

# Zinc Sorption by Six Agricultural Soils Amended with Municipal Biosolids

Antoine Karam, Lotfi Khiari, Bruno Breton, Alfred Jaouich

**Abstract**—Anthropogenic sources of zinc (Zn), including industrial emissions and effluents, Zn-rich fertilizer materials and pesticides containing Zn, can contribute to increasing the concentration of soluble Zn at levels toxic to plants in acid sandy soils. The application of municipal sewage sludge or biosolids (MBS) which contain metal immobilizing agents on coarse-textured soils could improve the metal sorption capacity of the low-CEC soils. The purpose of this experiment was to evaluate the sorption of Zn in surface samples (0-15 cm) of six Quebec (Canada) soils amended with MBS (pH 6.9) from Val d'Or (Quebec, Canada). Soil samples amended with increasing amounts (0 to 20%) of MBS were equilibrated with various amounts of Zn as ZnCl<sub>2</sub> in 0.01 M CaCl<sub>2</sub> for 48 hours at room temperature. Sorbed Zn was calculated from the difference between the initial and final Zn concentration in solution. Zn sorption data conformed to the linear form of Freundlich equation. The amount of sorbed Zn increased considerably with increasing MBS rate. Analysis of variance revealed a highly significant effect ( $p \leq 0.001$ ) of soil texture and MBS rate on the amount of sorbed Zn. The average values of the Zn-sorption capacity of MBS-amended coarse-textured soils were lower than those of MBS-amended fine textured soils. The two sandy soils (86-99% sand) amended with MBS retained 2- to 5-fold Zn than those without MBS (control). Significant Pearson correlation coefficients between the Zn sorption isotherm parameter, i.e. the Freundlich sorption isotherm ( $K_F$ ), and commonly measured physical and chemical entities were obtained. Among all the soil properties measured, soil pH gave the best significant correlation coefficients ( $p \leq 0.001$ ) for soils receiving 0, 5 and 10% MBS. Furthermore,  $K_F$  values were positively correlated with soil clay content, exchangeable basic cations (Ca, Mg or K), CEC and clay content to CEC ratio. From these results, it can be concluded that (i) municipal biosolids provide sorption sites that have a strong affinity for Zn, (ii) both soil texture, especially clay content, and soil pH are the main factors controlling anthropogenic Zn sorption in the municipal biosolids-amended soils, and (iii) the effect of municipal biosolids on Zn sorption will be more pronounced for a sandy soil than for a clay soil.

**Keywords**—Metal, recycling, sewage sludge, trace element.

## I. INTRODUCTION

THERE is an increasing interest in land application of municipal sewage sludge or biosolids (MBS) obtained from wastewater treatment plants due to their content of

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valuable components such as organic matter (OM) and plant nutrients [1]-[3].

Several studies revealed that application of MBS would improve several chemical properties of soils [2], especially those affecting heavy metal mobility in soil. Many soil parameters, such as pH, cation-exchange capacity (CEC) and the contents of OM, phyllosilicates, carbonates and variable charge minerals including iron and manganese oxides can reduce the environmental risk caused by heavy metal pollution [4]-[7].

The addition of alkaline-stabilized MBS or Ca-MBS to acid soils would add a lot of Ca compounds to the soil, leading to increase base saturation and exchangeable Ca in soils, and consequently soil pH [8], [9]. In soil environments, pH is considered to be the primary factor that controls adsorption of heavy metals and their availability [10]-[12]. MBS would also increase soil OM [1]. OM of the MBS is a very important adsorptive medium for trace metals in the soil [13].

While there have been numerous studies on the adsorption of zinc (Zn) by soils without amendments [11], [14], few reports have been devoted to studying adsorption of anthropogenic Zn by MBS-amended soils. Recently, [15] highlighted the importance to evaluate the land capacity to receive biosolids, and to compare the metal adsorption capacity between amended and non-amended soil. Solids added to soil with organic wastes could provide exchangeable sites for metal adsorption [16]. Anthropogenic sources of Zn, including industrial emissions and effluents, Zn-rich fertilizer materials and pesticides containing Zn can contribute to increasing the concentration of soluble Zn at levels toxic to plants in acid sandy soils. The application of MBS which contain metal immobilizing agents on coarse-textured soils could improve the metal sorption capacity of the low-CEC soils.

