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Modified coconut coir to remove hexavalent chromium from aqueous solution

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Abstract

Hexavalent chromium (Cr(VI)) was removed from aqueous solution by adsorption using chemically modified lignin-rich low-cost coconut coir. The equilibrium time, adsorption isotherm and adsorption kinetics at certain conditions such as pH, particle size and the initial concentration of adsorbate were investigated. The coconut coir was modified by sodium chlorite for adsorption studies. The scanning electron microscopic image depicted the morphology of SCM-CC of different particle sizes whereas the elemental composition of this sort of bioadsorbent was determined by using energy dispersive spectroscopic data. Fourier transform infrared (FTIR) spectrometry analysis indicated that the carbonyl (C=O) groups and hydroxy (O–H) groups from the lignin structure in coconut coir may be involved in the mechanism of Cr(VI) adsorption. The maximum Cr(VI) adsorption capacity of sodium chlorite modified coconut coir (SCM-CC) was found to be 28.03 mg Cr(VI) g^{-1} SCM-CC at pH 2.00 for the particle size of 75-100 μm with the dose of 7.5 g/L. The equilibrium was established within three hours resulting in the maximum removal (99.92%) of chromium. Freundlich isotherm was better fitted than Langmuir isotherm at pH 2.00 on the basis of the regression coefficient (R^2). The kinetic data well-fitted with Ho's pseudo second order kinetics. The SCM-CC can be treated as an excellent adsorbent for the remediation of chromium toxicity.

Keywords: Coconut coir; sodium chlorite; chromium (VI); adsorption; isotherm; removal

Introduction

The heavy metals (e.g., As, Pb, Cd, Cr etc.) induce toxic impacts on all the living entities including humans if they are thrown to the water without proper treatment. Among the three common stable valence states: 0, 3+ and 6+ the hexavalent chromium (Cr(VI)) has been found to impose the greatest threat to the living organism. It can naturally be found in the crust of the earth, although it is primarily emitted to the environment as a result of anthropogenic activities. The researchers (Mukherjee, 1998; Kotas and Stasicka, 2000; Ellis *et al.,* 2002; Paustenbach *et al.,* 2003; Krystek and Ritsema, 2007) have extensively reviewed the occurrence and distribution of chromium in the environment. Serious health problem is caused by the uncontrolled release of Cr(VI) in the environment without appropriate treatment. It causes respiratory, skin (Bruynzeel *et al.,* 1988), carcinogenic (Dayan and Paine, 2001), renal (Kirschbaum *et al.,* 1981),

hepatic (Pascale *et al.,* 1952), gastrointestinal, cardiovascular (Loubieres *et al.,* 1999), hematological, genotoxic and mutagenic effects on human body.

Several studies have been carried out so far in order to find effective adsorbent to mitigate chromium from polluted water. Raji and Anirudhan (1998) used batch process for removing Cr(VI) by polyacrylamide-grafted sawdust and studied kinetics and thermodynamics. The process was found to be exothermic with a maximum adsorption of 91.0% at 30 °C for an initial concentration of 100 mg/L at pH 3.00. Coconut coir was also used as bioadsorbent in order to remove Cr(VI) from laboratory waste water (Gonzalez *et al.,* 2008). The research revealed that about 17% of Cr had been removed from sulphochromic waste water solution by dried coconut coir whereas 72% of Cr was removed by hydrated coconut coir.

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Research for investigating the biosorption of chromium from industrial waste water was also done of by using pistachio hull waste biomass (Moussavi *and* Barikbin, 2010). Inorganic mineral as well as activated carbon are also being used for the removal of chromium from waste water. Wanees *et al.* (2012) investigated the adsorption potential of activated carbon and bentonite for the removal of Cr(VI) ions from waste water. They conducted comparative study of the adsorption of Cr(VI) on these two surfaces at different conditions, i.e., contact time, adsorbent dose, temperature, pH etc. through thermodynamic and kinetic study. Bentonite was found to be more effective adsorbent in removing Cr(VI). In comparison with the adsorbents (carbon and bentonite) mentioned above, the adsorbent (sodium chlorite treated coconut coir (SCM-CC)) used in this study is more cost effective and easily available in order to carry out the present work.

