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Effect of Process Parameters on Adsorption of Cadmium from Aqueous Solutions by Activated Carbon Prepared from *Bauhinia purpurea* Leaves

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ABSTRACT

Adsorption is one of the most versatile and widely used techniques for the removal of toxic metals from the aqueous solutions. Cadmium is the toxic metal selected for adsorption in this study and adsorbent of interest was activated carbon prepared from *Bauhinia purpurea* leaves as a plant biomass. The following process parameters were investigated during the batch adsorption process: The adsorption efficiency and cadmium deposition on the adsorbent surface were increased from 15.95 to 89.98%, 0.074 to 0.418 mg/g, and 7.27 to 89.97%, 0.034 to 0.418 mg/g with increasing contact time from 2 to 50 min and pH from 2 to 6.5, respectively. The adsorption efficiency was decreased from 89.98 to 45.96% and the amount of cadmium deposited on the surface of the *ACBPL* adsorbent was increased from 0.418 to 1.032 mg/g with an increase in solute concentration. The adsorption efficiency of cadmium decreased from 98.41 to 84.51% and 0.457 to 0.392 mg/L with an increase in particle size from 74 to 177 µm. The rate of adsorption of cadmium was increased from 61.31 to 96.34% with an increase in dosage of the *ACBPL* adsorbent from 0.05 to 0.15 g. The adsorption efficiency and cadmium deposition was decreased from 89.98 to 97.90%, 0.418 to 0.454 mg/g as the temperature increased from 303K to 323K. The variation of thermodynamic energy parameters ($\Delta G°$, $\Delta H°$ and $\Delta S°$) with the solution temperature described that the adsorption process is endothermic, spontaneous at high temperatures and non-spontaneous at low temperatures. The positive value of ΔS, reflecting the high degree of disorder in the adsorbent/adsorbate interface that is formed during the transition.

INTRODUCTION

Freshwater is a limited resource in many parts of the world. The major fact for this paucity of freshwater is not only due to demand for water, but also due to pollution of freshwater ecosystems. Due to the pollution created by human beings, the usable water has decreased drastically and the cost of purifying the water has increased dramatically. The rapid pace of population growth, industrialization and improper planning of urban development activities have been severely contributing to the contamination of water and soil systems. The persistence of heavy metals (zinc, copper, nickel, lead, chromium, cadmium, mercury, etc.) in the aquatic ecosystems results in a serious threat because of their toxicity, bioaccumulation, and bio-magnification in the food chains (Momodu et al. 2010, Rao et al. 2010). Among all the heavy metals, Cd(II) is the predominant one as it is generated from a majority of industrial operations such as metal plating industries, electroplating industries, petrochemical industries, refining of ores, and battery production (Fu et al.

2011, Akunwa et al. 2014). Cadmium is discharged into the environment due to the industrial processing of metals like zinc, aluminium, etc. The cadmium enters the food chain through the uptake of contaminated water and soil by the plants. A 0.005 mg/L is set as the maximum cadmium concentration in the domestic water supplies by the World Health Organization and the EUD erective. Excessive cadmium concentration in drinking water can lead to a number of health effects, mainly cancer, malfunctions of human kidneys, damage of bones, and many other ailments (Akunwa et al. 2014, Debasree et al. 2014). Formation of stones in the kidney and calcium metabolism is caused due to the excessive intake of cadmium. Adsorption is one of the most versatile and widely used techniques for the removal of metal ions. Activated carbon is the most effective and widely used adsorbent, because it has large specific surface area, micro-porous structure and excellent adsorption ability (Kannan et al. 2005). Activated carbon has been the water industry's standard adsorbent for the reclamation of municipal and industrial wastewater for potable use for decades. Despite its pro-

lific use in the water and wastewater treatment plants, activated carbon remains an expensive material. In recent years, research interest in the production of low cost (Akunwa et al. 2014, Kannan et al. 2005) activated carbon produced from diverse materials has grown. Low cost activated carbons are prepared by thermo-chemical methods from various materials and plant biomass to remove heavy metals from effluents (Venkatesan et al. 2013). The aim of the present study is to investigate the feasibility of alternative, low cost and novel adsorbents for efficient removal of cadmium from an aqueous solution. From the survey of the literature, no information is available for the adsorptive removal of cadmium by activated carbons prepared from *Bauhinia purpurea* leaves as adsorbent. The present work focuses on the adsorption process parameters. Initial metal ion concentration, pH, temperature and adsorbent dosage are the input variables considered for the optimization of heavy metal removal. Thermodynamic studies have been used to evaluate the thermodynamic energy parameters and compare the adsorption capacities of metals used with activated carbon prepared from *Bauhinia purpurea* leaves.

