The effects of chemical remediation treatments on the extractability and speciation of cadmium and lead in contaminated soils

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Abstract

Two rural soils contaminated by cadmium (Cd) and lead (Pb) were used to evaluate the effect of different chemical treatments on changes in speciation and extractability of Cd and Pb, and in phytoavailability to wheat. Triplicates of seven chemical treatments were tested to compare and evaluate the remediation techniques for contaminated soils using pot experiments. Treatments applied were calcium carbonate, a high quantity of phosphate salt, hog composts, iron oxide, manganese oxide, zeolite, and unamended control. Wheat (*Triticum aestivum*) was planted in the different amended soils for a further one month to evaluate the effectiveness of treatments on uptake of Cd and Pb by the wheat shoots. Results indicated that addition of calcium carbonate, manganese oxide, or zeolite reduces the extractability of Cd or Pb in both soils, and significantly reduce the uptake of Cd and Pb by wheat shoots. Changes in the extractability and metal sequential fractionations indicate that the exchangeable (or available) form of Cd and Pb in two soils can be transformed into unavailable forms after these amendments. © 2000 Elsevier Science Ltd. All rights reserved.

1. Introduction

Heavy metal contamination in the natural environment is a major problem for human health and environmental quality (Purves, 1985; Chen et al., 1996; Ma and Rao, 1997). Geochemical forms of heavy metals existing in the soils affect their solubility, which may directly influence their phytoavailability (Xian, 1987). In last decade, many researchers have attempted to assess phytoavailability of heavy metals in contaminated soils using metal sequential extraction methods (Xian, 1987, 1989). Most of the heavy metals are persistent in soil because of their immobile nature. Cadmium is known as more mobile and soluble than many other metals in soils, but Pb is well known to be relatively immobile and unavailable for plant uptake. Lead is a concern for human health when deposited on plant surfaces (Adriano,

1986). When agricultural soils are contaminated by heavy metals discharged from chemical plants, plant foliage is damaged and both growth rate and crop production are also retarded (Adriano, 1986). The effect of ionic strength and pH on the sorption of Cd and Pb is widely variable for different soils (Naidu et al., 1994). Higher concentrations of heavy metal soils may increase uptake of these elements by crops and potentially affect human health via food chains (Alloway, 1990). Cd and Pb may cause serious problems through food chains (Jackson and Alloway, 1992).

About 100 ha of rural soils contaminated with Cd and Pb were identified by the Environmental Protection Administration of Taiwan (EPA-Taiwan) in 1988. The concentration of Cd in brown rice growing at contaminated sites was higher than the critical health concentration of Cd (0.5 mg/kg dry weight) issued by Department of Health of Taiwan (Chen, 1991; Chen et al., 1996). The mean total concentrations of Cd in brown rice and soils in two of these contaminated sites are 1.5–3.0 mg/kg and 4.7–378 mg/kg. The mean total concentrations of Pb in

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brown rice and soils are 1.1–8.4 mg/kg and 25.8–3140 mg/kg, respectively (Chen, 1991; Chen et al., 1996). The contamination of Cd and Pb in other rural soils of Taiwan was also caused by the waste water discharged from the chemical plants in the industry park.

Many field and pot experiments on soil remediation techniques have been conducted at these two polluted sites (Chen et al., 1994; Lee and Chen, 1994; Wang et al., 1994; Chen and Lee, 1997). The soil remediation techniques include (1) chemical stabilization method to reduce the solubility of heavy metals by adding some non-toxic materials into the soils, (2) removal of polluted surface soils and replacement with clean soils, (3) covering the original polluted soil surface with clean soils, (4) on-site chemical leaching with acid agents, (5) dilution method, mixing polluted soils with surface and subsurface clean soils to reduce the concentration of heavy metals, or (6) phytoremediation by plants such as woody trees. The chemical stabilization method was evaluated as the most cost-effective remediation techniques for these contaminated sites, but techniques involving removal of polluted soils and addition of clean soils to the surface, or phytoremediation were also recommended (Chen et al., 1997).

