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Full Length Research Paper

Removal of chromium from an aqueous solution using *Azadirachta indica* (neem) leaf powder as an adsorbent

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In the present investigation, *Azadirachta indica* (neem) leaf powder is used as an adsorbent for the removal of chromium from aqueous solutions. The equilibrium studies are systematically carried out in a batch process, covering various process parameters that include agitation time, adsorbent size and dosage, initial chromium concentration, volume of aqueous solution and pH of the aqueous solution. Adsorption behavior is found to follow Freundlich and Langmuir isotherms. The adsorption mechanism is described by a pseudo second order kinetics.

Key words: Adsorption, neem, chromium, kinetics.

INTRODUCTION

Advances in science and technology have brought tremendous progress in many spheres of development, but in the process, also contributed to degradation of environment all over the globe due to very little attention paid to the treatment of industrial effluents. Industrial pollution continues to be a potential threat affecting the water. The discharge of non-biodegradable heavy metals into water stream is hazardous because the consumption of polluted water causes various health problems. Waste streams containing heavy metals such as Cu, Zn, Ni, Pb, Cd, Cr are often encountered in various chemical industries. Among these heavy metals, pollution by chromium is of considerable concern as the metal is used in electroplating, leather tanning, metal finishing, and chromate preparation. Chromium occurs in aqueous systems in trivalent and hexavalent forms. But the latter form is of particular concern due to its greater toxicity. When chromium enters the gastric system, epigastric pain, nausea, vomiting, severe diarrhea, corrosion of skin, respiratory tract and lungs carcinoma are noticed. The discharge limit of chromium from industries is less than 1 mg/L. Chromium is hazardous to health when its limit in potable water exceeds 0.5 mg/L (D.W.S, 1991).

Various methods adopted for the removal of heavy metals from industrial effluents include chemical precipitation, membrane separation, ion exchange and adsorption. In case of adsorption, the generally used adsorbents like activated carbon, silica, alumina etc- are expensive. This has prompted the use of various materials as adsorbents in order to develop cheaper alternatives. Natural materials available in large quantities or waste products may have the potentiality of high uptake of metals. They can be disposed off without regeneration due to their lower cost. A variety of adsorbents like chitosan (Schmuhl et al., 2001), cupress female cone (Murugan and Subramaniyan, 2003), coconut jute carbon (Chand et al., 1994), activated carbon from different materials (Kob-

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Nomenclature: b = Langmuir constant, g^{-1}; C_o = Initial concentration of chromium (VI) in aqueous solution, mg/L; C_t = Concentration of chromium (VI) in aqueous solution after time t min, mg / L; C_e = Equilibrium concentration of chromium (VI) in aqueous solution, mgL; d_p = Adsorbent size, µm; k_f = Freundlich constant, mg/g; K_{ad} = Lagergren first order rate constant, min^{-1}; K = Second order rate constant, $g.mg^{-1}.min^{-1}$;K_L = Langmuir constant, mg/g; m = Amount of adsorbent taken per 1 L of aqueous solution, g/L; n = Freundlich constant, dimensionless; q_t = Amount of Cr(VI) adsorbed per unit mass of adsorbent at t min, $(C_o\text{-}C_t)/m$, mg/g; q_e =Amount of Cr (VI) adsorbed per unit mass of adsorbent at equilibrium,$(C_o\text{-}C_e)/m$, mg/g; q_m = Maximum amount of Cr (VI) adsorbed per unit mass of adsorbent, mg/g; R = Correlation coefficient; R_L = Separation factor, $1/(1+bC_e)$; t = Agitation time, h; V = Volume of the aqueous solution, ml; w = Adsorbent dosage, g.

Table 1. Experimental conditions investigated

Parameter	Values investigated
Agitation time, t, h	0.5, 1, 2, 3, 4, 5, 6, 7 & 8
Adsorbent size, d_p, µm	106, 150 & 212
Adsorbent dosage, w, g	1, 2, 4 & 6
Initial concentration of chromium in aqueous solution, C_o, mg/L	25, 50, 75, 100 & 125
Volume of the aqueous solution, v, ml	50, 100, 150, 200 & 250
pH of the aqueous solution	3, 4, 5, 6, 7, 8 & 9

