

Understanding the Adsorption Efficiency of Chitosan Coated Carbon on Heavy Metal Removal

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Abstract: In the present study, the performance of chitosan coated carbon was evaluated for the removal of chromium (VI) and cadmium (II) from its aqueous solution. The chitosan coated carbon was prepared and characterized by using the various analytical tools such as FTIR, TGA, DSC and XRD. Batch adsorption experiments were performed in order to examine the effects of initial concentration, pH, and adsorbent dose and contact time for the removal process. The metal ion removal was pH-dependent. The optimum pH was found to be 5.0 for Cr (VI) and 5.5 for Cd (II). Experimental data were analyzed by Langmuir and Freundlich adsorption isotherms. The characteristic parameters for each isotherm and related correlation coefficients have been determined. The isotherm study revealed that the adsorption equilibrium is well-fitted to the Freundlich isotherm for both the metals. The kinetics study revealed that adsorption of Cr (VI) and Cd (II) onto chitosan coated carbon follows pseudo-second-order kinetics. The results showed that chitosan coated carbon were a favorable adsorbent for both the metals.

Index Terms: Chromium; Cadmium; Chitosan coated carbon; Adsorption isotherms and Kinetics.

Introduction:

The contamination of water by toxic heavy metals is a worldwide problem [1]. Mining activities, agricultural runoff, industrial and domestic effluents are mainly responsible for the increase of metallic species released into the environment [2]. The intake of polluted water may cause serious problems to all living beings. The present research work focuses on two heavy metals - chromium and cadmium, both of which are highly toxic. **Chromium (Cr)** exists in the following oxidation states (di-, tri-, penta-, and hexa) trivalent chromium together with the hexavalent state can be mainly present in the aquatic environment. Trivalent chromium acts as the micro nutrients which is essential for glycolysis whereas hexavalent chromium is 500 times toxic than trivalent chromium. It is carcinogenic and mutagenic as well as being a strong oxidizing agent which irritates the plant and animal tissues even in smaller quantities. It diffuses rapidly through the soil and aquatic environments as well as readily passes through the skin [3].

Cadmium (Cd) exists in +2 oxidation state in the polluted environment. It is carcinogenic to human and also considered to be toxic for plants [4]. A considerable amount of cadmium is present in tobacco smoke and it is reported that the amount of cadmium inhaled from each pack of 20 cigarettes is approximately 16 µg [5]. Cadmium is an industrial and environmental pollutant that creates adverse effects on a number of organs in humans [6]. It is taken up by the human body from the environment through pulmonary and renal pathways. Its harmful effects such as “itai-itai” disease, renal damage, emphysema, hypertension and testicular atrophy [7]. Kidney is one of the major targets of chronic toxicity [8] and it affects the kidney cytochrome [9]. It is also reported that high level exposure to cadmium increased the risk factor for breast cancer [10]. Hence, toxic metals should be removed from the effluent before discharging them into the soil and aquifers.

Among the various methods, adsorption is one of the effective separation processes for a wide variety of applications and is recognized as an effective, economical method for the removal of pollutants from wastewaters. Activated carbon is the most widely used adsorbent [11] which has a number of adsorptive sites that makes it a good adsorbent, but it is quite expensive and, possibly, not cost-effective in the treatment of large wastewater volumes. That is why, in the last few years, considerable attention has been paid to low-cost biosorbents-as an alternative to reduce the cost of adsorption systems.

Many naturally occurring polymeric materials (from marine waste) have been investigated for assessing their suitability in controlling water pollution. Among them chitosan, a hydrophilic natural polymer produced by alkaline de-acetylation of chitin is chosen for this study. It is characterized by a high content of nitrogen present as amine groups capable of adsorbing the metal ions through several mechanisms – including chemical interactions, such as chelation, electrostatic interactions or ion-exchange. Several studies have shown that chitosan is very efficient in removing various toxic and strategic metals, such as chromium, cadmium, mercury, molybdenum, uranium, vanadium, platinum and palladium [12],[13].

The present study is undertaken with the following objectives: (1) to prepare chitosan coated carbon by increasing the times of coating from 3 to 5 times and its characterization. (2) To investigate the use of chitosan coated carbon as an adsorbent for Cr(VI) and Cd(II); (3) to study the effect of different experimental parameters such as pH, adsorbent dose, and contact time, (4) to comprehensively evaluate the effect of several pretreatments on Cr(VI) and Cd(II) uptake by chitosan coated carbon adsorbent.

Experimental

Preparation of carbon

The coconut shell was broken into small pieces and ground well. This was burnt at a very high temperature for 2-3 hours. The surface of carbon obtained was activated by shaking the same with 7% sulphuric acid for 24 hours. The carbon was then washed several times with de-ionised water till there is no acid in the water. The sulphonated carbon was dried in hot air for over 5 hours at 110°C.

