# EFFECT OF INDUSTRIAL-COMMUNAL SEWAGE SLUDGE TREATMENT ON THE Cd AND Cr FRACTIONS OF SOIL AND ON PLANT UPTAKE

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The Cd and Cr fractions (mobile: 1M $NH_4NO_3$-soluble, mobilisable: ammonium acetate + EDTA-soluble, "total": cc. $HNO_3$ + cc. $H_2O_2$-soluble) of the experimental soils and the Cd and Cr contents of spring barley grain and straw were examined in a pot experiment to investigate the effect of applying industrial-communal sewage sludge with metal contents exceeding the permissible limit. The four experimental soils were acidic sand, calcareous sandy, calcareous chernozem loamy and brown forest soil or acidic loam. The sludge was applied at rates of 0, 2.5, 5, 10 and 20 g sludge D.M. /kg air-dry soil in four replications. The mobile fraction of both elements changed only on sandy soils significantly. Regression analysis on the soil Cr and Cd fractions and the barley grain and straw element contents revealed that in the case of Cd the soil mobile fraction is the most suitable to predict the plant concentration. The changes in plant Cr concentrations were not coherent, thus the regression with soil Cr concentration did not give reliable results. Only a negligible proportion of the elements added with the sludge appeared in mobile form in the soil. This ratio is smaller in the case of Cr where only 0.02 % of the sludge Cr content can be found in the soil mobile Cr fraction. Since Cd is more mobile element therefore, 2.5 % of the added Cd can be found in the mobile fraction. Our results indicated that in case of sewage sludge application on field it is important to consider not only the element contents, but also other parameters (pH, texture, organic matter), as these may influence the dissolution of contaminants. The Cd and Cr content of the investigated sludge may cause adverse effects mainly on light textured soils.

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## Introduction

The sewage sludge volume is supposed to be grown up very high with increasing drainage and capacity of wastewater treatment. In Hungary, the amount of sewage sludge dry matter is 150-160 thousand tons per year at present. This value is supposed to be increased to 350-400 thousand tons by 2015 .[1]

The most cost effective treatment of sludge is agricultural utilisation.[2] However, the sewage sludge may contain high concentrations of potentially toxic elements in it. The contamination of soils with these elements continues to be a matter of great concern because of their persistence in soils and increased uptake by crop plants even many years after sludge applications.[3,4,5,6]

The sorption of these elements in soil depends on the chemical properties of the elements and the composition of the soil. In the three characteristic elements fractions in soil – total (cc. $HNO_3$ + $H_2O_2$ extracted), mobilisable ($NH_4OAc$ + EDTA soluble) and mobile ($NH_4NO_3$ soluble) – the last two have important role from the environment protection point of view. These fractions significantly influence the plant element uptake (thus the contamination in food chain) and the element concentration of soil irrigated water and groundwater despite its concentration may be only negligible as compared to the total fraction. The ratio between the differently soluble fractions depends on the soil and element properties either.[7,8,9,10,11]

The effect of sewage sludge loads on soil composition and plant element uptake was investigated by various authors[12,13,14] in a pot experiment. In these experiments the relations between the total and mobile element contents and barley element composition were investigated. This work focuses on the effects of industrial-communal sewage sludge on mobile Cr, Cd fractions, mobility in soil and its uptake by barley.

## Methods

### Experimental design

A pot experiment was set up for determining the effect of sewage sludge loads on soils and plants.[13,14] Four soils were used from the plough layer (0-20 cm) of the field experimental stations of Institute for Soil Sciences and Agricultural Chemistry. Parameters of the investigated soils are shown in Table 1. The four experimental soils were brown forest soil with alternating thin layers of clay, or acidic sand (Nyírlugos), calcareous sandy soil (Őrbottyán), calcareous chernozem loamy soil (Nagyhörcsök) and brown forest soil, or acidic loam (Gyöngyös/Tasspuszta).

The soils were treated with industrial-communal sludge. The pre-treatment of the sludge compost was as follows: the compost was dried then sieved 3 times for homogenisation.

