time 150 min, shaking speed 150 rpm and temperature 25 °C). Figure 6 describes how adsorption efficiency affected by initial concentration of Cr (VI). The result showed that the adsorption efficiency of the adsorbent decreases with increasing initial concentration of Cr (VI). At optimum condition (initial concentration of 10 mg/L), the adsorbent had removed 99.86% of Cr (VI) from the solution. At the initial concentration of Cr (VI) higher than 10 mg/L, the removal efficiency had significantly reduced from 99.86% to 31.61%. One potential reason for the higher removal efficiency at lower Cr (VI) concentrations might be the enhanced ion mobility in the more diluted solutions. Meanwhile, the decrease in adsorption efficiency at higher initial Cr (VI) concentrations may be due to the limited availability of active binding sites on the adsorbent, given a constant adsorbent mass.

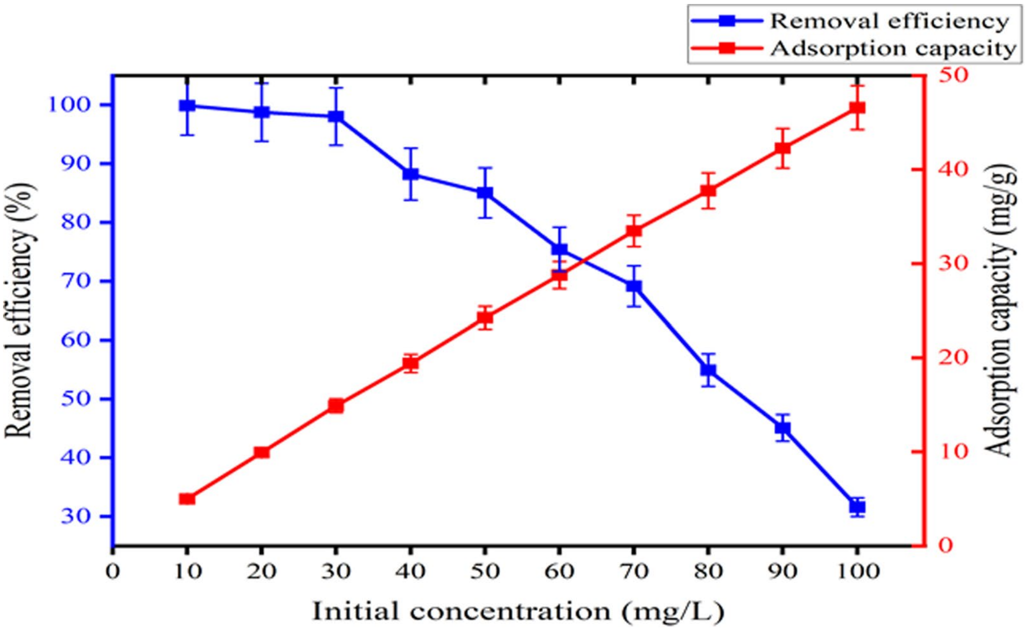

Fig. 6 Effect of initial concentration of Cr (VI) on adsorbent's removal efficiency

