

Removal of Cr(VI) from aqueous solution using coir pith biochar – An eco-friendly approach

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Coir pith biochar obtained by pyrolysis process is used for Cr(VI) removal from aqueous solution. The biochar has 0.27% moisture and 61.0% carbon contents. Scanning electron micrograph (SEM) of the coir pith biochar shows irregular plates with several developed mesopores over the surface. The coirpith sample optimized with different pH conditions, Cr(VI) concentration and adsorbent dosage. Under optimized condition of pH =7, concentration = 100 mgL⁻¹ and dosage 0.05g, the coir pith biochar removed 99.6% (100 mg L⁻¹) of Cr(VI) from aqueous solution. The Langmuir isotherm model and pseudo-second order rate expression show satisfactory fit to the equilibrium adsorption data of coir pith biochar. Fourier transform infrared spectroscopy of the coir pith biochar confirm the ionic interaction between Cr(VI) and surface functional groups. The results of the study indicate that coir pith biochar is a potential adsorbent for the removal of Cr(VI) from aqueous solution.

Keywords: Biochar, Chromium, Coir pith, FTIR, Sorption

Heavy metal pollution is one of the major threats to our ecosystem, and it increases with the advancement of technology. Metals like Pb, Cd, Cu, As, Cr, Ni, Zn and Hg have been recognized as hazardous heavy metals and have the tendency to bioaccumulate in biotic communities^{1,2}. Cr, a common pollutant, is introduced into the ecosystem due to the discharge of a variety of industrial wastewaters³⁻⁵. In the ecosystem, Cr exist in different oxidation states, with the trivalent (Cr(III)) and hexavalent (Cr(VI)) forms being the most stable. Cr(III) is an essential element necessary for biotic communities for the metabolism of lipids and glucose but is detrimental at high concentrations⁶. On the other hand, Cr(VI), a non-essential metal, is considered more toxic than Cr(III) as it induces mutations, liver damage, pulmonary congestion, and cancer in biotic communities^{7,8}. Furthermore, it has high solubility and mobility compared to Cr(III). Thus, it is necessary to remove Cr(VI) from contaminated wastewater before their discharge into the ecosystem. Conventional methods used for the removal of heavy metals include coagulation, ion exchange, solvent extraction, electrolysis, reverse osmosis, and electrochemical

precipitation⁹⁻¹¹. However, these methods are expensive, high energy processes that generate secondary wastes and are also ineffective in the removal of metal ions at lower concentrations¹². Adsorption is a widely accepted physiochemical method for the removal of heavy metals from wastewater because of its low cost, eco-friendly nature, and ease of operation¹³. Different types of natural and synthetic adsorbents have been used effectively and economically for the removal of Cr from wastewaters and simulated effluents^{5,14-19}.

Coir pith is an agro-industrial waste generated during the extraction of coir fiber from mature coconut husk. It does not carry any direct commercial value and is mostly dumped on road sides. Phenol and tannins leached from the coir pith contaminate the nearby agriculture soils and decrease the agricultural productivity²⁰. Furthermore, the high lignin content and slow degradation rate of coir pith create environmental pollution²¹. The estimated annual production of coir pith in India is about 7.5 million tons²². Therefore, it is important to recycle the coir pith effectively to reduce soil contamination and environmental pollution.

Biochar, a carbon-rich fine grained residue, is produced when a biomass is pyrolysed ($< 700^{\circ}\text{C}$) in the absence of oxygen or under low oxygen pressure. Recently, biochar has received considerable interest because of its ability to ameliorate the physical and biological properties of the soil²³. Woolf *et al.*²⁴ reported biochar to mitigate CO_2 emission from soil. Biochar has antagonistic effects on phytoaccumulation by decreasing metal availability and increasing root surface²⁵. Also, it has been widely used for the adsorption of heavy metals from aqueous solution because of its high surface area, porous nature, active functional groups, and presence of alkali cations^{26,27}. Abdelhafez and Li²⁸ reported that sugar cane biochar removed 86.96 mg g of Pb(II) from aqueous solution. Biochar prepared from oily seeds of *Pistacia terebinthus* L. removed 3.53 mg g of Cr(VI) under optimized conditions, and the adsorption capacity is higher than some chars and activated carbons reported in the literature²⁹. Pan *et al.*³⁰ reported that biochars prepared from peanut, soybean, canola and rice straws have great adsorption capacities ($Q_{\text{max}} = 0.48, 0.33, 0.28$ and 0.27 mol kg) for Cr(III) under acidic conditions. It is well known that the rate of metal adsorption onto biochar varies according to the carbon content, surface functional groups, coordination to π electrons (C=C), precipitation of insoluble matters such as carbonate, phosphate, and hydroxide, and electrostatic interactions^{26,27}. Hence, the objectives of the present study were to (i) synthesize and characterize biochar from coir pith, (ii) evaluate the potential of coir pith biochar (C-BC) for the removal of Cr(VI) from aqueous solution, (iii) assess the influence of experimental variables on Cr(VI) adsorption onto C-BC, and (iv) explore adsorption kinetic and isotherms to understand the mechanism of Cr(VI) removal.