The mixture of air-dried soil (< 5 mm particle size) and the air-dry compost was dispensed into 10-liter pots (10 kg soil). To take soil water supply under control the pots were in field situation, but under a shed. Soils were irrigated by deionised water as per the plants need. Applied sewage sludge loads were the following: 0, 5, 10, 20 g sludge D.M./kg air-dry soil. Number of treatments was: 4 soil x 4 load level = 16, with 4 replications.

**Table 1.** Selected properties of soil samples[12]

| Parameter | Soils | | | |
|---|---|---|---|---|
| | Acid sand | Calcareous sand | Calcareous loam | Acid loam |
| pH ($H_2O$) | 5.4-5.8 | 7.8-8.3 | 7.8-8.1 | 6.6-6.8 |
| pH (KCl) | 3.9-4.8 | 7.3-7.6 | 7.5-7.6 | 5.8-6.3 |
| $CaCO_3$ % | - | 10-13 | 8-10 | - |
| Clay (<0.002 mm, %) | 3-4 | 4-5 | 20-24 | 40-45 |
| Organic matter % | 0.5-0.8 | 0.6-0.8 | 2.6-3.0 | 3.0-3.5 |

The soil and sludge mixing was followed by 1-month incubation, then spring barley (30 seeds per pot on 3th May 1999) and after its harvest and removal of the roots (26th July 1999) peas (7 seeds per pot) were sown. The barley had 3 months growing period. Soil sampling was taken after pea harvest on the 20th October. Composite soil samples consisted of 20 cores/pot and all above-ground total plant mass was used for analysis and yield assessment. Before sampling of the soil it was sieved so as the plant residues would be removed. In this paper only the results about barley are shown.

#### Chemical analysis

Determination of plant, soil and sewage sludge pseudo total element concentrations was carried out with ICP-AES method after microwave teflon bomb digestion with cc. $HNO_3 + H_2O_2$.[15]

From the soil samples the pseudo total, the mobilisable: 0.5 M $NH_4$-acetate + 0.02 M EDTA extractable[16] and the mobile: 1 M $NH_4NO_3$ extractable[17] element concentrations were measured by ICP-AES method.

Measurement of pH was carried out in 1:2.5 soil 1 M KCl solution after 24 hours of mixing. The soil organic matter content was measured by oxidation with $K_2Cr_2O_7$ according to the method of Tyurin.[18] The total N content was measured after cc. $H_2SO_4 + H_2O_2$ digestion according to the modified method of Kjeldahl.[19] The $CaCO_3$ content of the soil was determined according to.[20]

## Results and discussion

The $NH_4NO_3$-soluble mobile fraction showed significant changes in function of sludge loads only in the case of B, Cd, Co, Cr, Cu, Mn, Mo, Ni, Sr and Zn. The results concerning the Cu, Zn, Mn, Ni and Co elements are discussed by us.[21] Thus in this work the results of only the Cd and Cr elements are presented.

The Cr and Cd concentration limit values for agricultural application of sewage sludge[22] were exceeded 1.8 and 3.5 times respectively in the sludge treatments. Thus the sludge applied in this experiment could not be used legally in the field. Table 2 shows the element loads.

For the understanding of the changes in $NH_4NO_3$-soluble mobile fraction it is necessary to investigate the changes in soil properties caused by sludge application. The sewage sludge influenced significantly the pH only in the case of the two sandy soils. The pH of acidic sandy soil increased from 5.9 to 6.2 (Table 3). While the pH of calcareous sand decreased from 7.8 to 7.4. The pH increasing effect of sludge has also been observed in the experiment of earlier workers.[23]

**Table 2.** Cd and Cr content of sludge loads (kg $ha^{-1}$)

| Element | Limit value* | Load level (g sludge D.M./kg soil) | | | |
|---|---|---|---|---|---|
| | | 2.5 | 5 | 10 | 20 |
| Cr | 10 | 13 | 26 | 53 | 106 |
| Cd | 0.15 | 0.26 | 0.53 | 1.05 | 2.1 |

*limit values for sewage sludge agricultural application, total fraction [22] (kg/ha/year)

