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Removal of Cr(VI) and Hg(II) from Aqueous Solutions Using Fly Ash and Impregnated Fly Ash

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ABSTRACT

The potential of impregnated fly ash (IFA), as low-cost adsorbent, was evaluated for the removal of Cr(VI) and Hg(II) from aqueous solutions by using batch equilibration technique. Effect of various physicochemical parameters such as initial concentration, contact time, temperature, and pH on the adsorption was investigated in order to optimize the conditions for adsorption. IFA showed enhanced tendency for adsorption. Adsorption efficiency was found to be dependent on the initial concentration and the adsorption behavior followed the Langmuir adsorption model. The adsorption capacity (Q_0) of FA, IFAAl, and IFAFe for Cr(VI) was found to be 1.379, 1.820, and 1.667 mg g^{-1} and that of Hg(II) was

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11.00, 12.50, and 13.40 mg g^{-1}. Rate constants were evaluated in terms of a first-order kinetic model. Thermodynamic parameters such as free energy (ΔG°), enthalpy (ΔH°), and entropy (ΔS°) for the adsorption process were calculated. The adsorption was found to be endothermic in both the cases.

Key Words: Langmuir adsorption model; First-order kinetic model; Thermodynamic parameters.

INTRODUCTION

The environment and all the life forms on earth face a very serious threat as a result of heavy metal pollution due to rapid industrialization and the increase in the world population. Unlike organic pollutants, the majority of which are susceptible to biological degradation, metal ions are not easily converted into harmless end products. The metals that cause serious concern include Cr, Hg, Zn, Ni, Cu, and Cd, which are commonly associated with pollution and toxicity problems.[1] Chromium, which is known to be a carcinogen, has various applications in a variety of industries such as textiles, fertilizers, leather tanning, electroplating, and metal finishing. Chromium can be present in solution in various forms. Electroplating and metal finishing wastes primarily contain Cr(VI) species, whereas textile and tanning waste contain either Cr(VI) or Cr(III).[2] Chromium-(VI) is about 100 times more toxic than Cr(III).

According to the US Environmental Protection Agency, the permissible limit of Cr(VI) in effluent is 0.05 mg L^{-1}. In India, about 150,000 ton $year^{-1}$ of Cr-containing sludge is released into the environment from tannery effluents alone.[3] The toxicological effects of mercury are neurological damage, including irritability, paralysis, blindness or insanity, and chromosome breakage.[4] The health hazards due to the toxic effect of mercury at Minamata, Japan, and in North America are well known. The World Health Organization (WHO) has fixed the maximum allowable limit of mercury in potable water at 1 μg d^{-1} and the maximum permissible tolerance level of intake by humans at 43 μg d^{-1}.[5,6] To curtail heavy metal pollution, many processes have been developed, which include chemical precipitation, electrode deposition, solvent extraction, ion-exchange, activated carbon adsorption,[7] and biological methods.[8] The main limitations of these techniques are their low efficiency in metal ions removal at trace level and the high cost. Hence, adsorption is reported to be the most effective method for this purpose, at low concentration.[9]

In recent years, research interest has increased in terms of production of low-cost alternatives to activated carbon, which remains an expensive material in spite of its prolific use. Both powdered and granular activated carbon have been used for the adsorptive removal of Pb, Cd, Hg(II), and similar heavy metals.[10] Fly ash (FA) is a solid waste produced in large quantities during the burning of municipal waste, fossil fuel, and coal in thermal power plants. The huge deposits of FA around thermal power plants pose problems in its disposal. Besides its utilization in preparing bricks, portland cement, etc., it has also been used in wastewater treatment.[11] A potential advantage of FA as an adsorbent is that it could easily be solidified after metal ion adsorption by treatment with lime and water. Thus, FA may serve as an effective and inexpensive fixation agent for heavy metal removal from water. For example, when metal-loaded FA was added up to 20% by mass to portland cement-based formulations, the fixed metal ions did not leach out from the solidified concrete blocks over extended periods.[10,12]

Previous studies[13–15] have shown that Al/Fe species are very efficient adsorbing chemicals for potable water treatment. The combination of FA and oxides of Al/Fe can form a new type of adsorbent with optimum adsorption properties. The aim of the present investigation is to compare sorption of Cr(VI) and Hg(II) on FA and fly ash impregnated (IFA) with Al or Fe in the aqueous media and the effect of impregnation on improving the efficacy of the surface for Cr(VI) and Hg(II) removal.

