

# Sorption of Cd(II), Pb(II), and Cr(VI) Metal Ions from Wastewater using Bottom Fly Ash as a Low Cost Sorbent

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## Abstract

Fly ash was used as a low-cost sorbent for the removal of Cd(II), Pb(II), and Cr(VI) ions from aqueous solutions. The influence of pH, initial concentration of metal ions, equilibrium contact time, and temperature was studied using batch sorption experiments. The equilibrium for Cd(II), Pb(II), and Cr(VI) ions was attained in 90, 100, and 120 minute contact times, respectively. The optimum pH values for Cd(II), Pb(II), and Cr(VI) removal were found to be 8.5, 6.5, and 2, respectively. The sorption equilibrium data for present sorbent-sorbate system were tested by both Langmuir and Freundlich models. The sorption kinetics fitted the first-order kinetic model. The values of  $\Delta G^\circ$  and  $\Delta H^\circ$  for Cd(II) and Pb(II) ions indicate that the adsorption process is spontaneous and exothermic, while for Cr(VI) ions it is endothermic.

**Keywords:** Cd(II), Pb(II), Cr(VI), Fly ash, Adsorption, Langmuir and Freundlich isotherms.

*JEPS (2009), Vol. 3, pp. 124 – 132.*

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## 1. Introduction

The presence of heavy metals in the environment is a major concern due to their toxicity and non-biodegradability. Consequently, improved and innovative methods of wastewater treatment are continuously being developed to deal with removal of these components. Numerous methods employed for separation and recovery of heavy metals from aqueous solutions include precipitation, ion exchange, evaporation, reverse osmosis, electrodialysis, membrane separation, solvent extraction, phytoextraction [1-4]. However, these methods have shown limited success. Although traditional solvent extraction processes have been widely utilized, they have less applicability toward dilute solutions and are economically infeasible because of the loss of solvent after use. Ion exchange resins can remove metal ions substantially; however, they do not show mechanical strength, due to swelling of the polymeric skeleton, and low selectivity. Many chelating resins have been reported but show limited applicability because of their poor hydrophilicity, small surface area, slow rate of adsorption, and lack of regenerability. Adsorption, on other hand, is a most promising technique employing solid sorbents to remove heavy toxic metal ions from wastewaters. The adsorption process provides an attractive alternative for the

treatment of contaminated waters, especially if the sorbent is inexpensive and does not require an additional pretreatment before its application. Fly ash is a hazardous voluminous solid waste by-product from coal-fired Power Plants. Currently, approximately 100 million tons of fly ash is being generated annually in India and only 15% of the quantity generated is gainfully utilized. Disposal of a growing amount of fly ash is a serious environmental problem and alternate utilization of the material has to be developed. The fineness of the fly ash particles and the inherent large surface area together with residual carbon content makes it a good choice for use as a potential low-cost adsorbent. Literature survey indicates that fly ash may be utilized beneficially for the removal of metal ions from wastewaters [5-10]. In this paper, the adsorption properties of the coal fly ash with respect to cadmium(II), lead(II), and chromium(VI) in solution were studied. These metals were chosen due to their solubility over a wide range of pH and their presence in several industrial wastewaters.

## 2. Materials and Methods

Samples of fly ash were collected from Indraprastha Thermal Power Plant in New Delhi, India. They were washed several times with distilled water and dried at 110 °C for 24 h, sieved to desired particle sizes (75, 150, and 300  $\mu\text{m}$ ), and finally stored in vacuum desiccators. Stock

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solutions of the metal ions were prepared in double distilled water. The chemical analysis was done following standard methods [11,12]. Surface area was determined by the N<sub>2</sub>-BET method employing a Quantasorb surface analyzer, QS/7. Porosity was measured by mercury intrusion porosimeter (model Micrometric 9310), while the mean particle diameter was determined with the Laser particle size analyzer (Malvern 3600). The FTIR spectrum of the fly ash sample was recorded on a Perkin Elmer spectrophotometer model 1620, while X-ray diffraction (XRD) was recorded with Philips Diffraction Unit model 1830 using Cu-K $\alpha$  radiation. Absorbance measurements were made with a Jasco-7800 UV-VIS spectrophotometer. pH measurements were made using a pH-meter. pH of the solution was adjusted, wherever required, using 0.1 M HCl or NaOH. The scanning electron micrograph was obtained with a scanning electron microscope (Model JEOL JSM 840).