A contradictory result like in the case of calcareous soils in this experiment was revealed by other workers[24] that sludge with higher pH than soil may decrease soil pH by their high Al and Fe content. The $CaCO_3$ concentration was affected by sludge only on calcareous sandy soil. At the same time the sludge increased the organic matter content in the sandy soils from 0.8 to 1 %. The significant increment in organic matter can be observed on the calcareous loamy soil also: the concentration reached 3.3 % in the 20 g sludge/kg soil compared to the control. The changes in organic matter content may have an influence on pH also by their functional groups.[25]

**Table 3.** Effect of sludge loads on soil properties

| Soils | Load level (g sludge D.M./kg soil) | | | | | $LSD_{5\%}$ | Mean |
|---|---|---|---|---|---|---|---|
| | 0 | 2.5 | 5 | 10 | 20 | | |
| | pH ($H_2O$) | | | | | | |
| Acid sand | 5.9 | 5.8 | 5.8 | 6.1 | 6.2 | | 5.9 |
| Calcareous sand | 7.8 | 7.8 | 7.8 | 7.7 | 7.4 | 0.2 | 7.7 |
| Calcareous loam | 7.8 | 7.8 | 7.8 | 7.7 | 7.7 | | 7.8 |
| Acid loam | 6.9 | 6.8 | 6.9 | 6.9 | 6.9 | | 6.9 |
| | $CaCO_3$ % | | | | | | |
| Acid sand | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | | 0.0 |
| Calcareous sand | 13.1 | 13.3 | 12.7 | 13.1 | 12.5 | 0.6 | 12.9 |
| Calcareous loam | 8.3 | 8.5 | 8.4 | 8.7 | 8.5 | | 8.5 |
| Acid loam | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | | 0.0 |
| | Organic matter % | | | | | | |
| Acid sand | 0.8 | 0.8 | 0.8 | 0.9 | 1.0 | | 0.9 |
| Calcareous sand | 0.8 | 0.8 | 0.8 | 0.9 | 1.0 | 0.2 | 0.9 |
| Calcareous loam | 2.9 | 3.0 | 3.0 | 3.2 | 3.3 | | 3.1 |
| Acid loam | 3.6 | 3.5 | 3.6 | 3.7 | 3.7 | | 3.6 |

The total and mobile Cd and Cr fractions increased significantly on each investigated soil (Table 4 and 5). The mobile fractions of the two elements showed an increment only on the sandy soils. On the loamy soils no concentration change can be observed, however the control concentrations of mobile Cd and Cr are in the same magnitude of order in each soil. In the case of both elements the mobile concentrations are the highest on acidic sand.

The mobile Cd concentration changes did not follow the volume of sludge loads (Table 3). In the control treatment the soils Cd concentration was statistically the same and on sludge treated soils only the acidic sand differed significantly from the other soils. The increment in mobile

Cd concentration was significant only on the sandy soils. On acidic sand the mobile Cd concentration increased significantly after the addition of the 2.5 g sludge/kg soil load but the higher loads did not modify this value significantly. A potential explanation of this phenomenon is the increase of pH that could reduce the solubility of this element.[26] The $NH_4NO_3$ soluble Cr concentration was statistically the same on each control soil. The sludge loads increased this value on the sandy soils. However, the Cr(III) is one of the least mobile element in soil. Thus the increment may probably refer to the increased amount of Cr(VI) that can be mobile in the present conditions.[12] At the same time the lack of the toxicity of the sludge to barley is against this theory. Based on the above the Cd and Cr content of the investigated sludge may cause adverse effects principally on light textured soils but in this experiment these could not be revealed because the barley yield increased significantly as a result of sludge treatments.[12]