Surface coating of carbon with chitosan

About 400 ml of chitosan gel was diluted with water and heated to 40-50°C. About 400 g of activated coconut shell carbon was slowly added and mechanically agitated using a rotary shaker at 150 rpm for 24 hours. This gel coated activated carbon was washed with de-ionised water and dried. This process was repeated to get 1, 2, 3, 4 and 5th coating at chitosan on activated carbon. 3, 4 and 5th coating carbon were used for the studies.

Characterization

FTIR measurements of the prepared polymeric samples was recorded by Fourier transform infra-red spectrophotometer (FT-IR) using the Perkin Elmer 200 FTIR spectrophotometer, in the range of 400-4000 cm^{-1} at 25°C with a resolution of 4 cm^{-1} . The thermal analysis (TGA and DSC) was carried out on a Perkin Elmer thermal analysis instrument. For DSC analysis the pierced lid in the nitrogen atmosphere at a heating rate of 10° K/min was used. The XRD Pattern of the various polymeric blend samples were tested by an X-ray scattering SHIMADUZ XD-DI Diffractometer using Ni filter Cu K α radiation source ($\lambda=0.154\text{nm}$), set as scan rate =10°/min, using a voltage of 40kv and a current of 30mA.

Stock solution

A stock solution of chromium (VI) ions and cadmium (II) ions were prepared separately by dissolving the potassium dichromate and cadmium chloride of 200 mg each per liter using twice-distilled water and standardized after preparation. 1:1 hydrochloric acid and 1% sodium hydroxide solutions were used for pH adjustment. The exact concentration of each metal ion solution was calculated on mass basis and expressed in terms of mg L^{-1} . The required lower concentrations were prepared by dilution of the stock solution.

Adsorption experiments

Batch studies were performed with different concentrations of potassium dichromate to investigate the extent of adsorption. Synthetic solutions of Cr (VI) ions and Cd (II) ions were taken in separate stopper bottles and agitated with the chitosan coated carbon at 30°C in orbit shaker at a fixed speed of 160 rpm. The extent of heavy metal removal was investigated separately by changing the adsorption dose, contact time of shaking and changing the pH of the solution. After attaining the equilibrium, adsorbent was separated by filtration using filter paper and aqueous phase concentration of metal was determined with atomic adsorption spectrophotometer (Varian AAA 220FS).

Results and Discussion

FTIR:

FTIR data revealed very important information about the interaction and polymeric association between chitosan and carbon. All the samples demonstrated similar characteristic peaks showing that similar changes were taken place during the coating of carbon onto chitosan, as presented in the figures 1a – 1c. The FTIR spectrum of pure chitosan sample shows a predominant broad band at around 3440 cm^{-1} corresponding to the stretching vibration of O-H and N—H. The peaks at 2924 and 2846 cm^{-1} are typical of C—H stretch vibration, while peaks at ~1639, 1540 and 1317 cm^{-1} are characteristic of amides I, II and III, respectively and the bands at 1153 and 1088 cm^{-1} are indicative of C—O stretching vibrations of the glycoside linkage.

Now, on interpreting the FTIR spectra of chitosan coated carbon, the spectrum shows the absorption of both chitosan and carbon confirming the successful coating of chitosan using carbon. The bands at around 2924 cm^{-1} and 2857 cm^{-1} correspond to the asymmetric and symmetric stretching. The peaks in the range of 1628 cm^{-1} , 1151 cm^{-1} , 1383 cm^{-1} , 1017 cm^{-1} and 886 cm^{-1} were

assigned to N-H bending, C-N stretching, O-H in plane bending, C-C-C Skeletal in the backbone and CH₃-C-OH stretching respectively. Bands around 652 cm⁻¹ and 479 cm⁻¹ indicate the presence of OH and C-C bending vibrations respectively.

