

Research Article

Monometal and Competitive Adsorption of Cd, Ni, and Zn in Soil Treated with Different Contents of Cow Manure

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This study was conducted to assess the monometal and competitive adsorption of Cd, Ni, and Zn in the soil incubated with different contents of decayed cow manure (T_1 : 0, T_2 : 25, and T_3 : 50 tha^{-1}) for 90-d at 25°C. Sorption isotherms were characterized using the linear Freundlich equation. Most sorption isotherms were well described by the Freundlich equation (maximum $R^2 = 0.988$ and minimum $R^2 = 0.618$) and the monometal and competitive adsorption isotherms of Zn, Cd, and Ni followed the L-curve type (L-2). Results showed that the mono- and multimetal sorption amounts increased with an increase in organic amendment content as the sorption capacities for Cd, Ni, and Zn followed the following sequence: $T_2 \geq T_3 > T_1$. This sequence was consistent with the CEC and particularly pH for the three soils. All soils showed greater sorption capacity for Zn than the other trace elements as the sorption sequence in T_1 was $\text{Zn} > \text{Cd} > \text{Ni}$, while in both T_2 and T_3 was $\text{Zn} > \text{Ni} \geq \text{Cd}$. Therefore, the metal-binding sites in OM were more selective for Zn and Ni than Cd. Competition significantly reduced metals K_d , especially for Cd and Ni.

1. Introduction

Heavy metal pollution of soils has become a dangerous problem in agricultural production around the world in the past few decades, as a result of anthropogenic activities, such as mining or industrial activities and improper use of heavy metal-enriched materials in agriculture, including chemical fertilizer and pesticides, industrial effluents, sewage sludge, and wastewater irrigation [1, 2]. Anthropogenic trace elements are easily accumulated in the surface soil [3], leading to serious environmental concerns [4]. There is concern that increased anthropogenic inputs of trace elements in soils may result in transport of these metals in the soil profile, leading to the increased concentrations of trace elements in the ground or surface waters [5]. The movement of trace elements in soil is greatly affected by their physicochemical forms in the soil solid phase [6] and adsorption [4]. The most important process that affects heavy metal availability and mobility is sorption onto soil solid phases. Sorption of heavy metals by soil depends on factors such as the nature and content of the mineral and organic constituents, the nature

and concentration of the metal, the composition of the soil solution, and pH [7, 8].

Organic matter is one of the major contributors to the ability of soils for retention of heavy metals in an exchangeable form. Moreover, organic matter also improves soil fertility and structure and other soil properties. The effect of organic matter on the reduction of metals in soil solutions is highly complex. It depends on the other soil components and the chemistry of the metals and also the characteristics of the OM, particularly with respect to its degree of humification, content of heavy metals, salts, and its effect on soil pH [9–14]. Application of fresh manure can increase heavy metal mobility in soil due to the production of soluble organic compounds which form complexes with the metals [9, 12], while the humic substances which constitute a major part of the OM of compost, peats, or decayed manure can reduce metal solubility and bioavailability by adsorption and by forming stable complexes with metals [11, 12]. It was reported that heavy metals' adsorption onto soil constituents declines with decreased organic matter content in soils [15, 16]. The ability of OM to bind heavy metal ions can be

attributed to their high content of oxygen-containing functional groups, including carboxyl, phenol, hydroxyl, enol, and carbonyl structures of various types [17]. Clemente et al. [18] showed that cow manure and compost can be useful for the immobilization of heavy metals in calcareous-contaminated soils, particularly manure, an organic material rich in P, which plays an important role in the reduction of their solubility. OM has been of particular interest in studies of heavy metal fixation in soils due to the tendency of transition metals to form stable complex with organic ligands [19].

Heavy metal adsorption and hence their plant availability do not only depend on soil constituents (inorganic and organic), but also on the nature of metals involved, and on their competition for soil sorption sites. Usually when competitive sorption of metals is compared with their monometal behavior, it is found that their adsorption is lower in the competitive systems [20]. More strongly sorbed metals, such as lead and copper, are less affected by competition than mobile metals, such as cadmium and zinc [21, 22]. However, the effect of competition among poorly sorbed metals, such as Cd, Ni, and Zn, especially in organic amended soils, has not been documented. Moreover, it is not clear how the competitive adsorption of poorly sorbed heavy metals affects their behavior and availability over time.

