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The Adsorption and Desorption Characteristics of EDTA–Chelated Copper Ion by Activated Carbon

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ABSTRACT

This paper addresses the effect of EDTA, a strong agent, on the removal of copper ion from solutions using activated carbon adsorption. Experimental studies indicate the presence of EDTA significantly altered the adsorption behavior of copper on the activated carbon due to the formation of copper chelate species in the solution. The adsorption isotherms and kinetics were found to be strong functions of solution pH and the ratio of copper ion and EDTA concentrations. Adsorption of EDTA–Cu chelates was found to be more favorable than those of free copper ion and unbound EDTA species in the solution. Experimental results indicated that the desorption of chelated copper ion from activated carbon by NaOH and $HClO_4$ solutions was influenced by the initial adsorption conditions. A significantly higher quantity of copper ion was recovered with $HClO_4$ than with NaOH. Combining the adsorption and desorption data of copper and EDTA, and an understanding of the species distribution of copper in the presence of EDTA, the behavior of the adsorption of EDTA–chelated copper on the activated carbon was described. The predominant adsorbed copper species was the chelated form, $CuEDTA^{2-}$, which can be adsorbed on activated carbon surfaces with either the Cu end or the EDTA end bonding directly to the surface.

Key Words. Copper–EDTA removal; Adsorption; Activated carbon

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INTRODUCTION

The removal of heavy metals has been a serious concern for the treatment of a variety of industrial wastewaters, especially for the wastewaters from some metal plating and finishing industries that may contain complexing ions such as ethylenediaminetetraacetate (EDTA), tartrate, ammonia, etc., which may inhibit the removal of heavy metals by conventional precipitation treatment. Hence the adsorption processes have been proposed as an alternative separation technique. Several studies have shown that the presence of organic complexing agents in solutions may enhance the adsorption characteristics of metal species onto solid surface. Huang and coworkers (1–4) studied several heterogeneous metal–liquid–solid aqueous systems, and they indicated that the adsorption of metal species by various adsorbents could be improved substantially by the presence of complexing agents. They found that adsorbent type, pH, and surface loading were the most important factors affecting the extent of metal removal. Bhattatcharyya and coworkers (5, 6) also reported that the adsorption of heavy metal by activated carbon was enhanced by the presence of organic ligands and was a function of metal and ligand types, ligand-to-metal ratio, pH, and metal chelate species distribution, and proposed that the adsorption capacity was related with the electrostatic interaction between the charges of solutes and the surface of activated carbon. Contrarily, a few researchers have reported that the presence of ligand might inhibit the adsorption of heavy metals onto solid surfaces. Benjamin and Leckie (7) found that complexation by nonadsorbing ligands, such as Cl^- and SO_4^{2-}, would decrease trace metal adsorption on oxide solids, such as γ-Al_2O_3, α-SiO_2, etc., because of the competition for metal ions between the ligands and the surface adsorption sites. Peters and Shem (8) found the presence of EDTA decreased the affinity of lead to be adsorbed onto soil, thus EDTA could be used to extract adsorbed lead from soil. Benjamin and Leckie (9) proposed a conceptual model to account for these changes. Their model considers complexed species to be either "metal-like" or "ligandlike" depending on whether adsorption of the chelated metal increases or decreases with increasing pH, respectively.

In this study an attempt was made to examine factors which govern the mode and extent of the adsorption and chemical desorption behaviors of copper–EDTA complexes with an activated carbon surface. Combining these desorption data and adsorption results, an adsorption mechanism is proposed based on a consideration of electrostatic interaction and complexation of species.

EXPERIMENTAL

Stock solutions used in this investigation were prepared with reagent-grade chemicals and RO-deionized water. Standard acid and base solutions ($HClO_4$ and NaOH) were prepared weekly for pH adjustment. The granular activated carbon Norit PK 3-5 was used as received without further treatment; its characteristics are shown in Table 1. The pore size distribution of this carbon was determined by the Barrett–Joyner–Halenda (BJH) method using a Micromeritics ASAP 2000 analyzer, and the pH_{zpc} was determined by direct titration.

Batch adsorption experiments were performed for all equilibrium studies. After mixing for a 24-hour period, the equilibrium pH values were measured and the sample was filtered for copper and EDTA analyses. Kinetic experiments were carried out with equimolar metal–ligand solutions, the solution pH was held constant throughout the experiment, and continuous mixing was maintained. At intermittent periods of reaction time a portion of the reactor solution was adopted and filtered for copper and EDTA analyses. For desorption experiments, activated carbon adsorbed Cu–EDTA at constant pH values was desorbed by NaOH and $HClO_4$ solutions with different sequences. In all the above experiments the temperature of solution was maintained at 25.0 ± 0.5°C. The concentrations of copper were analyzed by a GBC 904AA atomic adsorption spectrophotometer, and the concentrations of EDTA were determined with an O.I.C. 700 total organic carbon analyzer.