In the present study raw coconut coir was modified by sodium chlorite to enhance the adsorption capacity which was not good enough in the previous study for the removal of Cr(VI) by this sort of bioadsorbent (Gonzalez *et al.,* 2008). Besides, the adsorption of Cr(VI) on SCM-CC was also investigated under several experimental conditions such as contact time, particle size, pH and the initial concentration of adsorbate solution. The major goal has been the optimization of conditions of adsorption to efficiently remove Cr(VI) from aqueous solution

Materials and methods

Chemicals and instrumentation

Analytical grade potassium dichromate $(K_2Cr_2O_7)$ (Scharlau Chemicals, Spain) was used as the source of Cr(VI) in this research work. Coconut coir (CC) collected from the local market was treated by sodium chlorite $(80\%NaClO_2)$ (BDH Laboratory Supplier, England) for using as adsorbent for this experiment.

Single beam UV-visible recording spectrophotometer (OPTIZEN POP, Korea) was used to analyze Cr(VI). IR spectra of SCT-CC were recorded before and after adsorption using a FTIR spectrophotometer (Model: Frontier FT-NIR/MIR, Perkin Elmer, USA).

Processing of coconut coir

Fibrous coir purchased from the local market was washed with tape water for several times to remove dust and pigments and was again washed with distilled water for three times and was air-dried followed by drying in oven at 110ºC and the resulting crispy materials were grinded. This untreated coconut coir (UCC) was used for the further treatment.

Modification of coconut coir

According to the matrix designed by Box–Behnken experimental design (Liu *et al.,* 2012), the previously obtained UCC was stirred with 6.2 g of sodium chlorite in the presence of 9.5 mL of acetic acid for about 60 min at 150-200 rpm with designed reaction temperature 90°C. The treated CC was washed with distilled water until the filtrate had reached pH 6–7, then dried at 70ºC to a constant weight, screened through sieve and stored in desiccators for further use. The modified coconut coir thus obtained was termed as sodium chlorite modified coconut coir (SCM-CC).

Batch adsorption studies

About 0.3g of SCM-CC (particle size of 75-100 µm measured by using metallic sieve) was charged in reagent bottles containing Cr(VI) solution of concentration about 200 ppm (prepared from stock solution of 1000 ppm) at pH 2±0.02. These bottles were shaken in the mechanical shaker at room temperature ($\approx$ 28.0±0.5ºC). After a particular interval of time the reagent bottles were withdrawn from the shaker. The supernatant was centrifuged to obtain a clear solution. The experiment was repeated for the SCM-CC of particle size 100-200 and 200-300 m.

Analysis and characterization of surface

Chromium concentration in the solution was determined by using UV-visible spectrophotometer. The λmax of the solution was found to be within 360-370 nm at all the pH values under observation. Adsorption kinetics was investigated using three different particle sizes, 75-100, 100-200 and 200-300 µm of SCM-CC at room temperature (28.0±0.5ºC).

The surface of the SCM-CC of different particles sizes before and after adsorption of Cr(VI) was characterized by FTIR (Frontier FT-NIR/MIR (Perkin Elmer, USA)) spectroscopy in order to find out whether any shifting of IR peak due to the interaction of Cr(VI) with SCM-CC occurred or not. The scanning electron microscopic (SEM) images were recorded to investigate the morphology of the surface by using a JSM-5600LV SEM instrument (JEOL, USA) with gold film. The samples were coated with platinum to avoid charging. The elemental composition of the bioadsorbent (SCM-CC) was determined by utilizing energy dispersive spectroscopy (EDS).

Results and discussion

Characterization of surface

FTIR analysis

FTIR spectra are commonly used to confirm the presence of functional groups on the surface of bioadsorbent and thus the FTIR spectra of bare SCM-CC and Cr(VI) adsorbed SCM-CC at different particle sizes were investigated and are shown in Fig. 1. The band for the stretching vibration of –OH in cellulose of CC was found to become broader at 3439.7 cm^{-1} in the case of bare SCM-CC, which might be due to the formation of hydrogen bond after treatment. This broader band became comparatively sharper after the adsorption of Cr(VI) onto SCM-CC of different particle sizes.

However, the FTIR spectra of –OH group for Cr(VI) adsorbed SCM-CC of different particle seizes were found to be quite similar. It indicates that the chemical interaction between the SCM-CC and Cr(VI) is not affected by changing the particle size of the surface. Another band at 1612.8 cm^{-1} indicates the existence of C=C in bare SCM-CC. This band becomes more intense after the adsorption of Cr (VI) onto SCM-CC.

