

# The comparison of extraction methods for evaluating some heavy metals in polluted soils

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## ABSTRACT

The extractability of Cd, Co, Cr, Ni and Pb was evaluated using ammonium chloride, calcium chloride, strontium chloride and DTPA extractants in this research. The eight surface soils were used to assess plant available metals with different extraction methods. The amounts of metal extracted were related to pseudototal contents, determined after microwave digestion using  $\text{HNO}_3$ . Quantification of dissolved metals was used by ICP-MS matrix-matched standards. The obtained results indicated a high variability of metal extraction depending on extraction procedure, source of pollution, and nature of the soil. The results showed that the extractability for calcareous soils was best determined by DTPA. In comparison of chloride salts, a higher efficiency of extraction with ammonium chloride for these soils was found.

**Keywords:** ICP-MS; heavy metals; polluted soils; pollution; extraction methods; mobility

Many soils in developing and industrialized countries are affected by acid deposition, mine waste disposal, utilization of organic refuses such as sewage sludge, industrialization and urbanization that could be inputs of pollutants and especially heavy metals to the soil. The heavy metals may be retained by soil components, may exchange or precipitate or coprecipitate as sulfides, carbonate and/or Fe or Mn oxides or hydroxides. In arid zone soils, the presence of carbonate minerals effectively immobilizes heavy metals by providing an absorbing surface and by buffering pH at high values where precipitation takes place.

The chemical forms of heavy metals in the solid phase can strongly influence their behavior, such as their mobility, toxicity, bioavailability and chemical interactions, since metal speciation has deterministic control over physical, chemical, and biological processes that lead to transport and transformation of heavy metals among environmental compartments (air, water, biota, soil/sediment) in complex ecosystems (Allen et al. 1995).

Mobility of heavy metals relates to their capacity to pass from one soil compartment to another where the elements is bound more or less energetically, the ultimate mobile compartment being the soil solution which determines the bioavailability of heavy metals. Extraction of heavy metals by unbuffered salt solutions ( $\text{CaCl}_2$ ,  $\text{NH}_4\text{Cl}$ , etc.) is a rapid and simple way to evaluate their phytoavailability (Beckett 1989). However, in some cases, salt solutions do not reflect the plant available metals (Gupta and Atten 1993), so that must be used other extractants based on DTPA or hydroxylamine that are more predictive of phytoavailability. This research was

designed to determine the extractability of some heavy metals in polluted soils.

## MATERIAL AND METHODS

In this research, eight surface soils (0–25 cm) were selected on the basis of the diversity of pollution sources (industrial and agricultural activities). The soils were classified as non-calcareous (soils 1 to 3) and calcareous (soils 4 to 8). The soil samples were taken from the Bursa plain, Turkey. Soil 1 was polluted by acidic industrial wastewater. Soils 2 and 3 are typical sewage sludge amended soil. Soils 4 to 6 are sewage sludge amended calcareous soils irrigated by water with increasing level of Cd contamination (25, 85 and 210 mg/l). Soils 7 and 8 represented typical calcareous agricultural soils without anthropogenic pollution.

The following soil characteristics were evaluated: Particle size distribution determined by the hydrometer method (Gee and Bauder 1982), pH in a 1:2 soil:water ratio (McLean 1982), organic carbon (Nelson and Sommers 1982), calcium carbonate (Nelson 1982), electrical conductivity (SCS 1972) and CEC (Rhoades 1982). Pseudototal contents of metals were determined following microwave digestion using concentrated  $\text{HNO}_3$  and analysed by ICP-MS (Krishnamurti et al. 1994).

