Effect of Dissolved Organic Matter from Sludge and Sludge Compost on Soil Copper Sorption

L. X. Zhou and J. W. C. Wong*

ABSTRACT

Interaction of Cu with dissolved organic matter (DOM) is an important physicochemical process affecting Cu mobility in soils. The aim of this study was to investigate the effects of DOM from anaerobically digested dewatered sludge and sludge compost on the sorption of Cu on an acidic sandy loam and a calcareous clay loam. In the presence of DOM, Cu sorption capacity decreased markedly for both soils, especially for the calcareous soil. The Cu sorption isotherms could be well described by the Freundlich equation ($r^2 = 0.99$), and the binding intensity parameter of soils in the presence of sludge DOM was lower than compost DOM. An increase in DOM concentration significantly reduced the sorption of Cu by both soils. Within the Cu and DOM concentration range studied, the decrease in Cu sorption caused by sludge DOM was consistently greater than that of compost DOM. This might be attributed to the greater amount of hydrophobic fraction of DOM in the compost. Moreover, the reduction of Cu sorption caused by DOM was more obvious in the soil with higher pH. In addition, the sorption of Cu increased with an increase in pH for both soils without the addition of DOM, while Cu sorption in the presence of DOM was unexpectedly decreased with an increase in pH at a pH >6.8. This implied that DOM produced by sludge or other C-enriched organic wastes heavily applied on calcareous soils might facilitate the leaching loss of Cu because of the formation of soluble DOM–metal complexes.

Heavy metal contamination has received much attention with regard to plant uptake and contamination of ground water or surface waters because of sludge application (Cunningham et al., 1975; Riekerk and Zasoski, 1979). Evidence for metal translocation has been reported in numerous long-term sludge application experiments (Li and Shuman, 1996; Streck and Richter, 1997). Downward migration was observed 7 yr after sludge application where soluble Cu, Zn, and Cd were greater at the depth of 40 to 60 cm in the sludge-treated soil than in the untreated soil (Campbell and Beckett, 1988). Robertson et al. (1982) suggested that the decrease in soil pH following sludge application was the major reason for the higher mobility of heavy metals in sludge-treated soils. However, in some field experiments, leaching of Cu, Cd, and Pb in sludge-treated soils was reported even though soil pH was enhanced (Darmody et al., 1983; Williams, 1987).

The incorporation of C-rich sludge into soils has been shown to increase the amount of dissolved organic matter (DOM) in soils (Baham and Sposito, 1983; Lamy et al., 1993). Dissolved organic matter can facilitate metal transport in soil and ground water by acting as a *carrier* through formation of soluble metal–organic complexes (McCarthy and Zachara, 1989; Temminghoff et al., 1997). The drained ground water of a field plot receiving the highest application of sludge contained about twice the Cd concentration of the control plot during the first few weeks following sludge disposal (Lamy et al., 1993). Among most metals, Cu can form strong bonds with organic matter, which migrates into the subsoil without being stripped from the stable complex by soil sorption sites (del Castilho et al., 1993; Wu et al., 1999). Darmody et al. (1983) also noted that many metals were mobile in a silt loam receiving heavy sludge application, and Cu had greater downward movement than the other metals 3 yr after the initial application. Other authors have also reported that DOM from sewage sludge or animal manure was able to mobilize Cu sorbed on soil or sludge (Gerritse et al., 1982; McBride et al., 1997).

Composting is a common practice before sludge is applied to soil to eliminate pathogens and achieve biological transformation of the organic matter. Research has shown that the composting process alters greatly the composition and characterization of DOM of organic waste (Liang et al., 1996; Chefetz et al., 1998; Baziramakenga and Simard, 1998). Undoubtedly, soils amended with sludge and sludge compost display different physicochemical properties, especially in terms of DOM composition in soil, which will affect behavior of metals in soils.

Therefore, the objectives of this study were to quantitatively compare the effect of DOM derived from sludge and sludge compost on the sorption of Cu on a calcareous and an acid soil and to investigate the effect of DOM concentration and pH on Cu sorption by the two soils. This would provide information for assessing the potential of sludge and compost DOM in facilitating metal transport in sludge-treated soils.

