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Sorption of lead, mercury and cadmium ions in multi-component system using carbon aerogel as adsorbent

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Abstract

In the present study, adsorption of three metal ions Pb(II), Hg(II) and Cd(II) on carbon aerogel a new form of activated carbon has been investigated in mono- and multi-component (binary and tertiary) system. Batch experiments were also carried out for mono- and multi-component systems with varying metal ion concentration (mg/l) to investigate the competitive adsorption characteristics. Many adsorbents have been studied for their adsorption properties pertaining to mono-component solutions of metal ions. However, to treat wastewater with new materials, their performance needs to be ascertained in multi-component system. The scanning electron micrographs (SEM) and EDAX spectrum of carbon aerogel surfaces before and after the adsorbent was equilibrated with the metal ion solution clearly establishes the presence of the metal ions and some surface modifications can be observed on the carbon aerogel particles adsorption with (i) surface chemistry of the pellets on the surface of carbon aerogel and (ii) inside layers of the carbon aerogel. Applicability of the isotherm models namely Freundlich and Langmuir to predict the equilibrium uptake of Pb(II), Hg(II) and Cd(II) in mono-component, binary and tertiary system has also been tested. Langmuir and Freundlich models are found to generally represent the experimental though but not consistently.

Keywords: Carbon aerogel; Mono- and multi-component; Isotherms; Adsorption behavior

1. Introduction

In recent years, the presence of heavy metals in industrial wastewater and effluents have gained of importance due to their more stringent discharge norms increased usage and their high toxicity cumulative adverse characteristics. This increased awareness and concern about environmental pollution and of the stringent national and international legislations has also contributed to more efforts of research work in this area, specially in finding better and more efficient technique for separating these contaminants from effluent water.

A practical consideration to the problem reveals that most of the effluent solutions represent a case of multimetal situation rather than monometal situation. In such a scenario it becomes essential to study the effects of the presence of co-cations on the adsorption capacity of an adsorbent for a particle contaminant.

Researchers have examined in the past, the adsorption behavior of the adsorbent with respect to a particular metal in presence of other metal ions. Banat et al., 2001; Schiewer and Volesky, 1995; Mohan and Singh, 2002; Sag et al., 1998; Sanchez et al., 1999; Benguella and Benaissa, 2002; Ma and Tobin, 2003; Lee et al., 2004; Li et al., 2004, have successfully completed their studies in multimetal adsorption system focusing on metal sorption in binary system using multi-component Langmuir or Freundlich isotherm models using adsorbents, heat treated bentonite, marine algae, activated carbon derived from bagasse, *C.vulgaris* and *R.arrhizus*, *Cymod-ocea nodosa*, chitin, peat biomass, algal biomass and Phanerochaete chrysoporium, respectively [1–9].

Mohapatra and Gupta has reported the biosorption of Zn(II), Cu(II) and Co(II) onto *O. angustissima* immobilized biomass from single, binary and ternary metal solutions, as a function of pH and metal concentrations via response surface methodology. They have concluded that Freundlich model can appropriately described adsorption equilibrium for both mono-component and binary system. Furthermore, these researchers have also

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concluded that in the binary metal solutions, both metal uptake and adsorption yield for one kind of ions decreased with increasing concentration of the other metal ions [10].

Petrus and Warcho have Ternary and quaternary ion-exchange equilibria between heavy metal solution (Pb^{2+}, Cd^{2+}, Cu^{2+}) and Na-form of clinoptilolite. These researchers demonstrated the applicability of various models such as Langmuir, Competitive Langmuir, Wilson and thermodynamic sorption models for modelling of competition sorption data [11]. Saeed et al. have concluded the potential of application for the simultaneous removal of several heavy metals contained in effluent solution using crop milling waste (black gram husk) as adsorbent [12].