While numerous studies have been conducted to understand monometal and competitive adsorption of trace elements in pure minerals and other soil components, their noncompetitive and competitive adsorptions in the presence of different contents of animal manure are barely known and there is limited information on the effects of the manure on the availability of metals in contaminated soils. Therefore, the objectives of present study were to evaluate the adsorption of Cd, Ni, and Zn applied as single or together affected by adding different contents of cow manure to soil after an incubation period of 90-d from the addition date of cow manure and also their Freundlich isotherms. Changes in the physical and chemical nature of the manure-soil mixture after the incubation period were also quantified.

2. Materials and Methods

2.1. Sample Collection. Soil samples were collected from the surface layer (0–30 cm) of the field in Agricultural Faculty of Shahid Chamran University of Ahvaz, air-dried at room temperature, and sieved through a 2 mm plastic sieve. A subsample of soil was used to determine chemical and physical properties.

Decayed cow manure sample obtained from the Ahvaz ranches was used as the organic amendment, air-dried, and sieved through a 2 mm plastic sieve to increase the active surface area of the amendment particles. Some of the chemical and physical characteristics of the soil and cow manure are reported in Table 1.

2.2. Preparation of Treatments and Heavy Metal Solutions. The sampled soil was used to fill greenhouse pots and three levels of cow manure including 0 (control), 25, and 50 tha^{-1} were amended to soils. Soil-manure mixtures were placed

TABLE 1: Some chemical and physical properties of the soil and manure.

Variable	Soil	Manure
pH	7.16	7.9
EC (ds/m)	1.9	10.8
OM (%)	0.16	0.41
Zn (mg Kg^{-1})	2.47	48.4
Cu (mg Kg^{-1})	0.047	14.5
Fe (mg Kg^{-1})	1.15	293.7
Cd (mg Kg^{-1})	<0.05	<0.05
Ni (mg Kg^{-1})	<0.05	<0.05
CEC (cmolc kg^{-1})	15.3	—
Soil texture	Loam	—
Soil classification	Torrertic Haplustepts	—

in plastic bags, wetted to 65–70% of their water holding capacity, and then incubated for 90 days in a temperature-controlled chamber at 25°C. During this period, soil-manure mixtures were weighed and rewetted so as to maintain the soil moisture content constant, as appropriate. Soil samples were air-dried after 90 days and prepared for next experiments. Three replicates were arranged for each treatment.

Ni, Cd, and Zn were, respectively, used as NiCl_2 , CdCl_2 , and ZnCl_2 in varying concentrations, including 10, 25, 30, 40, 50, and 100 mg L^{-1} . Stock solutions of the metal salts were prepared in distilled water.

2.3. Noncompetitive Adsorption Experiments. 2 gr from each air-dried soil sample was weighed and poured into acid-washed polyethylene tubes and 20 mL of solution of Cd^{+2} , Ni^{+2} , or Zn^{+2} , in the above concentrations, individually added to the tubes. Then, the tubes were shaken at 150 rpm (rate per minute) with a rotator agitator for 24 h, as the equilibrium time, at 25°C. The soil samples dissolved in metal solutions (1:10 w/v) were centrifuged initially at 3000 rpm for 15 min to remove soil. Then, the supernatant was filtered through filter paper (Wathman filters Number 42). Cd, Ni, and Zn concentrations in the supernatant were measured by atomic adsorption spectrophotometer (model: Unicam 939).

2.4. Competitive Sorption Experiments. Competitive adsorption isotherms were performed in the same way, but by adding Cd, Ni, and Zn at a 1:1:1 mole ratio.

Monometal and competitive adsorption experiments had been conducted in constant pH. The control of pH was done by acid or base solutions and both experiments were carried out in a background electrolyte of 0.01 M CaCl_2 and were replicated three times.

2.5. Sorption Equations. The amount of trace elements sorbed by soil was calculated with the following equation:

$$Q = \frac{(C_o - C_e)V}{M}, \quad (1)$$

where Q is the amount of adsorbed species (mg kg^{-1}), C_o is the initial concentration of the species in solution (mg L^{-1}), C_e is the equilibrium concentration of the species in solution (mg L^{-1}), V is the solution volume (mL), and M is the weight of air-dried soil (kg).