MATERIALS AND METHODS

***Bauhinia purpurea* leaves:** The adsorbent, *Bauhinia purpurea* leaves used in the present study were collected from Rajam and Srikakulam, Andhra Pradesh, India. *Bauhinia purpurea* is used in several traditional medicine systems to treat various diseases. Water extracts of *Bauhinia purpurea* leaves have been shown to have anti-ulcer activity in animals in the 'ethanol-induced gastric ulcer model. *Bauhinia purpurea* may possess antibacterial, antidiabetic, analgesic, anti-inflammatory, anti-diarrheal, anticancerous, nephroprotective, and thyroid hormone-regulating activity (Zakaria et al. 2011, Kumar et al. 2011).

Chemicals and reagents: The chemicals of AR/LR grades supplied by different standard manufacturing industries are given in Table 1.

Preparation of adsorbent: The *Bauhinia purpurea* leaves were washed several times with deionized water until the wash water contains no dirt. The washed leaves were completely dried under sunlight for 30 days and cut into small pieces and powdered using domestic mixer. The powder was dried in an oven at 100°C for 120 min. The dried material was now placed in the cooker for carbonization and it was heated continuously for 10 min. The dried material was activated to 650°C for 120 min in a covered silica crucible by heating in a muffle furnace. After that, 100 g of carbon powder was taken and mixed with 100 g ZnCl$_2$ dissolved in 500 mL of distilled water containing 22 g of HCl. The chemically treated material was left overnight and carbonized.

Table 1: Chemicals and reagents.

Reagent	Company	Purity
Cadmium Nitrate Cd (NO$_3$)$_2$	S. D. Fine Chem. Pvt. Ltd.	99.5%
Hydrochloric Acid (HCl)	Sarabai Company, India	35%
Zinc Chloride (ZnCl$_2$)	Fischer Inorganics Ltd.	70%

Table 2: Range of adsorption process parameters covered in the present study.

Process parameter	Cadmium	
	Min	Max
Contact time, t (min)	2	80
Initial metal ion concentration of the solution, C_i (mg/L)	2	10
Solution pH	2	10
Average particle size of the adsorbent, d (μm)	74	177
Dosage of the adsorbent, w (g)	0.025	0.15
Temperature, T (K)	303	323

Carbonized material was refluxed with 10% HCl solution for 180 min and filtered. This was followed by drying of the material at 100°C for 24 hrs. The dried product was sieved to desired particle size range of 74-177 μm (Fig. 1).

Preparation of metal solutions: Adsorbate solutions of cadmium with a concentration of 1000 mg/L were prepared separately by dissolving 2.7442 g of 100% Cd(NO$_3$)$_2$ in 1000 mL of double distilled water. From the standard stock solution, working solutions of lower concentrations were prepared with cadmium (2, 4, 6, 8 and 10 mg/L), used for batch experiment. After adsorption, the final effluent solution was analysed by atomic absorption spectrophotometer of Perkin Elmer model-3100, a flame type AAS.

Batch adsorption experimental studies: The adsorption study of cadmium was conducted in the exploratory conditions of various effective parameters like pH 2-10, contact time of 2-120 min, metal ion concentration of 2-10 mg/L, the dosage of the adsorbent 0.025-0.15 g and the particle size of the adsorbent from 74 (200 mesh)-177 (80 mesh) μm. Agitation speed of 250 rpm was kept constant in the orbital shaker with the suitable time interims from 2 -120 min. The mixed adsorbent solutions were taken out and filtered through Whatman filter paper and analysed for metal ion concentration in an Atomic Absorption Spectrophotometer (Perkin Elmer model-3100). Batch experiments were conducted at various temperatures (303 K-323 K) of the metal solution using orbital shaker with optimum contact time of 50 min at pH of 6.5. Samples were analysed by AAS for metals to assess the thermodynamic parameters and study the feasibility of the process with temperature.