The purpose of this study has been to investigate the adsorption characteristics of three heavy metals Pb(II), Hg(II) and Cd(II), onto carbon aerogel in single, binary and tertiary systems. The main objectives of these competitive sorption studies were:

(a) To assess the efficacy of CA for removal of Pb(II), Hg(II) and Cd(II) in multimetal adsorption systems.
(b) To provide a quantitative estimation of adsorption capacity of CA in competitive sorption system using Langmuir model.

The applicability of the isotherm models namely Freundlich and Langmuir to predict the equilibrium uptake of Pb(II), Hg(II) and Cd(II) in mono-component, binary and tertiary systems has also been analyzed using the results for these investigations.

2. Experimental

2.1. Materials (adsorbent)

The carbon aerogel used in the study is the commercial product of Lawrence Livermore National Lab., California U.S.A. Carbon aerogels are derived from sol–gel polymerization of metal alkoxides or certain multifunctional organic monomers, followed by supercritical drying with CO_2 [13]. Carbon aerogel properties are in many respects better than those of their inorganic counterparts. Aerogel are micro cellular foam materials; they are quite porous and have an area-to-mass ratio of 700 $m^2\,g^{-1}$ and controllable pore size distribution ($\leq$50 nm) [14].

2.2. Adsorbate

Pb(II), Hg(II) and Cd(II) metal ions were studied, their main characteristics being summarized in Table 4. All the chemicals or reagent used were AR grade.1000 mg/l of adsorbate stock solution was prepared by dissolving metal salts in one percent HNO_3 solution to prevent hydrolysis formation. Salts used were $Pb(NO_3)_2$ for Pb(II), $HgCl_2$ for Hg(II), $CdSO_4$ for Cd(II), with high degree of purity. The stock solutions of Cu(II), Pb(II), and Hg(II) were diluted with distilled water to obtain standard solutions. Metal ions were analyzed with a GBC 935 Atomic adsorption spectrophotometer (AAS) at their respective wavelength.

Table 1
Physico-chemical characteristics of the carbon aerogel

Parameter	Value
BET Surface area (m^2/g)	700
pH (2% slurry)	8.32
Conductivity (S/cm)	50
Decolorizing power (mg/g)	93
Apparent density (g/ml)	0.644
Ion exchange capacity (meq/g)	0.01
Particle size (μm)	Less than 500
pH pzc	4.87
Moisture (%)	18
Acidic surface functional groups (meq/g)	
Carboxyl group	0.784
Lactonic group	0.015
Hydroxyl group	0.007
Carbonyl group	0.003
Basic surface functional groups (meq/g)	0.45
Porosity parameters of carbon aerogel	
Micropore	
a_{micro} (m^2/g)	300
V_{micro} (ml/g)	0.10
Mesopore	
a_{meso} (m^2/g)	402
V_{meso} (ml/g)	1.36

2.3. Characterization of adsorbent

The adsorbent was characterized with intend of assessing its various physical and chemical properties, so that a better explanation of adsorption mechanism can be provided. Carbon aerogel was analyzed for various parameters like pH, conductivity, moisture content, ion exchange capacity, apparent density and decolorizing power by using standard methods [15]. Studies are available which have investigated its application in electro-sorption of metal ions from aqueous medium [16,17]. The physico-chemical properties of the adsorbent are summarized in Table 1. The surface area was determined by nitrogen adsorption isotherm at 77 K using BET equation. The acidic and basic surface functional groups were determined by titration using the Boehm method [18]. The pH of the point of zero charge, pH $_{PZC}$ (above which the total surface of the carbon is negatively charged) was determined using so-called pH drift method [19]. The surface morphology and metal competition of CA equilibrated with metal ions Pb(II), Hg(II) and Cd(II) at pH 4.5 were visualised by using a scanning electron microscope (SEM). The SEM micrographs for the sample were obtained with a JSM-840 JEOL microscope at 200× magnifications. Energy dispersive X-ray analysis (EDAX) of the samples (with and without metal ions) was also carried out using ISIS-200 energy dispersive spectrometer (EDS) technique for qualitatively analyzing the elemental constitution of the samples.

