



Residual Effects of Long-term Biosolids Application on Concentrations of Carbon, Cadmium, Copper, Lead and Zinc in Soils of Two Regions of the United States

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ABSTRACT

Biosolids are used to improve soil physical and chemical properties. Analysis of biosolids-amended soils from multiple regions of United States using consistent analytical methods is lacking. This Study determined long-term biosolids application on soil pH, electrical conductivity (**EC**), carbon, cadmium, copper, lead, and zinc concentrations in soils from two regions in United States. At one region, little difference was observed in pH and EC between biosolids-amended surface soils and control, the second region ranged from 5.46 to 7.87 and 50.2 to 402 $\mu\text{S cm}^{-1}$ respectively. Trace metal levels at this region ranged from 0.76 to 3.79, 8.7 to 54.1, 15.2 to 53.9, and 26 to 207 mg kg^{-1} for **Cadmium, Copper, lead** and **Zinc** respectively; **with its** carbon levels ranging from 14.5 to 90.0 g kg^{-1} . Metal levels were within the standards for residential occupation with the 40 **Code of Federal Regulations** (CFR) Part 503 and should not affect soil and groundwater quality.

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Introduction

Biosolids consist of mainly organic material resulting from stabilized primary and secondary sludge from wastewater treatment facilities by the process of both aerobic and **anaerobic** digestions like lime stabilization, thermal drying, composting, etc. Characteristic of biosolids vary depending on the process used for pathogen reduction (Brown et al., 2003; Hirpassa and Codling 2019; Wang et al. 2008). Biosolids are rich in plant nutrients and have been used as soil amendments to improve soil physical and chemical properties (Hirpassa and Codling 2019). Biosolids have also been shown to improve soil fertility and increase soil organic carbon (Brown et al. 2011; Granato et al. 2004; Li and Evanylo 2013; Sharma et al. 2017; Wuest and Reardon 2016; Zhai et al. 2014). Wijesekara et al. (2017) observed a 45% increase in soil total organic carbon in the surface layer (0–15 cm) of a biosolids-amended soil, attributing the increase to a direct contribution from residual carbon from the biosolids application and to increased biomass production of crops grown on the amended soil. Biosolids may also increase water infiltration and reduce erosion due to the hydrological effects of biosolids on runoff (Moffet et al. 2005; Zartman et al. 2012). Although biosolids application is an effective method of supplying plant nutrients, increasing soil organic carbon, and reducing runoff and erosion, it can also increase heavy metal concentrations in soils (Codling 2014; Mossa et al. 2020). Barbarick, Ippolito, and Wesfall (1998) reported that after five applications of biosolids, significant accumulation of trace elements was observed in the plow layer but not (except for Zn) in the subsoil. Sloan, Dowdy, and

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Abbreviations: BC, Biosolids Compost; BCF, Biosolids Compost + Fertilizer; CFR, Code of Federal Regulations

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Dolan (1998) observed that **Cadmium** (Cd), **Chromium** (Cr), **Copper** (Cu), **Nickle** (Ni), **lead** (Pb), and **Zinc** (Zn) concentrations in biosolids-amended soil were much higher than in the control. However, some other studies reported that long-term biosolids application did not increase concentrations of heavy metals to toxic levels (Gaskin et al. 2003; Sukkariyah et al. 2005a) and even after a 17 year period of biosolid applications, there was negligible movement of trace metals through the soil profile and consequently little risk of contamination of ground water at the studied site (Sukkariyah et al. 2005b). Even a study on locations of high rates of biosolids observed results suggesting that the long-term application of biosolids at high loading rates does not result in trace metal pollution of groundwater (Oladeji et al. 2012). The degree of heavy metal accumulation in biosolids-amended soil depends on the biosolids source and on soil properties. For example, biosolids from sanitary wastewater generally have lower levels of heavy metals compared to those contaminated by industrial waste (Silveira, Alleoni, and Guilherme 2003). However, in the **United States**, industrial waste is omitted from wastewater treatment plant (WWTP) for the production of biosolids by the Wastewater Pretreatment Regulations; noting that a wastewater treatment plant is a facility in which a combination of various processes (e.g., physical, chemical and biological) are used to treat industrial wastewater and remove pollutants (Hreiz, Latifi, and Roche 2015). The purpose of this study was to determine residual concentrations of carbon and heavy metals in long-term biosolids-amended soils from two regions in the United States, using the same extracting methods for all samples.

Materials and methods

Soil collection

Soils for this study were collected from several sites in the states of New Jersey and Virginia with histories of biosolids application.

In the spring of 2017, soil samples were collected from agricultural fields and reclaimed lands at six locations in New Jersey: Hunterdon, Salem, Gloucester-E, Gloucester-H, Middlesex and Monmouth. Soil properties and histories of biosolids application as well as the cropping history of the fields are presented in Table 1. Soil samples were collected from two depths (0–15 and 15–30 cm) from each field/site. Each sample was a composite of six 1.5-cm diameter cores.