ya, 2004; Karthikeyan et al., 2005; Hamadi et al., 2001; Mor et al., 2006; Demirbas et al., 2004), sugar cane bagasse (Sharma and Forster, 1994a), leaf mould (Sharma and Forster 1994b), coconut tree saw dust (Selvi et al., 2001) were reported in literature for removal of chromium. The treated saw dust of Indian rose wood (Garg et al., 2004) was effective at a pH of 3 and the optimum agitation time of 60 min. was observed for the removal of chromium. The adsorption of chromium was investigated with pine needles and wool (Dakiky et al., 2002), soya cake (Daneshvar et al., 2002), waste pomace from olive oil factory (Malkoc et al., 2006) and peat (Ulmanu et al., 2003). The maximum uptake of 86.42 moles of chromium (VI) per liter of wet grape stalk waste (Flol et al., 2004) was recorded and the adsorption followed pseudo second order kinetics. The eucalyptus bark (Sarin and Pant, 2005) was effective at a pH of 2 and at a concentration of 200 ppm for the removal of chromium (45 mg/g). With tamarind hull (Verma et al., 2006), 99% (70 mg/g) of chromium was removed at a pH of 1. The adsorption data were also obtained with Turkish fly ash (Bayat, 2002), *Neurospora crassa* (Tunali, 2005), cationic starch maleate (Xing et al., 2006), raw rice bran (Oliviera et al., 2005), *Terminalia arjuna* nuts (Mohanty et al., 2005), *Aspergillus flavus* (Deepa et al., 2006), tannin immobilized on collagen (Liao et al., 2004), fungi biomass (Keskinkan et al., 2004), London plane leaves (Aoyama, 2003), calcined bauxite (Baral et al., 2007) etc. The interesting features of the new adsorbents are their high versatility, metal selectivity and high uptake. The present investigation looks into a specific process, for the removal of toxic element, chromium by adsorption using low cost adsorbent developed from abundantly available neem leaf powder, a plant material (Venkata et al., 2005).

Preparation of the adsorbent

The neem belongs to meliaceae family and is native to Indian sub-continent. Its seeds and leaves have been in use since ancient times to treat a number of human ailments and also as a household pesticide. The tree is also known as an air purifier. The medicinal and germicidal of the neem tree have been put to use in a variety of applications. The mature neem leaves used in the present investigation are collected from the available trees in Engineering College area of Andhra University, Visakhapatnam. They are washed thrice with water to remove dust and water soluble impurities and are dried until the leaves become crisp. The dried leaves are powdered and further washed with distilled water till the washings are free of colour and turbidity. Then the neem leaf powder is dried and sieved to different (212, 150, 106 µm) size fractions using a rotap sieve shaker. The resulting neem powder fractions are preserved in glass bottles for use as an adsorbent. The presence of niacin, proline, glutamic acid, aspartic acid, glutamine, tyrosine and alanine which contain polar groups like $-NH_2$, -COOH, -OH etc. in neem powder (Battacharya and Sarma, 2004; Neem foundation, http://www.neemfoundation.org, 1997) contribute to the negative surface charge. The ingre-dients contribute an electro negativity of 35.1%.

Experimental procedure

5.462 g of 99% $K_2Cr_2O_7$ (analytical grade) is dissolved in 1 liter (L) of double distilled water to prepare 1000 mg/L of chromium stock solution. The pH of the aqueous solution is varied by adding the required amounts of 1N H_2SO_4 and 1N NaOH. 75 mg/L chromium aqueous solution is prepared by diluting 75 ml of 1000 ppm chromium stock solution with distilled water in 1000 ml volumetric flask. 50 ml of the aqueous solution containing 75 mg/L chromium (VI) is treated with 2 g of 212 µm size adsorbent in a 250 ml conical flask for 30 min by shaking at 160 rpm on an orbital shaker at room temperature. The sample is allowed to settle and then it is filtered through a Whatman filter paper. The filtrate of the sample is analyzed in an AAS (Perkin Almer, model - 3100, 357.9 nm wave length) for the final concentration of chromium in aqueous solution. The percentage removal of chromium (VI) is calculated as $(C_o\text{-}C_t) \times 100/C_o$. The same experimental procedure is repeated for different agitation times and also for the other adsorbent sizes (150 and 106 µm). The agitation time for equilibrium to be established between the solution and adsorbent is noted. The effects of other parameters such as initial concentrations of chromium in the aqueous solution, volume and pH of the aqueous solution on % removal of chromium (VI) are obtained at the equilibrium agitation time by following the procedure described above. The values of variables studied in this investigation are shown in Table 1.