MATERIALS AND METHODS

Soil, Sewage Sludge, and Compost Samples

A calcareous clay loam (Typic Ustochrepts) and an acidic sandy loam (Typic Acrorthox) were taken from the top 20 cm soil in Northern Suburbs, Beijing and New Territory, Hong Kong, respectively. Soil samples were air-dried, ground to pass through a 1-mm sieve, and stored in plastic bottles until used. Anaerobically digested dewatered sludge was collected from the Taipo wastewater treatment plant, Hong Kong, and stored at 4°C until used. The sludge compost was mature compost produced from our previous composting experiments using

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L.X. Zhou, College of Resources and Environmental Sciences, Nanjing Agricultural Univ., Nanjing 210095, P.R. China; J.W.C. Wong, Dep. of Biology, Hong Kong Baptist Univ., Kowloon Tong, Hong Kong SAR, P.R. China. Received 23 Dec. 1999. *Corresponding author (jwcwong@hkbu.edu.hk).

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Abbreviations: DOM, dissolved organic matter; DOC, dissolved organic carbon; FAAS, flame atomic absorption spectrometer; HiA, hydrophilic acid; HiB, hydrophilic base; HiN, hydrophilic neutral; HoA, hydrophobic acid; HoB, hydrophobic base; HoN, hydrophobic neutral.

Table 1. Selected physicochemical properties of soils, sewage sludge, and sludge compost.

Samples	pH	Organic C	Total N	Total P	Sand	Silt	Clay	$CaCO_3$	CEC†	Total Cu
		$g\ kg^{-1}$			%				$cmol_c\ kg^{-1}$	$mg\ kg^{-1}$
Acidic sandy loam	4.5	14.0	0.91	0.31	46	43	12	0	7.0	5.0
Calcareous clay loam	7.9	17.5	0.85	0.68	26	47	27	10.5	16	23.6
Sludge	7.8	484	65	19.4	–	–	–	–	–	329
Sludge compost	6.8	601	20	8.5	–	–	–	–	–	150

† Cation exchange capacity.

the above sludge (Wong et al., 1997). Selected physicochemical properties of the soils, sludge, and compost are given in Table 1. Soil texture was determined by using the pipette method (Gee and Bauder, 1996). Sample pH was quantified, using a suspension of 1:1 (solid to water) for soil and 1:5 for organic waste, with an Orion (Beverly, MA) 902A Ionanalyzer. Cation exchange capacity (CEC) of each soil sample was obtained by exchanging the samples with NH_4OAc at pH 7 (Sumner and Miller, 1996). Total organic carbon (OC) was determined by the Walkley and Black wet dichromate oxidation method (Nelson and Sommers, 1996). Total N was determined by the Kjeldahl digestion–distillation method (Bremner, 1996). Total P was determined by H_2SO_4–$HClO_4$ digestion and analyzed by the molybdenum blue color method (Kuo, 1996). The carbonate content was calculated from the amount of CO_2 released by reaction with HCl (Loeppert and Suarez, 1996). Total Cu was determined with a flame atomic absorption spectrometer (FAAS) (Varian [Mulgrave, VIC, Australia] Spectra AA-20) after digestion in concentrated HNO_3.

Extraction and Characterization of Dissolved Organic Matter

Sludge and sludge compost were extracted with double-distilled water using a solid to water ratio of 1:10 (w/v) on a dry weight basis on a reciprocal shaker at 200 rpm for 16 h at 20°C. After the suspension was centrifuged at 12 000 × *g* for 20 min, the supernatant was filtered through a 0.45-μm sterilized membrane (GN-6 Metrice; Gelman Sciences, Ann Arbor, MI). The filtrates were stored at 4°C and analyzed for pH (Orion 902A Ionanalyzer), total dissolved organic carbon (DOC) using a total organic carbon (TOC) autoanalyzer (TOC-5000A; Shimadzu, Kyoto, Japan) and Cu with a FAAS (Varian Spectra AA-20). The selected properties of the DOM are listed in Table 2.