Experimental Section

Materials

Analytical grade chemicals were used in the experiment. Stock solution of Cr(VI) was prepared by dissolving 2.828 g of $\text{K}_2\text{Cr}_2\text{O}_7 \cdot \text{H}_2\text{O}$ (Himedia Laboratories, India) in double distilled water, and working concentrations ($50\text{-}250$ mg L^{-1}) were prepared by diluting the stock solution.

Synthesis and characterization of biochar

Biochar samples were prepared according to Shenbagavalli and Mahimairaja³¹. Briefly, coir pith was collected from Thondamuthur village of

Coimbatore, Tamil Nadu, India. The coir pith was washed extensively with deionized water, oven dried at 60°C for 24 h, and used for the production of biochar using a small scale biochar producing plant (Safire Scientific Company, Tamil Nadu Agriculture University, Coimbatore, Tamil Nadu, India). The produced biochar were sieved (< 0.25 mm) and used for the characterization and adsorption studies.

Proximate and ultimate factors such as moisture content, volatile matter, ash content, and fixed carbon content of the C-BC were determined according to American Society for Testing and Materials (ASTM)³². The size of the C-BC particles and zeta potential were determined using a particle size analyzer Horiba, SZ-100 (Japan). Morphological features and elemental composition of the biochar before and after Cr(VI) adsorption was obtained through scanning electron microscopy-energy dispersive spectroscopy (SEM-EDS) (Quanta FEI 250, Czechoslovakia). Fourier transform infrared spectroscopy (FTIR) spectra of the biochar before and after adsorption of Cr(VI) were obtained on a spectrometer (Shimadzu, Model 8400S) in a diffuse reflectance mode at a resolution of 8 cm^{-1} in KBr pellets. Scanning was done in the range of 400 to 4000 cm^{-1} .

Batch experiments for Cr(VI) removal

Batch experiments were performed according to Deveci and Kar²⁹. Briefly, 0.05 g of C-BC was agitated with 100 mL of metal solution individually at $32 \pm 1^{\circ}\text{C}$ in a rotary shaker. The shaking speed was maintained at 100 rpm throughout the study. Samples were collected at the predetermined time intervals, and the metal solution was separated from the adsorbent by centrifugation at 6000 rpm for 5 min. The supernatant was analyzed for the residual chromium concentration using UV-Vis spectrophotometer (UV-1800 Shimadzu, Japan) at 540 nm, after complexation with 1,5-diphenyl carbazide. Experimental variables considered were (i) the effect of pH on the adsorption capacities, (ii) dosage of C-BC (0.05 to 0.25 g), (iii) and initial chromium concentration (50 to 250 mg L^{-1}).

Data evaluation

Adsorption kinetics was performed using both pseudo first- and second-order kinetics using the following equation³³:

Pseudo first order:

$$\log (q_e - q_t) = \log (q_e) - (k_1/2.303)t \quad \dots (1)$$

Pseudo second order:

$$t/q_t = 1/k_2 q_e^2 + (1/q_e)t \quad \dots (2)$$

where q_e is the amount of metal adsorbed at equilibrium (mg g^{-1}), q_t the amount of metal adsorbed at time t (mg g^{-1}), k_1 (min^{-1}) the pseudo first-order rate constant of the equation calculated from the slope of the plot $\log(q_e - q_t)$ versus t , and k_2 ($\text{g mg}^{-1} \text{min}^{-1}$) the pseudo-second-order rate constant. A plot of t/q_t versus t yields a straight line with a slope of $1/q_e$. The value of k_2 is determined from the intercept of the plot.

