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Research Article

Adsorption of Chromium (VI) Ions by Riverbed Sand from Mullai Periyar, Tamilnadu

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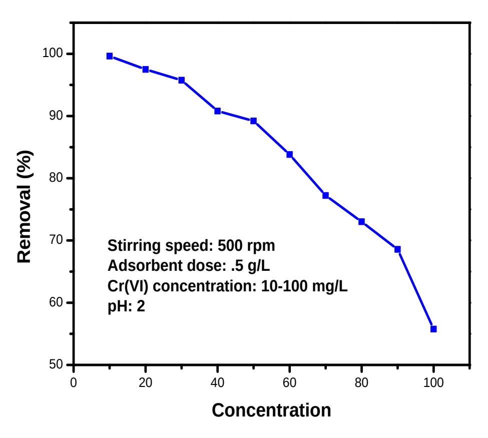
Abstract

In this study, removal of chromium (VI) ions from aqueous solutions are investigated using riverbed sand from mullai periyar river basin near Gudalur, Theni district, Tamilnadu. During the removal process, batch technique is used, and the effect of pH, Cr (VI) ions concentration, adsorbent dosage, agitation time and agitation speed on adsorption efficiency are studied. Langmuir, Frendulich, Tempkin and Dubinin-Radushkevich (D-R) isotherms are applied in order to determine the efficiency of riverbed sand used as an adsorbent. Kinetic studies showed that the adsorption followed pseudo second order reaction. Studies revealed that intra-particle diffusion played important role in the mechanism of Cr (VI) ions adsorption. The results obtained in this study illustrate that riverbed sand is expected to be an effective and economically viable adsorbent for hexavalent chromium removal from industrial waste water.

Keywords: Adsorption, equilibrium, kinetics and chromium (VI) ions



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Introduction

The presence of heavy metals in drinking water sources and in edible agricultural crops can be harmful to human. It is well known that heavy metals can be toxic that is they damage nerves, liver and bones also they block functional groups of vital enzymes [1]. Heavy metals are found in water, air and soil. The major sources of heavy metals in water and soil are waste water streams from many industrial processes [2]. Heavy metals like chromium, copper, lead, zinc, mercury, cadmium etc. are present in waste water from several industries such as metal cleaning and plating baths, refineries, paper and pulp, tanning, dyes and pigments, wood preserving, glass, ceramic paints, catalysis chemical manufacturing etc. in which chromium (VI) is present from 5 to 220 mg/dm^3 which leave into environment [3-5]. In aqueous phase chromium mostly exists in two oxidation states, namely, trivalent chromium [Cr^{+3} and $Cr(OH)^{2+}$] and hexavalent chromium ($HCrO^{4-}$, CrO_4^{2-}or $Cr_2O_7^{2-}$, etc). Most of the hexavalent compounds are toxic, carcinogenic and mutagenic. For example it was reported that $Cr_2O_7^{2-}$ can cause lung cancer [6, 7].

Chromium (III) and Chromium (VI) have major environmental significance because of their stability in the natural environment. Hexavalent Chromium [Cr (VI)] is known to have 100 fold more toxicity than trivalent Chromium [Cr (III)] because of its high water solubility and mobility as well as easy reduction [8]. International agency for research on cancer has determined that Cr (VI) is carcinogenic to humans. The toxicological effect of Cr (VI) originates due to oxidizing nature as well as the formation of free radicals during the reduction of Cr (VI) to Cr (III) occurring inside the cell [9]. Therefore, the World Health Organization (WHO) recommends that the toxic limits of Cr (VI) in waste water at the level of 0.05 mg/L, while total Chromium containing Cr (III), Cr (VI) and other species of chromium is regulated to be discharged below 2 mg/L [10].

Several methods utilized to remove Cr (VI) from aqueous solutions/wastewater include: reduction followed by electrochemical precipitation, chemical precipitation, chemical oxidation– reduction, ultrafiltration, ion exchange, reverse osmosis, solvent extraction, electrodialysis, electrochemical, coagulation, evaporation and adsorption [11]. Most of these methods suffer from drawbacks such as high capital and operational costs and problems in the disposal of the residual metal sludges [12]. The high cost of activated carbon sometimes limits its applicability for heavy metal removal [13]. Therefore, the interest of researchers is increasing using alternative materials, which are quite low cost, easily available and extremely effective adsorbents.

