



Full Length Article

Fractionation and Availability of Cadmium to Wheat as Affected by Inorganic Amendments

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ABSTRACT

Cadmium (Cd) is potentially toxic heavy metal that enters the food chain from the soil through crop uptake. Availability of metal ions in contaminated soils can be reduced by the addition of organic and inorganic amendments. In this study, effect of different inorganic amendments on fractionation and availability of Cd to wheat was evaluated. Inorganic amendments viz. lime, gypsum, diammonium phosphate (DAP) and potassium dihydrogen phosphate (KH₂PO₄) was used at different rates. Maximum reduction in Cd in grain (75%) and straw (64%) was observed where KH₂PO₄ were applied at 2000 mg kg⁻¹ followed by the treatment, where DAP was applied at the rate of 2000 mg P kg⁻¹. Lime application significantly decreased Cd concentration by 54% and 64% in straw and grain as compared to control, while decrease with gypsum 41% in straw and 61% in grains. However, maximum ABDTPA extractable Cd was observed with gypsum. The effect of P levels on Cd concentration in straw and grain was more pronounced as compared to P sources. Major fraction of Cd in all the treatments resided in carbonate bound fraction followed by exchangeable fraction. However, maximum Cd concentration was found in carbonate bound fraction (53%), where lime was applied at the rate of 8%. Exchangeable fraction of Cd decreased by 38%, while increased the carbonate fraction by 52%, Fe/Mn oxide fraction by 20% and residual fraction by 23% over that of control samples was observed with the highest level (8%) of lime application. Sequential extraction data indicated that both the P sources at the highest rate of application (2000 mg P kg⁻¹) were equally effective in transforming readily available Cd fraction (AB-DTPA extractable plus exchangeable) to less mobile fractions (carbonate, Fe/Mn oxide, organic matter & residual).

Key Words: cadmium; Amendments; Fractionation; Wheat; AB-DTPA extraction

INTRODUCTION

Higher concentration of heavy metals in soils is one of the major problems for the produce quality and human health. With increasing public awareness of the implications of contaminated soil environment on human and animal health, there has been ever-increasing interest amongst the scientific community in developing technologies for remediation of metal contaminated soils. Soils have a natural capacity to attenuate the bioavailability and the movement of metals by involving different mechanisms like precipitation, adsorption and redox reactions. When metals in high concentration are present, the capacities of soils become insufficient and it is necessary to combat the situation (Ayuso & Sanchez, 2003). There are several options available to remediate and/or to restore the productivity of contaminated soils. Conventional methods include soil excavation and landfill of the top contaminated soils *ex situ*, which is highly effective but expensive (Ryan *et al.*, 2001). In recent years, phytoremediation has been widely considered as a cost-effective approach to remediate

metal or metalloid-contaminated soils (Chaney *et al.*, 1997). In general, phytoremediation is a slow process, which reduces its applicability.

Many studies have been performed during the last decade to evaluate the ability of different soil amendments to immobilize heavy metals in polluted soils. These soil amendments include organic materials like compost, peat, manures (Narwal & Singh, 1998; Li *et al.*, 2000), phosphate rocks (Chen *et al.*, 2000; Basta *et al.*, 2001), alkaline agents such as lime and beringnite (Mench *et al.*, 2000), zeolites (Gworek, 1992), clay minerals like sepiolite and zeolite (Oste *et al.*, 2002) and synthetic polymers like polyacrylate (Lindim *et al.*, 2001). Various mechanisms have been attributed to the effect of inorganic amendments on the immobilization of metals in soils. These include: enhanced metal adsorption (Adriano, 2001) and precipitation of metals as phosphates, hydroxides or carbonates (Basta *et al.*, 2001).

Liming can lead to the precipitation of metals as metal-carbonate and significantly decrease the exchangeable fraction of metals in contaminated soil (Knox *et al.*, 2001),

which could reduce the uptake by plants. Street *et al.* (1978) found that soil pH >7.25, decreased Cd solubility by forming CdCO₃, at a CO₂ level of 0.003 atm or higher and showed the expected hundred-fold decrease in solubility for each unit increase in pH.