The aim of the investigation was to evaluate the sorption of Zn in surface samples (0-15 cm) of six Quebec (Canada) soils amended with MBS (pH 6.9) from Val d'Or (Quebec, Canada).

## II. MATERIALS AND METHODS

### A. Soils

Samples of six soils representing different soil types, textures and reactions (Table I) were collected from the arable layer (0-20 cm) in different locations in Quebec (Canada). These are Des Crêtes sand (S1), St-Jude loamy sand (S2), St-Augustin loam (S3), St-Lambert silt loam (S4), Kamouraska silty clay loam (S5) and Ste-Rosalie heavy clay (S6). Soils S1 and S2 are classified as Humo-Ferric Podzols and the others

(S3 to S6) as Humic Gleysols.

The soils were air-dried, passed through a 2 mm sieve and characterized using standard methods of soil analysis [17]. Particle-size distribution (sand, silt, clay) was determined by a hydrometer method and soil OM by a modified Walkley and Black wet-combustion method. Soil pH was measured in water suspension using a ratio soil-water of 1:1 ( $pH_{\text{water}}$ ).

Exchangeable basic cations, e.g. calcium ( $Ca_{\text{ex}}$ ), magnesium ( $Mg_{\text{ex}}$ ) and potassium ( $K_{\text{ex}}$ ), were extracted with 1N ammonium acetate, pH 7, whereas cation exchange capacity (CEC), at pH 7.0, was determined by the  $Ca(OAc)_2+CaCl_2$  method. Selected physical and chemical characteristic of each soil is given in Table I.

TABLE I  
SELECTED PHYSICAL AND CHEMICAL PROPERTIES OF THE SOILS (S1 TO S6) AND  $K_F$  VALUES

Soil property	S1	S2	S3	S4	S5	S6
Sand (%)	99.2	85.6	56.2	36.9	17.6	4.3
Silt (%)	0.5	10.0	32.3	52.6	55.8	32.5
Clay (%)	0.3	4.4	11.5	10.5	26.6	63.2
Organic matter (g/kg)	0.5	12.4	38	32	22	9.5
pH (H <sub>2</sub> O)	5.6	5.5	6.2	6.8	7.1	8.0
Exchangeable Ca (cmol(+)/kg)	0.17	4.8	13.3	16.1	17.6	32.3
Exchangeable Mg (cmol(+)/kg)	0.04	0.27	0.83	1.05	2.09	7.50
Exchangeable K (cmol(+)/kg)	0.05	0.12	0.35	0.25	0.27	0.64
CEC (cmol(+)/kg)	2.63	6.2	15.3	17.4	20.0	40.5
$K_F$ values (mg/kg):						
soils without amendment	2.5	10.5	108.2	300.2	416.3	664.0
soils + 5% MBS	14.6	25.1	149.1	338.2	452.77	736.5
soils + 10% MBS	37.1	64.15	181.1	361.9	474.32	827.2
soils + 20% MBS	89.5	172.6	219.9	396.4	517.0	1091.0

### B. Municipal Biosolids (MBS)

The MBS used in this trial was obtained from an anaerobic sewage sludge digester at the Val d'Or Municipal Wastewater Treatment Plant, Val d'Or (Quebec, Canada). The sludge was oven dried at 105°C for 24hr, mixed and homogenized, and ground to pass a 2-mm sieve; its main characteristics are: pH (H<sub>2</sub>O) 6.95; organic carbon 100.2 g/kg; total Zn 560 mg/kg; total Ca 1.44%; total Mg 0.51%; total K 0.28%; total Al 0.76%, total Fe 2.61% and total Mn 0.13% [18].