TABLE 1
Characterization of Activated Carbon

Physical properties and specification	Norit PK 3-5
Total pore volume[a] (cm^3/g)	1.2
Pore volume of pores[b] between 17.0 and 3000.0 Å	0.600
Surface area (BET)[a] (m^2/g)	750
Surface area (BET)[c] (m^2/g)	587
pH value[a]	9–10
pH_{zpc}[d]	8.20 ± 0.05

[a] Data provided by the manufacturer (10).
[b] Determined by BJH cumulative desorption.
[c] Determined by BET.
[d] Determined by direct titration method.

RESULTS AND DISCUSSION

The experiments were conducted over a wide range of pH in order to study the effect of chemical species and the surface charge of the activated carbon on the adsorption of chelated copper in solution. The molar ratio of metal to ligand was also varied in order to investigate the competitive adsorption among Cu–EDTA complexes, free EDTA, and free copper.

The Effect of pH

Several experiments were conducted to study the adsorption behavior under various solution pH conditions for Cu^{2+} only, Cu–EDTA chelates, and EDTA only systems, and the results are shown in Fig. 1. In the absence of EDTA, copper removal from solutions by activated carbon

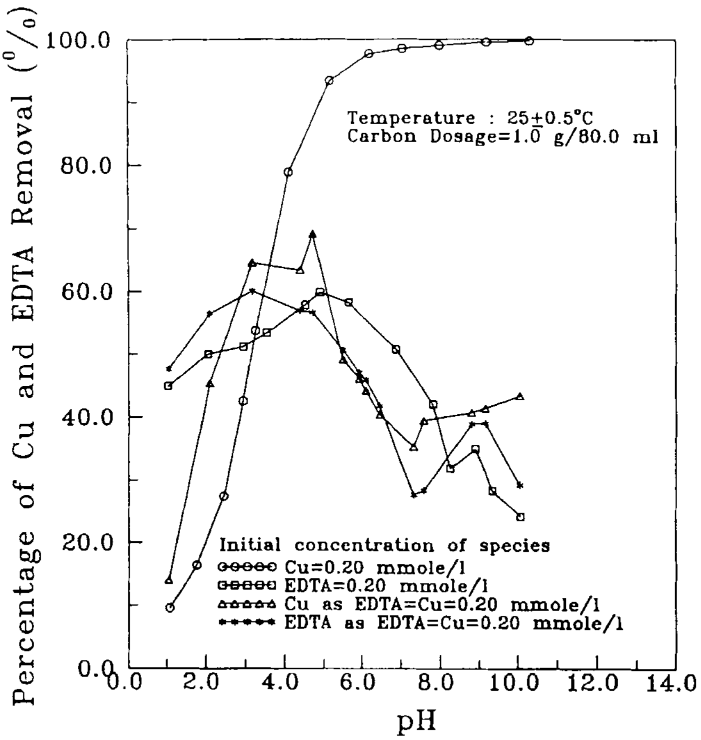


FIG. 1 Comparison of the removal efficiencies of copper and EDTA for different adsorbate systems as a function of pH.

adsorption was observed to increase with increasing pH value. The obviously increasing removal of copper ions in alkaline solutions may be due to the formation of copper hydroxide precipitates in solutions which start at a pH greater than 4. For EDTA/Cu = 1 systems, precipitation was completely inhibited, and the adsorption behavior was found to be highly influenced by solution pH. The calculated copper species distribution in the presence of EDTA as a function of pH, based on the mass balances of reacting species shown in Fig. 2, indicates that the free copper ion, Cu^{2+}, was the dominant species for pH less than 2, while the concentrations of negatively-charged $CuHEDTA^-$ and $CuEDTA^{2-}$ species were dominant for pH greater than 2. Since the pH_{zpc} of activated carbon was determined to be 8.20 ± 0.05, the carbon surface carries more positive charges in acidic solutions. The positively-charged Cu^{2+} species is assumed to exhibit a lower affinity for the positively-charged carbon surface in comparison with the negatively-charged $CuHEDTA^-$ and $CuEDTA^{2-}$ chelates. For solution pH greater than 6, the neutral and negatively-charged groups on the surface of activated carbon are assumed to increase gradually. Electrostatically, $CuEDTA^{2-}$ would be repelled from these negatively-charged surface groups or lack an electrostatic affinity for these