Cadmium has been identified as a major toxic heavy metal entering the food chain, directly through crop uptake and indirectly through animal transfer (Adriano, 2001). Health authorities in many parts of the world are becoming increasingly concerned about the effects of Cd on environmental and human health and its potential implications to international trade. The bioaccumulation of Cd in wheat and rice crops may have serious implications to animal and human health and to local and international cereal marketing (Nogawa & Kido, 1996). For these reasons, there is need to ensure that the Cd content of foodstuffs produced complies with regulatory standards. The objective of this study was to examine the effect of amendments on fractionation and bioavailability of Cd in soil.

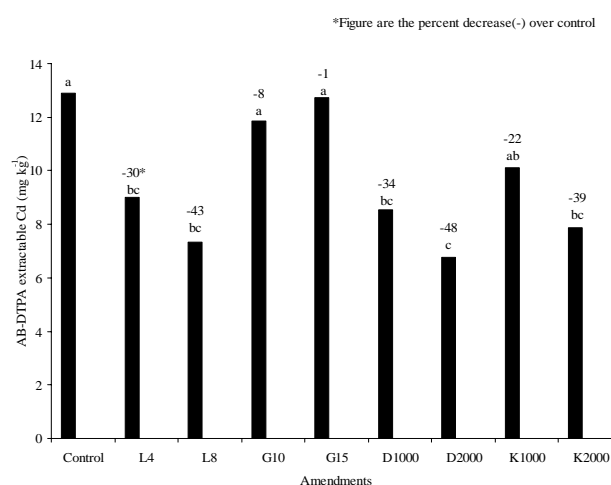
MATERIALS AND METHODS

Soil was collected from the farmer's field irrigated with city effluent since last decade. Soil was air dried and ground to pass through 2 mm sieve. Soil was analyzed for various physical and chemical properties such as texture, pH_s, EC_e, CEC, CaCO₃ by using methods as described by US Salinity Lab. Staff (1954), organic matter by method as described by Jackson (1962), AB-DTPA extractable Cd by Soltanpour (1985). Speciation of Cd in the soil was determined by method as given by Tessier *et al.* (1979). Brief description of different soil physical/chemical properties are given in Table I. Soil was contaminated with Cd @ 20 mg kg⁻¹ by using CdCl₂ and equilibrated for a period of 30 days with moisture maintained at field capacity. Pots were filled with soil @ 11 kg per pot. The treatments applied were: control, two levels of CaCO₃ (4 or 8 % by weight), two levels of CaSO₄ (10 or 15 me Ca 100g⁻¹ soil) and two levels of each diammonium phosphate (DAP) and potassium dihydrogen phosphate (KH₂PO₄) (1000 or 2000 mg P kg⁻¹ soil). After application of the treatments, soil was incubated for 30 days prior to crop sowing. The wheat crop was fertilized @ 300, 100 and 100 kg ha⁻¹ of NPK as urea, single super phosphate (SSP) and potassium sulphate (K₂SO₄), respectively. Whole of the SSP and SOP and urea @ 100 kg N ha⁻¹ was added at sowing, whereas the rest of nitrogen was applied in two equal splits. The crop was harvested at maturity and shoot and grain samples were collected for metal determination. Soil samples were also taken at the end of the experiment. Shoot and grain samples were digested in di-acid mixture (HNO₃ & HClO₄). The data were analyzed statistically following ANOVA techniques and DMR test was applied to differentiate the treatment effectiveness (Steel & Torrie, 1993).

Table I. Physical/chemical properties of soils

Parameter	Value
Textural Class	Loamy sand
pH _s	7.61
EC _e dS m ⁻¹	2.01
SAR (mmol L ⁻¹) ^{1/2}	1.60
CEC cmol _c kg ⁻¹	4.30
ESP %	4.65
OM %	0.58
CaCO ₃ %	0.72
AB-DTPA extractable Cd mg kg ⁻¹	0.18
Total Cd mg kg ⁻¹	2.12

Fig. 1. Effect of amendments on AB-DTPA extractable Cd. (Values followed by the same letters do not differ significantly at 5% level of probability)



