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Modeling Cadmium Adsorption by Activated Carbon Using the Langmuir and Freundlich Isotherm Expressions

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ABSTRACT

Cadmium adsorption isotherms were conducted using two commercially available powdered activated carbons (PACs). Isotherms were conducted at several pH values and metal and carbon concentrations. Both PACs removed significant amounts of cadmium, and removal was a strong function of solution pH: increasing the solution pH increased cadmium adsorption. Adsorption data at a specific pH were successfully modeled using both the Langmuir and Freundlich isotherms. Adsorption and surface precipitation were hypothesized to be the operative removal mechanisms. Cadmium removal is strongly related to the carbon's pH_{zpc}, acid-base characteristics, and surface charge–pH relationship. Surface area, an important adsorption parameter for organic adsorbates, does not appear to influence metal removal strongly.

INTRODUCTION

The presence of heavy metals in the environment is a major concern due to their toxicity to many life forms. Unlike organic pollutants, the majority of which are susceptible to biological degradation, heavy metals will not degrade into harmless end products. Thus, treatment of aqueous wastes containing soluble heavy metals requires concentration of the metals into a smaller volume followed by recovery or secure disposal.

Examples of wastes containing heavy metals include plating wastes, spent solvents, landfill leachates, and contaminated groundwater at abandoned hazardous waste sites. Precipitation is the most common method of metal removal for wastewaters with high heavy metal concentrations. In the future, as heavy metal discharge standards become more stringent, additional metal removal processes, downline from the precipitation process, will be required.

Heavy metals can be removed by adsorption on solid phases. Activated carbon, metal oxides, and ion-exchange resins have been used to remove and concentrate trace solutes from waste and drinking waters. Recently, Reed (1), Reed and Nonavinakere (2), Reed and Matsumoto (3), and Matsumoto et al. (4) reported that significant amounts of heavy metals were removed from solution by several commercially available powdered activated carbons. Reed and Nonavinakere (2) reported that several activated carbons were able to adsorb cadmium and nickel from waste streams containing several organic and inorganic ligands. Matsumoto et al. (4) reported that the addition of a Darco brand powdered activated carbon to a biological sequencing batch reactor successfully mitigated the adverse effects of several heavy metals on the biological removal of organic compounds. Preferential adsorption of the metals by the activated carbon, over that of the biological solids, was reported to be the cause of the improved treatment performance. Reed and Arunachalam (5) demonstrated the efficacy of using granular activated carbon columns for the removal of lead and cadmium from an aqueous waste stream. Based on these and other literature results (6, 7), the removal of heavy metals from aqueous waste by activated carbon appears to be a feasible treatment option.

In the environmental treatment field, adsorption by activated carbon is represented mathematically by either the Freundlich or Langmuir isotherms. Freundlich and Langmuir isotherm constants for numerous organic compounds are available in the literature. Thus, the decision on whether to consider activated carbon as a treatment option for organic compounds is fairly straightforward. Presently, there is no such database available for metal adsorption by activated carbon.

STUDY OBJECTIVES

If activated carbon is to be considered as a viable treatment method for metal-bearing waste streams, adsorption data and the corresponding isotherm parameters must be generated and made available to the scientific and engineering communities. With this in mind, the objectives of this study were to:

1. Investigate heavy metal removal by two commercially available activated carbons.
2. Determine to what extent the Freundlich and Langmuir isotherms can be used to represent the adsorption data.

SELECTED BACKGROUND INFORMATION

Heavy Metal Adsorption by Activated Carbon

Activated carbons develop a surface charge and exhibit amphoteric properties when added to water. This phenomenon is attributed to such surface functional groups as phenolic, carboxyl, *n*- and *f*-lactones, and quinone (8, 9). Surface groups responsible for the coordination and release of H^+ are also assumed to be responsible for the specific adsorption of metal ions. The existence of a surface charge causes an increase in counterions near the charged surface, forming an electric double layer (EDL). Thus, the free energy of adsorption can be defined as the sum of the free energy of chemical interaction at the solid surface and an electrostatic component:

$$\Delta G_{\text{adsorption}} = \Delta G_{\text{chemical}} + \Delta G_{\text{electrostatic}}$$

The surface charge is a function of pH. The pH at which the net charge on the solid surface is zero is referred to as the pH at the zero point of charge (pH_{zpc}). Below pH_{zpc}, the surface has a net positive charge; above pH_{zpc}, the surface has a net negative charge. Thus, for cations, as the pH increases, the electrostatic component of the free energy of adsorption increases.