Metal adsorption capacity: The amount of metal deposited on the adsorbent surface was determined by performing at different contact times (2-90 min), temperatures (303-323K) and pH (2-8) etc., for both the adsorbents. After experimentation, the amount of metal deposited on the adsorbent surface was calculated by using the following equation:

$$q_t = \frac{V(C_i - C_f)}{1000w} \qquad ...(1)$$

Where, q_t is the amount of metal deposited on the adsorbent surface (mg/g), C_i is the initial solute concentration in the solution before adsorption (mg/L), C_f is the final concentration of solute in the solution after adsorption (mg/L), V is the volume of metal solution (L) and w is the dosage of the adsorbent (g).

Thermodynamic studies: Thermodynamic studies provide information about the feasibility of the adsorption process. It also plays an important role in the study of the nature of adsorption process. The thermodynamic energy parameters like enthalpy change ($\Delta H°$), entropy change ($\Delta S°$), and Gibb's free energy ($\Delta G°$) are used to determine the spontaneity, heat change and affinity of the adsorption process. The Van't Hoff equation descries the thermodynamic parameters by using the following linear and nonlinear equations,

$$\frac{d \ln K_e}{dT} = \frac{\Delta H}{RT^2} \qquad ...(2)$$

$$\ln K_e = \frac{\Delta S^o}{R} - \frac{\Delta H^o}{RT} \qquad ...(3)$$

Where, R is gas constant (8.314 J/mol K) and K_e is the adsorption equilibrium constant equivalent to q_e/C_e. Van't-Hoff plot with $\ln K_e$ as a function of $1/T$ yields a straight line and $\Delta H°$ and $\Delta S°$ can be evaluated from the slope and intercept, respectively. The relationship between $\Delta G°$ and K_e is given by the following equation,

$$\Delta G^o = -RT \ln K_e \; \Delta G^o = -RT \ln K_e \qquad ...(4)$$

For significant adsorption to occur, the Gibb's free energy change of adsorption, $\Delta G°$, must be negative. As a thumb rule, a decrease in the negative value of $\Delta G°$ with an increase in temperature indicates that the adsorption process is more favourable at higher temperatures. This could be possible because the mobility of adsorbate ions/molecules in the solution increase with an increase in temperature and that the affinity of adsorbate on the adsorbent is higher at high temperatures. On the contrary, an increase in the negative value of $\Delta G°$ with an increase in temperature implies that lower temperatures make the adsorption easier. A negative value of $\Delta H°$ implies that the adsorption phenomenon

Fig.1: Activated carbons prepared from *Bauhinia Purpurea* leaves with average particlesize of 149 μm.

is exothermic while a positive value implies that the adsorption process is endothermic. In an endothermic process, the adsorbate species has to displace more than one water molecule for their adsorption and this results in the endothermicity of the adsorption process (Awwad et al. 2014, Venkatesan et al. 2013). A positive value of $\Delta S°$ reflects the affinity of the adsorbent towards the adsorbate species.

RESULTS AND DISCUSSION

Batch experiments were carried out to study the effect of process parameters such as contact time (t), solution pH, initial metal concentration (C_i), the dosage of the adsorbent (w), average particle size of the adsorbent (d) and temperature of the solution (Fig. 2). This section also deals with thermodynamic studies of the adsorption of cadmium using activated carbon prepared from *Bauhinia purpurea* leaves as an adsorbent.