The equilibrium data were modelled using the Langmuir isotherm which can be expressed by using standard straight-line equations¹⁷.

$$C_e/q_e = C_e/q_m + 1/K_L q_m \quad \dots (3)$$

where C_e (mg L^{-1}) and q_e (mg g^{-1}), are the solid phase concentration and the liquid phase concentration of adsorbate at equilibrium, respectively, q_m (mg g^{-1}) the maximum adsorption capacity, and K_L (L mg^{-1}) the adsorption equilibrium constant.

Freundlich equation can be expressed as:

$$\log q_e = \log K_F + (1/n) \log C_e \quad \dots (4)$$

where K_F ($\text{mg g}^{-1}(\text{L g}^{-1})^{1/n}$) is the Freundlich constant correlated to the sorption capacity and n the heterogeneity factor. K_F and n values are computed from the linear plot of $\log q_e$ versus $\log C_e$.

The linearized form of Tempkin equation is expressed as

$$q_e = B_T \ln K_T + B_T \ln C_e \quad \dots (5)$$

where $B_T = RT/b$, C_e is the equilibrium concentration of a metal in solution (mg L^{-1}), q_e the amount of Cr(VI) sorbed onto the C-BC (mg g^{-1}), K_T (L g^{-1}) the equilibrium potential corresponding to maximum binding energy, B_T (J mol^{-1}) the Tempkin constant relates to the heat of sorption, R the gas constant ($8.314 \text{ J mol}^{-1} \text{K}^{-1}$), and T the absolute temperature (K). Experiments were repeated twice, and mean values were considered. Blank experiments were carried out without C-BC to ensure that the sorption of Cr(VI) on the walls of flasks was negligible.

Results and Discussion

Characterization of biochar

The results of C-BC proximate analyses are depicted in Table 1. The pH of the C-BC was closest

to alkaline (7.7), which could be due to the detachment of alkali metals such as Ca and Mg from the coir pith. Abdelhafez and Li²⁸ observed similar results in the biochars generated from sugar cane bagasse and orange peel. Moisture content in the C-BC was observed as 0.27%, and the low moisture content indicates the good adsorptive capacity of C-BC³⁴. Fixed carbon content in the C-BC was high (61%), indicating the high carbonization process and providing good possibility for metals to be absorbed onto C-BC. The ash content of the C-BC was low (16%), and it could be due to the absence of oxygen during biochar preparation²⁸. Zeta potential study is a measure of charge stability and controls all particle-particle interaction within a suspension. From Table 1, it could be seen that the zeta potential of biochar was -26.3 mV, which indicates that the C-BC carries negative charge on their surface. The stability behaviour of biochar is found to be incipient instable. If the zeta potential is low, attraction exceeds repulsion and the dispersion will break and flocculate. The particle size ratio of C-BC is found to be 1.00.

Representative scanning electron micrograph of the C-BC is shown in Fig. 1a. The C-BC has irregular plates with several developed mesopores over the surface, which plays a vital role in many liquid-solid adsorption processes, probably due to pyrolysis²⁹. However, the nature of the mesopores C-BC was not altered after Cr adsorption process (Fig. 1b) and suggested that C-BC could be used as low cost adsorbent for several cycles in liquid-solid adsorption process. The energy dispersive spectra of C-BC before and after Cr adsorption are depicted in Fig. 2a and b. The spectra showed the presence of carbon and potassium in both test and control samples. However, the Cr peak (5.2 keV) was observed only in test sample, which confirms that C-BC had adsorbed Cr ions from aqueous solution. Numerous chemical groups such as carboxyl, hydroxyl, alkyl, sulfhydryl, aryl keto, phosphonate, sulfonate, and amide groups

Table 1 — Proximate characteristics of C-BC

S. No	Characteristics	Result
1	pH	7.7
2	EC (ds m^{-1})	2.87
3	Water holding capacity (%)	193.41
4	Moisture content (%)	0.27
5	Volatile content%	18
6	Fixed carbon content%	61
7	Ash Content%	16
8	Zeta potential (mV)	-26.3
9	Particle size (SP Area ratio)	1.00

are thought to contribute to the adsorption of metal ions in biochar. In order to understand the functional groups involved in the adsorption of Cr ions in C-BC, FTIR analysis was carried out, and the results are shown in Fig. 3. The bands at 3757 and 3425 cm^{-1} were assigned to the stretching of the O–H group because of inter- and intra molecular hydrogen

