Adsorption of Cr(VI) from Aqueous Solution onto a Mesoporous Carbonaceous Material Prepared from Naturally Occurring *Pongamia pinnata* Seeds

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Abstract

In the present study, adsorption of Cr(VI) from aqueous solution by mesoporous *Pongamia pinnata* Carbonaceous Material (PPCM) as an excellent adsorbent derived from natural source was investigated. PPCM was prepared using simple chemical activation method and Cr(VI) detection by spectrophotometrically. The synthesized mesoporous carbonaceous material was characterized by FT-IR, PXRD, SEM before and after adsorption. Batch mode experiments were used to analyze the effect of various parameters like effect of pH, initial Cr(VI) concentration, adsorbent dose, and contact time on the adsorption capacity of Cr (VI) on PPCM . It was found that, maximum Cr(VI) removal (99.8%) was observed at pH 1.0 with sorption capacity of 194.6 mg g^{-1}. The adsorption isotherms of Cr(VI) onto PPCM obtained were analyzed by Langmuir and Freundlich isotherm models and sorption data was better fitted by the Freundlich isotherm model. Adsorption kinetics were performed and obeys pseudo second order kinetic model. Experimental and kinetic results reveal that PPCM is a potential adsorbent in removal of Cr(VI) from waste water.

Introduction

Heavy metals in waste water are directly or indirectly discharged into the environment increasingly, especially in developing countries. Unlike organic contaminants, heavy metals are not biodegradable and tend to accumulate in living organisms and many heavy metal ions are known to be toxic or carcinogenic. In most cases, wastewaters often contain a lot of inorganic and organic pollutants. The major inorganic pollutants are toxic heavy metals such as Cr, Pb, Hg and Cd, which have become a serious problem in the aquatic environment due to their toxicity, bioaccumulation and persistence. And organic pollutants like phenol, polychlorinated biphenyls in water also have been causing serious environmental problems. Among these, Chromium is an essential nutrient for plant and animal metabolism. The two typical oxidative states of chromium ion in the environment are hexavalent, Cr(VI), and trivalent, Cr(III) ion, as all other oxidation states are not stable in aqueous solutions. These two oxidation states have widely contrasting toxicity and transport characteristics: hexavalent chromium is more toxic, carcinogenic and mutagenic [1,2] with high water solubility and mobility, and is a major pollutant in marine, ground, industrial and even treated wastewaters. While trivalent chromium is less soluble in water, less mobile and less harmful [3]. In contrast it is essential nutrient to normal carbohydrate, lipid and protein metabolism in the human body. Nowadays the removal of toxic contaminants from water remains a huge challenge for industrial and municipal wastewaters leading to an important problem in the field of waste water purification. Researchers have paid attention to it as it can cause sickness, even diseases such as cancer, and may prove fatal [4-6]. Chromium is used in various industrial applications, including the production of stainless steel, electroplating, metal finishing, battery manufacturing, mining, smelting, dyes and pigments, leather tanning , wood preserving, glass, ceramic paints, canning industries, electrical and electronics equipments and catalysis chemical manufacturing

due to its carcinogenic properties, thus, it causes many health problems for people (e.g., skin irritations; lung cancer; or kidney, liver, and gastric damage) [7]. Hence its concentration in drinking waters has been regulated in many countries. The World Health Organization (WHO) recommends a limit of 0.05 mg L^{-1} in drinking water (World Health Organization, 2006), value adopted by several national environmental agencies.

Conventional methods for removing Cr(VI) ions from industrial wastewater include reduction followed by chemical precipitation [8,9]. Advances in water and wastewater treatment technology need spur for the development of technologies that may be more effective and less costly. In general, a wide range of processes have been reported to eliminate the Cr(VI) from water and wastewater such as chemical precipitation, electrochemical reduction, sulphide precipitation, cementation, ion-exchange, adsorption, reverse osmosis, elecrodialysis, solvent extraction and evaporation, etc. [10]. Among the different treatments described above adsorption technology is one of the most recommended physicochemical treatment processes, where the substance called adsorbate accumulates at the interface of solid called adsorbent [11,12].

It is commonly used and applied for heavy metal ions removal from water samples and aqueous solutions. In addition, adsorption process

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is well recognized as one of the most efficient methods for removal of heavy metal ions from their matrices. Adsorption is attractive due to its merits of efficiency, economy and simple operation [13]. Activated carbon is commonly used as an adsorbent for water treatment because it has porous structure and large surface area [14,15].

