Efficient Removal Of Cd (Li) From Aqueous Solution Using Pyroaurite Type Sorbent, Ceralite, Activated Carbon, Goethite and Rice Husk: Adsorption Performance and Mechanism

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Abstract:

The use of pyroaurite type sorbents for removal of Cd(II) from aqueous solution has been studied. In batch tests, the sorbents IRA, PTS, PAC, PLC, FeO(OH) and RH could remove 95.3 ,93.8 , 93.9, 90.4, 75.1 and 59.5% cd (II) at initial metal concentration 0.1 mg/l, pH 6.0, temperature 25°C, agitation time 5 h, sorbent dose 5 g/l and rpm 150. The order of cadmium removal capacities for these chemical adsorbents was found to be: IRA > PTS > PAC > PLC > FeO(OH) > RH. The effects of various parameters affecting the adsorption such as initial metal concentration. Sorbent dose, contact time and pH were determined. Adsorption decreases with in cadmium concentration and pH but increases with increase in adsorbent dose and contact time. Equilibrium was attained in 5 h and the maximum removal was at pH 8. Thus, basic medium favoured the removal. The sorption data fitted very well to the Langmuir isotherm as well as the Freundlich equation.

Introduction

30-50% of the total Cd produced (17 x 10^3 tonnes per year) is used in plating and 20-30% in pigments. Its other uses are in Ni-Cd and Ag-Cd cells, stabilizers for plastics and in alloys. Cd, Pb and Hg are used in batteries. Cd, Cr, Cu and Zn are used in metal plating. Rubber tyres can contain 0.09 mg/g of Cd due to use of ZnO and zinc diallyl carbamates in the vulcanization process and Cd occurs with Zn in nature. The abrasion of tyres on the road adds Cd to street dust that comes to soil by wet and dry deposition processes. Though Cd^{2+} movement and plant availability in soils are small, its extreme toxicity can be a serious problem in soil. Water systems at several places have been found to be contaminated with cadmium (Griscom and Fisher, 2002; Harrington, 1987)

Cd damages kidney and causes itai-itai in which bones become fragile and muscles contracted with deformation and pain. A mineral salt solution added to the medium can enable growth of bacteria at higher concentrations of added lead. This is interesting to compare the results for another metal, or to find other suitable salts. The influence of 4 different cadmium salts such as $Cd(NO_3)_2$, $CdCl_2$. $CdsO_4$, and CdO on the resistance of different gram positive and gram-negative Species has been studied. For example, $CdCl_2$ is the most toxic cadmium salt whereas $Cd(NO_3)_2$, is efficiently absorbed by some strains. Metal ions have strong affinity for S containing ligands such as ---SH in cysteine, SCH, in methionine and amino acid residues in enzymes. Metalloenzymes contain metals in their structure. Their action is inhibited when an essential metal is replaced by a toxic metal of similar size and charge as Zn by Cd. Studies on the

treatment of effluents bearing heavy metals and organics reveal sorption to be the most promising technique because it is highly effective, cheap, easy and eco-friendly method among all physicochemical processes. Sorption of Cd(II) by various materials has been studied by previous researchers.

The major techniques used are precipitation, ion exchange, and reverse osmosis, electroflotation, solvent extraction etc. (Namasivayam and Ranganathan 1995) are attractive attention of scientists. Adsorption is one the physio-chemical treatment process found to be effective in removing heavy metals from aqueous solutions. According to (Bailey et al. 1999) An adsorbent can be considered as cheap or low cost if it is abundant in nature. The effect of natural and dynastic surface active substance on the electrochemical reduction of cadmium in natural waters has also been studied (Kozarac, 1986) However, the comparative far been adsorption behavior of common adsorbents of Cd(Il) has not so studied. So, the present study has been undertaken to investigate and compare the suitability of synthesized pyroaurite like compound (PLC), modified PLC, ie. Pyroaurite type sorbent (PTS) and adsorbents such as powdered activated carbon (PAC), rice husk (RH) and goethite (FeO(OH)) for the Cd(ll) removal from wastewater using batch experiments. A cation exchange resin Ceralite IR 120 (Na^+ form, 20-50 mesh) was also taken so that the sorption capacity of the synthesized sorbent can be compared (Pari and Durai, 2000; Rai et al., 1998; Rama and Rajeshwari, 2001).