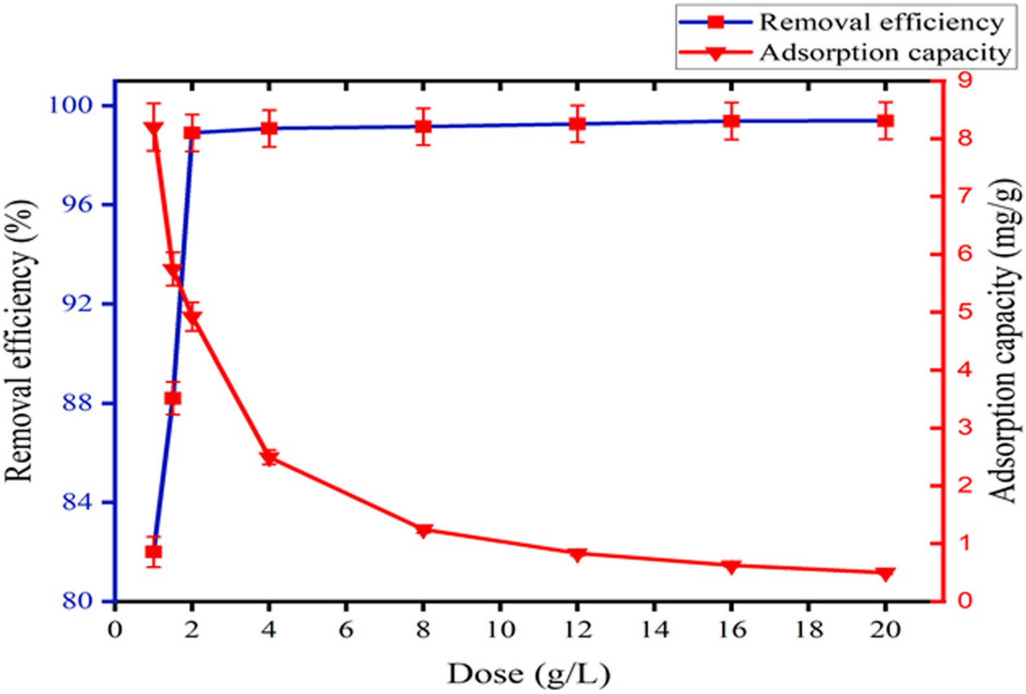

Fig. 7 Effect of adsorbent dosage on the removal of Cr (VI) by activated carbon

On the other hand, the adsorption capacity of the adsorbent was increased with increasing initial concentration of Cr (VI). In the initial concentration ranges between 10 and 100 mg/L, the adsorption capacity changed from 4.99 to 46.58 mg/g. This is likely due to the greater probability of collisions between the metal ions and the activated carbon [7, 34]. Generally, ion adsorption tends to occur more readily at higher initial concentrations due to the increased driving force required for the mass transfer of Cr (VI).

Effect of adsorbent dosage

The adsorbent doses of 1, 1.5, 2, 4, 8, 12, 16, and 20 g/L were tested to determine the effect of adsorbent dosage [12]. Figure 7 shows that at various dosages of activated carbon, there are significant variations in Cr (VI) removal efficiency and the capacities of adsorption. As the dosage of activated carbon was raised from 1 to 2 g/L, the Cr (VI) removal efficiency showed a significant increase and remained slightly constant above 2 g. There are more binding sites available for Cr (VI) adsorption at higher adsorbent doses, which causes the removal efficiency to rise until the dosage reaches saturation point. Therefore, 2 g/L has been chosen for the subsequent studies as optimum dose.

Conversely, the adsorbent dose influenced the adsorption capacity negatively (Fig. 7). The value of q decreased rapidly from 8.2 mg/g to 0.499 mg/g when the adsorbent dose changed from 1 to 20 g/L. When the amount of adsorbent is low, the active adsorption sites can quickly become saturated with adsorbates, leading to a decrease in the slope of the adsorption capacity curve as it approaches maximum capacity [9]. As the amount of adsorbent increases beyond a certain point, more adsorption sites become unable to interact with some adsorbate particles [7]. Additionally, as the quantity of adsorbents increases, their specific surface area decreases due to the ease of aggregation.

Effect of shaking speed

Shaking speed is a parameter that indicates the distribution of the adsorbent in the whole solution and the formation of the external boundary layer and preventing settling [24]. The dispersion of adsorbent facilitates the proper exposure of the adsorbent sites to the adsorbate ions. The effect of shaking speed on the removal efficiency of Cr (VI) was studied by varying the

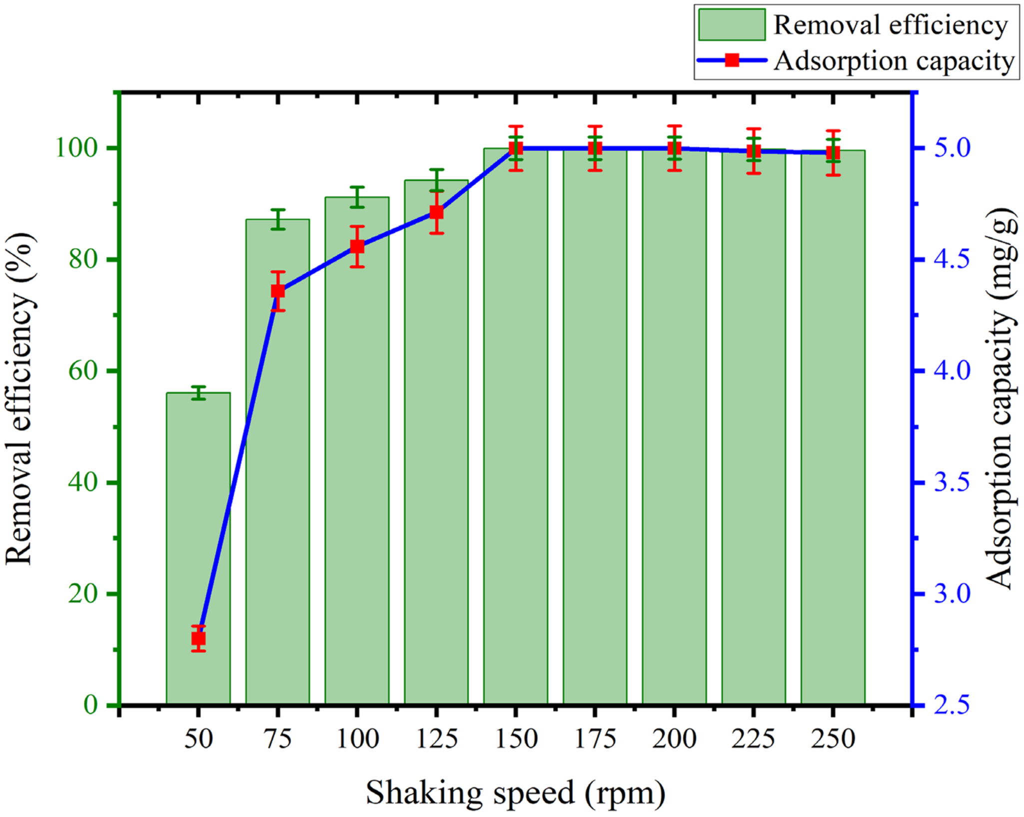

Fig. 8 Effect of shaking speed on the removal of Cr (VI) by activated carbon

speed of agitation from 50 to 250 rpm while keeping the other variable constant (pH 3, initial Concentration 10 mg/L, contact time 150 min, adsorbent dose 2 g/L and temperature 25 °C). Figure 8 describes the effect of shaking speed on adsorption capacity of the activated carbon.

