

Kinetics of copper desorption from soils as affected by different organic ligands

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ABSTRACT: Desorption of Cu and low molecular weight dissolved organics are the primary factors that impact fate and transport of Cu in soils. To improve predictions of the toxicity and threat from Cu contaminated soil, it is critical that time-dependent desorption behavior be understood. In this paper, the effect of organic ligands citrate, malate, and succinate on the kinetics of Cu desorption from contaminated soils varying widely in soil characteristics was investigated at 25° C and the soils used were referred to as clay, calcareous and sandy soils. The amount of Cu released by the used organic ligands varied greatly with physicochemical properties of the soils. The rate of Cu release by different extractants was in the order citric > malic > succinic, which was consistent with the stability constants of Cu complexes with these ligands. The modified Freundlich and the Elovich and Parabolic diffusion models were used to describe desorption of Cu²⁺ from the three studied soils as affected by the organic ligands. All of the models fit the data well with correlation coefficients ranging from 0.83 to 1.00 (P < 0.01). Each Model has a set of assumptions for the different physical and chemical properties of the systems to which they are being applied. The uses of these equations yield different magnitudes for the calculated variable, but the relationships between the soil + organic ligands and their effect (i.e., increase or decrease) on these variables are the same. Such information is critical, since Cu is used in a variety of industrial and manufacturing processes and is one of the most common contaminants found at hazardous waste sites.

Key words: Copper, kinetics, desorption, organic ligands

INTRODUCTION

Copper enters agricultural ecosystems through applications of Cu-containing fungicides, stable manures, and liquid or solid wastes from Cu-related mining and manufacturing. The accumulated Cu concentrations in agricultural soils have reached 110 to 1500 mg/kg, compared with 20 to 30 mg/kg background levels (Baker, 1990). Because copper is considered as a hazardous metal and is included in the EPA's list of priority pollutants, the USEPA and the European union have established maximum copper limits for soil and for biosolids to be applied to fields. The current soil cleanup criteria for Cu in the USA is 600 mg/kg (USEPA, 1999), and the upper limit of total soil Cu set by the European Union for receiving Cu-containing sewage sludge is 140 mg/kg (Ministry of Agriculture, Fisheries and Food, 1993). Fate and bioavailability of metals in soils are controlled by three main processes: (i) removal of metals from the soil solution by sorption onto soil particles, (ii) release of the metal from the soil particle to the soil solution

(desorption), and (iii) precipitation–dissolution of the metal as an independent phase in the soil matrix (Sposito, 1989; Sparks, 2003). However, researchers often rely on maximum adsorption levels and neglect desorption behavior to predict the fate and transport of contaminants such as Cu in the environment. This is unfortunate since once a soil is contaminated, desorption is an important process that controls the bioavailability of the contaminant. Thus, in order to improve remediation strategies, risk assessments, and make better predictions about the mobility of contaminants, it is critical that time-dependent metal desorption behavior on soil be understood. Low-molecular-weight organic acids originated from decomposition of soil organic matter in the upper soil horizons play an important role in the fate of metals (Glover, *et al.*, 2002; Collins, *et al.*, 2003). The anions of Low-molecular-weight organic acids can form soluble complexes with metal cations (Stevenson and Fitch, 1981), and modify the fixation of metals by soil organic matter, oxides, clays, and enhance their release (Chen, *et al.*, 2003). There were several reports on the effect of

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organic ligands on adsorption of metals (Wu, *et al.*, 2003), however, very little information about the effect of organic ligands on desorption of copper are available. In this study, therefore, we have investigated the effect of three different organic ligands on copper desorption behavior of selected Egyptian soils varying widely in chemical characteristics. Information obtained from this study will give insights to scientists and engineers that may lead to improved remediation strategies, disposal practices, and risk assessments.