Natural materials that are available in large quantities, or certain waste products from industrial or agricultural operations, may have potential as inexpensive sorbents. Due to their low cost, after these materials have been expended, they can be disposed of without expensive regeneration. Most of the low cost sorbents have the limitation of low sportive capacity and thereby for the same degree of treatment, which poses disposal problems. Therefore, there is need to explore low cost adsorbent having high contaminant sorption capacity [14]. Consequently numerous low cost alternatives have been studied including Beech sawdust [15], eucalyptus bark [16], green algae [17], seaweeds [18], coir pitch [19], peanut husks carbon [20], Zeolite tuff [21], activated carbon fabric cloth [22], bagasse fly ash [23], activated slag [24] , sand [25, 26] etc. New economical, easily available and highly effective adsorbents are still needed. Riverbed sand has the potential to sequester heavy metal ions from solutions. Abundant availability, high sorption capacity, cost-effectiveness, high cation exchange capacity and renewability are the important factors making these materials as economical alternatives for water treatment and waste remediation.

In this study, low cost locally available riverbed sand was studied for its potential use as an adsorbent for removal of Cr (VI) ions from aqueous solution. Factors affecting adsorption, such as initial Cr (VI) ions concentration, pH, contact time and adsorbent dose and agitation speed were evaluated. The equilibrium of adsorption was modeled by using the Langmuir, Freundlich, Tempkin and Dubinin-Radushkevich isotherms models. The kinetic parameters and intraparticle diffusion were determined for the Cr (VI) ions with riverbed sand waste system. Results of this study will be useful for future scale up using this riverbed sand material as a low cost adsorbent for the removal of heavy metal ions from its aqueous solution.

Materials and Methods

Adsorbent collection and preparation

The riverbed sand was collected from Mullai Periyar near Gudalur, Theni District, Tamilnadu, India. The soil samples were initially sun dried for 7 days followed by drying in hot air oven at 383±1 K for 2 days. The dried soil was crushed and sieved to give a fraction of 150 mesh screen and then stored in sterile, closed glass bottles till further investigation [25].

Adsorbate preparation

Stock solution (100 mg/L) of Cr (VI) was prepared by dissolving $K_2Cr_2O_7$ in double distilled water. The solution was further diluted to the required concentration before use. Before mixing the adsorbent, the pH of each Cr (VI) ions solution was adjusted to the required value by 0.1 M NaOH or 0.1 M HCl solution.

Batch adsorption studies

Batch adsorption experiments were carried out in round bottom flasks with 200 mL of working volume, with a concentration of 10 mg/L. A weighed amount (0.5 g) of adsorbent was added to the solution. The flasks were agitated at a constant speed of 500 rpm for 60 minute in a magnetic stirrer at 303 K. The influence of pH (2.0–8.0), initial Cr (VI) ions concentration (10, 20, 30, 40, 50,60,70, 90, 100 mg/L), contact time (15, 30, 45, 60, 75, 90, 105, 120, 135 min), adsorbent dose (0.1, 0.15, 0.2, 0.3, 0.4, 0.5, 0.75, 1, 1.25 g) and agitation speed (100, 200,300,400, 500, 600, 700, 800 rpm) were evaluated during the present study. Samples were collected from the flasks at predetermined time intervals for analyzing the residual chromium concentration in the solution. The residual amount of chromium in each flask was investigated using UV-Vis spectrophotometer. The percentage of removal and quantity adsorbed, q_e (mg/g) was calculated from the following equation:

$$R(\%) = \frac{(C_0 - C_t)}{C_0} \times 100 \qquad (1)$$

$$q_e = \frac{(C_0 - C_e)V}{m} \qquad (2)$$

Where, C_0 and C_e are initial and equilibrium concentrations of Cr (VI) ions in mg/L respectively. V is the volume of the solution and m the weight of the adsorbent used.

The effect various operational parameters such as of initial concentration of Cr (VI) ions, contact time, initial pH, adsorbent dosage and agitation speed was investigated by varying any one of the parameters and keeping other parameters constant.

Adsorption isotherms

An adsorption isotherm study represents the equilibrium to determine the efficacy of adsorption relationship between the adsorbate and concentration in the liquid phase and that on the adsorbates surface at a given conditions. A number isotherm has been developed to describe equilibrium relationships. In this study, Langmuir, Freundlich and Temkin and Dubinin-Radushkevich models were used to determine the adsorption equilibrium between the adsorbent and metal ions.