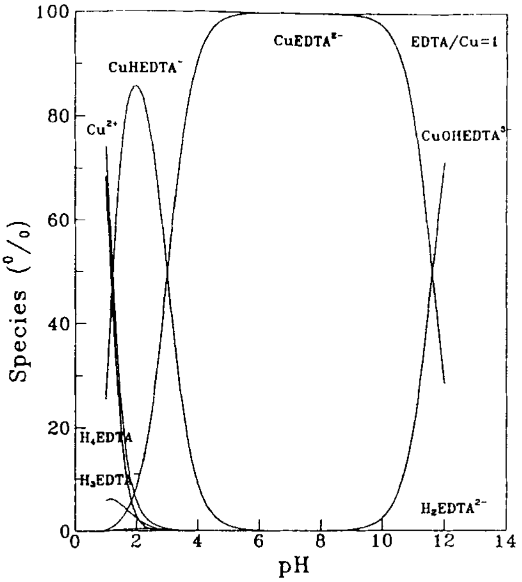
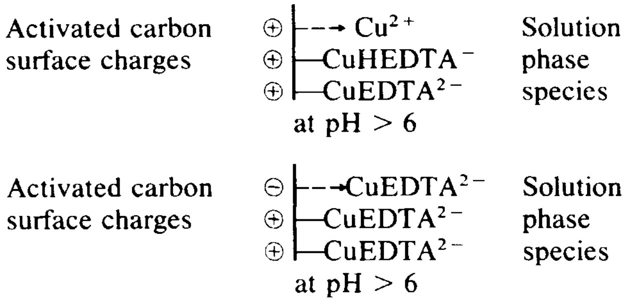
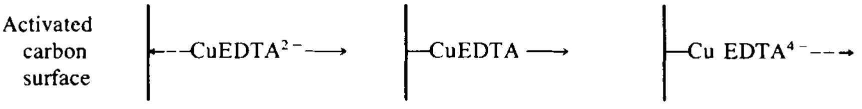


FIG. 2 Species distribution of Cd–EDTA as a function of pH.

neutral surface groups and thus reduce the extent of adsorption of chelated copper at higher pH values, as shown in Fig. 1. This electrostatic interaction is as follows.



A similar pH effect on the adsorption of ligand chelated copper was also obtained for the Cd–EDTA–activated carbon system by Jevititch and Bhattatcharyya (5) and for the Cu–NTA–Al_2O_3 system by Elliott and Huang (2). Figure 1 also indicates that the extent of adsorption of EDTA was lower than that of copper for EDTA/Cu = 1 systems for pH larger than 6. This is because the EDTA is dissociated from the adsorbed chelated copper species, $CuEDTA^{2-}$, due to the competition for the coordination sites of copper ion between the functional groups on the negatively-charged activated carbon surface and EDTA. The reaction steps are as follows.



$CuEDTA^{2-}$ attracted to the surface of activated carbon: Step 1

$CuEDTA^{2-}$ adsorbed on the surface of activated carbon: Step 2

EDTA dissociated from the adsorbed CuEDTA species: Step 3

For pH less than 2, the extent of adsorption of EDTA was higher than that of copper for the EDTA/Cu = 1 system. Thus it can be seen that the positively-charged activated carbon surface exhibits a higher affinity for the negatively-charged $CuHEDTA^-$ and H_3EDTA^- than the free positively-charged Cu^{2+} species.

Several equilibrium experiments were performed at various pH values to establish the adsorption isotherm. The Langmuir and Freundlich equations were both found to adequately describe the experimental equilibrium data of EDTA–chelated copper adsorbed by activated carbon as summarized in Tables 2A and 2B. From the Langmuir and Freundlich constants it was observed that the adsorption capacity of chelated copper at pH 3.10 and 4.50 was higher than that of other pH values, and the adsorption capacity of EDTA was higher than of Cu at lower pH values. The preferable electrostatic interaction between the positively-charged surface of activated carbon and negatively-charged EDTA species $CuHEDTA^-$ and H_3EDTA^- were dominant.

TABLE 2A
Langmuir Constants of Copper and EDTA at Different pH Values for Cu–EDTA Systems[a]

	Cu			EDTA		
pH	*Q* (mmol/g)	*b* (L/mmol)	*r*	*Q* (mmol/g)	*b* (L/mmol)	*r*
1.00 ± 0.01	—	—	—	-6×10^{-3}	5.02	0.95
2.00 ± 0.02	—	—	—	-47×10^{-3}	2.22	0.97
3.10 ± 0.05	45×10^{-3}	29.85	0.996	17×10^{-3}	18.14	0.81
4.50 ± 0.10	27×10^{-3}	38.01	0.999	50×10^{-3}	5.41	0.83
6.00 ± 0.05	20×10^{-3}	65.80	0.990	27×10^{-3}	14.06	0.95
7.50 ± 0.10	21×10^{-3}	27.13	0.995	12×10^{-3}	98.02	0.87
8.20 ± 0.05	18×10^{-3}	19.88	0.990	12×10^{-3}	44.52	0.94

[a] *r* = correlation coefficient. A dash indicates the adsorption quantity was too low for the parameter to be calculated.