Four different extraction methods were used to establish the extractability and predict the phytoavailability of heavy metals for these soils (Table 1). Each extraction method was performed in duplicate in 50 ml polycarbonate centrifuge tubes. All the extracts were centrifuged for 10 min at 3000 rpm,

Table 1. Summary of the extraction methods used for soils

Extractant	Ratio (w/v)	Time (h)	Temperature (°C)	Pool	Reference
1M NH <sub>4</sub> Cl	1:6	16	20	neutral salt, soluble/exchange	Krishnamurti et al. (1995)
0.01M CaCl <sub>2</sub>	1:5	16	20	neutral salt, soluble/exchange	Whitten and Ritchie (1991)
0.01M SrCl <sub>2</sub>	1:7	2	20	neutral salt, soluble/exchange	Ahnstrom and Parker (1999)
0.005M DTPA	1:2	2	20	chelating extra available pool	Lindsay and Norvell (1978)

and the supernatant filtered into volumetric flasks containing 10 µg In/Rh/l. All the extracts and standard solution were acidified to 1% HNO<sub>3</sub>. Inductively coupled plasma mass spectrometry (ICP-MS) of Ultramass 700 Varian Model determinations were obtained with matrix-matched standards.

## RESULTS AND DISCUSSION

The physical and chemical properties of the studied soils were presented in Table 2. These soils showed a wide range of physical and chemical properties. Clay content of soils ranged from 5.1 to 35.0%. The silt and sand contents varied from 14.1 to 69.1% and from 17.2 to 80.4%, respectively. The pH values of soils ranged from 3.1 to 8.73. The low pH of soil 1 is due to acidic pollution from

industrial wastewater. Electrical conductivity values ranged from 0.29 to 3.40 dS/m. The highest EC value occurred in the soil 1 that was polluted by acidic wastewater. CEC varied from 9.2 to 32.5 cmol(+)/kg. Organic carbon content ranged from 0.54 to 2.46% with the highest level for the sewage sludge amended soils.

The results of extractable Cd, Co, Cr, Ni and Pb for the different extractants are presented in Table 3. The values are expressed as percentages of the pseudototal content in these soils.

### Cadmium

Nakhone and Young (1993) stated that the lability of Cd ranged from 3 to 102% of total Cd (HNO<sub>3</sub> extractable digest). Cd was the most extractable

Table 2. The chemical and physical properties of the experimental soils

Features	Non-calcareous			Calcareous				
	soil 1	soil 2	soil 3	soil 4	soil 5	soil 6	soil 7	soil 8
Sand (%)	29.1	60.4	80.4	18.1	17.2	18.4	33.4	32.2
Silt (%)	60.3	21.5	14.1	67.7	69.1	71.5	61.5	32.8
Clay (%)	10.6	18.1	5.5	14.2	13.7	10.1	5.0	35.0
pH (1:2)	3.1	6.43	6.54	8.09	8.16	7.95	7.83	8.73
EC (dS/m)	3.4	0.39	0.73	0.74	0.63	1.01	1.52	0.29
Total CaCO <sub>3</sub> (%)	2.0	3.6	3.7	52.1	50.5	49.9	37.3	43.6
Organic C (%)	1.15	0.54	0.71	2.07	1.97	2.46	1.11	0.63
CEC [cmol(+)/kg]	36.9	14.3	9.2	32.0	32.5	31.9	26.7	14.6
Total Cd (mg/kg)	14.0	7.0	215.0	76.0	100.0	191.0	4.8	3.4
Total Co (mg/kg)	8.4	6.0	22.0	11.0	10.3	11.1	7.5	7.8
Total Cr (mg/kg)	28.4	17.9	5.6	28.7	31.1	33.8	22.8	20.7
Total Ni (mg/kg)	17.5	8.4	43.1	37.5	38.2	56.2	30.2	27.4
Total Pb (mg/kg)	282.0	29.0	41.0	221	35.0	36.0	26.0	18.0