RESULTS AND DISCUSSION

Dry matter yield of wheat. There was significant effect of different amendments on dry matter yield of wheat (Table II). Dry matter yield ranged between 19.41 to 31.0 g pot⁻¹. Lime @ 4 and 8% increased dry matter yield from 19.41 (control) to 24.27 and 31.0 g pot⁻¹, respectively. Gypsum @ 10 and 15 me 100 g⁻¹ produced dry matter yield of 23.54 and 22.34 g pot⁻¹, respectively. The DAP @ 1000 and 2000 mg P kg⁻¹ soil increased dry matter yield from 19.41 g pot⁻¹ (control) to 21.39 and 28.94 g pot⁻¹. The KH₂PO₄ @ 1000 and 2000 mg P kg⁻¹ soil increased dry matter yield to 22.73 and 29.85 g pot⁻¹, respectively. Increase in yield may be attributed to alleviating the Cd toxicity in soil owing to amendments application (Bolan *et al.*, 2003a & b) as Cd concentration in straw and grain decreased by 35 and >50% respectively Table II. Our findings are conformity with those of Chen *et al.* (2000), Friesel *et al.* (2004) and Zhu *et al.* (2004) who also reported that application of lime and phosphate amendments significantly increased dry matter yield of fescue grass (*Fescue rubra* L.) and wheat (*Triticum aestivum* L.) compared to un-amended control.

Cd concentration in wheat straw. The amendments significantly decreased the concentration of Cd in straw

Table II. The effect of amendments on pH, dry matter yield, Cd in grain and straw of wheat

Treatment	pH _s	Dry matter (g pot ⁻¹)	Cd concentration (mg kg ⁻¹)	
			Grain	Straw
Control	7.61bc*	19.41c	15.13a	37.42a
L ₄ (Lime 4%)	7.67ab	24.27b	6.47bc	22.18bc
L ₈ (Lime 8%)	7.70a	31.00a	5.46bcd	17.36cd
G ₁₀ (Gyp.10 me Ca 100 g ⁻¹)	7.61bc	23.54b	7.41b	21.91bc
G ₁₅ (Gyp.15 me Ca 100 g ⁻¹)	7.60c	22.34bc	5.92bcd	24.32b
D ₁₀₀₀ (DAP 1000 mg P kg ⁻¹)	7.62bc	21.39bc	5.89bcd	21.91bc
D ₂₀₀₀ (DAP 2000 mg P kg ⁻¹)	7.60bc	28.94a	4.20cd	16.83cd
K ₁₀₀₀ (KH ₂ PO ₄ 1000 mg P kg ⁻¹)	7.63bc	22.73bc	5.32bcd	20.57bc
K ₂₀₀₀ (KH ₂ PO ₄ 2000 mg P kg ⁻¹)	7.62bc	29.19a	3.83d	13.35d

*Values in a column followed by same letters do not differ significantly at 5% level of probability.

(Table II). The concentration of Cd in straw ranged between 13.35 to 37.42 mg kg⁻¹. The concentration of Cd in straw with lime @ 4% and 8% treated soils was 41 and 54% lower than that in the control plants, respectively. Gypsum addition @ 10 and 15 meq Ca 100 g⁻¹ soil decreased concentration of Cd in straw by 41% and 35% over that in control. Addition of DAP @ 1000 and 2000 mg P kg⁻¹ soil decreased straw Cd concentration by 41 and 55%, respectively compared to that in the control plants. The concentration of Cd in straw with KH₂PO₄ @ 1000 and 2000 mg P kg⁻¹ was 45 and 64% lower than that in the control, respectively. The decrease in concentration may be due to growth dilution, which occurred with an increase in biomass production (Lee *et al.*, 2004) and partially decreased Cd concentration in soil solution (Table II) with all the amendments through formation of less soluble compounds like Cd₃(PO₄)₂ and CdCO₃ (Bolan *et al.*, 2003a). Lee *et al.* (2004) and Zhu *et al.* (2004) reported that lime and phosphorus significantly decreased the concentration of heavy metals including Cd in wheat shoots.