SEM analysis

The surface morphology of SCM-CC with different particle sizes was observed executing SEM analysis. The tube like coir was found in all the cases (Fig. 2 (a-c)). Moreover, the clear porous structure was noticed after the adsorption of Cr(VI) onto SCM-CC of different particle sizes. The porosity evolved in the coir matrix was found to differ for different
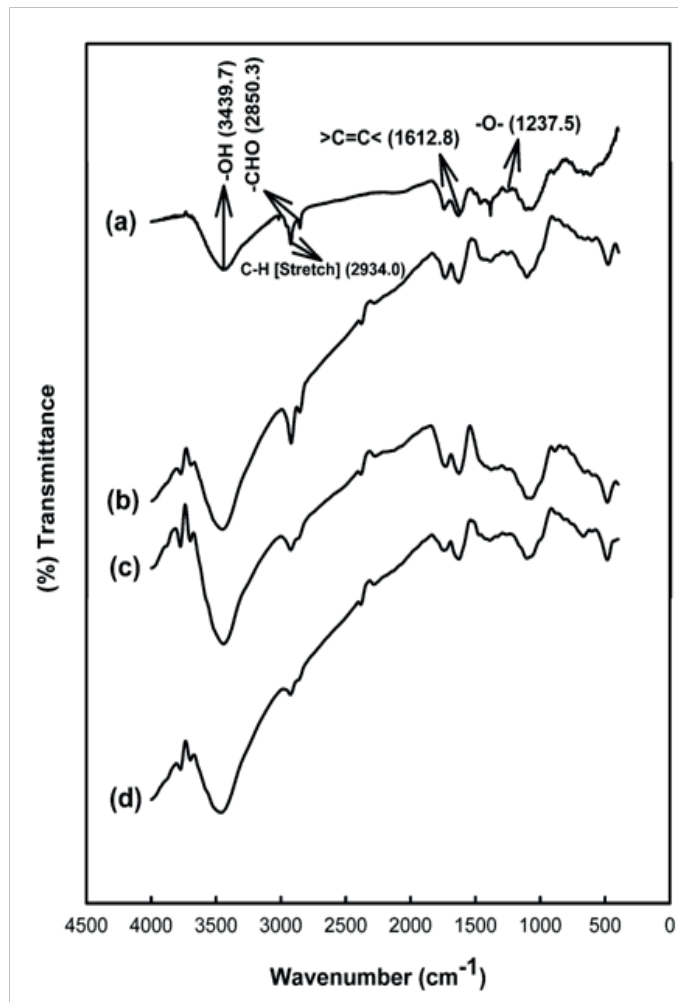

Fig. 1. FTIR spectra of (a) bare SCM-CC and Cr(VI)-adsorbed SCM-CC on (b) 75-100 µm, (c) 100-200 µm and (d) 200-300 µm of SCM-CC

particle sizes. The result showed the dissimilarity to that reported before in the case of coconut coir pith (Sheel *et al.,* 2016). Scanning electron microscopic (SEM) photographs revealed the porosity and adsorptive nature of CC.

Due to the evolution of porous site the surface of SCM-CC became more conducive to Cr (VI) to adsorb onto it rather than raw coconut coir.

Adsorption behaviour

Equilibrium time

The q_t (the amount of Cr(VI) adsorbed) vs. time curve (Fig. 3) for the adsorption of Cr(VI) on SCM-CC was found to be
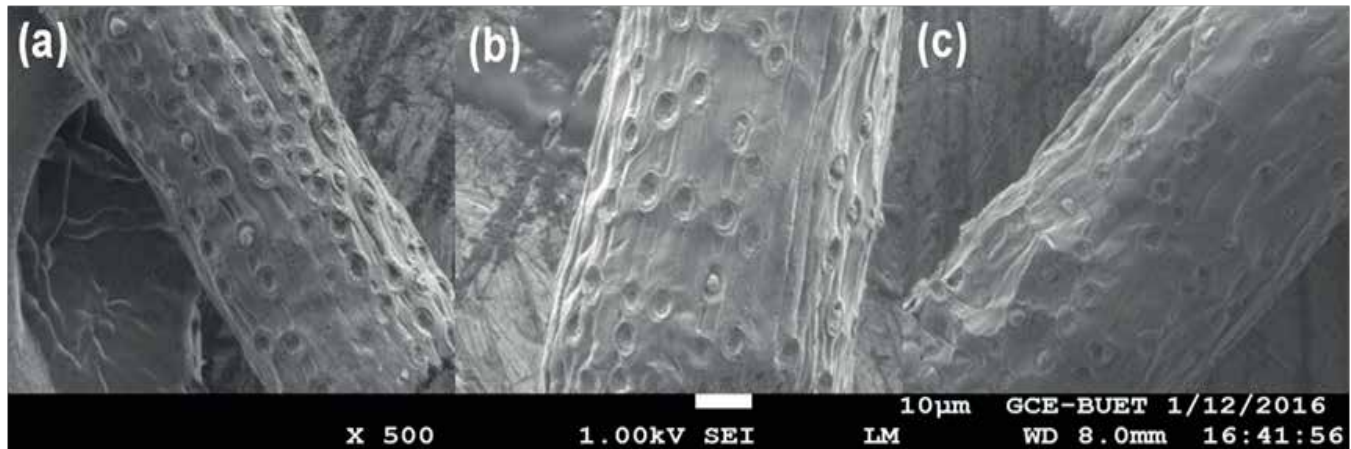
Fig. 2. Scanning electron microscopic (SEM) images for different particle sizes of SCM-CC, (a) 75-100 µm, (b) 100-200 µm and (c) 200-300 µm at 500 × magnification