Freundlich Isotherm

The Freundlich expression [27] is an empirical equation based on multilayer sorption to a heterogeneous surface and is expressed by the following equation:

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \qquad (3)$$

Where, q_e and C_e are the amount of adsorbed adsorbate per unit weight of adsorbent and unadsorbed adsorbate concentration in solution at equilibrium, respectively. K_F and 1/n are Freundlich constant characteristics of the system, which are determined from the log q_e vs.log Ce

Langmuir adsorption:

Langmuir monolayer adsorption isotherm is very useful for predicting adsorption capacities and also interpreting into mass transfer relationship. The isotherm can be written as follows:

$$\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{C_e}{q_m} \qquad (4)$$

The constant K_L (L/g) is the Langmuir equilibrium constant, and the K_L/a_L gives the theoretical monolayer saturation capacity, Q_0. These Langmuir parameters were obtained from the linear correlations between the values of C_e/q_e and C_e. Generally, the Langmuir equation applies to the cases of adsorption on completely homogeneous surfaces where interactions between adsorbed molecules are negligible [28].

Dubinin-Raduskevich Isotherm

This isotherm can be used to describe adsorption on both homogenous and heterogeneous surfaces. The Dubinin–Radushkevich equation has the following form:

$$\ln q_e = \ln q_m - \beta\varepsilon^2 \tag{5}$$

Where, q_m is the Dubinin–Radushkevich monolayer capacity (mmol/g), β a constant related to sorption energy, and ε is the Polanyi potential which is related to the equilibrium concentration as follows:

$$\varepsilon = RT\ln(1+\frac{1}{C_e}) \tag{6}$$

Where, R is the gas constant (8.314 J/mol K) and T is the absolute temperature. The constant b gives the mean free energy, E, of adsorption per molecule of the adsorbate when it is transferred tothe surface of the solid from infinity in the solution and can be computed using the relationship:

$$\mathrm{E} = \frac{1}{\sqrt{2\beta}} \tag{7}$$

The magnitude of E is useful for estimating the mechanism of the adsorption reaction. In the case of E < 8 kJ/mol, physical forces may affect the adsorption. If E is in the range of 8–16 kJ/mol, adsorption is governed by ion exchange mechanism, while for the values of E >16 kJ/mol, adsorption may be dominated by particle diffusion [28].

Temkin Isotherm

This isotherm describes the behavior of adsorption systems on heterogeneous surfaces, and it has generally been applied in the following form:

$$q_e = B\ln A + B\ln C_e \tag{8}$$

Where, RT/b =B, the adsorption data can be analyzed according to the above equation. Therefore, a plot of q_e versus ln C_e enables to determine the constants A and B [29].

Adsorption kinetics

The rate and mechanism of adsorption depends upon various factors like physical and chemical properties of adsorbents as well as mass transfer process. In order to investigate the adsorption of Cr (VI) ions on the surface of riverbed, different kinetic models are proposed to examine the controlling mechanism of adsorption process. In this study pseudo-second-order kinetic model and intra-particle-diffusion are investigated to find the best fitted model for the experimental data.

Pseudo first-order equation: The pseudo first-order equation of Lagergren [30] is given as follows:

$$log(q_e - q_t) = log(q_e) - \frac{k_1}{2.303}t \tag{9}$$

Where q_t and q_e are the amounts of Cr (VI) ions adsorbed at time t and equilibrium (mg/g), respectively, and k_1 is the pseudo first-order rate constant for the adsorption process (1/min). The linear graph of $\ln(q_e - q_t)$ vs. t shows the applicability of first order kinetic.

Pseudo second-order equation: This chemisorption kinetic rate equation is expressed as follows:

$$\left(\frac{t}{q_t}\right) = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}(t) \tag{10}$$

Where, k_2 is the equilibrium rate constant of pseudo second order equation (g/mg min). The linearity of t /q_t vs t suggests the best fitted with pseudo-second order kinetic [31].