Adsorption of metal cations onto hydrous solids is strongly dependent on the pH of the solution (10). The dependence of metal adsorption on pH is due in part to the amphoteric nature of hydrous solids and the solution chemistry of the metal ion. For metals in a ligand-free system, adsorption generally increases with solution pH. The fraction of metal ions removed from solution increases from near zero to 1 over a narrow pH range. The metal removal curve versus pH is referred to as the "pH–adsorption edge."

Langmuir and Freundlich Isotherms

Adsorption data are usually mathematically represented by either the Langmuir or Freundlich isotherms. In the following paragraphs, brief descriptions of the Langmuir and Freundlich isotherm are given.

Langmuir Isotherm

Consider the surface coordination reaction

$$X + C \Leftrightarrow XC \tag{1}$$

where C is the equilibrium solution phase concentration of the adsorbate and X is an adsorptive site on the solid. The mass law for this reaction can be written as

$$K_{ads} = \{XC\}/\{X\}\{C\} \tag{2}$$

and the mass balance for X is

$$\Gamma_{max} = X + XC \tag{3}$$

Assuming that the thermodynamic activities of the surface sites are proportional to their concentration and that the total number of adsorptive surface sites is constant, the equations representing the mass balance on X and the mass action law can be combined to form the familiar Langmuir isotherm

$$\Gamma = \frac{\Gamma_{max}K_{ads}C}{1 + K_{ads}C} \tag{4}$$

where Γ represents the amount of pollutant that is sorbed on the solid (i.e., XC). Γ is often referred to as the surface concentration or the surface density, and it has units of either mg/kg or mol/kg. C has units of either mol/L or mg/L. Γ_{max} is the maximum surface density at monolayer coverage.

The Langmuir isotherm assumes that the free energy of adsorption is independent of surface coverage. Saturation of the solid surface ($\Gamma = \Gamma_{max}$) with a monolayer coverage of the adsorbate at high C values and linear adsorption at low C values are predicted by the Langmuir isotherm. In general, Γ_{max} and K_{ads} are functions of pH, ionic media, and ionic strength. The effects of the electric double layer and the amphoteric behavior of the surface on adsorption are contained in Γ_{max} and K_{ads}.

Values of K_{ads} and Γ_{max} can be determined graphically by linearizing and rearranging Eq. (4):

$$\frac{C}{\Gamma} = \frac{1}{K_{ads}\Gamma_{max}} + \frac{C}{\Gamma_{max}} \tag{5}$$

When C is plotted versus C/Γ, Γ_{max} is equal to the inverse of the slope and K_{ads} is determined from the slope and intercept. Note that X, the "concentration" of surface sites, does not enter explicitly in the calculations.

Freundlich Isotherm

The Freundlich isotherm was originally derived as an empirical relationship. Later, however, it was shown that the Freundlich isotherm can be derived from the assumption that the probability of a bond having an adsorption energy between ΔG and $\Delta G + d\Delta G$ is proportional to $\exp(-\Delta G/RT)$ (11). The mathematical expression of the Freundlich isotherm is

$$\Gamma = AC^{1/n} \tag{6}$$

where C and Γ have the same definitions presented for the Langmuir isotherm. A and n are constants that can be related to the strength of the adsorptive bond and bond distribution, respectively (11). If $n < 1$, bond energies increase with surface density; if $n > 1$, bond energies decrease with surface density; and when $n = 1$, all surface sites are equivalent. When $n = 1$ the Freundlich isotherm is mathematically equivalent to the Langmuir isotherm when either $\Gamma_{\max}$ goes to infinity or $K_{\text{ads}} \ll 1$. The parameter A is equivalent to K_{ads} in the Langmuir isotherm. The situation $n > 1$ is most common and may be due to a distribution of surface sites or any factor that causes a decrease in adsorbent–adsorbate interaction with increasing surface density (e.g., unfavorable adsorbate–adsorbate interactions on the solid surface). The Freundlich isotherm does not predict saturation of the solid surface by the adsorbate, thus infinite surface coverage is predicted mathematically.

The Freundlich isotherm is often used to model limited segments of experimental data. Values of A and n at various values of pH and ionic strengths are usually required to describe a complex system. The effects of the EDL and the amphoteric behavior of the surface on adsorption are contained in the coefficients A and n. Values of A and n may be calculated by taking the logarithm of Eq. (6):

$$\log \Gamma = (1/n) \log \mathrm{C} + \log A \tag{7}$$

and plotting $\log \Gamma$ versus log C. The slope is equal to $1/n$ and the intercept to $\log A$.