Dissolved organic matter obtained from sludge and sludge compost was characterized by fractionation into hydrophilic and hydrophobic fractions using the method developed by Leenheer (1981), with slight modification. Dissolved organic matter solution was first pumped through the first glass column (Column I, 20 × 230 mm) packed with XAD-8 resin (Fluka, Buchs, Switzerland) by a peristaltic pump at a flow rate of 2 mL min^{-1}. Hydrophobic base (HoB) of DOM sorbed on the XAD-8 resin was then backflush-eluted with 0.25 bed volume of 0.1 *M* HCl, followed by 1.5 bed volume of 0.01 *M* HCl. The effluent from Column I was acidified to pH 2 with 6 *M* HCl and then passed through the second XAD-8 resin-packed column (Column II) followed by a 0.01 *M* HCl rinse. Hydrophobic acid (HoA) retained in Column II was desorbed by backflush elution with 0.1 *M* NaOH. The effluent through Column II was considered the hydrophilic fraction. The hydrophobic neutral fraction sorbed on the XAD-8 resin was calculated by subtracting the sum of HoB, HoA, and hydrophilic fraction from the total input DOC of the extract. The effluent that contained only the hydrophilic fraction from Column II was pumped through a third column (Column III) containing a strongly acidic cation exchange resin, Dowex-50wx8 resin (Fluka). Hydrophilic base (HiB) was desorbed by forward elution with 1.0 *M* NaOH from Column III. The effluents from Column III containing hydrophilic acid (HiA) and neutral (HiN) were then passed through a weakly basic, polyamine anion exchange resin, Biorad (Hercules, CA) AG 3x4 (Column IV). The HiA fraction retained in this resin was eluted with 1 *M* HNO_3. The HiN was obtained from the effluent of Column IV. Each fraction obtained was determined for DOC.

Copper Sorption Experiment

Preliminary experiments were performed to determine the time required to achieve equilibrium for Cu sorption by the soil samples, which could be reached after 1.5 h. Therefore, an equilibrium time of 2 h was chosen in the present study. A soil sample of 0.4 g was weighed into a 50-mL polyethylene centrifuge tube and shaken with 20 mL of 0.01 *M* KCl solution (background electrolyte for maintaining stable ionic strength) at 200 rpm for the sorption experiment. The KCl solution consisted of 0, 12.5, 25, 50, 100, 200, 400, 600, and 800 mg Cu L^{-1} as $CuCl_2 \cdot 5H_2O$ with or without the addition of 300 mg C L^{-1} of DOM derived from sludge and sludge compost. All treatments were done in triplicate. All suspensions were adjusted respectively to pH 4 for the acidic sandy loam and pH 7 for the calcareous clay loam with HCl or NaOH to minimize the error caused by pH fluctuation. The soil suspensions were shaken on a reciprocal shaker at 200 rpm for 2 h. The tubes were then centrifuged at 3500 × *g* and filtered through Whatman No. 2 filter paper. Copper concentration in the filtrates was determined with a FAAS. The quantity of Cu sorbed was calculated by subtracting the Cu in the equilibrium solution from total initial Cu. Another series of experiments was performed to evaluate the effect of DOM concentration on Cu sorption and desorption. A soil sample of 0.4 g was shaken with 20 mL of solution containing 40 mg Cu L^{-1} and different DOM concentrations of 0, 50, 100, 150, 200, 300 and 400 mg C L^{-1} (a concentration range found in soils receiving slight to heavy application of organic waste) at

Table 2. Parameters of the Freundlich equation for Cu sorption by soils in the presence and absence of 400 mg C L^{-1} of dissolved organic matter (DOM).

	Acidic sandy loam			Calcareous clay loam		
	No DOM	Compost DOM	Sludge DOM	No DOM	Compost DOM	Sludge DOM
k	52.1	32.1	10.7	2776	379	47.9
l/*n*	0.786	0.850	1.003	0.301	0.552	0.873
r^2†	0.976	0.991	0.995	0.942	0.958	0.930

† Significant at $p < 0.01$.