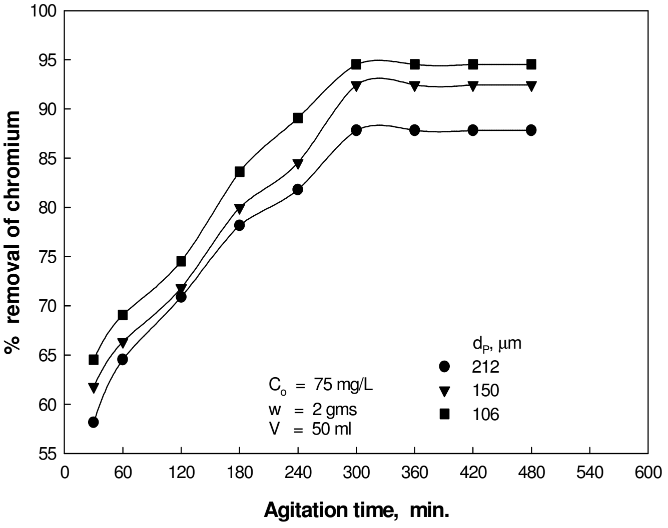

Figure 1. Effect of agitation time on % removal of chromium.

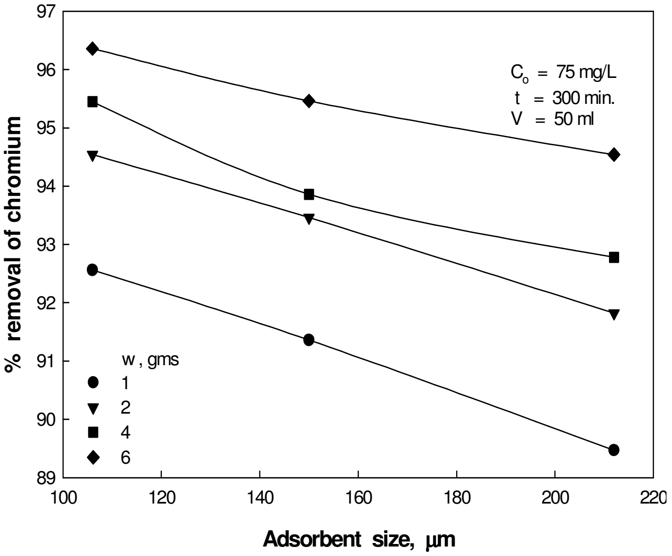

Figure 2. Variation of % removal of chromium with adsorbent size.

RESULTS AND DISCUSSION

Effect of agitation time

The equilibrium agitation time is determined by plotting the % removal of chromium against agitation time for different adsorbent sizes as shown in Figure 1. The % removal of chromium increases up to 5 h of agitation time and there after no further increase is recorded as reported earlier (Sarma and Battacharya, 2005) for chromium removal by neem leaf powder. The equilibrium agitation time for adsorption of lead by neem leaf powder is also reported as 5 h (Battacharya and Sarma, 2004).

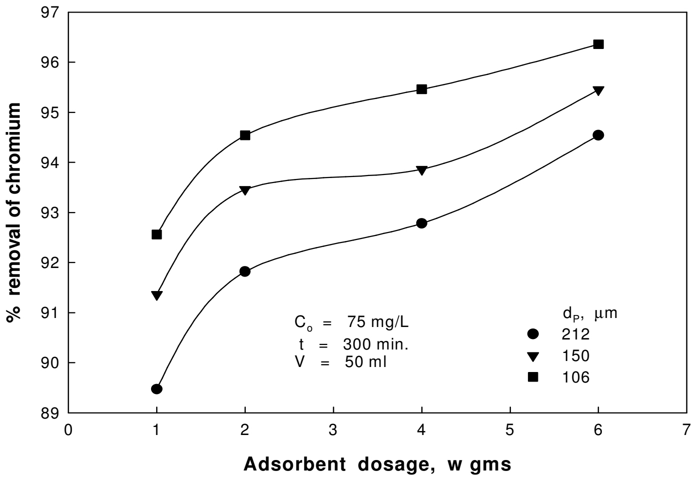

Figure 3. % removal of chromium as a function of adsorbent dosage.