Aqueous Chemistry of Cadmium

The hydrolysis and precipitation of the metal ion affects adsorption by changing the concentration and form of soluble metal species that are available for adsorption. In the absence of complexing agents, the solubility of cadmium is governed by hydroxide or carbonate concentration. The following reactions are then responsible for the behavior of cadmium in solution,

$$Cd^{2+} + H_2O \Leftrightarrow Cd(OH)^{+} + H^{+}, \quad pK_1 = 9.0 \qquad (8)$$

$$Cd^{2+} + 2H_2O \Leftrightarrow Cd(OH)_2 + 2H^{+}, \quad pK_2 = 19.1 \qquad (9)$$

$$Cd^{2+} + 3H_2O \Leftrightarrow Cd(OH)_3^{-} + 3H^{+}, \quad pK_3 = 30.3 \qquad (10)$$

$$Cd^{2+} + 2H_2O \Leftrightarrow Cd(OH)_2(s) + 2H^{+}, \quad pK_s = 13.6 \qquad (11)$$

$$Cd^{2+} + CO_3^{-2} \Leftrightarrow CdCO_3(s), \quad pK_s = 11.3 \qquad (12)$$

Equilibrium constants in Eqs. (8) through (12) are from Westall et al. (12). Based on thermodynamics, cadmium solubility is controlled by $CdCO_3(s)$. However, Boomhower (13) has demonstrated that cadmium solubility is controlled by cadmium hydroxide. The experiments in the present study were conducted in a manner similar to those conducted in the Boomhower study. The theoretical soluble cadmium speciation diagram, in the absence of precipitation (i.e., Eqs. 8 through 10), is presented in Fig. 1. Hydrolysis of cadmium becomes significant at approximately pH 8. Also presented in Fig. 1 are the theoretical and experimental precipitation curves for $Cd(OH)_2(s)$. Precipitation begins at pH values of 9.2 and 8.6 for cadmium concentrations of 5 mg/L (4.45×10^{-5} M) and 50 mg/L (4.45×10^{-4} M), respectively. The solid lines represent the results using Eqs. (8) through (11). Based on the closeness of the theoretical results to the experimental data, it was confirmed that cadmium solubility was controlled by $Cd(OH)_2(s)$.

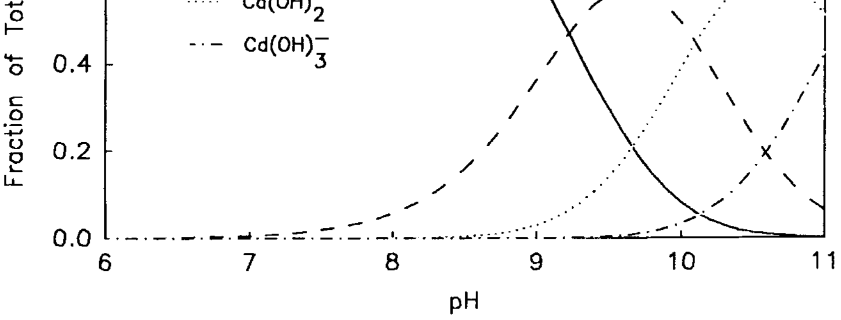


FIG. 1A

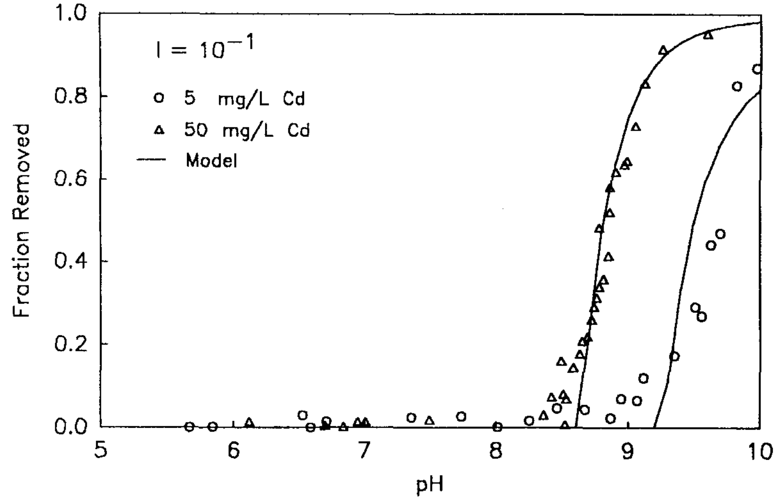


FIG. 1B

EXPERIMENTAL APPROACH AND METHODS

Two commercially available powdered activated carbons (PACs) were investigated: Darco HDB (American Norit Company) and Nuchar SN (Westvaco Company). Selected properties of the study carbons are presented in Table 1. Both carbons underwent the washing procedure suggested by Huang (14) prior to all experiments. The procedure consisted of washing the carbons with 0.5 N NaOH and then rinsing them with distilled water until the conductivity was less than 10^{-3}. The wash procedure was then repeated using 0.5 N HNO_3. The activated carbons were stored in 0.1 N $NaNO_3$.