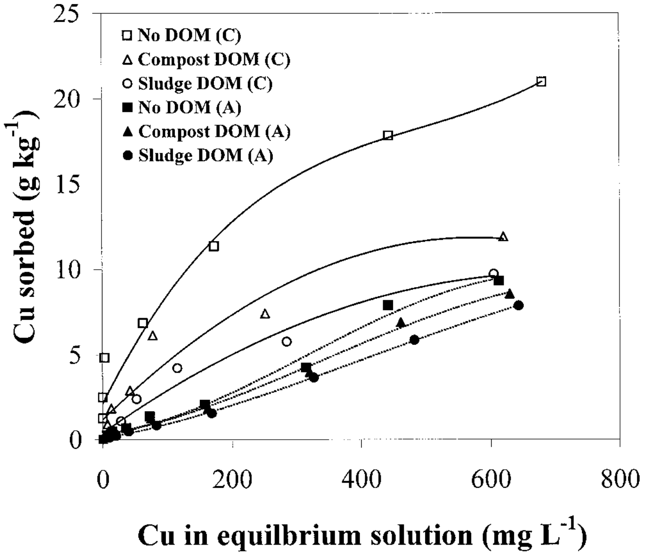


Fig. 1. Copper sorption isotherm of the acidic (A) and calcareous (C) soils with and without the addition of 400 mg C L^{-1} of the sludge or sludge compost dissolved organic matter (DOM).

a background ionic strength of 0.01 *M* KCl. The rest of the procedure, including pH adjustment and centrifugation, were the same as the above experiment. The residues obtained after sorption were washed three times using double-distilled water, then 20 mL of 1 *M* $MgCl_2$ (pH 7) was added into the tube and shaken for 2 h. After the suspension was centrifuged and filtered, Cu in the filtrate was determined to obtain desorption of Cu previously sorbed by soils.

The effect of pH on Cu sorption in the presence and absence of DOM was also performed in this study. A soil sample of 0.4 g was shaken with 20 mL of solution containing 0.01 *M* KCl and 40 mg L^{-1} of Cu in a centrifuge tube with or without the addition of 400 mg C L^{-1} DOM. The pH of all suspensions was adjusted to various levels ranging from pH 2 to 10. In this study, the concentration of DOM-born Cu was much lower than the Cu concentration added as $CuCl_2 \cdot 5H_2O$, so that the effect of DOM-born Cu on the Cu sorption experiment was negligible.

RESULTS AND DISCUSSION

Copper Sorption Isotherm

Figure 1 gives the equilibrium isotherms of Cu sorption of the two soils with or without the addition of DOM derived from sludge or sludge compost. The amount of Cu sorbed increased with an increase in equilibrium Cu concentrations and eventually attained a plateau value at high equilibrium Cu concentrations. The sorption capacity of the calcareous soil for Cu was much higher than that of the acidic sandy loam at the same equilibrium concentration of Cu, which might be because of the higher pH, clay mineral and carbonate contents, and higher CEC of the calcareous soil (Table 1). More variable negative charges and sorption sites were formed in the soil of higher pH and clay mineral contents, which favor Cu sorption. On the other hand, the higher pH and carbonate contents may also facilitate Cu precipitation as $Cu(OH)_2$ and/or $CaCO_3$ (Lindsay, 1979). In the presence of DOM, the Cu sorption capacity decreased markedly for both soils, but the effect on the calcareous soil was greater than that on the acidic sandy loam. Dissolved organic matter derived from sludge compost had a smaller effect in reducing the sorption capacity for Cu than that of sludge for both soil types.

The Cu sorption data were plotted according to the linear Freundlich equation:

$$\log (x/m) = \log K + 1/n \log C$$

where x/m is the amount of Cu sorbed (mg kg^{-1}), C is the equilibrium Cu concentration (mg L^{-1}), K is the equilibrium partition coefficient, and $1/n$ is the sorption intensity.

The Cu sorption isotherms conformed better to the Freundlich equation than to the Langmuir equation (data not shown) as indicated by the high values for the correlation coefficient of determination ($r^2 > 0.938$). Other studies also reported that the Freundlich equation described Cu sorption for soils better than the Langmuir equation (Atanassova and Okazaki, 1996). The Cu sorption behavior of the acidic sandy loam can be described better by the Freundlich model than that of the calcareous soil. The calculated parameters of the Freundlich sorption isotherms are listed in Table 2. Generally, the higher the sorption intensity parameter ($1/n$), the lower the binding affinity of soil with Cu. The equilibrium partition coefficient (k) is positively related to the Cu sorption capacity of soils. The Cu sorption capacity and the binding affinity of the calcareous clay loam were higher than that of the acidic sandy loam as indicated by the higher K but lower $1/n$ value. The Cu sorption capacity and binding energy calculated for the two soils with the DOM treatments decreased in the following order: no DOM > compost DOM > sludge DOM. The role of DOM in reducing Cu sorption could be due to the formation of soluble Cu–organic complexes, because Cu can be strongly bound by organic matter (Stevenson and Ardakani, 1972).

