Journal of Applied Sciences

ISSN 1812-5654

ANSInet
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http://ansinet.com

Journal of Applied Sciences 7 (2): 242-247, 2007
ISSN 1812-5654

The Use of Natural Clay Deposits for Controlling the Hazards of Irrigating Calcareous Soils with Zinc and Cadmium Rich Water

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Abstract: An experiment was conducted to study the effect of adding natural clay deposits to two calcareous soil differ in $CaCO_3$ level and irrigated with metal rich water on the distribution of different chemical forms of, Zn and Cd. The results indicate that the water soluble form of the studying heavy metals was redistributed to other forms by the addition of natural clay deposits. The addition of clay deposits led to a significant decrease in water extractable form of both studied metals. The highest reduction was attained on the addition of 0.6% clay deposits in the two studying soils. This result suggests that the clay minerals in the used deposits have strong effect on decreasing water extractability of both studied metals. However, the other chemical forms (i.e., exchangeable, carbonate, Fe and Mn oxides, organically bond and residual forms of Cd and Zn) were significantly increased with increasing the rate of deposits application for both studied soils. The increased in metal extractability due to clay deposits application leads to using such materials for the isolation of mine wastes with high metallic contents and due to their relatively low hydraulic conductivity and their good sportive characteristics, clay deposits can be used as a physical and chemical barrier too and to impede the dispersion of metals from pollution sources to the surface and ground water bodies. Therefore, the use of clay deposits is a promising tool for a reduction of the water extractability and possibly toxicity of heavy metals in the contaminated soil.

Key words: Calcareous soil, natural clay deposits, chemical forms of Zn and Cd, sequential extraction, mobility index

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INTRODUCTION

The continued industrialization and mining activities has led to extensive environmental problems and may result in soil heavy metals contamination. A great deal of attention should be paid to overcome and/or avoiding heavy metals pollution. In this respect, reducing heavy metal solubility and bioavailability in the contaminated area without removing them from the soil is one of the common practices in decreasing the negative impacts on the environment and improving soil quality. On the other hand, the increasing problems of dispersion of heavy metals in the environment as the result of mining activities has made that the removal of mine wastes has received considerable attention due to the toxic nature of the potential leachates formed by water circulation. In consequence, the disposal safe of wastes containing heavy metals has become to be a matter of special interest and adsorption of heavy metals on solid substrates is being studied extensively (Garcia and Page, 1978; Anderson and Rubin, 1981; Kinniburgh and Jakson, 1981; Bourg, 1983; Cavallaro and McBride, 1984; Elliott *et al*., 1986; Vega *et al*., 1995). Inorganic substrates include oxides and clay minerals; ion exchange resins and biomass. Clay minerals, because of their low permeability, play an important role, as physical barriers, for the isolation of metal from metal rich wastes (La Grega *et al*., 1994); but chemical barrier too, as consequence of the ability of some types of clay minerals to adsorb heavy metals and to avoid their environmental dispersion (Vega *et al*., 1995). In addition, the fate and transport of a metal in soil and ground water depends significantly on the chemical form and speciation of the metal (Allen and Torres, 1991; Allen *et al*., 1994). The mobility of metals in ground water systems is hindered by reactions that cause metals to adsorb or precipitate, or chemistry that tends to keep metals associated with solid phase and prevent them from dissolving. These mechanisms can retard the movement of metals and also provide a long-term source of metal contaminants (NRC, 1994). Therefore the aim of the present work is to further evaluate to what extent did natural clay deposits contribute on avoiding the hazard that might arise from additions of high contaminated water to calcareous soils. As well as, to study the effect of the application of varied rates of natural clay deposits on the changes in the distribution of different chemical forms of Cd and Zn throughout calcareous soil columns.