Cadmium was chosen as the study heavy metal because of its high solubility and widespread industrial use. Cadmium was analyzed by flame atomic adsorption spectrophotometry using Method 310A of *Standard Methods* (15) and a Perkin-Elmer 560 Atomic Adsorption Spectrophotometer. A wavelength of 228.8 μm was used and, if necessary, samples were diluted with distilled water. All samples were filtered through 0.45 μm membrane filters and acidified prior to cadmium measurement.

A short equilibrium period of 6 hours was used for the metal adsorption experiments. Cadmium adsorption kinetic experiments were conducted to determine the magnitude of the error associated with using a short equilibrium time. The results of the kinetic experiments, which have been reported elsewhere (3), indicate that the error associated with using the

TABLE 1
Properties of Darco HDB and Nuchar SN[a]

Carbon	Ash (%)	S_a[b] (m^2/g)	Density (kg/m^2)	pH$_{zpc}$	Particle size distribution, (% through) 100 Mesh	200 Mesh	325 Mesh
Darco HDB	NA[c]	600–650	500	7.45	99	95	90
Nuchar SN	3–5	1400–1800	350	5.35	95–100	85–95	65–85

[a] Manufacturer's data except pH$_{zpc}$.
[b] Surface area: Nitrogen BET method.
[c] NA: Not available.

short equilibrium time was significant only at high adsorbate/adsorbent mass ratios. For Darco HDB, the average and largest difference in metal removal between the 6 hour and 2 day isotherm experiments were 3 and 9%, respectively. Results from isotherm experiments conducted at adsorbate/adsorbent ratios similar to those that did not reach equilibrium in the kinetic experiments were not included in isotherm parameter calculations. For Nuchar SN, the largest difference was less than 4% and the average difference was 2%. Using an isotherm period shorter than the equilibrium period results in a conservative estimate of the adsorptive capabilities of the carbon.

Adsorption isotherms were conducted at cadmium concentrations of 5, 10, and 50 mg/L as functions of solution pH. Carbon concentrations varied between 0.5 and 12 g/L. Experiments were conducted in the absence of carbon at cadmium concentrations of 5 and 50 mg/L to determine the removal of cadmium from solution by precipitation. Cadmium nitrate was used as the source of Cd. All experiments were conducted at an ionic strength of 0.1 ($NaNO_3$). Predetermined volumes of cadmium, carbon, and $NaNO_3$ stock solutions were added to a 1-L volumetric flask such that upon dilution to 1 L with distilled water the desired ionic strength and cadmium and carbon concentrations were achieved. Fifty milliliter aliquots were withdrawn and placed in 75 mL Nalgene bottles. The pH was adjusted by adding varying amounts of either NaOH (0.1 N) or HNO_3 (0.1 or 1.0 N) to the individual samples using micropipets. The samples were sealed and placed on a mechanical shaker and agitated for 6 hours. At the end of the 6-hour period, the samples were removed, the pH of the suspension was measured, and the samples were filtered and analyzed for cadmium. Carbon concentrations were determined using Method 209A of *Standard Methods* (15). At least four bottles from each adsorbate/adsorbent ratio run were measured for carbon concentrations. The temperature ranged between 20 and 25°C.

RESULTS AND DISCUSSION

Results from cadmium adsorption experiments were initially presented as the fraction of total cadmium removed versus solution pH (i.e., as pH–adsorption edges). Examples of pH–adsorption edges are presented in Fig. 2 for 5 mg/L Cd. The fraction of cadmium adsorbed increased from