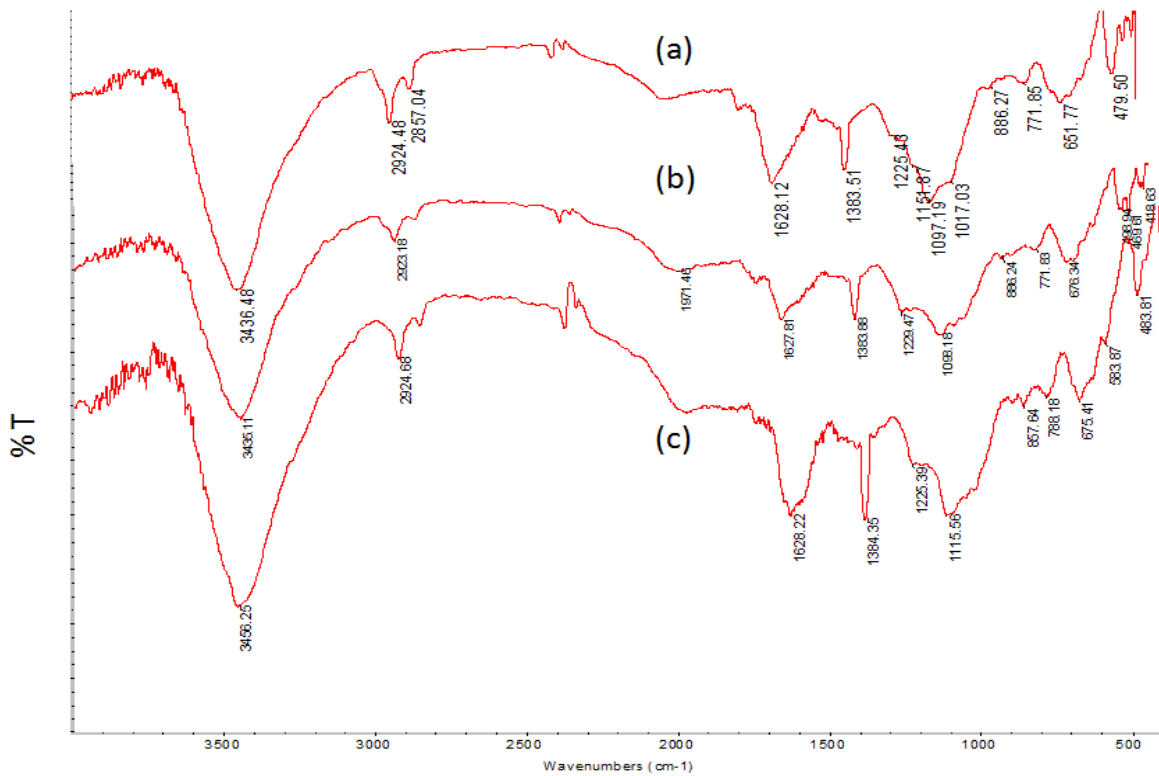


Figure 1: FTIR spectra of chitosan coated carbon (a) 3 times coated (b) 4 times coated (c) 5 times coated

DSC analysis

Figures 2a – 2c, represent the DSC details of chitosan coated carbon 3rd, 4th and 5th time. The effect of carbon coating onto the chitosan were reflected in the thermal behavior of the samples. DSC shows one broad endothermic peak for all the samples, at the temperatures of about 75.04°C, 75.04°C and 78.27°C indicating the crystallization temperature. This is due to the loss of water. The single glass transition temperature was observed at 163.31°C, 210.54°C and 180°C respectively showing the compatibility of the sample. On comparing the DSC curve of the prepared samples with pure chitosan, the samples show a shift in the glass transition temperature. From the above results, it is concluded that the coating of carbon onto the chitosan increases the thermal stability.

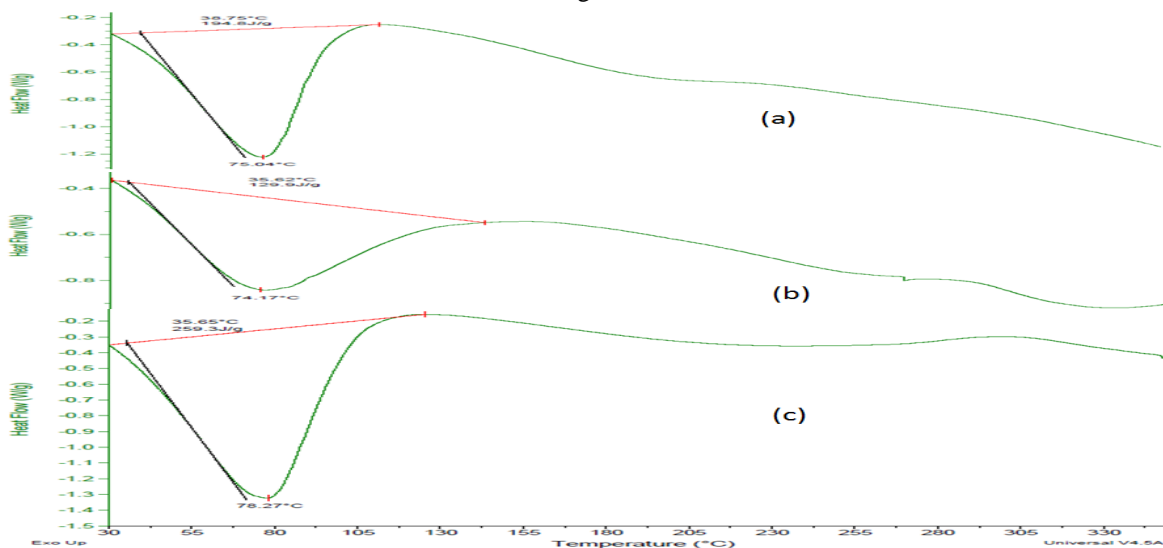


Figure 2: DSC of chitosan coated carbon (a) 3 times coated (b) 4 times coated (c) 5 times coated

TGA studies

The TGA thermogram details of chitosan coated carbon samples were represented in the Figures-3a – 3c. Generally, three stages of decomposition were attributed. The first stage of transition falls in the range of 30°C -110°C shows about 4 - 6% weight loss due to the evaporation of loosely bound water molecules from the samples. The second stage-a predominant one, was the maximum weight loss which had taken place indicating the breaking of the polymer structure. The maximum decomposition taking place at the temperature range from 150 to 450°C for the prepared samples chitosan coated with 3rd, 4th and 5th time using carbon, around 69.014%, 65.19% and 72.65% of the sample had disintegrated at the temperature of 789.96°C, 756.66°C and 743.28°C respectively. In the third stage, the breakdown of the polymeric backbone took place [14].