MATERIALS AND METHODS

Soils

Samples of three surface soils (0-30 cm) which differed in location, origins, texture and calcium carbonate content were used. One was a clay alluvial soil (Torrifluvents), the second was a calcareous soil (Calciorthids) and the third was a sandy loam desert soil (Quartzipasmment). Hereafter, the soils will be

referred to as clay, calcareous and sandy soils respectively. The soils were air-dried, passed through a 2- mm sieve and stored in plastic bags before use. The pH values of the soils were measured in a 1:2 soil-water, conductivity (EC) was measured in the soil paste extracts. The organic matter content was determined by the method of Walkley and Black (Nelson and Sommers, 1982) and the cation exchange capacity by the method of Thomas (1982). Particle size analysis was determined by the hydrometer method (Black, 1965). Selected chemical and physical properties of the three soils studied are shown in Table 1.

Organic acids

Organic acids that occur naturally in soils and differ in number of functional groups were selected for the study. The selected organic acids are citric, malic and succinic. Table 2 lists some of the characteristics of the organic acids.

Table1: Selected chemical and physical characteristics of the three soils

Type of soil	pH	E.C dS/m	Clay	Silt	Sand kg/g	Total Carbonate	Organic matter	CEC cmol (p ⁺)/kg
Clay	8.40	0.75	446.00	140.00	419.00	30.00	14.00	33.00
Calcareous	7.90	4.54	268.00	64.00	449.00	260.00	2.10	18.00
Sandy	8.00	1.12	194.00	13.00	793.00	190.00	7.90	11.00

Table 2: Relevant properties of low-molecular- weight organic acids used in the study

Organic acids	Chemical formula	Molecular weight (g)	Ligand form	pk _a [*]
Malic	HO ₂ CCH ₂ CH(OH)CO ₂ H	134.09	H ₂ L	3.46,5.1
Succinic	HOOCCH ₂ CH ₂ COOH	118.09	H ₂ L	4.16,5.61
Citric	C(OH)(COOH)(CH ₂ COOH) ₂	192.13	H ₃ L	3.13,4.78, 6.43

* pk_a values were adopted from Serjeant and Dempsey (1979)

Table3: Kinetic modeling parameters and correlation coefficients for Cu desorption from the three studied soils as affected by different organic ligands at 298 K

Organic Ligands	Elovich			Parabolic diffusion			Modified Freundlich		
	a [mmol Cu/kg]	α [mmol Cu/ kg/min]	R ² *	K _d [mmol Cu/kg]	a [mmol Cu/ kg/min]	R ² *	1/m	K _d *10 ³ min	R ² *
Sandy soil									
Malic	1.15	2.22	0.950	2.51	0.19	0.833	0.22	1.53	0.946
Citric	1.35	13.64	0.975	3.99	0.16	0.891	0.15	2.93	0.974
Succinic	1.04	0.95	0.914	1.99	0.19	0.865	0.33	0.20	0.824
Calcareous soil									
Malic	1.16	1.98	0.966	2.40	0.19	0.830	0.23	1.33	0.939
Citric	1.53	37.73	0.964	3.92	0.14	0.847	0.13	3.27	0.923
Succinic	1.35	0.79	0.955	1.33	0.17	0.915	0.29	0.53	0.909
Clay soil									
Malic	1.10	1.47	0.925	2.47	0.14	0.865	0.22	1.13	0.798
Citric	1.73	45.38	0.956	3.66	0.12	0.811	0.13	3.07	0.909
Succinic	1.00	1.00	1.000	1.82	0.15	0.834	0.26	0.27	0.862

All coefficient of determinations (R²) are significant at the p = 0.01 level

Kinetic experiments (Desorption)

Sub-samples of the soils were treated with 150 ug/g Cu as CuSO₄. After adding Cu, the soils were incubated at field capacity for 6 wks, during which the soils were subjected to four wet-dry cycles. After 6 weeks the soils were air dried, crushed to pass through a 2mm sieve and stored in polyethylene bag for chemical analysis. Kinetic batch experiments were carried out by adding 10 g of contaminated soil samples to 40 mL of organic acid (3mM) in a reaction vessel unit. The mixtures were stirred and an aliquot of about 10 ml was removed and filtered at selected time intervals [5, 10, 20, 40, 60, 120, 240, 480 minute]. The filtrate was analyzed for Cu using atomic absorption spectrometry (AAS) and a number of different kinetic models were applied to describe the desorption. The kinetic experiments were carried out at 25 °C temperatures. The samples were centrifuged for 15 minutes equilibration filtered, and pH was measured. Copper in the extract was determined using atomic absorption spectrometry (AAS).