Concentration of Cd in wheat grains. The amendments had significant effect on decreasing the concentration of Cd in grains compared to that in the control plants (Table II). Lime @ 4% and 8% decreased grain Cd concentration by 57% and 64% compared to that in control samples. Gypsum @ 10 and 15 me Ca 100 g⁻¹ soil decreased concentration of Cd in grains by 51% and 61% compared to that in the control samples. The concentration of Cd in grain samples with DAP @ 1000 and 2000 mg P kg⁻¹ was 61 and 72% lower than that in the control. The KH₂PO₄ @ 1000 and 2000 mg P kg⁻¹ soil (T₇) decreased grain Cd by 64 and 74% compared to that in the control samples, respectively. The decrease in concentration may be due to growth dilution, which occurred when there was an increase in yield owing to application of lime, P and compost etc. (Lee *et al.*, 2004). Chen *et al.* (2000), Lee *et al.* (2004) and Zhu *et al.* (2004) reported that amendments (lime, ZnO, P & compost) significantly decreased the concentration of Cd in wheat grains compared to that of control plants.

AB-DTPA extractable Cd. The amendments significantly decreased AB-DTPA extractable Cd compared to that in the control pots (Fig. 1), which ranged between 6.77 to 12.90 mg kg⁻¹. Lime addition @ 4% and 8% decreased Cd by 30

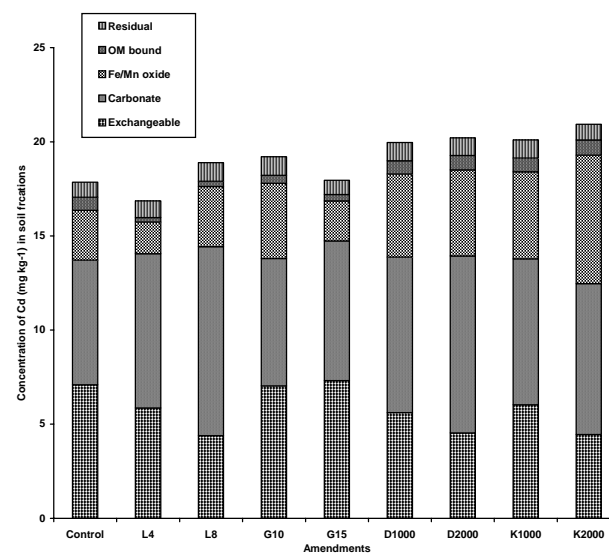
Table III. Effect of different amendments on the percent relative distribution (%) of sequential extractions of Cd in soil

Treatments	Exchangeable	Carbonate fraction	Fe/Mn bound fraction	OM bound fraction	Residual Fraction
Control	40 ^a	37	15	4	4
L ₄	35	49	10	1	5
L ₈	23	53	17	1	5
G ₁₀	37	36	21	2	5
G ₁₅	41	41	12	2	4
D ₁₀₀₀	28	42	22	4	5
D ₂₀₀₀	22	47	23	4	5
K ₁₀₀₀	30	39	23	4	5
K ₂₀₀₀	21	38	33	4	4

^aThis is the percentage of ratio of concentration of each fraction divided to sum of five fractions

^bThe percentage is the ratio of sum of concentration of Cd in five fractions to total content of Cd extracted with aqua regia.

Fig. 2. Effect of amendments on concentration of Cd in soil fractions



and 43%, respectively compared to that of the control. The increase in soil pH with lime (Table II) might cause in precipitation of Cd with carbonates and reduced the

solidities of Cd in soil. Gypsum decreased AB-DTPA extractable Cd compared to control, which was 11.87 and 12.74 mg kg⁻¹ with 10 and 15 me 100 g⁻¹, respectively. This increase with higher gypsum rate seems due to decreased Cd²⁺ adsorption resulting from increased Ca²⁺ competition (Naidu *et al.*, 1996). The concentration of Cd with DAP @ 1000 and 2000 mg P kg⁻¹ was 48 and 34%, respectively lower than that in the control samples. The KH₂PO₄ @ 1000 and 2000 mg P kg⁻¹ soil decreased AB-DTPA extractable Cd by 39 and 22% compared to that in the control samples, respectively. Naidu *et al.* (1996) and Bolan *et al.* (1999) reported that increasing rate of phosphate application caused a significant increase in Cd²⁺ adsorption by soil colloids. Therefore, decrease in AB-DTPA extractable Cd may be attributed to an increase in negative charge to affect more Cd adsorption. The precipitation of Cd as Cd(OH)₂ and Cd(PO₄)₂ has also been reported by Bolan *et al.* (2003a) with the application of P amendments.