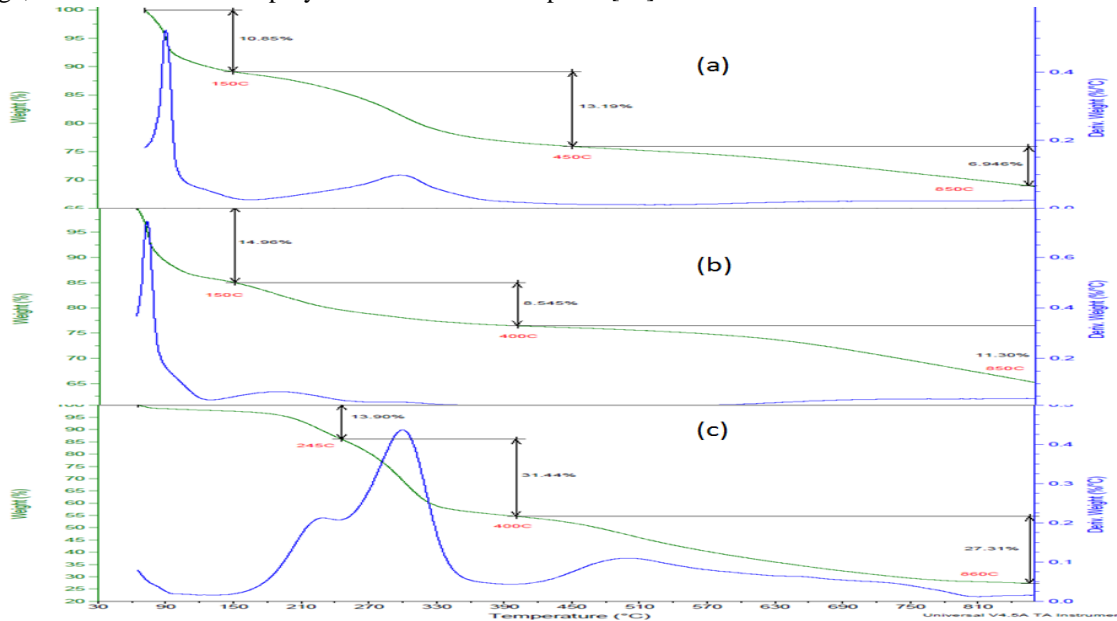


Figure 3: TGA of chitosan coated carbon (a) 3 times coated (b) 4 times coated (c) 5 times coated

XRD studies

Figure 4a – 4c displays the diffraction patterns of the chitosan coated carbon 3rd, 4th and 5th time. Similar strong reflections are observed for all the samples with $2\theta=24^\circ$ and 42° which is associated with the crystalline region. When compared to pure chitosan, the qualitative changes were observed, the 10° and 20° reflection for pure chitosan is absent confirming that the coated carbon onto chitosan changes the morphology. The broad peaks in the range of $16^\circ - 30^\circ$ and $35^\circ - 50^\circ$ shows samples having amorphous nature. The results indicate that there is a decrease in chitosan crystallization on coating with carbon. The decrease in the crystalline nature makes it the best adsorbent for the heavy metal remediation.

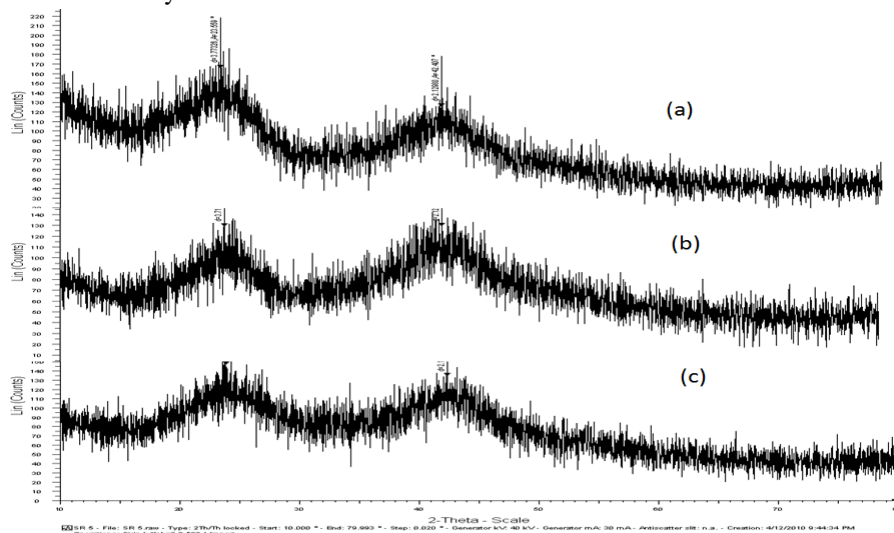


Figure 4: XRD of chitosan coated carbon (a) 3 times coated (b) 4 times coated (c) 5 times coated