Kinetic modeling

Three Kinetic Models were applied to the desorption data to better understand the effects of organic ligands and time on the desorption process. The three equations are as follows.

Elovich Equation (Chien and Clayton, 1980, Elkhatib and Hern, 1988)

$$q = (1/\alpha) \ln(a\alpha) + (1/\alpha) \ln t \quad (1)$$

Where:

q = the amount of adsorbate released in time t;

a = a constant; and

α = a constant, related to the initial velocity of the reaction.

Thus a plot of q versus $\ln t$ should give a linear relationship with the slope of $(1/\alpha)$ and intercept of $(1/\alpha) \ln(a\alpha)$.

Parabolic diffusion equation (Laidler, 1965)

This equation may be written:

$$q = a + k_d t^{1/2} \quad (2)$$

Where:

q = the amount of Cu desorbed in time t.

a = a constant.

k_d = apparent diffusion rate coefficient.

Thus, a plot of "q" $t^{1/2}$ should provide a linear relationship if the reaction conforms to parabolic diffusion law.

Modified Freundlich Equation

The modified Freundlich equation (Kuo and Lotse, 1973) used in the form:

$$q = k_d C_i t^{1/m} \quad (3)$$

Where:

q = desorbed Cu (mmol/kg)

C_i = initial Cu concentration (Mm)

t = reaction time (min)

k_d = desorption rate coefficient (min^{-1}), and

1/m = constant.

The parameter $k_d C_i$ and 1/m were calculated from the intercept and slope, respectively of the linear plots. These equations have been widely applied in desorption studies in soils and soil components, even though, the equations are empirical and yield only apparent rate parameters. Finding a meaningful model that can accurately describe these data is extremely difficult because of the heterogeneous nature of soil. Therefore, the use of these models in this study was only for qualitative comparison between soil and organic ligands combination.

Statistical analysis

All data were processed by Microsoft Excel (Microsoft, 2000), and the regression of linear and other statistical analyses were conducted using the programs of SAS Release 6.12 (SAS Institute, 1996).

RESULTS**Physicochemical properties of soils**

Table 1 displays physical and chemical properties of the studied soils. Organic matter content is approximately 14, 2.1 and 7.9 g/kg in clay, calcareous and sandy soils, respectively. The clay soil contains much less calcium carbonate percentage than the other two soils (calcareous soil samples had the highest). The clay soil samples display the highest CEC while sandy soil samples had the lowest (clay, 33; calcareous 18; and sandy 11 cmol (p^+)/kg).

Copper desorption vs time

The effect of time on desorption of Cu from the three studied soils by using different organic extractants is displayed in Fig. 1. A nearly linear relationship of Cu release versus time was observed in the soil samples studied for all the extractants (citric, malic, and succinic acids). Desorption of Cu was least in clay soil while the sandy soil exhibited the highest release, copper extractability from calcareous soil was intermediate

(Fig. 1). For the different soils, Cu desorption followed the release order: citric acid > malic acid > succinic acid. The sequence of amounts of Cu released by these ligands is generally in accord with the stability constants of Cu complexes with these ligands (Table 2). The percentage of Cu released from the surface of soil system is also consistent with the stability constants of the complexes of Cu with those ligands. Citrate ligands released 28 % of added Cu, about 23 % of Cu added was released by malic acid while 19 % was released by succinic acid (Table 2). The introduction of organic ligands to the desorption system may cause the change of the surface characteristics of soils, competitive distribution of metal ions between soil surface and organic ligands in the solution, and partly dissolution of minerals in soils. Overall, the release of Cu was greatly enhanced in the presence of citrate ligand under the reaction conditions we studied. To further assure the effectiveness of organic ligands on Cu release, the results were modeled using several kinetic equations.

Kinetics

The use of kinetic models was for qualitative comparison of data sets. Often, multiple models can fit the same set of kinetic data (Ogwada and Sparks, 1986); therefore, we have intended only to investigate the efficacy of these models in pointing out differences in Cu release trends. Table 3 gives the values of kinetic variables in the three equations applied to the experimental data.