### 2.1 Reagents

The stock solutions of cadmium(II), lead(II), and chromium(VI) were prepared using cadmium chloride, lead nitrate, and potassium dichromate (all Merck, India).

### 2.2 Batch adsorption studies

Batch adsorption studies were carried out using 1.0 g of adsorbent with 50 mL of solutions containing metal ions of desired initial concentrations at constant temperature in a 250 mL Erlenmeyer flask. The flasks were shaken for a predetermined time interval in a constant temperature water bath shaker. The adsorbent and solution were separated by centrifugation at 1,400 rpm. The residual concentrations of Cd<sup>2+</sup> and Pb<sup>2+</sup> in the centrifugate were determined spectrometrically by the dithizone method at 518 and 510 nm, while that of Cr<sup>6+</sup> by the diphenylcarbazide method at 540 nm. The kinetic studies were carried out with the same equipment and similar conditions. The sample mass was 1.0 g and the volume of the metal solution was 50 mL. The original pH of the metal ion solution was adjusted to the desired value by adding required quantities of decimolar solutions of sulphuric acid or sodium hydroxide. Effects of initial dye concentration, particle size, and pH at different agitation times were also studied following the same experimental procedures. All measurements were at least duplicated to ensure accuracy.

## 3. Results and Discussion

### 3.1 Characterization of the adsorbent

The adsorbent was characterized by chemical analysis, IR spectrum, and X-ray diffraction (XRD). The results of chemical analysis revealed the following composition of the fly ash (%): SiO<sub>2</sub>, 60.10; Al<sub>2</sub>O<sub>3</sub>, 18.60; CaO, 6.30; Fe<sub>2</sub>O<sub>3</sub>, 6.40; MgO, 3.60, and loss on ignition (LOI), 4.90

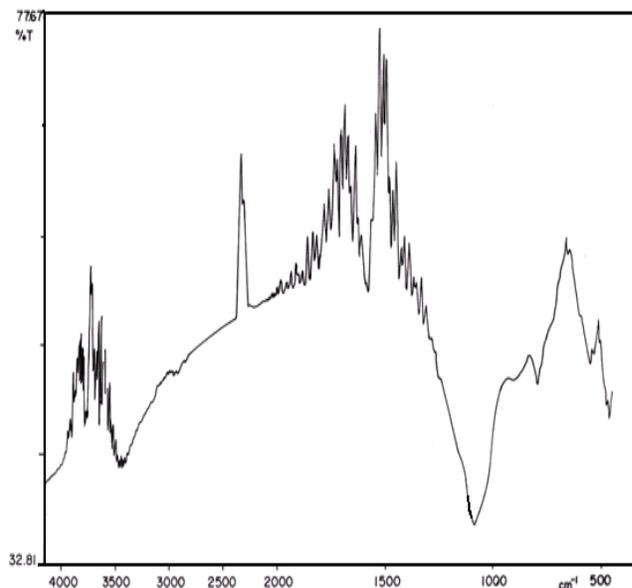


Figure 1: FT-IR spectrum of fly ash.

wt% and surface area, 40.16 m<sup>2</sup>g<sup>-1</sup>; porosity, 0.43; and bulk density, 3.51 g/cm<sup>3</sup>. The FT-IR spectrum of fly ash (Figure 1) displayed a number of absorption peaks, reflecting the adsorbent's complex nature. It showed distinct absorption bands at 558 cm<sup>-1</sup>, which may be ascribed to Si-O-Al stretching vibrations, a characteristic of quartz. The adsorption bands at 3,454-3,891 cm<sup>-1</sup> due to O-H stretching vibrations indicated the presence of kaolinite. The strong band at 1,481 cm<sup>-1</sup> suggested the presence of Fe-O bend and at 1,661 cm<sup>-1</sup> indicated the presence of Ca-O/Mg-O bend. The XRD diagram of fly ash is shown in Figure 2. X-ray diffraction (XRD) studies indicated the presence of haematite [ $d(\text{\AA})$  2.6876, 1.6970],

