

Mobilisation of Cu from soils by dissolved natural and processed humic substances

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Dissolved humic substances (HS) may increase the dissolution of heavy metals (HM) and hence augment HM mobility in soils by forming dissolved HM–HS complexes, but the role of HS composition, which depends on the origin of HS, is uncertain. In order to determine the influence of HS composition / origin on HM dissolution, two HM-polluted soil samples were extracted in the batch mode by three different HS solutions containing ~25 mM dissolved organic carbon (DOC) in 0.05 mol/l KNO₃ at pH = 6 for totally 10 weeks with weekly replacements of HS solutions. Two natural HS solutions (MEMO(beech)-HS and MEMO(spruce)-HS) were obtained from litter layers under beech and Norway spruce. Processed HS (Bio-HS) is cow slurry, which has been treated by oxidative, hydrolytic destruction in order to dissolve organic compounds and remove inorganic compounds such as HM. DOC, Cu concentrations and pH were determined in clear extracts obtained each week by centrifugation.

The investigation showed that natural as well as processed HS possess a high capacity to dissolve HM in polluted soils. In fact, up to 50% of total Cu was extracted after 10 weeks from strongly contaminated soil (CRC), indicating that HS has a substantial potential as a cleaning agent for remediation of HM-polluted soils. The origin of HS influenced the extracted amounts, which decreased in the order MEMO(spruce)-HS > MEMO(beech)-HS > Bio-HS, but only in the extraction of the CRC soil, while the three HSs extracted similar percentages from moderately Cu-polluted agricultural soil (Cu-P). Accordingly, HM nature, maybe HS origin and soil composition affect HM mobility and must be taken into consideration if HS is to be used as a cleaning agent in the remediation of HM-polluted soils.

Key words: copper, dissolved humic substances (HS), dissolved organic carbon (DOC), heavy metal mobilisation, soil

INTRODUCTION

Research of HM in soils continues to receive increasing attention due to a deeper understanding of their toxicological importance for ecosystems, agriculture and human health (Alloway, 1995).

The use of phosphate and lime fertilizers, pesticides, application of sewage and manure, and atmospheric deposition represent the main inputs of HM into agricultural soils (Sukreeyapongse et al., 2002).

The environmental risk caused by HM depends on their mobility and bioavailability (Khan et al., 2006; Zehl, Einax, 2005) in soil, which in turn are affected by variables such as pH, soil aeration status, content of clays and pedogenic metal oxides, and the content of solid and dissolved organic carbon (DOC) (Strobel et al., 2005). The extent to which the metals are mobilized depends on the properties of soil. If land use is changed from agriculture to forestry, the choice of tree species can markedly affect various soil

processes and properties, such as pH, weathering, soil structure, transport of solutes, microbial activity, and the properties of soil organic matter (Andersen et al., 2004).

The choice of tree species affects the pH and organic content of the soil as well as the build-up extent of the litter layer and its properties. The pH of the forest floor is generally lower under conifer than under deciduous forest (Raulund-Rasmussen, Vejre, 1995). Many investigations have shown that the solubility of Cu in the soil increases as the pH decreases (McBride, 1989). Even though the acidification of the subsoil and the subsequent mobilization of the metal is slow, this process can eventually lead to groundwater pollution (Andersen et al., 2004).

The different build-up of organic matter in the mineral soil and in the litter layer, as well as differences in the microbial activity sustained by different tree species affect the amount of DOC in the soil solution. Many authors have found a higher DOC concentration under conifers than under broad-leaved trees (Robertson et al., 2000); also Strobel et al., (1999) found a higher DOC concentration in the A-horizon under spruce than under beech (Andersen et al., 2004).

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Humic acids (HA) in sewage sludge influence the behavior of HM in soil environments. Khan et al. (2006) and McBride et al. (1997) suggested that mobile organically complexed forms of HM could account for the migration of substantial fractions of HM out of the surface soil several decades after sludge application. HA are humified, and major fractions of DOC contain acidic groups such as carboxylic and phenolic functional groups, which can form complexes with HM ions and thus result in an increasing bioavailability of HM (Senesia et al., 2003; Marschner, Kalbitz, 2003). HA compete for free metal ions to form soluble complexes and reduce metal adsorption onto the surface of soil particles, and the organometallic complexes of HA and HM could be taken up by plant roots (Evangelon et al., 2004; Khan et al., 2006).