Materials and Method
Cd (II) Solution
All reagents were of analytical grade. In order to avoid interference by other elements in wastewater, the experiments were conducted with aqueous solutions of pentavalent cadmium in redistilled water. Synthetic samples of various initial cd (II) concentrations were prepared from the stock solution that was in turn prepared by dissolving 1.000 g of Cd metal in a minimum volume of (1: 1) HCl and then diluting it to 1 L with 1 % (v/v) HCl. Four standard solutions of 0.1, 1, 5 and 10 mg/l concentrations of Cd (II) were prepared by diluting the stock solution of 1 g/1. Double distilled water was used to dilute the solution.

Preparation of Pyroaurite like Compound
Mg Cl2 (0.15 mol) and FeCl3 (0.075 mol) were dissolved into 200 ml of distilled water. 10-wt% NaOH aqueous solution was then added drop wise into the solution while stirring vigorously until the pH of the solution reached above 13. The solution was kept at room temperature for 24h with vigorous stirring. The obtained precipitate was washed with a large amount of distilled water to wash away residual.

Table 1. Characteristics of sorbents

Characteristics	IRA	PTS	PLC	PAC	FeO (OH)	RH
Density (glcc)	2.11	2.20	2.50	1.40	2.49	1.09
Bulk density (g/ml)	1.3	1.6	1.8	1.2	1.7	0.9
Moisture(%)	10.5	11.31	16.31	3.61	15.00	8.20
Ash (%)	10.7	9.65	12.65	3.32	12.32	2.30
Volatile matter (%)	9.3	6.74	5.74	9.39	6.08	10.02
Combustible matter (%)	55.6	64.16	54.16	84.85	57.54	89.02
Loss of ignition	4.52	5.02	4.02	3.83	5.96	9.02
Chemical Analysis						
SiO_2 (%)	9.3	2.00	2.00	7.05	33.92	18.35
Al_2O_3 (%)	12.0	0.02	0.02	2.05	12.44	1.83

CaO (%)	.31	0.12	0.12	0.04	4.15	1.35
Fe_2O_3(%)	0.22	5.32	5.02	1.23	12.26	2.51
MgO(%)	0.11	3.02	3.00	1.10	2.36	1.04
Surface area (m^2/g)	730	590	470	538	378	198

Batch Studies

Batch tests were conducted at constant temperatures under steady state and transient rate batch conditions.

Capacity Studies

The capacity studies were carried out by transient batch tests. In20 numbers (4 concentration of 4 toxics each + 4 controls one for each) of 100- ml capacity PVC bottles with screw caps), 0.5 g of sorbent was added to each bottle of 4 sets, each set having 5 bottles 4 for different concentrations of a toxic and 1 for its control. 100 mi of the toxic solution was added to each bottle. The solution was buffered with 0.07 M sodium acetate – 0.03M acetic acid, pH 8. The bottle was shaken in a reciprocating shaker at 150 rpm for 5 h at room temperature, and the concentration of a toxic element was measured after 1, 3, 5, and 24 h until the solution reaches equilibrium. The controls without sorbent demonstrated toxic uptake due to the sorbent, not form other sources such as the walls of the container, centrifuge tube etc. The contents were centrifuged at 2000 rpm for 10 min and the supernatant liquid was filtered using a 0.45 mm membrane filter. The filtrate was analysed for the studied ions (APHA 1998) The initial and final concentrations of the solutions were determined using a Perkin Elmer Analyst 100 AAS. Experiments were triplicated and results averaged. Unless otherwise stated all experiments were conducted at room temperature.

The uptake of toxic ions on the sorbent at initial concentrations 0.1,1,5 and 10 mg/l were investigated. To study the effects of the sorbent dose 0.1,1,5. 10 and 20 g sorbent/ l of toxic solution was taken. To examine the effect of pH, the pH was varied from 2 to 8 when metal ions largely precipitate. To examine whether the filters used during the tests absorbed or released any Pb, several filtered and unfiltered samples were compared.

Sorption capacity (SC) of the substrate was expressed in term of amount of toxic sorbed on the unit sorbent mass (mg/g) and sorption efficiency of the system (% Removal) was indicated as the percent removal of toxic ions relative to the initial amount.

$$SC = \frac{Co - Ce}{Cs} = \frac{(Co - Ce)V}{m}$$

$$\% \, Removal = \frac{Co - Ce}{Co} \times 100$$

Where Co and Ce are respectively, the initial or original and final or equilibrium concentrations of toxic ions in solution (mg/l) and Cs is the concentration of the sorbent in the mixture (g/l), m is the mass of sorbent and V is the volume of the solution. The data were analysed using isotherm, kinetic and thermodynamic equations.