Elovich equation

The empirical Elovich equation (Chien and Clayton, 1980) was used to describe the Cu desorption rate. Elovich equation plots of Cu desorbed vs. \ln of reaction time at 298 K for sandy, calcareous and clay soils as affected by citric, malic and succinic acid are shown in Fig. 2. A linear relationship exists between 'q' and ' $\ln t$ ' for each of the three organic acids used. Elovich equation parameters were determined from the slope and intercept of the linear plots. Parameters for the three soils and three organic acids are shown in Table (3). The " α " values listed in Table (3) for calcareous, sandy and clay soils were the highest in the presence of citric acid. In Elovich equation, a decrease in " α " indicates a reduction in the desorption reaction rate (Chien and Clayton, 1980) therefore the rate of Cu desorption from the soils studied increased as a result of using citric acid followed by malic acid and succinic

acid. Correlation coefficients relating Cu released to \ln reaction time are also given in table 3. In all cases R^2 values were significant at 0.01 level. Therefore, the Elovich equation appeared to be successful in describing the kinetics of Cu desorption from the studied soils as affected by the three organic ligands. The Elovich equation (Eq. (2)), fit all of the soil + organic ligands samples ($r^2 > 0.914$). The information provided by α suggested that the presence of citric acid will increase Cu desorption; however, these parameters are dependent on soil type and reaction conditions.

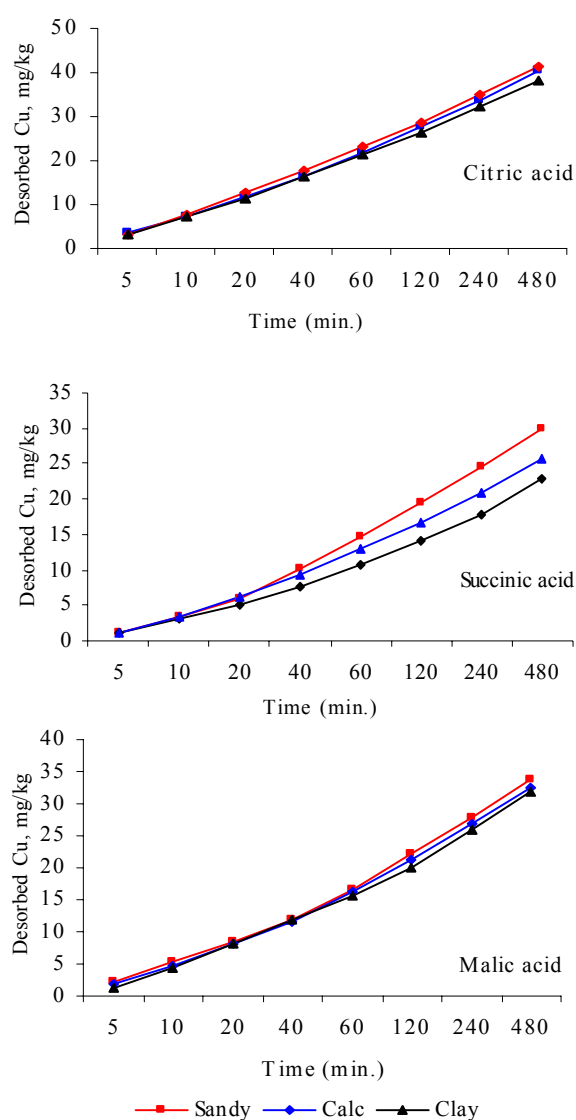


Fig. 1: Effect of different extractant and time on cumulative desorption of Cu from the three studied soils