The results indicated that the adsorption capacity increased from 2.8 mg/g to 4.99 mg/g as the shaking speed was raised from 50 to 150 rpm. Additionally, the adsorption efficiency improved from 56% to 99.87% in the same range of shaking speed. Increasing the shaking speed enhances metal ion diffusion to the adsorbent surface and spreads the sorbent surface, making active sites available and increasing the adsorption rate [28]. But, it remained relatively constant after shaking speeds of 150 rpm and followed by a slight decrease at speeds exceeding 200 rpm.

The reduction in adsorption at highest shaking speed is due to increased kinetic energy at high shaking speed, which can lead to the desorption of already adsorbed metal ions due to vigorous collisions between adsorbate and adsorbent particles [20]. The results indicated that increasing shaking speed enhances adsorption up to an optimum level, while excessive agitation disrupts the process. Thus, a shaking speed of 150 rpm is optimal for Cr (VI) removal, ensuring effective interaction between the adsorbate and adsorbent.

Effect of contact time

Contact time is one of factors affecting adsorption capacity and efficiency. Studies shown that as contact time increases, so does adsorption capacity [11]. The effect of contact time was studied by varying in the range of 30–300 min, with an interval of 30 min while keeping other variables constant. Figure 9 illustrates that the initial stages of Cr (VI) adsorption on the sample was found to be rapid. After 90 min, it started to gradually increase, and after 150 min, it started to stay relatively constant. This result shows that adsorption starts out quickly because there is a large enough adsorption sites available, but it gradually shows down until equilibrium is reached. Between 30 to 150 min, the adsorption capacity changed from 4.1 mg/g to 4.96 mg/g and remains constant beyond 150 min. This indicates that adsorption initially occurs rapidly at first due to the availability of active binding site, but slows down over time as the active sites of the adsorbent become filled by the Cr (VI) ions [11].