The objective of the present work is to investigate the adsorption of Cr(VI) on PPCM system under visible light. The influence of pH, temperature, contact time, initial concentration, and dose of adsorbent on Cr(VI) adsorption from synthetic Cr(VI) solution were studied. Both Kinetic and equilibrium isotherm models were applied to establish the rate of adsorption, adsorption capacity and mechanism of Cr (VI) adsorption on activated carbon.

Experimental

Materials

Commercially pure potassium dichromate ($K_2Cr_2O_7$, AR 99%, Sigma-Aldrich), 1:1 sulphuric acid (H_2SO_4, AR 98%, Sigma-Aldrich), hydrochloric acid (HCl, AR 99%, Sigma- Aldrich), Sodium hydroxide (NaOH, AR 99%, Sigma-Aldrich), 1, 5 Diphenyl carbazide (1,5 DPC AR 99%, Sigma-Aldrich). All chemicals were used as such without further purification.

Preparation of PPCM adsorbent

The Pongamia seeds were collected from local market Bengaluru, India and washed with distilled water two to three times for removing dust particles and dried at 120ºC for 6 h. The dried sample was mechanically milled and sieved and then it is treated with 1:1 H_2SO_4 98% (w/w). The powder was then soaked in concentrated sulphuric acid (H_2SO_4) at 200 ºC for 24 h. Then this carbon was washed to get it acid free and its pH was checked. The carbonized adsorbents were dried and rewashed many times until its pH reaches 7. After this the activated carbon was deacidified by use of distilled water and sodium bicarbonate solution in later stages. Deacidification was done in following manner. After 25-30 times washing carbon was treated with aqueous ammonia solution in such a fashion that it does not affect the surface properties. Then carbon was dried to 80 ºC for 24 h to remove the moisture.

Preparation of adsorbate

A metal stock solution of Cr(VI) containing 1000 mg/L was prepared by dissolving 2.8289 g potassium dichromate ($K_2Cr_2O_7$) in 1000 ml de-ionized water. The metal solution was further diluted using distilled water to obtain required standard solutions in the range of 10 - 100 mg/L. The solutions were stored in a plastic sealed beaker. The pH adjustment is carried out by using 0.5 N HCl and 0.5 N NaOH solutions. Freshly prepared solutions were used for all experiments.

Chromium analysis method

For the determination of Cr(VI) in the aqueous solution, 0.25% solution of 1,5 diphenyl carbazide in 50% acetone was prepared as required. The standard solution for the calibration of spectrophotometer (Spectronic 20, Bausch and Lomb, USA) was in the range of 0.2 mgL[-1] of dichromate. To about 5 ml of this solution was added sufficient 1M H_2SO_4 to make the concentration about 0.1M, then to this solution added 3 ml of diphenyl carbazide ($C_{13}H_{14}N_4O$) reagent and it is made upto 25 ml with water. The pink-violet (diphenylcarbazone) coloured complex was formed when 1,5 diphenyl carbazide was added into Cr(VI) in acidic solution and concentration was determined spectrophotometrically (SL- 159 UV- Visible spectrophotometer, ELICO, India) at 540 nm after 20 min.

Characterization of adsorbent

The prepared PPCM adsorbent was characterized by powder X-ray diffractometer (XRD, PAN alytical X'Pert Pro. Cu Kα (1.541 Å) radiation with nickel filter is used to obtain diffraction data in predicting crystallanity and phase puity of the adsorbent material. Surface morphology was analyzed by Scanning Electron Microscopy (SEM, Model: SU-70, Hitachi, Japan). The FT-IR studies have been performed on a Perkin Elmer Spectrometer (Spectrum 1000) with KBr pellet technique in the range of 400-4000 cm[-1]. Remi centrifuge was used to separate Cr(VI) solution from adsorbent.

Results and Discussion

FT-IR spectroscopic studies

FTIR spectra analyses at wave numbers ranging from 400 to 4000 cm[−1] of the PPCM before and after adsorption process were recorded and shown in the Figure 1a and 1b. Results are indicated that there are a number of important functional groups on the surface of the adsorbents. The broad adsorption band around 3432cm[-1] (Figure 1a) could to be attributed to the O-H stretching of the adsorbed water molecules and intra-molecular hydrogen bonding and bonded to the surface of fresh adsorbent [16-18]. The bands between 2850 and 2950 cm[-1] observed in Figure 1a are due to the symmetrical and assymetrical stretching of -CH vibrations of -CH_3 and -CH_2 groups. The sharp band of carbonyl stretching(C=O) observed at 1708 cm[-1] [19,20]. Moreover, a board adsorption band at wave numbers ranging 2854 to 3000 cm[-1] (with a maximum at 2925 cm[-1]) was observed in the spectrum of fresh adsorbents, demonstrating that C-H groups are present on the surface of PPCM. Also a wide adsorption band at wave numbers ranging 1449-1708 cm[-1] may relate to the presence of phenolic groups.