Adsorption equilibrium time is defined as the time required for heavy metal concentration to reach a constant value. In the first 30 min the % removal is so rapid 58 - 65% removal is noticed for three adsorbent sizes. With 2 g of 106 µm adsorbent size, the removal of chromium is increased from 65 to 94.5% in the next 4.5 h of agitation. Different equilibrium agitation times are reported in literature for the removal of chromium. With the waste pomace from olive oil industry (Malkoc et al., 2006) the equilibrium agitation time is found to be 3 h. It is 1.5 h with neurospora crassa (Tunali et al., 2005), 2 h with activated alumina and activated charcoal (Mor et al., 2006), 20 min with cationic starch maleate (Xing et al., 2006), 60 min with raw rice bran (Oliviera et al., 2005), 100 min with tamarind hull (Verma et al., 2006) and 7.5 h with *Terminalia arjuna* nuts (Mohanty et al., 2005).

Effect of adsorbent size and dosage

The variations in percentage adsorption of chromium in aqueous solution with adsorbent size are presented in Figure 2. The % removal of chromium increases from 87.8 (1.65 mg/g) to 94.5% (1.77 mg/g) for 2 g of dosage at 5 h equilibrium agitation time with decrease in the adsorbent size from 212 to 106 µm. This phenomenon is expected as the decrease in the size of the adsorbent results in the increase of the surface area of it, thereby the number of active sites are better exposed to the adsorbate. Hamadi et al. (2001) reported increased uptake capacities from 27.74 to 29.93 mg/g by decreasing the adsorbent size from 0.9 to 0.38 mm. Increased uptake capacities from 10.6 to 59.4 mg/g were recorded by Demirbas et al. (2004) by decreasing the size of the activated carbon obtained from agricultural waste from 1.4 to 1.25 mm. Figure 3 shows the % removal of chromium with adsorbent dosage at equilibrium agitation

Table 2. Metal uptake capacities (mg/g) for initial chromium concentration of 75 mg/L

Adsorbent size, µm	Adsorbent dosage, g			
	1	2	4	6
212	3.2	1.65	0.87	0.58
150	3.4	1.73	0.88	0.6
106	3.48	1.77	0.9	0.6

Table 3. Metal uptake capacities (mg/g) for 106 µm size

Initial chromium concentration in the Aqueous solution(mg/L)	Adsorbent dosage, g			
	1	2	4	6
25	1.18	0.6	0.3	0.2
50	2.34	1.19	0.6	0.4
75	3.48	1.78	0.9	0.6
100	4.37	2.25	1.14	0.77
125	4.92	2.51	1.34	0.92

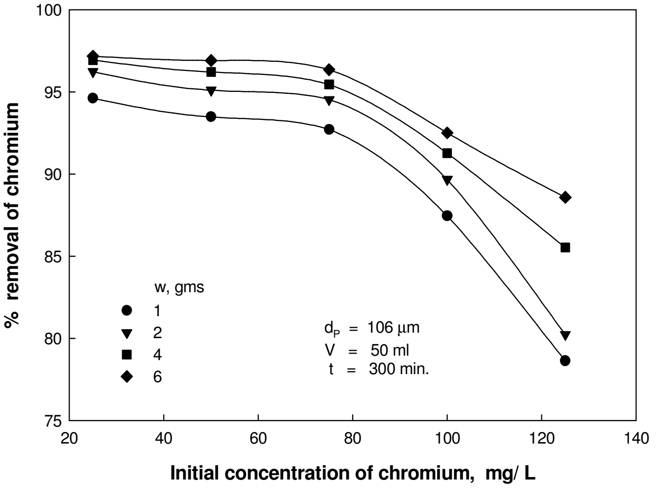

Figure 4. Influence of initial concentration of chromium on % removal of chromium

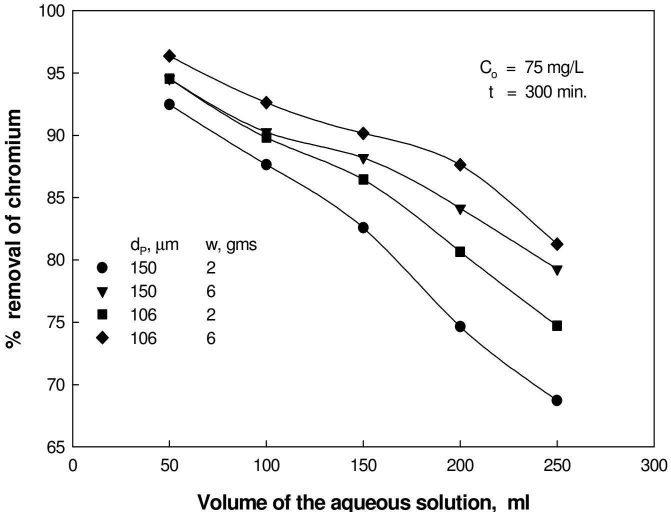

Figure 5. Change in % removal of chromium with volume of the aqueous solution.