similar with the usual curves. After the quick increase of the amount adsorbed at the beginning, the adsorption of Cr(VI) on SCM-CC reached the equilibrium within 3 h at smaller particle sizes (i.e., 75-100 & 100-200 µm).

However, at larger particle size (such as 200-300 ?µm) the system took about 4 h to attain the equilibrium. Two different observations may be pointed out here. In one case, the initial increase of the amount of Cr(VI) adsorbed onto SCM-CC was sharp to eventually attain the steady value. Gradual increase in the curve followed by the slow attainment of the steady value was observed in the other case.
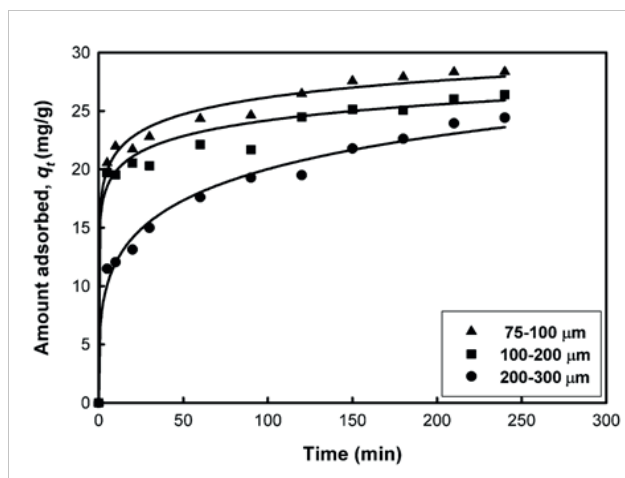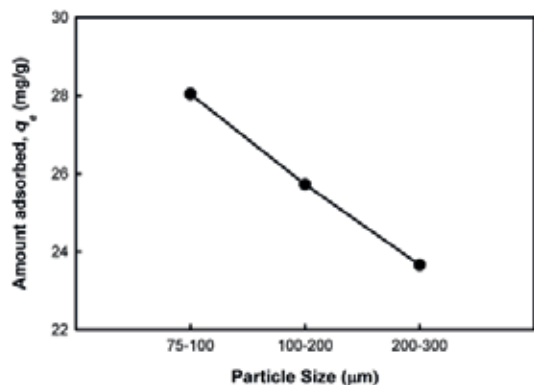

Fig. 3. Variation of the amount of Cr(VI) adsorbed on SCM-CC of different particle sizes as a function of time at pH 2.00 at room temperature (28.0±0.5ºC)

Effect of particle size

The adsorption capacity of SCM-CC against Cr(VI) was found to be influenced by the particle size of SCM-CC (Fig. 4). The amount of Cr(VI) adsorbed on SCM-CC was observed to be the highest (28.03 mg/g) for the particle size of 75-100?µm and the lowest (23.66 mg/g) for that of 200-300?µm. The percent removal of Cr(VI) from aqueous solution was found to be maximum 99.92% at lower pH by using SCM-CC of particle size 75-100 µm. The particle size is inversely correlated with the external surface area per g of adsorbent. Smaller the particle size higher will be the value of external surface area or vice versa. High value of external surface area is attributed to the high adsorption capacity of the adsorbent. Therefore, the SCM-CC of smaller size (75-100µm) exhibited the maximum removal of Cr(VI) compared with that of larger size (100-200 or 200-300 Cm). This is in a good agreement with the observation by Panda *et al.,* 2011.