Results and Discussion

Batch Process

Effect of Initial Concentration and Contact Time

Sorption capacity is found to decrease with increase in metal concentration. The higher uptake at lower initial concentration can be attributed to the availability of ore isolated cadmium cations. The sorbents IRA, PTS, PAC, PLC, FeO(OH) and RH could remove 95.3, 93.8. 93.9, 90.4, 75.1 and 59.5 % Cd(II) at

initial metal concentration 0.1 mg/l, pH 6.0, temperature 25°C, agitation time 5 h and adsorbent dose 5 g/l. removal capacities for these chemical adsorbents was found to be: IRA > PTS > PAC > PLC > FeO(OH) > RH. The effect of various parameters affecting the adsorption such as initial metal Concentration, adsorbent dose, contact time and pH was determined.

Table 2. Removal % of Cd at different concentrations and at sorbent dose 5 g/l, pH 6.0, contact time 5 h, temperature 25°C and rpm 150

Concentration, mg/l	IRA	PTS	PAC	PLC	FeO(OH)	RH
0.1	95.3	93.8	93.9	90.4	75.1	59.5
1	93.3	92.3	89.3	89.1	60.3	56.0
5	93.4	88.7	87.6	86.3	56.3	55.3
10	90.3	85.2	84.2	81.0	51.1	50.2

Sorption rate is very rapid during initial period of contact and about 60 per cent of sorption on IRA, PTS, PAC and PLC is reached within an hour. However, equilibrium was attained within 5 h (Ainsworth, 1994)

Table 3. Removal % of Cd at different agitation times and at initial concentration 10 mg/l, sorbent dose 5 g/l, pH 6.0, temperature 25°C and rpm 150

Contact time (h)	IRA	PTS	PAC	PLC	FeO(OH)	RH
1	63.9	64.1	60.8	62.1	38.1	31.3
3	70.2	67.5	69.3	65.3	42.5	38.3
5	90.3	85.6	84.2	81.0	51.1	50.2
24	91.1	86.3	85.8	82.3	52.3	53.6

Adsorption decreases with rise in Cd concentration but increases with increase in adsorbent dose and contact time.

Effect of Adsorbent Dose

The effect of adsorbent dose on the adsorption of cadmium using different adsorbents at pH 6 and temperature 25°C is depicted in Table 4. It clearly indicates that the percent Cd(II) removal increases with increase in adsorbent

Table 4. Removal % of Cd at different sorbent doses and at initial concentration 10 mg/1, pH 6.0, contact time 5 h temperature 25°C and rpm 150

sorbent dose (g/l)	IRA	PTS	PAC	PLC	FeO(OH)	RH
0.1	75.6	68.2	65.8	65.1	33.4	28.3
1	78.9	80.2	73.8	75.8	43.3	35.1
5	90.3	85.6	84.2	81.0	51.1	50.2
10	95.4	92.7	93.3	89.2	59.2	54.4
20	95.8	93.2	93.9	90.5	60.4	55.6

dose. It is obvious from Table 4. that as the sorbent dose increases, the percentage of Cd(II) removal also increases, but after an optimum dose of 10 g sorbent per litre of Cd(II) solution there is no appreciable change in removal. Further, at lower sorbent dose the adsorbate is more easily accessible and because of

this, removal per unit weight of adsorbent is higher. The initial rise in adsorption with adsorbent or adsorbate concentration is probably due to bigger Driving force and lesser surface area. Larger surface area of sorbent and smaller size of adsorbate favour adsorption. The rate of adsorption is high in the beginning as sites are available and unimolecular layer increases. Adsorption and desorption occur together, and rates become equal at a stage called adsorption equilibrium when isotherms are applied. That is why there is little increase in % removal on increasing adsorbent dose from 10 to 20 g/ml of Cd(II) solution. The subsequent slow rise in curve may be due to adsorption and intra-particle diffusion taking place simultaneously with dominance of adsorption. With rise in sorbent dose there is less commensurate increase in adsorption resulting from lower adsorptive capacity utilization of sorbent. This is called 'solid concentration effect' meaning overcrowding of particles.