Intra-particle equation: Kinetic data can also be analyzed by an intra-particle diffusion kinetic model [32], formulated as follows:

$$R = k_{id}(t)^a \tag{11}$$

A linearised form of the equation is followed by

$$\log R = \log k_{id} + a\log(t) \tag{12}$$

Where, R is the percent Cr (VI) ions adsorbed
t is the contact time (h)
a is the gradient of linear plots
k_{id} is the intraparticle diffusion rate constant (h^{-1})
a depicts the adsorption mechanism
k_{id} may be taken as a rate factor, i.e., per cent Cr(VI) adsorbed per unit time.

Results and discussion

Effect of adsorbent dosage

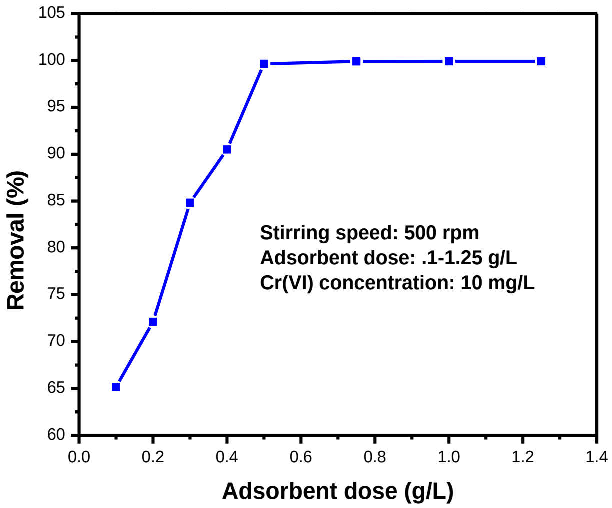

Figure 1 Effect of adsorbent dosage on Cr (VI) removal

The effect of the adsorbent dose on the adsorption of Cr (VI) ions was studied and the relationship between the percentage removal of Cr (VI) ions and amount of riverbed sand is shown in **figure 1.** It can be seen that as the amount of the riverbed sand increases from 0.1 to 1.25 g/L the percentage of adsorption increase from 0.1 to 0.5 g/L. Further increases of adsorbent amount beyond 0.5 g/L do not affect the adsorption significantly this observation can be explained in terms of availability of active sites on the adsorbent surface. The increasing trend in adsorption percentage as adsorbent dosage increases might be attributed to the increases of surface area and presence of additional availability sites [33]. The adsorbent dosage was inversely proportional to the adsorption capacity of riverbed sand this is because increase in adsorbent dosage might have led to the overlapping of surface area due to the restricted area made available and therefore led to reduction of effective surface area which plays an important role in metal uptake [34, 35].

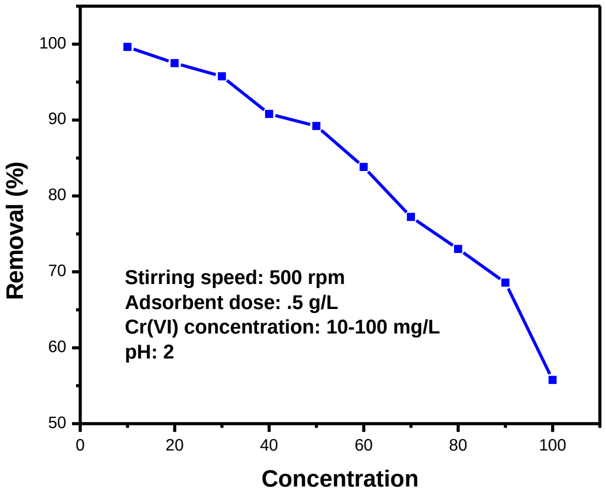

Figure 2 Effect of initial concentration Cr (VI) removal

Effect of initial Cr (VI) concentration

Adsorption experiments at initial Cr (VI) concentrations from 10 to 100 mg/l were also performed with fixed dosages of riverbed sand. The results indicated that percentage Cr (VI) removal decreased as the initial concentration of Cr (VI) was increased. **Figure 2** shows that when the initial Cr (VI) ion concentration increased from 10 – 100 mg/L, Cr (VI) adsorption removal decreased from 99.67% to 55.74% and the uptake capacity of riverbed sand increased. The decrease in removal percent was due to the saturation of the sorption sites on adsorbents. In addition, the increasing in uptake capacity of riverbed sand with increasing of Cr (VI) ion concentration is due to higher availability of Cr (VI) ions in the solution, for the adsorption and this can be attributed to an increase in deriving force to overcome the all mass transfer resistance of metal ions between the aqueous and solid phases resulting in higher probability of collision between Cr (VI) ions and adsorbents. This also results in higher metal uptake. It is clear from the figure that in initial stages the removal/adsorption percentage is higher in lower ranges of concentration.