Effect of contact time

It has been observed that at a constant concentration of metal ions and fixed amount of adsorbent, the adsorption efficiency increases with increasing the contact time up to a certain level and then it reaches the equilibrium. Fig.5 shows that adsorption rate first increased rapidly as the contact time increases, but after reaching the optimum time of about 300 min there is no significant increase. The effect may be due to the saturation of adsorption sites with metal ions on the solid particle. The optimum contact time for chromium (VI) was 300 min. with 59.2% adsorption and 300 min. for cadmium (II) with 71.5% adsorption. The slight decrease in adsorption after optimum contact time may be due to the breakage of newly formed weak adsorption bonds due to constant shaking.

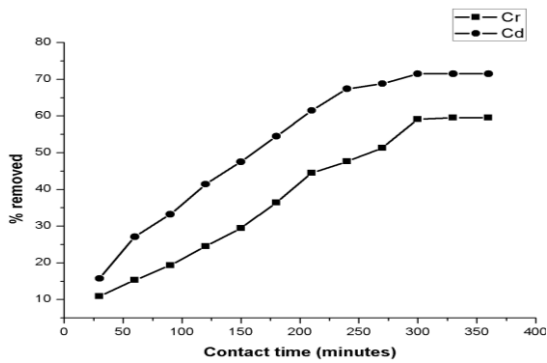


Figure 5: Effect of contact time

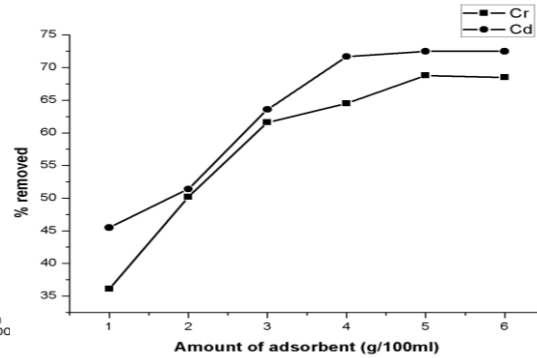


Figure 6: Effect of adsorbent dose

Effect of adsorption mass

The effect of the adsorbent dose was studied at room temperature by varying the sorbent amounts from 1 to 6 g. For all these runs, initial concentration of the metal ions was fixed. Figure 6 shows the adsorption of Cr(VI) and Cd(II) ions increase rapidly with increase in the amount of adsorbent due to greater availability of the surface area at higher concentration of the adsorbent. The significant increase in uptake was observed when the dose was increased from 1 to 4 g. Any further addition of the adsorbent beyond this, did not cause any significant change in the adsorption. This might be due to overlapping of adsorption sites as a result of overcrowding of the adsorbent particles. These results indicate that removal efficiency is directly related to the number of available adsorption sites. Once equilibrium is attained, there is no effect on adsorption efficiency.

Effect of pH

The adsorption process is strongly affected by the pH of the solution. The effect of pH change on adsorption was studied for both metals by changing the pH of the contents from 4-8, using dilute solutions of HCl and NaOH.

It has been observed from fig. 7 that maximum adsorption of both metals took place in acidic media. Maximum adsorption for Cr (VI) was observed at pH 5 and cadmium at pH 5.5. Published literature showed that at very high and very low pH values, the surface of adsorption was surrounded mainly by H⁺ and OH⁻ ions. These positively and negatively charged ions may compete with the metal ions and as a result, adsorption decreases. That is why metal ions show low adsorption at very high and low pH [15]. On other hand, sometimes, precipitation of metal ions as hydroxide also occurs at high basic pH values, which is not feasible for good adsorption.

Influence of pH on adsorption phenomenon also related with the functional groups present on the bio-adsorbent. The potential binding sites on the bio-adsorbents might be carbohydrates, amino groups, hydroxide groups and carboxylic groups. These functional groups might dissociate or ionize at different pH values. So, the surface chemistry of the functional groups also play an important role in the adsorption process [16].