time for different adsorbent sizes. The % removal of chromium from the aqueous solution increases from 92.7 (3.48 mg/g) to 96.4% (0.6 mg/g) with an increase in the adsorbent amount from 20 to 120 g/L for 106 µm size. Such behavior is obvious since the metal uptake of the adsorbent increases with the increase in dosage. This is so because the number of active sites available for metal increases with the increase in the amount of adsorbent. Malkoc et al. (2006) observed higher % removal from 51.7 (10.34 mg/g) to 98.6 % (6.57 mg/g) as the dosage is increased from 5 to 15 g/L. By increasing the *Terminalia arjuna* nuts dosage from 0.5 to 3.5 g/L, the % removal increases from 78 to 100% (Mohanty et al., 2005). Deepa et al. (2006) increased the dosage of *Aspergillus flavus* from 4 to 20 mg/L and the % removal was increased from 29 to 65%. By increasing the dosage of treated saw dust from 2 to 10 g/L, Garg et al. (2004) reported increased removal from 65.4 (3.7 mg/g) to 100% (10 mg/g). The metal uptake capacity in the present investigation for various adsorbent sizes, dosages and C_o values are compiled in Tables 2 and 3.

Effect of initial Cr (VI) concentration and volume of aqueous solution

The effect of initial concentration of chromium is shown in Figure 4. Results from these plots indicate that % removal decreases from 94.6 (1.18 mg/g) to 78.6% (4.92 mg/g) as the initial concentration of chromium is increased from 25 mg/L to 125 mg/L for 1 g of 106 µm size adsorbent at equilibrium agitation time. However, the change in % removal is nominal for C_o between 25 and 75 mg/L. Evidently such a behavior can be attributed to the maintenance of fixed number of binding sites in the dosage while increasing the concentration. Mohanty et al. (2005) increased the initial concentration of chromium from 10 to 30 mg/L and noticed the decreased adsorption from 100 to 94%. The adsorption of chromium is decreased from 61 to 30.4%, as the initial concentration of waste pomace of olive oil factory was increased from 50 to 200 mg/L (Malkoc et al., 2006). As the initial concentration of cationic starch maleate was increased from 10 to 45 mg/L, the uptake capacities were decreased from 33 to 24 mg/g (Xing et al., 2006). Figure 5 shows the change in % adsorption of chromium with variations in volume of aqueous solution from 50 to 250 ml for adsorbent sizes, 150 and 106 µm and two adsorbent dosages of 2 and 6 g. From the plots, it is clear that % re-