Effect of pH

The effect of pH on the adsorption of Cr (VI) on riverbed sand is investigated and shown in **Figure 3**. The adsorption behavior of Cr (VI) was studied in pH range of 2 to 8. Removal of Cr (VI) was high in acidic and low in alkaline medium. Removal decreased from 99.99% to 68.66% by increasing pH of the solution from 2 to 8 at 10 mg/L initial concentration, 30 ℃, 100 μm particle size, 500 rpm agitation speed and 0.5 g/L adsorbent dose. The removal efficiency was highly pH dependent, the percentage uptake of Cr (VI) decreased gradually with an increase in pH. Figure 3 shows the effect of pH on Cr (VI) removal efficiency.

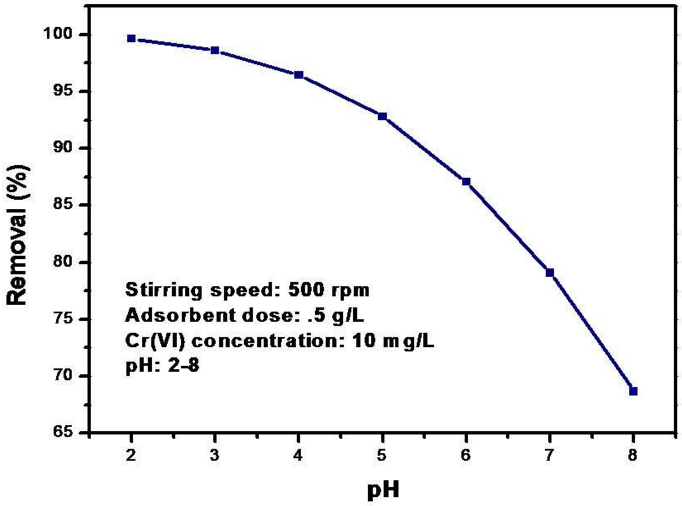

Figure 3 Effect of pH on Cr (VI) removal

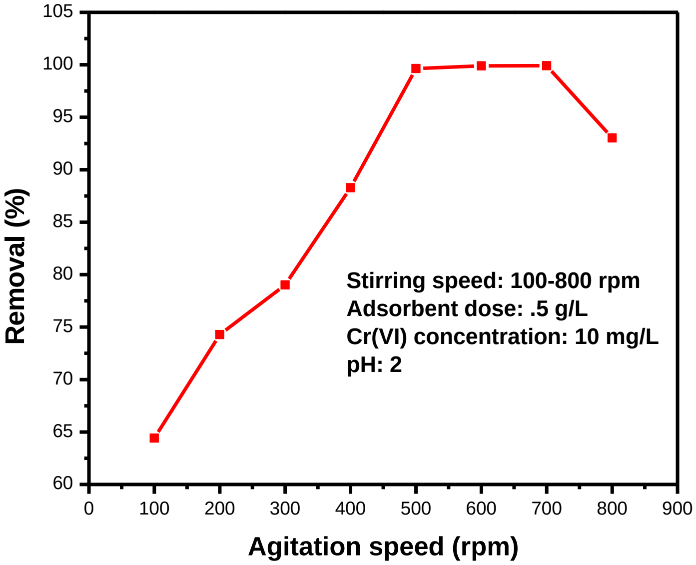

Figure 4 Effect of agitation speed on Cr (VI) removal

Effect of agitation speed

The effect of agitation speed rate on Cr (VI) adsorption is shown in **Figure 4** and it appears agitation speed has pronounced effect on the amount of Cr (VI) adsorbed. As the agitation speed increased from 100 to 500 rpm, the percentage removal increased from 64.5 % to 99.99 %. However beyond 500 rpm, the adsorption percentage remained constant and the agitation speed of 500 rpm was selected in subsequent analysis. The increase in adsorption capacity at a higher agitation speed could be explained in terms of the reduction of boundary layer thickness around the adsorbent particles [36]. Therefore, with increasing agitation speed the concentrations of Cr (VI) ions near the adsorbent surface would be increased. A higher agitation speed also encourages a better mass transfer of Cr (VI) ions from bulk solution to the surface of the adsorbent and shortened the adsorption equilibrium.