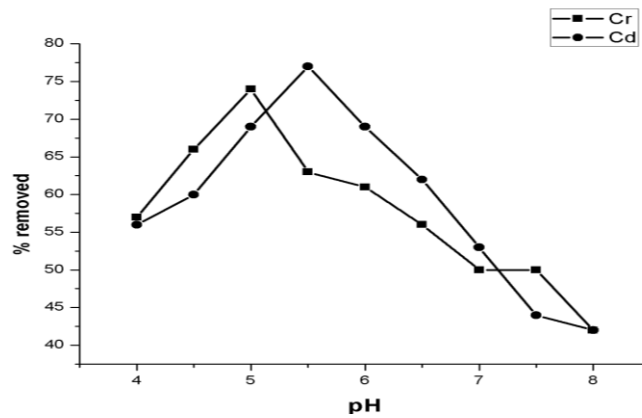


Figure 7: Effect of initial solution pH

Adsorption isotherms – Langmuir and Freundlich

The adsorption isotherm is fundamental in describing the interactive behavior between solutes and adsorbent [17],[18]. According to Ofomaja and Ho, the isotherm yields certain constant values, which express the surface properties and affinity of the adsorbent. It also plays an important role in the design of an adsorption system. The Langmuir and Freundlich models are often used to describe equilibrium adsorption isotherms. The most widely used Langmuir equation, is valid for monolayer sorption onto a surface with a finite number of identical sites. It is assumed that once a metal ion occupies a site, no further adsorption can take place at that site [19].

The Langmuir equation, which is also based on the assumption that there is no reaction between the molecules adsorbed on neighboring sites, is given by:

$$C_{eq}/C_{ads} = [1/Qb] + [C_{eq}/Q] \quad (1)$$

where Q is the maximum adsorption at monolayer ($mg\ g^{-1}$), C_{eq} is the equilibrium concentration of metal ions (ppm), C_{ads} is the amount of metal ions adsorbed per unit weight of chitosan coated carbon at equilibrium concentration ($mg\ g^{-1}$) and b is the Langmuir constant related to the affinity of binding sites ($ml\ mg^{-1}$) and is a measure of the energy of adsorption. A linearized plot of C_{eq}/C_{ads} against C_{eq} gives the Langmuir constant Q and b .

The widely used empirical Freundlich equation based on sorption on a heterogeneous surface is given by:

$$C_{ads} = K C_{eq}^{1/n} \quad (2)$$

The linearised form of this equation is expressed as

$$\log C_{ads} = 1/n \log C_{eq} + \log K \quad (3)$$

where K and n are Freundlich constants indicating adsorption capacity ($mg\ g^{-1}$) and intensity, respectively. K and n can be determined from a linear plot of $\log C_{ads}$ against $\log C_e$. The calculated results of the Langmuir and Freundlich isotherm constants are given in Table 1.

Table 1- Adsorption isotherm constant, C_{max} and correlation coefficients

Metal ions	Langmuir constants				Freundlich constants		
	K_L (dm^3/g)	b (dm^3/mg)	C_{max} (mg/g)	R^2	K	n	R^2
Cr(VI)	4.522	0.002745	1647.36	0.8107	0.2760	1.0735	0.9998
Cd(II)	3.648	0.004385	831.93	0.8496	0.3909	1.1299	0.9999

From the table, it is found that the adsorption of Cr (VI) and Cd (II) ions onto the adsorbent correlates well with the Freundlich equation as compared to Langmuir equation under the various concentration ranges studied. Also, the Freundlich constant n shows the feasibility of heterogeneous adsorption.

The essential features of a Langmuir isotherm can be expressed in terms of a dimensionless constant separation factor or equilibrium parameter, R_L that is used to predict if an adsorption system is “favourable” or “unfavourable” [20]. The separation factor, R_L is defined by:

$$R_L = 1 / (1 + bC_f)$$

where C_f is the final Cr (VI) concentration (ppm) and b is the Langmuir adsorption equilibrium constant (ml mg^{-1}). The parameter indicates the Effect of separation factor on isotherm shape

R_L value Type of isotherm

$R_L > 1$ Unfavourable

$R_L = 1$ Linear

$0 < R_L < 1$ Favourable

$R_L = 0$ Irreversible

according to Table 1 and 2 . The values of R_L calculated for different initial metal ion concentration are given in Table: 2. If the R_L values are in the range of $0 < R_L < 1$, it indicates that the adsorption of Cr (IV) and Cd (II) onto chitosan coated carbon is favourable. Thus, chitosan coated carbon is a favourable adsorbent.