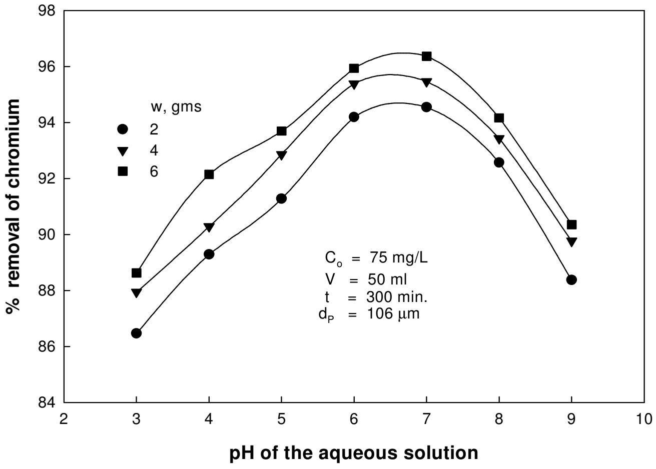

Figure 6. Effect of pH of aqueous solution on % removal of chromium

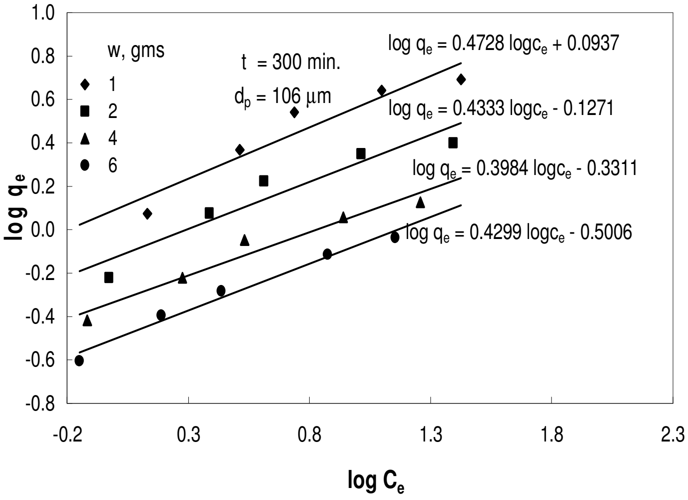

Figure 7. Freundlich isotherm for adsorption of chromium using neem leaf powder

moval of chromium decreases gradually from 94.5 (1.77 mg/g) to 79.3% (7.43 mg/g) for 2 g of 106 µm size with an increase in the volume of the aqueous solution. The reason may be attributed to an increase in chromium metal concentration with an increase in volume of the water and the unchanged area of contact of the adsorbent. The % removal of chromium is found to be maximum (96.4%) for 6 g of 106 µm adsorbent size.

Effect of pH of the aqueous solution

pH is an important factor controlling the process of adsorption as it affects the surface charge of the adsorbents, the degree of ionization and the species of adsorbate.

The effect of pH on adsorption of chromium is shown in Figure 6. The % removal of chromium increases from 86.5 to 94.5% for 2 g of 106 µm adsorbent with an increase in pH from 3 to 7 as reported earlier (Sarma and Battacharya, 2005). It is conformed that adsorption increases with the decrease in acidity. At low pH, hydrogen ions compete with chromium ions for appropriate sites on the adsorbent. As pH approaches to 7, the competition of hydrogen ions becomes negligible and more chromium ions are bound to the adsorbent. The % removal decreases as pH increases beyond 7. The maximum removal of chromium (3 mg/g) was reported at a pH value of 7.4 for treated bauxite (Baral et al., 2006). With chitosan as adsorbent (Sarin and Pant, 2005), the maximum uptake capacity (50 mg/g) was noted at a pH of 5. The fungi biomass (Keskinkan et al., 2004) removed 64 mg/g at a pH of 4.8. The maximum removal of chromium was reported at a pH of 4 with cationic starch maleate (Xing et al., 2006).

The principal driving force for metal ion adsorption is the electrostatic interaction that is, attraction between adsorbent and adsorbate. The greater the interaction the higher the adsorption of heavy metal. The neem leaf powder contains 35.1% electronegative components. In the present investigation, the maximum % removal of chromium is 97% (7.43 mg/g). The reason can be attributed to the higher electro negativity of the adsorbent, the neem leaf powder.

Adsorption isotherms

The empirical Freundlich relationship (Freundlich, 1907), does not indicate a finite uptake capacity of the adsorbent. This relationship can be reasonably applied to the low or intermediate concentration ranges. Freundlich isotherm equation is given by:

$$q_e = k_f \, C_e^n \qquad (2)$$

and is linearized as:

$$\log q_e = \log k_f + n \log C_e \qquad (3)$$

The present data, plotted in Figure 7, shows good linearity for Freundlich relationship (correlation coefficient, R = 0.94 - 0.98). Linearity of the relationship indicates strong binding of Cr (VI) to the adsorbent .The slope of the isotherm (n) varies between 0.4 and 0.47 (0.43 is the mean value) fulfilling the condition of $0<n<1$ for favorable adsorption.

The following equations are obtained from the graph:

$$\log q_e = 0.4728 \log C_e + 0.0937 \qquad (4)$$
$$\log q_e = 0.433 \log C_e - 0.1271 \qquad (5)$$
$$\log q_e = 0.3984 \log C_e - 0.3311 \qquad (6)$$
$$\log q_e = 0.4299 \log C_e - 0.5006 \qquad (7)$$