HM cations in soil respond differently to changes in pH and presence of ligands. Copper forms strong complexes with organic ligands in soils and soil solutions (McBride et al., 1997; Strobel et al., 2001). HM form strong complexes with solid and dissolved organic compounds, and they are strongly adsorbed onto soil mineral surface (Strobel et al., 2001).

The aim of the research was to evaluate the capacity of humic substances (HS) of different origin (natural and processed) to dissolve (extract) Cu and, more specifically, to test the possibility of using HS as a cleaning agent in the remediation of soils polluted with Cu.

MATERIALS AND METHODS

Soil samples

The study soils included a strongly polluted soil deposited at a landfill site at Copenhagen Recycling Centre (this soil is called CRC soil) and a moderately Cu-polluted agricultural soil from the University Experimental Station at Tåstrup (called Cu-P soil). The soils were air-dried and sieved (mesh 2 mm). Soil pH was determined potentiometrically by means of a combination electrode in a suspension of soil and 0.01 M CaCl₂ at a soil : solution (mass : volume (m / V)) ratio of 1 : 2.5. The texture was determined by use of the combined hydrometer and sieving methods (Day, 1965). Calcium carbonate in the CRC soil was determined volumetrically by a calcimeter (Allison, Moodie, 1965). Organic carbon was determined by dry combustion at 1250 °C in oxygen (ELTRA, 1995); the C content of the CRC soil was corrected for inorganic C (CaCO₃). Total HM contents were determined by ICP-OES on digests obtained by boiling the soil with HNO₃ (ratio 1 : 1 (m / V)) for 8 h (Tjell, Hovmand, 1978).

Humic substance (HS) solutions

Three HS solutions were used, including two from forest litter under beech and Norway spruce (called MEMO(beech)-HS and MEMO(spruce)-HS, respectively) and from processed cow slurry (called Bio-HS). Beech-HS and Spruce-HS solutions were isolated by centrifugation at 4000 g for 30 min, followed by filtration (Durapore Membrane, PVDF, hydrophilic, 0.45 µm) and treatment with H⁺-saturated cation-exchange resin (Dowex 50W-X) to reduce metal content as described by Strobel et al. (2001b). Bio-HS was prepared by oxidizing hydrolytic degradation of an alkaline cow slurry heated under pressure and limited

O₂ inlet for a few hours as described by BioCorrection (2007) and Popov et al. (2002).

The concentration of DOC was determined by a Shimadzu TOC-500 Total Organic Carbon Analyzer. HM contents in the three HS solutions diluted to 25 mM DOC were determined by graphite furnace atomic absorption spectrometry (GFAAS) on a Perkin Elmer 5100, Zeeman 5100, HGA 600 instrument with an AS 60 autosampler.

All results of DOC and extracted HMs were evaluated by statistical calculations estimating the standard deviation (SD) of three sample replicates.

Extraction

The extractions were carried out in a batch mode with a solution : soil ratio of 5 : 1 (m / V) in triplicates with solution renewal each week (168 h) for a total of 10 weeks. Batch extraction is preferable according to Heil et al. (1999), and one week between solution renewals was chosen because 4–6 days had been found necessary for equilibration (Strobel et al., 2001a). The extraction solutions comprised the three HS solutions diluted to contain 25 mM DOC in a 0.05 mol/l KNO₃ matrix and as control solely 0.05 mol/l KNO₃, all adjusted to pH 6.0. The extractions were carried out in 50 ml polypropylene centrifuge tubes with screw-caps, where 5.000 g of soil was shaken end-over-end with 25.00 ml of extraction solution for one week. After centrifugation at 3000 g for 10 min, 10.00 ml clear supernatant was removed and diluted 10 times with a mixture of 0.2% HNO₃ and 0.05 mol/l KNO₃. The concentrations of Cu were determined in this dilution by GFAAS. In another 10.00 ml clear supernatant portion, the DOC concentration and pH were determined as described above. Twenty millilitres of fresh extraction solution was pipetted into the centrifuge tube, and extraction continued for another week (168 h). A total of 10 extractions (10 × 168 h) were performed with each of the two soils and the three HS solutions and the control.