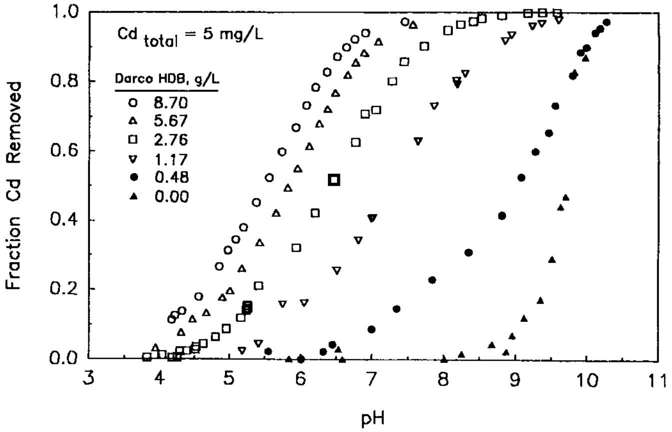


FIG. 2A

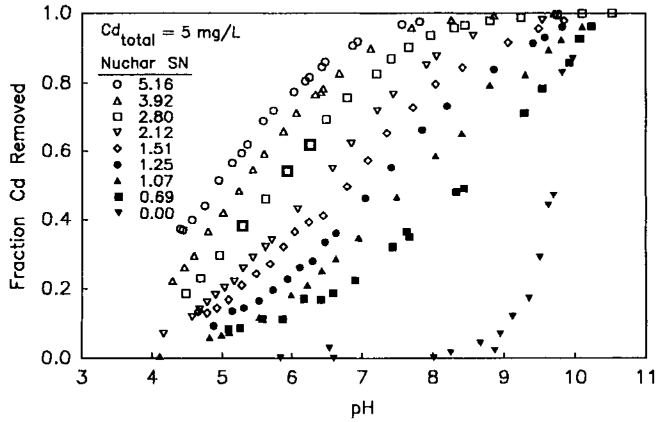


FIG. 2B

near zero to 1 over a relatively narrow pH range (2–3 pH units). The removal in the absence of carbon via solution precipitation is also presented in Fig. 2. For almost all the carbon concentrations employed, removal was complete prior to the pH at which solution precipitation begins (approximately pH 9.2 for 5 mg/L Cd). Thus, the majority of cadmium was removed from solution via interaction with the carbon surface. Cadmium removal was most likely a combination of adsorption and surface precipitation. The pH at a charged surface can be estimated by using the Boltzman equation (10):

$$\{H_s^+\} = \{H_b^+\} \exp(-F\Phi/RT) \tag{12}$$

where R, F, and T are the ideal gas constant, Faraday constant, and absolute temperature, respectively. Φ is the potential difference across the surface–liquid interface in volts. When Φ is positive (positive surface charge, $\mathrm{pH} < \mathrm{pH}_{\mathrm{zpc}}$), the surface pH is greater than the solution pH and the concentration of OH^- is higher at the surface than in solution. Thus, the probability of precipitation occurring at the surface is greater than in solution. The opposite is true when Φ is negative ($\mathrm{pH} > \mathrm{pH}_{\mathrm{zpc}}$). However, it should be pointed out that at high pH values, the amount of cadmium present in solution in precipitated form is negligible because the samples were acidified to pH $\approx$ 2 prior to cadmium analyses. Obviously, interactions between the surface and cadmium species exist that make residing on the surface more thermodynamically advantageous. The authors are presently conducting experiments to determine the exact form of the "sorbed" metal.

Cadmium removal data at pH values of 6, 6.5, 7, 7.5, and 8 were calculated from the pH–adsorption edges in Fig. 2 using linear interpolation. These data were then used in the Langmuir or Freundlich isotherms calculations. Cadmium adsorption data are presented in Fig. 3 in Langmuir isotherm form (i.e., as Γ_{Cd}, mg Cd adsorbed/g carbon, versus $\mathrm{Cd}_{\mathrm{equil}}$, mg/L). Values of $\Gamma_{\max}$ and K_{ads} calculated using Eq. (5) and the data in Fig. 3 are presented in Table 2. Langmuir isotherms are presented as solid lines in Fig. 4. For the majority of pH values tested, the Langmuir isotherm successfully simulated metal adsorption. $\Gamma_{\max}$ and K_{ads} generally increased with solution pH, reflecting the increased ease of removal as surface precipitation becomes operative. $\Gamma_{\max}$ appears to have been reached at all pH values presented for Nuchar SN and for pH values of 6, 6.5, and 7 for Darco HDB. $\Gamma_{\max}$ increased more rapidly with increased pH for Darco HDB than for Nuchar SN. For all but pH 6, $\Gamma_{\max}$ for Darco HDB is greater than that of Nuchar SN, indicating Darco HDB has a larger capacity for cadmium adsorption than Nuchar SN.