MATERIALS AND METHODS

Soil samples: Two separate surface soil samples were collected in Mars 15, 2005 from Derab and Al-Thomama area (about 40 km Southwest of Riyadh and about 30 km, Northeast of Riyadh, Saudi Arabia respectively. Soil samples were air-dried, ground and thoroughly mixed to pass through a 2 mm sieve. Also, at the time of collecting soil samples, natural clay deposits were collected from Dorma in the central part of Saudi Arabia, (at Dorma-Marratt road) about 140 km from Riyadh city. Then crushed and prepared for the chemical, physical and mineralogical analysis as well as to be used in the experiment.

Analysis of soil and natural clay deposits: The physical and chemical properties of the studied soils and deposits were determined according to Rainwater and Thatcher, (1979) for the determination of soluble SO_4^{2-}, Page *et al.* (1982) for the determination of chemical, physical properties of the used soil samples and natural clay deposits, (Hossner, 1996) for the total heavy metals in soils and clay deposits with $HF-H_2SO_4-HClO_4$ extraction. Also, XRD for the used clay deposits was done according to (Kunze and Dixon, 1994; Moore and Reynolds, 1997). The obtained data are given in Table 1 and 2.

Experimental procedure: A laboratory experiment was conducted-in September, 20, 2005-at the growth chamber of the Department of Soil Science, College of Food Sciences and Agriculture, King Saud University, Riyadh, Saudi Arabia. Glass columns of (10 cm internal diameter and 50 cm length with an end cap covered with filter paper for an outlet) were used in this study Each soil column was packed ten times from a height of 10 cm to obtain a final height of soil in each column to 30 cm with an approximately bulk density of 1.65 g cm^{-3}. The treatments were: Two soils differ in their $CaCO_3$ contents (6.81 for Al Thomama soil and 19.62% for Derab soil), three rates of natural clay deposits namely, ND_0 (0.0), ND_1 (0.3%) and ND_2 (0.6%.). The clay deposits were thoroughly mixed with the upper 10 cm depth of the soil columns. Soil columns were arranged in a completely randomized block design with four replicates. The soil columns were subjected to the intermittent leaching for ten times (seven days intervals) using metal rich water containing 300 ppm (Zn and Cd, as NO_3 salts) in amount equivalent to, the field capacity of soils plus 50% of it. At the end of the experiment, each soil column was sampled at three subsequent depths i.e., 0-10, 10-20 and 20-30 cm from top to bottom, respectively. The soil of each depth was air dried ground, thoroughly mixed and stored for the chemical analyses. Also, the sequential extraction procedure of Salubu *et al.* (1998) modified from Tessier *et al.* (1979) was used to estimate the chemical forms of the studied metals in sex steps)ig, F1-water soluble; F2-Exchangeable; F3-Carbonate bond; F4-Fe and Mn Oxide bond; F5-Organically bond and F6-Residual) as described by Al-Wabel and El-Maghraby (2006) in the following steps:

Step No.	Fraction	2 g soil sample from each depth was sequentially extracted with	Centrifuge/filtrate
1	F1-Water soluble	20 mL DI water for 1 h at 20°C-Rolling table	10000 rpm, in 30 min
2	F2-Exchangeable	20 mL 1M NH_4 OAC (pH 7) for 2 h at 20°C-Rolling table	10000 rpm, in 30 min
3	F3-Carbonate bond	20 mL 1M NH_4 OAC (pH 5) for 2 h at 20°C-Rolling table	10000 rpm, in 30 min
4	F4-Fe and Mn Oxide bond	20 mL 0.04 M $NH_2OH.HCl$ in 25% (v/v) Acet (pH 3) for 6 h at 80 Co-shaking water bath	10000 rpm, in 30 min
5	F5-Organically bond	15 mL 30% H_2O_2 (adj pH 2) for 5.5 h at 80°C and 5 mL 3.2 M NH_4OAcet 20 Co in 20% (v/v)HNO_3, shaking water bath and rolling table.	10000 rpm, in 30 min
6	F6-Residual	7 M HNO_3 for 6 h at 80°C	Filtrate