Based on Cu concentrations determined after each week, the accumulated metal release was calculated by the formula:

$$Y_n = X_n \times 5 - X_{n-1} + Y_{n-1},$$

where Y is accumulated release in µmol/kg, X is metal concentration in µmol/l, n is number of extractions (= weeks) and 5 is the solution to soil ratio in l/kg; X_{n-1} accounts for the Cu content in 5 ml of solution per 5 g soil left over from the previous extraction.

RESULTS

Soil characteristics

The CRC soil is pH-neutral due to carbonate content, while the Cu-P soil is acidic. The carbonate content in the CRC soil is calculated as CaCO₃, but the presence of minor amounts of HM carbonates, e. g., CuCO₃ cannot be excluded. The organic carbon and clay contents are similar in both soils, but the CRC soil is richer in silt as compared to the Cu-P soil (Table 1).

The total Cu contents are much higher in the CRC soil than in the Cu-P soil (Table 2).

pH was slightly increasing, but close to 6.0 throughout 10 weeks of extraction of the Cu-P soil, except extraction with Bio-HS in which pH was about 7.0 pH.

Table 1. Selected characteristics of the CRC and Cu-P soils, and HS

Material	*Particle size distribution (%)			pH (CaCl ₂)	OC, %	DOC, mM	¹ CaCO ₃ (%)	Cu	Cd	Ni	Pb
	Clay (2µm)	Silt (2–20 µm)	Sand (20–2000 µm)					(mg/kg)			
CRC	10	16	74	7.4	1.4		5.8	770	10	9.4	323.3
Cu-P	10	6	84	5.3	1.2		nd	82	0.62	5.1	19
Bio-HS	nr	nr	nr	6.7		330	nr	nr	nr	nr	nr
MEMO(beech)-HS	nr	nr	nr	5.79		96	nr	nr	nr	nr	nr
MEMO(spruce)-HS	nr	nr	nr	5.16		79	nr	nr	nr	nr	nr

¹ Calculated as calcium carbonate.

* The soils were distributed to particle size according to classification of Day, 1965.

nr – no results, nd – not detected.

Table 2. Total Cu contents (CuTotal) in CRC and Cu-P soils together with uncorrected and corrected accumulated releases after 10 weeks (Cu10) by three HS as well as uncorrected and corrected percentages of Cu10 to CuTotal (in parentheses). In the calculation of corrected Cu10, 10-week releases were recalculated assuming a HS content of 25.0 mM DOC

Heavy metal	Cu _{total} µmol/kg	Cu ₁₀ (Bio-HS)		Cu ₁₀ (MEMO(beech)-HS)		Cu ₁₀ (MEMO(spruce)-HS)	
		Uncorr.	*Corr.	Uncorr.	*Corr.	Uncorr.	*Corr.
		µmol/kg (%)		µmol/kg (%)		µmol/kg (%)	
CRC soil							
Cu	12118	3597 (30)	4408 (36)	5403 (45)	5425 (45)	6486 (54)	5791 (48)
Cu-P soil							
Cu	1291	364 (28)	446 (35)	431 (33)	433 (34)	434 (34)	388 (30)

* Bio-HS contains 20.4 mM DOC; MEMO(beech)-HS contains 24.9 mM DOC; MEMO(spruce)-HS contains 28.0 mM DOC.