2.4. Methodology

2.4.1. Mono-component adsorption of metal ions

Batch mode adsorption studies of Pb(II), Hg(II) and Cd(II), were carried out at 37 °C using 50 ml of metal ion solution with concentration ranging between 5 to 70 mg/l and 100 mg of adsorbent and agitated using mechanical shaker at 120 rpm. After equilibrium adsorption (20 h) the samples were withdrawn and separated. All the experiments were carried out at initial pH of 4.5, (adjusted using dilute HNO_3 or NaOH), where the adsorption is significant but below the pH where metal hydroxide precipitation occurs. The final (residual) metal concentrations (C_e) for different metal systems in test solution were analyzed, leading to the respective calculated values for adsorbent metal uptake. Metal uptake or surface loading, q_e for each sorption system was calculated using the general definition

$$q_e(\text{mg/g}) = \frac{V(C_i - C_e)}{W} \tag{1}$$

where, C_i is the initial metal concentration in solution (mg) V is the volume (L) and W is the mass of adsorbent (mg).

2.4.2. Binary and tertiary adsorption metal ions

Binary and tertiary component adsorption systems were prepared by solubilizing a combination of either Cd^{2+}–Pb^{2+}, Pb^{2+}–Hg^{2+} or Hg^{2+}–Cd^{2+} or Cd^{2+}–Pb^{2+}–Hg^{2+} with presence of each metal [Cd(II)/Pb(II)/Hg(II)] with other metal/metals present in equal concentrations (mg/l) that is, 1:1 or 1:1:1 system. Binary and tertiary adsorption of metal ions was conducted with the same operating conditions as for mono-component adsorption in terms of volume (50 ml), activated carbon weight (100 mg), pH (4.5), agitation time (20 h) and agitation rate (120 rpm). Initial individual concentrations of three metal ions (in binary or tertiary system) ranged from 5 to 70 mg/l. Obtained results were modelled using Langmuir and Freundlich equations.

3. Result and discussion

3.1. Mono-component adsorption of metal ions

The equilibrium isotherms were determined with respect to each metal ion, i.e. Pb(II), Hg(II) and Cd(II) in a mono-component system. Fig. 2 shows the isotherms determined for mono-component metal ion system.

For the mono-component isotherms, when concentration is expressed in mg/l, the adsorption capacities in mg/g increase in the following the order: Hg(II) > Pb(II) > > Cd(II) (Fig. 1). Adsorption isotherms were modeled following Freundlich and Langmuir equation [20,21] as described below:

A. Langmuir equation:

$$\frac{C_e}{q_e} = \frac{1}{Q_0 b} + \frac{C_e}{Q_0} \tag{2}$$

where, C_e is the final (residual) concentration (mg/l), q_e is the amount of metal uptake at equilibrium (mg/g) and Q_0 (mg/g) and b (l/mg) are Langmuir constants related to adsorption capacity and energy of adsorption, respectively. The essential characteristics of a Langmuir isotherm generally expressed in terms of dimensionless constant called equilibrium parameters, R_L, which is defined by

$$R_L = \frac{1}{1 + bC_0} \tag{3}$$

where, b is defined as Langmuir constant (l/min) and C_0 as initial mercury concentration (mg/l). R_L values were calculated using Eq. (3) for Cd(II), Pb(II) and Hg(II) ions. The value was found to be between 0 and 1 at different initial concentrations indicating favourable adsorption of all three metal ions in mono-component system [22].

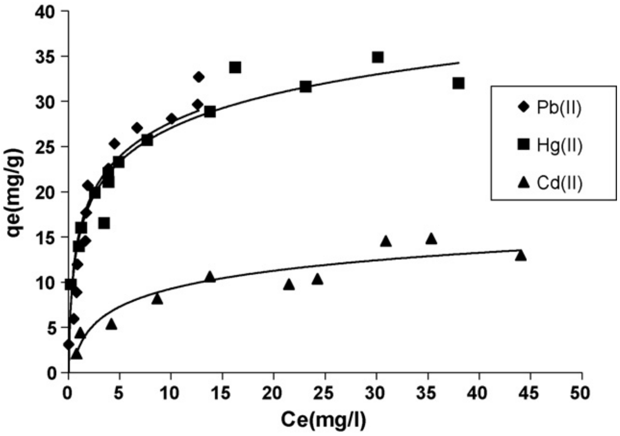

Fig. 1. Isotherm curves for lead (II), mercury (II) and cadmium (II) in mono-component system using carbon aerogel.

