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**EFFECTS OF CHELATING AGENTS
ON COPPER, LEAD AND ZINC SOLUBILITY
IN POLLUTED SOILS AND TAILINGS
PRODUCED BY COPPER INDUSTRY**

**WPŁYW DODATKU ZWIĄZKÓW KOMPLEKSUJĄCYCH
NA ROZPUSZCZALNOŚĆ MIEDZI, OŁOWIU I CYNKU
W GLEBACH SKAŻONYCH
ORAZ OSADACH POFLOTACYJNYCH GÓRNICICTWA MIEDZI**

Abstract: The main aim of this paper was to examine the effects of chelating agents on the mobilization of heavy metals from polluted soils and tailings generated by copper ore processing. Ammonium salts (chloride and nitrate(V)), organic acids (citric and tartaric), amino acids (glycine and histidine) and synthetic chelating agents EDTA (*ethylenediaminetetraacetic acid*) and EDDS (*ethylenediaminedisuccinic acid*) were applied to soil suspension at the rate of 5 mmol · kg⁻¹ and shaken. Similar tests were performed also with two kinds of sewage sludge, at various stages of biochemical stability. Examined were 8 samples collected from the dumping sites Żelazny Most and Wartowice, as well as 2 samples of polluted soils from the vicinities of copper smelters Legnica and Głogów were tested.

Total concentrations of metals in tailings were: 1300–3800 mg Cu · kg⁻¹, 80–300 mg Pb · kg⁻¹ and 23–35 mg Zn · kg⁻¹, whereas the concentrations of those metals in soils were: 1270–1500 mg Cu · kg⁻¹, 304–336 mg Pb · kg⁻¹ and 80–89 mg Zn · kg⁻¹. Efficiency of metals extraction from soils and tailings varied between tested soils and tailings material, metals and chemical agents applied. EDTA appeared the most effective in solubilizing Cu, and extracted 7–39 % of total Cu and 2–18 % Pb from various samples. The efficiency of Cu solubilization from soils and tailings by other agents was in the following order: citric acid (for tailings) > glycine > histidine > EDDS > citric acid (for soils) > tartaric acid > ammonium chloride > ammonium nitrate. The order for extraction of two other metals differed from that obtained for Cu. The tests with sewage sludge did not prove essential increase of Cu and Pb solubility, despite considerable amounts of dissolved organic matter present in one sludge. This effect can be explained by the sorption of chelated forms of metals by highly humified organic matter.

Keywords: soil, heavy metals, solubility, chelates, complexing, sewage sludge

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Copper industry produces huge amounts of wastes, in particular those obtained from flotation process, that are dumped in tailings dumping sites. There are seven such objects in Lower Silesia, of which the largest is still operating, whereas other 6 have been closed and partly reclaimed [1]. Reclamation of those objects is difficult, mainly because of unsuitable physical and chemical properties of tailings, such as lack of organic matter, shortage of bioavailable nutrients (N and P) and high concentrations of metals, in particular Cu and Pb [2]. Grain size composition of tailings depends on the properties of gangue rocks accompanying copper ores, and varies strongly: from sand and sandy silt to silty loam and clay. Tailings material contains certain amounts of limestone or dolomite, and therefore indicates alkaline or neutral reaction. For this reason, heavy metals are believed to remain there in insoluble and non-phytoavailable forms, and their presence should not cause the problems with biological reclamation. Application of organic matter will be needed for successful vegetation on tailings, to supply necessary macro- and micronutrients as well as to improve the physical properties of deposited material. Various organic materials are usually considered as a source of organic matter for reclamation of bare soils or landfills; there are numerous reports on the application of peat or lignite, as well as waste materials, such as manure, sewage sludge or composts. It should be pointed out, however, that poorly humified organic material should not be considered for reclamation of soils rich in heavy metals because of a risk of metals mobilization. Metals solubility may dramatically increase in the presence of low molecular weight organic compounds that act as chelating agents, and may transform metals into soluble chelates both in acidic and neutral or alkaline conditions [3–5]. It has been proved that organic acids present in plant root exudates may act as compounds chelating soil aluminum, iron and heavy metals; similar effects may be caused by fulvic acids and low molecular acids or amino acids produced in considerable amounts during the transformation of soil or waste organic matter, eg before sewage sludge or organic wastes become biochemically stabilized or before the compost reaches the stage of full maturity.