Table 1: Physical and chemical properties of the investigated soils

A) physical properties

	Particle size distribution (%)							
Soil	Sand	Silt	Clay	Texture	Field capacity (%)	Wilting point (%)	Available water (%)	$CaCO_3$ (%)
Al-Thomama	93	1	6	Sandy	11.22	5.82	5.80	6.81
Derab	90	2	8	Sandy	12.41	6.42	5.99	19.62

B) Chemical properties

			Soluble cations (meq L^{-1})			Soluble anions meq L^{-1}		
Soil	pH	EC (dS m^{-1})	(Ca^{+2} +Mg^{2+})	Na^+	K^+	HCO^{3-}	Cl-	SO_4^{-2}
Al-Thomama	8.31	3.61	11.4	23.4	2.06	15.19	16.45	4.63
Derab	8.42	3.94	14.7	22.3	2.82	12.33	18.98	6.63

	Total concentration (mg kg^{-1})							
Soil	Mn	Zn	Cu	Cd	Ni	Pb	(OM g kg^{-1})	(CEC Cmol kg^{-1})
Al-Thomama	55.7	6.3	2.9	1.4	7.06	2.9	6.2	4.92
Derab	104.3	10.7	3.1	1.6	8.11	5.5	11.1	9.93

Table 2: Physical and chemical properties of the used natural clay deposits

A) Physical properties

Particle size distribution (%)						
Sand	Silt	Clay	Texture class	Field capacity (%)	Hydraulic conductivity (cm s^{-1})	$CaCO_3$ (%)
23	16	61	Clay	30.2	10^{-9}	9.3

B) Chemical properties

		Soluble Cations (meq L^{-1})			Soluble anions (meq L^{-1})		
pH	EC dS m^{-1}	(Ca^{2+}+ Mg^{2+})	Na^+	K^+	HCO_3^-	Cl^-	SO_4^{2-}
8.36	2.35	17.5	4. 33	8.00	1.75	11.85	14.3

C) Fertility status

Total concentration (mg kg^{-1})								
Mn	Zn	Cu	Cd	Al_2O_3	Fe_2O_3	OM (%)	CECC mol kg^{-1}	Surface area (m^2 g^{-1})
156.46	137	67	Tr.	801	1325	4.2	56.54	149.26

D) Mineral composition

Mineral composition
Smect. +++, Kaol. ++, Iliite +, Quartez +, etc.,

Where: +++means High from 40 to 75%, ++means Moderate from 20 to 40%, +means Low from 10 to 20%

The contents of the studied metals in the solutions were determined by the ICP-AES (Prkin Elemer, Model 4300 DV). Also, the mobility indexes as well as, the amount of extracted metals in each fraction, as a percentage of total metal content, were calculated according to Kashem and Singh (2002).

Quality control: The accuracy of the heavy metal determinations based on the well-defined extraction procedures and was verified using CRM TIL1 (Canadian Certified Material, Ottawa, Canada). All the chemicals were purchased from E. Merck (A.R., 99.9%). The used columns and vessels were previously washed with 30% HNO_3, then rinsed three times with deionized water and dried in an oven. All stages of sample preparation, analysis and measuring were carried out in a clean environment.

Statistical analysis: The obtained data were statistically analyzed according to Snedecor and Cochran (1973) and the differences among treatments were tested by LSD test at (p = 0.05).

RESULTS AND DISCUSSION

The water soluble and exchangeable heavy metals are a very important pool regarding their toxicity and bioavailability. Data in Table 3 and Fig. 1 clearly appear that, the metals in water soluble and exchangeable forms are very low, in the untreated soil samples, compared to the other metal forms. Possibly due to the complexes of metals with organic matter, sorption on oxides and clays and precipitate as carbonates, hydroxides and phosphates. These data were stood in a good agreement with the water extractable fraction reported by (Tiller *et al.* (1984), Stockmeyer and Kruse (1991), Vega *et al.* (1995), Kashem and Singh (2001) Kabala and Singh (2001) and Al-Wabel and El-Maghraby (2006). On the other hand, clay deposits treatment lead to a significant decrease in