B. Freundlich isotherm

$$\log_{10}\left(\frac{x}{m}\right) = \log_{10} K_f + \frac{1}{n}(\log_{10} C_e) \tag{4}$$

where, C_e is the equilibrium concentration (mg/l), x is the amount of metal removed (mg), m is weight of adsorbent used (g) and K_f ($\text{mg}^{1-1/n}\,\text{l}^{1/n}\,\text{g}^{-1}$) and n are constants incorporating all factors affecting the adsorption capacity and an indication of the favourability of metal ion adsorption onto adsorbent, respectively According to Treybal (1980) it has been shown using mathematical calculations that n values between 1 and 10 represent beneficial adsorption [23].

Maximum adsorption capacity, Q_0 (mg/g) calculated from Langmuir isotherm and Freundlich parameter K_f as presented in Table 2 are in increasing order of Hg (II) [34.96 mg/g,

Table 2
Equilibrium models for adsorption of lead, mercury and Cadmium onto carbon aerogel in mono-component system

Metal ion	Langmuir parameters			Freundlich parameters		
	Q_0 (mg/g)	b (l/mg)	r^2	K_f (mg/g l/g)	$1/n$	r^2
Pb(II)	34.72	0.027	0.980	2.91	0.418	0.934
Hg(II)	34.96	0.483	0.992	3.19	0.258	0.946
Cd(II)	15.53	0.1641	0.956	1.40	0.540	0.836

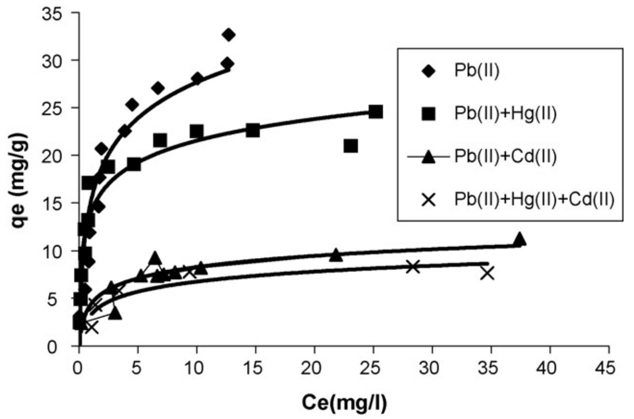

Fig. 2. Lead (II) adsorption onto carbon aerogel in Binary and tertiary system with mercury and cadmium.

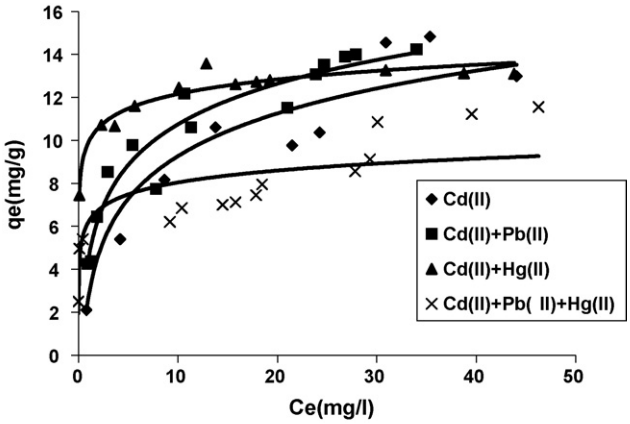

Fig. 4. Cadmium (II) adsorption onto carbon aerogel in Binary and tertiary system with mercury and lead.