In this study, we examined the effect of copper, lead and zinc solubilization from tailings, as well as from soils highly polluted by copper smelting processes, caused by the application of substances that may act as chelating agents, and might be introduced to soil or tailings, by accident or on purpose, during their reclamation. Inorganic compounds containing ammonium ion that may act as Cu-chelating ligands [3, 5], ie, pure organic acids and amino acids, synthetic chelating compounds, as well as two kinds of sewage sludge and sewage sludge extracts were investigated. In the light of the literature, it is not clear whether the application of sewage sludge into soil (or soil parent rock) containing high concentrations of metals will or not lead to increased metal solubility and bioavailability. There are many reports proving, that sewage sludge application poses the risk of increasing metal solubility in soils [6–9]. Accordingly, legal regulations, introduced in Poland and in other countries, have established maximum permissible contents of heavy metals in soils to be amended with sewage sludge. On the other hand, however, the introduction of organic material, such as compost or sewage sludge, causes an increase of the soil sorption capacity, and therefore leads to the immobilization of potentially soluble metals [10–12]. Thus, metals

solubility and bioavailability will decrease rather than increase after the application of sewage sludge. Several authors point out, however, that metal sorption by the solid phase and complexation by dissolved organic ligands must be studied simultaneously, because both processes may counteract one another [13, 14]. This statement prompted us to examine changes in the solubility of metals in polluted soils and in tailings after the application of various pure chelating substances, as well as sewage sludge at various stages of biochemical stability.

Material and methods

Investigations dealt with 8 samples of tailings collected from the surface layers (0–20 cm) in two dumping sites: the operating one Zelazny Most and Wartowice, officially called Iwiny III, closed 30 years ago and not reclaimed. Additionally, 2 soil samples were collected from the surface layers (0–20 cm) of soils in the vicinities of copper smelters Legnica (soil L) and Glogow (soil G). Basic physicochemical and chemical properties of tailings and soils were determined according to the common methods used in soil science [15]. Grain size distribution was analysed by areometric-sieve method according to Polish Norm PN-R-4033, and assigned to the textural groups determined by USDA [16]. The properties of tailings collected from those two objects differed strongly, which was mainly caused by different geological conditions of two copper mining centers [1, 3]. The differences within the groups of four samples collected from the same object were small (with standard deviations below 20 %), which confirms their relative homogeneity. Therefore, all results presented in the paper will be given as mean values for four samples. The tailings from Zelazny Most (described further as tailings ZM) were light in texture and had grain size composition of sand. They contained 1–5 % (with the mean value: 3 %) of clay fraction (< 0.002 mm) (Table 1).

Table 1

Basic properties of soils and tailings tested in the study

Soil / tailings material	Texture	Share of particles [mm]		Organic carbon C _{org} [g · kg ⁻¹]	pH	CaCO ₃ [%]	CEC [mmol(+) · kg ⁻¹]	Total contents of metals		
		< 0.02	< 0.002					Cu	Pb	Zn
		[%]								
Soil L	silty loam	41	11	9.1	6.0	0	125	1270	336	80.3
Soil G	sand	18	4	7.5	6.4	0	96.3	1490	304	89.0
Tailings ZM	sand	8	3	6.5	7.5	9.5	n.d.*	1309	301	22.9
Tailings W	silty loam	64	11	11	7.7	56	n.d.	3780	77.8	35.4

* n.d. – not determined.