Table 3. Percentage of total metal content released by each extraction procedure

Metal	Extractant	Non-calcareous			Calcareous				
		soil 1	soil 2	soil 3	soil 4	soil 5	soil 6	soil 7	soil 8
Cd	NH <sub>4</sub> Cl	3.90	0.47a	1.94	11.7	15.6	23.6	2.28	0.63
	CaCl <sub>2</sub>	2.97a	0.10b	0.33	0.54	0.41	0.31	0.22	0.22a
	SrCl <sub>2</sub>	3.28a	0.20b	0.57a	0.91	1.46	2.65	3.30	0.13a
	DTPA	2.45	0.51a	0.57a	56.3	95.3	81.5	9.11	1.63
	<i>F</i> -anova	**	**	***	***	***	***	***	***
Co	NH <sub>4</sub> Cl	20.4a	0.31a	1.52	0.38a	0.33a	0.35a	0.40a	0.39
	CaCl <sub>2</sub>	22.0a	0.15a	1.05a	1.26	0.18a	0.15a	0.10	0.08a
	SrCl <sub>2</sub>	21.1a	0.78	0.82a	0.22a	0.22a	0.24a	0.26a	0.14a
	DTPA	19.0a	2.09	1.06a	8.21	9.37	7.56	5.49	6.47
	<i>F</i> -anova	ns	**	*	***	***	***	***	***
Cr	NH <sub>4</sub> Cl	0.93	1.20b	0.95a	3.28	1.39	1.91	2.09	5.93
	CaCl <sub>2</sub>	0.02a	0.02a	0.04	0.09a	0.05a	0.05a	0.05a	0.03a
	SrCl <sub>2</sub>	1.53	0.81b	3.65	1.06	0.83	1.35	1.45	1.28
	DTPA	0.08	0.07a	0.81a	0.04a	0.09a	0.07a	0.05a	0.03a
	<i>F</i> -anova	**	**	***	***	***	**	**	***
Ni	NH <sub>4</sub> Cl	11.4	1.31	3.04	0.61a	0.43a	0.40	0.57	0.30
	CaCl <sub>2</sub>	7.91a	0.04	1.40a	0.71a	0.10b	0.06a	0.10a	0.08a
	SrCl <sub>2</sub>	7.15a	0.85	0.84b	0.26	0.20ab	0.21a	0.26a	0.02a
	DTPA	4.00	9.30	1.21ab	1.90	1.98	1.28	3.07	1.84
	<i>F</i> -anova	***	***	**	**	*	**	**	**
Pb	NH <sub>4</sub> Cl	0.49	0.48	0.16a	0.04a	0.31a	0.05a	0.04a	0.03a
	CaCl <sub>2</sub>	0.04a	0.05a	0.07	3.67	0.10	0.04a	0.04a	0.05a
	SrCl <sub>2</sub>	0.02a	0.03a	0.24ab	0.22a	0.31a	0.34	0.14a	0.21
	DTPA	0.02a	5.14	0.33b	9.37	8.47	8.41	6.11	10.50
	<i>F</i> -anova	**	***	*	***	***	***	***	***

\*, \*\*, \*\*\* indicate significant differences at  $p = 0.05, 0.01, 0.001$ , respectively

Figures within vertical columns followed by the same letter are not statistically different ( $p = 0.05$ )

element with DTPA from the contaminated calcareous soils, which corresponds to the findings of Lindsay and Norvell (1978). Young et al. (2000) in comparison of labile Cd with Cd dissolved in four extractants showed that weak extractants like CaCl<sub>2</sub> and KNO<sub>3</sub> consistently underestimated labile Cd. In this research, very high recovery of total Cd (57 to 96%) was determined with DTPA for calcareous soils, especially for soils 4 to 6, where Cd pollution was due to irrigation with polluted water and organic amendments. Mahler (1988) stated that both native and sludge-derived Cd was mainly in the carbonate fraction in calcareous soils. It ap-

pears that high extraction percentages of Cd with DTPA are closely related to the chelating capacity of the Lindsay-Norvell solution while maintaining the soil pH at alkaline values to optimize cation extraction. It was observed that an increase of the exchangeable Cd fraction with increasing organic matter in the soil (from soil 4 to 6), which is a consistent result with He and Singh (1993).