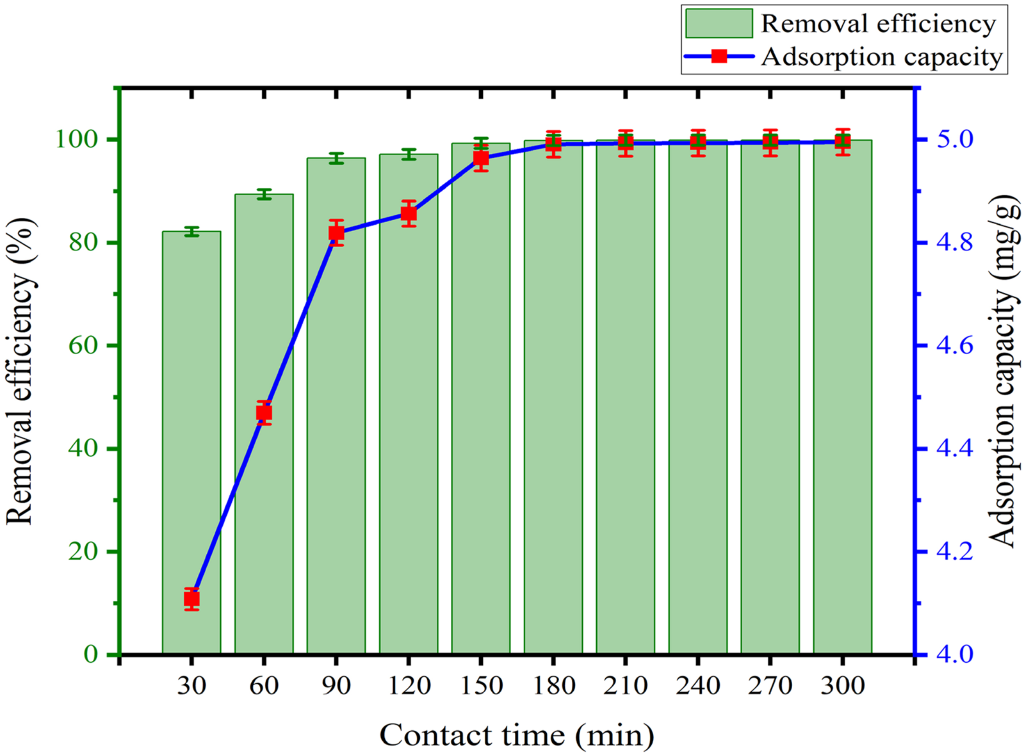

Fig. 9 Effect of contact time on the removal of Cr (VI) by activated carbon

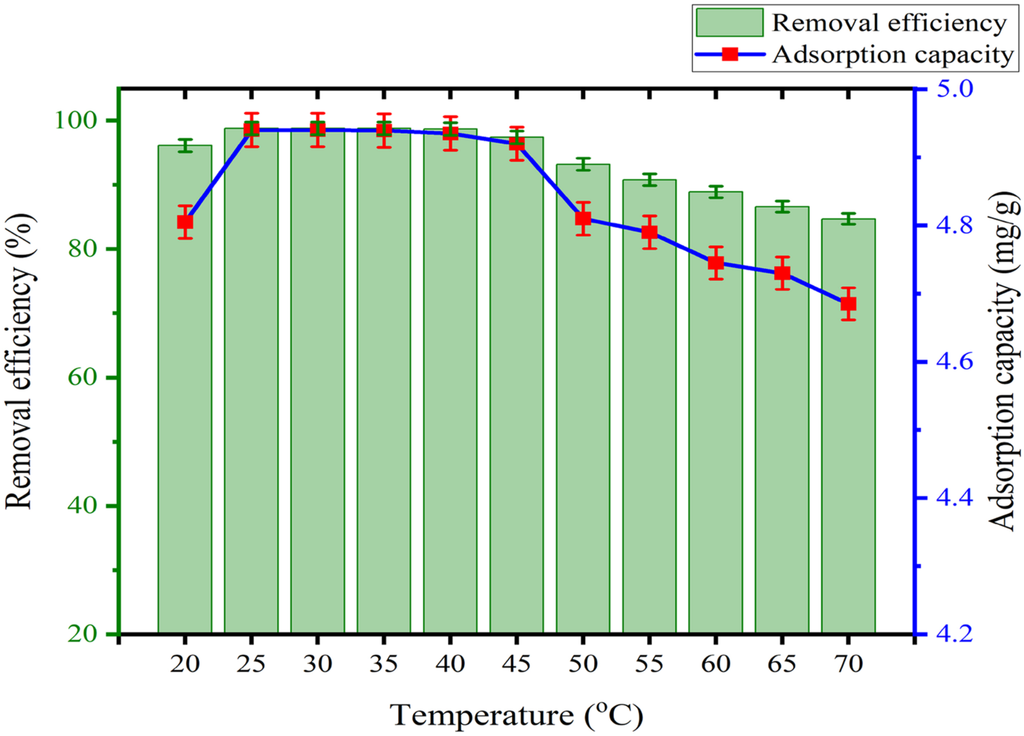

Fig. 10 Effect of temperature on the removal of Cr (VI) by activated carbon

Effect of temperature

Temperature is an important parameter in determining the metal adsorption mechanism because it changes molecular interaction and solubility [13]. The effect of temperature on the removal efficiency of Cr (VI) was studied by varying the temperature from 20 °C to 70 while keeping the other variable constant at their optimum conditions. Temperature could have a positive or negative impact on biosorption within a given interval [20]. As shown in Fig. 10, the adsorption of Cr (VI) ions on adsorbent increased rapidly with an initial increase in temperature and later declined with a further increase in temperature. The adsorption capacity was increased 4.8 mg/g to 4.94 mg/g, when the temperature was increased from 20 °C to 25 °C and remained constant in the temperature range 25 °C to 40 °C. Similarly, adsorption efficiency was changed from 96.1% to 98.8% in the range of temperature 20 °C to 25 °C.

The enhancement in the adsorption efficiencies for some range of temperature may be due to the chemical interaction between adsorbate and adsorbents, the creation of some new adsorption sites, or the increased rate of intraparticle diffusion of Cr (VI) ions into the pores of the adsorbents [13]. Further increases in temperature beyond 40 °C resulted in a decline in removal efficiency and adsorption capacity. The reduction in adsorption percentage after a temperature of 40 °C may be due to desorption caused by increased available thermal energy that may damage the active binding sites of adsorbents [28]. Increasing temperatures induce higher mobility of the adsorbate, causing desorption. Therefore, at the optimum temperature (25 °C), the removal efficiency and adsorption capacity were found to be 98.8% and 4.94 mg/g, respectively.

From this study process, the combination of ion exchange and chemical adsorption mechanism were expected. There is an ion exchange mechanism as the –OH and $-PO_4H_2$ functional groups allow for the exchange of ions between the carbon surface and Cr (VI) anions. This process involves the interaction between the carbon surface charges and the negatively charged Cr (VI) species (like $HCrO_4^-$). On the other hand, the partial reduction of Cr (VI) to Cr (III) on the carbon surface before undergoing complexation with oxygen-containing functional groups indicates a chemical reaction taking place. So, there is a chemisorption which typically involves strong chemical bonds (covalent or ionic) between the adsorbate (Cr species) and the adsorbent (carbon surface). Hence, the process involves a combination of ion exchange and chemical adsorption, with chemical adsorption being more dominant due to the reduction and complexation reactions.