The texture of tailings Wartowice (tailings W) was much heavier, determined as silty loam, with 11 % of mean clay contribution, and very high amount (64 %) of the sum of fractions < 0.02 mm. Tailings ZM contained lower amounts of total Cu (0.13 %) than the tailings W (0.38 %), but the mean contents of total Pb were higher in the tailings

ZM ($301 \text{ mg} \cdot \text{kg}^{-1}$) than in tailings W ($78 \text{ mg} \cdot \text{kg}^{-1}$). Two soil samples tested in this study (soil L and G) also differed in their textures, that were determined as sand and silty loam, respectively. Concentrations of Cu and Pb in both soils were comparable with those in tailings ZM (Table 1).

Easily soluble forms of metals were determined in soil and tailings material in the common extraction tests with $1 \text{ mol} \cdot \text{dm}^{-3} \text{ NH}_4\text{NO}_3$ and $0.01 \text{ mol} \cdot \text{dm}^{-3} \text{ CaCl}_2$ [2]. The tests of metals extractability with chelating agents were performed as batch extractions (m/v : 1/5), in which the chelating agents were applied to soil suspension at the rate $5 \text{ mmol} \cdot \text{kg}^{-1}$ soil. The following agents were applied: ammonium chloride and ammonium nitrate(V), citric and tartaric acids, amino acids: glycine and histidine and synthetic chelating agents EDTA (*ethylenediaminetetraacetic acid*) and EDDS (*ethylenediaminedisuccinic acid*). Similar tests were performed also with two kinds of sewage sludge applied at the rate 1 g d.m. per 20 g soil d.m. Sewage sludge material was obtained from two municipal wastewater treatment plants, as freshly produced and mechanically dewatered (sludge 1) and stabilized in methane digestion tank (sludge 2). Basic properties of sludge material, as well as dissolved organic matter content (DOM) and metals contents are presented in Table 2.

Table 2

Basic properties of sewage sludge applied

Sewage sludge	OM [g · kg ⁻¹]	pH	CaCO ₃ [%]	CEC [mmol(+) · kg ⁻¹]	DOM [g C · kg ⁻¹ d.m.]	Total contents of metals		
						Cu	Pb	Zn
						[mg · kg ⁻¹]		
1	600	6.0	0	680	56	462	144	820
2	550	6.1	0	750	41	365	155	931

In order to estimate the pure effects of metal mobilization, ie, and to avoid subsequent sorption of soluble chelates by solid sludge organic matter, parallel tests were carried out in which sludge water extracts, obtained from the same amounts of sludge, were used. The extracts were prepared by shaking sludge with water (1/10, *d.m./v*), centrifuging and filtering prior to application to soil suspension.

Results and discussion

Easily soluble forms of metals, as determined by common single extraction method by $0.01 \text{ mol} \cdot \text{dm}^{-3} \text{ CaCl}_2$, proved that all metals examined are poorly soluble both in tailings and in polluted soils. Copper forms extracted by $0.01 \text{ mol} \cdot \text{dm}^{-3} \text{ CaCl}_2$ remained below 0.4 % of their total contents in soils and below 0.2 % in tailings, which corresponded with absolute values of $4.0\text{--}5.2 \text{ mg} \cdot \text{kg}^{-1}$ and $0.6\text{--}2.1 \text{ mg} \cdot \text{kg}^{-1}$, respectively (Table 3). Similarly low were contributions of soluble Pb, and absolute amounts of soluble Zn, in soils and tailings. Attention should be paid to strikingly high amounts of “easily soluble” Cu, ie, that extracted both from soils and tailings with $1 \text{ mol} \cdot \text{dm}^{-3} \text{ NH}_4\text{NO}_3$, which is the standard method in several countries [2].

Particularly high amounts of Cu were extracted from tailings (61–66 % of total Cu), and this fact may be explained by formation of Cu ammine complexes $[\text{Cu}(\text{NH}_3)_4]^{2+}$ under alkaline conditions [3–5], as well as by the formation of HNO_3 as a product of ammonium nitrate hydrolysis. Similar data have already been reported [2, 5].