Effect of agitation time

The percentage removal of metal ions at different time intervals were determined and plotted against time was shown in **figure 5**. It is clear from the figure that adsorbed amount of Cr (VI) constantly increases with increasing time and then attains equilibrium. It happens because the rate of meal ions binding with microspheres is more at initial stage which gradually decreases and remains constant after an optimum adsorption. Equilibrium agitation time is defined as the time required for the heavy metal concentration to reach a constant value.

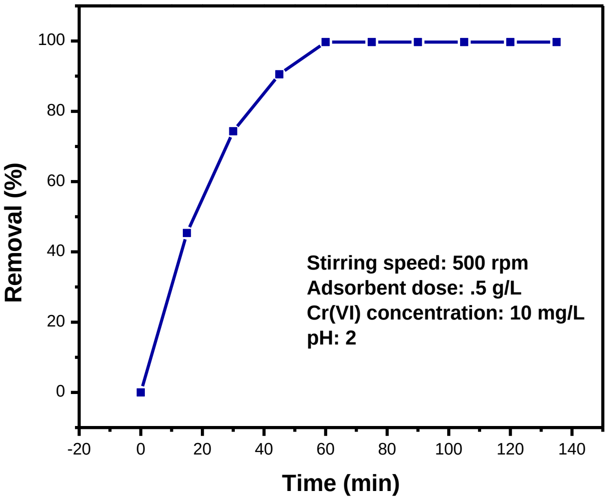

Figure 5 Effect of contact time on Cr (VI) removal

Adsorption isotherm

Equilibrium data, commonly known as adsorption isotherms, are basic requirements for the design of adsorption systems. Adsorption isotherm also usually describes the equilibrium relationship between adsorbent and adsorbate. The correlation coefficient (r^2) value for the equilibrium curve is the most significant parameter to optimize the design of an adsorption system to removal of metal ions from effluents. Hence, the correlation of equilibrium data using either a theoretical or empirical equation is essential for the adsorption interpretation and prediction of the extent of adsorption.

The linear plot of Langmuir isotherm for Cr (VI) adsorption and the calculated parameters along with regression coefficients are shown in **Figure 6** and **Table 1**, respectively. Maximum adsorption capacity, q_m, for complete monolayer coverage are found 2.6809 mg/g for 0.5 g/l. R^2 values approaching to one (0.999), clearly suggest that Langmuir isotherm follows a good relation of Cr (VI) ions adsorption with riverbed sand. 'K_L' is the adsorption constant related to the affinity of binding sites (l/g) and lower value of 'K_L' (l/g for three doses) indicate that the particles radius of riverbed sand were small toward adsorption [36]. The value of R_L (0.057) revealed that Cr (VI) adsorption on riverbed sand is favorable as the values lies between 0 and 1.

The linear plot of Freundlich equation for Cr (VI) adsorption and the calculated parameters are shown in **Figure 7** and **Table 1**, respectively. K_F is a Freundlich constant that shows adsorption capacity on heterogeneous sites with non-uniform distribution of energy level and n shows the intensity between adsorbate and adsorbent. Compared with the correlation coefficient value of linear plot of Langmuir isotherm, which of Freundlich model was found to less satisfactory. In this study the n values are greater than unity indicating chemisorption. Isotherms with n>1 are classified as L-type isotherms reflecting a high affinity between adsorbate and adsorbent and it is indicative of chemisorption.