TABLE 2B
Freundlich Constants of Copper and EDTA at Different pH Values for Cu–EDTA Systems[a]

	Cu			EDTA		
pH	*K* (L/g)	1/*n*	*r*	*K* (L/g)	1/*n*	*r*
1.00 ± 0.01	—	—	—	2.67×10^{-3}	1.72	0.93
2.00 ± 0.02	8.32×10^{-4}	1.60	0.86	0.04	1.30	0.89
3.10 ± 0.05	0.22	0.80	1.00	0.24	0.92	0.80
4.50 ± 0.10	1.24	0.68	0.99	0.21	1.05	0.86
6.00 ± 0.05	1.60	0.54	0.99	0.48	0.81	0.98
7.50 ± 0.10	0.70	0.74	0.97	1.31	0.52	0.96
8.20 ± 0.05	0.51	0.72	0.94	0.91	0.55	0.96

[a] *r* = correlation coefficient. A dash indicates the adsorption quantity was too low for the parameter to be calculated.

The kinetic experiments were conducted to study the temporal adsorption behavior, and the results shown in Fig. 3 indicating that the equilibrium was approached in the early stages of adsorption, around 40–50 minutes of contact time. In these experiments the stirring rate was high enough so that the boundary layer resistance was negligible. Hence, an intraparticle diffusion controlled model was used to investigate the solution pH effect on the diffusion rate of adsorption behavior. The semi-infinite linear diffusion model (11) was modified from the Cottrell equation (12) and can be used to calculate the diffusion-limited current, $i_d(t)$, for a planar microelectrode:

$$i(t) = i_d(t) = \frac{ZFAD^{1/2}C_0}{\pi^{1/2}t^{1/2}} \tag{1}$$

The rate of adsorbate, $dn/dt = i(t)/ZF$, diffusing to the surface is considered to be proportional to the bulk concentration of adsorbate, C:

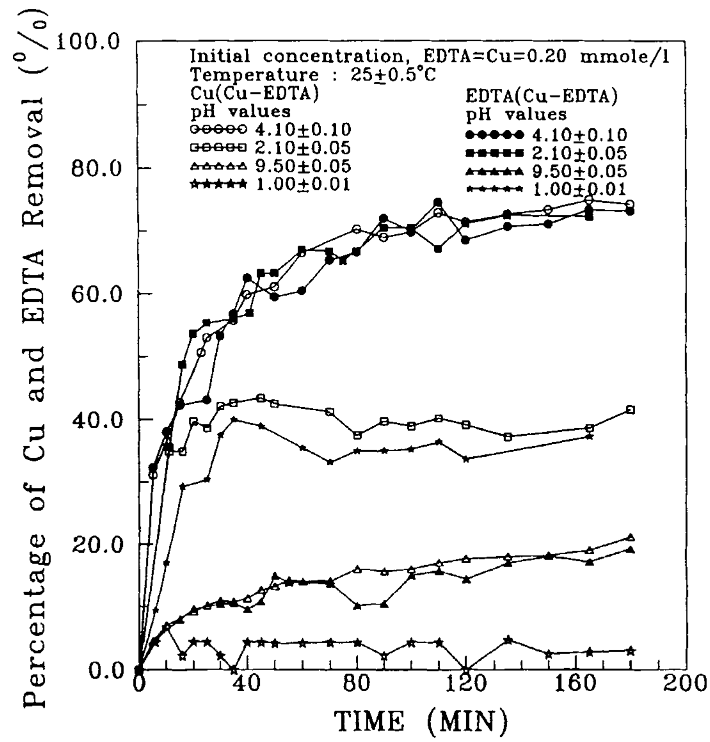


FIG. 3 Effect of pH on the kinetic removal of copper by activated carbon.