The relation between the concentration of dissolved and adsorbed heavy metal was expressed by the Freundlich isotherm. The linear form of the Freundlich isotherm is given by

$$\log Q = \log K_F + \frac{1}{n} \log C, \quad (2)$$

where Q (or x/m) is the amount of metal adsorbed per gram of sorbent (mg kg^{-1}), C is the equilibrium concentration of the adsorbate (mg L^{-1}), and K_F and n are the Freundlich constants related to adsorption capacity and adsorption intensity, respectively. Freundlich parameters can be obtained by plotting $\log Q$ versus $\log C$, with $1/n$ being the slope and $\log K$ being the intercept of the line. K_F is the Freundlich coefficient, related to the total sorption capacity of the soil and n is a constant that typically has a value of less than 1.

Distribution coefficient, K_d , is an index of a metal's potential mobility and calculated with the following equation:

$$K_d = \frac{\text{Equilibrium metal concentration adsorbed}}{\text{Equilibrium metal concentration in solution}}. \quad (3)$$

The distribution coefficient represents the sorption affinity of the metal cations in solution for the soil solid phase and can be used to characterize the mobility and retention of trace elements in a soil system. Low distribution coefficients indicate that most of the metals present in the system remain in the solution and are available for transport, chemical processes, and plant uptake [23], whereas higher values indicate lower mobility and higher retention of metals in the soil. K_d is positively related to metal sorption capacity of soils. Also, the higher the sorption intensity parameter ($1/n$), the lower the binding affinity (n) of soil with metal. The sorption isotherms were obtained for each soil sample by equilibrating 2 g soil with 20 mL of solutions containing concentrations of 10, 25, 30, 40, 50, and 100 mg L^{-1} of mono- and three metals, separately. Statistical analysis was performed using the Excel programs.

3. Results and Discussion

3.1. Incubation Effects on the Soil Characteristics. As can be seen in Table 1, the cow manure has the near-neutral pH, low salinity and is unpolluted with heavy metals. Therefore, adding the manure to soil led to increase of the soil OM and slight change in the soil pH. Addition of manure amendment affects the properties of soils. The treatments of the soil with different contents of decayed cow manure resulted in increases at pH in the range from 7.56 (25 tha^{-1}) to 7.54 (50 tha^{-1}); when compared to the control soil (7.16), the EC increased to 3.12 and 4.34 dS^{-1} in response to treatments with 25 tha^{-1} and 50 tha^{-1} manure amendment, respectively. Thus, the primary change of soil during incubation and hence

TABLE 2: Some variable properties of soil due to addition of manure in different levels at the end of incubation period.

Variable	T_1	T_2	T_3
pH	7.16	7.56	7.54
EC (ds/m)	1.9	3.12	4.34
CEC (cmolc kg^{-1})	15.3	16.4	16.6
OM (%)	0.16	0.23	0.28

T_1 : control, T_2 : soil treated by 25 tha^{-1} manure, T_3 : soil treated by 50 tha^{-1} manure.

the processes most likely to influence metal sorption were OM and consequently CEC, that the latter factor is affected by the soil OM content. Raising the soil OM content can increase soil CEC, a factor which may affect both soluble and exchangeable metal levels [12, 24] (see Table 2).

3.2. Effect of Contents of Cow Manure and Initial Concentration of Metals on the Sorption Isotherms. Monometal and competitive Zn, Cd, and Ni adsorption isotherms for T_1 , T_2 , and T_3 were obtained based on the Freundlich equation after the 90-d incubation with cow manure. Monometal (Figure 1) and competitive (Figure 2) sorption isotherms by the three soil treatments exhibited differences in shape and in amount retained. Sorption isotherms provide important information about the soil immobilization capacity and the strength with which the sorbate is held onto the soil [25]. On the y -axis S represents the metal concentration sorbed onto solid phases and on the x -axis C represents metal equilibrium concentration in solution.

Experimental data from the adsorption tests for all metals and all treatments in both systems (monometal and competitive) gave a satisfactory fit (Table 3) to the Freundlich model (maximum $R^2 = 0.988$ for Cd-monometal at T_2 and minimum $R^2 = 0.618$ for Cd-competitive at T_1).

