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Metal Adsorption by Activated Carbon: Effect of Complexing Ligands, Competing Adsorbates, Ionic Strength, and Background Electrolyte

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Abstract

Cadmium and nickel adsorption isotherms were performed using three activated carbons in ligand-free systems and in the presence of EDTA, succinic acid, PO_4 (Ni only), NH_4BF_4 (Cd only), competing metals (Ni and Cd), and differing ionic strengths and background electrolytes. Generally, all carbons removed metals from the ligand-free systems. Four scenarios were forwarded which described metal adsorption in the presence of various organic and inorganic compounds. The effect of organic and inorganic ligands on metal removal was dependent on the carbon, metal, and ligand type and concentration. The presence of a second heavy metal did not affect removal of the primary metal ion. Increasing ionic strength decreased metal removal for all carbons and metals investigated while the type of background electrolyte had no effect on metal removal.

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.

Numerous wastes contain heavy metal(s), organic pollutants, and relatively inert inorganic ions. Examples of such wastes are electro- and electroless 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. However, the effectiveness of precipitation methods depends strongly on the composition of the wastewater. The presence of

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organic and inorganic compounds can reduce the heavy metal removal efficiency. Additional metal removal processes, downline from the precipitation process, will be required as discharge standards become more stringent.

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 and Matsumoto (*1–3*) and Matsumoto et al. (*4*) reported that significant amounts of heavy metals were removed from solution by several commercially available powdered activated carbons. 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. Based on these and other literature results (*5*, *6*), the removal of heavy metals from aqueous waste by activated carbon appears to be a feasible treatment option.

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STUDY OBJECTIVES

Presently, there are limited data available in the literature describing heavy metal removal by activated carbon from a complex waste stream. If activated carbon treatment systems are to be considered as a viable treatment method for metal-bearing waste streams, additional experimental data must be gathered which quantifies metal adsorption in the presence of organic and inorganic ligands, competing heavy metals, and variable ionic strengths and background electrolytes. Therefore, the objectives of this study were to:

1. Investigate metal removal by several commercially available activated carbons in a "ligand-free"environment.
2. Examine the effects organic and inorganic ligands, competing metals, varying ionic strength, and background electrolytes have on metal removal by activated carbon.
3. Forward generalized scenarios describing the metal adsorption phenomenon in the absence and presence of various secondary compounds.

Because of space limitations, the data produced in fulfilling the first two objectives cannot be presented in its entirety. Thus, only data which illustrates generalized adsorption scenarios will be presented. The complete data set from this study has been published elsewhere (*7*).

SELECTED BACKGROUND INFORMATION

Activated carbons when added to water develop a surface charge and exhibit amphoteric properties. This phenomenon is attributed to surface functional groups, such as phenolic, carboxyl, *n*- and *f*-lactones, and quinone (*8*, *9*). The surface groups which are responsible for the coordination and release of H^+ are also assumed to be responsible for the specific adsorption of metal ions. The existence of the surface charge causes an increase of 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.

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

Mass actions laws, based on the principles presented above, have been used to successfully model metal adsorption by activated carbons (*2*, *3*).

Adsorption of metal cations onto hydrous solids is strongly dependent on the pH of the solution. Solution pH has been identified as the variable governing metal adsorption onto hydrous solids (*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 one over a narrow pH range. The metal removal curve versus pH is referred to as the "pH–adsorption edge."

The presence of organic and inorganic compounds, competing metals, varying ionic strength, and background electrolyte can alter metal removal from that observed in the ligand-free system. A metal ion which has been complexed in solution may adsorb more strongly, weakly, or the same as an uncomplexed metal species. Factors determining the effect ligands have on metal adsorption include type and concentration of ligand and metal, adsorbent type and concentration, and solution pH. In systems with more than one adsorbable ion, competition between the adsorbates for surface sites may occur. The degree of competition is dependent on the type and concentration of the competing ions, number of surface sites, and the affinity of the surface for each metal ion. The ionic strength and background electrolyte composition can affect metal adsorption by altering soluble metal chemistry and the structure of the electric double layer which surrounds the solid surface. Several scenarios illustrating the effect a secondary compound(s) can have on metal adsorption are discussed in the remaining portion of this section. A typical pH–adsorption edge for each scenario, including one representing a ligand-free system, are presented in Fig. 1.