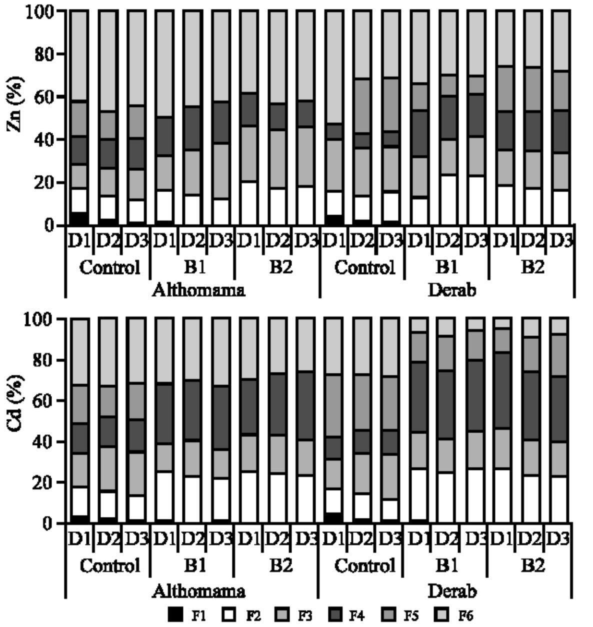

Fig. 1: Distributions of the different chemical forms of Cd and Zn (as a percent of their total contents) throughout calcareous soil columns amended with Natural clay deposits treatment, Control = 0, B1 = 0.3% and B2 = 0.6% soil column depths: D1 = 0-10, D2 = 10-20, D3 = 20-30 cm, Chemical forms: F1 = water soluble, F2 = Exchangeable, F3 = Carbonate form, F5 = Fe and Mn oxide bond, F6 = Residual

Table 3: Fractional distribution of Zn and Cd in different soil depths of calcareous soil columns as affected by Natural clay deposits application