Effect of contact time: The adsorption efficiency and metal accumulated on the adsorbent surface determined at different contact times are shown in Figs. 2 & 3. The percentage removal and cadmium accumulated on the adsorbent surface were increased with an increase in contact time from 2 to 50 min and thereafter reached plateau after attaining equilibrium at 50 min. The rapid adsorption at the early stages of contact period indicates the availability of more number of active sites on the surface of the adsorbent and also due to the thermal-chemical treatment process during the preparation of activated carbon from *Bauhinia purpurea* leaves. At later stages of adsorption, the external adsorbent surface was saturated with cadmium molecules, thus the adsorption proceeds on interior walls of the adsorbent surface. The slower adsorption rate towards equilibrium was due to the availability of less number of vacant sites on the adsorbent surface (Fu et al. 2011). After equilibrium, the adsorption rate was very slow with longer contact time, concluded the possible involvement of chemical bonding between cadmium
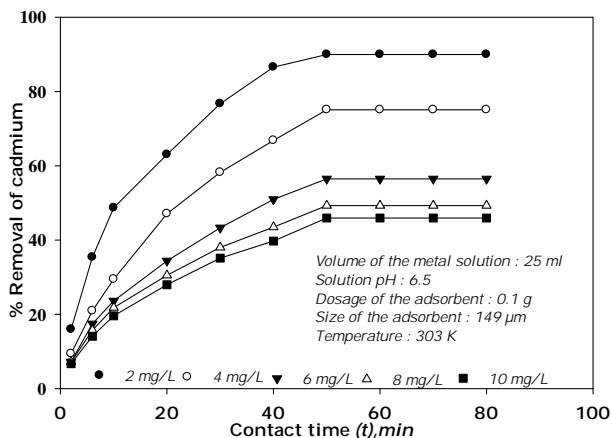
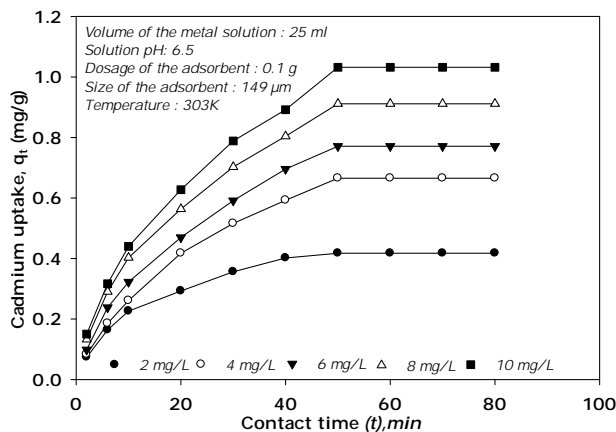Fig. 2: Effect of contact time on % removal of cadmium.

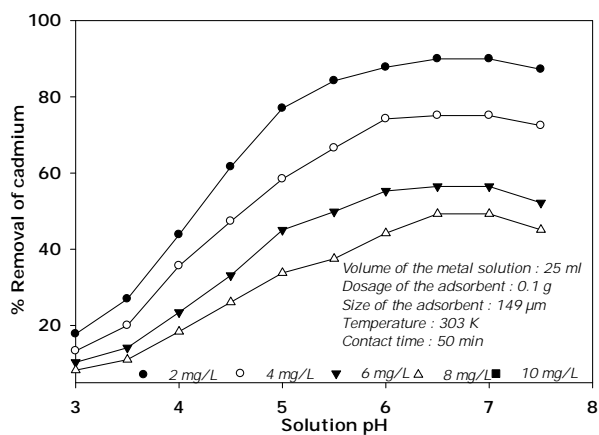Fig. 3: Effect of contact time on cadmium uptake.

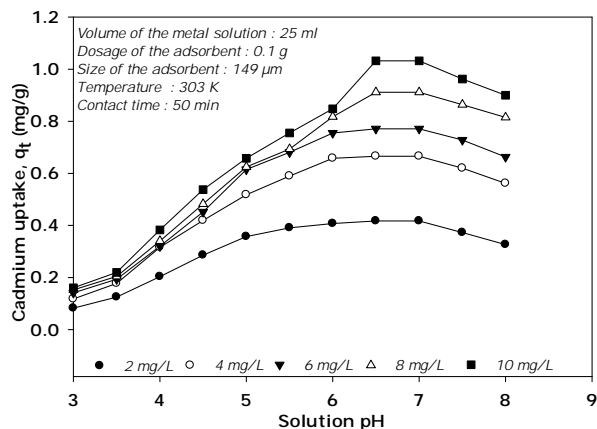
Fig. 4: Effect of solution pH on % removal of cadmium.

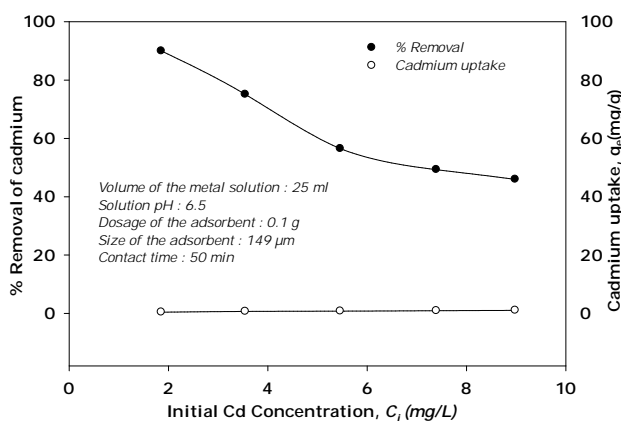Fig. 5: Effect of solution pH on cadmium uptake.