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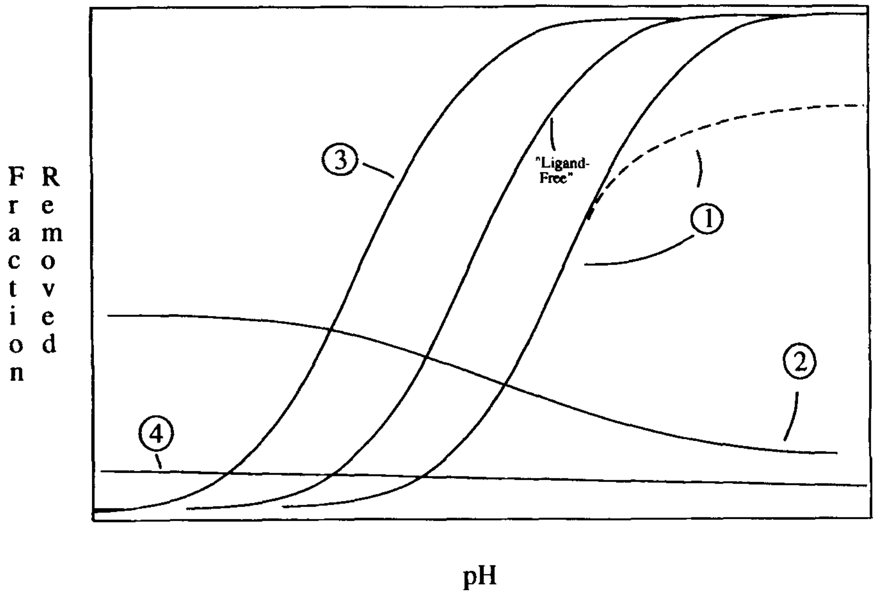

FIG. 1. Typical pH–adsorption edges for a ligand-free system and for Scenarios 1 through 4.

Scenario 1

For Scenario 1, the general shape of the pH–adsorption edge remains unchanged compared to the ligand-free system except that the adsorption edges move to a higher pH region. A plateau, defined as the leveling off of the pH–adsorption edge at a fraction removed <1.0, may also be observed (dotted line). Several circumstances can cause this behavior. The adsorbate/adsorbent concentration ratio is increased, decreasing the number surface sites per unit of metal ion. The secondary compound competes with the solid surface for the metal ion, decreasing the amount of metal ions available for interaction with the solid surface. The secondary compound may be adsorbed by the solid, decreasing the number of surface sites available for metal removal. Increasing the ionic strength will alter the metal's solution chemistry and the electric double layer. The increased amount of electrolyte can "swamp" the surface of the solid, decreasing metal access to the carbon surface.

Scenario 2

In Scenario 2, the metal-ligand complex adsorbs via a ligand–solid interaction (or equivalently, the ligand adsorbs on the solid and the metal ion reacts with the solid-ligand complex). The shape of the pH–adsorption

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edge is radically different from that observed in the ligand-free system. Adsorption increases at lower pH values and decreases at higher pH values compared with the ligand-free case.

Typically, at low pH values the surface of carbons is positively charged while at high pH values the surface is negatively charged. Metal-organic complexes are often negatively charged over a wide pH range (e.g., EDTA-metal complexes). In a ligand-free system, the electrostatic force is usually repulsive at low pH values (positively charged metal species and surface), decreasing the adsorption energy. For a negatively charged metal-ligand complex the electrostatic force is attractive, increasing adsorption energy. At higher pH values the negatively charged carbon surface and metal-ligand complex produce a repulsive electrostatic force which can decrease adsorption compared to the ligand-free system.

In addition to the electrostatic forces, the magnitude and sign of chemical interaction term will also be dependent on pH. The effect this has on metal adsorption is more difficult to predict because the carbon's affinity for the metal, ligand, and metal-ligand complex has not been quantified. Further research on this topic is recommended.

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Scenario 3

In Scenario 3, unique complexation between the metal, ligand, and carbon surface causes an increase in metal removal at all pH values. The general shape of the pH–adsorption edge is similar to the ligand-free system. This phenomenon is rarely observed. A possible example of this behavior is when at low pH values the carbon surface carries a positive charge and the metal-ligand complex is negatively charged while at higher pH values the signs of the charges are reversed. Thus, the electrostatic force is attractive (or near zero) over a wide pH range. Again, it must be noted, that the chemical affinity between the surface and the metal-ligand complex will play a major role in determining the shape of the removal curve.

Scenario 4

In Scenario 4, adsorption is suppressed at all pH values. The presence of a strong metal complexer (e.g., EDTA) or a large amount of a weak complexing agent or competing adsorbate can cause this behavior. A chelating agent may complex the metal ion to such an extent that the "free" metal concentration decreases, causing a decrease in adsorption (provided the resulting metal-ligand complex is not adsorbed). Also, a secondary compound that is strongly adsorbed by the solid can decrease the number of sites available for metal interaction.