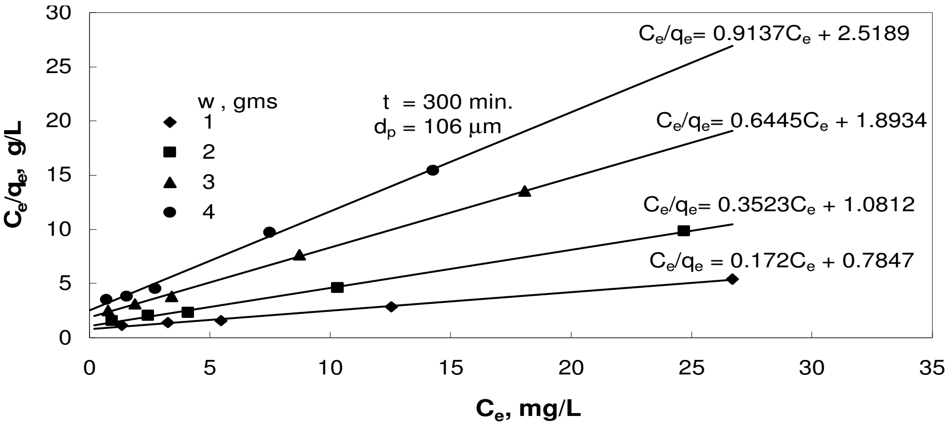

Figure 8. Langmuir isotherm for adsorption of chromium using neem leaf powder.

Table 4. q_m, k_f, n and R values obtained in the present investigation

Neem leaf powder ($g.L^{-1}$)	Langmuir isotherm		Freundlich isotherm		
	q_m, mg/g	R	k_f	n	R
20	49.15	0.99	1.2407	0.47	0.94
40	25.1	0.99	0.7462	0.43	0.95
80	13.63	0.99	0.4665	0.4	0.96
120	9.23	0.99	0.3157	0.43	0.98

Table 5. q_m, k_f and n values obtained with other adsorbents

Adsorbent	Langmuir isotherm	Freundlich isotherm	
	q_m, mg/g	k_f ,min^{-1}	slope, n
Activated alumina & charcoal (Mor et al., 2006)	0.1422 & 0.033	3.1262 & 0.9614	3.488 & 1.6162
Treated bauxite (Baral et al., 2007)	1.8	-0.2878	0.429
Terminalia arjuna nuts (Mohanty et al., 2005)	28.4	2.72	1.815

The present adsorbent seems better with respect to slope than those reported in Table 5. Langmuir isotherm (Langmuir, 1918) is the most widely used two-parameter equation. The relationship is of the form:

$$q_e/q_m = bC_e/ (1+bC_e) \quad (8)$$

Langmuir isotherm for the present data is drawn in Figure 8 between C_e and C_e/q_e. q_m is calculated from the slope ($1/q_m$). The isotherm lines have good linearity (correlation coefficient, R~0.99) indicating strong binding of Cr (VI) ions to the surface of neem leaf powder. The separation factor, R_L obtained between 0.105 and 0.795 for various adsorbent dosages shows favorable adsorption ($0<R_L<1$). The following equations are obtained from Figure 8.

$$C_e/q_e = 0.172\, C_e + 0.7847 \quad (9)$$
$$C_e/q_e = 0.3523\, C_e + 1.0812 \quad (10)$$
$$C_e/q_e = 0.6445\, C_e + 1.8934 \quad (11)$$
$$C_e/q_e = 0.9137\, C_e + 2.5189 \quad (12)$$

The values of q_m, k_f, n and R obtained from Langmuir and Freundlich isotherms are tabulated below in Table 4.

The values of q_m, k_f and n obtained from Langmuir and Freundlich isotherms for other adsorbents are compiled in Table 5.

The mean q_m values in the present investigation is better than those reported in Table 5.

The adsorption capacities of various adsorbents used for the removal of chromium reported in the literature are compared along with the present investigation and shown in Table 6.

Studies on kinetics of adsorption

The order of adsorbate-adsorbent interactions has been

Table 6. Uptake capacities for chromium removal with various adsorbents

Adsorbent	Maximum uptake capacity, mg/g
Chitosan cross linked and Non-cross linked (Murugan and Subramaniyan 2003)	50 and 78
Cupress female cone (Chand et al., 1994)	119.4
Cement kiln dust (Karthikeyan et al., 2005)	33
Eucalyptus bark (Hamadi et al., 2001)	45
Hevea brasilinesis saw dust activated carbon (Mor et al., 2006)	44.05
Tyres activated carbon (Demirbas et al., 2004)	58.50
Leaf mould (Sharma and Forster 1994)	43.10
Sugar cane baggasse (Sharma and Forster 1994)	13.40
Coconut tree saw dust (Selvi et al., 2001)	3.60
Treated saw dust of Indian rose wood (Garg et al., 2004)	10
Pine needles and Wool (Dakiky et al., 2002)	5.36 and 8.66
Soya cake (Daneshvar et al., 2002)	0.288
Neem leaf powder (present investigation)	7.43