EXPERIMENTAL

All chemicals used were of analytical grade (E. Merck, India). Stock solutions of $K_2Cr_2O_7$ and $HgCl_2$ were prepared in double-distilled water. pH measurements were carried out with an Elico digital pH meter (model No LI-120). The concentrations of Cr(VI) and Hg(II) were determined on a GBC-911A (Australia) ultraviolet (UV)-visible spectrometer. The average particle size was measured with the COULTER LS 230 particle size analyzer. Infrared spectra were recorded on a FT-IR spectrometer (JASCO FT-IR-420). Scanning electron microscopy (SEM) studies were carried out using a Philips XL-30 (Netherlands) instrument. Surface area measurements were done by BET adsorption method using a surface area unit fabricated at ACC-RCD Thane, India.

Material Development

FA was obtained from the Thermal Power Plant of Century Rayon, Shahad (Maharashtra). This was passed through standard test sieve No. 150, which has an average particle diameter of 100 μm. The uniform sample of FA, thus, obtained was impregnated with 0.1 M $Al(NO_3)_3$ or 0.1 M $Fe(Cl)_3$.[16] These are denoted as IFAAl and IFAFe, respectively.

Kinetic Studies

For kinetic studies, experiments were carried out by shaking 1 g of the adsorbent (FA, IFAAl, and IFAFe) with 300 mL of Cr(VI) solution or 500 mL of Hg(II) solution containing known concentrations of metal ions, in a fully baffled reactor fitted with an overhead stirrer (100 rpm) and a temperature controller ($\pm 2^{\circ}$C). The pH of the solution was adjusted initially using 0.1 M NaOH and HCl, and maintained throughout. The solutions were shaken for total period of 1 hr. After definite intervals of time, the adsorbents were separated from the solution by centrifuging (8000 rpm) for 10 min and the concentrations of Cr(VI) and Hg(II) were determined in the supernatant using diphenyl carbazide set at a wavelength of 540 nm and zincon method set at wavelength of 600 nm.[17,18]

Adsorption Studies

Batch experiments were carried by shaking 0.1 g of the adsorbent (FA, IFAAl, and IFAFe) with 100 mL of aqueous solution using a series of Erlenmeyer flasks of 250 mL capacity. The effect of pH was studied by adjusting the pH of the solution with 0.1 M HCl and 0.1 M NaOH. After shaking for 1 hr on the orbital shaker (100 rpm), the solutions were centrifuged, the final pH of the solution was measured, and the supernatant liquid was analyzed for Cr(VI) and Hg(II) spectrophotometrically.

RESULTS AND DISCUSSION

Characterization of the Adsorbents

The FA was found to be stable in water, dilute acids, and alkali. Neither of the metal ions (Al^{3+}, Fe^{3+}) was eluted from IFA with cold or hot water or cold dil HCl but got eluted with hot dil HCl. The characteristics of FA, IFAAl, and IFAFe are given in Table 1.[19] The FT-IR spectrum (Fig. 1) of FA (A) shows strong bands at 1260, 1100, and 800 cm^{-1}, which could be attributed to Si–O stretching vibrations, characteristic of quartz. The absorption bands at 3696 and 3670 cm^{-1} indicate the presence of Kalonite. The bands at 3620, 3542, and 3409 cm^{-1} are attributed to $-$OH stretching vibrations. It can be seen from the FT-IR spectrum of IFAAl (B) that the band at 1260 cm^{-1} that was present in FA has disappeared and the intensity of the bands at 1100 and 800 cm^{-1} has considerably decreased. This can be attributed to the replacement of surface Si–OH by $Si{-}OAl^{2+}$. Also two new bands are observed, a weak band at 1417 $cm^{-1}$and another at 512 cm^{-1}, which can be attributed to Al–O vibration.[20,21] The SEM photomicrographs of untreated FA and

Table 1. Characteristics of FA, IFAAl, and IFAFe.

Constituents	Weight (%)
SiO_2	52.0
Al_2O_3	27.0
Fe_2O_3	6.0
MgO	0.70
CaO	0.11
TiO_2	1.01
Loss on ignition	13.18
Mean particle size	
FA	47.90 μm
IFAAl	55.87 μm
IFAFe	50.11 μm
Surface area	
FA	$3.12\,m^2\,g^{-1}$
IFAAl	$4.43\,m^2\,g^{-1}$
IFAFe	$3.73\,m^2\,g^{-1}$

IFAAl are shown in Figs. 2 and 3, respectively. Figure 2 shows that FA has a rough surface and it also clearly reveals the porous nature of FA. Figure 3 shows that FA has been completely modified with aluminum hydroxide. As a result, the surface texture of FA has changed after the treatment and the surface has become smoother due to aluminum hydroxide coating.