Removal of Cr (VI) from real Tannery wastewater

This study is undertaken to investigate the adsorption capability of activated carbon produced from cabbage waste as an effective adsorbent for treating real tannery wastewater collected from the Batu tannery, Addis Ababa, Ethiopia. All the experiments with actual effluent were conducted under optimized parameters based on studies carried out with synthetic wastewater (stock solution tests), namely pH 3, adsorbent dose 2 g/L, contact time 150 min, shaking speed 150 rpm, and temperature 25 °C. Initially, the concentration of Cr (VI) in real wastewater was 0.32 mg/L and decreased to 0.05 mg/L after treatment with a very impressive removal efficiency of 83.81%. This findings is higher than the study result conduct by Dagnew and Bantie [9] who had reported a removal efficiency of 72.41% for Cr (VI) from real tannery wastewater using activated carbon derived from orange peel. But, relatively lower than similarly study by Temesgen [38] who demonstrated activated carbon produced from cactus and achieved a removal efficiency of 88.7% for Cr (VI) in tannery wastewater under optimized conditions. The comparison shows that the amount (mass) of adsorbent used in this study is less than that of the earlier studies reported in Table 1 except the study by [31] who had used 1.25 g/L of activated carbon derived from banana peduncle.

On the other hand, the removal efficiency of non-activated carbon was 73.43% for the Cr (VI) stock solution and 28.13% for the tannery effluent. These values are significantly lower compared to the removal efficiency achieved using H_3PO_4-activated carbon. This demonstrates that activation with H_3PO_4 enhances pore formation and introduces functional groups that are crucial for the adsorption of Cr (VI).

Adsorption isotherm and kinetics model

The adsorption equilibrium was investigated by fitting the experimental data to the linear equations of the Langmuir (Eq. 5) and Freundlich (Eq. 6) isotherm models [24].

$$\text{Langmuir isotherm model}: \frac{C_e}{q_e} = \frac{1}{K_L q_o} + \frac{C_e}{q_o} \tag{5}$$

Freundlich isotherm model (6)

where, K_L is the Langmuir adsorption constant, q_m (mg/g) is the maximum adsorption capacity for monolayer formation on the adsorbent, C_e (mg/L) is the adsorbate concentration in the solution at equilibrium, and q_e (mg/g) is the amount of adsorbate adsorbed. K_F is the number of ions adsorbed onto the beads and defined as the adsorption or distribution coefficient. The value of 1/n, when it gets closer to zero and becomes more heterogeneous, is used to represent surface heterogeneity. A key feature of the Langmuir isotherm is its ability to predict the affinity between the sorbent and the adsorbate using a dimensionless constant known as the separation factor (R_L), represented by (Eq. 7).

Table 1 Cabbage waste-derived activated carbon's Cr (VI) removal efficiency compared to other adsorbents

S.No	Materials used to derive adsorbent (activated carbon	pH	Dosage (g/L)	Time (min)	Removal Efficiency (%)	References
1	Cactus	3	2.5	60	99.5	[38]
2	Eichhornia crassipes	5	10	90	98.4	[11]
3	Orange peel	2	2.5	90	94.74	[9]
4	Tella residue	2	2.5	240	98.43	[22]
5	Pea seed Shell	3	2.5	60	99.88	[22]
6	Potato peel	2	6	280	99.52	[40]
7	Manjistha	2	2.5	180	95	[44]
8	Rice Husk	2	6	280	99.88	[40]
9	Banana peduncle	4	1.25	90	73.78	[31]
10	Chat stems (*Catha edulis*)	4	30	180	97.03	[6]
11	Sugarcane bagasse	2	7.5	20	98	[35]
12	Groundnut shell	8	2	120	87.6	[7]
13	Teff husk	1.9	20.2	124	95.56	[2]
14	Hyacinth roots	3	14	120	84.23	[24]
15	Maize straw	2	4	2880	99.9	[41]
16	Cabbage waste	3	2	150	99.87 (S*)	This study
17	Cabbage waste	3	2	150	83.81 (Rt*)	This study