**Table 4.** Effect of industrial-communal sewage sludge loads on soil Cd fractions and barley grain and straw Cd concentration (total, mobilisable and plant concentrations are based on the data of Kádár and Morvai[12])

| **Soils** | **Load level (g sludge D.M./kg soil)** | | | | | **LSD$_{5\%}$** | **Mean** |
|---|---|---|---|---|---|---|---|
| | 0 | 2.5 | 5 | 10 | 20 | | |
| | **Cd load, µg Cd kg$^{-1}$ soil** | | | | | | |
| | 0 | 88 | 175 | 350 | 700 | | |
| Mobile ($NH_4NO_3$ soluble) Cd concentration of soil (µg kg$^{-1}$) | | | | | | | |
| Acid sand | 4 | 19 | 20 | 15 | 18 | 6 | 15 |
| Calcareous sand | 1 | 4 | 1 | 8 | 8 | 6 | 4 |
| Calcareous loam | 2 | 1 | 3 | 3 | 3 | n.s. | 2 |
| Acid loam | 7 | 4 | 4 | 4 | 6 | n.s. | 5 |
| LSD$_{5\%}$ | 12 | | | | | | |
| Total (cc. $HNO_3$+$H_2O_2$ soluble) Cd concentration of soil (µg kg$^{-1}$) | | | | | | | |
| Acid sand | 68 | 105 | 153 | 210 | 355 | 96 | 178 |
| Calcareous sand | 134 | 169 | 182 | 310 | 569 | 178 | 273 |
| Calcareous loam | 234 | 369 | 410 | 440 | 659 | 100 | 419 |
| Acid loam | 510 | 564 | 706 | 804 | 1061 | 102 | 729 |
| LSD$_{5\%}$ | 95 | | | | | | |
| Mobilisable ($NH_4OAc$ + EDTA soluble) Cd concentration of soil (µg kg$^{-1}$) | | | | | | | |
| Acid sand | 26 | 60 | 67 | 158 | 237 | 59 | 110 |
| Calcareous sand | 80 | 115 | 109 | 236 | 394 | 81 | 187 |
| Calcareous loam | 176 | 187 | 214 | 330 | 447 | 51 | 271 |
| Acid loam | 305 | 364 | 424 | 496 | 508 | 64 | 419 |
| LSD$_{5\%}$ | 20 | | | | | | |
| Barley grain Cd concentration (µg kg$^{-1}$) | | | | | | | |
| Acid sand | 38 | 55 | 65 | 66 | 82 | 22 | 61 |
| Calcareous sand | 18 | 31 | 14 | 24 | 45 | 16 | 26 |
| Calcareous loam | 18 | 23 | 21 | 24 | 44 | 16 | 26 |
| Acid loam | 38 | 34 | 37 | 43 | 46 | n.s. | 40 |
| LSD$_{5\%}$ | 14 | | | | | | |
| Barley straw Cd concentration (mg kg$^{-1}$) | | | | | | | |
| Acid sand | 0.16 | 0.26 | 0.33 | 0.37 | 0.42 | 0.05 | 0.31 |
| Calcareous sand | 0.15 | 0.17 | 0.17 | 0.23 | 0.30 | 0.05 | 0.20 |
| Calcareous loam | 0.15 | 0.14 | 0.13 | 0.17 | 0.22 | 0.04 | 0.16 |
| Acid loam | 0.24 | 0.26 | 0.263 | 0.28 | 0.29 | n.s. | 0.27 |
| LSD$_{5\%}$ | 0.02 | | | | | | |

n.s.: not significant

**Table 5.** Effect of industrial-communal sewage sludge loads on soil Cr fractions and barley grain and straw Cr concentration (total, mobilisable and plant concentrations are based on the data of Kádár and Morvai[12])