Some chemical techniques for immobilizing metals in soils are application of dolomite, phosphates, or organic matter residues into the polluted soils to reduce the soluble concentration of heavy metals in soils by precipitation, adsorption, or complexation (Impens et al., 1991; Mench et al., 1994; Chen and Lee, 1997). Application of calcium carbonate materials significantly reduces the solubility of heavy metals in contaminated soils (McBride and Blasiak, 1979; Sommers and Lindsay, 1979; McBride, 1980; Kuo et al., 1985; Chen et al., 1997; Liu et al., 1998). Many reports also indicated that application of hydrous iron oxides or manganese oxides mixed into contaminated soils could reduce the concentration of soluble Cd or Pb in soils (McKenzie, 1980; Kuo and McNeal, 1984; Tiller et al., 1984; Khattak and Page, 1992; Mench et al., 1994; Chen et al., 1997).

The objectives of this study are (1) to compare the extractability of Cd and Pb in two contaminated soils treated with different chemical materials, (2) to evaluate the effectiveness of different chemical treatments on the uptake of Cd and Pb by wheat, and (3) to study the changes in the speciation of Cd and Pb by sequential fractionation methods after application of different chemical treatments.

2. Materials and methods

2.1. The contaminated soils

Two rice-growing soils, a clayey soil of the Chungshing series at the Chungfu site and a sandy soil

of the Tsaota series at the Tatan site, were selected for this study. These soils were contaminated with Cd and Pb, which were discharged from two chemical plants in northern Taiwan (Chen, 1991; Chen et al., 1996). The area of the Chungfu contaminated site is about 80 ha, and that of the Tatan site is about 20 ha.

2.2. Soil properties

The surface soils (0–20 cm) of contaminated sites were sampled, air-dried, ground through a 2 mm sieve, and stored in plastic bags prior to laboratory analysis and use in pot experiments. Soil pH values were determined using glass electrodes in a soil:water ratio of 1:1 or a soil:1M KCl ratio of 1:2.5 (McLean, 1982). The particle size distribution of two soils was analyzed by the pipette method (Gee and Bauder, 1986). Organic carbon was determined by the Walkley–Black wet combustion method (Nelson and Sommers, 1982). Exchangeable cations (K, Na, Ca, and Mg) and cation exchange capacity (CEC) were determined using ammonium acetate (pH 7) (Rhoades, 1982).

2.3. Pot experiments

Seven chemical treatments were used to compare and evaluate the effectiveness of chemical remediation techniques. Soils (500 g) were put in polyethylene pots. The soils were treated by one of the following methods: (1) calcium carbonate (CaCO_3) added to increase soil pH to 7.0, (2) a high quantity of calcium phosphate (10 mg P), (3) 2% dry hog composts (10 g), (4) 1% iron oxide (5 g), (5) 1% manganese oxide (5 g), (6) 1% synthetic zeolite (5 g, CEC of zeolite is 220 cmol(+)/kg zeolite, Sigma Chemical Company, USA), and (7) un-amended control. Each treatment was performed in triplicates and incubated for two months at room temperature (25°C). The water content of pot experiments was controlled by weighting the pot every three days. After incubation for two months, wheat (*Triticum aestivum*) was planted and an adequate amount of chemical fertilizer was also applied to the soils to support their normal growth. The amount of chemical fertilizers added to pots included 125 mg N (ammonium nitrate), 54 mg K (potassium sulfate), 53 mg P (calcium phosphate), and 30 mg Mg (magnesium sulfate). The soil pH was not effected by these treatments. The pots were laid out in a random complete block design (RCBD) under glasshouse conditions. Plants were grown for one month before harvesting. The height of the wheat shoot was only about 20 cm. The harvested shoots were oven-dried at 60°C for 72 h, then ground to pass a 40 μm sieve. 0.5 g of ground plant material was digested with a 10 ml conc. $\text{H}_2\text{SO}_4\text{:HClO}_4$ mixture (92:8, v/v) for 1 h. The digestion solutions were filtered with a Whatman No. 42 filter paper and diluted

to 50 ml. The concentration of Cd and Pb in the digests was determined by atomic adsorption spectrophotometry (Hitachi 180-30 type).