In Figure 1b, there is a significant decrease in the peak intensities after adsorption, indicating the activity and role of functional groups in adsorption of Cr(VI) is a chemical adsorption process. From the discussions, it is evident that, the presence of function groups could be effective in adsorbing Cr(VI) from aqueous solution.

Powder X-ray diffraction studies

The X- ray diffraction measurements were carried out to examine the structure of the adsorbent before and after adsorption (Figure 2). Figure 2a shows the X-ray diffraction patterns of PPCM before and after Cr(VI) adsorption. It can be seen from the XRD patterns that there is significant difference in the intensity of the peaks and it is also interesting to note that there is shift in the diffraction pattern towards higher angle after adsorption. The enhancement in intensity may be due to heating of the PPCM to dry the sample and the shift in peak is attributed to the adsorption of the metal on to the surface of the adsorbent. Similar results were also reported by Xue et al. in their studies on Removal of Cu(II) from aqueous solution by adsorption onto functionalized SBA-16 mesoporous silica [21].

Morphological analysis

Scanning electron micrographs (SEM) of the PPCM before and after adsorption process are shown in Figure 3a and 3b respectively. It is clear from Figure 3a that the SEM figures that the PPCM surfaces were rough, mesoporous and contained pores of different size. After the adsorption (Figure 2b) the pores packed with Cr(VI) ions. The micrographs signified that the Cr(VI) ions had densely and homogeneously adhere to the surface of PPCM adsorbent due to the physical adsorption by electrostatic force or due to the covalent binding between Cr(VI) ions and adsorbent. The biosorbent materials prepared
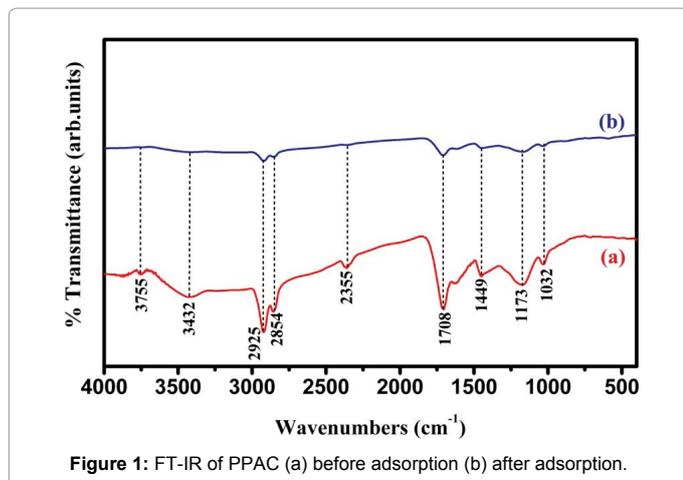
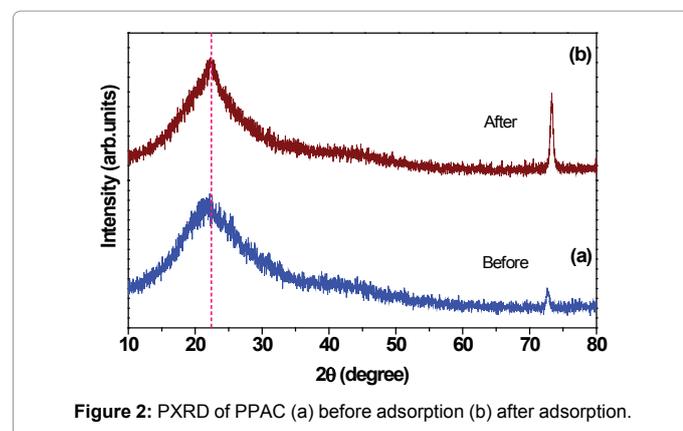**Figure 1:** FT-IR of PPAC (a) before adsorption (b) after adsorption.