Fig. 6: Effect of initial concentration on % removal and cadmium uptake using *ACBPL* as an adsorbent.
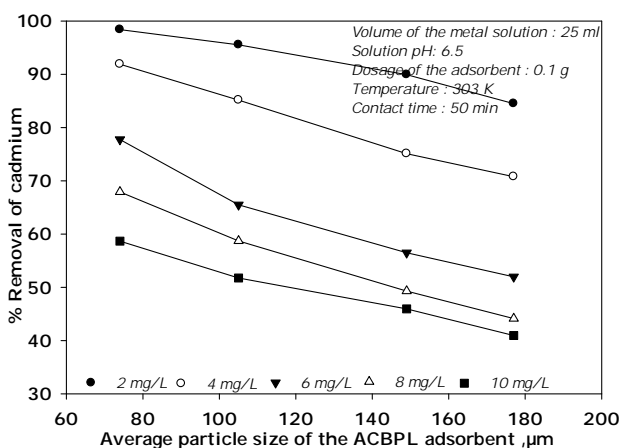

Fig. 7: Variation of % removal of cadmium with average particle size of the adsorbent.

molecule and adsorbent surface. The adsorption efficiency and cadmium accumulated on adsorbent surface were increased from 15.95 to 89.98% and 0.074 to 0.418 mg/g, respectively with increasing contact time from 2 to 50 min for 2 mg/L initial concentration. A similar procedure was repeated for 4, 6, 8 and 10 mg/L metal concentrations of cadmium and the results showed the same contact time of 50 min.

Effect of solution pH: The adsorption process, mostly affected by changing the pH of metal solution. The pH of the solution will affect the surface charges of the adsorbent as well as the degree of ionization of different metal ions in the aqueous solution. The effect of pH on adsorption of Cd at constant temperature (303 K) for varying concentrations and contact time of 50 min is shown in Figs. 4 and 5. It was observed that as the pH of the solution varied from 3 to 5, the adsorption efficiency was very low because repulsion was taking place between the solution and *ACBPL* adsorbent. The adsorption efficiency and cadmium deposition were highly increased with an increase in pH from 5 to 6.5, because the effluent solution was exposed to the negative charges and strongly attracted the Cd^{+2} ions with the adsorbent surface (Leandro et al. 2014). The increase in solution pH from 6.5 to 8 leads to undergo hydrolysis process forming a precipitation of cadmium hydroxide $(Cd(OH)_2)$ and carbonate $(Cd(CO_3)_2)$ in the metal solution (Bohli et al. 2013). Therefore the adsorption efficiency and cadmium deposition increased from 7.27 to 89.97% and 0.034 to 0.418 mg/g respectively, with an increase in solution pH from 2 to 6.5 for 2 mg/L of the initial concentration.

Effect of initial concentration of metal in solution: At low initial metal concentration, numbers of unoccupied active sites are available on the adsorbent surface for the adsorption of metal from the solution. Under these conditions, the ratio of available adsorption active sites to the solute concentration was higher and hence leads to higher adsorption efficiencies. When the initial solute concentration increases, the active sites required for the adsorption of the solute will not be sufficient to accommodate the increased solute concentration in the solution. This led to the available active sites on the adsorbent surface completely filled with the solute and decreased the adsorption efficiency. The Fig. 6 shows that the adsorption efficiency of cadmium was decreased from 89.98 to 45.96% and the amount of cadmium deposited on the surface of the *ACBPL* adsorbent was increased from 0.418 to 1.032 mg/g with an increase in solute concentration. At lower concentrations, all cadmium ions present in the solution could interact with the binding sites and accordingly the rate of adsorption was higher than those at higher initial cadmium ion concentrations (Umar et al. 2015). At higher concentrations, the lower adsorption yield

is due to the saturation of adsorption sites.