2.4. Extraction of Cd and Pb in soils

Extraction of Cd and Pb in the experimental soils was determined by several different extraction solutions for the seven treatments (Lakanen and Ervio, 1971; Norvell, 1984). These extraction reagents included distilled water (Mench et al., 1994), 0.1 M $\text{Ca}(\text{NO}_3)_2$ (Mench et al., 1994), 0.05 M EDTA (pH 7.0) (Norvell, 1984), 0.43 M HOAc (Mench et al., 1994), and 0.1 M HCl (EPA-ROC, 1994). The extraction solutions were filtered with a Whatman No. 42 filter paper and <0.45 μm millipore filter paper. Total concentrations of Cd and Pb in soils were determined by aqua regia digestion for 2 h and filtered with a Whatman No. 42 filter paper, then diluted to 50 ml (EPA-ROC, 1994). The concentrations of Cd and Pb in the filtrate were determined by flame atomic absorption spectroscopy (Hitachi 180-30 type).

2.5. Sequential extraction of Cd and Pb in soils

Sequential extraction of Cd and Pb in contaminated soils treated with different amendments was undertaken (Mench et al., 1994). The procedure was conducted in four steps, assuming the forms of Cd and Pb extracted were (1) water soluble, exchangeable, weakly bounded with organic matter, and carbonate fractions, extracted with 0.11 M HOAc by shaking for 16 h, (2) occluded Fe or Mn oxide fraction, extracted with 0.1 M hydroxy ammonium chloride by shaking for 16 h, (3) organically bound and sulphide fractions, extracted with 30% hydrogen peroxide and 1 M ammonium acetate by shaking for 16 h, and (4) structurally bound in residual fraction, digested with aqua regia. Concentrations of Cd and Pb in these sequential fractions were deter-

mined by atomic absorption spectrophotometry (Hitachi 180-30 type).

2.6. Statistical analysis

The variance and significant differences of concentrations of Cd and Pb in different chemical treated soils were analyzed by ANOVA (SAS Institute, 1982). The statistical significance in this analysis was defined at $P < 0.1$.

3. Results and discussion

3.1. Soil properties and concentrations of Cd and Pb at the contaminated sites

Some selected soil properties of both contaminated sites are shown in Table 1. Soil pH values were 5.0 (H_2O) and 4.3 (KCl) at Chungfu, and 5.4 (H_2O) and 4.8 (KCl) at Tatan. The organic carbon content and CEC of Chungfu soil were higher than those of Tatan soil. The other major difference in soil properties of these two polluted soils was the particle size distribution. The texture of Tatan soil is sandy loam with a low clay content (135 g/kg) and high sand content (742 g/kg). The texture of Chungfu soil is slit clay with a high clay content (406 g/kg). The differences in CEC and clay content between the two contaminated soils could imply that Chungfu soil has a potentially stronger adsorption of heavy metals than Tatan soil.

Concentrations of Cd and Pb in Chungfu soil extracted by 0.1 M HCl are 4.56 and 11.3 mg/kg; in Tatan soil, 15.3 and 398 mg/kg, respectively. Total concentrations of Cd and Pb digested by aqua regia in contaminated Chungfu soil were 5.47 and 39.2 mg/kg, and those of Tatan soils were 18.6 and 611 mg/kg, respectively. These results indicated that Tatan soil was more

Table 1

The soil properties and concentration of Cd and Pb in both contaminated soils for pot experiments^a

Soil	pH			Particle size analysis (g/kg)				Extractable ^b (mg/kg dry soils)		Total content ^c (mg/kg dry soils)	
	H_2O	KCl	OC	sand	silt	clay	CEC	Cd	Pb	Cd	Pb
Chungfu	5.0	4.3	24	113	481	406	12.4	4.56 (8%) ^d	11.3 (10%)	5.47 (10%)	39.2 (8%)
Tatan	5.4	4.8	13	742	123	135	4.5	15.3 (8%)	398 (5%)	18.6 (10%)	611 (12%)

OC: organic carbon content g/Kg; CEC: cation exchangeable capacity (cmol(+)/kg dry soils).

^a Pot experiments are triplicates.

^b Extracted using 0.1M HCl.

^c Digestion using aqua regia.

^d CV(%): coefficient of variation (%) in the parenthesis.

contaminated than Chungfu soil (Chen, 1991; Chen et al., 1996).