Table 3

Extractable metals determined in the common tests of extractability (mean values)

Soil / tailings material	Easily soluble metals in soils and tailings [$\text{mg} \cdot \text{kg}^{-1}$], as extracted by					
	$1 \text{ mol} \cdot \text{dm}^{-3} \text{NH}_4\text{NO}_3$			$0.01 \text{ mol} \cdot \text{dm}^{-3} \text{CaCl}_2$		
	Cu	Pb	Zn	Cu	Pb	Zn
Soil L	149	5.3	22.3	5.2	1.1	8.9
Soil G	96.5	2.3	9.6	4.9	0.95	5.9
Tailings ZM	807	8.2	0.3	2.1	1.7	0.4
Tailings W	2590	4.3	1.8	0.6	0.9	0.2

High mobilization affinity of Cu present in tailings, and to lesser extent in soils, as caused by the solutions containing high concentrations of ammonium cations, was not reflected however when ammonium salts were applied at low ratio of $5 \text{ mmol} \cdot \text{kg}^{-1}$. In that case, the amounts of Cu extracted from tailings and soils remained very low, ie, in the ranges $0.9\text{--}6.4 \text{ mg} \cdot \text{kg}^{-1}$ and $1.25\text{--}1.4 \text{ mg} \cdot \text{kg}^{-1}$, respectively. Ammonium salts (both chloride and nitrate) applied at that ratio, appeared to have the poorest capacity to mobilize Cu, if compared with other compounds tested in the study.

The most efficient chelating agent was EDTA, that applied at the rate of $5 \text{ mmol} \cdot \text{kg}^{-1}$ was able to solubilize $254\text{--}507$ and $284\text{--}287 \text{ mg Cu} \cdot \text{kg}^{-1}$ from tailings and soils, respectively (Table 4). Those amounts corresponded to the range: from 7 % (tailings W) up to 39 % (tailings ZM) of total Cu.

Table 4

Amounts of metals extracted from tailings with chelating agents applied at the ratio of $5 \text{ mmol} \cdot \text{kg}^{-1}$

Chelating agent	Tailings Zelazny Most			Tailings Wartowice		
	Cu	Pb	Zn	Cu	Pb	Zn
	[$\text{mg} \cdot \text{kg}^{-1}$]					
EDTA	507	49.9	1.0	254	1.4	1.5
EDDS	97	2.5	2.1	85	1.1	0.9
Glycine	284	0.8	0.2	215	0.4	0.7
Histidine	98	1.1	0.8	101	0.8	0.6
Citric acid	431	9.3	2.6	248	0.4	0.6
Tartaric acid	134	4.8	1.6	6.9	0.7	0.2
NH_4NO_3	3.0	1.1	0.3	0.9	0.4	0.6
NH_4Cl	6.4	1.7	0.5	3.9	0.5	0.8

The orders of mobilizing capacities were different for various materials (tailings vs soils) as well as for various metals. Different reactions of chelating agents with various materials may be partly explained by their various pH-related characteristics [4, 5]. Copper was mobilized from tailings by various chelating agents according to the order: EDTA > citric acid > glycine > tartaric acid (*case of tailings ZM*) >> histidine > EDDS > tartaric acid (*case of tailings W*) >> NH₄Cl > NH₄NO₃ (Table 4).

The order of the capacity to mobilize Cu from polluted soils was as follows: EDTA >> EDDS / glycine / histidine >> citric acid / tartaric acid >> NH₄Cl > NH₄NO₃ (Table 5). None of the compounds proved to mobilize Cu from polluted soils with the efficiency comparable to that of EDTA.