Effect of average particle size of adsorbent: The number of active sites per unit volume of adsorbent for solid-liquid interface highly influences the adsorption efficiency and metal deposited on *ACBPL* surface. The contact of the solid-liquid is higher for smaller particles when compared with larger particles, which shows that there was more rapid adsorption and high mass transfer to enhance the adsorption process (Venkatesan et al. 2013). Figs. 7 & 8 show that the removal efficiency of the cadmium adsorption process was increased with the decrease of the particle size of activated carbon of *Bauhinia Purpurea* leaves indicating that the cadmium ion adsorption occurs by a surface phenome-non. It reveals that the percent removal and cadmium accumulated on the activated carbon of *Bauhinia Purpurea* leaves was decreased from 98.41 to 84.51% (at t : 50 min, pH : 6.5 and T : 303 K) and 0.457 to 0.392 mg/L respectively, with an increase in particle size from 74 to 177 μm for 2 mg/L of cadmium concentration in solution.

Effect of dosage of adsorbent: The dosage of the adsorbent is an important parameter to determine the capacity of an adsorbent for a given initial concentration of the effluent. The rate of adsorption of cadmium was increased from 61.31 to 96.34% with an increase in dosage of the *ACBPL* adsorbent from 0.05 to 0.15 g at an initial concentration of 2 mg/L, pH 6.5 and contact time of 50 min as shown in Fig. 9. Similar trends were obtained for the initial metal concentration of 4, 6, 8 and 10 mg/L of cadmium by varying dosage of the *ACBPL* adsorbent from 0.05 to 0.15 g. Fig.10 shows that the amount of cadmium deposited on the *ACBPL* adsorbent surface was decreased from 1.14 to 0.30 mg/g with an increase in dosage of the adsorbent from 0.05 to 0.15 g/25 mL of solution. These results illustrate that the percent removal was increased with an increase in dosage of the *ACBPL* adsorbent, because of an increase in the number of available active sites on the adsorbent surface. The decrease in metal deposition on the adsorbent surface with an increase in dosage of the adsorbent could be due to the reduction in the available surface area of the adsorbent for the adsorption of metals due to overlapping of adsorbent particles (Mona et al. 2014).

Effect of temperature: Temperature plays an important role in the adsorption, since it is an energy dependent mechanism which involves the binding of metal onto cell wall. It alters the cell wall stability, configuration and ionization of the functional groups on the cell wall surface. The above factors parallelly affect the binding sites on the adsorbent cell wall which results in the reduction of metals. These changes may play positive or negative roles in the adsorption of metals. The temperature has two major effects on the
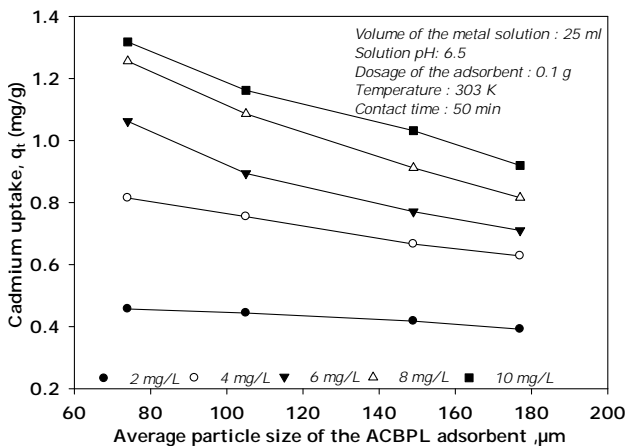
Fig. 8: Variation of cadmium uptake with average particle size of the adsorbent.
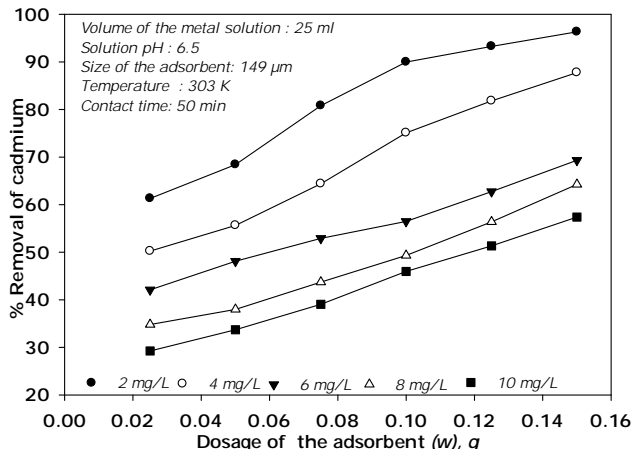