Figures 1 and 2 showed that the sorption amount increased with an increase in equilibrium Zn, Cd, and Ni concentrations and approached a plateau value at higher equilibrium concentrations for the soils. Therefore, the monometal and competitive adsorption isotherms of Zn, Cd, and Ni for T_1 (a), T_2 (b), and T_3 (c) were of the L-curve type (L-2), which is characterized by an initial slope that does not increase with the concentration of the added metal in the soil solution. This explains the higher adsorption at lower concentrations, which then decreases as the concentration increases [26]. The isotherm shapes at low concentration range, do not change markedly between T_1 , T_2 , and T_3 , despite the significant increase of organic matter at the end of incubation period, whereas they vary with increasing the heavy metal concentrations in all treatments.

The sorption amount increased with an increase in organic amendment content. Organic matter is considered to play an important role in reducing plant uptake of heavy metals from soils due to its high CEC and complexing ability. Many authors have found that high organic matter content or addition of organic matter by organic amendments decreased

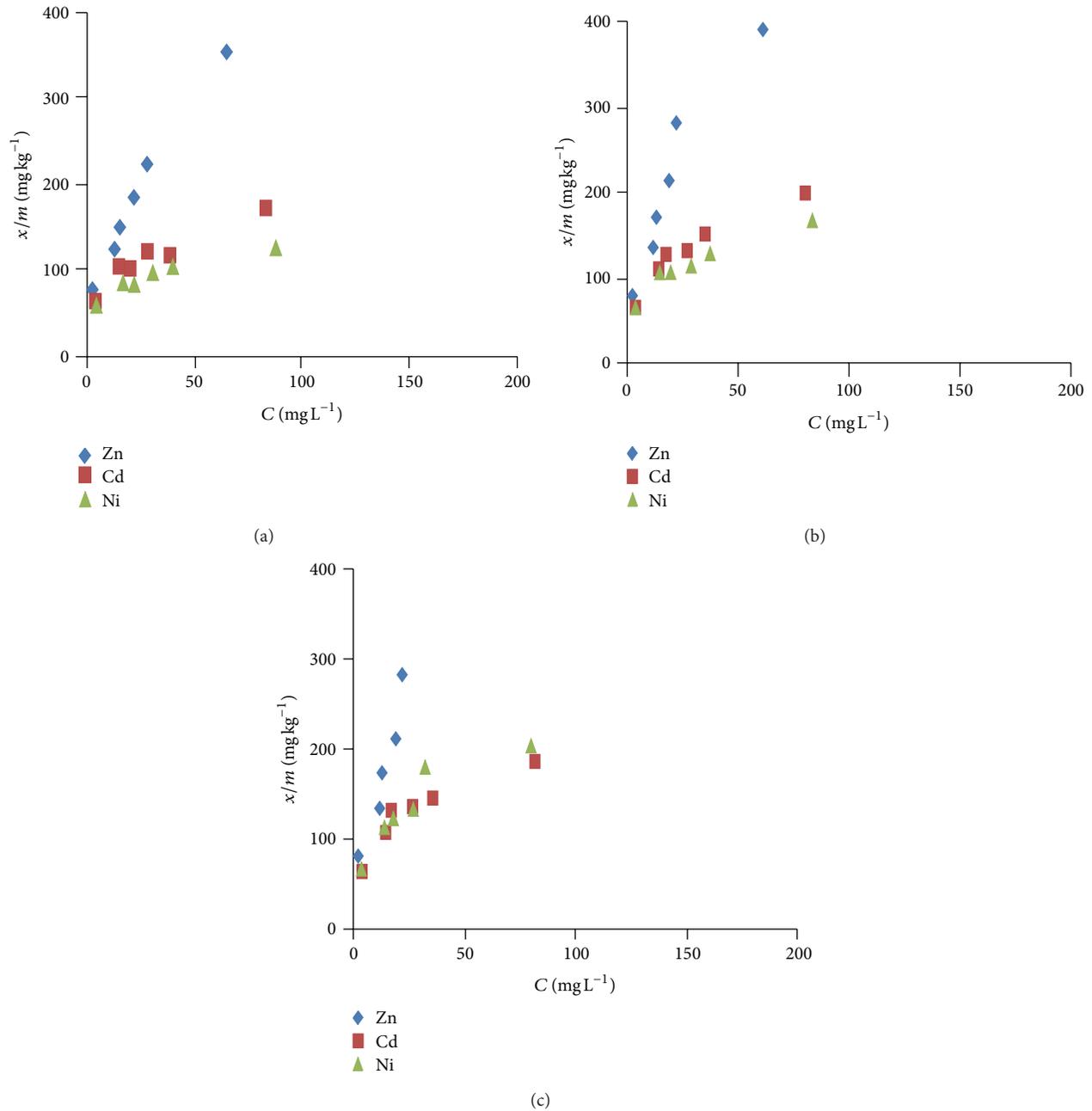


FIGURE 1: Noncompetitive adsorption isotherms of Zn, Cd, and Ni in soil control (a), T_2 (b), and T_3 (c) samples. Data are means of triplicate determinations.

the heavy metals concentration in solution [14, 27–29]. This effect is attributed to the high CEC of organic matter and its ability to form chelate complexes with metals. Haghir [30] concluded that the decreased plant availability of heavy metal concentration with higher levels of organic matter added was predominantly due to the effect of increasing soil CEC.

The adsorption values in the treatments were positively correlated with manure contents, indicating that the organic manure has the capacity of metal adsorption. As seen in Table 3, K_F was increased with increasing amount of the manure in soil; hence the adsorption sequences for three

treatments in both noncompetitive and competitive systems were found: $T_2 \geq T_3 > T_1$. This sequence is consistent with OM and CEC sequences and also is consistent with pH sequence in the treatments. Clemente et al. [18] in their study on the short-term effects of two different OM amendments (fresh cow manure and a mature compost) found that the concentrations of Zn extracted with CaCl_2 were significantly different in every sampling in control, manure-, and compost-treated samples. The main effect of manure treatment on Zn was the significant decrease of NaOH- and EDTA-extractable concentrations of this element