(S*) = from synthetic wastewater; (Rt*) = from real tannery wastewater

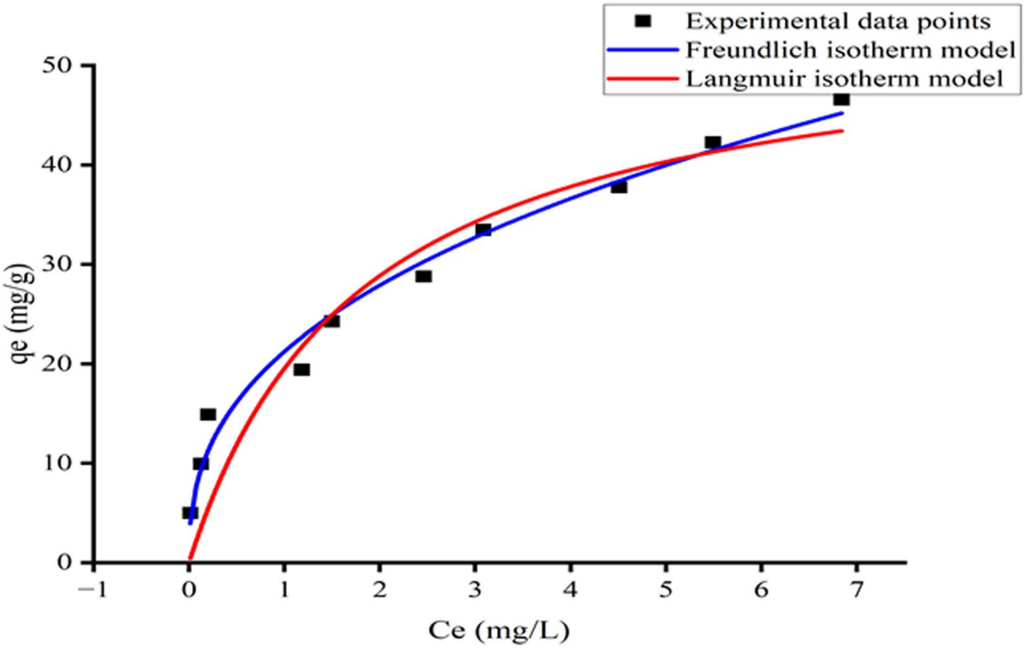

Fig. 11 Freundlich and Langmuir isotherm model

Table 2 Adsorption isotherm models parameters for Cr (VI)

Adsorbent	**Langmuir isotherm model**					**Freundlich isotherm model**			
Parameters	q_{max}	K_L	R^2	X^2	R_L	K_F	$\frac{1}{n}$	R^2	X^2
Value	54.89	0.55	0.89	22.43	0.02–0.15	21.25	0.39	0.98	3.87

$$R_L = \frac{1}{1 + K_L C_0} \tag{7}$$

where, Co (mg/L) is the initial concentration. In the case favorable adsorption, R_L ranges from 0 to 1, whereas $R_L > 1$ denotes unfavorable adsorption, $R_L = 1$ is linear adsorption, $R_L = 0$ denotes irreversible adsorption processes, and $0 < R_L < 1$ denotes favorable adsorption.

According to the data presented in Fig. 11 and Table 2, the Freundlich model with $R^2 = 0.98$ compared to Langmuir with $R^2 = 0.89$ can better describe the adsorption of Cr (VI) ion by activated carbon. This indicates the applicability of the heterogeneous surface and multilayer adsorption process of the Cr (VI) on the surface of activated carbon derived from cabbage waste. In the present study, R_L was found to be 0.02—0.15 indicating favorable adsorption processes; whereas the value of $\frac{1}{n}$ was found to be 0.39. The value $\frac{1}{n}$ is usually less than 1 because sites with the highest binding energies are utilized first, and followed by weaker sites [24].

In this work, kinetic studies were conducted using the 50 mL solution containing 2 g/L of activated carbon separately at a constant concentration of 10 mg/L at pH 3. The impact of adsorption time was studied for 30–300 min. After shaking at 150 rpm for the specified time, the solutions were filtered, and the concentrations of Cr (VI) were determined. Equation (3) was utilized to analyze the adsorbed amount (q_t) of Cr (VI) onto activated carbon at time t.

The adsorption kinetic models employ the following equations: Eq. (8) and (9) provides the pseudo first and pseudo second order adsorption kinetics.

$$\ln(q_t - q_e) = ln(q_e) - K_1 t) \tag{8}$$

$$q_t = \frac{K_2 q_e^2 t}{1 + K_2 q_e t} \tag{9}$$

where, q_t the adsorption capacity (mg/g) at any time t (min), q_e adsorption capacity at equilibrium, K_1 (min^{-1}) is pseudo first order rate constant and K_2 (min^{-1}) is pseudo second order rate constant.

To determine which kinetic models gives the best fit, both models are compared based on the correlation