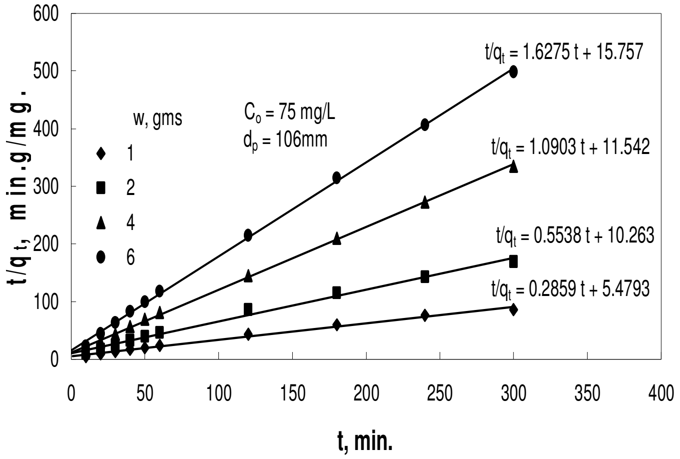

Figure 9. Second order kinetics for adsorption of chromium.

described using various kinetic models. Traditionally, the pseudo first order model derived by Lagergren finds wide application. In the case of adsorption preceded by diffusion through a boundary, the kinetics in most cases follows the pseudo first order rate equation of Lagergren:

$$dq_t/dt = K_{ad}\,(q_e - q_t) \qquad (13)$$

Plot of log $(q_e - q_t)$ versus t gives a straight line for first order kinetics and the adsorption rate constant, K_{ad} is computed from the plot. Lagergren plot of log $(q_e - q_t)$ versus agitation time t, for the present data is not linear. Hence, pseudo first order kinetics can not describe the mechanism of Cr (VI)-neem leaf powder interactions. On the other hand, several authors have shown that pseudo second order kinetics can describe these interactions very well in certain specific cases. The pseudo second order kinetics is given by:

$$dq_t/dt = K\,(q_e - q_t)^2 \qquad (14)$$

Rearranging the above equation, we get in the linear form

$$t/q_t = 1/(Kq_e^2) + (1/q_e)\,t \qquad (15)$$

If the pseudo second order kinetics is applicable, the plot of (t/q_t) versus t gives a linear relationship that allows computation of q_e and K. The pseudo second order model which considers the rate-limiting step as the formation of chemisorptive bond involving sharing or exchange of electrons between the adsorbate and the adsorbent is therefore applied.

In the present studies the kinetics is investigated with 50 mL of aqueous solution of initial Cr (VI) concentration of 75 mg/L at room temperature with four different adsorbent dosages of 1, 2, 4 and 6 g and with three adsorbent sizes of 106, 150 and 212 µm in the agitation time interval of 10 to 300 min. Figure 9 indicates the suitability of pseudo second order rate equation. The second order rate constant is in the range of 0.0136 to 0.1681 g. $mg^{-1}min^{-1}$. The suitability of second order rate equation for the present data indicates chemisorption as the rate controlling step (Battacharya and Sarma, 2004). The suitability of second order rate equation was reported in the literature for the removal of chromium for the adsorbents: treated bauxite (Baral et al., 2006), activated carbon from agricultural waste (Demirbas et al., 2004), used tyres and saw dust (Hamadi et al., 2001) and raw rice bran (Oliviera et al., 2005). From the Figure 9, the following pseudo second order rate equations are obtained.

For w = 1 g and $d_p = 106\ \mu m$ $\quad t/q_t = 0.2859\ t + 5.479$ (16)

For w = 2 g and $d_p = 212\ \mu m$ $\quad t/q_t = 0.5538\ t + 10.263$ (17)

For w = 4 g and $d_p = 106\ \mu m$ $\quad t/q_t = 1.0903\ t + 11.542$ (18)

For w = 6 g and $d_p = 106\ \mu m$ $\quad t/q_t = 1.6275\ t + 15.757$ (19)

Conclusions

The equilibrium agitation time for the adsorption of chromium is 5 h. The percentage removal of chromium in aqueous solution is increased with a decrease in adsorbent size and an increase in the adsorbent dosage. The increases in initial concentration of chromium and volume of the aqueous solution result in a decrease in % removal of chromium. % removal of chromium is increased up to a pH of 7. The adsorption data fit in both Freundlich and Langmuir isotherms and is well described by pseudo second order kinetics.

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