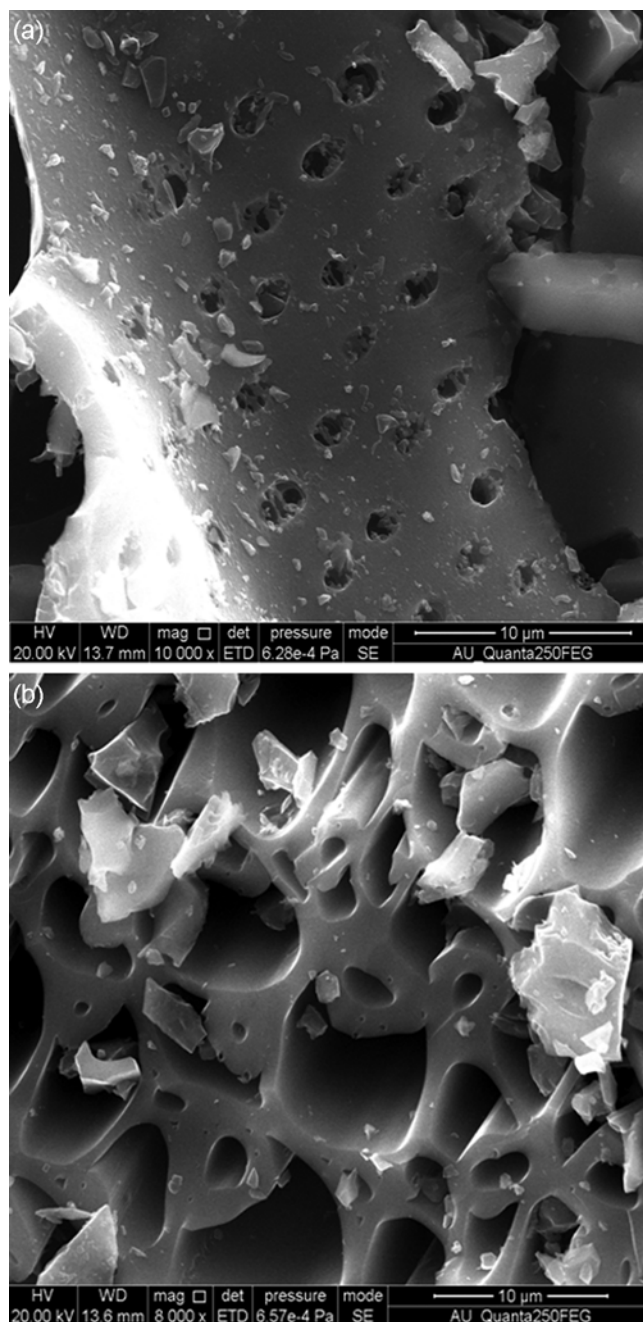


Fig. 1 — Scanning electron micrographs of C-BC before and after Cr(VI) adsorption. The C-BC has irregular plates with several developed mesopores over the surface. a) Before Cr(VI) adsorption, b) After Cr(VI) adsorption

bonding of polymeric compounds³⁵. A peak observed at 2924 cm^{-1} was due to the vibration of metal alkyl compounds. Moreover, C–H aldehyde and aryl keto groups stretching were observed at 2854 and 1689 cm^{-1} , respectively. Peaks observed at 1581, 1442, and 879 cm^{-1} could be due to the vibrations in carboxylic acid groups³⁶. The FTIR peaks of C-BC is similar to those of biochars derived from agriculture biomass^{29,30}. Consequently, the results showed the presence of different surface functional groups in the C-BC that are capable of adsorbing Cr ions from the aqueous solution. However, after Cr adsorption, the band assigned to the carboxylic acid group shifted from 1442 to 1458 cm^{-1} . The electrostatic attraction and chemical bonds formed between carboxylic acid group and Cr ions were responsible for the variation in vibration frequency. Furthermore, new hydroxyl and amino group stretching peaks were observed at