MATERIALS AND METHODS

Three commercially available activated carbons were investigated: Darco KB, Darco HD4000 (American Norit Company Inc.), and Calgon Filtrasorb 4000 (F400) (Calgon Corporation). Darco HD4000 and Calgon F400 are granular carbons and Darco KB is a powdered carbon. Carbons were ground to 325 mesh size for adsorption experiments. Selected properties of the three carbons were provided by the manufacturers and are given in Table 1.

Cadmium and nickel were chosen as the study heavy metals. EDTA, a strong complexing agent, and succinic acid, a weak organic acid, were chosen as the organic compounds. Phosphate and ammonium fluoroborate were selected as the study inorganic compounds. Phosphate is present in electroless nickel plating baths and has been reported to be adsorbable by activated carbons (*11*), and ammonium fluoroborate is a constituent of some cadmium plating baths. Cadmium and nickel were employed in competing metal experiments. Ionic strengths of 0.01 and 0.1 were investigated, and $NaClO_4$ and $NaNO_3$ were used as the study background electrolytes.

Metal content was determined by flame atomic absorption spectrophotometry using a Perkin-Elmer 3000 Atomic Absorption Spectrophotometer. Wavelengths of 228.8 and 232 nm were used for cadmium and nickel, respectively. If necessary, samples were diluted with distilled water prior to metal analysis. All samples were filtered through 0.45 μm membrane filters prior to analysis.

NaOH and HNO_3 were used for pH adjustment. An Orion Research combination pH electrode (Model #810200) was used to measure pH. A temperature of ~22°C was used for all experiments. Prior to adsorption experiments, the carbons were hydrated for approximately 12 hours in either a 0.01 or 0.1 *N* $NaClO_4$ solution. The only exception to the hydration method was for the background electrolyte experiments in which $NaNO_3$ was used. In these experiments, Darco KB was hydrated for approximately 12 hours in 0.01 *N* $NaNO_3$.

For the ligand-free system, adsorption isotherms were conducted at several pH values and metal and carbon concentrations. Cadmium and nickel

TABLE 1
Selected Properties of the Study Carbons

Carbon	Ash (%)	pH	Molasses RE	Moisture (%)
Darco KB	1.6	4.7	288	23
Darco HD4000	23	5.2	NA	4.4
Calgon F400	7.2	8.2	38	1.9

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concentrations of approximately 10 and 50 mg/L and carbon concentrations of 0.5, 1, 3, and 5 g/L were employed. The ionic strength was held constant at 0.01 *N* (as $NaClO_4$). Isotherms were also conducted without carbon to determine the removal which would occur via solution precipitation. Predetermined volumes of metal and carbon stock solutions were added to a 0.5-L volumetric flask such that upon dilution to 0.5 L the desired metal and carbon concentration would be achieved. Fifty milliliter aliquots were placed in 75 mL Nalgene bottles and varying amounts of either NaOH (0.1 or 1.0 *N*) or HNO_3 (0.1 or 1.0 *N*) were added to the individual samples. 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 was measured, the samples were filtered, and the filtrate was analyzed for either cadmium or nickel.

EDTA and succinic acid were investigated for both metals, while PO_4 and NH_4BF_4 were studied for nickel and cadmium, respectively. Metal–ligand molar ratios of 1:0.1, 1:1, and 1:10 were employed. The isotherm procedure was similar to that described in the preceding paragraph, except that carbon concentrations of 1 and 5 g/L were used. Experiments were also conducted without carbon for each metal:ligand ratio investigated to determine the effect the ligand had on metal precipitation.

The effect of competing metals on individual metal removal was assessed by conducting batch adsorption isotherms with varying amounts of cadmium and nickel. The isotherm procedure employed was similar to the one described earlier except only 5 g/L of the particular carbon was employed. To determine the effect of a competing metal on Cd removal, experiments were conducted at 10 mg/L Cd in the presence of either 0, 5, or 10 mg/L Ni. At 50 mg/L Cd, isotherms were conducted with either 0, 25, or 50 mg/L Ni present. A similar experimental design was used to determine the effect cadmium had on nickel removal. At 10 mg/L Ni, either 0, 5, or 10 mg/L Cd was present, and at 50 mg/L Ni, either 0, 25, or 50 mg/L CD was present.