The contrast in removal capability of the two carbons can also be ascer-

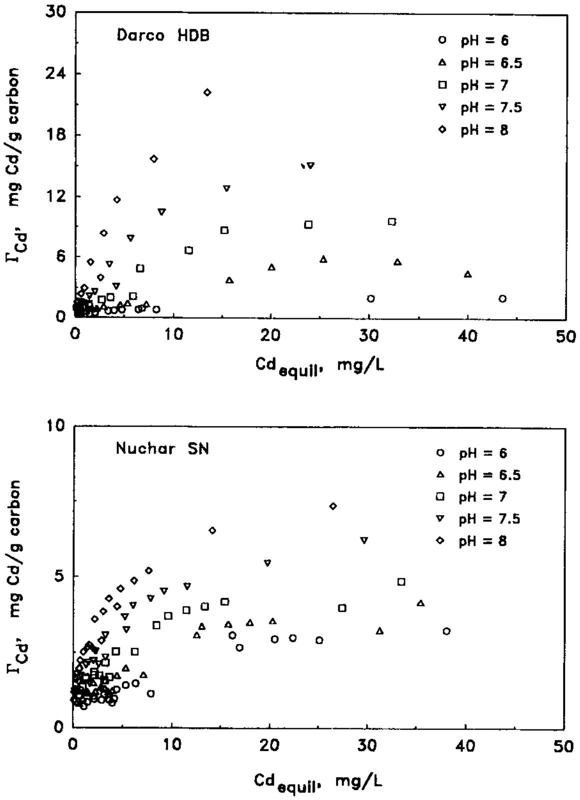


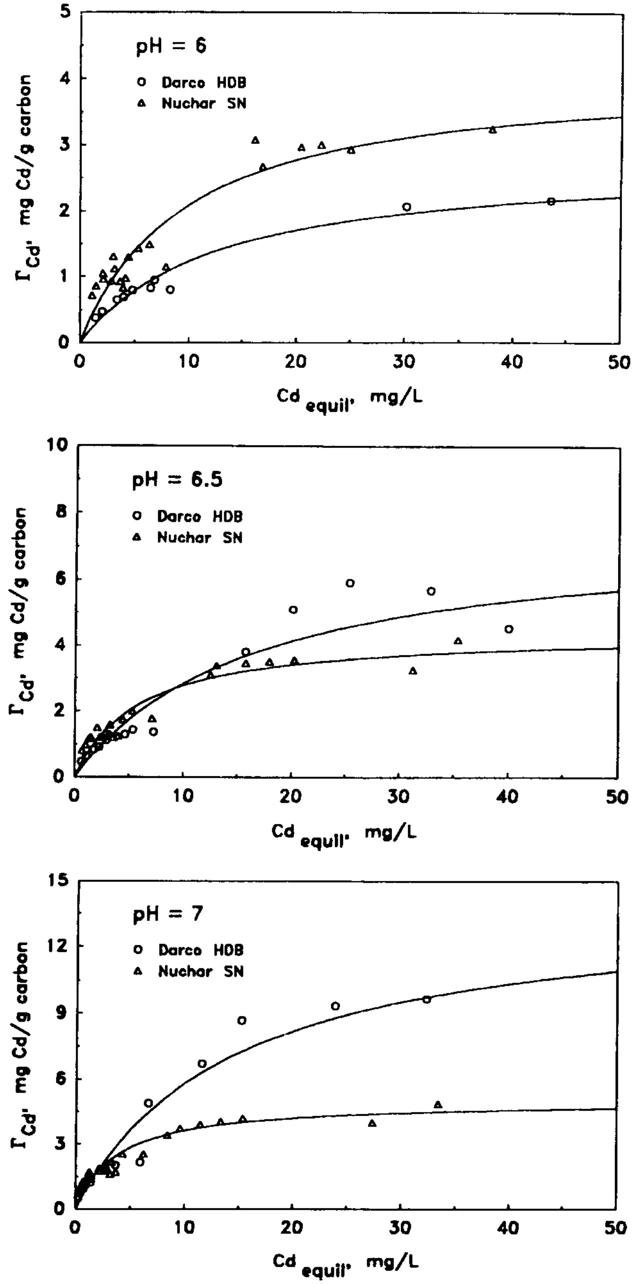
FIG. 3

TABLE 2

Values of Γ_{max} and K_{ads} for the Langmuir Isotherm

Carbon	pH	Γ_{max}[a]	K_{ads}
Darco HDB	6	2.79	0.079
	6.5	7.65	0.059
	7	14.0	0.071
	7.5	19.5	0.115
	8	28.9	0.157
Nuchar SN	6	4.12	0.103
	6.5	4.43	0.168
	7	5.02	0.262
	7.5	6.58	0.282
	8	7.67	0.412

[a] mg Cd/g carbon.