3.19] > Pb(II) [34.72 mg/g, 2.91] > Cd(II) [15.53 mg/g, 1.40] which is consistent with the equilibrium adsorption isotherm curves shown in Fig. 1. Determination coefficient, r^2 of both, Freundlich and Langmuir equations, is near to unity, indicating the good agreement of experimental data with both the isotherm models in case of mono-component system.

3.2. Adsorption of metal ions in binary and tertiary system

Binary adsorption studies are particularly important for assessing the degree of interference posed by common metal ions in adsorptive treatment of wastewaters. The binary system adsorption isotherms for metal ion Pb(II), Hg(II) and Cd(II) have been given in Figs. 2–4, respectively. As depicted in the figures, for each metal ion, co-adsorption induces a decrease in equilibrium adsorption capacity but the percentage of decrease depends on co-metal ion present in the system.

Adsorption behaviors of the metal ions in binary system have been modeled using Langmuir and Freundlich equation as presented in Table 3. Faur-Brasquet et al. reported competitive adsorptive of Pb(II), Hg(II) and Cd(II) using activated carbon cloth on the basis of ionic properties of metal ions [24]. The hydrated radii of metal ions: Pb^{2+} (0.401 nm), Hg^{2+} (0.422 nm) and Cd^{2+} (0.426 nm); therefore Pb^{2+} have more accessibility to the surface and pores than other two cations which leads to the higher adsorption of lead (II). The ionic properties of three adsorbate metal ions [Pb(II), Hg(II) and Cd(II)] have been provided in Table 4. Lead (II) has the maximum hydrated radii and molecular weight in comparison to Hg(II) and Cd(II).

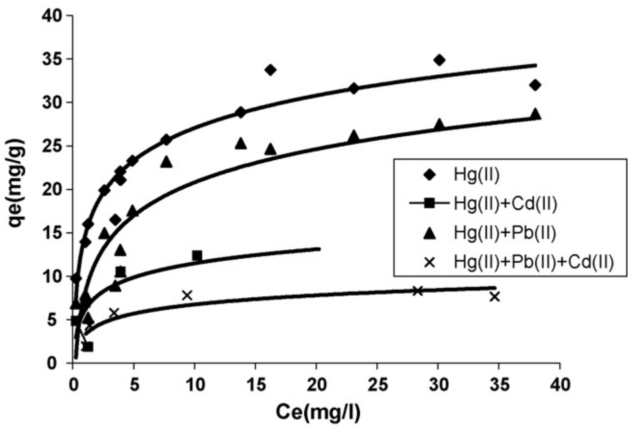

Fig. 3. Mercury (II) adsorption onto carbon aerogel in Binary and tertiary system with cadmium and lead.

As depicted in the figures, for each metal ion, co-adsorption induces a decrease in equilibrium adsorption capacity but the percent of decrease depends on co-metal ion present in the system Kandah et al., Singh and Yenkie, Flogeac et al. and Sharma et al., has reported similar results using waste ship manure waste, granular activated carbon columns, France soil and shelled Moringa oleifera seeds, respectively, as adsorbents [25–28].

Previous studies have explained the removal of metal ion in adsorption competitive systems on the basis of comparative assessment of their initial adsorption rates [29,30].

Adsorption behavior of metal ions in binary system was modeled using Langmuir and Freundlich isotherm equation as predicted in mono-component system. Values of Langmuir and Freundlich parameter for each metal ion of binary system, that is, Cd(II) + Pb(II), Pb(II) + Hg(II) and Hg(II) + Cd(II), and their respective determination coefficient r^2 values are presented in Table 3.

Langmuir equation was also applied with respective lead isotherm in presence of mercury and cadmium for a binary and tertiary system. For instance, Q_0 maximum adsorption capacity of Pb(II) was found to 23.31, 12.38 and 8.4 mg/g for binary systems [Pb(II) + Hg(II) and Pb(II) + Cd(II)] and tertiary system [Pb(II) + Hg(II) + Cd(II)], respectively. It can be easily observed that sorption is decreased in the presence of other bivalent metal ions. This is due to screening effect of surface charge produced by the added metal ions [7,11].