The effect of ionic strength and background electrolyte on metal removal was determined for Darco KB only. Ionic strengths of 0.01 and 0.1 (as $NaNO_3$) were used. For background electrolyte studies, 0.01 *N* $NaClO_4$ or $NaNO_3$ was used. For both sets of experiments, 3 and 5 g/L of Darco KB and 10 and 50 mg/L of either cadmium or nickel were used. Adsorption isotherms were conducted as described earlier.

RESULTS AND DISCUSSION

In this section, experimental results are presented for each of the scenarios discussed earlier and for the competing metal and background electrolyte studies. An exception is Scenario 3 behavior, which was not ob-

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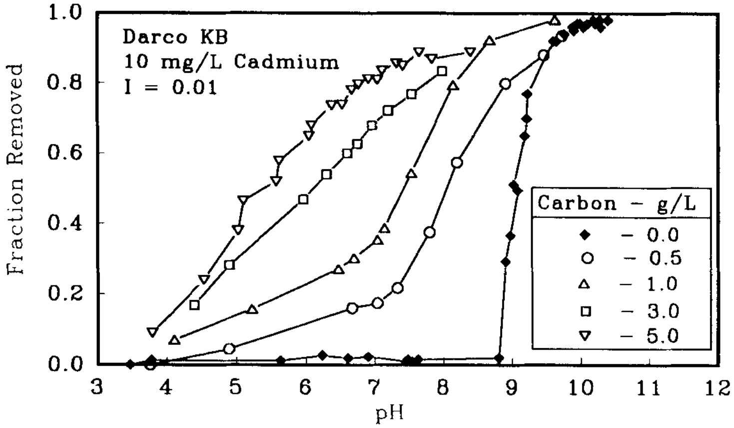

FIG. 2. pH-adsorption edges for Darco KB and 10 mg/L cadmium.

served in this study. For ease of presentation, results illustrating Scenarios 1, 2, and 4, and the effects of competing metals and background electrolyte studies are discussed individually.

Scenario 1

The pH-adsorption edges for 10 mg/L cadmium are presented in Figs. 2 and 3 for Darco KB and Darco HD4000, respectively. The pH-adsorption edges move to higher pH values as the adsorbate/adsorbent ratio increased.

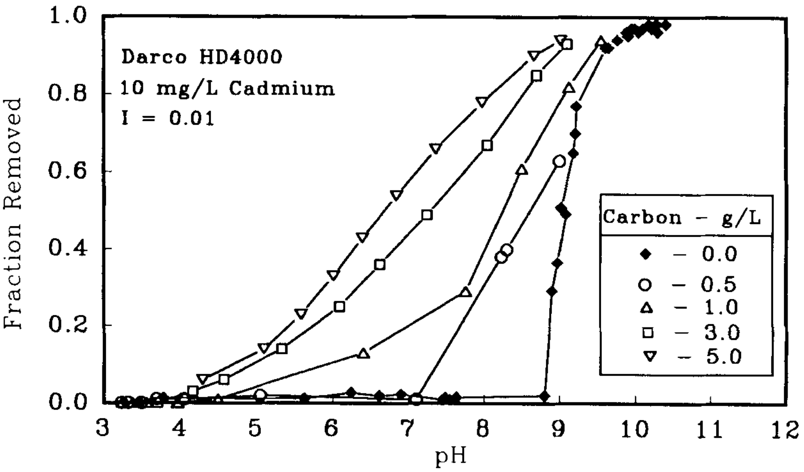

FIG. 3. pH-adsorption edges for Darco HD4000 and 10 mg/L cadmium.

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Also presented in Figs. 2 and 3 is the removal which would occur in the absence of carbon via solution precipitation. Precipitation occurred at approximately 8.5 for 10 mg/L cadmium. Darco KB and Darco HD4000 removed significant amounts of heavy metals from solution at pH values less than the pH at which precipitation occurs. Similar results were observed for the remaining carbon–metal systems.

Examples of the effects organic and inorganic ligands can have on metal removal are presented in Fig. 4 for Calgon F400–10 mg/L Ni–1:1 Ni:PO_4 and in Fig. 5 for Darco KB–50 mg/L Cd–1:1 Cd:EDTA. For the F400 system, the addition of PO_4 shifted the pH–adsorption edge to higher pH values (i.e., decreased adsorption). For Darco KB, the presence of EDTA caused both a slight shift of the removal curve to higher pH values and a plateau at higher pH values.