Effect of pH

100 ml solution of 10 mg/l Cd(II) concentration was adjusted to pH 2 to 8 with HCV/NaOH and treated with 0.5 g of sorbent at rpm 150 for 5h. The influence of pH of solution on the extent of adsorption of metals is depicted in Table 5. The Cd(II) removal is highly dependent on pH of the medium which affects the surface charge of the adsorbent and degree of ionization. The maximum removal was at pH 8. Thus, basic medium favoured the removal.

Table 5. Removal % of Cd at different pH values and at initial concentration 10 mg/l, sorbent dose 5 g/l, contact time 5 hrs, temperature 25^0C and rpm 150.

pH	IRA	PTS	PAC	PLC	FeO(OH)	RH
2	74.6	70.2	72.3	68.1	46.5	38.3
4	81.2	81.3	76.1	76.3	50.3	43.7
6	90.3	85.6	84.2	81.0	51.1	50.2
8	94.6	91.2	90.3	89.1	60.9	52.4

Most of these sorbents consist of oxides of silicon, aluminium, iron, calcium, magnesium etc. Many researchers have found that anion adsorption sites on such minerals like alumina and clay are aquo groups ($-M-OH_2-$) and hydroxy groups ($-M-OH$). The surface chemistry of an oxide in contact with an aqueous solution is determined to a large extent by deprotonation or a hydroxyl ion association reaction. The presence of oxides of alumina, calcium and silicon of the adsorbents develop charge in contact with water according to pH of the solution (Fraysse et al.2000). in the present study, the optimum pH for cadmium removal was found in the basic medium. The reason may be attributed to the large number of OH^- ions in basic medium that support sorption through precipitation of metal ions. 169 Above and below this pH, the extent adsorption was considerably low. Except silica, all other oxides (the major constituent of flyash being alumina) posses positive charge for a pH range of interest. As the pH decreases below 4.5, Al_2O_3 is dissolved as A and subsequently the surface of, Al_2O_3, becomes further positively charged with decrease in pH. As pH increases, the hydroxy group ($-M-OH$) goes on disappearing and forming increasingly negatively charged surface. n addition, OH also competes for the available sites left on the surface. The zero-point charge of SiO_2, Al_2O_3 and CaO are 2.2, 8.3 and 11.0 respectively. Thus, the negatively charged silica surface sites of adsorbents get neutralized by cations such as metal or H ions, thereby reducing hindrance to diffusion of anions present in the aqueous phase.

Effect of Temperature

The effect of temperature on cadmium adsorption on sorbents was studied by conducting the batch adsorption at different temperatures of 15, 20, 25 and 35°c. With increase in temperature, the adsorption came down rapidly indicating the adsorption process to be exothermic in nature. The mean value of standard entropy change DH remained in the range -27.75 to 21.85 kl/mol conforming the absorption process to be exothermic. The considerable low values of DH indicates work interaction between metal and he sorbents. This also explained the rapid decrease in adsorption with increasing temperature.

Table 6. Removal % of Cd (II) at different temperatures and at initial concentration 10 mol, initial sorbent dose 5 g/l, pH 6.0, contact time 5h rpm 150.

Temp., °C	IRA	PTS	PAC	PLC	FeO(OH)	RH
15	96.2	94.1	91.8	86.9	69.1	62.7
20	94.5	90.7	89.5	85.6	63.4	59.8
25	90.3	85.6	84.2	81.0	51.1	50.2
35	79.1	71.6	69.5	65.7	48.9	45.1

The evaluation of the thermodynamic parameters shows that cadmium ions bind moderately to the sorbent surface by a chemisorptive process and is accompanied by decrease in enthalpy, entropy and Gibbs free energy. Thus, the adsorption process is exothermic and spontaneous as well as thermodynamically favourable.