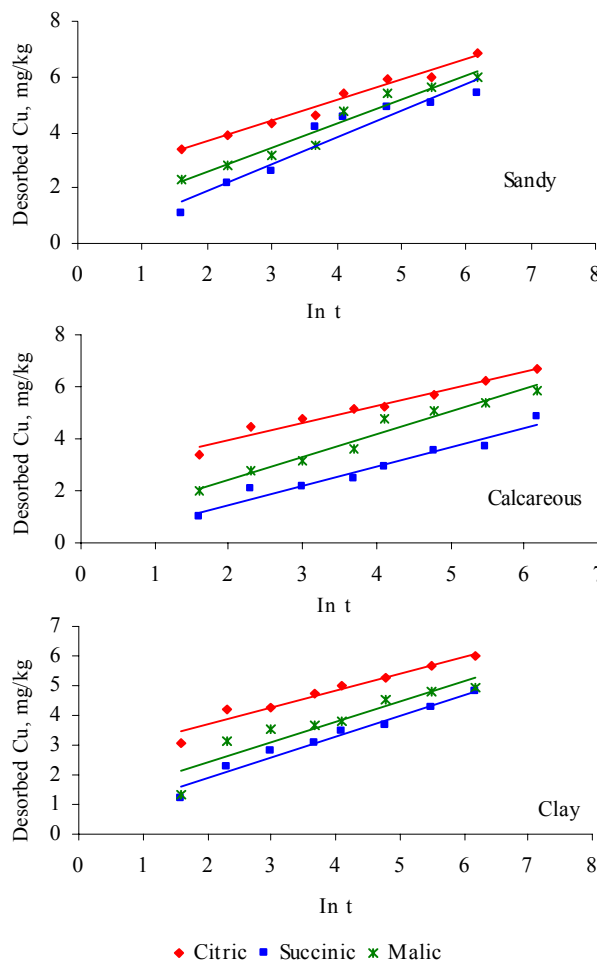


Fig. 2: Elovich equation plots for Cu desorption from the three studied soils as affected by different organic ligands

Parabolic diffusion equation (Laidler, 1965)

The Parabolic Diffusion Equation was also used to describe the kinetic data of Cu release. Parabolic Diffusion plots for Cu released from the soils as affected by three organic acids at 298 K are shown in Fig. 3. Linear relationships exist between Cu released and $t^{1/2}$ for the three organic acids studied. Parabolic Diffusion law parameters were determined from the slope and intercept of the linear plots. Parameters of the three soils as affected by citric, succinic and malic acid are shown in Table (3). The apparent Cu diffusion rate coefficient " k_d " in the parabolic diffusion law is considered a measure of the relative rate of Cu release. The " k_d " values were higher in the sandy soil than the values in the calcareous and clay soil. The " k_d " values followed the trend sandy soil > calcareous > clay. The

lower values in the clay soil are attributed to its higher organic matter, clay content and cation exchange capacity.

The " k_d " values were also higher in the presence of citric acid in all the soil samples studied than the corresponding values in the presence of malic and succinic acids. Correlation coefficients relating Cu released to square root of reaction times were significant at 0.01 level in most cases and they are given in Table 3. Therefore, based on coefficient of determination, the parabolic diffusion law was also successful in describing the kinetics of Cu released by the three studied soils as affected by three organic ligands. The parabolic diffusion model (Eq. 1) seemed to best fit the sandy systems; however, linear relationships, an indicator that desorption processes