Natural	Soil depth (cm)	Zn							Cd						
		F1	F2	F3	F4	F5	F6	MI	F1	F2	F3	F4	F5	F6	MI
		mg kg^{-1}							mg kg^{-1}						
Al-Thomama Soil															
ND0	0-10	2.9	6.0	5.6	6.7	9.0	21.7	0.39	2.5	6.5	7.8	7.3	9.8	16.1	0.51
	10-20	1.3	7.0	7.8	7.6	7.7	27.9	0.37	1.1	7.6	11.9	8.3	8.4	18.5	0.58
	20-30	0.9	7.4	9.5	9.2	10.4	29.7	0.36	0.7	8.0	13.8	10.0	11.3	20.4	0.54
ND1	0-10	2.0	31.5	32.3	36.1	61.2	38.9	0.48	1.8	34.3	18.2	42.1	34.2	11.2	0.62
	10-20	0.6	25.2	38.6	37.6	53.4	29.2	0.54	0.5	25.3	20.0	33.5	27.2	6.9	0.68
	20-30	0.3	21.0	46.0	33.4	47.1	27.1	0.63	1.1	27.1	16.3	38.1	30.9	10.9	0.56
ND2	0-10	0.2	41.8	53.7	31.5	39.9	40.3	0.86	0.1	44.4	31.8	47.6	35.4	17.4	0.76
	10-20	0.1	32.8	50.0	22.6	36.4	44.4	0.80	0.1	35.4	26.0	44.5	29.6	9.5	0.74
	20-30	0.1	29.9	41.9	20.2	31.2	35.1	0.83	0.0	27.4	20.4	38.5	22.9	7.3	0.70
Derab soil															
ND0	0-10	3.1	7.9	17.2	4.9	20.6	16.9	0.67	2.9	7.8	9.8	6.9	20.1	17.8	0.46
	10-20	1.3	8.7	15.5	4.4	18.5	22.2	0.56	1.3	8.3	13.1	8.0	18.1	18.5	0.51
	20-30	1.1	11.6	16.9	4.9	20.3	25.0	0.59	0.9	7.7	16.5	8.4	19.8	21.3	0.51
ND1	0-10	2.1	30.6	45.5	51.8	33.0	80.7	0.47	2.2	47.4	33.0	63.3	27.1	13.1	0.80
	10-20	0.9	57.3	40.5	47.6	25.5	73.4	0.67	0.6	39.0	27.2	52.1	28.2	13.4	0.71
	20-30	0.7	49.8	38.6	42.8	18.6	64.9	0.71	1.3	44.4	30.9	59.2	25.0	10.2	0.81
ND2	0-10	0.2	45.6	42.1	44.0	51.8	63.2	0.55	0.2	50.9	35.4	69.5	23.6	8.7	0.85
	10-20	0.1	36.8	38.8	39.4	44.1	56.6	0.54	0.1	44.2	30.8	62.0	31.8	17.2	0.68
	20-30	0.1	30.3	31.3	35.4	32.9	50.8	0.52	0.0	32.8	22.9	46.0	27.8	11.7	0.65
Statistical analysis LSD at 5% level for															
Soil (A)		0.5	8.6	9.7	1.6	7.5	7.3	0.222	0.2	4.6	4.0	6.1	3.3	0.6	0.06
ND. (B)		0.6	10.5	11.9	1.9	9.2	8.9	0.272	0.3	5.7	4.9	7.4	4.0	0.7	0.07
Depth(C)		0.6	10.5	11.9	1.9	9.2	8.9	0.272	0.3	5.7	4.9	7.4	4.0	0.7	0.07
A*B		0.9	14.8	16.8	2.8	13.0	12.6	0.385	0.4	8.0	7.0	10.5	5.7	1.0	0.10
A*C		0.9	14.8	16.8	2.8	13.0	12.6	0.385	0.4	8.0	7.0	10.5	5.7	1.0	0.10
B*C		1.1	18.2	20.5	3.7	15.9	15.4	0.471	0.5	9.9	8.5	12.9	6.1	1.2	1.22
A*B*C		1.5	25.8	29.0	4.8	22.5	21.8	0.667	0.7	13.9	12.1	18.2	9.9	1.7	0.17

F1: Water soluble, F2: Exchangeable. F3: Carbonate bond, F4: Oxide bond, F5: Organic bond, F6: Residual, MI: Mobility Index; ND0, ND1 and ND2 Natural clay deposits = 0.0, 3.0 and 6.0%, respectively

the water soluble forms of both Zn and Cd. The decrease of water extractable forms of both studied metals in the soil treated with clay deposits was greater than the other metal forms. This result indicates that the water soluble forms of heavy metals were redistributed to other forms by the addition of clay deposits. As compared with untreated soil, the addition of 0.6% clay deposits significantly decreased water extractable Zn and Cd in the upper soil segment (0-10 cm) of both studied. These results suggest that, the clay minerals found in the used clay deposits have strong effect on decreasing the water extractability of Zn and Cd. This may be rendered to that, the clay deposits well known as a good adsorbents for heavy metals (Tiller *et al*., 1984; Stockmeyer and Kruse, 1991). On the other hand, Smectittes (which found in the used clay deposits, Table 2) have a typical layered silicate structure consisting of an octahedral sheet sandwiched between two tetrahedral sheets. The interlayer space-an essential characteristics of clay minerals form this group-is easily accessible to water and another polar liquids, also, Smectitte group minerals have a good cation exchange capacity due to the presence of hydrated cations as Ca^{2+}, Na^{+}, K^{+}, etc. In the layer surfaces and this cations can be easily exchanged by heavy metals (such as Zn and Cd) compensating the negative charge as follows:

2 Smectitte-Na+Me2+ -Smectitte-Me+2 Na+.

Also, because of the increase of the specific area as was found in this study (149.26 $m^2\ g^{-1}$ Table 2). In this respect Vega *et al*. (1995), indicated that, the sorption tends to increase when particulate size of clay minerals decreases. Although, the application of clay deposits decreased the water extractability of Zn and Cd, the other metal forms were significantly increased over the rate of clay deposits applications. This reflected on increasing the amounts that retained from both studied heavy metals in the deposits treated soil as compared to the untreated one. The order of fractions content found in the upper soil segments of the untreated soils (in most cases) was as follows:

Zn: residual>organic>carbonate>
oxide>exch.>w. soluble (Table 3).