Dissolved Organic Matter Concentration Effect

Increasing the DOM concentration caused a significant reduction on the sorption of Cu by both soils (Fig. 2). A significant negative linear correlation between DOM concentration and Cu sorption was observed for these treatments at the DOM concentration range studied ($r = -0.938$ and -0.915 for sludge DOM and sludge compost DOM in the acidic sandy soil, respectively; $r = -0.990$ and -0.996 in the calcareous soil, respectively [$p < 0.05$]). Sorption was decreased by 7.3 and 12.4% with a DOM increment of 100 mg C L^{-1} for sludge compost and sludge DOM, respectively, in calcareous soil, and correspondingly 1.5 and 6.8% in acidic sandy soil, based on the linear regression equations obtained in Fig. 2. When DOM concentration added was raised to 400 mg C L^{-1}, there was no Cu being sorbed by the acidic sandy soil with sludge DOM treatment, while in calcareous soil Cu sorption was reduced by 47 and 28% for sludge and sludge compost DOM treatments, respectively. Sludge DOM had a more significant effect in reducing Cu sorption than that of compost DOM and the effect was more pronounced for calcareous soil than acidic soil.

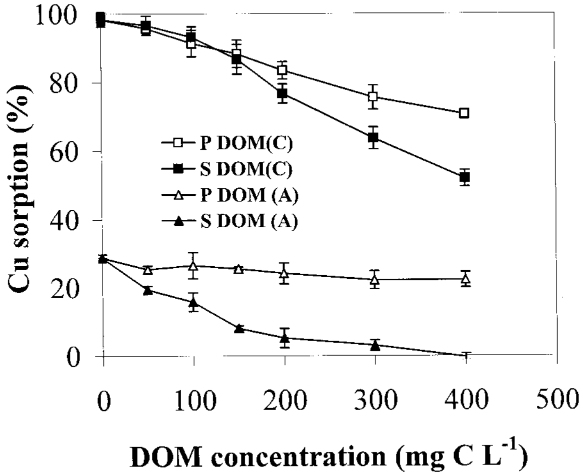


Fig. 2. Effect of concentration of dissolved organic matter (DOM) derived from sludge (S) and sludge compost (P) on the Cu sorption onto the acidic (A) and calcareous (C) soils with an initial Cu concentration of 40 mg L^{-1}.

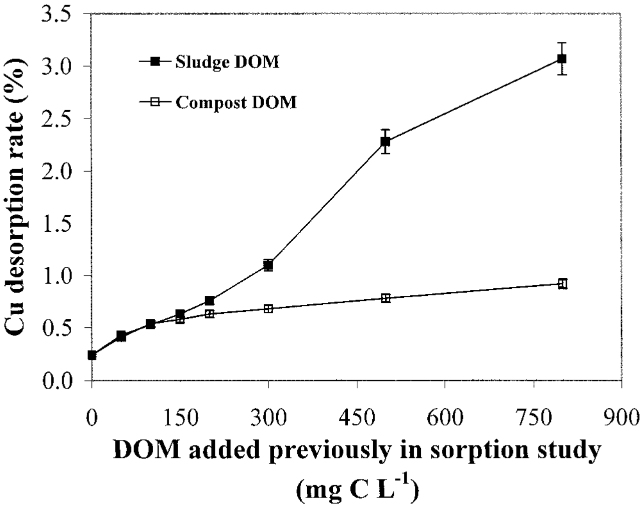


Fig. 3. Desorption of Cu sorbed under the addition of various dissolved organic matter (DOM) concentrations and with an initial Cu concentration of 40 mg L^{-1} by the calcareous clay loam through 1 *M* $MgCl_2$ (pH7) extraction for 2 h.

The origin and concentration of DOM affected not only the Cu sorption of the two soils, but the desorption of Cu sorbed previously in the sorption study (Fig. 3). A relatively higher desorption was found for Cu sorbed in the presence of a higher concentration of DOM, especially for sludge DOM treatment. This might result from a stronger binding affinity of Cu with sludge DOM, and as a result it hinders partly the precipitation of Cu as $Cu(OH)_2$ under such a high-pH condition, as compared with compost DOM. In addition, it was assumed that the higher affinity of the Cu–compost DOM to the soil matrix may also contribute to this phenomenon. This implied that Cu mobility might be facilitated greatly in the soil receiving DOM, especially for sludge DOM, because of the reduction of Cu sorption onto soil and the relatively ready desorption of Cu sorbed previously in the presence of DOM.