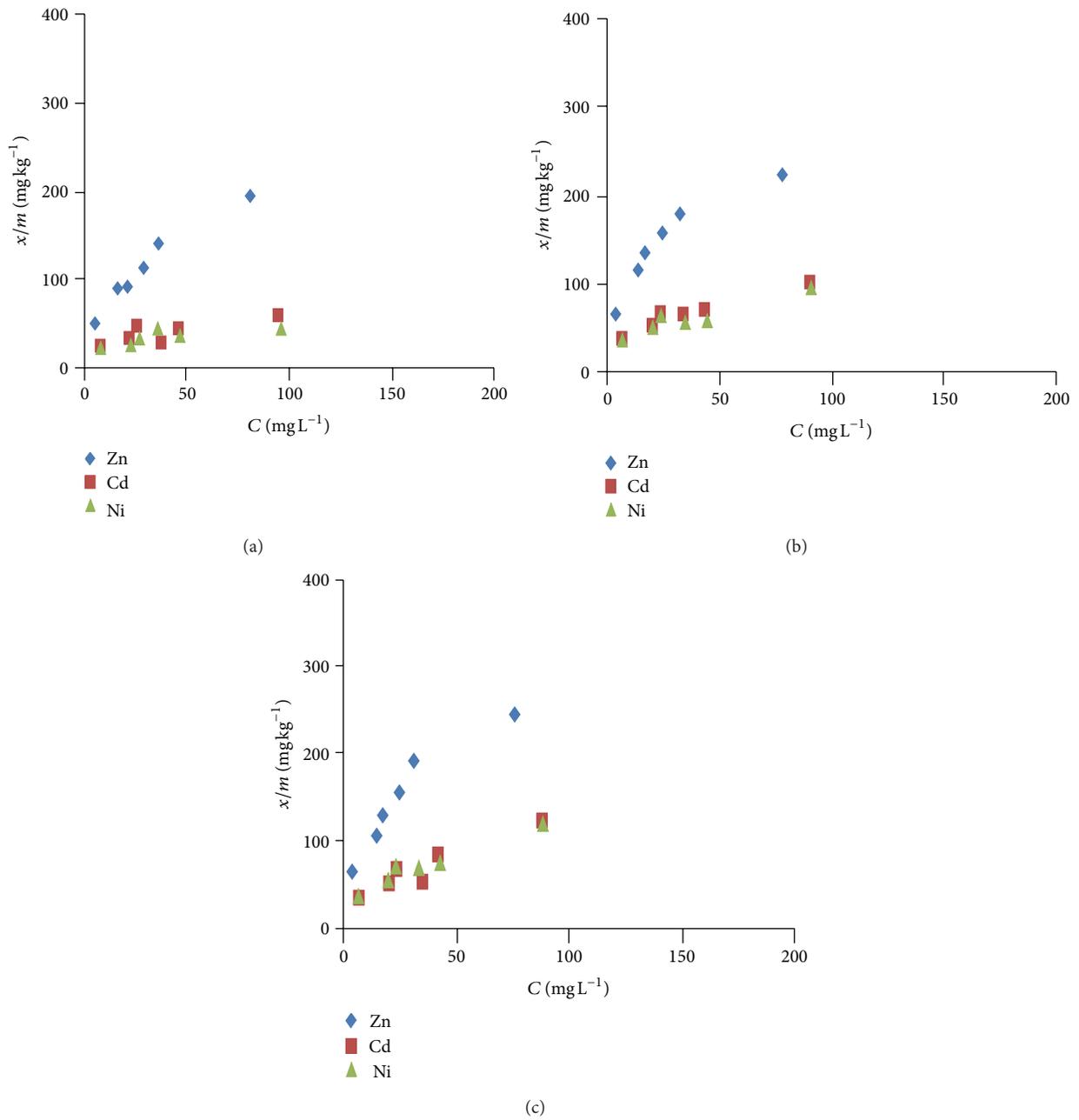


FIGURE 2: Competitive adsorption isotherms of Zn, Cd, and Ni in soil control (a), T_2 (b), and T_3 (c) samples. Data are means of triplicate determinations. Coefficients of variation were less than 10%.

TABLE 3: The Freundlich constants and R^2 values obtained from the Freundlich isotherm in single and multimetal systems.

	K_F	T_1 N	R^2	K_F	T_2 N	R^2	K_F	T_3 N	R^2
Zn-monometal	50.815	2.299	0.94	56.234	2.150	0.935	57.147	2.127	0.916
Zn-competitive	24.21	2.101	0.985	43.752	2.538	0.983	38.904	2.304	0.968
Cd-monometal	46.025	3.46	0.959	46.773	2.994	0.988	46.238	3.021	0.962
Cd-competitive	15.031	3.448	0.618	21.183	2.915	0.953	15.667	2.273	0.855
Ni-monometal	43.351	4.184	0.984	48.97	3.650	0.981	46.025	2.841	0.975
Ni-competitive	13.899	3.663	0.74	20.606	3.125	0.861	19.724	2.445	0.951

T_1 : control, T_2 : soil treated by 25 tha⁻¹ manure, T_3 : soil treated by 50 tha⁻¹ manure.

with respect to control soil after incubation period, showing a decrease in metal availability shortly after amendment addition to soil. Petruzzelli et al. [31] and Agbenin and Olojo [32] report significant decreases in the adsorption of heavy metal on soil after the chemical removal of organic matter.

