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Fate of biosolids Cu and Zn in a semi-arid grassland

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ABSTRACT

Biosolids land application applies varying trace metal amounts to soils. Measuring total soil metals is typically performed to ensure environmental protection, yet this technique does not quantify which soil phases play important metal release or attenuation roles. We assessed the distribution of biosolids-borne Cu and Zn associated with soluble/exchangeable, specifically adsorbed/carbonate-bound, amorphous and crystalline Mn/Fe oxyhydroxide-bound, residual organic, and residual inorganic phases. Biosolids were surface-applied (no incorporation) to experimental plots, at the Meadow Springs Ranch (40 53′46″N, 104 52′28″W) which is owned by the city of Fort Collins, CO, USA, in 1991 at rates of 0, 2.5, 5, 10, 21, and 30 Mg ha^{-1}. Plots were split in half in 2002, with one-half receiving biosolids at rates identical to 1991 rates. In 2003, 0–8, 8–15, and 15–30-cm soil depths were collected and subjected to 4 M HNO_3 digestion and sequential fractionation. The 4 M HNO_3 extraction suggested downward Cu transport, while Zn was immobilized in the soil surface. The sequential extraction procedure, more sensitive to changes in soil metal pools, suggested that repeated biosolids application did not affect vertical Zn movement, but did increase the downward transport potential of organically complexed Cu. In the given time, organically complexed Cu was likely mineralized and subsequently associated with soil mineral oxide phases. Because bioavailability of Cu is associated with dissolved phases, and soluble/exchangeable Cu concentrations were below detection limits in the subsoil, a reduction in environmental quality should be minimal. Still, we advocate that on coarse-textured semi-arid soils, biosolids application rates should match the plant N needs to avoid potential downward trace metal transport.

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1. Introduction

Biosolids land application is a major method of disposal in the US, with approximately 50% land applied (US EPA, 2007a); in US Environmental Protection Agency (US EPA) Region 8, which encompasses Colorado, Montana, North Dakota, South Dakota, Utah, and Wyoming, 85% is land applied (US EPA, 2007b (http://www.epa.gov/unix0008/water/)). This recycling method can greatly benefit municipalities by recycling plant nutrients in an environmentally sound manner when applied at agronomic rates (Barbarick et al., 1992). Our 12-year biosolids study with the city of Fort Collins, Colorado has provided valuable information on the effects of biosolids application to a semi-arid grassland soil. For example, Harris-Pierce (1994) studied surface biosolids application with no incorporation of rates up to 30 Mg ha^{-1} to a city of Fort Collins owned semi-arid grassland. The author noted increasing concentrations of total Cd, Cu, Mo, and Zn in the soil 0–8-cm depth associated with increasing biosolids application rates. There was some evidence that NO_3–N and salts were leaching to the lowest depth sampled (15–30 cm), with increased leaching associated with increasing biosolids application. Sullivan et al. (2005) revisited the research site, noting that ammonium bicarbonate-diethylenetriaminepentaacetic acid (AB-DTPA) extractable Cu and Mo increased with biosolids application rates up to of 30 Mg ha^{-1} in the 0–15-cm depth. Lacking from these studies, however, was a more detailed analysis pertaining to the fate and transport of biosolids-borne metals within this long-term biosolids-amended semi-arid grassland soil.

The limited studies pertaining to biosolids application in semi-arid settings have focused primarily on total and plant-available soil metal concentrations. Fresquez et al. (1990) studied the effects of increasing biosolids surface application with no incorporation (22.5, 45.0, and 90.0 Mg ha^{-1}) on the 0–15-cm soil depth of degraded semi-arid grassland. The authors observed that DTPA-extractable Cu and Zn concentrations increased with increasing biosolids application in each of four years following biosolids

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application; increases were probably the result of biosolids movement and consequently incorporation into the soil, increased decomposition by microorganisms, and subsequently a decrease in soil pH (Fresquez et al., 1990). Walter et al. (2006b) studied HNO_3–HCl (i.e. total) and DTPA-extractable heavy metal soil concentrations one and five years following 0, 40, 80, and 120 Mg ha^{-1} biosolids application to a degraded Mediterranean soil. Total soil metals did not significantly increase over time. However, an increase in DTPA-extractable Cu and Zn concentrations with increasing biosolids rates five years following application was observed, potentially due to organic matter degradation and incorporation of soluble organic matter into the soil. Brenton et al. (2007) studied trace element leaching through intact columns of semi-arid rangeland soils amended with up to 90 Mg ha^{-1} of surface-applied biosolids. Leachate Cu and Zn concentrations were not affected by biosolids application.

Many biosolids trace metal research studies have focused on soils in agricultural settings. Emmerich et al. (1982a) studied biosolids mixed with soil in the top 15 cm of a column study and found little metal movement below the incorporation zone. They attributed the lack of movement, and thus fate, to metals shifting towards more stable forms after soil incorporation (Emmerich et al., 1982b). Dowdy et al. (1991) and Sloan et al. (1998), however, observed increased trace metal concentrations with depth in biosolids-treated agricultural soil as compared to a control soil. McBride et al. (1999) noticed metal transport into groundwater below an orchard soil amended with 244 Mg ha^{-1} biosolids, associating metal movement with dissolved organic matter. Al-Wabel et al. (2002) utilized soils from a long-term biosolids-amended agricultural site, noting that 26.8 Mg ha^{-1} biosolids increased both dissolved organic carbon and Cu in column effluents. A positive correlation between Cu and dissolved organic carbon was also observed.