Effect of Contact Time and Initial Metal Ion Concentration

The adsorption experiments were carried out in the concentration range of 2–10 mg L^{-1} for Cr(VI) at pH 2.0 and 20–40 mg L^{-1} for Hg(II) at pH 5.8 at 30°C. The equilibrium was established in 30 min for Cr(VI) and 20 min for Hg(II) and the equilibration period was found to be independent of concentration. As the initial concentration of Cr(VI) increased, the amount adsorbed on FA, IFAAl, and IFAFe increased from 0.30 to 1.10 mg g^{-1}, 0.41 to 1.50 mg g^{-1}, and 0.39 mg g^{-1} to 1.23 mg g^{-1}, While the amount of Hg(II) adsorbed increased from 5.60 to 10.10 mg g^{-1}, 5.90 to 11.4 mg g^{-1}, and 6.40 to 12.30 mg g^{-1}.

Effect of Temperature

The adsorption studies were carried out at three different temperatures: 30°C, 45°C, and 60°C. With increase in temperature from 30°C to 60°C,

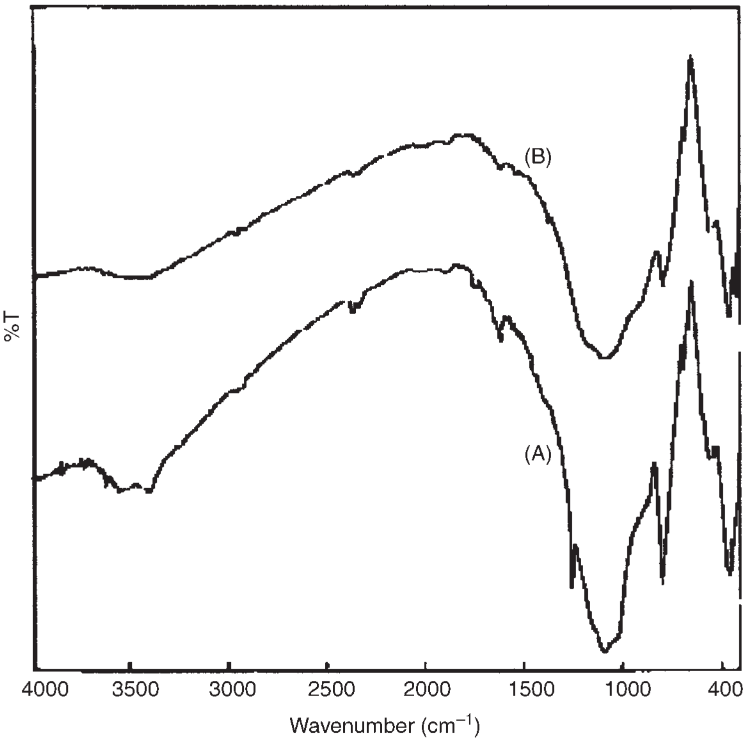

Figure 1. FT-IR spectra of (A) FA and (B) IFAAl.

the adsorption of Cr(VI) and Hg(II) by FA, IFAAl, and IFAFe increased at a concentration of 5 mg L^{-1} and a pH of 2.0 for Cr(VI) and 30 mg L^{-1} and 5.8 for Hg(II) (Figs. 4 and 5). The equilibrium time is independent of solution temperature. The same trend was observed for IFAAl and IFAFe as well (plots not given). The removal of Cr(VI) by IFAAl and IFAFe increased from 0.80 to 0.92 mg g^{-1} and 0.70 to 0.84 mg g^{-1} and the removal of Hg(II) increased from 8.40 to 10.50 mg g^{-1} and 9.70 to 11.40 mg g^{-1}. The observations indicate the adsorption to be endothermic for Cr(VI) and Hg(II) on all three adsorbents. The increase in the uptake of Cr(VI) and Hg(II) with increasing temperature might also be due to the enhanced rate of intraparticle diffusion of the adsorbate, as diffusion is an endothermic process. The increase in the adsorption behavior also suggests that the number of active surface centers available for adsorption increases with increasing temperature.[22]

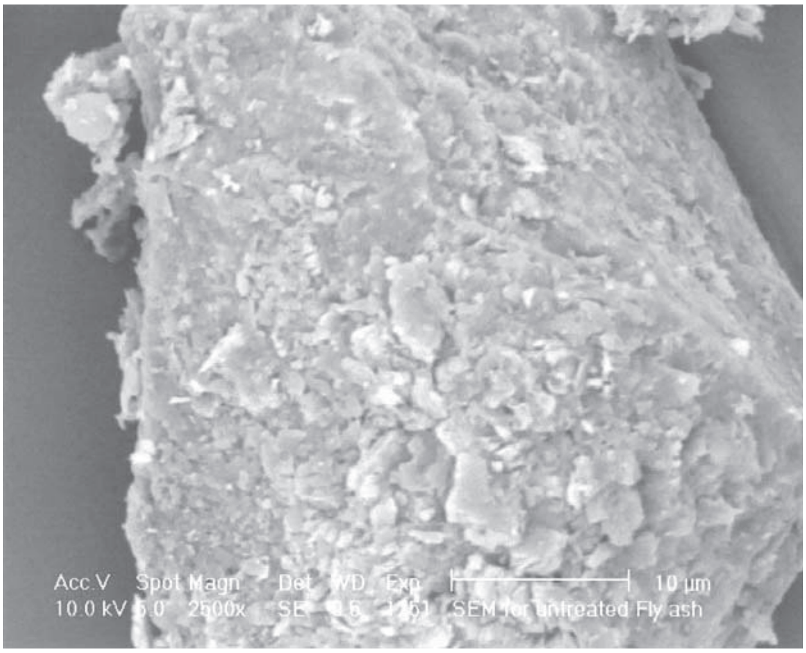

Figure 2. SEM micrograph of FA.