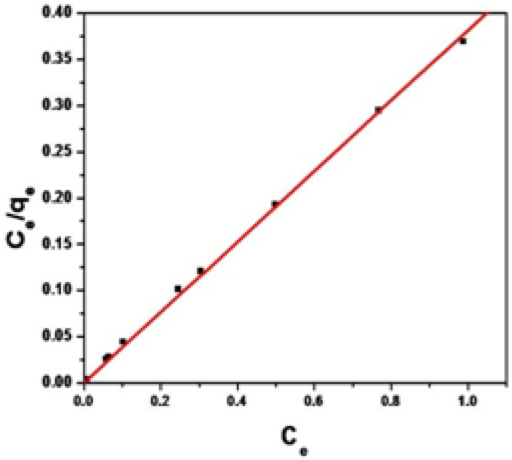

Figure 6 Linear plot of Langmuir isotherm

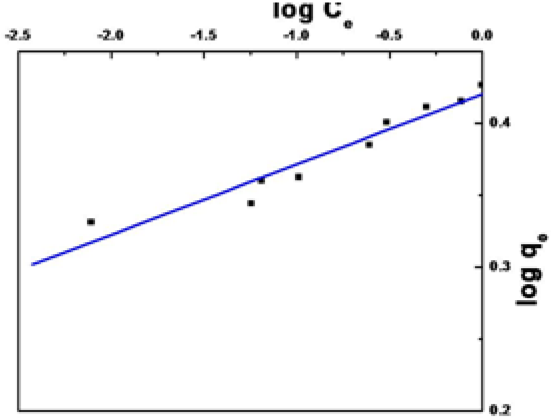

Figure 7 Linear plot of Frendulich isotherm

Figure 8 and **Table 1** shows Temkin isotherm and calculated constant of adsorption (b_T). Linear plots for Tempkin adsorption isotherm, which consider chemisorptions of an adsorbate onto adsorbent, fit quite with correlation coefficients ≥ 0.919. This further supports the findings that the adsorption of Cr (VI) ions onto riverbed sand is a chemisorptions process.

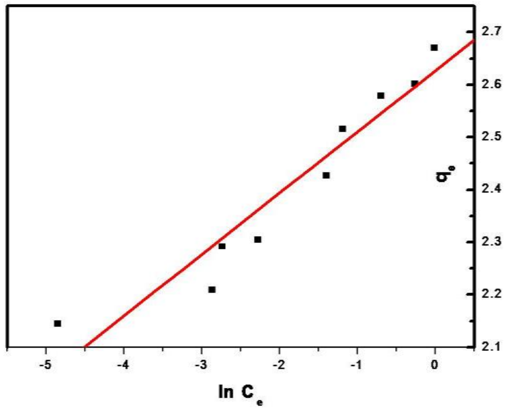

Figure 8 Linear plot of Tempkin isotherm

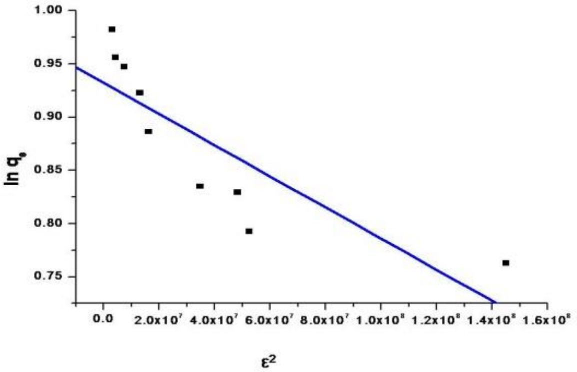

Figure 9 Linear plot of D-R isotherm

The slope and intercept of plots of ln q_e versus ε^2 were used to calculate the D-R isotherm parameters β and q_m show in **Table 1 and figure 9**. The q_m values are not consistent with the q_m values previously determined for Langmuir isotherm. The magnitude of the correlation coefficients for D-R isotherm model is lower when compared to other three isotherm models. This suggests that the Cr (VI) ions adsorption onto riverbed sand is not a physical process.

Table 1 The Isotherm constants for Cr (VI) adsorption onto riverbed sand

Isotherm models	parameters	Results
Langmuir isotherm	R^2	0.999
	K_L	7.668
	q_m (mg/g)	2.809
	R_L	0.057
Freundlich isotherm		
	R^2	0.928
	K_F	1.521
	n	20.83
Tempkin isotherm	R^2	0.919
	B_T	320.21
	A	2.627
D-R isotherm	R^2	0.709
	β	0.001
	q_m (mg/g)	0.932
	E (kJ/mol)	22.361

Kinetic studies

Values of correlation coefficients for these equations determined by non-linear regression for all kinetic models, pseudo-first and pseudo-second order and intra-particle equation for adsorption of Cr (VI) ions onto riverbed sand are listed in **Table 2** and **Figures 10, 11, 12**. The correlation coefficient (r^2) for the linear plots of pseudo-second order reaction plots is better than the pseudo first-order plots.