| Soils | Load level (g sludge D.M. /kg soil) | | | | | LSD$_{5\%}$ | Mean |
|---|---|---|---|---|---|---|---|
| | 0 | 2.5 | 5 | 10 | 20 | | |
| | Cr load, µg Cr/kg soil | | | | | | |
| | 0 | 4405 | 8810 | 17620 | 35240 | | |
| Mobile ($NH_4NO_3$ soluble) Cr concentration of soil (µg/kg) | | | | | | | |
| Acid sand | 5 | 9 | 12 | 17 | 18 | 5 | 12 |
| Calcareous sand | 5 | 6 | 5 | 8 | 11 | 2 | 7 |
| Calcareous loam | 8 | 7 | 5 | 7 | 6 | n.s. | 7 |
| Acid loam | 5 | 9 | 5 | 5 | 5 | n.s. | 6 |
| LSD$_{5\%}$ | 5 | | | | | | |
| Total (cc. $HNO_3$ + $H_2O_2$ soluble) Cr concentration of soil (mg/kg) | | | | | | | |
| Acid sand | 11 | 13 | 15 | 21 | 27 | 5 | 17 |
| Calcareous sand | 13 | 16 | 16 | 22 | 35 | 11 | 20 |
| Calcareous loam | 36 | 42 | 47 | 43 | 58 | 6 | 45 |
| Acid loam | 50 | 52 | 65 | 66 | 77 | 5 | 62 |
| LSD$_{5\%}$ | 7 | | | | | | |
| Mobilisable ($NH_4OAc$ + EDTA soluble) Cr concentration of soil (mg/kg) | | | | | | | |
| Acid sand | 0.08 | 0.16 | 0.22 | 0.55 | 0.92 | 0.18 | 0.39 |
| Calcareous sand | 0.09 | 0.11 | 0.10 | 0.18 | 0.27 | 0.06 | 0.15 |
| Calcareous loam | 0.09 | 0.07 | 0.10 | 0.10 | 0.15 | 0.03 | 0.10 |
| Acid loam | 0.13 | 0.15 | 0.23 | 0.23 | 0.22 | 0.07 | 0.19 |
| LSD$_{5\%}$ | 0.09 | | | | | | |
| Barley grain Cr concentration (mg/kg) | | | | | | | |
| Acid sand | 0.26 | 0.29 | 0.22 | 0.24 | 0.21 | n.s. | 0.24 |
| Calcareous sand | 0.35 | 0.26 | 0.34 | 0.21 | 0.27 | n.s. | 0.28 |
| Calcareous loam | 0.24 | 0.22 | 0.23 | 0.17 | 0.16 | n.s. | 0.21 |
| Acid loam | 0.17 | 0.15 | 0.17 | 0.18 | 0.21 | n.s. | 0.17 |
| LSD$_{5\%}$ | 0.08 | | | | | | |
| Barley straw Cr concentration (mg/kg) | | | | | | | |
| Acid sand | 1.26 | 0.77 | 1.09 | 1.66 | 0.73 | n.s. | 1.10 |
| Calcareous sand | 0.91 | 0.77 | 1.09 | 1.19 | 1.72 | 0.51 | 1.14 |
| Calcareous loam | 1.37 | 0.75 | 0.80 | 0.63 | 0.85 | 0.40 | 0.88 |
| Acid loam | 1.06 | 0.80 | 1.06 | 0.76 | 0.84 | n.s. | 0.91 |
| LSD$_{5\%}$ | 0.22 | | | | | | |

n.s.: not significant

The regression analysis between soil element fractions and plant element concentrations involving the four soils and the five sludge loads resulted that in the case of Cd the $NH_4NO_3$ soluble mobile fraction is the best predictor for the plant Cd concentration. The straw Cr concentration could not be determined from the soil Cr fractions in this experiment. In the case of grain Cr concentration the total Cr fraction proved to be the best predictor. However, the grain Cr concentration did not change significantly and the equation shows negative correlation between the plant and soil Cr concentration. Thus, this connection indicates only that the total element fraction is not suitable to predict plant element content. The regression equations are the following:

$$[Cr]_{grain} = -0.002\,[Cr]_{total} + 0.3 \qquad R^2 = 0.47^{**} \qquad (1)$$

$$[Cd]_{grain} = 2.51\,[Cd]_{mobile} + 0.02 \qquad R^2 = 0.73^{***} \qquad (2)$$

$$[Cd]_{straw} = 9.24\,[Cd]_{mobile} + 0.17 \quad R^2 = 0.49^{***} \quad (3)$$

These results reinforce the results of previous studies that showed the $NH_4NO_3$ soluble element fraction as a suitable predictor of plant element concentrations.[27,28] This fact is in favour of this extraction method despite the controversies.[29]