3.2. Extraction of Cd and Pb

The extracted concentrations of heavy metals in these two polluted soils treated with different amendments can be compared and evaluated by different extractants, such as distilled water, 0.1 M $\text{Ca}(\text{NO}_3)_2$, 0.05 M EDTA (pH 7.0), 0.43 M HOAc, and 0.1 M HCl. Table 2 indicated that there were significant decreases in the extractability of Cd following amendment with calcium carbonate, manganese oxide or zeolite; this is based on the concentrations extracted by EDTA and HOAc ($P < 0.1$). There are no significant differences in Cd extraction from Tatan soil extracted with $\text{Ca}(\text{NO}_3)_2$ or HCl. These results are at variance with some other published papers which indicate that application of lime can significantly reduce the solubility of Cd in contaminated soil, especially at slightly contaminated sites (McBride, 1980; Christensen, 1984; Kuo et al., 1985; Mench et al., 1994; Liu et al., 1998). Other studies have indicated that application of hydrous iron or manganese oxides can significantly reduce solubility of Cd in soil (Kuo and McNeal, 1984; Tiller et al., 1984; Fu et al., 1991; Khattak and Page, 1992; Mench et al., 1994; Lee, 1996). In the present study, the extraction of Cd in soils treated with calcium carbonate to increase soil pH to 7 is almost lowest than other amendments, therefore soil pH is regarded as a key factor controlling extractability of Cd and Pb in both contaminated soils (Table 2).

Table 3 also indicates that a significant decrease in extractable Pb concentration in soils following treatments with either calcium carbonate, manganese oxide or zeolite ($P < 0.1$). The changes in extractability of Pb were based on $\text{Ca}(\text{NO}_3)_2$ and EDTA extracts for Chungfu soil and $\text{Ca}(\text{NO}_3)_2$, EDTA, and HOAc extracts for Tatan soil. These results agree with other findings that application of lime (McBride, 1980; Kuo et al., 1985; Mench et al., 1994; Liu et al., 1998) and hydrous iron or manganese oxides (Kuo and McNeal, 1984; Tiller et al., 1984; Khattak and Page, 1992; Mench et al., 1994; Lee, 1996) can significantly reduce solubility of Pb in soil. It was also indicated that application of zeolite could significantly reduce the solubility of Pb in the soil (Gworek, 1992). The extractable Pb concentration in seriously contaminated Tatan soil was elevated ($>250 \text{ mg/kg}$).

3.3. Uptake of Cd and Pb by wheat

Uptake of Cd and Pb by wheat (*Triticum aestivum*) grown in the two polluted soils amended with different materials is shown in Table 4. Results indicate that treatment with calcium carbonate, manganese oxide or zeolite can significantly reduce plant uptake of Cd and Pb ($P < 0.1$). Table 4 also shows that treatment with iron oxide does not affect plant uptake of Cd and Pb. From these pot experiments, it was concluded that remediation with manganese oxide, calcium carbonate or zeolite can significantly reduce uptake of Cd and Pb in wheat.

Table 2

Cadmium concentrations extracted by different extractants in both contaminated soils treated with different amendments

Treatment ^a	Cd concentrations extracted by single extraction solution (mg/kg dry soils)				
	Water	$\text{Ca}(\text{NO}_3)_2$	EDTA	HOAc	HCl
<i>Chungfu soil</i>					
Control	ND	4.31 a	4.09 a	3.75 a	4.56 a
FO	ND	4.11 b	4.09 a	3.13 bc	4.45 ab
MO	ND	4.11 b	3.86 ab	2.92 c	4.23 bc
CaCO_3	ND	3.95 b	3.42 c	3.13 bc	4.17 c
Phosphate	ND	3.34 b	3.98 ab	3.34 b	4.34 abc
Compost	ND	3.13 b	3.87 ab	3.13 bc	4.56 a
Zeolite	ND	4.05 b	3.76 b	2.92 c	4.17 c
<i>Tatan soil</i>					
Control	0.12 a	13.7 a	15.1 a	17.0 a	15.3 a
FO	0.12 a	13.7 a	15.1 a	16.0 bc	15.3 a
MO	0.12 a	13.7 a	14.0 ab	15.3 c	15.8 a
CaCO_3	ND b	13.7 a	11.3 c	16.2 c	15.8 a
Phosphate	ND b	13.2 a	14.0 ab	16.2 bc	15.8 a
Compost	ND b	13.2 a	12.4 bc	14.9 c	15.3 a
Zeolite	ND b	13.7 a	12.9 bc	17.0 ab	15.8 a

Data are expressed as mean value of triplicates. Difference letters in a column indicate significant difference between treatments at $P = 0.1$.