Sorption values from T_2 to T_3 were decreased slightly, that attributed to increase of pH. Increases in pH decrease surface potential and proton competition and thus favor metal binding [33]. Adding the organic material to soil and the subsequent incubation conditions may influence the pH and therefore modify metal speciation [13]. Walker et al. [34] found that cow manure is capable of preventing soil acidification and decreasing heavy metal bioavailability. Also, they expressed that the increase of soil pH caused by manure addition is the main factor reducing metal availability. Ram and Verloo [35] found that farmyard manure and peat soil enhanced the mobility of Cd at lower pH and decreased it at higher pH.

The steep slope of the Zn and Cd isotherms in contrast to the gentle slope of the Ni isotherm in the control treatment indicated a stronger affinity of the soil for Zn and Cd than Ni. On the basis of K_F values, the following selectivity sequence for T_1 was found: Zn > Cd > Ni (Table 3), which may be attributed to their adsorption affinities and their first hydrolysis equilibrium constant: (9.0) Zn > (9.9) Ni \geq (10.1) Cd. Similar result was also reported by Antoniadis et al. [36], who studied monometal and competitive adsorption of Cd, Zn, and Ni by a soil before and after one-year incubation with sewage sludge and observed the decreasing sequence adsorption: Zn > Cd > Ni.