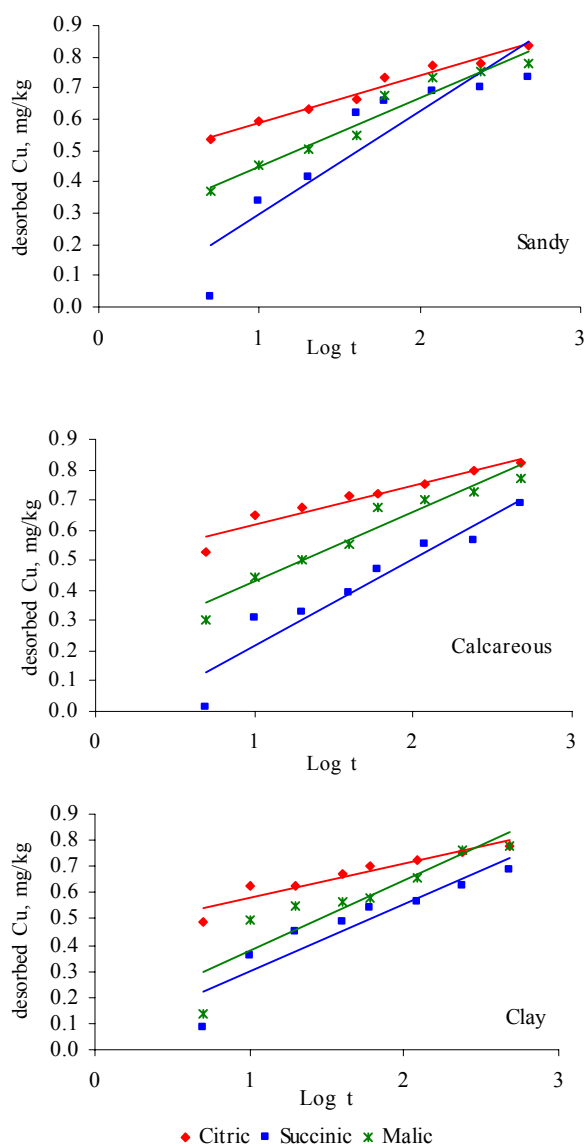


Fig. 3: Parabolic diffusion equation plots for Cu desorption from the three studied soils as affected by different organic ligands

are diffusion controlled (Toor and Bahl, 1999), are evident in the other two soils as well. The diffusion coefficient " k_d " is greater in the soils with a light texture (sandy) and lower organic matter content, as soils with more heterogeneity are more likely to have an increase in transport-limited processes. Calculated diffusion coefficients were higher for soil + citric acid, indicating that diffusion rates were faster. In the case of succinic acid, the diffusion coefficients were much lower; however, the model still yields a straight line. Such an

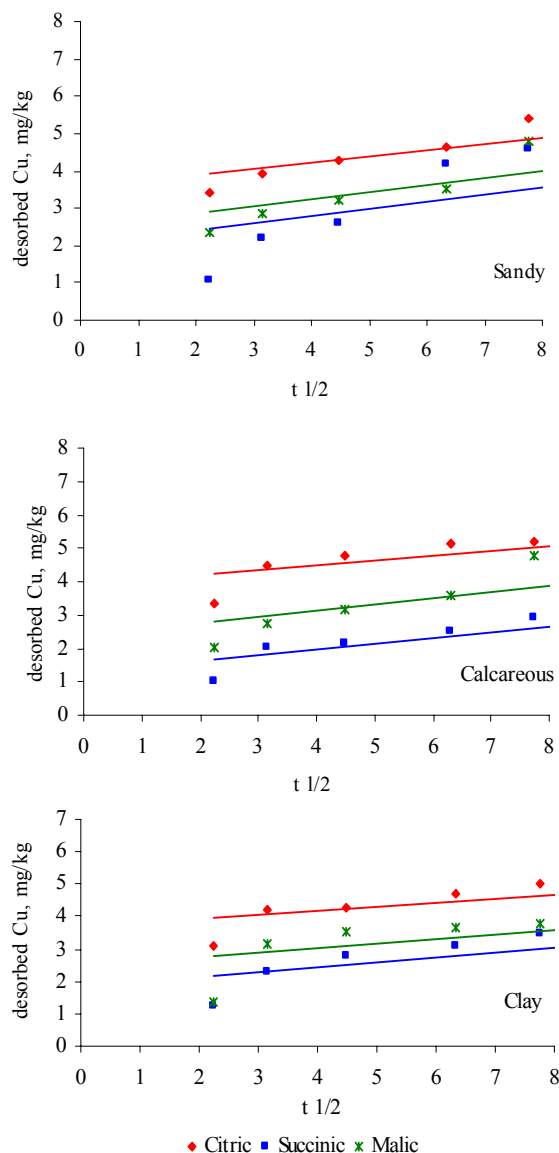


Fig. 4: Modified Freundlich equation plots for Cu desorption from the three studied soils as affected by different organic ligands

observation may suggest that diffusion influenced Cu desorption to a smaller extent than in the case of citric acid study, however, we cannot determine the type of diffusion that is taking place in either case.