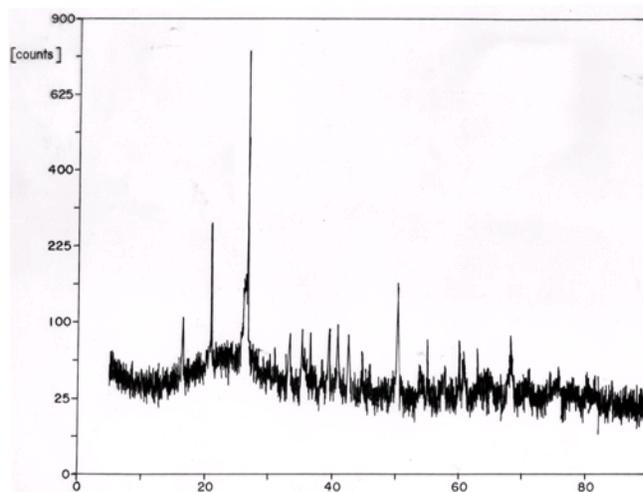


Figure 2: XRD diagram of fly ash.

kaolinite [ $d(\text{\AA})$  2.3384], calcite [ $d(\text{\AA})$  2.2787, 2.1241], and quartz [ $d(\text{\AA})$  4.2408, 3.4100, 1.8152] minerals in the ash sample. The scanning electron micrograph (SEM) of fly ash [13] revealed the presence sporadically of particles of irregular size.

### 3.2 Effect of contact time and concentration

The results show that the extent of adsorption increases rapidly in the initial stages and becomes slow in the later stages until saturation. It is clear from the results (Table 1 and Figure 3) that the time required to attain equilibrium of

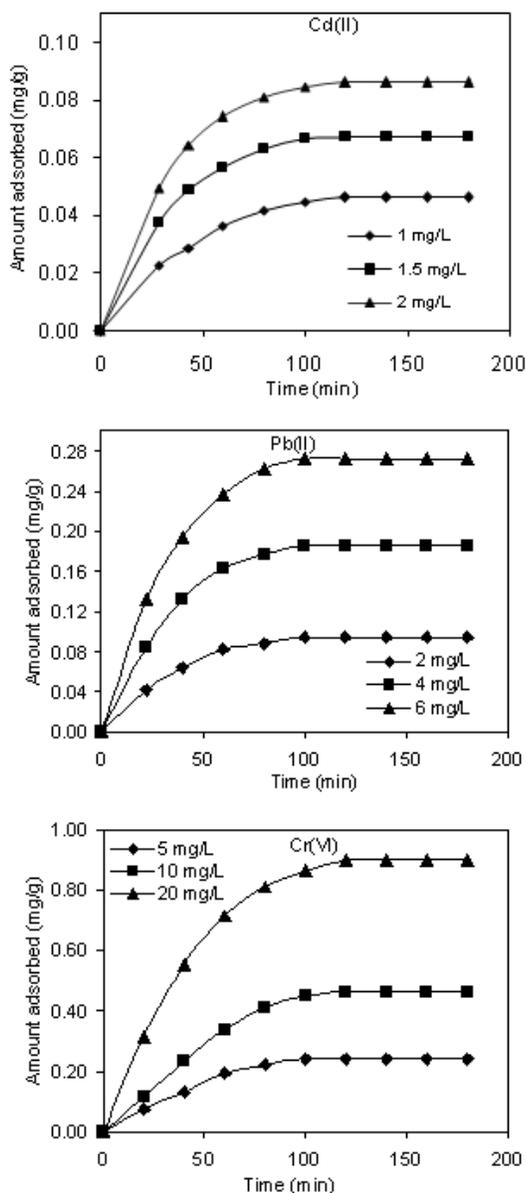


Figure 3: Effect of time and concentration on removal of metal ions.

adsorption of Cd(II), Pb(II), and Cr(VI) on fly ash is 90, 100, and 120 minutes, respectively, at all concentration ranges. This indicates that the equilibrium time is independent of the initial sorbate concentration. However, the extent of removal is highly dependent of concentration. The percentage removal decreased with increase in initial concentration of metal ions. For instance, the percentage removal of Cd(II) at pH 8.5 decreased from 88.00 to 74.00 when concentration was increased from 1.00 to 2.00 mg/L. Similarly, the percent uptake of Pb(II) at pH 6.5 by fly ash decreased from 83.00 to 70.33. Pb(II) concentration of 2.00 to 6.00 mg/L while that of Cr(VI) at pH 2.0 decreased from 90.80 to 86.40 at Cr(VI) concentration of 5.00 to 20.00 mg/L at 30 °C. This observation may be ascribed to reduction in immediate solute (metal ions) sorption, owing to lack of available active sites required for high initial concentration of metal ions.