Ni sorption increased with an addition of cow manure to soil more than Cd sorption, as in treatments of 25 and 50 tha^{-1} ; sorption sequences followed this sequence: Zn > Ni \geq Cd. Zn and Cd were sorbed more favorably than Ni on inorganic surfaces of soil, but organic matter favored retention of Ni over Cd. When OM was added to the soil, $K_{d,Zn}$ (monometal system) increased 9.6% and 12.18% at T_2 and T_3 , and $K_{d,Ni}$ increased 11.5% and 5.9%, respectively, as compared to the control, whereas $K_{d,Cd}$ had no significant change as compared to the control. The higher increasing of $K_{d,Zn}$ and $K_{d,Ni}$ than $K_{d,Cd}$ suggested that the metal binding sites in OM were more selective for Zn and Ni than Cd. Also, these results suggest that Ni has higher affinity for organic activity sites than for inorganic sites, whereas Zn would prefer both inorganic and organic sites. The higher affinity of the control soil for Zn and Cd is probably due to the existence of a greater number of active sites with high specificity for these metals, so when they are present, these sites would not be occupied by other cations [37].

3.3. Effect of Competition on the Sorption Isotherms and the Freundlich Constants. The presence of other metals reduced the amount of each metal sorbed compared to monometal system, so the adsorption capacity (K_d) of each metal in the multimetal condition was lower than that in the monometal condition. This suggests that the metals were competing for the same binding sites as one another, though the total amount of added metals was not so high as to occupy a

large part of the available surface adsorption sites. Significant inhibitory effects of competitive metals on the adsorption of a particular metal have also been reported by Basta and Sloan [27].

Although K_d values indicated a reduction in metal adsorption due to competition sorption, they were evident mostly at the higher end of equilibrium concentrations. Thus, at low metal concentrations, effects of competition were not very strong. This seems to concur with the work of Saha et al. [38], who found no evidence of metal (Cd, Zn, and Pb) competition at low concentrations. They explained that at low added metal concentrations, metals are mainly adsorbed onto specific adsorption sites, while at higher metal inputs, soils lose some of their ability to bind heavy metals as adsorption sites overlap, becoming thus less specific for a particular metal. This, in turn, induces a reduction in metal sorption. Although competition reduced sorption of all three metals, the magnitude of these effects was different for each metal. $K_{d,Zn}$ decreased by nearly 51.3% at T_1 due to competition, by 21.2% at T_2 , and by 31.9% at T_3 . This compares to competition-induced reductions of around 67.3%, 54.7%, and 66.1% for Cd at T_1 , T_2 , and T_3 , respectively, and 67.9%, 57.9%, and 57.1% for Ni at T_1 , T_2 , and T_3 , respectively. Thus, the effect of competition in reducing the sorption of metals followed the following order: Cd \geq Ni > Zn. This suggests that upon coaddition of the three metals to the soil, Zn, and to a less extent Ni, became preferentially adsorbed at the expense of Cd. This is likely to have been the result of differences in the nature of the dominant sorbing surfaces for each metal [32].

4. Conclusion

The monometal and competitive adsorption behavior of Cd, Ni, and Zn in soil, affected by different contents of decayed cow manure (0, 25, and 50 tha^{-1}), was investigated in the present study. Evidences showed that soils which received decayed manure exhibited higher CEC and pH in general terms. Therefore, the organic matter had metal immobilization effect after the incubation.

Monometal system K_d values in control soil followed the following order: Zn > Cd > Ni. In amended soils, Zn and particularly Ni sorption values increased significantly, while Cd did not after 90-d incubation. The adsorption sequences in T_2 and T_3 were in the order of Zn > Ni \geq Cd. The adsorption capacity (K_d) of each metal in the multimetal condition was lower than that in the monometal condition. Low values of K_d for Cd and Ni in competitive system indicate that most Cd and Ni remain in the solution and are available for transport, chemical processes, and plant uptake. The effect of competition in reducing the sorption of metals followed the following order: Cd \geq Ni > Zn. Most sorption isotherms for trace elements were adequately described by the Freundlich equation and were of the L-curve type.

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