DOC in extracts

DOC in control (0.05 mol/l) was almost zero during the whole period of extraction of both soils (Fig. 1, a and b). Extraction of Cu-P soil (Fig. 1a) with all three HS solutions showed an increasing DOC content during the first three weeks; then DOC stabilized, respectively to 20.4 mM for Bio-HS, 28.0 mM for MEMO(spruce)-HS and 24.9 mM for MEMO(beech)-HS. In contrast, DOC content in the extracts from CRC soil was lower than the original extractant concentration at the beginning of extraction (Fig. 1b). While MEMO(spruce)-HS and MEMO(beech)-HS tend to reach the maximum concentrations after 5–7 weeks and to exceed the original concentrations, the concentration in the Bio-HS extracts remained slightly lower than the original concentration (20.4 mM) throughout the extractions.

Cu in extracts

Cu concentrations in extracts from the CRC soil increased during the first 4–5 weeks and then gradually decreased reaching almost constant values after 8 weeks (Fig. 2a). The accumulated releases follow S-shaped curves starting to increase after two weeks and gradually leveling off towards the end of extraction (Fig. 2b).

Cu concentrations in extracts and accumulated release from Cu-P soil follow curves resembling those from CRC soil, but concentrations and accumulated release were much lower and the releases started earlier than with the CRC soil (Fig. 2 c and d).

DISCUSSION

The amounts of Cu extracted by HS-containing extractants are seen in Fig. 2 to be much higher than the amounts extracted in control. In fact, higher amounts of Cu were extracted by control

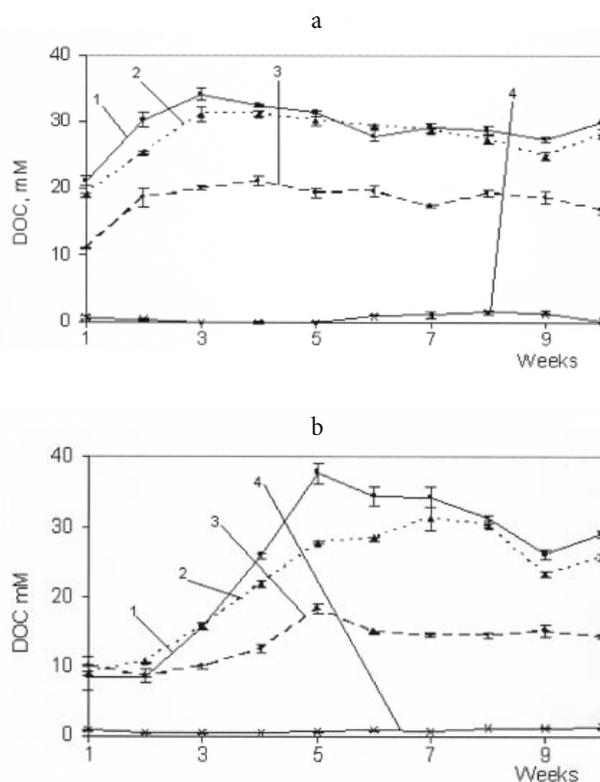


Fig. 1. DOC concentrations in solutions in contact with Cu-P soil (a) and CRC soil (b).

Extractants:

---■--- (1) MEMO(spruce)-HS; ...▲... (2) MEMO(beech)-HS;
-●- (3) Bio-HS; ---x--- (4) control.

The error bars indicate standard deviation (SD).