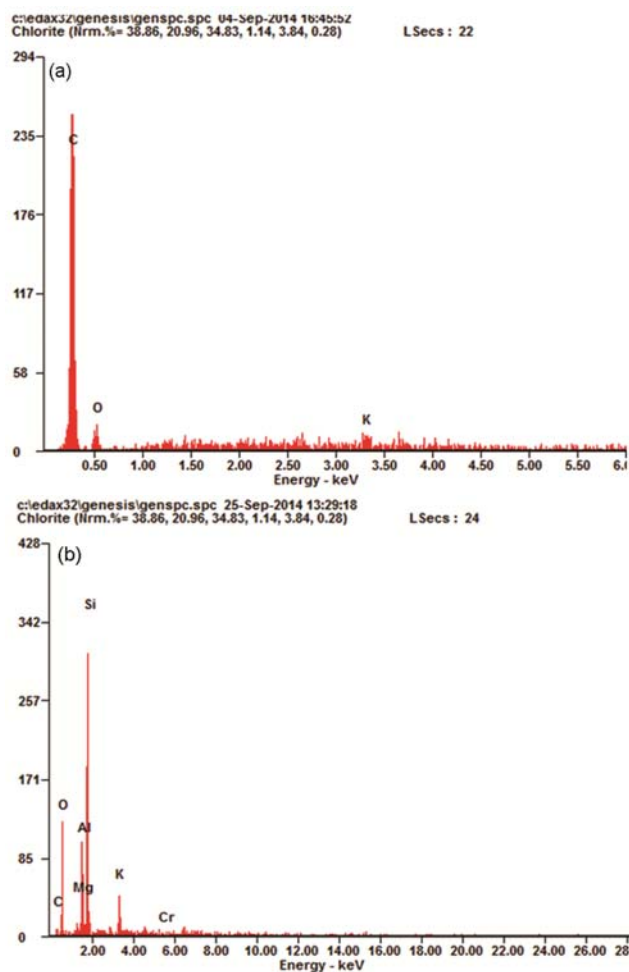


Fig. 2 — SEM-EDS spectra of C-BC before and after Cr(VI) adsorption. a) Before Cr(VI) adsorption, b) After Cr(VI) adsorption

2376 and 1242 cm^{-1} suggesting that Cr adsorption can affect and alter the surrounding environment of the functional groups. The results are in agreement with previous studies indicating the involvement of carboxyl, hydroxyl, and amino groups in Cr adsorption onto biochars^{29,30}.

Effect of Cr(VI) concentration

Adsorbate dosage is considered as an important parameter for effective adsorption processes, as it provides a driving force to overcome all mass transfer resistance of metal ions between the aqueous and solid phase¹⁷. Thus, the effect of initial Cr(VI) concentration (50 to 250 mg L^{-1}) was studied with the optimized parameters (0.05 g of biochar, pH 7.0), and the results are presented in Fig. 4a. A minor decrease (0.4-2%) in the removal percentage was observed according to the increasing concentration of Cr(VI) (50 to 250 mg L^{-1}). The increased sorption at lower concentrations could be due to the availability of more active sites present in biochar. In the later stage, saturation of these active sites and the steric repulsion between solute molecules slow down sorption process and, thereby, decrease the removal capacity. A similar trend of Cd adsorption onto peanut husk biochar was reported by Cheng *et al.*³⁷.

Effect of adsorbent dosage

Adsorbent dosage is considered as an important parameter in the sorption processes as it determines the adsorbent-adsorbate equilibrium of the system³³. Thus, to determine the optimum adsorbent dosage, experiments were carried out with different C-BC

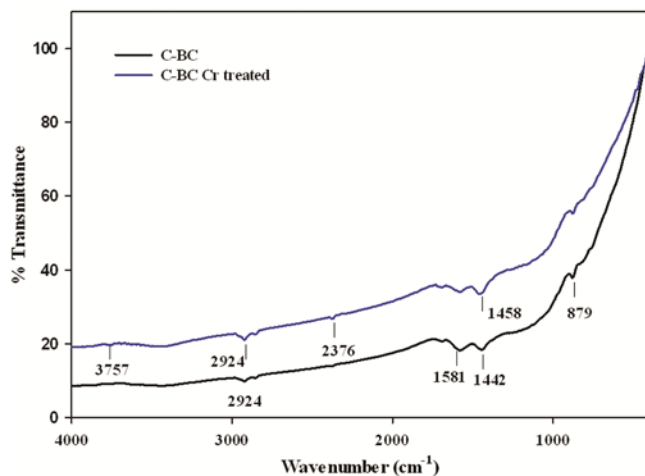


Fig. 3 — FTIR spectra of C-BC before and after Cr(VI) adsorption. A minor shift in the carboxyl groups and new amine peaks was observed in Cr(VI) treated C-BC.

dose (0.05 - 0.25 g), and the results are depicted in Fig. 4b. It was evident from the figure that the removal capacity of Cr(VI) was not significantly altered by C-BC dose, and more than 99% removal was observed in all the dosages. The high adsorption was mainly due to the availability of more active sites and adsorptive surface area present in the C-BC.