In Fig. 6 the pH–adsorption edges for 5 g/L Darco KB and 50 mg/L Cd at ionic strengths of 0.01 and 0.1 are presented. As the ionic strength increased, metal removal decreased. This is an important observation because a number of metal-bearing waste streams (e.g., landfill leachates, metal-plating wastes) contain high amounts of total dissolved solids.

Scenario 2

Calgon F400 pH–adsorption edges for 10 mg/L Cd–1:10 Cd:EDTA and 10 mg/L Ni–1:10 Ni:EDTA are presented in Figs. 7 and 8, respectively.

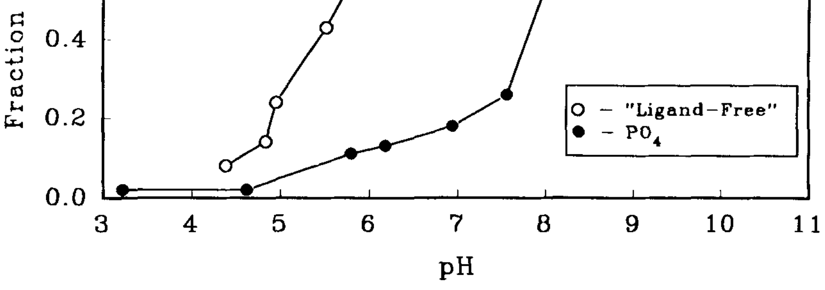

FIG. 4. Calgon F400 pH–adsorption edges at 10 mg/L nickel. Comparison of ligand-free and 1:1 Ni:PO_4 molar ratio systems.

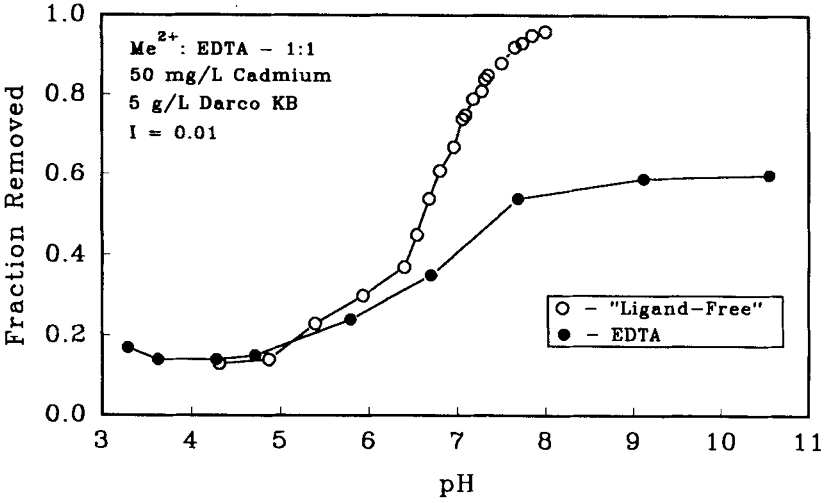

FIG. 5. Darco KB pH–adsorption edges at 50 mg/L cadmium. Comparison of ligand-free and 1:1 Cd:EDTA molar ratio systems.

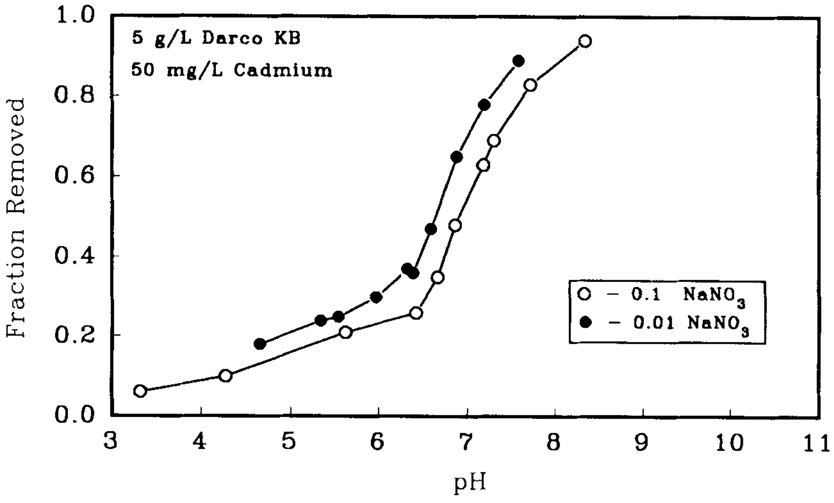

FIG. 6. Darco KB pH–adsorption edges for 50 mg/L cadmium at ionic strengths of 0.01 and 0.1.