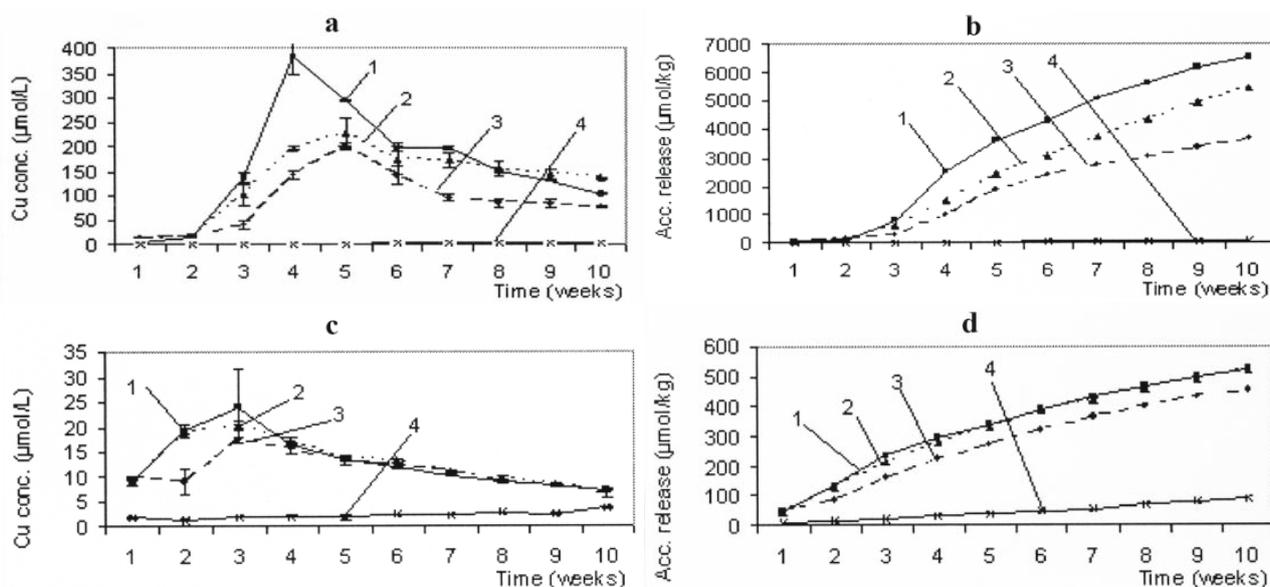


Fig. 2. Cu concentrations in extracts (a) and accumulated release (b) from CRC soil and in extracts (c) and accumulated release (d) from Cu-P soil.

Extractants: ----■---- (1) MEMO(spruce)-HS; ...▲... (2) MEMO(beech)-HS; -.-●-.- (3) Bio-HS; ----x---- (4) control.

The error bars indicate standard deviation (SD)

from the Cu-P soil than from the CRC soil, which can be ascribed to the lower pH of extracts from Cu-P soil than from CRC soil. Another difference between the two systems is the lower HS concentrations in extracts from CRC soil than from the Cu-P soil, particularly in the first extracts (Fig. 1). This difference is undoubtedly due to precipitation of Ca-HS or adsorption of HS onto CaCO_3 , which is present in the CRC soil but not in the Cu-P soil (Table 1).

The observed capacity of HS to mobilize HM is in accordance with numerous previous investigations showing increased concentrations of HM in HS-containing extracts from natural soils (Andersen et al., 2004; Khan et al., 2006; Strobel et al., 2001 a, b). However, compared to these naturally (and diffusely) contaminated soils, the CRC soil originates from point contaminated grounds with much higher contents of HM of anthropogenic origin. The Cu-P soil is point-contaminated, but only the Cu content is markedly elevated as compared to general contents in diffusely contaminated agricultural Danish soils.

In order to facilitate a comparison between extractabilities of HM from the two soils and the capacities of the three HS to mobilize HM, total HM contents taken from Tables 1 are shown in Table 2 together with accumulated HM releases after 10 weeks, all in $\mu\text{mol/kg}$.

The accumulated releases in Table 2 were corrected in two ways. Firstly, the HM amounts released into control solution after 10 weeks were subtracted from the amounts released to HS-containing extracts. These values are called 'Uncorr' (Table 2). Secondly, to allow for different HS concentrations in the extractants (Bio-HS contains 20.4 mM DOC, MEMO(beech)-HS contains 24.9 mM DOC and MEMO(spruce)-HS contains 28.0 mM DOC), accumulated releases were recalculated for HS solutions containing 25.0 mM DOC, assuming a linearity between the amounts extracted and DOC concentration; these values are called 'Corr' (Table 2). In addition to accumulated releases in $\mu\text{mol/kg}$, the percentages of total HM contents released during 10 weeks are shown in parentheses.