Fig. 1 presents isotherms curves obtained for co-adsorption of Pb(II), Hg(II) and Cd(II) in tertiary adsorption system of Pb(II) + Hg(II) + Cd(II).

The equilibrium adsorption capacity for tertiary system increases in the order of Cd(II) > Hg(II) > Pb(II). Adsorption

Table 3
Modeling the results of adsorption isotherm of mono-component, binary and tertiary system

Single metal system	Metal ion	Freundlich parameters			Langmuir parameters		
		K_f	$1/n$	r^2	Q_0	b	r^2
	Cd(II)	1.40	0.540	0.836	15.53	0.1641	0.956
	Pb(II)	2.91	0.418	0.934	34.72	0.027	0.980
	Hg(II)	3.19	0.258	0.946	34.96	0.483	0.992

Binary metal system	Metal ions	Freundlich parameters			Langmuir parameters		
		K_f	$1/n$	r^2	Q_0	b	r^2
Cd(II) + Pb(II)	Cd(II)	5.06	0.3033	0.881	15.432	0.255	0.9773
	Pb(II)	4.047	0.2885	0.859	12.376	0.237	0.987
Pb(II) + Hg(II)	Pb(II)	11.49	0.2967	0.86	23.31	1.88	0.991
	Hg(II)	8.122	0.3696	.8137	32.05	0.212	0.9698
Hg(II) + Cd(II)	Hg(II)	6.587	0.3058	0.758	17.98	0.472	0.9148
	Cd(II)	9.66	0.0955	0.94	13.304	1.818	0.9993

Tertiary metal system	Metal ions	Freundlich parameters			Langmuir parameters		
		K_f	$1/n$	r^2	Q_0	b	r^2
Cd(II) + Pb(II) + Hg(II)	Cd(II)	2.377382	1.855	0.9839	11.57	0.1843	0.910
	Pb(II)	4.44	0.182	0.858	8.401	0.56	0.991
	Hg(II)	3.81	0.105	0.88	2.197	0.360	0.991

Table 4
Ionic properties of metal ion

Property	Cd	Pb	Hg
Formula	Cd(II) from $CdSO_4$	Pb(II) from $Pb(NO_3)_2$	Hg(II) from $HgCl_2$
Mol.Wt.	–	207	200
Electronic configuration	$[Kr]\ 4d^{10}5s^2$	$6s^2 4f^{14} 5d^{10} 6p^2$	$5s^2 5p^6 5d^{10} 6s^2$
Ionic radii (Å)	0.97	1.12	1.02
Hydrated radii (Å)	4.26	4.01	4.22
Pauling EN	1.69	1.87	1.9

behavior of metal ions in tertiary system was modeled using Langmuir and Freundlich isotherm equation as predicted in mono-component and binary system. Values of Langmuir and Freundlich parameters for each metal ion of tertiary system, that is, Pb(II) + Hg(II) + Cd(II), and their respective determination coefficient r^2 values are presented in Table 3.

4. Conclusions

The equilibrium sorption of three metal ions—lead, mercury and cadmium onto carbon aerogel has been studied for three mono-component and binary systems and ternary. Studies on equilibrium quantitative sorption in mono-component, and binary system have revealed that competition has occurred. The results show that the perfectly adsorption of the following metals in the order of Hg(II) > Pb(II) > Cd(II) in a single systems. In the binary and tertiary systems, the sorption is suppressed by the presence of other metal ions in aqueous solution.

The equilibrium data have been analyzed for each metal ion in each system using Langmuir and Freundlich models. Both Langmuir and Freundlich models can be used to fit the data and estimate model parameters but certainly not in all the cases of adsorbent and adsorbate system. The presence of the three metal ions even in competitive presence of each other onto CA has evidenced using SEM and EDAX analysis.

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