Cd fractionation in soil. For studying speciation of Cd in soils, the widely used procedure of Tessier *et al.* (1979) was followed. The procedure yielded 94 to 122% recovery of the Cd in soil (Table III). This range of recovery, as validated by a separate total digestion with aqua regia analysis, indicated a fairly accurate series of extractions. The effect of different soil amendments on the distribution of Cd into five operationally defined fractions (Exchangeable, carbonate bound, Fe/Mn oxide bound, organically bound & residual) is discussed below.

Exchangeable Cd. Exchangeable fraction is considered readily bioavailable forms in soils (Xian, 1989) and was significantly affected by amendments in soil. Exchangeable Cd ranged from 4.38 to 7.31 mg kg⁻¹ soil (Fig. 2). The lowest Cd concentration (mg kg⁻¹) was observed with lime @ 8% (4.38) followed by KH₂PO₄ @ 2000 mg P kg⁻¹ (4.45), DAP @ 2000 mg P kg⁻¹ (4.53), DAP @ 1000 mg P kg⁻¹ (5.60), lime @ 4% (5.86), KH₂PO₄ @ 1000 mg P kg⁻¹ (6.03), gypsum @ 10 me 100 g⁻¹ (7.04), control (7.10) and gypsum @ 15 me 100 g⁻¹ (7.31).

Lime @ 4 and 8% decreased the percent distribution of Cd into exchangeable fraction from 40% (control) to 35 and 23% of the total Cd, respectively (Table III). These observations are in line with those of Chen *et al.* (2000), Bolan *et al.* (2003b), Zwonitzer *et al.* (2003) and Zhu *et al.* (2004). The distribution of Cd in exchangeable fraction with gypsum @ 10 and 15 me 100 g⁻¹ was 37 and 41% of the total Cd compared to that of the control (40%). The increase in concentration of Cd in exchangeable fraction was probably due to decreased Cd²⁺ adsorption resulting from increased Ca²⁺ competition (Naidu *et al.*, 1996). Bolan *et al.* (2003b) observed a greater increase in Cd²⁺ adsorption in the KOH-treated than the Ca(OH)₂ treated soil, which was attributed to the greater competition of Ca²⁺ for adsorption. The phosphate amendments significantly decreased exchangeable Cd compared to control. The distribution of Cd with DAP @ 1000 and 2000 mg P kg⁻¹ was 28 and 22%, respectively of the total. In KH₂PO₄ treated soil @ 1000 and

2000 mg P kg⁻¹, Cd in exchangeable fraction was 30 and 21% of the total compared to control (40%). This corroborates the observations made by others for Cd that with increasing phosphate addition, decrease in concentration of Cd in soluble + exchangeable fraction might be due to precipitation of Cd as Cd₃(PO₄)₂ and phosphate-induced Cd²⁺ adsorption (Levi-Minzi & Petruzzelli, 1984; McGowen *et al.*, 2001; Hettiarachchi *et al.*, 2002; Bolan *et al.*, 2003a). Bolan *et al.* (2003b) observed that approximately 36-52% of the phosphate-induced surface charges contributed for Cd²⁺ adsorption.

Carbonate bound Cd. The amendments differed significantly to affect carbonate bound Cd (Fig. 2) and concentration in carbonate fraction ranged between 6.60 to 10.03 mg kg⁻¹. The maximum concentration (mg kg⁻¹) was observed with lime @ 8% (10.03) followed by DAP @ 2000 mg P kg⁻¹ (9.39), DAP @ 1000 mg P kg⁻¹ (8.26), lime @ 4% (8.19), KH₂PO₄ @ 2000 mg P kg⁻¹ (8.02), KH₂PO₄ @ 1000 mg P kg⁻¹ (7.74), gypsum @ 15 me 100 g⁻¹ (7.42), gypsum @ 10 me 100 g⁻¹ (6.76) and control (6.61). The relative percent distribution of Cd into carbonate fraction (Table III) was 49 and 53% with lime @ 4 and 8% of the total, respectively. In limed soils, the activities of free Cd²⁺ and OH⁻ ions, CO₂ partial pressure, control the precipitation of Cd as CdCO₃ (octavite) and Cd(OH)₂ (Street *et al.*, 1978). However, Street *et al.* (1978) observed precipitation of Cd as CdCO₃ only in a sandy soil having low organic matter and low CEC. Gypsum application @ 10 and 15 meq Ca 100 g⁻¹ soil increased CO₃⁻ bound Cd to 36 and 41%, respectively of the total Cd. Among phosphate amendments, maximum increase in carbonate bound Cd was observed with DAP @ 2000 mg P kg⁻¹ soil and minimum with KH₂PO₄ @ 2000 mg P kg⁻¹. An increase in carbonate bound Cd after lime and P application was also observed by Bolan *et al.* (2003a) and Zwonitzer *et al.* (2003).