Adsorption Kinetics

The adsorption of Cr(VI) and Hg(II) can be considered as a reversible reaction. With the reaction in both directions being first-order, the rate equation may be expressed as:[23]

$$\log(q_\mathrm{e} - q) = \frac{\log q_\mathrm{e} - k^1}{2.303 \times t} \tag{1}$$

where q is the amount ($\mathrm{mg\,g^{-1}}$) adsorbed at time t and q_e is the amount ($\mathrm{mg\,g^{-1}}$) adsorbed at equilibrium.

And

$$k^1 = k_1\left(1 + \frac{1}{K_\mathrm{c}}\right) = k_1 + k_2 \tag{2}$$

where

$$K_\mathrm{c} = \frac{C_\mathrm{ae}}{C_\mathrm{se}} = \frac{k_1}{k_2} \tag{3}$$

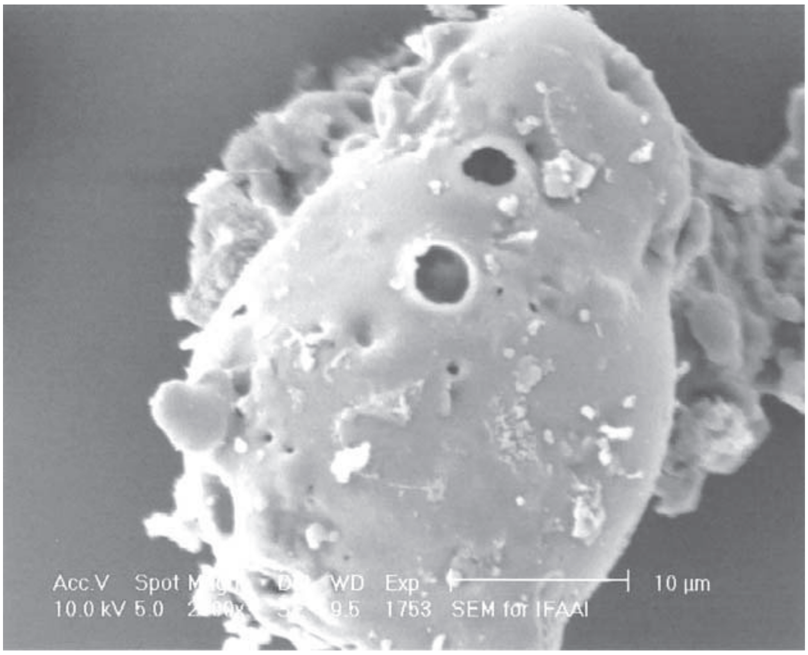

Figure 3. SEM micrograph of IFAAl.

k^1 is the overall rate constant, k_1, k_2 are the rate constant of forward and reverse reactions, C_{ae} is the equilibrium concentration ($\mathrm{mg\,L^{-1}}$) of Cr(VI) or Hg(II) on the adsorbent, and C_{se} is the equilibrium concentration ($\mathrm{mg\,L^{-1}}$) of Cr(VI) or Hg(II) in the solution.

The overall rate constant of adsorption at different temperatures was determined from the slopes of the straight line plots of $\log(q_e - q)$ vs. t (Table 2). The observation indicates that removal of Cr(VI) and Hg(II) is favored at higher temperatures.

The energy of activation was determined from the slope of the Arrhenius plot of $\ln k^1$ vs. $1/T$ (Table 3).