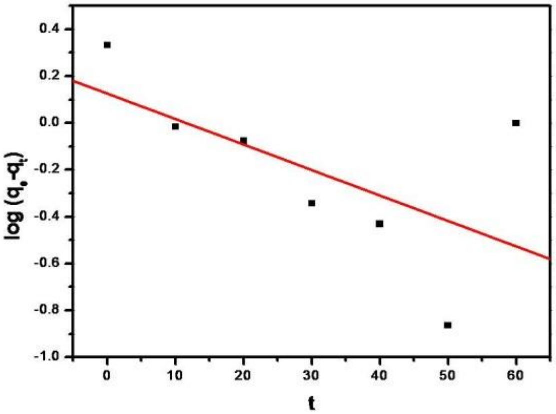

Figure 10 Linear plot of Pseudo-first order model

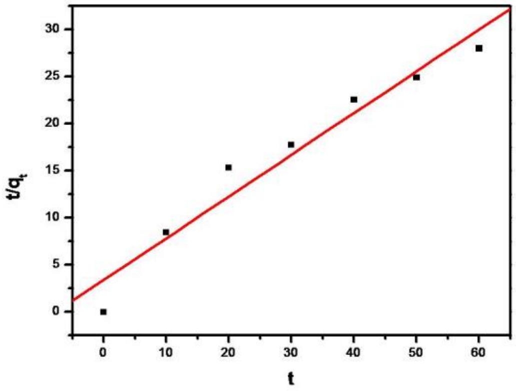

Figure 11 linear plot of pseudo-second order model

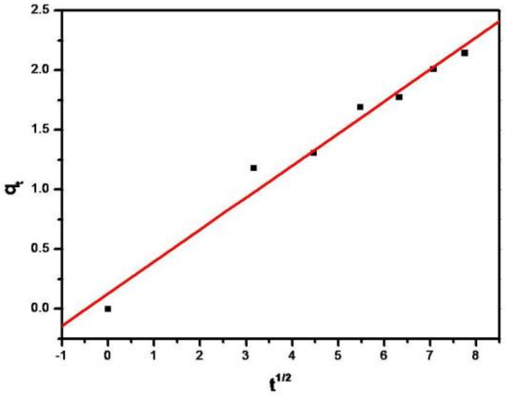

Figure 12 plot of intra-particle diffusion model

Table 2 Different kinetic model parameters

Models	**Parameters**	
	$q_{e,\ exp}$ (mg/g)	2.1443
Pseudo-first order model	R^2	0.373
	k_1 (min^{-1})	0.02303
	$q_{e,\ cal}$ (mg/g)	1.1331
Pseudo-second order model	R^2	0.950
	h (mg/g min)	0.2934
	k_2 (g/mg min)	0.0576
	$q_{e,\ cal}$ (mg/g)	2.2573
Intra-particle diffusion model	R^2	0.976
	K_{id} (mg/g $min^{-0.5}$)	0.269
	A (mg/g)	0.125

This shows that the pseudo-second order kinetic model explains the sorption in better way. The pseudo-second order model is based on the assumption that the rate determining step may be a chemical sorption involving valance forces through sharing or exchanging electrons between sorbent and sorbate. If the intraparticle diffusion involved in the adsorption process, then the plot of the square root of time versus the uptake would result in a linear relationship and the intraparticle diffusion would be controlling step if this line is passed through the origin. When the plots donot pass through the origin, this indicative of some degree of boundary layer control and this further shows that the intraparticle diffusion is not the only rate controlling step, but also other process may control the rate of adsorption [37]. The values of k_{id} were calculated from the slope of such plots and the r^2 values led to the conclusion that the intraparticle diffusion process is the rate-limiting step. Higher values of k_{id} illustrate an enhancement in the rate of adsorption, whereas larger k_{id} values illustrate a better adsorption mechanism, which is related to an improved bonding between Cr (VI) ions and the adsorbent particles.

Conclusion

The present study focuses on adsorption of Cr (VI) ions from aqueous solution using the riverbed sand as an effective adsorbent. Adsorption of Cr (VI) ions is found to be effective in the lower pH range. Equilibrium isotherm data were fitted using different models. Among them models, Langmuir model and Temkin model are in good agreement with the experimental data with high R^2. Kinetic study showed that the pseudo-second order model is appropriate to describe the experimental and film diffusion might be involved in the sorption process.

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