### 3.3 Sorption kinetics

It was found that the kinetic data best fitted to the following kinetic equation for adsorption.

$$\log (q_e - q) = \log q_e - K_{ad} \times t / 2.303$$

where  $q_e$  and  $q$  (mg/g) are the amounts of metal ions at equilibrium and at any time  $t$ , respectively. The  $K_{ad}$  values at different concentrations are calculated from the slope of the linear plots of  $\log (q_e - q)$  vs.  $t$  (Figure 4). The kinetic parameters revealed that the adsorption followed the first-order kinetic rate model.

### 3.4 Isotherm models

The adsorption data were analyzed by different sorption isotherms, namely, Langmuir and Freundlich.

The adsorption data for different metal ion concentrations at various temperatures followed the rearranged Langmuir isotherm [14]:

$$C_e / q_e = 1/Q_0 b + C_e / Q_0$$

where  $C_e$  is the equilibrium concentration (mg/L) of metal in the solution,  $q_e$  the amount of metal ion adsorbed at equilibrium (mg/g), and  $Q_0$  and  $b$  are Langmuir constants related to adsorption capacity and energy of adsorption, respectively. The maximum sorption capacity ( $Q_0$ ) represents monolayer coverage of sorbent with sorbate while  $b$  represents enthalpy of sorption. The values of  $Q_0$  and  $b$  are obtained from the slopes and intercepts of the linear plots of  $C_e/q_e$  vs.  $C_e$  (Figure 5). The dimensionless factor,  $R_L$ , is calculated from the expression,  $R_L = 1/(1+bC_i)$ . The Langmuir parameters,  $r^2$  and  $R_L$  values are given in Table 2. The adsorption capacity,  $Q_0$ , for cadmium and lead decreased from 0.969 to 0.172 mg/g and