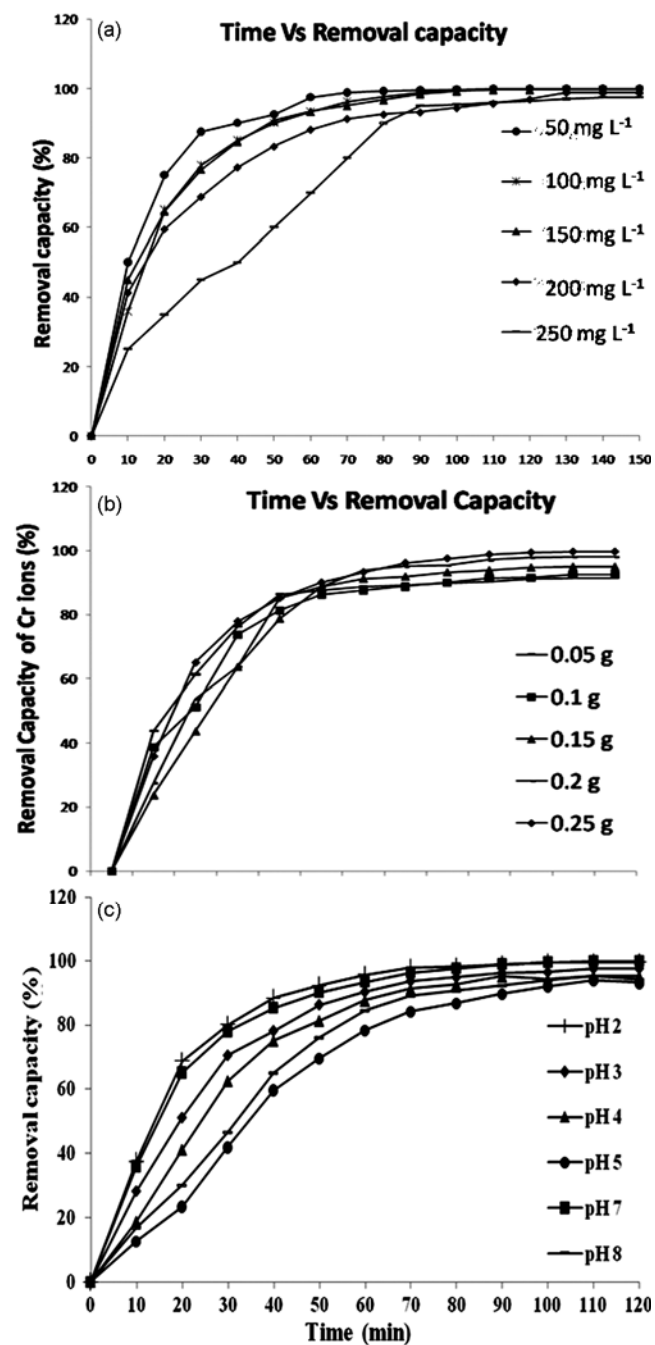


Fig. 4b — Influence of C-BC dosage on adsorption of Cr(VI). A minor increase in adsorption efficiency was observed with increase in adsorbent dose.

Thus, further experiments were carried out using the 0.05 g L⁻¹ as the adsorbent dosage. The results are consistent with previous study reporting increased Cr(VI) adsorption onto biochars derived from oily seeds of *Pistacia terebinthus* L.²⁹.

Effect of pH

pH of the solution is considered as one most significant parameter as its affects the interaction between adsorbent and adsorbate by altering the surface charge and functional groups^{5,33}. Moreover, pH influences the degree of ionization of the materials present in the solution. Thus, 0.05 g of the C-BC was mixed with 100 mL of Cr(VI) solutions (100 mg L⁻¹) at different pH values (2-8), and the results are depicted in Fig. 5. The pH of the solution was altered after the addition of C-BC. From Fig. 4c, it was observed that sorption of chromium takes place at all pH ranges (2-8) with removal capacity of more than 88%. The maximum adsorption (99.6%) was observed at neutral pH (7) and could be due to protonation of both amine and carboxyl groups in the adsorbent, eventually leading to strong electrostatic attraction between the chromate ions and C-BC³⁸. Furthermore, the competition between the OH⁻ ion and the chromate ions weakens neutral pH resulting in a higher uptake of chromium³⁸. This was supported by the results of FTIR where a shift in the carboxyl group and new amine peaks was observed at 1442 and 1242 cm⁻¹, respectively. Thus, further experiments were carried out at neutral pH 7. However, deprotonation of carboxylic group in acidic pH (3-6)