Langmuir Adsorption Isotherm

The results of the adsorption studies of Cr(VI) and Hg(II) on FA, IFAAl, and IFAFe were well fitted in the rearranged Langmuir equation:

$$\frac{C_e}{q_e} = \frac{1}{Q_0}b + \frac{C_e}{Q_0} \tag{4}$$

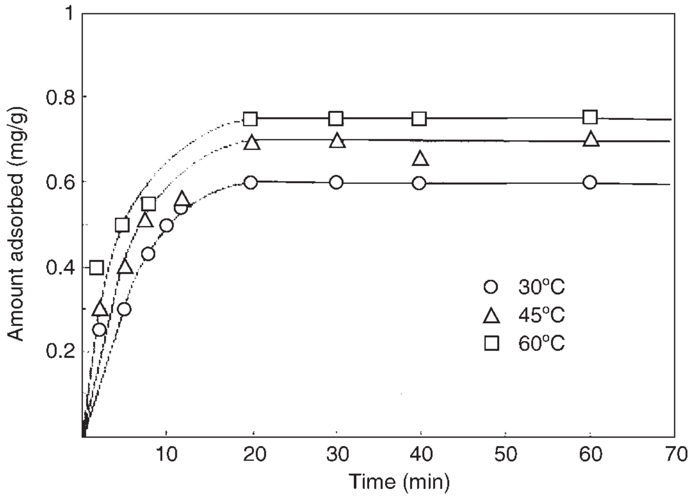

Figure 4. Effect of temperature on the Cr(VI) uptake by FA.

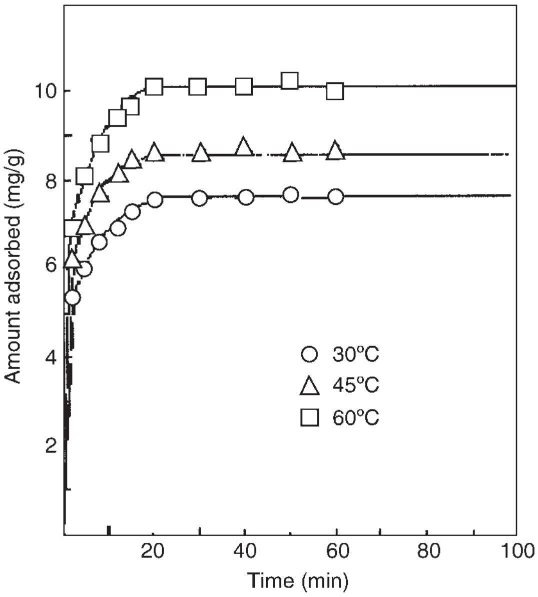

Figure 5. Effect of temperature on the Hg(II) uptake by FA.

Table 2. Rate constant values for Cr(VI) and Hg(II) adsorption.

Temperature (°C)	k^1 (min^{-1})					
	Cr(VI)			Hg(II)		
	FA	IFAAl	IFAFe	FA	IFAAl	IFAFe
30	0.111	0.176	0.167	0.118	0.141	0.165
45	0.138	0.207	0.179	0.132	0.158	0.178
60	0.167	0.230	0.216	0.158	0.191	0.204

where q_e is the equilibrium concentration of adsorbate in the solid phase (mg g^{-1}), C_e is the equilibrium concentration of adsorbate in the liquid phase (mg $^{-1}$L), and Q_0, b are the Langmuir constants.

The linear plots C_e/q_e vs. C_e show that the Langmuir isotherm model satisfactorily describes the adsorption of Cr(VI) and Hg(II) on FA, IFAAl, and IFAFe. The values of Q_0 and b determined from the slope and intercept of the plot are given in Table 4.

Thermodynamic Parameters

The various thermodynamic parameters were calculated from standard equations.[24]

$$\Delta G^\circ = -RT \ln K_c \tag{5}$$

$$\Delta H^\circ = \frac{R(T_2 T_1)}{(T_2 - T_1)} \ln\left(\frac{K_{c_2}}{K_{c_1}}\right) \tag{6}$$

$$\Delta S^\circ = \frac{(\Delta H^\circ - \Delta G^\circ)}{T} \tag{7}$$

where K_{c_1}, K_{c_2}, are the equilibrium constants at T_1 and T_2.

Table 3. Activation energy (E_a) values for the adsorption of Cr(VI) and Hg(II).

	E_a (kJ mol^{-1})		
	FA	IFAAl	IFAFe
Cr(VI)	21.54	16.75	19.15
Hg(II)	15.96	12.77	9.57

Table 4. Values of Langmuir constants.

	Q_0 (mg g^{-1})		b (L mg^{-1})	
	Cr(VI)	Hg(II)	Cr(VI)	Hg(II)
FA	1.379	11.00	0.207	0.900
IFAAl	1.820	12.50	0.183	0.089
IFAFe	1.667	13.40	0.194	0.0829

The free energy (ΔG°) decreases with increase in temperature for Cr(VI) and Hg(II), which indicates that the sorption process becomes more favorable at higher temperature. The negative value of ΔG° in case of Cr(VI) and Hg(II) adsorption on IFAAl and IFAFe at lower temperature suggests the process to be spontaneous as compared to FA (Tables 5 and 6). The positive value of, ΔH° for Cr(VI) and Hg(II) confirms endothermic nature of the adsorption (Tables 5 and 6).