$$\frac{dn}{dt} = \frac{AD^{1/2}C}{\pi^{1/2}t^{1/2}} \tag{2}$$

It is assumed that the concentration remains constant ($C = C_0$), and that transport by diffusion is rate controlling. But the concentrations of adsorbates were obvious changing during the experiments, and thus Eq. (2) was modified:

$$\frac{dC}{dt} = \frac{AD^{1/2}C}{V\pi^{1/2}t^{1/2}} \tag{3}$$

where dC/dt is the changing rate of solution concentration and is equal to $(1/V)(dn/dt)$; V is the volume of solution. On integration, Eq. (3) gives

$$\ln(C_0/C) = \left(\frac{A}{V\pi^{1/2}}\right)D^{1/2}t^{1/2} \tag{4}$$

The slope of the linear plot of $\ln(C_0/C)$ versus $t^{1/2}$ is defined as the rate parameter, $k_a[= (A/V\pi^{1/2})D^{1/2}]$, which was calculated for various pH values as shown in Table 3. It indicates that the adsorption rate of chelated copper at pH 4.10 was higher than that at other pH values, and also shows that the adsorption capacity of EDTA was higher than of copper in lower pH ranges. Hence, based on these equilibrium and kinetic constants, it was concluded that the optimum pH range for the adsorption of chelated copper and EDTA by activated carbon is about 2 to 5.

The Effect of Ligand-to-Metal Ratio

Figure 4 shows the effect of the amounts of EDTA and copper on the adsorption of Cu as a function of pH. Comparing the experimental results for EDTA/Cu = 1 and EDTA/Cu = 3, an increase of excess EDTA concentration results in a decrease of copper adsorption. Conceptually, this can be visualized as a competition for surface sites between the

TABLE 3
Rate Parameter of Cu and EDTA at Different pH Values for Cu–EDTA Systems

	Cu		EDTA	
pH	Rate parameter	r	Rate parameter	r
9.50 ± 0.05	0.020	0.99	0.001	0.85
4.10 ± 0.10	0.139	0.99	0.122	0.99
2.50 ± 0.05	0.044	0.97	0.131	0.97
1.00 ± 0.01	0.004	0.16	0.052	0.94

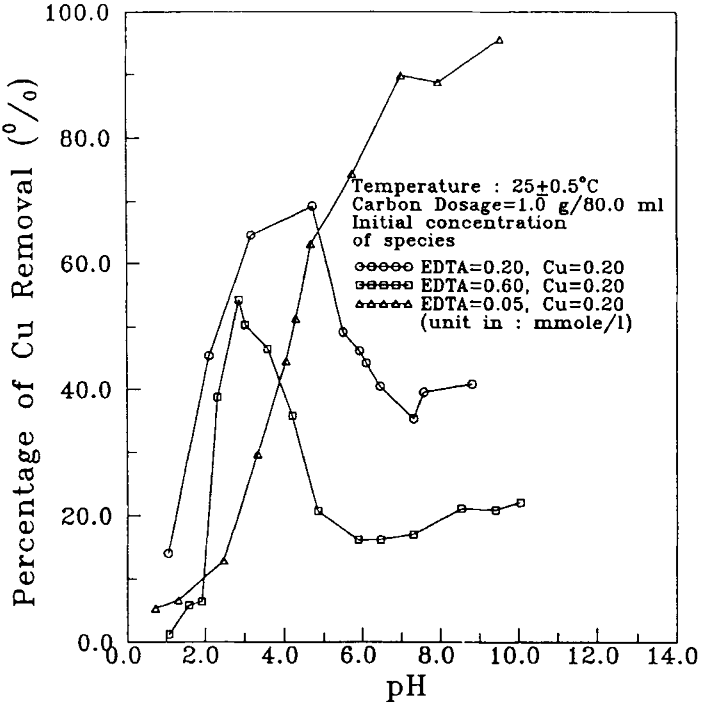


FIG. 4 Effect of EDTA/Cu molar ratio on the removal of copper as a function of pH.

EDTA-chelated copper species and the free EDTA species. Similar findings were reported by Elliott and Huang (2) for the Cu-NTA-Al_2O_3 system.

For EDTA/Cu < 1, when the concentration of EDTA is less than that of copper, the removal of copper from solution by the formation of hydroxide precipitates at higher pH conditions must be considered. However, for the experimental results from precipitation-free situations (pH < 4), a predominant portion of the copper in solution is supposed to exist as the positively-charged free copper species Cu^{2+}, which exhibits a lower affinity for the positively-charged surface of activated carbon compared to the negatively-charged EDTA-chelated copper species as shown in Fig. 4. Thus, most of the copper removed was in the form of CuEDTA chelates, the free copper species remaining in solution. Therefore, the greater the EDTA concentration, the greater the fraction of copper ion present in the chelated form which prefers to be adsorbed by activated carbon for EDTA/Cu ≦ 1. But when the copper in solution exists totally as the chelated form ($CuHEDTA^-$ and $CuEDTA^{2-}$), additional EDTA introduced to the solution occupy the surface adsorption sites and reduce the extent of adsorption of chelated copper.