Modified Freundlich equation

The Modified Freundlich Equation (Kuo and Lotse, 1973) was also used to describe the Cu desorption data. Modified Freundlich plots of log Cu desorbed vs. log of reaction time for the sandy soil, calcareous soil and

clay soil at 298 K as affected by the three organic ligands are shown in Fig. 4. Linear relationships exist between log Cu released and log t for the three soil studied with three organic acids. The modified Freundlich equation parameters " $k_d C_0$ " and " $1/m$ " were calculated from the intercept and the slope of the linear plots. The values of desorption rate coefficient " $k_d C_0$ " and " $1/m$ " for the soils are shown in Table (3). In general, the desorption rate coefficient " k_d " values listed in Table 3 as a result of citric acid addition were the highest in comparison with the succinic acid and malic acids in the same soil. Coefficient of determination relating log of Cu released to log of the reaction time are given in Table (3). In most cases, R^2 values were significant. Therefore, based on coefficient of determination, the modified Freundlich equation was also successful in describing the kinetics of Cu release from the studied soils.

DISCUSSION AND CONCLUSION

A detailed investigation has been made to study the effect of time on desorption of Cu from three different soils by using different organic extractants. The quantity of Cu released from soils followed the trend Sandy > Calcareous > Clay. The low Cu desorption associated with the clay soil suggests that most Cu ions are specifically chemically sorbed by both organic matter and layer silicate clays. Organic matter at low levels (Alcacio, *et al.*, 2001) has strong affinity for Cu. McBride (1994) proposed possible binding configurations of Cu^{2+} on complexes of oxide minerals and organic matter: (i) Cu^{2+} is bonded to the mineral surfaces only (inner-sphere complex); (ii) Cu^{2+} is bonded to the organic matter that is adsorbed by the oxides at high levels (Type B ternary complex); and (iii) Cu^{2+} acts as a bridge cation between the oxides and the organic matter that is adsorbed at low levels (Type A ternary complex). The higher soil CEC generally coexisted with higher content of clay and organic matter, and showed strong ability on the sequestration of metals (Stewart, *et al.*, 2003) and vice versa. The clay soil under study showed the highest values for organic matter, clay content and cation exchange capacity whereas sandy soils showed the lowest values of these properties (Table 1). For all the studied soils, Cu desorption followed the release order: citric acid > malic acid > succinic acid. The difference in the effect on Cu desorption by organic acids is related to their chemical structures. Among the organic ligands

used, ions of malic and citric acids carrying two or three $-COO$ groups can form chelates with 5- or 6-membered ring structure. These chelates are much more stable than succinate complexes. The greater stability of the citrate- and malate- Me^{z+} complexes led to the greater efficacy of citric and malic acids in desorbing Cu from soils than succinic acid. Besides, succinic acid with a smaller acid dissociation constant (pK_a value 4.16) provided fewer anions for complexing with Cu than the other two acids (pK_a values 3.13 and 3.46), and thus may show a smaller effect on metal desorption. In addition, major forms of citric acid at high pH changed from H_3L , H_2L^- , and HL^{2-} to L^{3-} . HL^{2-} and L^{3-} (L^{3-} , HL^{2-} and L^{3-} always showed greater chelating ability with heavy metals than H_3L and H_2L^-), leading to larger desorption amount of Cu, in the presence of HL^{2-} and L^{3-} than in the presence of H_3L and H_2L^- (Martell and Smith, 1977). Data presented here show that organic ligands are effective chelators of sorbed metals and these ligands may play an important role in the mobilization and phytoavailability of Cu in soils. The empirical Elovich, the Parabolic Diffusion and the Modified Freundlich models were used to describe the Cu desorption rate. Based on coefficient of determination, the three models were all successful in describing the kinetics of Cu release from the studied soils. All of the models fit the data well, and each has a set of assumptions for the different physical and chemical properties of the systems to which they are being applied. The uses of these equations yield different magnitudes for the calculated variable, but the relationships between the soil + organic and their effect (i.e., increase or decrease) on these variables are the same. Information obtained from this study will give insights to scientists and engineers that may lead to improved remediation strategies, disposal practices, and risk assessments.

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