Effect of pH

Removal of pollutants from wastewaters by adsorption is highly dependent on pH, which affects the surface charge of the adsorbent, and the degree of ionization and speciation of the adsorbate.[25] The plots of amount adsorbed at equilibrium at various pH values are shown in Figs. 6 and 7. The adsorption increased with increasing pH for Hg(II) and in case of Cr(VI) there was an initial increase up to pH 2.0 followed by a continuous decrease. This observation can be explained in terms of the surface charge of the adsorbent. At low pH, the surface has high positive charge density due to the presence of large number of protonated silanol ($SiOH_2^+$) and aluminol ($AlOH_2^+$) groups that bind electrostatically with the $HCrO_4^-$, which is the dominant chromium species at the system concentration and pH.[26] When pH of the solution is

Table 5. Thermodynamic parameters at different temperatures for Cr(VI)adsorption.

	ΔG° (kJ mol^{-1})			ΔH° (kJ mol^{-1})	ΔS° (J mol^{-1} K^{-1})
	30°C	45°C	60°C		
FA	1.021	0.353	−0.074	11.948	36.0
IFAAl	−0.336	−1.072	−1.513	9.041	32.0
IFAFe	0.336	−0.353	−0.893	10.367	34.0

Table 6. Thermodynamic parameters at different temperatures for Hg(II) adsorption.

	ΔG° (kJ mol^{-1})			ΔH° (kJ mol^{-1})	ΔS° (J mol^{-1} K^{-1})
	30°C	45°C	60°C		
FA	−0.067	−0.781	−2.002	18.587	62.0
IFAAl	−0.608	−1.294	−2.346	17.086	58.0
IFAFe	−1.523	−2.240	−3.192	15.450	56.0

increased, the negative charge density on the surface of the adsorbent increases as the adsorbent surface contains SiO^- and AlO^- groups and, hence, due to electrostatic repulsion between $HCrO_4^-$ and negatively charged groups on the adsorbent, the extent of adsorption is low. At $pH < 6.0$, the majority of Hg(II) exits as $Hg(Cl)_2$, $HgCl^+$, and $Hg(OH)^+$. The Hg(II) is adsorbed on the adsorbent as $HgCl_2$ with the subsequent reduction of $HgCl_2$.[27]

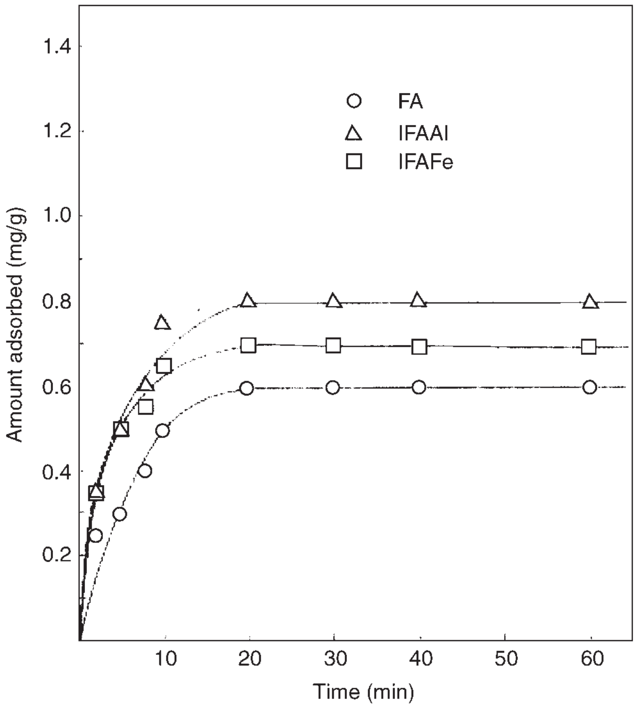

Figure 6. Adsorption kinetics of Cr(VI) on FA, IFAAl, and IFAFe.

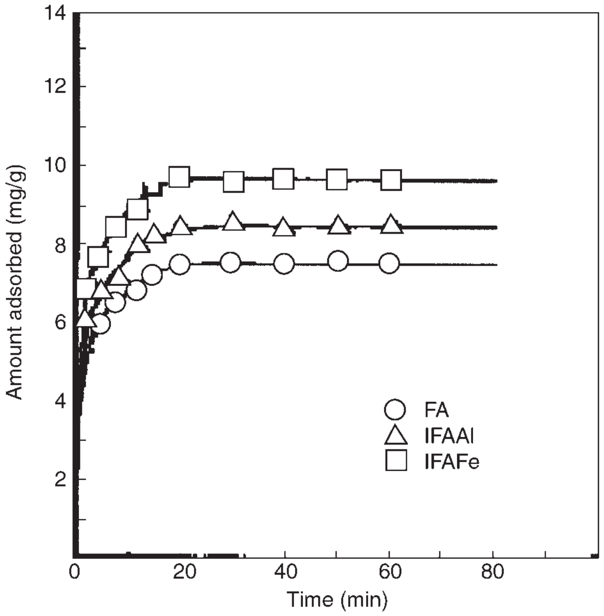

Figure 7. Adsorption kinetics of Hg(II) on FA, IFAAl, and IFAFe.