The ammonium was the most efficient chloride salt for extracting Cd for the non-calcareous soils. This could be due to pH of the soil-NH<sub>4</sub>Cl suspension being similar to soil pH, and also to the similar order of magnitude of the stability con-

stant of Cd-chloride complex and Cd-organic acid complex for many of the low molecular-weight organic acids that exist in the rhizosphere (Smith and Martell 1977). The influence of the cation in the chloride salts was minimal in the very acidic conditions (soil 1).

Simple linear correlation analysis shows a highly significant relationship for Cd-NH<sub>4</sub>Cl and Cd-DTPA extracts with soil organic carbon content between Cd in the different extracts and selected soil properties (Table 4). The obtained results indicated that cadmium was the most easily extractable metal studied.

### Chromium

The most stable form in soil is Cr<sup>3+</sup> and at slightly acidic to alkaline pH values, ionic Cr<sup>3+</sup> species precipitate with precipitation being favoured by the presence of Fe (Cifuentes et al. 1996). This low solubility is consistent with the very low amounts (from 0.04 to 5.95% compared to total content in

soils) of Cr in the soluble-exchangeable pool. DTPA extracted less Cr than the chloride salts. Comparing these salts, we can observe that ammonium chloride and in lesser extent strontium chloride were more efficient extracting agent for Cr than calcium chloride. The ammonium chloride seems to be the best extractant for evaluating the extractability of Cr for calcareous soils.

### Cobalt

The different behaviors were observed for Co extraction depending on soil properties for the non-calcareous soils. The soil 1 induced a very high level of soluble Co compared with soil 3 with its near neutral pH. The influence of soil pH on soluble-exchangeable Co was very high for almost all the extractants and especially for the neutral chloride salts. The extractable Co pool was closely related to the electrical conductivity (i.e. salinity) of the soil. Extractability of Co was very low, except

Table 4. Pearson correlation coefficients between selected soil properties and different extracting methods

Metal	Extractant	Some soil properties						
		sand	silt	clay	pH	CaCO <sub>3</sub>	OC	CEC
Cd	CaCl <sub>2</sub>	ns	ns	ns	-0.87**	ns	ns	0.92**
	DTPA	ns	0.72*	ns	ns	0.75**	0.92**	ns
	NH <sub>4</sub> Cl	ns	0.72*	ns	ns	ns	0.95***	ns
	SrCl <sub>2</sub>	ns	ns	ns	ns	0.75*	ns	0.78**
Co	CaCl <sub>2</sub>	ns	ns	ns	-0.92**	ns	ns	0.92**
	DTPA	ns	ns	ns	ns	ns	ns	0.82*
	NH <sub>4</sub> Cl	ns	ns	ns	-0.91**	ns	ns	0.93**
	SrCl <sub>2</sub>	ns	ns	ns	-0.92**	ns	ns	0.92**
Cr	CaCl <sub>2</sub>	0.80*	ns	ns	ns	ns	ns	ns
	DTPA	0.80*	ns	ns	ns	ns	ns	ns
	NH <sub>4</sub> Cl	ns	0.82*	ns	ns	ns	ns	ns
	SrCl <sub>2</sub>	0.71	ns	ns	ns	ns	ns	ns
Ni	CaCl <sub>2</sub>	ns	ns	ns	-0.92**	ns	ns	0.91**
	DTPA	ns	ns	ns	ns	ns	ns	ns
	NH <sub>4</sub> Cl	ns	ns	ns	-0.96**	ns	ns	0.89**
	SrCl <sub>2</sub>	ns	ns	ns	-0.95***	ns	ns	0.91**
Pb	CaCl <sub>2</sub>	ns	ns	ns	ns	ns	ns	ns
	DTPA	ns	ns	ns	ns	ns	ns	ns
	NH <sub>4</sub> Cl	ns	ns	ns	ns	ns	ns	ns
	SrCl <sub>2</sub>	ns	ns	ns	ns	0.74*	ns	ns

\*, \*\*, \*\*\* indicate significant differences at  $p = 0.05, 0.01, 0.001$ , respectively, and ns = non significant

for the DTPA extraction from calcareous soils. This result could mainly reflect the low levels of pollution by this metal and also the high proportion of soil Co present in non-available pools compared with Co pseudo total concentration in soils.