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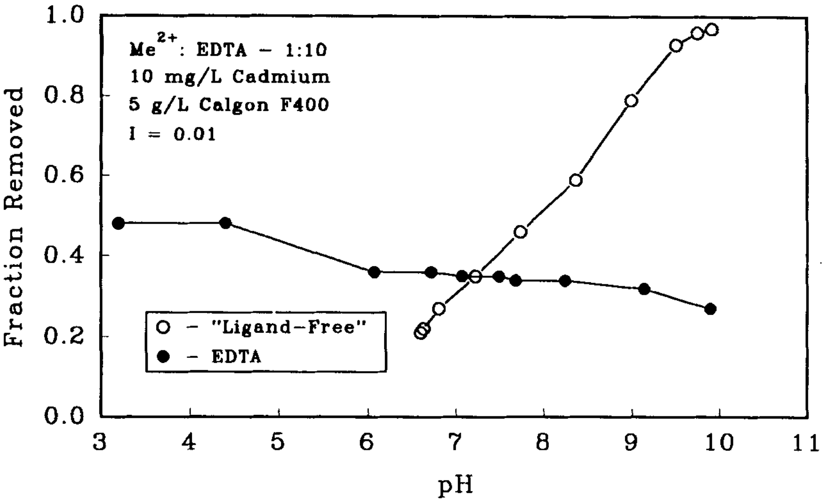

FIG. 7. Calgon F400 pH–adsorption edges at 10 mg/L cadmium. Comparison of ligand-free and 1:10 Cd:EDTA molar ratio systems.

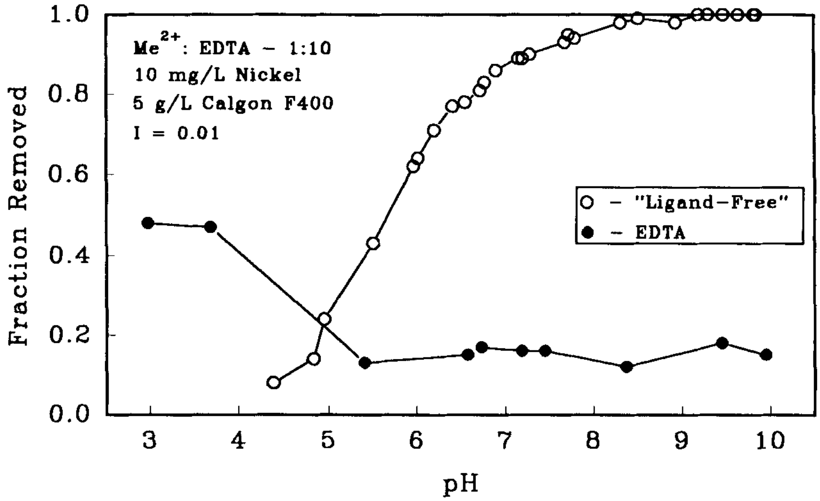

FIG. 8. Calgon F400 pH–adsorption edges at 10 mg/L nickel. Comparison of ligand-free and 1:10 Ni:EDTA molar ratio systems.

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For both systems, removal increased at lower pH values and decreased at higher pH values over that observed in the ligand-free system. The pH_{zpc} (pH at which the net surface charge is zero) for Calgon F400 was reported to be 10.4 (*9*). Below a pH of 10.4 the surface has a net positive charge, and above a pH of 10.4 the surface has a net negative charge. The EDTA complexes of cadmium and nickel carry a negative charge for the pH range investigated in this study. Thus, at lower pH values the electrostatic force is attractive. As the pH increases, this attractive force decreases until it becomes repulsive. As mentioned previously, metal adsorption cannot be explained solely by electrostatic theory. Chemical interactions between the carbon surface and a particular metal-ligand complex can also vary with pH. Also, the degree to which a carbon adsorbs the ligand will influence the extent of metal-ligand adsorption. Further laboratory research and model development and calibration are required for this phenomenon to be better understood.

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Scenario 4

In Scenario 4, metal removal is negligible over a wide pH range. An example of this phenomenon is presented in Fig. 9 for Darco KB-50 mg/L Cd-1:10 Cd:EDTA. The high concentration of EDTA decreased metal removal compared to the ligand-free system. At lower pH values there is a small amount of removal, indicating limited Scenario 2 behavior.