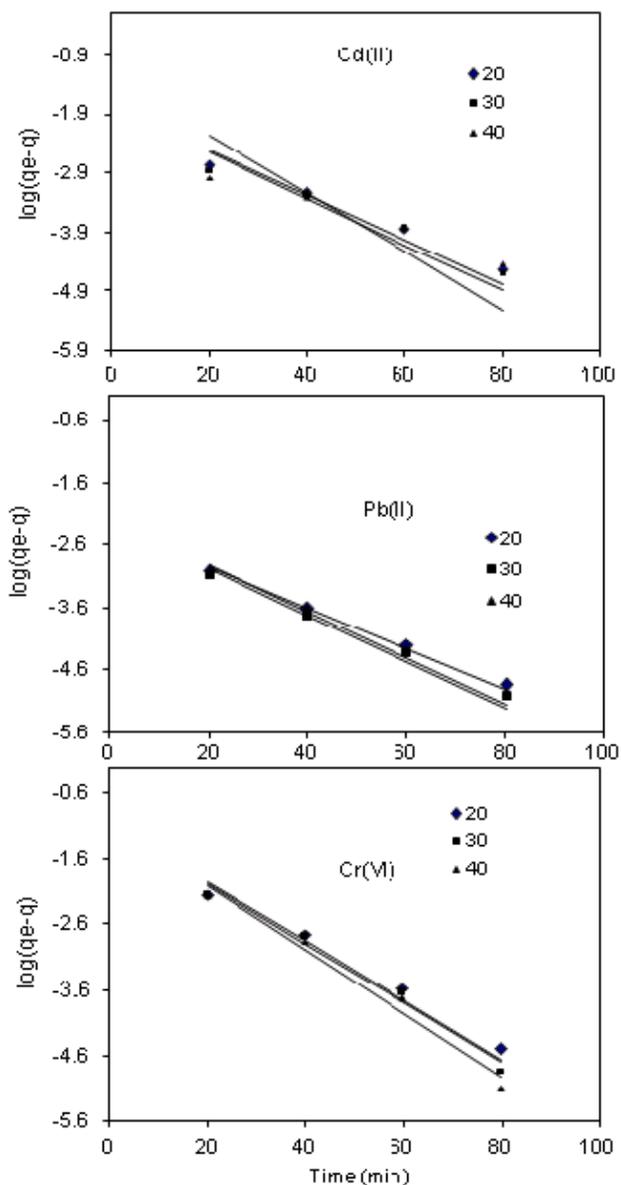


Figure 4: Lagergren Plot for sorption of metal ions.

Table 1. Effect of contact time and concentration				
Metal Ions	Initial Equilibrium Concentration (mg/L)	Amount Adsorbed (mg/g)	Removal %	Time (min.)
Cd(II)	1.0	0.044	88.00	90
	1.5	0.058	77.33	90
	2.0	0.074	74.00	90
Pb(II)	2.0	0.083	83.00	100
	4.0	0.148	74.10	100
	6.0	0.211	70.33	100
Cr(VI)	5.0	0.227	90.80	120
	10.0	0.445	89.00	120
	20.0	0.864	86.40	120

Table 2. Langmuir and Freundlich constants of adsorption					
Langmuir constants					
Sorbate	Temp. (°C)	Q <sub>0</sub> (mg/g)	b	r <sup>2</sup>	R <sub>L</sub>
Cd(II)	20	0.969	0.0524	0.9606	0.0394
	30	0.218	0.2595	0.9471	0.0529
	40	0.172	0.2940	0.9972	0.0028
Pb(II)	20	1.234	0.0439	0.9106	0.0894
	30	0.892	0.0529	0.9605	0.0395
	40	0.343	0.1525	0.9720	0.0280
Cr(VI)	20	6.227	0.0074	0.9188	0.0812
	30	12.984	0.0037	0.9948	0.0052
	40	14.664	0.0035	0.8251	0.1749
Freundlich constants					
Sorbate	Temp. (°C)	K <sub>F</sub>	1/n	r <sup>2</sup>	
Cd(II)	20	0.0482	0.9350	0.9995	
	30	0.0452	0.7303	0.9967	
	40	0.0391	0.7114	0.9999	
Pb(II)	20	0.0540	0.8792	0.9958	
	30	0.0478	0.8425	0.9998	
	40	0.0498	0.6780	0.9829	
Cr(VI)	20	0.0504	0.9249	0.9999	
	30	0.0505	0.9626	0.9998	
	40	0.0525	0.9622	0.9999	

from 1.234 to 0.343 mg/g, respectively as the solution temperature was increased from 20 to 40 °C, thereby indicating an exothermic nature of the process. However, in the case of chromium, the Langmuir constant, Q<sub>0</sub>, increased from 6.227 to 14.664 mg/g as the solution thermodynamic parameters. The R<sub>L</sub> values between 0 and

1 (Table 2) indicate favorable adsorption for all the initial concentrations and temperatures studied. The Langmuir adsorption isotherms effectively described the adsorption data with all r<sup>2</sup> values between 0.8251 and 0.9948. The applicability of the Langmuir isotherm for the present system suggests the formation of monolayer covering on the surface of the sorbent.

### 3.5 Freundlich isotherm

The Freundlich adsorption isotherm [15] was applied for the adsorption of metal ions by fly ash. The Freundlich isotherm is represented by the equation:

$$\log q_e = \log K_F + 1/n \log C_e$$

where  $K_F$  and  $1/n$  are Freundlich constants related to adsorption capacity and intensity of adsorption, respectively. A plot of  $\log q_e$  vs.  $\log C_e$  (Figure 6) gives the values of  $K_F$  and  $1/n$ , which are determined from the intercept and the slope of the linear plots. Linear plots of  $\log q_e$  vs.  $\log C_e$  show that the adsorption of metal ions onto fly ash followed the Freundlich isotherm model. The calculated values of Freundlich constants,  $K_F$  and  $1/n$ , are given in Table 2. The adsorption is considered favorable when  $1/n$  values are in the range 0-1.