pH = 6
o Darco HDB
▵ Nuchar SN
Γ_{Cd}, mg Cd/g carbon
Cd equil, mg/L
0
1
2
3
4
5
10
20
30
40
50
pH = 6.5
o Darco HDB
▵ Nuchar SN
Γ_{Cd}, mg Cd/g carbon
Cd equil, mg/L
0
2
4
6
8
10
20
30
40
50
pH = 7
o Darco HDB
▵ Nuchar SN
Γ_{Cd}, mg Cd/g carbon
Cd equil, mg/L
0
3
6
9
12
15
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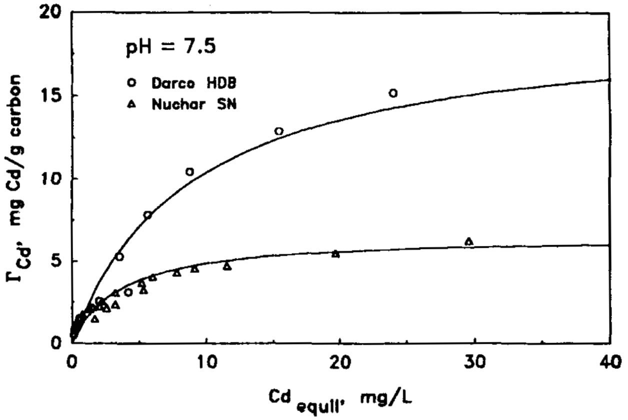
40
50

FIG. 4



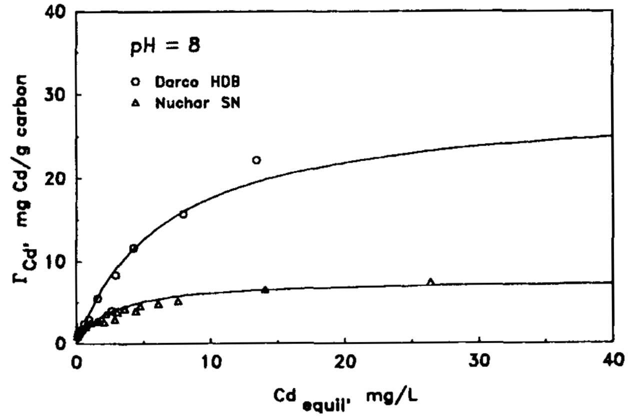


FIG. 4 Continued

tained from Fig. 4. At pH 6, Nuchar SN is superior to Darco HDB at removing cadmium from solution. At pH 6.5, both carbons have similar removal abilities up to a cadmium concentration of ~15 mg/L, after which Darco HDB is the better adsorbent. At pH values of 7, 7.5, and 8, the two carbons behave similarly for lower concentrations of cadmium. Darco HDB is the better adsorbent at larger soluble cadmium concentrations. This observation is interesting because Nuchar SN has a larger specific surface area, about 1600 m^2/g compared to about 650 m^2/g for Darco HDB. It appears that for heavy metals, unlike organic compounds, surface area may not be a critical adsorption parameter. As mentioned previously, metal removal by activated carbon is influenced by the carbon's acid-base characteristics, surface charge–pH relationship, and the type of surface sites. Based on acid-base titrations of the study carbons (8), Nuchar SN was found to be more acidic than Darco HDB. The larger concentration of OH^- at the surface of Darco HDB increases metal removal via surface precipitation. The surface charge of the carbon also affects metal removal. For Darco HDB, the surface has a net positive charge at $pH < pH_{zpc}$ (7.45) and a net negative charge at $pH > pH_{zpc}$. The pH_{zpc} for Nuchar SN is 5.35, and thus, for all the isotherms presented in this paper, Nuchar SN has a net negative charge. For the pH range investigated, cadmium exists either as a cation or neutral species (see Fig. 1). At low study pH values, the electrostatic force is repulsive for Darco HDB and attractive for Nuchar SN. The attractive electrostatic force between the Nuchar SN surface and cadmium species contributes to the superior performance of Nuchar SN over Darco HDB. At study pH values greater than the pH_{zpc} of Darco HDB, the combination of a basic surface and an attractive electrostatic force produces a better adsorbent. The sudden rise in K_{ads} for Darco HDB at $pH > pH_{zpc}$ (see Table 2) is a result of these phenomena. Finally, while the type and concentrations of surface sites affect metal removal, there is not sufficient information on the study carbons to make specific comments on this topic.