The differences in Cu sorption and desorption behavior caused by DOM of different original materials appeared to be closely related to the chemical components of DOM. Sludge compost DOM contained a relatively greater amount of high molecular weight hydrophobic fractions, especially hydrophobic acid (HoA) and hydrophobic neutral (HoN), but fewer hydrophilic fractions, especially hydrophilic base (HiB) and hydrophilic acid (HiA), than that of sludge DOM (Table 3). The Fourier transform infrared (FT–IR) spectrum showed in our previous study that the hydrophobic fraction had a considerably more aromatic acids or aromatic phenols, while the hydrophilic fraction was richer in carboxyl and polyhydroxyl groups. Sludge DOM appeared to have more C–N and C–O groups of a chelating feature, possibly from organic acid, amino acid, and amines, than compost DOM, especially for the HiA, HiB, and HoA fractions (Zhou et al., 2000). Keefer et al. (1984) pointed out that the HiB fraction was mainly comprised of N-containing groups including most amino acids, amino sugars, low molecular weight amines, and pyridine, while the HiA fraction contained the components of the –COO functional group, such as uronic acids, simple organic acids, and polyfunctional acids, which resulted in the higher affinity of Cu with HiB. In addition, many researchers also revealed that hydrophobic fractions of DOM bind more strongly on soils and minerals than the hydrophilic fractions (Gu et al., 1995; Jardine et al., 1989; Kaiser et al., 1997). Liang et al. (1996) found that increased sorption of the DOM extracted from the composted manure is probably due to an increase in molecular weight of the major DOM components. It is reasonable to presume that the reduction of mobile DOM in the soil receiving sludge compost DOM was due either to sorption of greater amounts of compost DOM in comparison with sludge DOM by the soils, or the formation of a Cu–compost DOM complex. The Cu–compost DOM complex could be sorbed repeatedly by the soils, although DOM sorption by soils was not measured in this study. In contrast, sludge DOM contained more HiB fraction, which was not readily sorbed by soils but could strongly associate with Cu. Thus,

Table 3. The characterization of dissolved organic matter (DOM) derived from sludge and sludge compost.

			Hydrophilic fraction†			Hydrophobic fraction‡		
DOM origin	**pH**	**Cu**	**HiA**	**HiB**	**HiN**	**HoA**	**HoB**	**NoN**
		μg per mg DOC	%					
Sludge	7.7	0.24	39.4a§	16.2b	4.18c	38.5a	0.81d	0.85d
Sludge compost	6.9	0.18	21.2b	2.57c	1.86cd	52.0a	0.43d	22.0b

† HiA, hydrophilic acid; HiB, hydrophilic base; HiN, hydrophilic neutral.
‡ HoA, hydrophobic acid; HoB, hydrophobic base; HoN, hydrophobic neutral.
§ Means followed by the same letter within each column are not significantly different at $P \leq 0.05$.

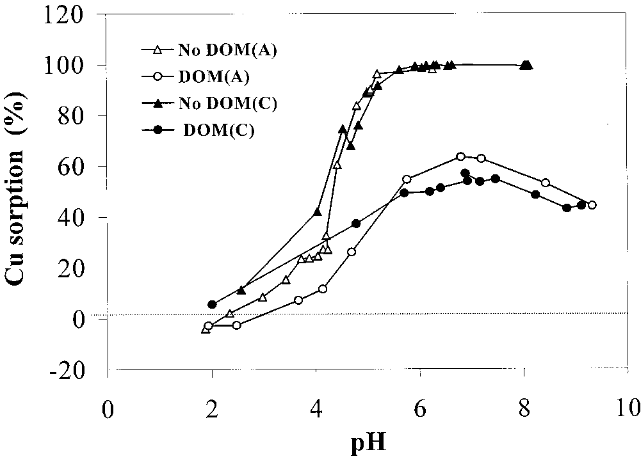
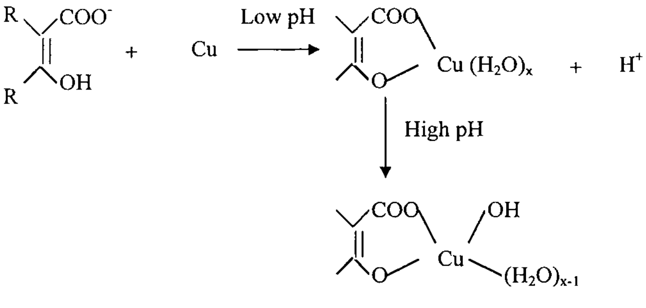