The adsorption of copper in the presence of various amounts of EDTA was studied, and the results are presented in Fig. 5. It was also shown that the removal efficiencies of copper decrease with increasing EDTA dosage when EDTA is in excess, and this decreasing tendency of copper removal decreases at higher carbon dosages. The experiments with a fixed initial EDTA dosage indicated that the adsorption capacity for copper decreased with increasing carbon dosage (Fig. 6), possibly because the high dosage of activated carbon offered sufficient surface sites for the various adsorbates, free copper ions, free EDTA species, and chelated copper species, and thus there was less competition among these species.

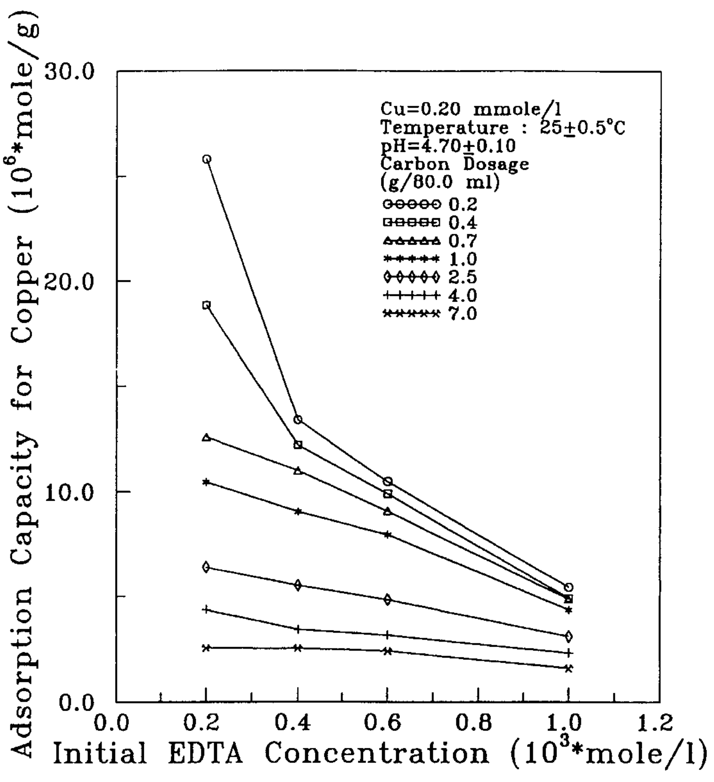


FIG. 5 Effect of initial EDTA concentration on the adsorption capacity for copper at various carbon dosages.

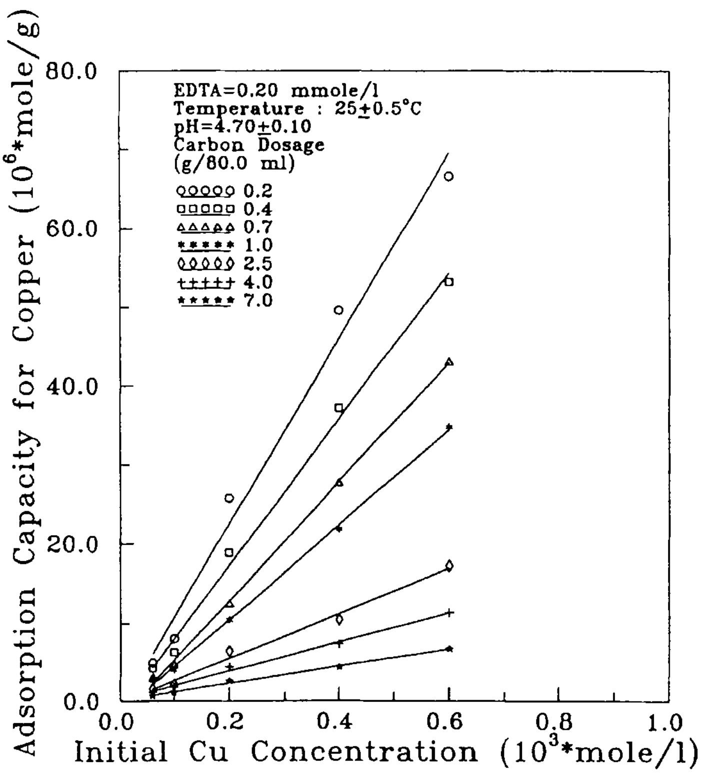


FIG. 6 Effect of initial copper concentration on the adsorption capacity for copper at various carbon dosages.