Comparing the concentrations in the mobile and total fraction it can be seen that only the trace of the elements added by sewage sludge appears in the mobile fraction. The ratio of two differently soluble element fractions can be defined as mobility. The mobility of an element in soil shows how soluble is the element in the soil solution [30]. The mobility of elements in our experiment was defined as a ratio of $NH_4NO_3$ extractable / pseudo total element content. The increase this value the more mobile is the given element. The mobility of an element may have a relation with the element uptake by plants. But it is necessary to consider that the plants metal content depends on plant species, physico-chemical properties of the soil, weather conditions, fertilisation, etc. Therefore plant responses to soil contamination should be always studied in a particular soil – plant system.[31] The calculation of mobility is:

$$M = 100\frac{\text{mobile}}{\text{pseudo total}} \quad (4)$$

where M is the mobility of a given element, “mobile” is the $NH_4NO_3$ extractable concentration and „pseudo total” is the result of extraction with cc. $HNO_3 + H_2O_2$ (mg kg$^{-1}$).

**Table 6.** Effect of industrial-communal sewage sludge loads on Cd and Cr mobility (Mobility = [mobile]/ [total]*100) in soil

| **Soils** | **Load level (g sludge D.M./kg soil)** | | | | | **LSD$_5$ %** | **Mean** |
|---|---|---|---|---|---|---|---|
| | 0 | 2.5 | 5 | 10 | 20 | | |
| Mobility of Cd | | | | | | | |
| Acid sand | 6.1 | 17.3 | 14.6 | 7.3 | 6.7 | 4.5 | 10.4 |
| Calcareous sand | 0.76 | 2.46 | 0.67 | 2.62 | 1.61 | n.s. | 1.62 |
| Calcareous loam | 0.88 | 0.26 | 0.63 | 0.78 | 0.46 | n.s. | 0.60 |
| Acid loam | 1.51 | 0.78 | 0.58 | 0.49 | 0.56 | n.s. | 0.78 |
| LSD$_{5\%}$ | 5.04 | | | | | | |
| Mobility of Cr | | | | | | | |
| Acid sand | 0.049 | 0.073 | 0.090 | 0.090 | 0.089 | n.s. | 0.078 |
| Calcareous sand | 0.037 | 0.040 | 0.031 | 0.037 | 0.035 | n.s. | 0.036 |
| Calcareous loam | 0.022 | 0.016 | 0.011 | 0.016 | 0.011 | n.s. | 0.015 |
| Acid loam | 0.011 | 0.018 | 0.008 | 0.008 | 0.007 | n.s. | 0.010 |
| LSD$_{5\%}$ | 0.024 | | | | | | |

n.s.: not significant

Table 6 shows, that mobility of Cd is at least one order of magnitude higher than that of Cr, which is in good agreement with earlier findings also.[32] The mobility has significantly maximum values in acidic sandy soil in the case of both elements. In most cases the mobility did not change in function of sludge loads. The only significant differences between treatments can be seen in the case of Cd mobility on acidic sand showing the highest mobility value at the 2.5 g sludge/kg soil load. Regarding the regression between Cd and Cr mobility and barley grain and straw concentrations only the mobility of Cd showed significant regression with the grain Cd concentration but the $R^2$ value (0.40) was below that seen in the case of Eqn. 2. Based on the above facts, the mobile fraction itself proved to be a better indicator of plant Cd concentration than mobility.

Investigating the relation between the mobile fraction and the total Cd and Cr amount of the sludge loads it can be seen that in function of the increasing sludge loads the ratio of mobile fraction to the added element amount became smaller. Thus, the higher the concentration in the soil the smaller the mobile fraction compared to the total. This ratio is smaller in the case of Cr where only 0.02 % of the sludge Cr content can be found in the soil mobile Cr fraction. Since Cd is more mobile element in that case this ratio is 2.5 %.

The above results indicated that in the case of sewage sludge application on field it is important to consider not only the element contents, but also other parameters (pH, texture, organic matter), as these may influence the dissolution of contaminants.

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