### C. Zn Sorption

Sub-samples of each soil were mixed with MBS (4 treatments) as follows: T0: 0% MBS (soil without amendment); T1: 5% MBS; T2: 10% MBS; T3: 20% MBS. Zinc sorption was suited using ZnCl<sub>2</sub> solution (25, 50, 100, 150, 250 and 500 mg Zn/L, corresponding to 375 – 7500 mg Zn/kg of soil), 0.01 M with respect to CaCl<sub>2</sub>. Thirty milliliters of solution were added to each MBS-amended soil sample (2 g, two replicates) in a polyethylene centrifuge tube and allowed to equilibrate for 48 hours at room temperature with occasional shaking. After equilibration, the samples were centrifuged at 2000 rpm for 10 min, decanted and supernatant solutions retained for Zn and pH ( $pH_{\text{equilibrium}}$ ) determination. As it is difficult, by means of adsorption curves, to distinguish between true adsorption and surface precipitation or polymerization, the term sorption is used here. According to [19], sorption is a general term that can be used when the retention mechanism of solute at a solid surface is unknown. The amount of Zn sorbed was calculated as the difference between the quantity added and that remaining in the supernatant solution. The data on Zn sorption of each soil were fitted to the linear form of the empirical Freundlich

sorption isotherm [19]:  $\log q = 1/n \log C + \log K_F$ , where q is the quantity of Zn sorbed (mg/kg), C is the equilibrium Zn concentration (mg/l),  $K_F$  (mg/kg) is Freundlich sorption coefficient, and n is a linearity factor [20], [21]. The concentration of cations in solution was measured using Perkin Elmer atomic absorption spectrophotometer.

### D. Statistical Analysis

Statistical analysis was performed using the Analysis of Variance and Linear Models (Pearson correlation) procedures [22]. The Pearson values (r) presented in Table II are significant at  $p \leq 0.05$  (\*), 0.01 (\*\*) or 0.001 (\*\*\*)

## III. RESULTS AND DISCUSSION

### A. pH

The  $pH_{\text{water}}$  values of S1, S2, S3 and S4 ranged from 5.6 to 6.8 and were lower than that of the MBS ( $pH_{\text{water}}$  6.95) by 1.35, 1.25, 0.75, 0.15 pH units, respectively (Table I). The pH values of S5 and S6 were higher than that of the MBS by 0.15 and 1.05 pH units, respectively (Table I). The  $pH_{\text{water}}$  values of fine-textured soils are higher than those of coarse-textured soils. Soil  $pH_{\text{water}}$  showed a tendency to increase with increasing MBS application rate (data not shown).

After the addition of ZnCl<sub>2</sub> solutions, the values of  $pH_{\text{equilibrium}}$  of MBS-amended soil suspensions varied between: (i) 6.1 (T0) and 7.4 (T0) for S1, (ii) 6.3 (T0) and 7.7 (T3) for S2, (iii) 7.1 (T0) and 7.7 (T3) for S3, (iv) 7.9 (T0) and 8.0 (T3) for S4, (v) 7.6 (T0) and 7.9 (T3) for S5, and (vi) 7.8 (T0) and 8.1 (T3) for S6. The values of  $pH_{\text{equilibrium}}$  increased in the following order: T0 (without amendment) < T1 < T2 < T3. Overall treatments,  $pH_{\text{equilibrium}}$  of soil samples increased with increasing MBS application rate, and was greatest for soils