Table 5

Amounts of metals extracted from soils with chelating agents applied at the ratio of 5 mmol · kg⁻¹

Chelating agent	Soil L			Soil G		
	Cu	Pb	Zn	Cu	Pb	Zn
	[mg · kg ⁻¹]					
EDTA	287	31.8	5.9	284	23.7	7.1
EDDS	46.3	0.8	1.7	51.7	0.6	2.3
Glycine	36.5	3.7	0.6	65.9	3.9	0.8
Histidine	40.1	0.3	0.45	50.0	0.5	0.7
Citric acid	7.5	1.3	0.4	9.2	1.3	1.3
Tartaric acid	9.6	2.6	0.4	13.1	2.9	1.8
NH ₄ NO ₃	1.2	0.2	0.2	0.3	0.35	0.8
NH ₄ Cl	1.4	0.2	0.25	0.5	0.25	0.6

The amounts of Pb and Zn solubilized from tailings and soils by chelating agents tested in this study were much lower than those of mobilized Cu. Application of EDTA led to the mobilization of considerable Pb amounts from the tailings ZM (49.9 mg · kg⁻¹) and from soils (23.7–31.8 mg · kg⁻¹). Those values corresponded to the range 8–16 % of total Pb. The mobilization of Pb from the tailings W was much less effective (1.4 mg · kg⁻¹, ie 1.8 % of total Pb).

The effects of Pb mobilization by other chelating agents were much poorer than those obtained with EDTA (Tables 4 and 5), and therefore will not be discussed in details. The effects of chelating agents on Zn solubility should also be considered as negligible, and the amounts of Zn released to the solutions were lower than those determined as easily soluble and exchangeable Zn, extracted by 0.01 mol · dm⁻³ CaCl₂.

Results obtained in the tests with two kinds of sewage sludge did not confirm the mobilization of metals from the tailings or soils after biosolids application (Table 6).

The amounts of Cu, Pb and Zn released from the solid to liquid phase of soil + sludge or tailings + sludge suspensions remained lower than those determined as easily soluble. These results support the opinions presented by various authors [10–12], who proved that biosolids application significantly reduced metals mobility and, accordingly, will

not cause environmental risk of metal mobilization from polluted soils or metals-rich wastes, such as tailings. On the other hand, however, the amounts of all metals released to the liquid phase after application of sewage sludge extracts were much higher than those observed when untreated sludge were applied in the tests (Table 6).

Table 6

Amounts of metals extracted from soils [$\text{mg} \cdot \text{kg}^{-1}$] with sewage sludge and sewage sludge extracts

Metal:	Cu	Pb	Zn	Cu	Pb	Zn
Application of:	Sewage sludge 1			Sewage sludge 2		
Soil L	0.4	0.45	3.2	3.5	0.2	3.7
Soil G	1.2	0.35	5.8	2.9	0.25	4.9
Tailings ZM	0.35	1.05	0.6	1.2	1.1	0.8
Tailings W	0.45	< 0.1	< 0.1	0.2	< 0.1	0.4
Application of:	Extract from sewage sludge 1			Extract from sewage sludge 2		
Soil L	4.9	1.2	4.0	5.5	1.4	4.5
Soil G	9.0	1.5	8.5	10.5	1.3	6.7
Tailings ZM	4.5	1.6	1.3	6.1	1.45	1.35
Tailings W	9.5	< 0.1	0.3	12.2	< 0.1	0.45

This observation stays in accordance with the hypothesis that easily soluble organic substances (DOM) present in various amounts in sewage sludge, do react with heavy metals, particularly with Cu and Pb, and form soluble complexes, like chelates, that potentially may be mobilized to soil solution. At the same time however, those potentially soluble complexes, are effectively adsorbed and immobilized by well-humified organic matter present in sewage sludge or in soil with high sorption capacity.

Therefore, application of sludge or sewage sludge or other organic waste rich in soluble fractions, to calcium carbonate rich soil or limed soil, at $\text{pH} > 6.8$ should be carried out carefully, because in such conditions, the formation of soluble DOM-metal complexes will be possible, which may lead to leaching of metals, especially Cu, from surface soils or reclaimed waste material such as tailings. Consequently, also the phytoavailability of Cu may vitally increase. Possible impacts of the latter effect should be tested in further experiments.