Regarding extraction of Cu from the CRC soil (Table 2), it may be seen that up to about half of the total contents of Cu can

be extracted by the MEMO-HS, while about one third of this metal is extracted by Bio-HS. For Cu, the extractability increased in the order: Bio-HS < MEMO(beech)-HS ~ MEMO(spruce)-HS; the similar extraction capacities of MEMO(beech)-HS and MEMO(spruce)-HS are most clearly shown with corrected percentages.

In contrast to extraction of the CRC soil, almost the same percentages of Cu were extracted by all three HS. In fact, the processed HS (Bio-HS) tends to be a slightly more effective extractant for Cu than the natural HS (MEMO(beech)-HS and MEMO(spruce)-HS).

The observed differences in Cu extractability of the two soil samples indicate that soil composition and HS origin must also be considered important for HM mobilization and should be taken into consideration if HS is used as a cleaning agent in the remediation of HM-polluted soils. For this purpose, other factors such as pH, HS concentration and temperature, which are known to affect HM mobility (Borggaard, Elberling, 2004), should be undoubtedly also taken into consideration, but their precise management in relation to soil remediation awaits further investigation.

CONCLUSIONS

Natural as well as processed HS possess a high capacity to dissolve HM in polluted soils. In fact, up to 50% of total Cu was extracted after 10 weeks from strongly contaminated CRC soil, indicating that HS has a substantial potential as a cleaning agent for remediation of HM-polluted soils. The origin of HS influenced the extracted amount, which decreased in the order MEMO(spruce)-HS > MEMO(beech)-HS > Bio-HS, but only in the extraction of the CRC soil, while similar percentages of the three HS were extracted from the Cu-P soil.

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Cu JUDRUMAS DIRVOŽEMIUOSE, VEIKIANT JUOS NATŪRALIOMIS IR IŠ PERDIRBTŲ ATLIEKŲ GAUTOMIS HUMUSO MEDŽIAGOMIS

Santrauka

Ištirpusios humuso medžiagos (HM) gali padidinti sunkiųjų metalų (SM) skaidymąsi, vadinasi, jos didina SM judrumą dirvožemyje, formuodamos ištirpusių SM–HM kompleksinius junginius, bet HM sudedamųjų dalių vaidmuo, kuris priklauso nuo HM kilmės, nėra žinomas. Siekiant nustatyti HM sudedamųjų dalių ir jų kilmės poveikį SM tirpumui, du SM užterštų dirvožemių mėginiai buvo ekstrahuojami, veikiant juos 10 savaičių trimis skirtingų HM tirpalais, kurių sudėtyje yra ~25 mM ištirpusios organinės anglies ir pH 6. Tirpalai buvo keičiami kas savaitę. Išcentrifuguotose ištraukose buvo nustatytos Cu koncentracijos ir pH. Dvi HM (MEMO(buko)-HM ir MEMO(eglys)-HM) buvo gautos iš buko ir Norvegijos melsvosios eglės miško pakločių. Antropogeninės kilmės HM (Bio-HM) oksidacinės hidrolizės būdu buvo perdirbtos iš karvių srutų.

Tyrimai parodė, kad natūralios, kaip ir perdirbtos, HM sugeba mobilizuoti (ištirpinti) SM užterštuose dirvožemiuose. Daugiau nei 50% viso Cu iš labai užteršto CRC dirvožemio buvo ekstrahuota po 10 savaičių. HM kilmė turėjo įtakos ekstrahuoto Cu koncentracijai, kuri mažėjo tokia seka: MEMO(eglys)-HM > MEMO(buko)-HM > Bio-HM, bet tik ištraukose su CRC dirvožemiu, tuo tarpu visos trys HM ištraukė panašius Cu kiekius iš vidutiniškai Cu užteršto žemės ūkio Cu–P dirvožemio. Taigi SM prigimtis, HM kilmė ir dirvožemio sudėtis turi reikšmės SM judrumui ir turi būti įvertintos jų reikšmės, jeigu HM naudojamos kaip dirvožemio gerinimo priemonė.

Raktažodžiai: varis, ištirpusios humuso medžiagos (HM), ištirpusi organinė anglis, sunkiųjų metalų mobilizacija, dirvožemis