Significant adsorption of Hg(II) does not occur at low pH due to the electrostatic repulsion between the adsorbent surface and $HgCl^+$ and $Hg(OH)^+$. At $pH > 6.0$, the dominant Hg(II) species is $Hg(OH)_2$. Teh $Hg(OH)_2$ is more strongly adsorbed to the adsorbent surface as compared to $Hg(Cl)_2$ with the subsequent reduction of $Hg(OH)_2$ and so the adsorption of Hg(II) increases with increasing pH, as has been observed in earlier studies.[28]

Effect of Impregnation by Al^{3+} and Fe^{3+} Ions

In comparison with FA, the extent of adsorption of Cr(VI) and Hg(II) is higher for IFA (Figs. 8 and 9). This is probably caused by newly developed active sites (Al or Fe hydroxide layers) formed due to the electrostatic interaction between $Al(OH)_3$, $Fe(OH)_3$, and SiO_2 on IFA.[29] Adsorbents with a surface layer of aluminum hydroxide can specifically adsorb metal cations.[30,31] The pH-dependent charge is located at the edge sites, where the surface hydroxyl groups can be protonated or deprotonated depending on the pH. Small heavy metal cations can be bound specifically by the edge hydroxyl groups of the Al or Fe in the hydroxide surface layer on the planar

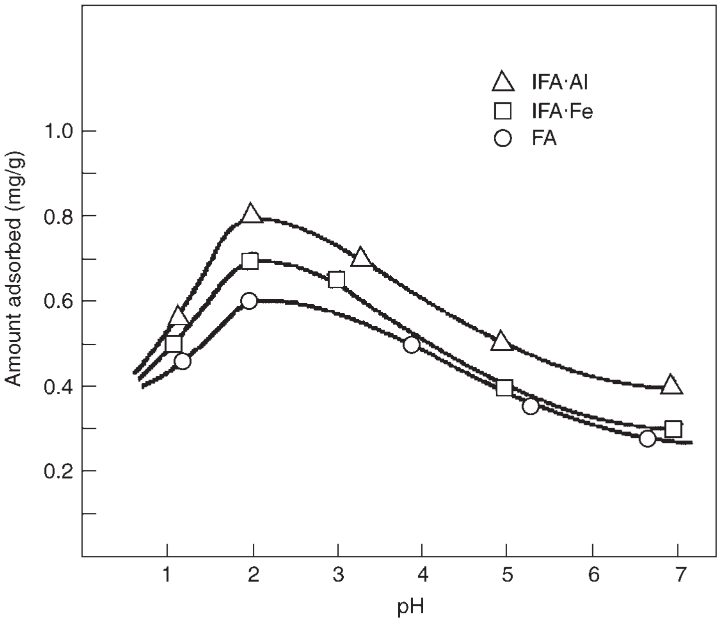

Figure 8. Effect of pH on Cr(VI) uptake of FA, IFAAl, and IFAFe.

surfaces of the modified FA. The low affinity sites on the permanently charged surface are widely replaced by high affinity Al(OH) functional groups with variable charge.[32] This is supported also by the fact that as compared to pure FA, the dissolved concentrations of Hg(II) are increased at low pH values when FA is treated with Al or Fe (Fig. 9).

Sorption Mechanism

The most abundant surface functional group participating in the surface complexation reactions on oxide surfaces is the hydroxyl group, which is amphoteric and extremely reactive. The adsorption of Hg(II) and Cr(VI) on hydrous metal oxide surface can be explained on the basis of surface complex formation and ligand exchange reactions. Consequently, the major adsorption schemes may be written as follows.

The adsorption of Cr(VI) can occur in aqueous solution as:

$$-\mathrm{SEOH} + \mathrm{H}^+ \Leftrightarrow -\mathrm{SEOH}_2^+$$

$$-\mathrm{SEOH}_2^+ + \mathrm{HCrO}_4^- \Leftrightarrow -\mathrm{SEOH}_2(\mathrm{HCrO}_4) \quad \text{or} \quad [\mathrm{SE}(\mathrm{HCrO}_4) + \mathrm{H}_2\mathrm{O}]$$