A Langmuir-type correlation for the relationship between the rate of copper removal and the carbon dosage was obtained as shown in Fig. 7. In order to explain this result, a 1:1 stoichiometry reversible reaction is proposed:

$$C + S \rightleftharpoons CS$$

where C, S, and CS are the concentrations of adsorbates in solution, vacant surface sites, and adsorbates on surface sites, respectively. The adsorption equilibrium constant can be expressed as

$$K_{\text{ads}} = \frac{CS}{C \times S} = \frac{CS}{(C_0 - CS) \times S} \tag{5}$$

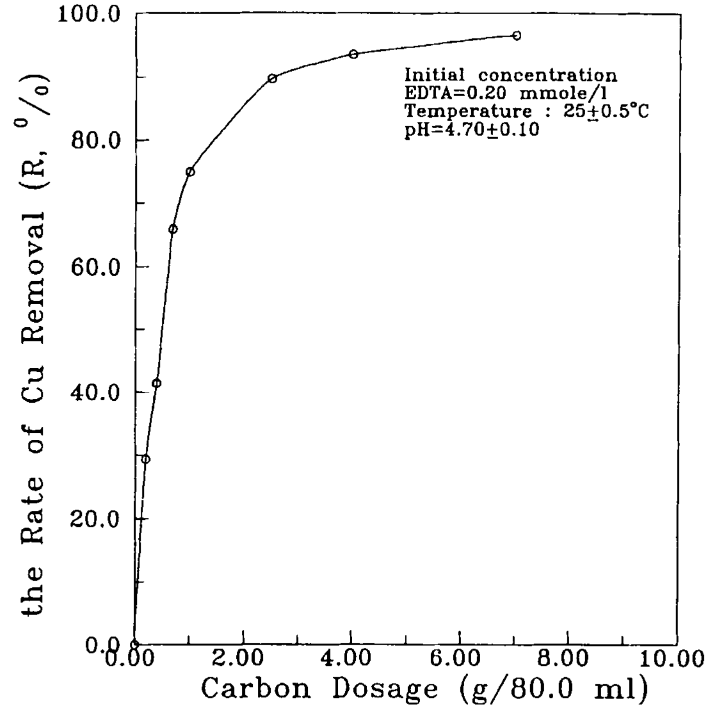


FIG. 7 Effect of carbon dosage on the rate of copper removal.

The rate of copper removal, R, is defined as

$$R = \frac{CS}{C_0} \tag{6}$$

which can be determined by the slope of a linear line for each carbon dosage in Fig. 6. Equation (5) can be rearranged as

$$\frac{R}{1 - R} = K_{\text{ads}} \times S$$

or

$$R = \frac{K_{\text{ads}} \times S}{1 + K_{\text{ads}} \times S} \tag{7}$$

which is a modified Langmuir adsorption isotherm. K_{ads} was determined to be about 0.5.

The results for the study of desorption of Cu–EDTA chelates on activated carbon are shown in Tables 4A and 4B. Investigation of the desorption quantity of copper and EDTA showed that more Cu^{2+} species were desorbed than EDTA species with 0.2 N $HClO_4$, and more EDTA species were desorbed than Cu^{2+} species with 0.2 N NaOH. This phenomena can be explained by the competition existing between the positively-charged copper ion and hydrogen ion for functional groups of the adsorbed EDTA at high acidic conditions. The following reaction scheme is suggested.

Main reaction: $\vdash$EDTA–Cu + $HClO_4 \rightarrow$ $\vdash$EDTA + Cu^{2+}

Side reaction: $\vdash$Cu–EDTA + $HClO_4 \rightarrow$ $\vdash$H + Cu–$EDTA^-$

At alkaline conditions, the OH^- ions can also significantly compete with negatively-charged EDTA species for the coordination sites of the copper species. The following reaction scheme is suggested.

TABLE 4A
Desorption Results of Activated Carbons Adsorbing Cu at Two Different Equilibrium Adsorption Condition

		Amount of desorbed species (mM/g)							
		Cu							
		Desorption sequence A,[a] 0.2 N, $HClO_4 \rightarrow$ 0.2 N, NaOH				Desorption sequence B,[b] 0.2 N, NaOH $\rightarrow$ 0.2 N, $HClO_4$			
		1st		2nd		1st		2nd	
Carbon dosage (g/80 mL)		$\times 10^3$	%	$\times 10^3$	%	$\times 10^3$	%	$\times 10^3$	%
Carbon A[c]	0.7	7.55	60.51	1.57	1.30	1.48	11.94	6.50	52.42
	1.0	7.24	58.86	1.10	0.87	0.92	7.45	7.03	56.69
	2.5	7.24	59.06	6.30	0.51	1.57	12.66	5.40	43.55
Carbon B[d]	0.7	5.96	74.90	0.15	1.94	0.94	11.75	4.48	56.00
	1.0	5.92	73.04	0.10	1.23	0.60	7.50	4.76	59.50
	2.5	5.79	72.73	0.05	0.57	0.47	5.93	4.07	51.07

[a] Sequence A for using desorbing solvent is adding 0.2 N $HClO_4$, after filtration solution and drying activated carbon, followed by adding 0.2 N NaOH.