Figure 2: PXRD of PPAC (a) before adsorption (b) after adsorption.
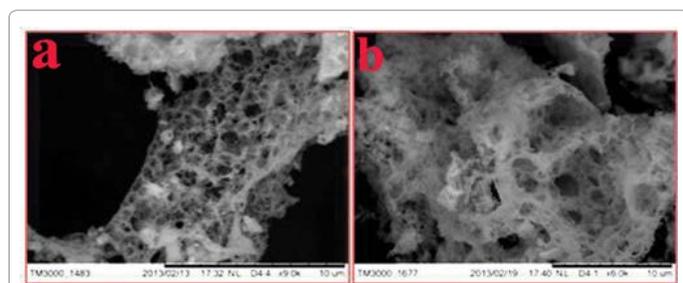

Figure 3: SEM micrographs of PPAC (a) before adsorption (b) after adsorption.

from agricultural wastes possess insoluble organic compounds and poly functional groups which interact with Cr(VI) ions with electrostatic force of attraction. Similar results were reported by Ilyas et al. [22] for the removal of Cr(VI) from aqueous solutions by peanut shell as adsorbent.

Batch Experiments

$$\% \text{Adsorption} = \frac{C_i - C_e}{C_i} \times 100 \qquad (1)$$

Where, C_i and C_e (mg/L) are the initial and equilibrium concentrations respectively.

The concentration of free chromium ions in the stock solution and unabsorbed Cr(VI) in the reaction medium was determined spectrophotometrically. The amount of Cr(VI) adsorbed by the PPCM and percentage removal of Cr (VI) are calculated using the following equation,

$$Q = \frac{C_o - C_e}{w} \times v \qquad (2)$$

Where, V is the volume of Cr(VI) sample solution (ml), W is the amount of adsorbent (g), Co and Ce (mg/L) are the initial and equilibrium metal concentrations respectively.

Effect of contact time

The adsorption of Cr(VI) onto PPCM was also studied as a function of contact time in order to find out the equilibrium time for maximum adsorption. For initial concentration of 10, 25 ppm, the uptake of Cr(VI) is very fast in the beginning and the equilibrium time were reached by in 60 minutes (Figure 4). For higher concentration equilibrium times were more than 150 minutes. The Cr(VI) ions adsorbed initially on the external surface area of PPCM which made the adsorption rate very fast. When the adsorption of the exterior surface reached saturation, the Cr(VI) ions entered into the pores of porous material and were adsorbed by the interior surface of the particles. This phenomenon took relatively less contact time. Similar phenomenon was reported in the literature for the adsorption of basic dye (methylene blue) on activated carbon prepared from rattan sawdust [23].

Effect of adsorbent dose

The effect of Cr(VI) adsorption on PPCM was investigated in the range of 0.1-0.5 g/l and the results were shown in Figure 5. The adsorption concentration increased by increasing the concentration of PPCM adsorbent from 0.1-0.5 g/l. Increase in adsorption with increase in adsorbent dose could be attributed to increased surface area and availability of mare adsorption sites [24]. Finally, the adsorbent dose of 0.5 g/l was chosen for further adsorption studies.

Effect of solution pH

The effect of initial pH on the adsorption behavior Cr(VI) on PPCM was examined in the pH range of 1.0 to 11.0 by using either by 0.1M HCl and 0.1M NaOH. The characteristic adsorption of metal ions at various pH values in the range was examined. The pH of the Cr(VI) plays an important role in the whole adsorption process and particularly on the adsorption capacity, influencing not only the surface charge of the adsorbent, the degree of ionization of the material present in the solution and the dissociation of functional groups on the active sites of the adsorbent [25]. The adsorption of Cr(VI) metal ion is highly pH dependent and the effect of solution pH on the amount of Cr(VI) adsorbed was studied by varying the initial pH under constant process parameters at equilibrium conditions. From Figure 6 it was observed that the adsorption was strongly on pH dependent. Experiments were performed by varying pH from 1 to 11 at room temperature and initial concentration of 10 mgL^{-1} and the agitation time was 60 min. The maximum removal efficiency (99.8%) of PPCM was found at pH 1 and at higher pH values the removal efficiency decreases gradually. The result is in good agreement with the previous report [26] This can be explained that since Cr(VI) exists in the form of $Cr_2O_7^{2-}$, $HCrO_4^-$, CrO_4^{2-}, $Cr_4O_{13}^{2-}$, $Cr_3O_{10}^{2-}$ etc. in acidic conditions, among these the predominant Cr(VI) species is $HCrO^{4-}$ which is favorable adsorbed since it has low adsorption free energy [27] and lowering of pH caused the surface of the adsorbent to become protonated to a higher extent and as a result of a strong attraction exists between these oxyanions of Cr(VI) and positively charged surface of the adsorbent. Hence uptake increases with decrease in the pH of the solution. Whereas at higher pH sorbent surface was negatively charged and there will be an abundance of negatively charged hydroxyl ions in aqueous solution. Both these factors can result in the decrease in uptake of Cr(VI).