More detailed biosolids-amended soil analyses have utilized sequential extraction procedures to operationally define soil metal pools. Sloan et al. (1997) determined the fate of biosolids-borne metals using a sequential extraction procedure and noted >75% of Cu and Zn was found in relatively stable soil fractions following applications of up to 224 Mg ha^{-1} on cultivated soils. Sukkariyah et al. (2005) utilized a sequential extraction on soils from an agricultural setting which received up to 210 Mg ha^{-1} biosolids, showing the greatest Cu and Zn concentrations were associated with soil metal oxides. Guerra et al. (2007) used a sequential extraction procedure to study the effects of biosolids-borne metals to a Mollisol under production agriculture. The authors observed increased Zn in labile fractions associated with biosolids application at 30 Mg ha^{-1}. Copper concentration in the organic matter phase increased following biosolids application, with Cu forming relatively strong complexes with biosolids fulvic acid functional groups (Guerra et al., 2007; Dahlgren et al., 1997). Berti and Jacobs (1996) also used a sequential extraction technique and showed that total biosolids applications of up to 690 Mg ha^{-1} on cropland increased Zn, and to a lesser extent Cu, in potentially plant-available (i.e. bioavailable) forms.

Sequential extractions are time consuming, yet research on soil trace metal behavior following years of biosolids application is needed to understand long-term effects on soils (Sukkariyah et al., 2005). Furthermore, such information is necessary for heavy metal environmental impact purposes as well as for enhancement of biosolids land application regulatory guidelines (Vaca-Paulin et al., 2006). This project presents a unique perspective because previous research utilizing sequential trace metal extraction techniques have mostly targeted biosolids land application in agricultural settings. We focused our efforts on Cu and Zn with the goal of identifying the dominant metal pools present in a semi-arid grassland soil receiving biosolids either once (in 1991) or twice (in 1991 and 2002), and to explain any discrepancies within or between various soil metal phases. Biosolids used in the current study contained Cu and Zn concentrations at an order of magnitude greater than other metals typically present in biosolids (e.g. Cd, Cr, Mo, Ni, and Pb), making it easier to detect and thus describe in the soils utilized. Our hypotheses were that both long-term (once) and short-term (twice) biosolids applications will: (a) cause persistent changes in soil Cu and Zn fractions, which will affect metal mobility, extractability, and fate; and (b) show the largest containing Cu and Zn pools residing in more resistant soil metal phases.

2. Materials and methods

2.1. Site description, biosolids, and soil sampling

The city of Fort Collins, CO, USA owns the 10,500 ha Meadow Springs Ranch (40 53′46″N, 104 52′28″W) and utilizes it for beneficial biosolids land application. In August 1991, 15 m × 15 m test plots were established at the Meadow Springs Ranch with treatments consisting of 0, 2.5, 5, 10, 21, and 30 Mg biosolids ha^{-1} surface-applied with no incorporation. All treatments were replicated four times in a randomized complete block design. In October 2002 the original plots were split in half. One-half received a second surface with no incorporation application using the same rates at the original plots, creating a split-plot in time study design.

Biosolids samples from the city of Fort Collins, Colorado wastewater treatment facility were collected before each application, kept refrigerated at approximately 3 °C, then total Cu, Zn, and additional metal composition was determined by $HClO_4$–HNO_3–HF–HCl digestion using inductively coupled plasma–atomic emission spectroscopy (ICP–AES; Table 1; Soltanpour et al., 1996). Biosolids pH was determined using a saturated paste extract (Rhoades, 1982), total N via LECO-1000 analysis (Nelson and Sommers, 1996; LECO Corp., St. Joseph, MI), NH_4–N and NO_3–N in a 2 M KCl extract (Mulvaney, 1996), and organic N via subtraction of inorganic from total.

The Meadow Springs Ranch is a semi-arid, shortgrass steppe rangeland community dominated by the perennial grasses blue grama (*Bouteloua gracilis* (H.B.K.) Lag. Ex steud) and western wheatgrass (*Pascopyrum smithii* (Rydb.) A. Love). The research site soil was an Altvan loam, fine-loamy over sandy or sandy-skeletal, mixed, mesic Aridic Argiustoll, 0–3% slopes. The Altvan series consists of deep, well-drained soils that formed in mixed alluvial deposits (NRCS, 1980). Three soil cores from each plot were collected in July 2003 from the 0–8, 8–15, and 15–30-cm depths, composited, placed in Ziploc® bags, then into coolers and returned to Colorado State University. Sampling depth was similar to that described by Harris-Pierce (1994). All soils were immediately air-

Table 1
Characteristics of the 1991 and 2002 Fort Collins, CO, USA biosolids applied to the Meadow Springs Ranch semi-arid rangeland experimental plots.

Parameter[a]	Soil	Biosolids application year	
		1991	2002
Zn (mg kg^{-1})	35	772	652
Cu (mg kg^{-1})	6.1	547	475
Pb (mg kg^{-1})	7.9	120	39
Cr (mg kg^{-1})	9.3	40	21
Ni (mg kg^{-1})	6.6	19	17
Mo (mg kg^{-1})	<0.1	16	19
Cd (mg kg^{-1})	0.3	5.0	2.6
pH	6.3	7.3	6.0
Organic N (%)	0.2	4.2	4.2
NH_4–N (mg kg^{-1})	5.4	3960	5440
NO_3–N (mg kg^{-1})	0.9	102	2.9

[a] All values are presented on a dry weight basis.

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