Effect of pH

The pH has the pronounced effect on surface phenomena. Adsorption of heavy metal is definitely dependent on pH of the adsorbate solution. With a view to determining the suitable pH for adsorption, tests were carried out taking initial concentration (200 ppm) of potassium dichromate. In present research it was also proved experimentally that the change in pH from lower to higher (2.00 to 7.00) caused the decrease in

Fig. 4. Variation of the equilibrium amount of Cr(VI) adsorbed per gram of SCM-CC with particle size at 2.00 at room temperature (28 ± 0.5°C)
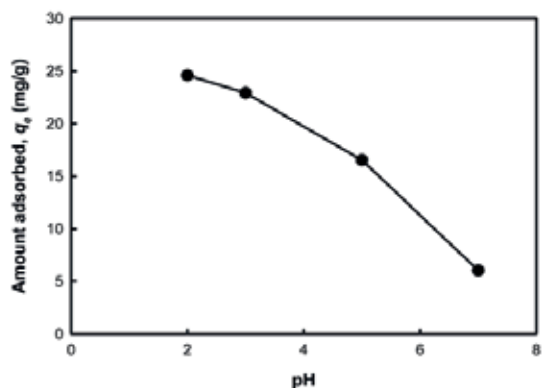
**Fig. 5. Effect of pH on Cr(VI) adsorption on SCM-CC at room temperature (28±0.5°C).**

the effective removal of Cr(VI) by adsorption. The amount adsorbed (q_e) of Cr(VI) onto SCM-CC was found to be the lowest at high pH value (pH 7.00) whereas low pH (pH 2.00) of the system exhibited the highest adsorption of chromium. The variation of equilibrium adsorbed amount (q_e) for SCM-CC with pH was shown in Fig. 5. Since dichromate is a composite anion, when the pH was increased surface become negative and because of this there was a repulsive force generated between the adsorbate and the surface. But as the pH was decreased to lower values, the surface became positively charged and negatively charged dichromate ion experienced the electrostatic force of attraction towards the surface to increase the amount of adsorbed species. This agrees well with Suksabye *et al.* (2007). The Cr(VI) adsorption efficiency of activated carbon based on coconut shell was reported to decrease with the increase of the system pH values from 2 to 10. Islam *et al.* (2016) showed the effect

of pH on the adsorption kinetics of Cr(VI) on sodium chlorite treated coconut coir. In that case the rate of adsorption was shown to decrease as the pH was tuned from lower to higher values.

Effect of initial concentration

The varying initial concentration of dichromate was found to have marked effect on the equilibrium amount of adsorbed Cr(VI). Experiments for the dye Cr (VI) were carried out by selecting a concentration range of 20-200 ppm with an adsorbent dosage of 0.3 g for SCM-CC at room temperature (28±0.5?ºC). The experimental results showed that an increase in the concentration caused an increase in the amount of the Cr(VI) adsorbed. This increase in adsorption of Cr(VI) with concentration is attributed to the retardation of resistance toward chromium uptake, which increases the diffusion of dichromate (Fig. 6). The uptake of Cr(VI) onto SCM-CC at pH 2.00 was found to increase further if the initial concentration Cr(VI) would have been increased much. On the other hand, at pH 3.00 the initial concentration of Cr(VI) was observed to have no effect on the amount adsorbed up to 100 ppm. However, the uptake of chromium at pH 3.00 started to increase after 100 ppm and remained constant at the proximity of 200 ppm.

This can be compared with the results obtained from the removal of hexavalent chromium-contaminated wastewater using activated carbon and bentonite *(*Wanees *et al.,* 2012) where the uptake of Cr^{6+} was found to increase with the initial concentration for both the adsorbents. In the present case, the relation between the amount adsorbed and the initial concentration followed the usual pattern in more acidic condition whereas the uptake of chromium was found to be
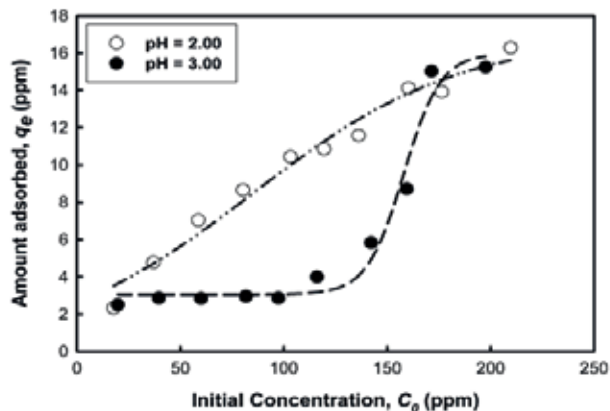
Fig. 6. Amount of Cr(VI) adsorbed on SCM-CC with varying initial concentration of dichromate at different pH values at room temperature (28±0.5°C)

independent on pH to a certain range of initial concentration of dichromate at less acidic medium. This trend of chromium uptake pattern remained same up to the concentration of 200 ppm after a quick rise in the range of 100 and 200 ppm.