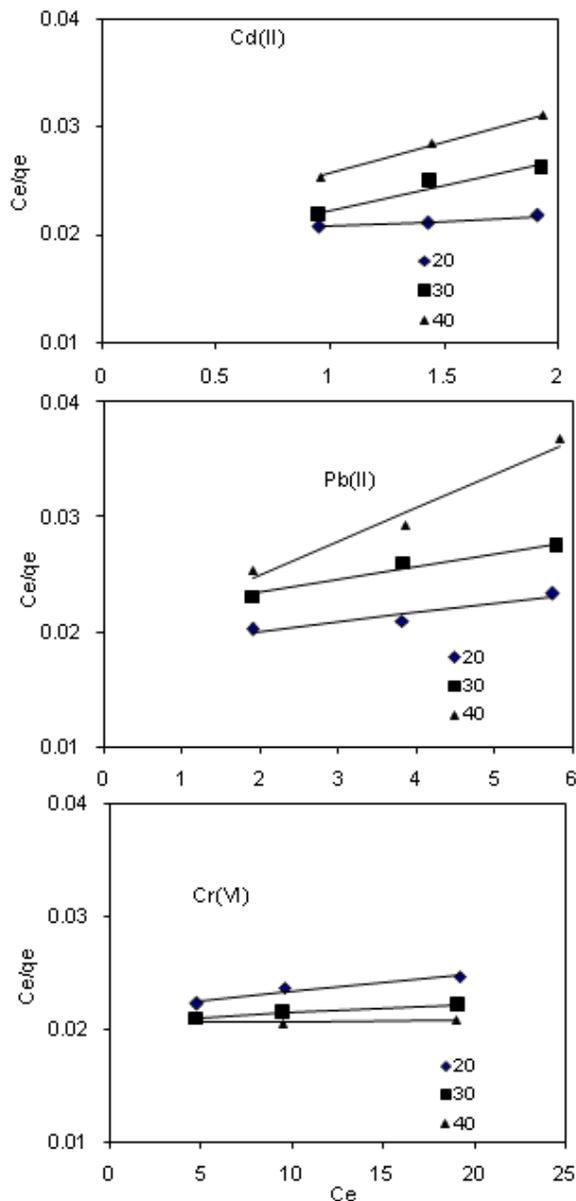


Figure 5: Langmuir plots for metal ion sorption

### 3.6 Effect of Temperature

The results of the studies on the influence of temperature on metal ions adsorption are presented in Figure 7. A study

of the temperature dependence of adsorption reactions gives valuable information about the enthalpy change during sorption. The effect of temperature on sorption rate was studied by carrying out experiments at 20, 30, and

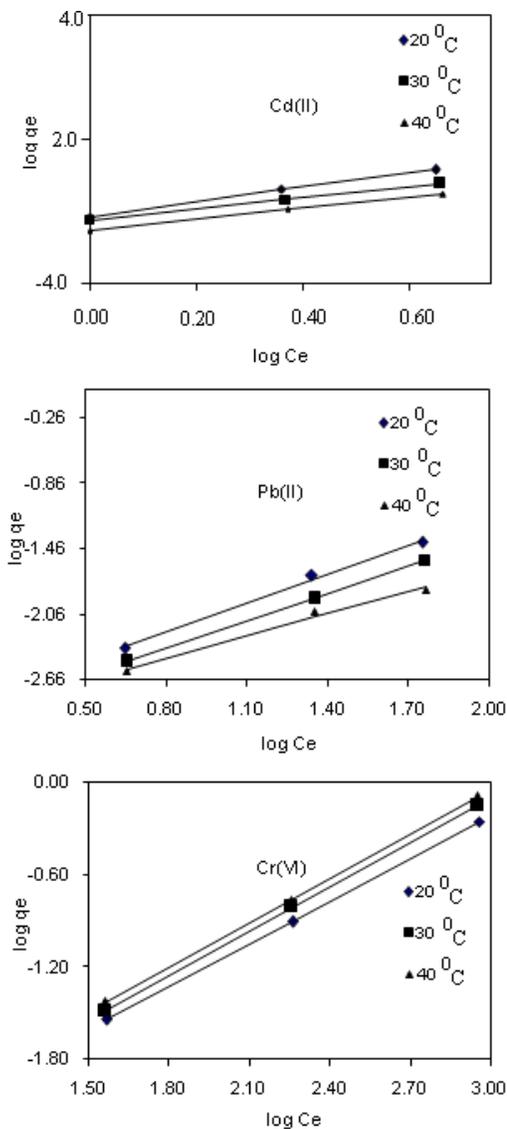


Figure 6: Freundlich plot for metal ions sorption onto fly ash.