Fe/Mn oxide bound Cd. A significant difference in Fe/Mn bound Cd existed between treatments (Fig. 2). Maximum concentration (mg kg⁻¹) was observed with KH₂PO₄ @ 2000 mg P kg⁻¹ (9.82) and minimum with lime @ 4% (1.69). The relative percent distribution of Cd into Fe/Mn oxide fraction (Table III) with lime @ 4 and 8% was 10 and 17% of the total Cd, respectively. There was inconsistent response of Fe/Mn oxide bound Cd to applied lime. Gypsum @ 10 and 15 me Ca 100 g⁻¹ soil increased 21% and decreased by 12% of the total Cd, respectively. Among phosphate amendments, KH₂PO₄ @ 2000 mg P kg⁻¹ soil increased Fe/Mn oxide occluded Cd to 33% of the total followed by 23% with DAP @ 2000 mg P kg⁻¹ and KH₂PO₄ @ 1000 mg P kg⁻¹ and 22% with DAP @ 1000 mg P kg⁻¹. Zwonitzer *et al.* (2003) observed that the addition of KH₂PO₄ affected significant reduction in exchangeable Cd and increase in carbonate, Fe/Mn oxide and organically bound fractions. Bolan *et al.* (2003a) also reported that P application as KH₂PO₄ significantly increased the concentration of Cd in Fe/Mn oxide fraction.

Organic matter bound cadmium. Amendments had

significant effect on organic matter bound Cd (Fig. 2). The concentration of Cd bound to organic matter ranged between 0.24 to 0.83 mg kg⁻¹. Maximum concentration (mg kg⁻¹) of organic matter bound Cd was with KH₂PO₄ @ 2000 mg P kg⁻¹ (0.81) and minimum with lime @ 4% (0.24). Organic matter bound Cd constituted 4% of the total in control soil (Table III). About 1% of the total Cd existed in organic matter fraction in lime treated soil. This may be due to that most of the Cd in limed soil was still in exchangeable, carbonate and Fe/Mn oxide fractions and little was occluded by organic matter. In gypsum treated soils organic matter bound Cd decreased from 4 (control) to 2% with gypsum @ 15 me 100 g⁻¹. With DAP @ 1000 and 2000 mg P kg⁻¹, OM bound Cd was 5% of the total, whereas in soil with KH₂PO₄ @ 1000 and 2000 mg P kg⁻¹ soil, OM bound Cd was 5 and 4%, respectively of the total Cd. This is similar to the observations made by others for both Cd and other metals in the presence of lime and other inorganic amendments, such as apatite and flyash (Pierzynski & Schwab, 1993; Knox *et al.*, 2001).

Residual cadmium. Residual Cd ranged from 0.74 to 0.98 mg kg⁻¹ (Fig. 2) with non-significant amendment difference. However, there was an increase in residual Cd for all the treatments compared to that of control. This is in conformity with Chen *et al.* (2000), Bolan *et al.* (2003a & b) and Zhu *et al.* (2004) who reported that lime, P and compost significantly increased residual Cd in soil.

CONCLUSION

There was significant decrease of Cd in AB-DTPA extracts following amendments lime and phosphates application. The amendments significantly reduced the concentration of Cd in wheat straw and grains. Exchangeable fraction of Cd in soil was transformed into unavailable forms like carbonate, Fe/Mn oxide, organic matter and residual fractions with lime, gypsum and P carriers. The effect of lime and P at their higher levels was more prominent compared to that of lower levels to affect a decrease in AB-DTPA Cd or Cd concentration in wheat straw and grains.

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