EDS analysis

EDS analysis was performed to determine the elemental composition of the biosorbents after metal ion adsorption. Carbon and oxygen are the major constituents in the biosorbents. Their features can be observed at 0.277 and 0.525 keV, respectively. As the surface of CC was modified by sodium chlorite, then the features of sodium and chlorine appeared at 1.041 and 2.621 keV respectively. Fig. 11 presents the EDS spectrum of SCM-CC following Cr(VI) adsorption, as an example. As shown in Fig. 7, new peaks appeared at 5.411 keV represents the feature of Cr. Overall, the appearance of EDS feature of Cr on the surface of biosorbents entails the ability of SCM-CC for metal ions binding.

Adsorption isotherm

The adsorption isotherm refers to the plot of the amount adsorbed against equilibrium concentration at constant temperature. The most favored approach to an investigation of the adsorption mechanism is a study of the isotherm. The implication of the adsorption isotherms in adsorption mechanisms is based on few aspects such as the shape of isotherm, the significance of the plateau in many isotherms, the orientation of the adsorbed molecules. The data of the adsorption isotherms obtained for the adsorption of Cr(VI) onto SCM-CC at pH 2.00 and 3.00 at room temperature (28 ± 0.5°C) were examined whether they are fitted to the Langmuir or Freundlich isotherms to understand the adsorption behavior.
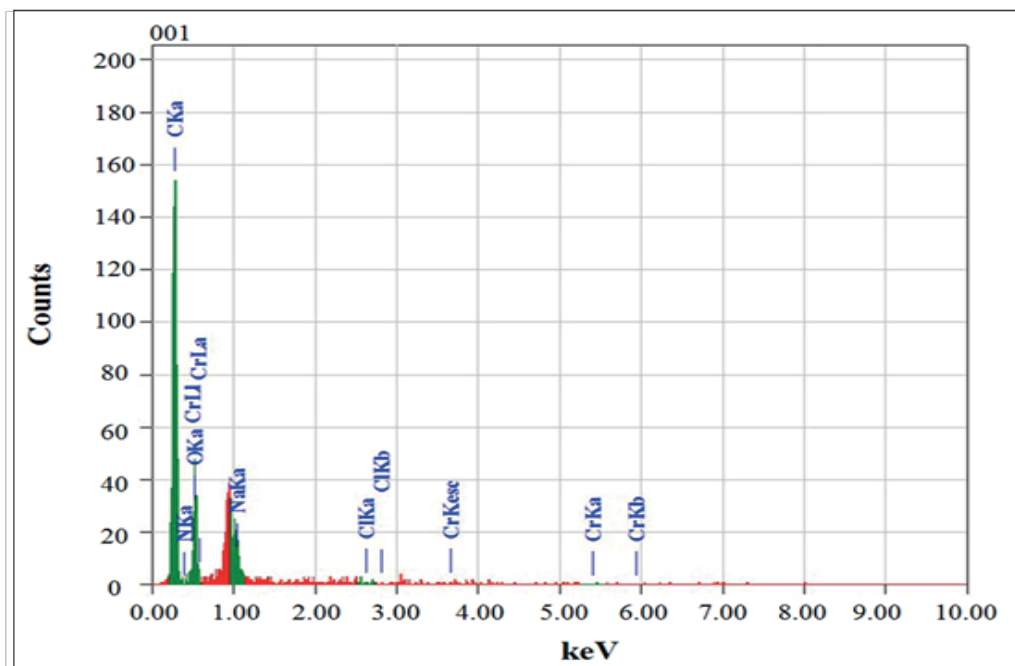

Fig. 7. Energy dispersive spectrum (EDS) of SCM-CC after interaction with Cr(VI)