[b] Sequence B for using desorbing solvent is adding 0.2 N NaOH, after filtration solution and drying activated carbon, followed by adding 0.2 N $HClO_4$.

[c] Adsorption condition of activated carbon: pH 2.50 ± 0.10, Cu adsorbed = 12.40×10^{-3}, EDTA adsorbed = 9.28×10^{-3} (mmol/g).

[d] Adsorption condition of activated carbon: pH 9.40 ± 0.10, Cu adsorbed = 8.00×10^{-3}, EDTA adsorbed = 3.50×10^{-3} (mmol/g).

TABLE 4B
Desorption Results of Activated Carbons Adsorbing EDTA at Two Different Equilibrium Adsorption Condition

Carbon dosage (g/80 mL)		Amount of desorbed species, mM/g							
		EDTA							
		Desorption sequence A,[a] 0.2 N, $HClO_4 \rightarrow$ 0.2 N, NaOH				Desorption sequence B,[b] 0.2 N, NaOH $\rightarrow$ 0.2 N, $HClO_4$			
		1st		2nd		1st		2nd	
		$\times 10^3$	%	$\times 10^3$	%	$\times 10^3$	%	$\times 10^3$	%
Carbon A[c]	0.7	3.42	36.50	—[e]	—	2.28	24.31	2.06	21.96
	1.0	2.80	29.84	—	—	1.55	16.52	1.03	10.98
	2.5	2.13	22.65	—	—	2.86	30.46	—	—
Carbon B[d]	0.7	2.36	67.20	0.28	8.10	2.14	61.19	—	—
	1.0	2.16	61.50	0.05	1.40	1.91	54.18	—	—
	2.5	1.58	44.90	0.27	7.60	1.83	52.26	—	—

[a-d] See Table 4A footnotes.
[e] Desorption quantity is negligible.

$$\text{Main reaction:} \quad \vdash\text{Cu-EDTA} + OH^- \rightarrow \quad \vdash\text{Cu-OH} + \text{EDTA}^-$$

$$\text{Side reaction:} \quad \vdash\text{EDTA-Cu} + OH^- \rightarrow \quad \vdash\text{OH} + \text{Cu-EDTA}^-$$

Based on the above explanation and the fact that neither $HClO_4$ nor NaOH can simultaneously desorb the same quantity of copper and EDTA, it can be further suggested that two possible conformations

$$\vdash\text{Cu-EDTA} \quad \text{or} \quad \vdash\text{EDTA-Cu}$$

of adsorbed complexes may exist simultaneously.

CONCLUSION

The adsorption of chelated copper on activated carbon varies over the entire pH range, possibly because the electrostatic interactions between the activated carbon surface and the dominant charged species is highly pH dependent. For pH > pH_{zpc}, the surface charge of activated carbon is assumed to be negative, and adsorption is usually favorable for positively-charged species present in the solution. But at pH < pH_{zpc}, the activated carbon surface is positively charged, which favors the adsorption of negatively-charged species from the solution. Thus, for the adsorption of a chelated metal, the optimum pH would be dependent on the surface charge characteristics of activated carbon and the species distribution of metal and ligand. Chelated copper species exhibit better adsorption characteristics than free copper species and enhance copper removal by activated carbon, but the presence of excess chelating agents may cause competition for the adsorption sites with the chelated copper species, thus decreasing the removal of copper.

Based on the adsorption and desorption characteristics of both copper and EDTA species, it is presumed that competition for adsorption exists between copper and hydrogen ions in acidic solutions, and exists between OH^- ions and EDTA species in alkaline solutions. Chelated metal ions can be adsorbed on an activated carbon surface with either the metal or the ligand end bonding directly to the surface.

NOMENCLATURE

A	the surface area of adsorbent
b	Langmuir constant (l/mM)
C	the concentration of adsorbates
C_0	the initial concentration of Cu–EDTA complex (mM)
CS	adsorbates on surface sites
D	diffusion coefficient (cm^2/min)
dn/dt	the number of moles of adsorbate diffusing to the surface of adsorbent per minute
F	Faraday's constant
$i(t)$	the diffusion-limited current
k	Freundlich constant (l/g)
k_a	rate parameter $(\text{minute})^{1/2}$
K_{ads}	the adsorption equilibrium constant
$1/n$	Freundlich constant
Q	Langmuir constant (mM/g)
R	the rate of copper removal
S	vacant surface sites
t	the experimental time (minutes)
V	the volume of solution
Z	charges of adsorbate

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