Cadmium adsorption data are plotted as Freundlich isotherms in Fig. 5 for both carbons at pH values of 6, 6.5, 7, 7.5, and 8. The solid lines represent the Freundlich isotherm equation. Values of A and n are presented in Table 3 for both carbons. Values of n at pH values of 6.5, 7, 7.5, and 8 for Darco HDB are similar (~1.56). Values of n for Nuchar SN are similar (~2.62) except at pH values of 6 and 6.5. Benjamin (11) re-

TABLE 3
Values of n and A for Freundlich Isotherm

Carbon	pH	n	A
Darco HDB	6	1.96	0.33
	6.5	1.55	0.56
	7	1.53	2.63
	7.5	1.57	2.06
	8	1.57	3.79
Nuchar SN	6	2.08	0.62
	6.5	2.34	0.48
	7	2.61	1.34
	7.5	2.58	1.76
	8	2.68	2.39

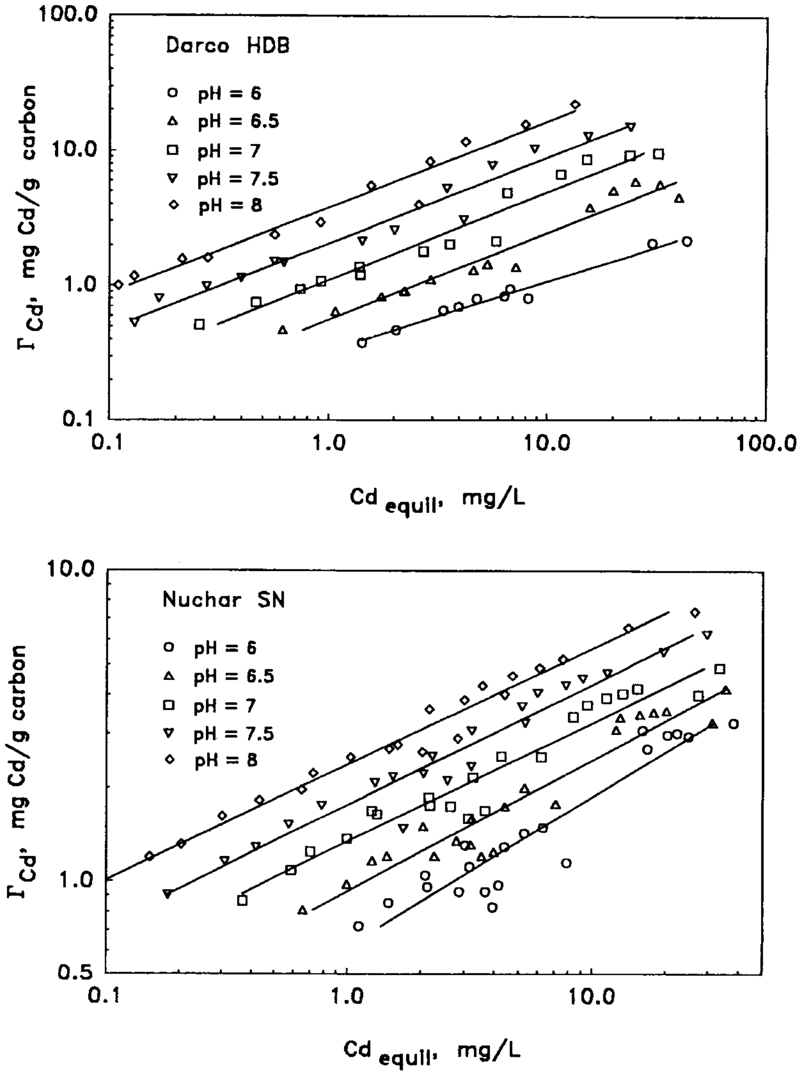


FIG. 5

ported that the greater the difference between *n* and 1, the wider is the distribution of surface bond energies. Thus, the surface of Nuchar SN may have a wider distribution of cadmium-surface bond energies than does Darco HDB.

The Langmuir and Freundlich isotherm models are not capable of accounting for change in removal with pH. If the effect of pH is to be explicitly accounted for, other models, such as the surface complex formation model (1, 3, 9), should be used.

SUMMARY

Both powdered activated carbons removed significant amounts of cadmium from solution. Cadmium removal was a strong function of solution pH. The fraction of cadmium removed increased from near zero to 1 over a relatively narrow pH range (2–3 pH units). Both the Langmuir and Freundlich isotherms successfully represented the adsorption phenomenon at a specific pH. While the isotherm models are not capable of describing removal as a function of pH, the results are useful because most treatment systems are operated at constant pH. Adsorption and surface precipitation are hypothesized to be the operative removal mechanisms. Cadmium removal is strongly related to the carbon's pH_{ZPC}, acid-base characteristics, and surface charge–pH relationship. Surface area, an important adsorption parameter for organic adsorbates, does not appear to strongly influence metal removal.

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