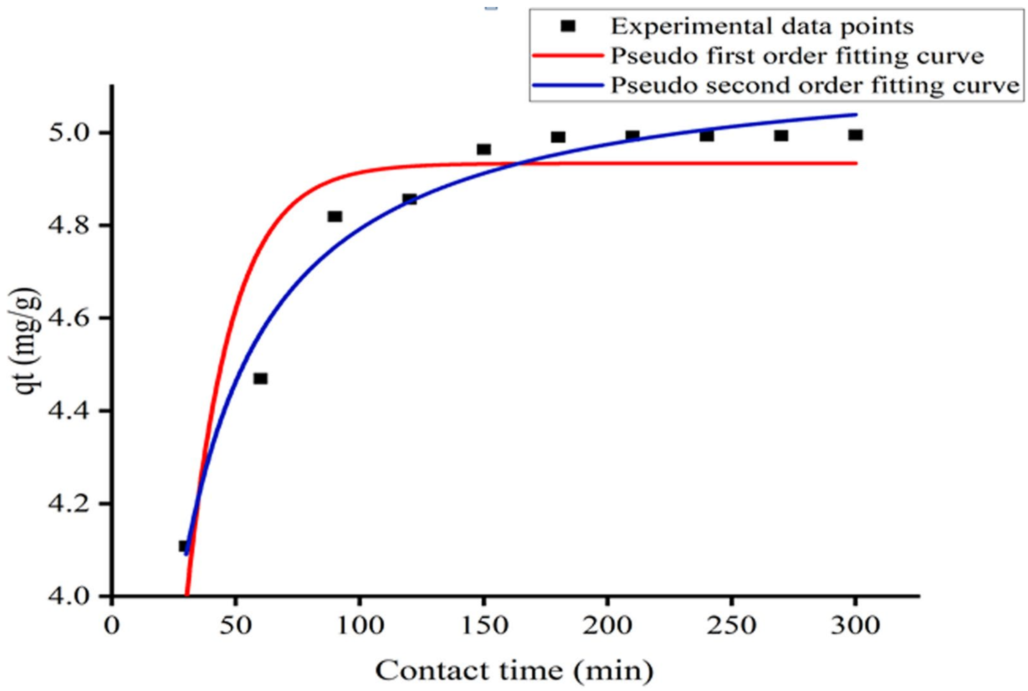

Fig. 12 Pseudo first and Pseudo second order adsorption kinetics model

Table 3 Pseudo first and Pseudo second order adsorption kinetic model

Kinetic model	Pseudo first order model				Pseudo second order model			
Parameters	q_e	K_1	R^2	χ^2	q_e	K_2	R^2	χ^2
Value	4.93	0.06	0.83	0.02	5.17	0.02	0.97	0.0027

coefficient (R^2), adsorption capacities, and chi-square (χ^2). The results indicated that the pseudo second order model described the adsorption process with a higher degree of compliance, expressed by $R^2 =$ 0.97, $\chi^2 =$ 0.0027, than the pseudo first order model, which presents $R^2 =$ 0.83, $\chi^2 =$ 0.02 in Fig. 12 and Table 3. Also, the equilibrium adsorption capacities (q_e) of experimental and predicted values showed better alignment with the pseudo second order model compared to the pseudo first order model for the adsorbent. Therefore, the pseudo second order kinetic model can be concluded as more suitable to describe the adsorption kinetics of Cr (VI) on adsorbent (activated carbon derived from cabbage waste).

Thermodynamic studies of adsorption

The thermodynamic parameters, including Gibbs free energy change (ΔG^0), enthalpy (ΔH^0), and entropy (ΔS^0), were calculated using the following equations (Eq. 10 and 11) [10]:

Table 4 Thermodynamic parameters for adsorption of Cr (VI)

Temperature (K)	ΔG^0 (KJ mol^{-1})	ΔH° (KJmol^{-1})	ΔS° (KJ·mol·K^{-1})
293.15	− 22.94	− 24.82	3.46
298.15	− 26.32		
313.15	− 27.44		
328.15	− 25.47		
343.15	− 25.42		

$$\Delta G^\circ = -RTlnKc \tag{10}$$

where Kc is the distribution constant for adsorption evaluated at each temperature using (Eq. 12), R is universal gas constant (8.314 J mol^{-1} K $^{-1}$), T is the absolute temperature in K (Kelvin).

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \tag{11}$$

$$\mathrm{Kc} = \frac{C_{Ae}}{C_e} \quad (12)$$

where C_{Ae} and C_e is the amount of Cr (VI) ion adsorbed on solid phase at equilibrium and the equilibrium concentration of Cr (VI) left in solution after adsorption in mg/L, respectively. The values of Gibbs free energy change (ΔG) for the adsorption of Cr (VI) by activated carbon at various temperatures are presented in Table 4. ΔH^0 and ΔS^0 were determined from the slope and intercept of the plot of ΔG versus T. Thermodynamic studies showed whether the adsorption is spontaneous ($\Delta G^\circ < 0$) or non-spontaneous ($\Delta G^\circ > 0$), endothermic ($\Delta H^\circ > 0$) or exothermic ($\Delta H^\circ < 0$), with positive or negative entropy values.

Accordingly, the values of ΔG°, ΔH° and ΔS° of the chromium adsorption process by activated carbon derived from cabbage waste represent spontaneous, exothermic and increased randomness of the system, respectively as described in Table 4. The negative values of the standard Gibbs energy change (ΔG°) indicates the feasibility and spontaneous nature of the adsorption process. Whereas, the negative values of the standard enthalpy change (ΔH°) indicate that the adsorption process was exothermic in nature and a given amount of heat is released during the binding of Cr (VI) ions on the surface of adsorbents. On the other hand, the positive values of the standard entropy change (ΔS°) shows the increased randomness at solid–liquid interfaces during the adsorption of Cr (VI) ions onto activated carbon derived from cabbage waste adsorbents.

Desorption study

According to Fig. 13, it seems that the activated carbon obtained from cabbage waste has a limited desorption capacity. Activated carbon made from cabbage waste adsorbent had the higher desorption percentage (13.09%) when using NaOH, followed by KOH (8.22%), HCl (4.61%), H_2SO_4 (3.04%), and HNO_3 (2.71%).