Langmuir isotherm

The Langmuir Isotherm equation:

$$x/m = aC_e/(1+bC_e) \qquad (1)$$

$$\therefore C_e/x/m = 1/kK_{eq} + C_e/k$$

$$or, \quad C_e/q_e = 1/q_mK_{eq} + C_e/q_m \qquad (2)$$

Freundlich isotherm

The Freundlich model was applied to calculate the adsorption data of CV, as per the given relation:

$$x/m = K_FC_e^{1/n} \qquad (3)$$

$$or \log (x/m) = \log K_F + 1/n \log C_e \qquad (4)$$

The adsorption isotherm found experimentally was fitted relatively more precise to Freundlich than to Langmuir isotherm for the experiment carried out with SCM-CC at pH 2.00 and 3.00. In the case of pH 2.00 the value of correlation coefficient, R^2, was only 0.946 (Fig. 8) for fitting with Langmuir equation whereas the isotherm was precisely Freundlich type depicted in Fig. 8. Here, the value of correlation coefficient, R^2, was found to be 0.976 (Fig. 9) for Freundlich isotherm. On the other hand, the adsorption isotherm was neither found to fit with Langmuir nor Freundlich isotherms (Fig. 8 and Fig. 9) at pH 3.00. In this case R^2 value was found to be 0.192 (Fig. 8) and 0.194 (Fig. 9) for fitting with Langmuir and Freundlich isotherm respectively.

Adsorption kinetics

The data obtained from the estimation of equilibrium time for the adsorption of Cr(VI) on SCM-CC of different
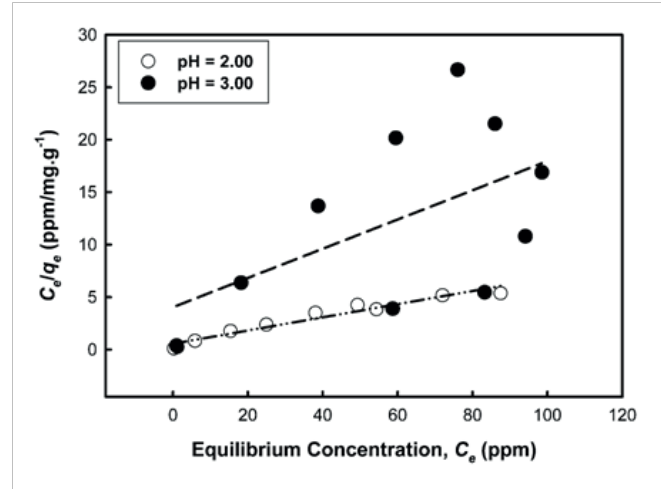
Fig. 8. log C_e/q_e vs. C_e for fitting with Langmuir isotherm for the adsorption of Cr(VI) on SCM-CC and SCT-CC at pH 2.00 (R^2= 0.946) and 3.00 (R^2= 0.192) at room temperature (28±0.5°C).
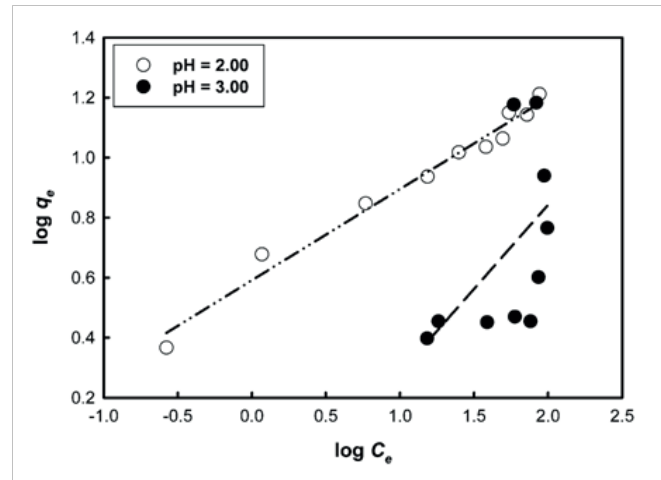
Fig. 9. log q_e vs. log C_e for fitting with Freundlich Isotherm for the adsorption of Cr(VI) on SCM-CC and SCT-CC at pH 2.00 (R^2= 0.976) and 3.00 (R^2= 0.194)

Table I. Langmuir and Freundlich coefficients for the adsorption of Cr(VI) on SCM-CC at pH 2.00 and 3.00 and at the temperature (28 ± 0.5°C)

pH	Langmuir Coefficients			Freundlich Coefficients		
	q_m (mg/g)	K_{eq} (L/mg)	R^2	K_F(L/g)	*n*	R^2
2.00	15.92	8.94	0.946	3.90	3.29	0.976
3.00	7.18	29.01	0.192	1.90	1.78	0.194

Table II. Pseudo first order and pseudo second order kinetic parameters for the adsorption of Cr(VI) onto SCM-CC of different particle sizes