Effect of initial concentration of Cr(VI)

The adsorption of Cr(VI) ions was carried out at different initial chromium ion concentrations ranging from 10 to 100 mgL^{-1}, at pH 1.0, at 250 rpm with 60 min contact time using PPCM. The results were shown in Figure 7, describing that the adsorption capacity increased with increase in initial Cr(VI) concentration.. A higher initial concentration provides an important driving force to overcome all mass transfer resistances of the pollutant between the aqueous and solid phases [28], thus increase the uptake of the Cr(VI) also increased with increasing the initial metal concentration tending to saturation at higher metal concentrations. As shown in Figure 7, for the initial Cr(VI) concentration of 10, 25, 50, 75 and 100 mgL^{-1}, the uptake capacity of PPCM could be 24, 63.8, 132.4, 205.9 and 278 mgg^{-1} respectively with corresponding saturation contact time.

Adsorption Isotherm Models

The adsorption isotherm indicates how adsorbate molecules partitioned between the adsorbent and liquid phase at equilibrium as a function of adsorbate concentration. In this study, equilibrium studies were carried out to understand the behavior of the adsorbent of Cr(VI) onto PPCM at equilibrium conditions by considering Langmuir and Freundlich adsorption isotherm models.

Langmuir isotherm model

The Langmuir equation is valid for monolayer adsorption onto a surface with a finite number of identical sites [29]. The Langmuir equation is applicable to homogeneous sorption where the sorption of each sorbate molecule on to the surface has equal sorption activation energy

The linear form of Langmuir isotherm equation is given as:

$$\frac{c_e}{q_e} = \frac{1}{k_L \times q\max} + \frac{c_e}{q\max} \tag{3}$$

Where q_e is the amount of chromium adsorbed per unit weight of adsorbent (mg/g), C_e the equilibrium concentration of chromium in the bulk solution (mg/l), q_{max} is the monolayer adsorption capacity (mg/g) and K_L is the Langmuir equilibrium constant (l/mg) related to the adsorption. Both are determined from a plot of C_e/q_e versus C_e (Figure 8)

The essential characteristics of a Langmuir isotherm can be expressed in terms of a dimensionless constant separation factor or equilibrium parameter, R_L [30], which is defined by the following equation:

$$R_L = \frac{1}{1 + (q\max \times K_L)C_o} \tag{4}$$

Where C_0 is the highest initial concentration and The value of separation factor, R_L indicates the isotherm type and nature of the adsorption process. Considering the R_L value, adsorption can be unfavorable ($R_L > 0$); linear ($R_L = 1$); favorable ($0 < R_L < 1$) or irreversible ($R_L = 0$) [31]. Therefore, as the R_L value closer to zero, the adsorption will be done better. In our experimental study, the R_L value 0.247 (Table 1) unfavourable adsorption, hence non homogeneous sorption.

Freundlich isotherm model

The Freundlich isotherm is purely empirical equation and it best describes adsorption onto a heterogeneous surface [32]. The linear form of the Freundlich equation is given as:

$$Inq_e = \ln K_F + \frac{1}{n}\ln C_e \tag{5}$$

Where q_e is the amount of chromium adsorbed at equilibrium (mol/g), C_e the equilibrium concentration of adsorbate solution (mol/l), K_F the Freundlich constant indicative of the relative adsorption capacity of the adsorbent and n is the Freundlich exponent, that represents how favorable is the adsorption process. The Freundlich exponent, n, should have values lying in the range of 1-10 for classification as favorable adsorption [33]. These parameters are determined from a plot lnq$_e$ versus lnCe (Figure 9). Thus the constants can be determined as shown in Figure 9 and Table 2. Values of KF and n were determined
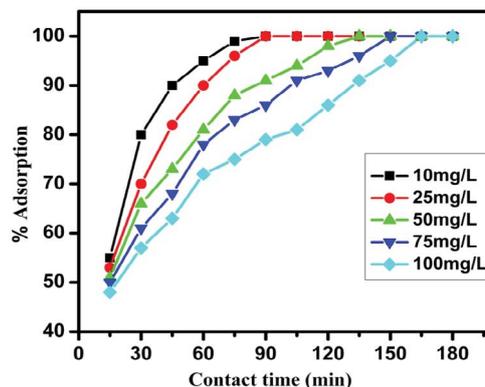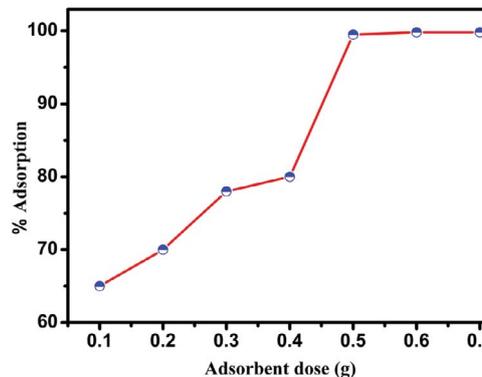

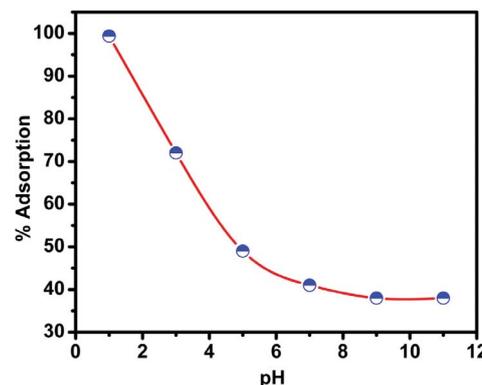**Figure 4:** Effect of contact time.

Figure 5: Effect of dosage.
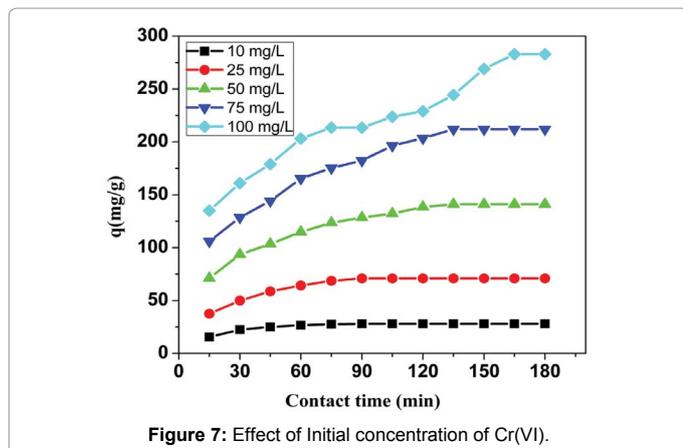

Figure 6: Effect of pH.

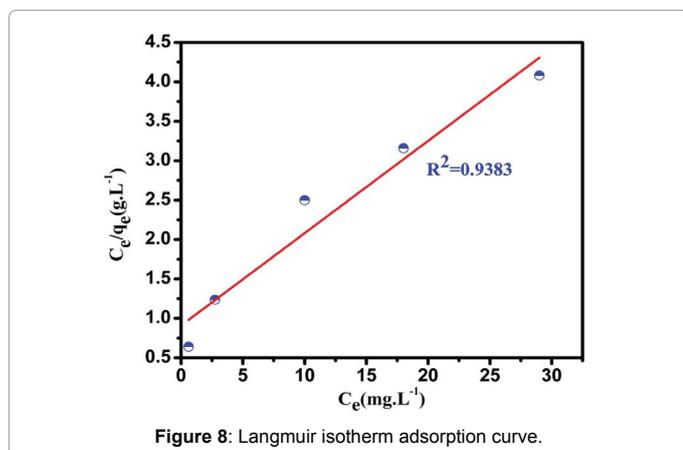**Figure 7:** Effect of Initial concentration of Cr(VI).

$R^2 = 0.9383$

Figure 8: Langmuir isotherm adsorption curve.

as 4.3732 and 1.58 respectively at 27°C with R^2 of 0.9976. The value of *n* greater than unity and R^2 value of the Freundlich plot is higher than the Langmuir, suggesting that the equilibrium adsorption of Cr(VI) onto PPCM could be best described with the Freundlich isotherm, because the correlation between calculated and experimental values as well as regression factors is in good agreement with Freundlich isotherm indicated that Cr(VI) was favorably adsorbed by PPCM.