### Lead

The soluble-exchangeable pool of lead was generally low due to the high percentage of insoluble forms in the soils. DTPA seems to be a better extractant for the majority of the soils, probably due to its capacity to dissolve Pb precipitates (Schalscha et al. 1980). Shuman (1988) stated that addition of organic matter to soil could decrease Pb lability probably by increasing CEC. Addition of soluble ligands in organic matter could decrease adsorption and increase leaching. Only for soil 1, which is very acidic, ammonium chloride was more efficient than DTPA. These results are consistent with the findings of Shuman (1988) who reported on increased mobilization of Pb in soil columns with hydrochloric acid but not with sulfuric acid. The correlation of soil properties with Pb extractability was also low, which is consistent with the low mobility of this metal. Williams et al. (1987) found that Pb availability to Swiss chard was unaffected by soil pH. Lead extractable by  $\text{SrCl}_2$  was slightly correlated with total and active calcium carbonate in the soils. Cabral and Lefebvre (1988) found that soils contaminated with Pb showed an accumulation of Pb in the sorbed/carbonate phase.

### Nickel

DTPA extracted more Ni than the chloride salts in calcareous soils. However, for non-calcareous soils, ammonium chloride extracted more Ni from soils 1 and 3. Li and Shuman (1996) reported that neutral salts such as  $\text{Mg}(\text{NO}_3)_2$  were equivalent to DTPA in estimating the extractability of Ni from polluted soils. A strong dependence of the amount of Ni extracted by chloride salts on the pH and salinity of soils (Table 4) was observed. Thus, with decreasing soil pH and increasing salinity, higher metal extraction percentages were obtained with these salts. There were significant differences between calcium and strontium chloride in the amount of Ni extracted, probably due to ionic radii. The efficiency of DTPA in extracting Ni was not affected by any soil property. A high percentage of extractable Ni occurred in soil 1. This soil had been polluted with sulfidic sediments deposited by wastewater flood with a high impact of soil acidity on nickel availability according with the findings of Adamo et al. (1996).

## CONCLUSION

Generally, the extractability of heavy metals is best determined with DTPA for calcareous soils. This may reflect the chelating capacity of the Lindsay-Norvell solution while maintaining soil pH at alkaline values to optimize cation extraction. For the non-calcareous soils, smaller differences were observed between extractants although ammonium chloride was the most effective extractant under acid conditions for heavy metals. The influence of soil pH and electrical conductivity on soluble-exchangeable heavy metals was noticeable for Cd, Co and Ni for all the extractants and especially for chloride salts.

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## ABSTRAKT

### Porovnání extrakčních metod pro hodnocení vybraných těžkých kovů ve znečištěných půdách

Byla hodnocena extrahovatelnost Cd, Co, Cr, Ni a Pb z půd roztokem chloridu amonného, chloridu vápenatého, chloridu strontnatého a DTPA. Pro hodnocení rostlinou přijatelného podílu kovů v půdách jednotlivými extraktanty bylo vybráno osm vzorků kontaminovaných půd. Extrahovatelné obsahy prvků byly vztaženy k pseudototálnímu obsahu prvků stanovenému po mikrovlnné digesti vzorků v HNO<sub>3</sub>. Pro kvantifikaci jednotlivých prvků v roztocích bylo použito metody ICP-MS s kalibrací pomocí standardů s modifikovanou maticí vzorku. Výsledky naznačily významnou variabilitu extrahovatelnosti prvků v závislosti na metodě extrakce, zdroji kontaminace a původu analyzované půdy a ukázaly nejlepší extrahovatelnost půd s vysokým obsahem vápníku pomocí roztoku DTPA. Při porovnání chloridových solí byla u těchto půd zaznamenána nejvyšší účinnost extrakce v případě roztoku chloridu amonného.

**Klíčová slova:** ICP-MS; těžké kovy; znečištěné půdy; kontaminace; extrakční metody; mobilita

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