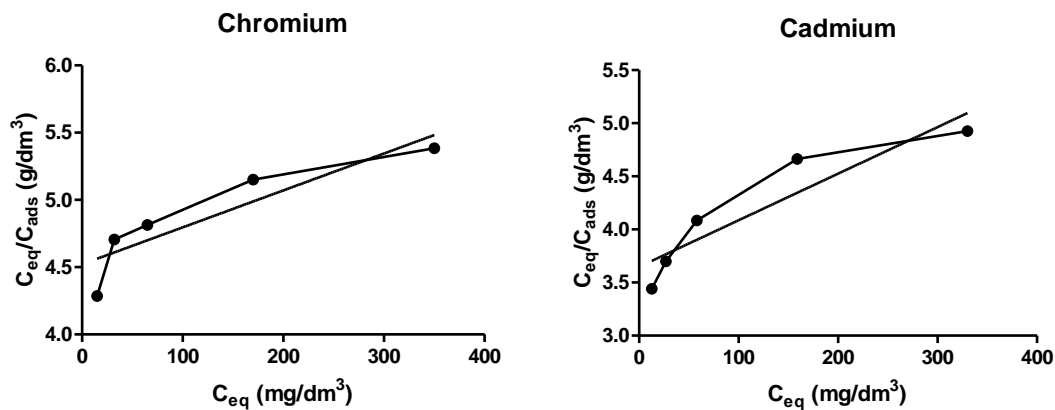


Figure 8: Langmuir isotherm for (a) Cr (VI) ion; (b) Cd (II) ion

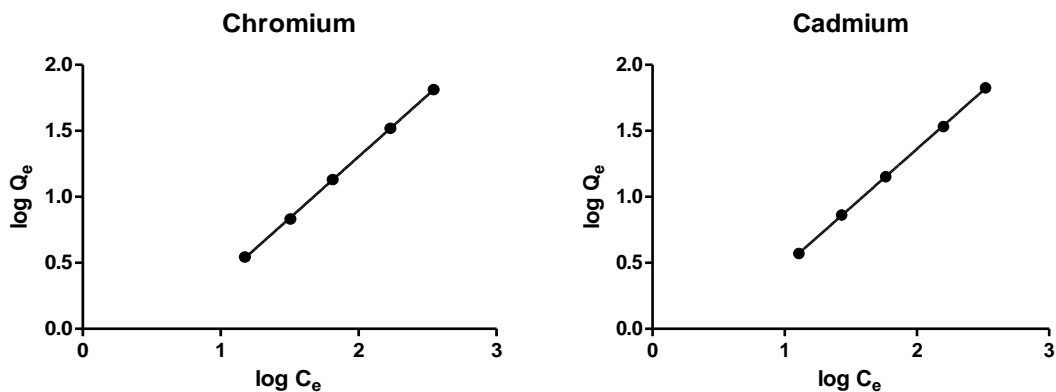


Figure 9: Freundlich isotherm for (a) Cr (VI) ion; (b) Cd (II) ion

Table 2 R_L values based on Langmuir adsorption

Metal ions	Initial concentration C_0 (mg/dm ³)	Final concentration C_f (mg/dm ³)	R_L values
Cr(VI) ion	1000	350	0.5100
	500	170	0.6818
	200	65	0.8486
	100	32	0.9192
	50	15	0.9604
Cd(II) ion	1000	330	0.4086
	500	159	0.5892

	200	58	0.7972
	100	27	0.8941
	50	12.8	0.9468

Kinetics Study:

The sorption data of Cr (VI) and Cd (II) uptake by chitosan coated carbon adsorbent is fitted using Lagergren pseudo-first-order model (Fig.10a and 10b) and pseudo-second-order model (Fig.11a and 11b). The linearized form of first order Lagergren equation is given as Eqn.4

$$\log(q_e - q_t) = \log q_e - \frac{k_{1,ads}}{2.303} t \tag{4}$$

The pseudo-second-order equation Eqn.5

$$\frac{t}{q} = \frac{1}{k_{2,ads} q_e^2} + \frac{t}{q_e} \tag{5}$$

where q_e is the mass of metal adsorbed at equilibrium (mg/g), q_t the mass of metal at time t (min.), $k_{1,ads}$ the first-order reaction rate of adsorption (per minute), $k_{2,ads}$ the pseudo-second-order rate constant of adsorption mg/g min^{-1} . A comparison between two kinetic models suggested (Table.3) that the coefficient of correlation (R^2) for the pseudo second-order kinetic model is much higher in comparison to pseudo-first-order model (2) the close agreement between the experimental q_e (mg/g) values and the estimated q_e (mg/g) values from pseudo second- order kinetic model. These facts obtained suggest that Cr and Cd kinetic data followed the pseudo-second order kinetic model which describes the biosorption as the rate limiting step [21].

Table 3 Comparison between Lagergren pseudo-first-order and pseudo-second-order kinetic models for Cr (VI) and Cd (II) sorption by chitosan coated carbon

Metal ion	Pseudo-first-order kinetic model			Experimental value	Pseudo-second-order kinetic model		
	q_e (mg/g)	k_1 (min^{-1})	R^2		q_e (mg/g)	k_2 ($\text{g mg}^{-1} \text{min}^{-1}$)	R^2
Cr(VI)	767.10	0.002924	0.9485	135	555.79	0.003217	0.7040
Cd(II)	505.70	0.004562	0.9665	148.8	206.95	0.004244	0.9757

Adsorption of Cr(VI) and Cd (II) onto chitosan coated carbon follows second order kinetics.