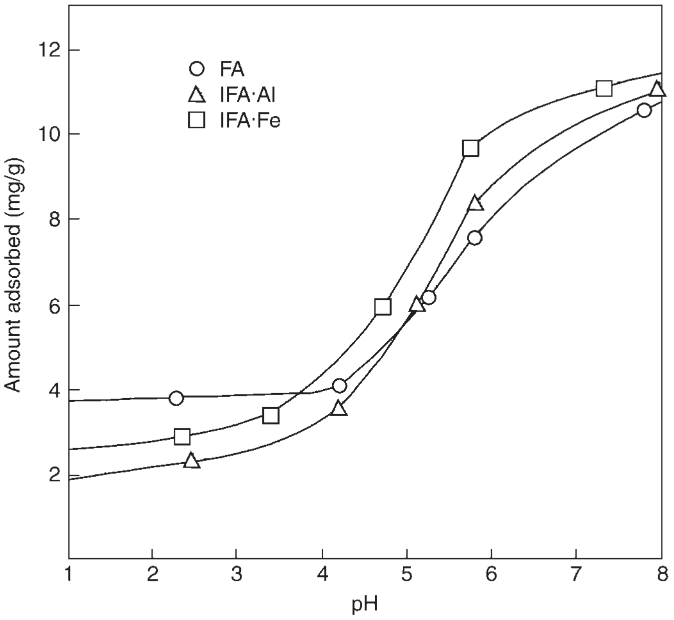

Figure 9. Effect of pH on Hg(II) uptake of FA, IFAAl, and IFAFe.

The adsorption of Hg(II) can occur in aqueous solution as:

$$-\mathrm{SEOH} + \mathrm{H}^{+} \Leftrightarrow -\mathrm{SEOH}_2^{+}$$
$$-\mathrm{SEOH}_2^{+} + 2\mathrm{Hg(Cl)}_2 \Leftrightarrow -\mathrm{SEOHg}_2\mathrm{Cl}_2 + 2\mathrm{HCl}$$

and

$$2(-\mathrm{SEOH}) + 2\mathrm{Hg(Cl)}_2 \Leftrightarrow (-\mathrm{SEO})_2\mathrm{Hg}_2(\mathrm{Cl})_2 + 2\mathrm{HCl}$$

over a pH range of 2.0–5.5.

Over a pH range from 5.5 to 8.0, the surface reactions can be expressed as:

$$-\mathrm{SEOH}_2^{+} + 2\mathrm{Hg(OH)}_2 \Leftrightarrow -\mathrm{SEOHg}_2(\mathrm{OH})_2 + 2\mathrm{H_2O}$$

and

$$2(-\mathrm{SEOH}) + 2\mathrm{Hg(OH)}_2 \Leftrightarrow (-\mathrm{SEO})_2\mathrm{Hg}_2(\mathrm{OH})_2 + 2\mathrm{H_2O}$$

where, $-\mathrm{SEOH}$ = Surface hydroxyl site and SE = Si, Al, or Fe.

Similar mechanism for Hg(II) adsorption has been postulated by Sanchez-Polo and Rivera-Utrilla.[27]

Cost Estimation

The cost of the cheapest variety of carbon available in India is approximately US$ 285 per ton. The waste FA costs almost nothing and considering the cost of transport, chemicals, electrical energy used in the activation process, and labor, the finished product would cost approximately US$10 per ton. Therefore, the developed IFA adsorbent may be considered as a good cost-effective replacement for commercially available carbon.

Application of the Developed Adsorption System to Industrial Wastewater

The developed IFA are very effective, economical, and rapid for the removal of Cr(VI). This system has successfully been tested to remove Cr(VI) from wastewater samples obtained from electroplating plants at MIDC, Badlapur (Maharashtra). The sample effluents were given the following treatment before subjecting them to adsorption.

The sample solution (7.6 mg L^{-1}, pH 3.4) was freed from all suspended particles by centrifuging at an agitation speed of 8000 rpm for 10 min. It was observed that the 0.5 g of developed adsorbent, IFAAl [which was found to be the most efficient adsorbent for Cr(VI)] was capable of adsorbing and retaining Cr(VI) up to 90% from 100 mL of the sample solution. Hence, the system can be developed and extended for further practical applications of wastewater treatment.

CONCLUSIONS

On the basis of the above results the following inferences can be drawn:

1. The sorption capacity of IFA for Cr(VI) and Hg(II) was found to be higher than that of FA.
2. Adsorption of Cr(VI) and Hg(II) follow the Langmuir isotherm model.
3. The negative value of ΔG° in case of Cr(VI) and Hg(II) adsorption on IFAAl and IFAFe at lower temperature suggests the process to be spontaneous as compared to FA.

4. The adsorption of Hg(II) and Cr(VI) on hydrous metal oxide surface can be explained on the basis of surface complex formation and ligand exchange reactions.
5. The studies reveal that IFA can be used efficiently as an adsorbent for the removal of Cr(VI) and Hg(II). The data obtained can be extended and further exploited for fabricating a continuous treatment plant for wastewater contaminated with Cr(VI) and Hg(II).

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