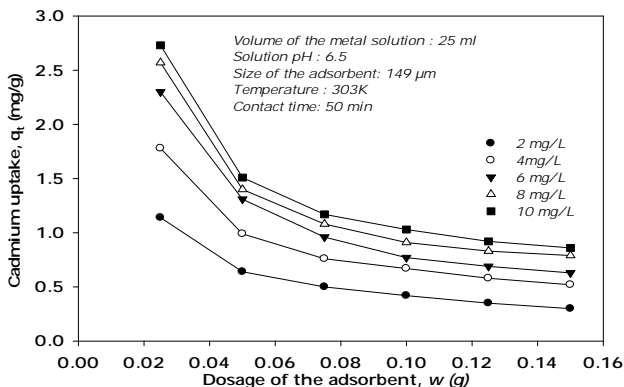Fig. 9: Effect of dosage of the adsorbent on % removal of cadmium.

Fig. 10: Variation of cadmium uptake on the adsorbent surface with dosage of the adsorbent.
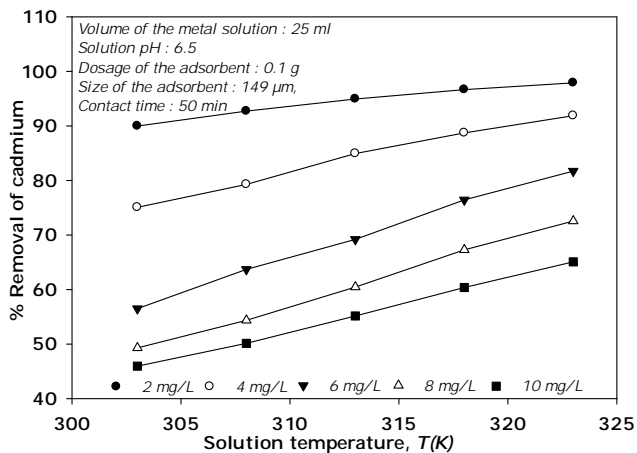

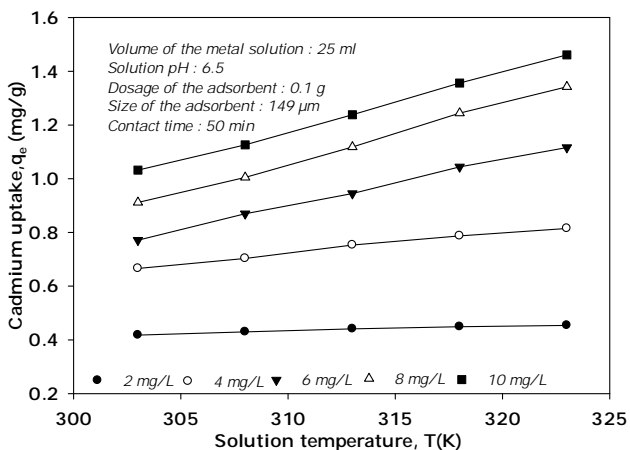Fig. 11: Effect of temperature on % removal of cadmium.

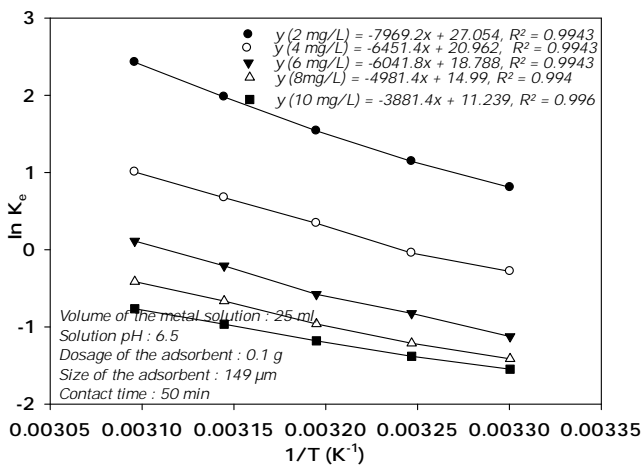Fig. 12: Effect of temperature on cadmium uptake.

Fig. 13: Estimation of thermodynamic energy parameters for cadmium adsorption process.

Table 3: Thermodynamic energy parameters for the adsorption of cadmium using *ACBPL* as an adsorbent.