40°C. The sorption of cadmium and lead ions on to fly ash decreased when temperature was increased from 20 to 40 °C, indicating that the adsorption process was exothermic in nature. The sorption of Cr(VI) by fly ash, however, increased with increase in temperature from 20 to 40 °C, indicating better adsorption at a higher temperature, so the reaction was endothermic. The increase in the amount of chromium ions sorbed at equilibrium with an increase in temperature may be either due to acceleration of some

originally slow adsorption steps or to the creation of some new sites on the sorbent surface [16].

### 3.7 Thermodynamic parameters

The variation in sorption with respect to temperature may be explained on the basis of thermodynamic parameters—namely changes in standard free energy ( $\Delta G^\circ$ ), standard enthalpy ( $\Delta H^\circ$ ), and standard entropy ( $\Delta S^\circ$ ) for adsorption of Cd(II), Pb(II), and Cr(VI).

$$\Delta G^\circ = -RT \ln K_1$$

$$\Delta H^\circ = -R (T_2 T_1 / T_2 - T_1) \times \ln K_2 / K_1$$

$$\Delta S^\circ = \Delta H^\circ - \Delta G^\circ / T$$

The values of thermodynamic parameters thus calculated are recorded in Table 3. It is obvious that the negative values of free energy change in all the systems indicate the spontaneous nature of the process. In such cases, the adsorptive forces are strong enough to cross over the potential barrier. The negative values of standard enthalpy change for Cd(II) and Pb(II) sorption suggest the exothermic nature of the process, whereas the positive value of enthalpy change indicates the endothermic nature of Cr(VI) removal. The negative values of  $\Delta S^\circ$  in the case of Cd(II) and Pb(II) sorption indicates faster interaction during the forward reaction (sorption). In the sorption process, the loss in degree of freedom of sorbate ions due to association, fixation, or immobilization of Cd(II) and Pb(II) is expected to be responsible for negative entropy change. However, the positive entropy change in the case of Cr(VI) suggests an increased randomness at the solid-solution interface during sorption. The sorbed species, which are displaced by the sorbate species, gain more translational entropy than is lost by the sorbate ions, thus allowing for the prevalence of randomness in the system [17,18].

**Table 3. Thermodynamics of adsorption**

Sorbate	Temp. (°C)	$-\Delta G^\circ$ (kJ/mol)	$-\Delta H^\circ$ (kJ/mol)	$-\Delta S^\circ$ (J/Kmol)	$K_{ad} \times 10^{-2}$ (min <sup>-1</sup> )
Cd(II)	20	7.503	1.334	71.10	3.76
	30	8.214	1.315	70.48	4.92
	40	8.919	-	-	3.87
Pb(II)	20	7.335	9.587	57.73	3.27
	30	7.912	7.245	50.00	3.74
	40	8.412	-	-	3.75
Cr(VI)	20	7.562	4.199	11.47	4.53
	30	7.676	4.956	8.97	4.88
	40	7.766	-	-	4.86