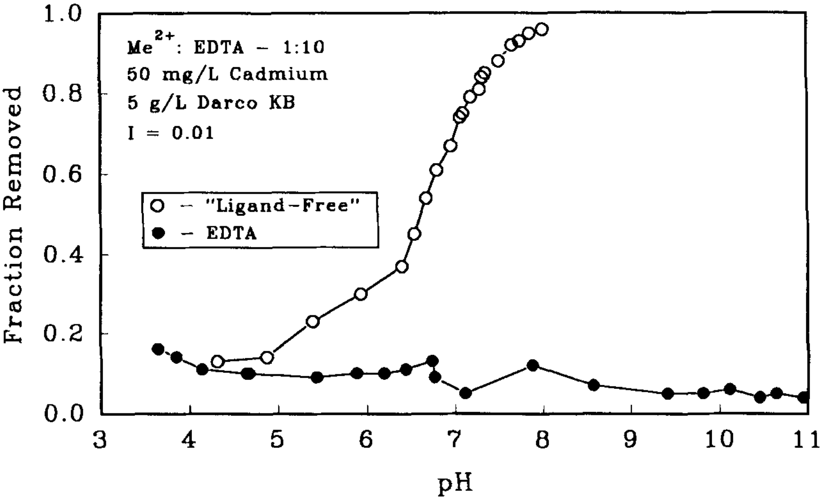

FIG. 9. Darco KB pH–adsorption edges at 50 mg/L cadmium. Comparison of ligand-free and 1:10 Cd:EDTA molar ratio systems.

Competing Metals and Background Electrolyte

The pH–adsorption edges for Darco KB and 10 mg/L Ni in the presence of 0, 5, and 10 mg/L cadmium are presented in Fig. 10. Nickel removal was not affected by the presence of cadmium. It is apparent that the sites that interact with nickel do not strongly interact with cadmium. In Fig. 11, the moles of cadmium and nickel removed by 5 g/L Darco KB at pH 7 are presented. Darco KB removed approximately the same amount of nickel from solution regardless of the initial nickel concentration. This suggests that nickel may specifically interact with a small number of surface sites. A similar observation cannot be made for cadmium. A fivefold increase in the initial cadmium concentration (10 to 50 mg/L) produced approximately the same increase in moles of cadmium removed (7.34×10^{-5} to 3.11×10^{-4} *M*). The increase in removal with increasing cadmium concentration indicates either that there are a large number of sites having a variable affinity for cadmium or that a portion of the cadmium removal is nonspecific in nature (i.e., similar to organic removal). This result was also observed for the remaining carbon–metal systems.

Regardless of the actual sorption mechanism, competition between the cadmium and nickel was minimal. This result is encouraging in that the decision to use activated carbon to treatment a multiple metal waste stream could be based on single metal adsorption isotherms. However, before

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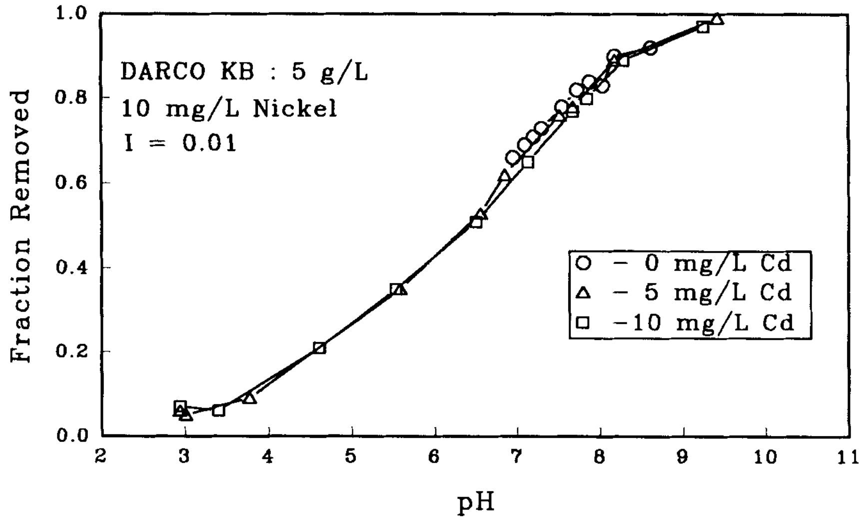

FIG. 10. Darco KB pH–adsorption edges at 10 mg/L nickel and 0, 5, and 10 mg/L cadmium. Effect of competing metals.