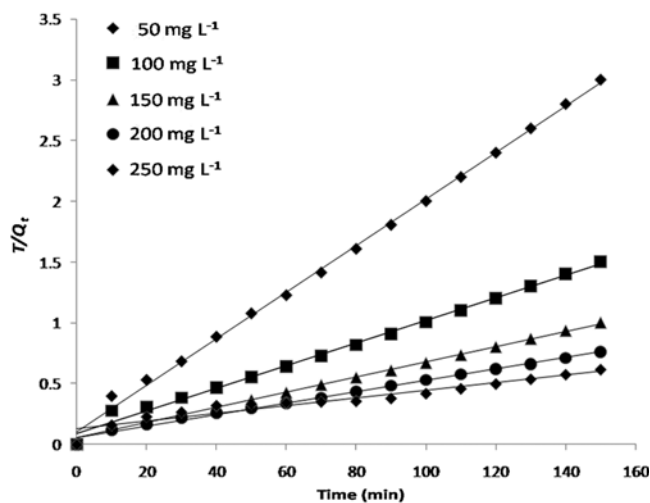


Fig. 5 — Pseudo first-order kinetic plot for adsorption of Cr(VI) onto C-BC at concentration=100 mg L⁻¹, dosage = 0.05 g and pH=7.

may faintly decrease the Cr(VI) adsorption onto C-BC. Similarly, increase of OH⁻ ion concentration in pH 8 and competition between the OH⁻ ion and chromate ions in alkaline solutions may barely hinder the interaction between Cr(VI) ions and C-BC. The results indicate that Cr(VI) adsorption is adaptable to wide range of pH and depends on the characteristics of biochar. The cellulose, lignin, tannins and polysaccharides present in the coir pith can bind with the Cr(VI) ions. Similar observations have been reported by Cheng *et al.*³⁷ for Cd adsorption onto peanut husk biochar.

Isotherm modeling

Adsorption isotherm helps understand the sorption behavior of the adsorption processes. Furthermore, it also provides the information about the distribution adsorbate between the solid phase and liquid phase when it reaches the equilibrium. Thus, the sorption experiments were carried out under optimized conditions (pH = 7; adsorbent dose = 0.05 g L⁻¹; Cr(VI) concentrations = 50-250 mg L⁻¹), and the equilibrium data were analyzed using Langmuir and Freundlich isotherms. Langmuir adsorption assumes that the adsorption occurs at specific homogenous site of the adsorbent and predicts the monolayer coverage. Also, it suggests that the sorption energy is constant and all the sites are energetically equivalent, and there is no interaction between adsorbed molecules on neighbouring sites³⁹. The important Langmuir parameter, q_m and K_L , were determined from the slope and intercept of the plot between C_e/q_e and C_e , and the results are presented in Table 2. Langmuir constant (q_m) and affinity constant (K_L) were used to compare the results, and it represents the adsorption capacity of the adsorbent. Mao *et al.*⁴⁰ reported that the adsorbent with high q_m and K_L values is generally desirable for the wastewater treatment process. The results reveal

Isotherm models	Parameters	C-BC
Langmuir model	q_m (mg g ⁻¹)	249.73
	K_L (L mg ⁻¹)	3.373
	R^2	0.992
	Freundlich model	K_F (mg g ⁻¹) (L mg ⁻¹) ^{1/n}
Temkin model	n	2.89
	R^2	0.96
	K_T	5.1212
	B_T	98.34
	R^2	0.9775