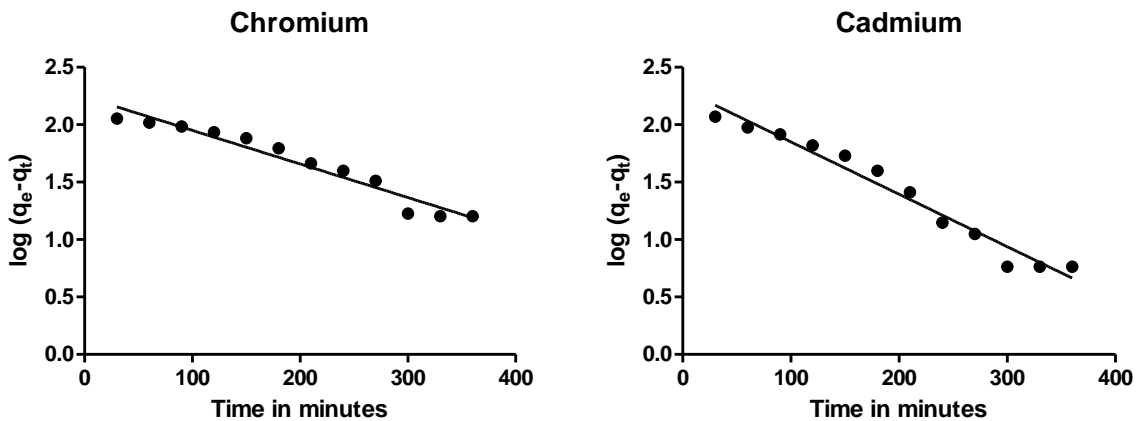


Figure 10: Pseudo-first-order kinetics plot (a) Cr (VI) ion; (b) Cd (II) ion

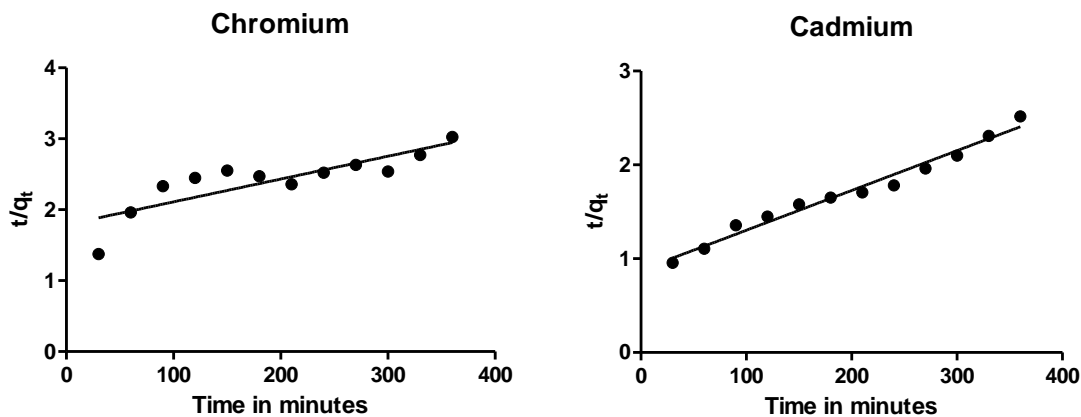


Figure 11: Pseudo-first-order kinetics plot (a) Cr (VI) ion; (b) Cd (II) ion

Free energy change (ΔG)

The change in the free Gibbs energy (ΔG) is calculated from the Langmuir constant using equation $\Delta G = -RT \ln K_L$ (where R is the universal gas constant (8.314 J/mol K), K_L is Langmuir constant and T is the absolute temperature). For Cr(VI) $K_L=4.522(\text{dm}^3/\text{g})$, and for Cd(II) $K_L= 3.648$. $T= 25^\circ\text{C}$.

Hence, $\Delta G= -313.636$ KJ/mol for Cr (VI)

$\Delta G= -268.99$ KJ/mol for Cd (II)

The negative value of ΔG indicates that the adsorption process of Chromium (VI) and Cadmium (II) onto chitosan coated carbon is feasible and spontaneous.

Conclusion

The adsorption of Cr (VI) and Cd (II) ions from aqueous solutions onto chitosan coated carbon was investigated. The experiments were performed in batch system, at room temperature (25 ± 0.5 °C), on analyzing the effects of the following variables: initial solution, pH, contact time and chitosan dosage. The results lead to the following,

- The adsorption of Cr (VI) and Cd (II) onto chitosan coated carbon increases by changing the pH from acid towards neutral, while the optimum pH value for Cr (VI) removal is found to be 5.0 and for Cd (II) was found to be 5.5
- The equilibrium state of the interaction between chromium, cadmium ions and the chitosan coated carbon surface is reached after a 300 min contact time.
- The removal of both metals increases with the chitosan coated carbon dosage increase.
- The application of the Langmuir and Freundlich isotherm models for the mathematical description of Cr (VI) and Cd (II) adsorption shows that the Adsorption equilibrium data well fitted to Freundlich than the Langmuir isotherm, the maximum adsorption rate (C_{max}) – of 1647mg/g for Cr(VI) (C_{max}) – of 831.93mg/g and occurring under the experimental conditions studied.
- The negative value of the free Gibbs energy (ΔG), indicating that the adsorption process of both metal ions onto chitosan coated carbon is feasible and spontaneous.
- The adsorption process describes adequately Pseudo-second-order kinetics.

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