C_i (mg/L)	ΔH^o (kJ/mol)	ΔS^o (kJ/mol.K)	$-\Delta G^o$(kJ/mol)				
			303K	308K	313K	318K	323K
2	66.255	0.224	7.08	5.87	4.68	3.44	2.22
4	53.636	0.174					
6	50.231	0.156					
8	41.410	0.124					
10	32.260	0.093					

Table 4: The optimal experimental values of the process variables and responses (% removal).

Process variable	Optimum value
Contact time (*t*), min	50
Solution pH	6.5
Initial metal concentration (mg/L)	4
Dosage of the adsorbent (g)	0.15
Solution temperature (K)	313
Maximum % removal	88.76

adsorption process. As the temperature increases, the rate of diffusion of adsorbate molecules across the external boundary layer and internal pores of the adsorbent particle increases (Safa et al. 2009). In addition, changing temperature will change the equilibrium capacity of the adsorbent for particular adsorbate. The rate of adsorption of cadmium and deposition of cadmium onto *ACBPL* adsorbent surface with changing temperature (303K to 323K) were increased from 89.98 to 97.90 % and 0.418 to 0.454 mg/g with an initial concentration of 2 mg/L (pH: 6.5 and t : 50 min) of cadmium solution. When the temperature increases, the pores enlarge, thereby more surface will be available for adsorption, diffusion and penetration of Cd^{+2} ions favouring adsorption and hence there is an increase in the % removal and metal accumulation on the adsorbent surface of cadmium ions. The results are shown in Figs. 11 & 12.

Thermodynamic studies: Thermodynamic energy parameters (ΔH°, ΔS°, and ΔG°) give the evidence of the direction of the adsorption process. The equilibrium constant (K_e) obtained from Eq. 3 was used to evaluate the thermodynamic energy parameters. The values of ΔH°, ΔS° and ΔG° were calculated from the slope and intercept of the linear Vant-Hoff's plot i.e., lnK_e vs $\left(\frac{1}{T}\right)$ as shown in Fig. 13. The estimated thermodynamic energy parameter values of ΔH°, ΔS° and ΔG° are presented in Table 3. The thermodynamic feasibility of cadmium adsorption process described that the % removal of cadmium increased with increasing temperature. According to Eq. (3), the values of ΔH°, ΔS° and ΔG° at various initial concentrations of cadmium with different temperatures are given in Table 3. The adsorption process is endothermic, spontaneous at high temperatures and non-spontaneous at low temperatures. Based on the experimental results, the *ACBPL* adsorbent is efficient for the removal of cadmium at higher temperatures (Awwad et al. 2014). The positive value of ΔS° reflects the high degree of disorder in the adsorbent/adsorbate interface that is formed during the transition.

CONCLUSIONS

The activated carbon prepared from *Bauhinia purpurea* leaves demonstrate a good capacity of adsorption of cadmium, highlighting its potential as an effective adsorbent for the treatment of industrial effluent. The following conclusions could be drawn from the present study on the removal of cadmium from aqueous solutions using adsorption technique:

1. The equilibrium contact time for the adsorption of cadmium onto activated carbon of *Bauhinia purpurea* leaves adsorbent was 50 min. Short equilibrium times indicate that the rate of adsorption is very fast for the cadmium.

2. It was observed that the maximum rate of adsorption was obtained at pH 6.5 for cadmium using activated carbon prepared from *Bauhinia purpurea* leaves. Further increase in solution pH showed that the % removal of cadmium was decreased.

3. The experimental results showed that % removal of cadmium was decreased with an increase in the initial concentration of the solution, but the actual amount of metal accumulated on the adsorbent surface per unit mass of adsorbent was decreased with an increase in the initial concentration of metal solution by using activated carbon of *Bauhinia purpurea* leaves.

4. The percent removal of cadmium was increased and metal uptake decreased with an increase in adsorbent dosage.

5. It was observed that the % removal and metal uptake of cadmium was decreased with an increase of average particle size of the adsorbent.

6. The percent removal and adsorption capacity of cadmium was increased with an increase of solution tem-

perature to indicate the endothermic nature of the adsorption process.

7. The calculated thermodynamic energy parameters (ΔH°, ΔG° and ΔS°) of cadmium adsorption for the same adsorbent with solution temperatures described that the adsorption process is endothermic, spontaneous at high temperatures and non-spontaneous at low temperatures. The positive value of ΔS°, reflecting the high degree of disorder in the solid/solution interface between adsorbate and adsorbent.

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