### 3.8 Effect of pH

The results of the sorption studies of Pb(II) and Cd(II) ions

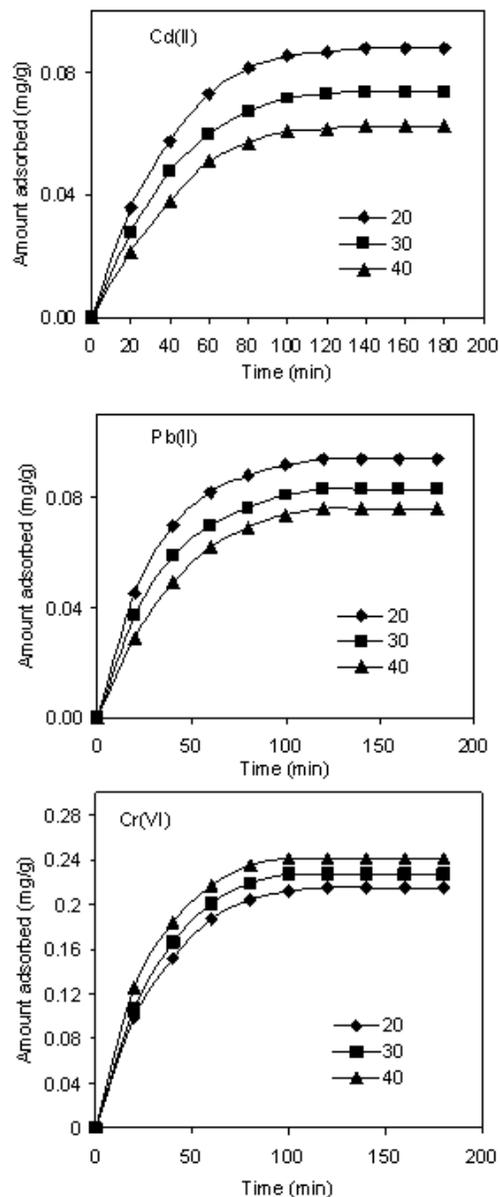


Figure 7: Effect of temperature on sorption of metal ions.

onto fly ash at pH values between 2.0 and 10.5 are given in Figure 8. The mechanism of sorption of various anionic and cationic species on such sorbents has been explained on the basis of competitive adsorption of  $H^+$  and  $OH^-$  ion with sorbents. It is a common observation that metallic oxide sorbents are favorable at low pH range due to the presence of  $H^+$  ion on the surface. However, in high pH range, due to accumulation of  $OH^-$  on the surface, cationic sorption is favored [19]. The sorbents used here were rich



anions of Cr(VI) that may be present in water are  $\text{Cr}_2\text{O}_7^{2-}$  and  $\text{HCrO}_4^-$ . The  $\text{HCrO}_4^-$  is the predominant species between pH 1.5 and 4.0, and  $\text{HCrO}_4^-$  and  $\text{CrO}_4^{2-}$  exist in equal amounts at pH 6.5. Below pH 1.5,  $\text{H}_2\text{CrO}_4$  is the dominating species. Further, at pH 2.0 the proton to  $\text{HCrO}_4^-$  concentration ratio is 1.0 and this ratio increases below pH 3.0.



The higher adsorption at a pH value around 2.0 or less indicates that the sorbent, in the present study, adsorbs  $\text{HCrO}_4^-$  anion and the sorption has been observed to increase as the proton to Cr(VI) ratio is one or more than one [25]. At lower pH values, the higher hydrogen ion concentration neutralizes the negative charges at the surface of internal pores, the structural hindrances to transport are reduced, and a few more new energetic sorption sites are developed. All these factors tend to increase the sorption with increasing hydrogen ion concentration. Under low pH all hexavalent chromium species may get converted into dichromate ions and the hexavalent chromium in this form may start oxidizing the sorbent surface and/or may reduce to trivalent chromium. When the hexavalent chromium concentration exceeds the proton activity, the total chromium sorption happens to be a combination of proton-hexavalent chromium sorption and hexavalent chromium hydrolytic sorption [26]. Therefore, it can be concluded that with an increase in hydrogen ion concentration, the negative charge over the surface of sorbent is neutralized and the surface provides a positive charge for  $\text{HCrO}_4^-$  anions to get adsorbed. It also makes available in the system the most favorable form of the sorbate molecules [27] i.e.,  $\text{HCrO}_4^-$ .

#### 4. Conclusion

The present study revealed that the adsorption of metal ions onto fly ash was dependant on contact time, initial metal ion concentration, adsorbent dose, and solution pH. The effect of temperature on the sorption of Cd(II), Pb(II), and Cr(VI) suggests that the increase in temperature lowers the extent as well as the rate of sorption of Cd(II) and Pb(II), whereas a high temperature favors the removal of Cr(VI). The sorption equilibrium data for the present sorbent-sorbate system followed Langmuir and Freundlich isotherm models. The maximum removal of Cd(II), Pb(II), and Cr(VI) occurs at pH 8.5, 6.5, and 2.0, respectively.

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