^a FO: iron oxides; MO: manganese oxides; ND: not detectable.

Table 3

Lead concentrations extracted by different extractants in both contaminated soils treated with different amendments

Treatment ^a	Pb concentration extracted by single extraction solution (mg/kg dry soils)				
	Water	Ca(NO ₃) ₂	EDTA	HOAc	HCl
<i>Chungfu soil</i>					
Control	ND	14.6 a	12.2 a	1.27 a	11.3 a
FO	ND	12.9 b	10.5 ab	1.27 a	11.3 a
MO	ND	8.59 c	6.13 c	1.27 a	6.14 b
CaCO ₃	ND	12.9 b	11.3 ab	1.27 a	10.5 a
Phosphate	ND	13.3 ab	12.2 a	1.27 a	11.3 a
Compost	ND	14.2 ab	10.5 ab	1.27 a	11.3 a
Zeolite	ND	12.9 b	8.72 b	1.27 a	10.5 a
<i>Tatan soil</i>					
Control	1.31 a	347 a	415 a	282 a	398 a
FO	ND b	343 a	381 b	282 a	398 a
MO	ND b	330 b	381 b	235 c	398 a
CaCO ₃	ND b	343 a	389 b	282 a	398 a
Phosphate	ND b	343 a	381 b	275 b	398 a
Compost	ND b	339 ab	398 ab	242 c	398 a
Zeolite	1.31 a	347 a	372 b	275 b	372 b

Data and symbol are expressed as Table 2.

^a FO: iron oxides; MO: manganese oxides; ND: not detectable.

Table 4

Uptake of Cd and Pb in wheat shoot grown in two contaminated soils treated with different amendments

Treatments ^a	Chungfu soil (mg (dry weight) total taken up/pot)	Tatan soil (mg (dry weight) total taken up/pot)
<i>Cadmium</i>		
Control	0.05 a	0.13 a
FO	0.02 bcd	0.13 ab
MO	0.02 bcd	0.10 b
CaCO ₃	0.01 d	0.10 b
Phosphate	0.04 ab	0.12 ab
Compost	0.03 abc	0.12 ab
Zeolite	0.02 cd	0.06 c
<i>Lead</i>		
Control	0.14 a	0.31 a
FO	0.02 b	0.25 ab
MO	0.05 b	0.21 b
CaCO ₃	0.05 b	0.15 c
Phosphate	0.02 b	0.23 b
Compost	0.02 b	0.18 bc
Zeolite	0.02 b	0.14 c

Data are expressed as mean value of triplicates. Difference letters in a column indicate significant difference between treatments at $P=0.1$.^a FO: iron oxides; MO: manganese oxides.

3.4. Sequential extraction studies

The concentrations of Cd and Pb found in different sequential extractions following treatment of the un-amended soil are shown in Table 5. Results indicate that

the soluble form of Cd, ranging from 56% to 76% of the total content, is the major chemical form in both contaminated soils, and the minor fractions are the organically bound form, sulphide form, and residual form. Table 5 also indicates that the residual fraction of Pb is the major chemical form (67% of total content) in Chungfu soil, which possess low Pb content, but the other three forms (which are soluble form, Fe- and Mn-bound form, and organically bound and sulphide fraction) of Pb are of almost equal importance (20%–30% of the total content for each form) in Tatan soil with elevated Pb content.

The changes of sequential fractions of Cd in Chungfu soil and Tatan soils after incubation for two months are shown in Table 6. Results indicate that exchangeable form of Cd, extractable by ammonium acetate, in Chungfu and Tatan soils amended with calcium carbonate or zeolite can be transformed to other three bounded forms. It was concluded that the forms of Pb in Chungfu soil amended with different materials were only changed to unavailable forms including organic, sulphide and residue fractions. The fraction of available form of Pb in Chungfu soil is very low (<1%). The exchangeable form of high level of Pb in Tatan soil can be slightly transformed to other three unavailable forms after amendments with manganese oxide, calcium carbonate, compost or zeolite. The changes of metal sequential extraction of Cd and Pb in both contaminated soils indicated that the exchangeable (or available) form of Cd and Pb can be significantly transformed to unavailable form after amendment with manganese oxide, calcium carbonate or zeolite.