receiving 20% MBS (T3) and lowest for soils without amendment (Fig. 1). The lowest  $pH_{\text{equilibrium}}$  values (6.1-7.7) were recorded in light soils (sand 85-99%) with low initial  $pH_{\text{water}}$ . The  $pH_{\text{equilibrium}}$  change is more pronounced in coarse-textured soils than in clayey soils. Comparing the T3 and T0 treatments, the S1 and S2 soils (initial soil  $pH_{\text{water}}$  5.5-5.6) demonstrated the larger increase in  $pH_{\text{equilibrium}}$  at 1.27-1.34 pH units, whereas the smaller was shown by the S4, S5 and S6 soils (initial soil  $pH_{\text{water}}$  6.8-8.0) at 0.12-0.38 pH units (Fig. 1). This indicates that MBS had sufficient lime value to neutralize soil acidity and to raise pH of soils containing high levels of sand with low buffering capacities (ability to resist pH change). Except by dilution of the soil mixed with the neutral-pH MBS, the increase in pH could be explained by the existence of base-forming cations (e.g. Ca, Mg, K) associated with oxides, hydroxides, or carbonates in the MBS. A dose-dependent increase of soluble Ca, Mg and K resulted from incorporation of MBS to soils (data not shown). Thereby, the soil pH was increased to some extent after the addition of MBS.

#### B. $K_F$

Significant Pearson correlation coefficients for the six soils ( $r = 0.90$  to  $0.99$ ) indicated that the sorption equilibrium of Zn fits the linear form of the empirical Freundlich sorption equation. The amount of sorbed Zn increased markedly with increasing application rate of MBS. The  $K_F$  values increased gradually with increasing MBS rate and were all higher for the soils amended with MBS than for the control where no amendment was added (Table I). This finding is consistent with results of other metal sorption studies using sewage sludge as soil amendment [16].

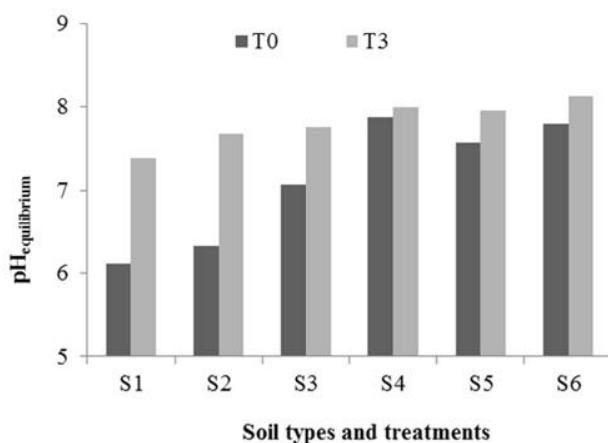


Fig. 1 Values of  $pH_{\text{equilibrium}}$  as a function of soil types and treatments

The two sandy soils (86-99% sand) amended with MBS retained 2- to 5-fold Zn than those without MBS. This result indicates that MBS had a high affinity for Zn retention in the soil. Antoniadis et al. [23] studied Cd, Ni, and Zn adsorption by a sewage sludge-amended soil (Typic Xerofluvent) before and after one-year incubation in both monometal and competitive systems. They found that sewage sludge-amended soils exhibited high affinity for Zn. The presence of organic

material in the MBS would provide sorption sites for Zn. It is known that OM or humic substances plays a significant role in the sorption of heavy metals [6]. According to many authors humic substances retain metallic ions mainly by pH-dependent formation of inner-sphere and/or outer-sphere complexes and cation exchange [6], [24]. As reported earlier, sewage sludge provides sites for metal adsorption [16], [25]. Zinc has been found to be adsorbed strongly by the humic acid-like fraction of municipal sewage sludge [26]. Several studies showed that by adding organic waste amendment to soil, its sorption capacity is enhanced [25], [27]-[29], justifying the increased use of such materials for environmental site remediation purposes [6], [15]. In a study on the rehabilitation of acid generating mine tailings pond, [18] showed that MBS had a great ability to bind heavy metals.