Cd: residual>organic>carbonate>
oxide>exch.>w. soluble (Table 3).

On the other hand, the distribution of solid-phase heavy metals (Zn and Cd) in the clay deposits treated soils, changes considerably with the rate of deposits application. Nevertheless, these changes varied with the fraction type, soil column depth and the element under study. However, the solid-phase chemical forms of each metal in the clay deposits treatments exhibited widely different patterns of elements fractions as shown in Fig. 1. The relatively order of fractions content found in case of applying clay deposits (in most cases) was as follows:

Zn: organic>residual>carbonate>oxide>exch.>w. soluble, (Table 3).

Cd: oxide>organic>exch.>carbonate>residual>w. soluble, (Table 3).

This was true for both studied soils, however, the values of the chemical forms of Zn were higher than those of Cd. This may be due to the relatively higher initial content of Zn in the used clay deposits (Table 2). Also, in the Derab soil, the obtained metal values in each form were some what higher, possibly due to its relatively higher initial contents of $CaCO_3$ and organic matter (Table 1). It is appear, therefore, that, the studied metals could found in immobile forms affecting by clay deposits applications. As, the values of the studied metals extracted with milder reagents (F1-F3) fractions (e.g., water soluble, exchangeable and carbonate forms) increased significantly with increasing rate of natural clay deposits application. On the other hand, the extractable amounts of the mobile fractions of Cd, found in Derab soil were relatively higher (Table 3), possibility due to its higher calcium carbonate content (19.62%) and probably Cd bond with Ca which could be released by 1MNH_4OAC at pH 5.0 (i.e., F3-carbonate form, Kashem and Singh, 2002). The carbonate form of Cd is relatively mobile in acidic conditions and within a few years it may convert to an exchangeable form (Chlopecka, 1993). From the above-mentioned results, one can say that:

- Most of water extracted elements were concentrated in the surface layer (0-10) of the untreated soils. While a relatively increases in the lower depths for the other chemical forms were obtained. Also, the water soluble and exchangeable fractions of Zn and Cd in the untreated soils, constituted the least proportion of elements fraction, as these forms were depended mainly on the studied soils properties, which differ in their $CaCO_3$ and OM contents (Table 1).
- The application of clay deposits has changed the solid phase containing Zn and Cd, this change suggests that the application of clay deposits would retained such metals from the polluted leachates.
- The calculated mobility indexes (Table 3) indicated that, the mobile forms (F1-F3 e.g., water soluble, exchangeable and carbonate) of each of the studied metals tends to increase by nearly (40% of the total metal content) in the Natural clay deposits treatments depending upon the type of element and/or the deposits application rate. On the contrary, the immobile forms (F4-F6 e.g., Fe-Mn oxide, organic and residual forms) of each of the studied metals represents (more than 70% of the total metal content) in the untreated soils.
- The experimental data obtained from interaction of metal solutions with the added clay deposits lead to consider this material as a potent adsorbent to retain heavy metals from polluted leachates. Therefore, the property of such materials to retain metals could be used for the isolation of mine wastes with high metallic contents.

CONCLUSIONS

According to the relatively low hydraulic conductivity of the used clay deposits (Table 2), as well as, their good sportive characteristics they can be used not only as a physical barrier, but, as a chemical barrier too, with the aim to impede the dispersion of metals from pollution sources to the surface and ground water bodies. In the case of security landfills, where residual leachates are always produced because of the natural humidity of the stocked wastes, clay deposits has an interesting potential application field in the environmental industry to avoid the dispersion of metals to the environment.

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