Sorption Capacity and Kinetics

The experimental data were analysed using four sorption kinetic models: the Pseudofirst order, the Ritchie second order, the modified second order and the Elowich equations (Cheung, 2001; Reddad,2002). The values of specific reaction rate or velocity coefficient k was calculated for all the sorbents, and they were found constant when calculated using the first order equation.

$$k = 2.303/t \ \log a /(a - x)$$

Where a mol/l is initial cadmium concentration from which x mol/l has been removed after 1 seconds. the data were also analysed using Langmuir and Freundlich equations.

$$\log a = log K_f + \frac{l}{n} \log c \qquad \text{Freundlich equation}$$

$$\frac{c}{a} = \frac{l}{Qb} + \frac{c}{Q} \qquad \text{Langmuir equation}$$

Where a (mg/g) is the metal sorbed per unit mass of soil (a= x/m where x mg of metal is sorbed on m grams of soil), k (mg/kg) and n are Freundlich constants related to adsorption capacity and adsorption intensity respectively, and q (mg/g) and b (kg/g) are Langmuir constants related to adsorption capacity of soil and adsorption maximum (energy of adsorption) respectively. Freundlich and Langmuir constants were calculated at four initial concentrations under optimal conditions. The sorption data fitted very well to the Langmuir isotherm as well as the Freundlich equation. The values of Q and k_f in Table 5.7 clearly indicate the sorptive capacities of different sorbents.

Table 7. Analysis of data using kinetic and isotherm models.

Sorbent	First order rate constant	Langmuir constants and R^2			Freundlich constants and R^2		
	K(l/min)	Q,mg/g	B,l/g	R^2	$K_{f,,}$mg/g	L/n	R^2
IRA	0.0099	10.9032	0.0825	0.9633	6.9245	0.0945	0.7925
PLC	0.0075	7.9035	0.0372	0.9635	4.9245	0.0345	0.7934
PTS	0.0097	9.0526	0.0754	0.9578	5.0235	0.0545	0.6523
PAC	0.0079	8.7222	0.0502	0.9223	4.7045	0.0512	0.6112
FeO(OH)	0.0045	5.2256	0.0417	0.9785	3.2246	0.0213	0.8312
RH	0.0035	2.9323	0.0208	0.9677	1.2345	0.0145	0.6612

Conclusions

In an attempt to evaluate the suitability of synthesized pyroaurite like compound (PLC), modified PLC, i.e., pyroaurite type sorbent (PTS) and adsorbents such as powdered activated carbon (PAC), rice husk (RH) and goethite), (FeO(OH)) and a cation exchange resin Ceralite IR 120 (N_i^+ form, 20-50 mesh) for the removal of divalent cadmium from aqueous solution, a study of these six sorbents was carried out by batch process. In batch tests, the sorbents IRA, PTS, PAC, PLC, FeO(OH) and RH could remove 95.3 ,93.8 , 93.9, 90.4, 75.1 and 59.5% cd (II) at initial metal concentration 0.1 mg/l, pH 6.0, temperature 25°C, agitation time 5 h, sorbent dose 5 g/l and rpm 150. The order of cadmium removal capacities for these chemical adsorbents was found to be: IRA > PTS > PAC > PLC > FeO(OH) > RH. The effects of various parameters affecting the adsorption such as initial metal concentration. Sorbent dose, contact time and pH were determined. Adsorption decreases with in cadmium concentration and pH but increases with increase in adsorbent dose and contact time. Equilibrium was attained in 5 h and the maximum removal Was at pH 8. Thus, basic medium favoured the removal. The sorption data fitted very well t to the Langmuir isotherm as well as the Freundlich equation. The values of Q and K_f clearly indicate the sorptive capacities of different sorbents are in the order as mentioned above. The experimental data were analysed using four sorption kinetic models: the pseudofirst order, the Ritchie second order, the modified second order and the Elowich equations. The values of specific reaction ate or velocity coefficient k were calculated for all the sorbents, and they were found constant when calculated using the first order equation The adsorption resulted in a significant decrease in Gibbs energy pointing to a spontaneous and energetically favourable process. The average value of standard entropy change was also negative showing that the adsorption of cadmium on Sorbents was accompanied by a decrease in entropy. The immobilization of the organic molecules on specific sites of the sorbent surface might have led to restoration of order (in comparison to the solution phase) resulting in a decrease of entropy. The evaluation of the thermodynamic parameters shows that cadmium ions bind moderately to the sorbent surface by a chemisorptive process and is accompanied by decrease in enthalpy, entropy and Gibbs free energy. Thus, the adsorption process is exothermic and spontaneous as well as thermodynamically favourable. The results showed variation of percent Removal at various pH values. A specific relation of optimum pH was observed. Cadmium showed the highest removal of 95.3% by batch process.

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