Adsorption Kinetics Studies

A kinetic study of adsorption is very much necessary as it provides the information about the mechanism of adsorption, which is practical decisive for adsorption and gives important information for designing and modeling the processes. The most common kinetic models for adsorption are pseudo first order and pseudo second order models [34-36]. The kinetic parameter, which is helpful for the prediction of adsorption rate. Thus, the effects of initial concentration, contact time, and adsorbent dosage were analyzed from the kinetic point of view. Preliminary studies on the adsorption rate show that the amount adsorbed increased with increased Cr(VI) concentration. The maximum amount of Cr(VI) ions was adsorbed within the first 60 min (70-90% of total metal ions adsorbed) and thereafter the adsorption proceeds at a slower rate until equilibrium reached. The equilibrium time was found to be at 90, 135, 150 and 165 min for the corresponding initial Cr(VI) of 10, 25, 50, 75 and 100 ppm concentration range studied. The rate of uptake increases as the PPCM dosage increases because it leads to more adsorption sites for adsorption.

All kinetic data for the adsorption of Cr(VI) ions onto PPCM,

calculated from the related plots, are summarized in Table 2. The validity of the exploited models is verified by the correlation coefficient, r^2. Comparison of the r^2 values for different models suggests that the pseudo-second-order kinetic model fits best since its highest value ($r^2 = 0.990$). Pseudo-second-order kinetic model implies that the predominant process here is chemisorption, which involves a sharing of electrons between the adsorbate and the surface of the adsorbent. Chemisorption is usually restricted to just one layer of molecules on the surface, although it may be followed by additional layers of physically adsorbed molecules [36]. In our case, two different kinetic models were applied in order to establish the best fit with experimentally obtained data.

Pseudo-first order kinetic model

The pseudo-first order kinetic model is frequently used in kinetic studies [36]. It is described by the following equation (Equation 6)

$$\frac{dqt}{dt} = k_1(q_e - q_t) \tag{6}$$

Where q_e and q_t are the amounts (mg/g) of adsorbate at equilibrium and at time *t* (min) respectively; and k_1 is the rate constant (1/min). Integration of Equation 6 at the boundary, $q_t = 0$ at t=0 and $q_t = q_t$ at *t=t*, gives (Equation 6).

$$\log(1 - \frac{q_t}{q_e}) = -(\frac{k_1}{2.303})t \tag{7}$$

The term k_1 (min^{-1}) is the first order adsorption rate constant, q_e the amount of metal adsorbed at equilibrium and qt is the amount adsorbed at time 't'. The first order equation did not apply throughout all the contact times in this work. It was applicable over the initial 15-45 min sorption period. Plot of log ($q_e - q_t$) versus "time" at different adsorbate concentrations (Figure 10) deviated considerably from the data after a short period. The calculated slopes and intercepts from the plots were used to determine the rate constant k_1 and equilibrium capacity (q_e). The values of k, q_e and regression co-efficient provided in Table 2.

Pseudo-second order kinetic model

At equilibrium, the amount of metal adsorbed on the adsorbent surface is greatly influenced by pseudo second order reaction. The rate is directly proportional to the number of active surface sites. The Pseudo-second order kinetic rate expression can be written as,

$$\frac{dqt}{dt} = k_2(q_e - q_t)^2 \tag{8}$$

Where k_2 is the rate constant (g/mg min). Integration of Equation 6 at the boundary, $q_t = 0$ at *t=0* and $q_t = q_t$ at *t=t* and then rearrangement to a linear form gives (Equation 4).

$$\frac{1}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}t \tag{9}$$

The value of k_2 and q_e for the pseudo second order adsorption and can be obtained from the slope and the intercept of the plot t/q_t v/s t, respectively. The validity of exploited models is verified in comparison of r^2, correlation coefficient values of different models suggests that, the pseudo second order kinetic model fits best since its highest value ($r^2 = 0.990$). This implies that chemisorption is predominating process which involves sharing of electron between adsorbate and adsorbent surface (Figure 11 and Table 2).