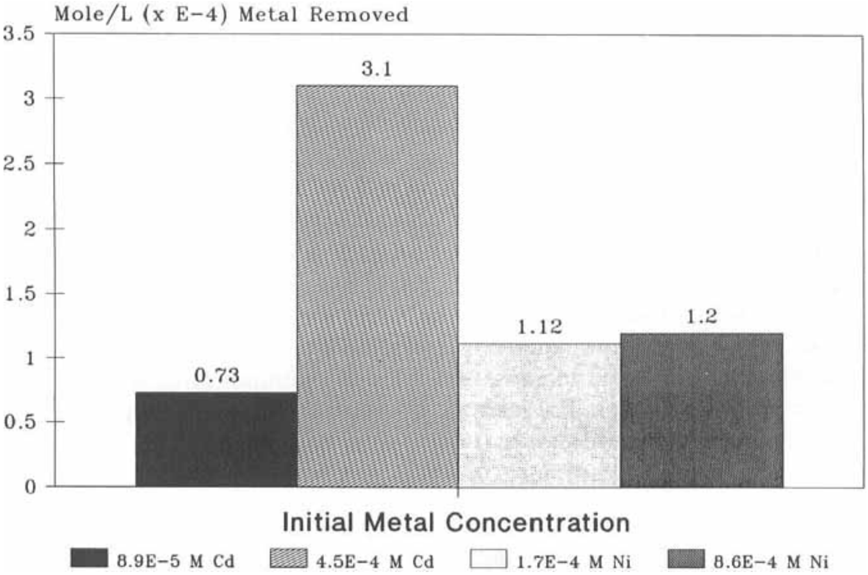

FIG. 11. Comparison of metal removal on a molar basis for Darco KB at pH 7. Effect of initial metal concentration.

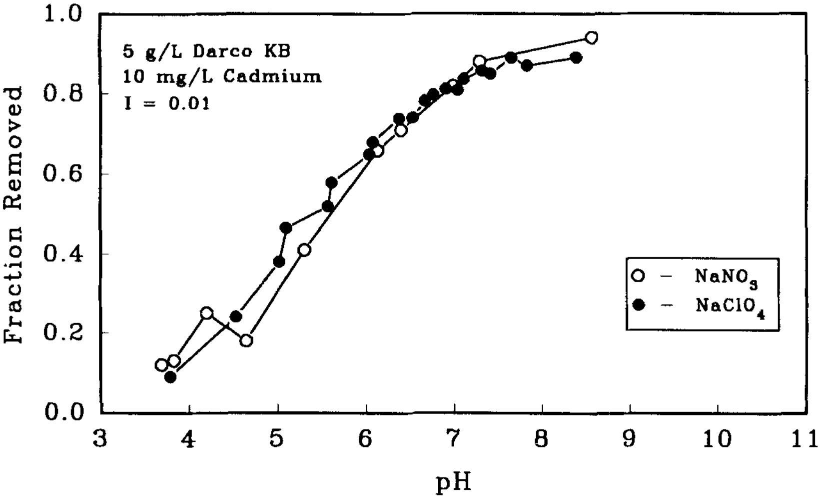

FIG. 12. Darco KB pH–adsorption edges for 10 mg/L cadmium. Effect of background electrolyte on metal removal.

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such a conclusion can be reached, further studies using other metals and carbons must be conducted.

The type of background electrolyte, either $NaNO_3$ or $NaClO_4$, did not significantly affect metal removal. An example of this phenomenon is presented in Fig. 12 for Darco KB and 10 mg/L Cd. Thus, the assumption that the background electrolyte is inert appears to be valid.

SUMMARY AND FURTHER RESEARCH

Summary

Cadmium and nickel adsorption isotherms were performed using three commercially available activated carbons, Darco KB (powdered), Darco HD4000 (granular), and Calgon F400 (granular) in ligand-free systems and in the presence of EDTA, succinic acid, PO_4 (Ni only), NH_4BF_4 (Cd only), competing metals (Ni and Cd), and differing ionic strengths and background electrolytes.

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Generally, all carbons removed metals from the ligand-free systems. The effect of organic and inorganic ligands on metal removal was dependent on the carbon, metal, and ligand type and concentration. Three of the four Scenarios forwarded to describe metal behavior in a complex waste stream were observed.

Increasing ionic strength decreased metal removal for all carbons and metals investigated, while the type of background electrolyte had no effect on metal removal. The presence of a second heavy metal did not affect removal of the primary metal ion.

Further Research

Subsequent to the conduct of this study, the following research needs have been identified:

1. Repeat batch adsorption experiments using other carbons, metals, and ligands.
2. Determine the efficacy of using a granular activated carbon (GAC) column to remove metal(s) and investigate regeneration schemes.
3. Repeat batch and GAC column experiments with an actual metal-bearing waste stream (e.g., landfill leachate).
4. Develop mathematical models that describe metal removal from batch and column reactors.

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