Table 3 — Adsorption kinetic model constants for Cr(VI) adsorption onto C-BC

Kinetic models	Parameters	Initial Concentration (mg L ⁻¹)				
		50	100	150	200	250
Pseudo first order	q_e (exp) (mg g ⁻¹)	49.94	99.62	149.37	197.5	243.75
	q_e (calc) (mg g ⁻¹)	48.52	111.25	130.24	170.18	282.21
	K_1 (min ⁻¹)	-0.0639	-0.0561	-0.0491	-0.0304	-0.0422
	R^2	0.988	0.972	0.976	0.987	0.943
Pseudo second order	q_e (exp)(mg g ⁻¹)	49.94	99.62	149.37	197.5	243.75
	q_e (max)(mg g ⁻¹)	52.198	101.82	159.7	213.36	315.01
	k_2 (g mg ⁻¹ min ⁻¹)	0.001	0.003	0.0007	0.0004	0.0001
	R^2	0.998	0.993	0.996	0.994	0.9120
	h	0.0589	0.2771	0.1127	0.0804	0.0251

that the C-BC has higher q_m and K_L values as compared to the other biochars. Biochar prepared from oily seeds of *Pistacia terebinthus* L. shows an inferior q_m value of 2.43 mg g⁻¹ in the adsorption of Cr(VI)²⁹. Peanut husk biochar exhibited an inferior q_m value of 28.99 mg g⁻¹ in the adsorption of Cd³⁷. Magnetic biochar derived from peanut hull exhibited an inferior q_m value of 77.54 mg g⁻¹ in the adsorption of Cd⁴¹. The differences in the adsorption capacity of biochars could be due to the differences in structure, functional groups, and sugars present in the raw materials, preparation conditions, and proximate characteristics.

Freundlich isotherm is an empirical equation that is used to describe the heterogeneity of the adsorbent surface energy by multilayer adsorption. The Freundlich constants, K_F and n , were determined from the linear plot of $\log q_e$ versus $\log C_e$, and the results along with the correlation coefficients (R^2) are presented in Table 2. The constants, K_F and n , indicates the binding capacity, and the favourability of adsorption process. The higher K_F value, 142.9 mg g⁻¹, indicates the high adsorption capacity of the C-BC. Temkin isotherm assumes that the heat of adsorption of all the molecules in the layer decreases linearly rather than logarithmically with the coverage of sorbate-sorbent interactions. Temkin constants, B_T and K_T , were determined from the intercept and slope of the plot between q_e and $\ln C_e$, and the results along with the correlation coefficients (R^2) are presented in Table 2. The higher B_T value suggests the favourability of Cr(VI) adsorption onto C-BC. The linear correlation coefficients (R^2) of the examined isotherm models indicate that the Langmuir model well describes the adsorption equilibrium data with higher correlation coefficients R^2 (0.992) than the

Freundlich and Temkin models. The results indicate that the Cr(VI) adsorption onto C-BC were homogenous, and the adsorbed Cr(VI) ions did not interact or compete with each other. Moreover, the model describes that the Cr(VI) adsorption was monolayer coverage⁴². The results are consistent with previous studies reporting the adsorption of Cr(VI) onto the biochar to follow Langmuir adsorption model^{37,41}.

Kinetic modeling

The kinetic modelings are often used to describe the efficiency of adsorption process, effectiveness of the adsorbate, and adsorption mechanisms. Thus, the equilibrium data were analyzed with the conventional pseudo first-order and second-order kinetic models, and the corresponding first-order curve is presented in the Fig. 5. Adsorption rate constants and their correlations co-efficients (R^2) were calculated from the curves and are summarized in the Table 3. From the kinetic data, it can be seen that the correlation coefficient for the pseudo first-order kinetic model is very low, indicating a poor fit of the model to the experimental data. However, the correlation coefficient value of pseudo second-order model ($R^2 = 0.99$) was close to unity, indicating the better fit of pseudo second-order model than pseudo first-order model. Moreover, the theoretical values of the q_e calculated from the pseudo second-order model were close to the experimental uptake values q_{ex} . The results of the kinetic analysis indicate that the rate of the adsorption was controlled by the chemisorptions processes. The results are in accordance with previous study reporting that the adsorption of Cr(VI) onto *Pistacia terebinthus* L. seeds biochar followed pseudo second-order kinetics²⁹.

Conclusion

The C-BC effectively removed (> 99%) of Cr(VI) from aqueous solution. The Cr(VI) removal rate is highly influenced by the pH, and the maximum removal was observed at pH 7. The ionic interaction between the Cr(VI) and functional groups present in the C-BC was confirmed by FTIR studies. The results indicate that C-BC is an efficient in-expensive adsorbent for the removal of Cr(VI) from aqueous solution. Moreover, recycling of coir pith waste reduces the environmental pollution and contamination.

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