This demonstrates that the adsorption was an irreversible chemisorption process [9]. Chemisorption indicates the existence of a strong chemical bonding between Cr (VI) and the activated carbon obtained from cabbage waste adsorbent, making it challenging to regenerate, recover, and reuse. Therefore, to prevent environmental pollution, the exhausted adsorbent should be properly disposed of.

Conclusion

This study employs activated carbon derived from cabbage waste treated with H_3PO_4 to effectively remove Cr (VI) ions from aqueous solutions and tannery wastewater using a batch adsorption technique. The maximum removal efficiencies and adsorption capacities

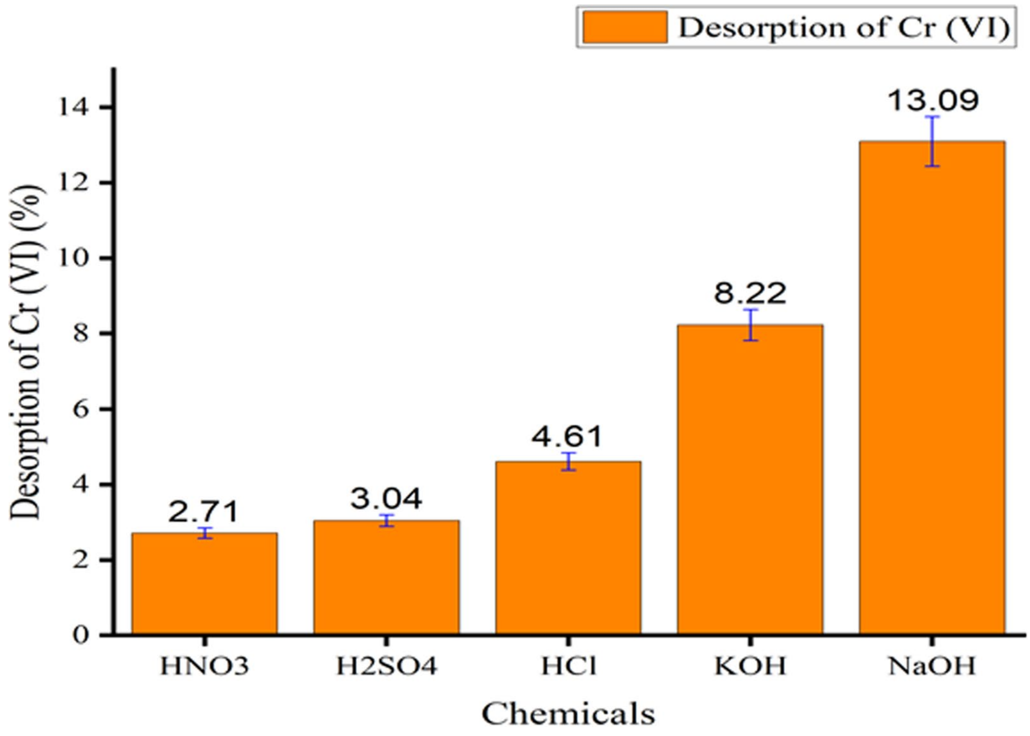

Fig. 13 Percentage of desorption of Cr (VI) with different chemicals

were investigated by optimizing parameters like pH, initial concentration, adsorbent dose, shaking speed, contact time and temperature. The adsorbent achieved maximum removal efficiency of 99.87% and 83.81% for Cr (VI) in aqueous solutions (synthetic wastewater) and real tannery wastewater, respectively, under optimum conditions.

Equilibrium observations confirmed the multilayer adsorption mechanism of Cr (VI) onto the adsorbent is more dominant and fixed extremely well in the Freundlich isotherm equation with adsorption capacity of 4.9 mg/g. Plots were drawn for the Langmuir and Freundlich isotherms. The value of R_L ranges from zero to one indicating favorable adsorption. A pseudo second order model was found to fit the Cr (VI) adsorption data. The findings of this study supported the effectiveness of the technique for the production of low cost carbon materials (adsorbents) and recycling of cabbage waste for many perspectives.

Abbreviations

Cr (VI)	Hexavalent chromium
FTIR	Fourier transform infrared analysis
XRD	X-ray powder diffraction
pHpzc	PH of point of zero charge

Acknowledgements

Authors would like to acknowledge Jimma University for fnancial and material support.

Author contributions

EB and KTA performed the experiments, carried out data analysis, and contributed to drafting and editing. SF, DD, and BN conceptualized, designed, supervised, reviewed, edited, and finalized the manuscript. All authors reviewed and approved the final manuscript.

Funding

Jimma University provided funding for this study. The funders had no involvement in the study's conceptualization, design, data collection and analysis, publication decision, or manuscript preparation.

Data availability

All relevant data were included in the manuscript. Additional datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Ethics approval and consent to participate

In accordance with institutional, national, and international regulations, we confirm that the necessary permissions were secured for the collection of wastewater samples from the tannery in Ethiopia. This was conducted in full compliance with local legislation and ethical standards to ensure proper adherence to all relevant guidelines.

Consent for publication

Not applicable.

Competing interests

The authors declare no competing interests.

Received: 3 January 2025 Accepted: 7 April 2025

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