Conclusions

In this study, the naturally occurring low cost carbonaceous mesoporous material was prepared by a simple chemical activation

Langmuir model				Freundlich model		
q_m (mg/g)	K_L (L/mg)	R^2	R_L	K_F (mg/g)	$1/n$	R^2
169.49	0.0098	0.9383	0.247	4.3732	0.63	0.9976

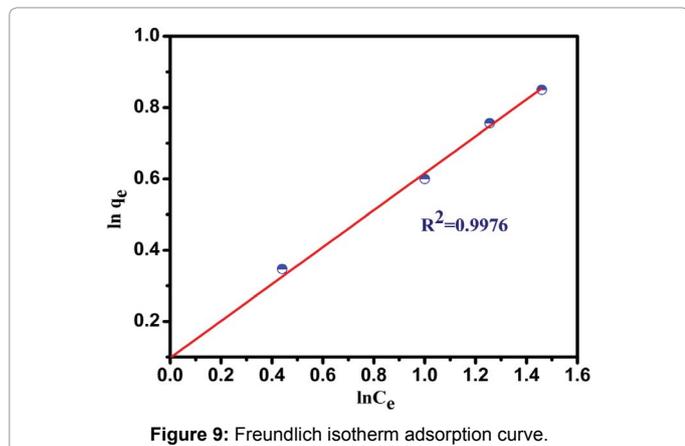**Table 1:** Indication of the isotherm type and nature of the adsorption process.

Figure 9: Freundlich isotherm adsorption curve.

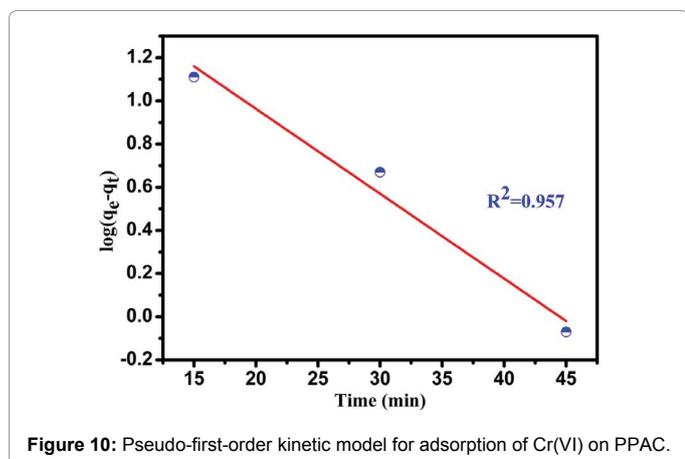| Pseudo first order kinetic model			Pseudo second order kinetic model		
q_e (mg g^{-1})	K_1 (min^{-1})	R^2	q_e (mg g^{-1})	K_2 (min^{-1})	R^2
52.49	1.19×10^{-2}	0.957	181.82	5.89×10^{-4}	0.990

Table 2: Determination of kinetic models.

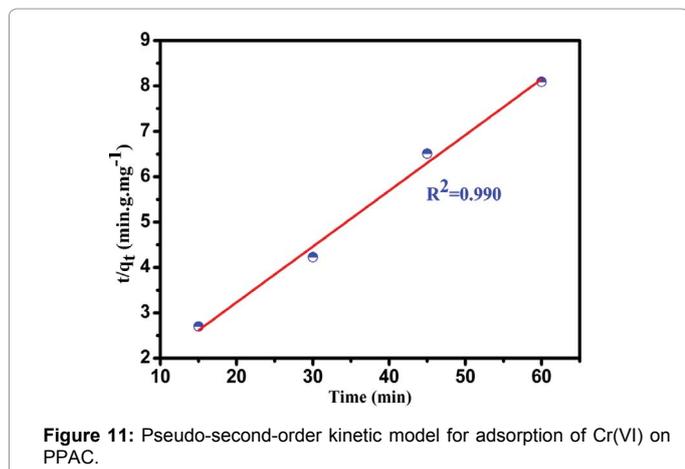**Figure 10:** Pseudo-first-order kinetic model for adsorption of Cr(VI) on PPAC.

Figure 11: Pseudo-second-order kinetic model for adsorption of Cr(VI) on PPAC.

method. The Cr(VI) ion adsorption activities were studied onto mesoporous carbonaceous material with various batch adsorption techniques with various experimental conditions. The proposed approach was verifying that, the uptake capacity of Cr(VI) was decreased with increase in pH and get saturated with increase in metal ion concentration. The Freundlich isotherm model is obeyed well to adsorption behavior of Cr(VI) ion than Langmuir model, suggesting that multilayer adsorption on to surface of PPCM was occurred. In addition to this, the kinetic data is fitted well with a pseudo-second order reaction model, suggesting that the uptake of Cr(VI) is mainly performing through chemisorption process. It is anticipated from our study, PPCM is expected to be an promising, highly efficient, cost effective and easily viable adsorbent for Cr(VI) from aqueous solution.

Acknowledgements

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