Conclusions

Heavy metals present in tailings, as well as metals present in soils polluted by the processes of copper smelting, may be released from the solid phase into soil solution by various chelating agents. The efficiency of metal solubilization will not be easy to predict, as it strongly depends on the kind of chelating agent, soil or waste properties as well as on the kind of metal. Of three metals examined, the highest affinity to be solubilized by chelating agents has undoubtedly Cu, and the most effective chelator was

EDTA. Several organic compounds, naturally occurring in the environment, such as organic acids or amino acids may also act as considerably effective metal-solubilizing agents. Therefore, special attention is needed when polluted soils or wastes would be treated with biosolids, that may contain soluble organic compounds. Environmental risk caused by metal solubilization when sewage sludge is applied, will be reduced by possible sorption of chelated metals by solid organic matter, and therefore sludge application acts as a factor immobilizing metals rather than mobilizing one. The results of this study may be helpful for planning treatments of chelate-induced soil decontamination or application of organic material in the processes of soil remediation and waste landfills revegetation.

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WPŁYW DODATKU ZWIĄZKÓW KOMPLEKSUJĄCYCH NA ROZPUSZCZALNOŚĆ MIEDZI, OŁOWIU I CYNKU W GLEBACH SKAŻONYCH ORAZ OSADACH POFLOTACYJNYCH GÓRNICICTWA MIEDZI

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Abstrakt: Celem pracy było badanie wpływu substancji kompleksujących na rozpuszczalność metali z zanieczyszczonych gleb i osadów poflotacyjnych górnictwa miedzi. Do testów ekstrakcji przeprowadzonych metodą wytrąsania w zawiesinie, zastosowano sole amonowe (chlórek i azotan (V)), kwasy organiczne (winowy i cytrynowy), aminokwasy (glicynę i histydynę) oraz syntetyczne substancje chelatujące EDTA (*kwasa wersenowy*, tj. *kwasa etylenodiaminotetraoctowy*) i EDDS (*kwasa etylenodiaminodibursztynowy*), w dawce 5 mmol · kg⁻¹. Wykonano także podobne testy z dwoma rodzajami komunalnych osadów ściekowych o różnym stopniu stabilizacji biochemicznej. W doświadczeniach badano osiem próbek osadów poflotacyjnych ze składowisk Żelazny Most oraz Wartowice oraz 2 próbki gleb zanieczyszczonych

z sąsiedztwa hut miedzi w Legnicy i Głogowie. Całkowite zawartości badanych pierwiastków w osadach wynosiły: Cu: 1300–3800 mg · kg⁻¹, Pb: 80–300 mg · kg⁻¹ oraz Zn: 23–35 mg · kg⁻¹. Zawartości tych pierwiastków w glebach wynosiły: Cu: 1270–1500 mg · kg⁻¹, Pb: 304–336 mg · kg⁻¹ oraz Zn: 80–89 mg · kg⁻¹. Skuteczność ekstrakcji metali z osadów i gleb była zróżnicowana. Najsilniej rozpuszczająco na Cu działał EDTA, który ekstrahował 7–39 % całkowitej zawartości Cu oraz 2–18 % Pb. Skuteczność rozpuszczania Cu z gleb i osadów pozostałymi odczynnikami układała się w szereg: kwas cytrynowy (dla osadów) > glicyna > histydyna > EDDS > kwas cytrynowy (dla gleb) > kwas winowy > chlorek amonowy > azotan amonowy. Dla pozostałych dwóch metali szereg ten nie był identyczny. Mimo znacznej zawartości łatwo rozpuszczalnej substancji organicznej w niestabilizowanych osadach ściekowych, w testach z osadami ściekowymi nie stwierdzono znaczącego wzrostu rozpuszczalności Cu i Pb. Praktyczne znaczenie rozpuszczania metali wskutek chelatowania może być ograniczone ze względu na silne sorbowanie połączeń metali przez szumifikowaną substancję organiczną.

Słowa kluczowe: gleba, metale ciężkie, rozpuszczalność, chelaty, kompleksowanie, osady ściekowe