Particle size	q_e (mg/g)	Pseudo -First -order kinetic model		Pseudo -Second-order kinetic model	
(?m)		k_1 (min^{-1})	R^2	k_2 (g /(mg min))	R^2
75-100	191.20	7.71 x 10^{-5}	0.926	4.66 x 10^{-2}	0.998
100-200	203.38	6.62 x 10^{-5}	0.942	4.48 x 10^{-2}	0.995
200-300	170.92	10.00 x 10^{-5}	0.942	2.06 x 10^{-2}	0.987
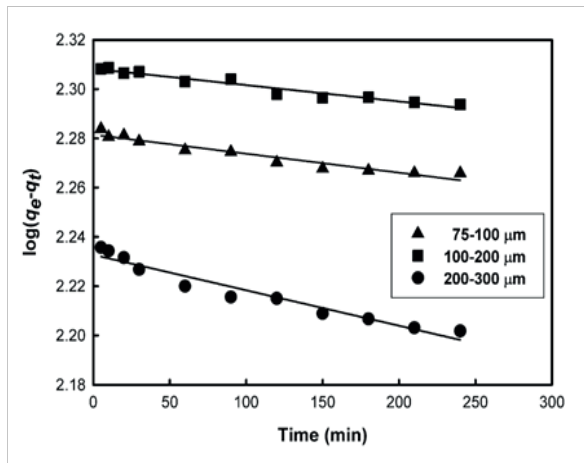
Fig. 10. log (q_e-q_t) vs. time for fitting with Lagergren equation of first order kinetics for adsorption of Cr(VI) on SCM-CC of different particle sizes, 75-100μm (R^2 = 0.926), 100-200μm (R^2 = 0.942) and 200-300μm (R^2 = 0.942) at pH 2.00.
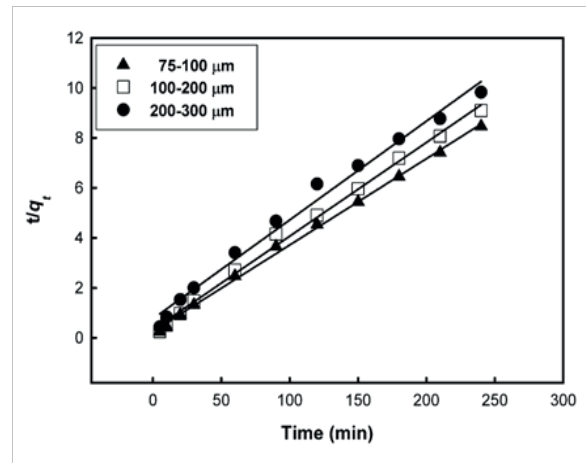
Fig. 11. t/qt vs. time for fitting with Ho's equation of pseudo second order kinetics for adsorption of Cr(VI) on SCM-CC of different particle sizes, 75-100μm (R^2 = 0.998), 100-200μm (R^2 = 0.995) and 200-300μm (R^2 = 0.987) at pH 2.00

particle sizes, 75-100?m, 100-200?m and 200-300?m were fitted in both Lagergren first order kinetic equation and Ho's pseudo-second order kinetic equation. The data for first and second order kinetics at different pH were depicted in the Fig. 10 and Fig. 11 respectively. The values of correlation coefficient, R^2, indicated in the Table 2 revealed that the adsorption kinetics of Cr(VI) onto SCM-CC mostly follows Ho's pseudo second order kinetics model for all particle sizes.

The kinetic model fitted with Lagergren first order,

$$\log(q_e\text{-}q_t) = \log q_e - k_1 t \qquad (5)$$

and Ho's pseudo-second order kinetic equation as follows:

$$t/q_t = 1/k_2 q_e^{2} + t/q_e \qquad (6)$$

In the above equation q_e and q_t denote the amounts adsorbed at equilibrium and at any time t, respectively, and k_1 and k_2 are the first order and second-order rate constants respectively.

Conclusions

Cr(VI) adsorps on SCM-CCT at pH 5.00 and 7.00 and the equilibrium is attained rapidly. The lower value of pH (2.00 and 3.00) facilitated the adsorption on

SCT-CC. The adsorption kinetics was found to be influenced by the action of the pH of the solution. The high value of the rate of adsorption was observed at pH 2.00 and 3.00, which fell drastically when the pH was increased to 5.00 and 7.00. The kinetic data indicated that the interaction between the Cr(VI) ($Cr_2O_7^{2-}$) and SCM-CC is fully electrostatic. Because of the electrostatic force of attraction between negatively charged adsorbate species and positively charged adsorbent enhanced the rate of adsorption at lower pH whereas the neutral pH showed very much weaker interaction between adsorbate and adsorbent.

Acknowledgement

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