Table 5

The distribution of metal sequential fractionation of cadmium and lead in two contaminated soils before treatments

Soil	Metal sequential fractionations by different reagents (mg/kg dry soils)				Sum of fractions (mg/kg dry soils)	Total content of elements (mg/kg dry soils)	Recovery of summation ^a (%)
	HOAc	HONH ₃ Cl	H ₂ O ₂ +NH ₄ OAc	AR			
106	<i>Cadmium</i>						
	Chungfu	3.26 (56%) ^b	1.86 (32%)	ND (<1%)	0.70 (12%)	5.82 (100%)	5.47
	Tatan	16.3 (76%) ^b	3.19 (15%)	1.14 (5%)	0.91 (4%)	21.5 (100%)	18.6
115	<i>Lead</i>						
Chungfu	ND (<1%)	ND (<1%)	7.25 (33%)	14.5 (67%)	21.8 (100%)	39.2	56
Tatan	187 (30%) ^b	148 (24%)	162 (26%)	126 (20%)	623 (100%)	611	101

AR: total contents of elements digested by aqua regia. ND: non-detectable (lower than method detection limit, MDL). The method detection limit of Cd and Pb are 0.2 and 1.00 mg/kg dry soils.

^aThe percentage is the ratio of sum of four fractions divided the total content of metal.

^bPercentage in the parenthesis is the percentage of ratio of concentration of each fraction divided to sum of four fractions.

Table 6

The relative distribution (%) of sequential extraction of cadmium and lead in both contaminated soils after different amendments

Soil	Treatments	Metal sequential fractionations by different reagents (%)				Recovery of summation ^a
		HOAc	HONH ₃ Cl	H ₂ O ₂ +NH ₄ OAc	AR	
<i>Cadmium</i>						
Chungfu	Control	54 ^b	20	13	13	119
	FO	50	25	13	13	118
	MO	50	28	14	7	110
	CaCO ₃	43	28	14	14	110
	Phosphate	62	9	9	19	90
	Compost	55	14	14	18	95
	Zeolite	48	17	17	17	99
Tatan	Control	75	13	6	6	114
	FO	76	12	6	6	110
	MO	73	13	7	7	111
	CaCO ₃	65	16	11	9	112
	Phosphate	72	14	7	7	107
	Compost	68	17	9	7	107
	Zeolite	73	12	9	7	114
<i>Lead</i>						
Chungfu	Control	<1	<1	38	62	93
	FO	<1	<1	50	50	93
	MO	<1	<1	57	43	94
	CaCO ₃	<1	<1	50	50	93
	Phosphate	<1	<1	33	67	85
	Compost	<1	<1	33	67	89
	Zeolite	<1	<1	33	67	92
Tatan	Control	29	24	26	20	90
	FO	27	24	28	21	92
	MO	21	25	32	22	88
	CaCO ₃	24	24	30	22	88
	Phosphate	28	25	27	21	86
	Compost	21	25	31	23	84
	Zeolite	25	23	31	21	86

AR: total contents of elements digested by aqua regia.

^aThe percentage is the ratio of sum of four fractions divided to total content of metal.

^bPercentage is the percentage of ratio of concentration of each fraction divided to sum of four fractions.

4. Conclusions

1. There are significant decreases in the extractability of Cd and Pb in both contaminated soils following amendments with calcium carbonate, manganese oxide, or zeolite ($P < 0.1$).
2. The chemical remediation methods, using manganese oxide, calcium carbonate or zeolite, can significantly reduce the uptake of Cd and Pb in wheat shoots ($P < 0.1$).
3. Exchangeable (or available) form of Cd and Pb in both contaminated soils can be transformed to unavailable forms after amendment with 1% manganese oxide or zeolite, or treatment with calcium carbonate to increase the soil pH to 7.

Acknowledgements

Funding of this research was supported by National Science Council, Executive Yuan, Republic of China (grants numbers of NSC 85-2621-P-002-004 and NSC 86-2621-P-002-006).

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