It is obvious that the effect of MBS on Zn sorption is more pronounced for sandy soils (Table I) and that the high clay soil S6, which had the highest pH (8.0), was by far the most sorptive. This result suggests that the immobilization of soluble added Zn by MBS-amended soils is mainly due to sorption properties of both MBS and soil type. Analysis of variance revealed a highly significant effect ( $p \leq 0.001$ ) of soil texture and MBS rate on the amount of sorbed Zn (data not shown). As mentioned by Alamgir [30], the soil texture is an important soil characteristic that influence heavy metal sorption. Assaad et al. [31] found that the amount of specifically Zn retained by three surface soil samples from Denmark varied with soil texture, initial Zn concentration and soil pH. The average  $K_F$  values of MBS-amended coarse-textured soils were lower than those of MBS-amended fine textured soils (Table I). These results confirm the fact that fine-textured soils often retain more  $Zn^{2+}$  ions than coarse-textured soils due to their higher content of phyllosilicate clay minerals [14], [16], [32], [33]. From the point of view of sorption properties of soils, the total amount of clay minerals in soil bulk has a major importance [34].

In general, highly significant Pearson correlation coefficients were obtained between  $K_F$  and soil pH, clay content, exchangeable basic cations (Ca, Mg or K), CEC and clay content/CEC ratio (Table II). The results indicate that the sorption of Zn is affected by chemical properties that are associated with soil buffer capacity (pH, clay, CEC). Among all the soil properties measured, soil pH gave the best significant correlation coefficients ( $p \leq 0.001$ ) for soils receiving 0, 5 and 10% MBS. Numerous works showed that Zn sorption was mainly influenced by the soil reaction [10], [11], [31]. Yli-Halla and Loeppert [35] found that pH was by far the most important variable explaining Zn sorption on some cultivated mineral soils from Finland. It is known that pH values regulate specific adsorption and complexation reactions in soils [6], [36]. An increase in pH may cause a decrease in the competition of  $H^+$  (and  $Al^{3+}$ ) ions for negative sorption sites [36], [37]. Moreover, several functional groups of OM such as carboxyl, phenolic, alcoholic and carbonyl dissociate with increasing pH, thereby enhancing their affinity for heavy metal retention [38]. As a consequence, Zn adsorption increases with the increase in soil pH. Furthermore,

the possibility of precipitation of inorganic and organic Zn compounds with increasing pH should not be neglected [37], [39], especially when heavy metal concentrations in soil solution are high [38].

TABLE II  
LINEAR CORRELATION COEFFICIENTS BETWEEN SOIL PROPERTIES (N=6 SOILS) AND  $K_F$

Soil property	T0	T1	T2	T3
Soil pH	0.992***	0.994***	0.988***	0.857*
Clay content	0.934**	0.939**	0.975**	0.958**
CEC	0.957**	0.965**	0.976***	0.946**
$C_{ex}$	0.957**	0.966**	0.971**	0.918**
$Mg_{ex}$	0.904*	0.911*	0.935**	0.037**
$K_{ex}$	0.857*	0.873*	0.892*	0.969*
Clay/CEC	0.868*	0.870*	0.873*	0.873*

The significant correlation between clay content and  $K_F$  is explained by the fact that the clay size fraction of soils contains clay minerals with high affinity for Zn. It is known that the clay size fraction of a soil has a high sorption capacity due to its large specific surface area and CEC [30]. As a result, clay size fraction retains high amount of metals when compared to sand size fraction [40]. It is obvious that the CEC and clay content of the soil can interact in the sorption of Zn as indicated by the significant positive correlation between  $K_F$  and clay/CEC. These results are in agreement with [14] which reported that the sorption Zn parameters of some gleysolic C horizons of Quebec were highly correlated with clay/CEC ratio.

#### IV. CONCLUSION

The application of MBS with higher pH than the targeted soils might have the potential to increase soil pH, especially at high application rate. The soils amended with MBS had a greater affinity for Zn than the soils without amendment. The results obtained show that adding MBS increases the metal sorption capacity of soils. The removal of soluble added Zn by MBS-amended soils is mainly due to sorption properties of both MBS and soil type. These results may have practical implications in metal-polluted site management for the reduction of trace metal mobility in drainage system and of water contamination risk.

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