Fig. 4. Effect of pH on Cu sorption onto the acidic (A) and calcareous (C) soils with or without the addition of 300 mg C L^{-1} of sludge dissolved organic matter (DOM).

sludge DOM had a stronger capability to reduce Cu sorption by soils than did compost DOM.

pH Effect

As shown in Fig. 4, the presence of sludge DOM could counteract partially the effects of pH change on Cu sorption by both acidic (pH = 4) and calcareous (pH = 7) soil. Increasing pH from 2 to 10 caused a sharp rise in Cu sorption for both soils receiving no sludge DOM. A sharp change in Cu sorption was found between pH 4 and 5. Maximum Cu sorption was obtained at pH > 5.8, in which more than 99% of Cu could be sorbed by the two soils of different clay content. This was caused mainly by the formation of $Cu(OH)_2$ at pH > 5.8, because in the present experiment, the pH at which $Cu(OH)_2$ precipitation occurred was calculated as 5.77 based on the $Cu(OH)_2$ solubility product of 2.2×10^{-20} (Lindsay, 1979). Addition of sludge DOM reduced the Cu sorption at each respective pH for both soils. The reduction was especially obvious with an increase in pH, which implied that DOM could bind with Cu more readily and strongly at a higher pH. However, at pH > 6.8, Cu sorption unexpectedly decreased with increases in pH in the presence of sludge DOM for both soils. Similar behavior was also observed by James and Barrow (1981). It was assumed that DOM might complex with Cu in different binding forms at various pH values as follows (McBride, 1994; Qin and Mao, 1993):



Hydroxyl groups bound with Cu could be easily ionized in high pH to yield a negative charge. Consequently, the Cu–DOM complex bearing a negative charge would be repelled by the soils of the same charge through which Cu sorption was reduced. Other workers have also reported different binding forms of Cu with organic ligands in various pH levels (Messori et al., 1997). Besides, the dissolved macromolecules exhibited different structures in aqueous solution in various pH conditions, which could modify the exposed surface area and alter the functional group chemistry of DOM. It has been observed that organic molecules dispersed into aggregates of smaller size (<0.1 μm) at high pH and that such constituents exhibited a high affinity toward Cu (Myneni et al., 1999). Thus, it is clear that Cu sorption by soils was simultaneously affected by both pH and DOM concentration at a lower soil pH. At a high-pH condition (>6.8), however, Cu sorption was predominantly affected by DOM due to a strong binding affinity of DOM with Cu. It was concluded that DOM released due to heavy application of sludge onto calcareous soil would result in a leaching loss of Cu. In fact, this hypothesis was supported by the findings of Karlik (1995), who found that liming would increase the leaching of DOM and some trace metals down the soil profile.

CONCLUSION

Our results confirmed that addition of DOM derived from sludge and sludge compost reduced the sorption of Cu by both an acid and a calcareous soil. The Cu sorption isotherm in the presence of either sludge or compost DOM could be well described with the Freundlich equation. The values of *K* of the Freundlich equation of sludge DOM were less than those of compost DOM for both soils, indicating that sludge DOM had a greater effect on reducing soil Cu sorption capability than did compost DOM. This could be attributed to the higher hydrophobicity of compost DOM than sludge DOM. There was less Cu being sorbed as DOM concentration increased. Unlike Cu sorption in soil without DOM addition, Cu sorption was unexpectedly decreased at pH > 6.8 with an increase in pH in the presence of sludge DOM. Therefore, application of sludge or C-rich organic waste on calcareous soil and the liming of sludge-amended soil at pH > 6.8 should be approached cautiously, because this may facilitate formation of soluble DOM–metal complexes and the leaching loss of metals from surface soils, especially for Cu.

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