In the fall of 2017, soil samples were collected from two locations in Virginia with histories of biosolids application. Location I, on a Fauquier silt loam, was at the Northern Piedmont Research Station in Orange, where an experiment had been established in 1999 with a complete block design and four replications, where each plot was 3.6 m wide and 7.5 m long. Treatments sampled for the current study were a control (commercial fertilizer), biosolids compost (4.48–11.21 Mg ha⁻¹), and biosolids compost (4.48–11.21 Mg ha⁻¹) + fertilizer (Table 2). Location II was in Charles City County on a Pamunkey sandy loam soil. Each plot was approximately 36 by 15 m in size. Treatments were 0 (control), 14, 43, 70 and 98 Mg ha⁻¹ anaerobically digested biosolids (Table 2). Experimental methods were as described by Li and Evanylo (2013) for Locations I and II, (Evanylo et al. 2008; Spargo, Evanylo, and Alley 2006; for Location I), and Daniels et al. (2001) for Location II.

Soil analysis

The soil was air-dried, passed through a 2-mm mesh sieve and stored for analyses. Soil pH was measured in a 1:1 soil: deionized water suspension after 1 h of equilibration. Electrical conductivity (EC) was determined in a 1:2 soil: deionized water solution after 1 h of equilibration. For total carbon (C) determination, soil samples were ground using a mortar and pestle to < 0.25 mm and analyzed using a rapid carbon sulfur analyzer (Elementar, Hanau, Germany) for which 30 mg of ground soil and 30 mg of conditioner (Tungsten VI and Wolfram VI oxides) were wrapped in tin foil prior to measurement. The detection limit of the instrument was 0.01 mg C, corresponding to a C concentration of 0.03% in a 30-mg sample (Codling and Eickhoff 2012). Total elemental composition of the soil



Table 1. Field area, soil texture, rate and date of biosolids application, and crops grown on biosolids-amended soil in New Jersey.

Sites	Field area (ha)	Soil Texture	Pre-application Soil pH	Application Rate		Quantity (dry ton)	Biosolids Application Date	Previous Lime Application	Crops Planted
				(Mg ha ⁻¹)	Metric Tonnes				
Hunterdon	22.9	Clay loam	5.8–6.4	11.8	299	271	Fall 1999	Not known	Beans
Salem	10.1	Sandy loam	5.8	9.2	103	93.4	June 2000	3-year Prior	Leaf vegetable
Gloucester-E	20.2	Loamy sand	5.9	11.2	250	226	April 2000	Not known	Corn
Gloucester-H	23.5	Loamy sand	5.0	12.3	318	289	April 2001	5-year prior	Vegetables
Middlesex	60.7	Clay		50.9	3404	3088	Feb./Mar 1999	None	Native grass mix
Monmouth	0.16	Not avail.	4.1	1.37	23.8	21.6	Mar. 1996	None	Native grass mix

Table 2. Soil texture, rate and dates of biosolids application, and crops grown on biosolids-amended soils at two locations in Virginia.

Location	Soil Series	Treatments	Frequency	Crops History
Location I	Fauquier silty clay loam	1. Fertilized control 2. Biosolids compost at the agronomic N rate of 4.48–11.21 Mg ha ⁻¹ (BC) 3. Biosolids compost at the agronomic N rate of 4.48–11.21 Mg ha ⁻¹ + fertilizer to meet crop needs (BCF)	2000, 2002 2003, 2004	The crop included pumpkin in 2000, Sweet corn 2001, and bell pepper in 2002. Corn was grown in 2003–2004, and soybean was grown in 2005. Cereal rye was planted in the autumn 2000–2005 as winter cover crop. Tall fescue was planted in 2006–2011.
Location II	Pamunkey sandy loam	0 (fertilized control), 14, 42, 70, and 98 Mg ha ⁻¹ anaerobically digested biosolids	1996	Crops included corn 1996, wheat in fall 1996, Soybean 1997, and cotton in 1998 until 2017.

From Li and Evanylo (2012).

was determined using Aqua Regia digestion (McGrath and Cunliffe 1985). Briefly, 5 ml of concentrated nitric acid (HNO₃) and 15 ml of concentrated hydrochloric acid (HCl) were added to 10 g of soil and refluxed for 2 h. The mixture was heated to dryness, 20 ml 3 N HCl was added and heated for an additional 2 h, then filtered, and the filtrate was brought to a volume of 50 ml with 0.1 N HCl. Elemental concentrations in the solution were determined using an inductively coupled plasma optical emission spectrophotometer (ICP-OES). For quality control, all samples were run in duplicate with blanks and a certified **National Institute of Standards and Technology** (NIST) standard (Buffalo River sediment 2704, National Institute of Standards and Technology Gaithersburg, MD) included after every 10 samples (Hirpassa and Codling 2019). Analysis of variance was used to determine statistically significant differences among treatments (SAS 2012). Separation of means was performed using Duncan's multiple range test at $P \leq 0.05$ (Steel and Torrie 1980).

Results and discussion

New Jersey

The New Jersey study sites were six agricultural fields that had received single applications of biosolids between 1996 and 2001 (Table 1) prior to being sampled for this study in 2017. Soil pH in the 0–15 cm depth ranged from 5.46 at the Gloucester-E site to 7.87 at the Salem site (Table 3). Soil pH changed significantly with increasing soil depth only for the Harrison (+0.78 unit) and Sayreville (–1.61 unit)

Table 3. Soil pH, electrical conductivity, and concentrations of C, Cd, Cu, Pb and Zn in soils collected from biosolids-amended fields in New Jersey.

Sites	Depth	pH	EC μS cm ⁻¹	C g kg ⁻¹	Cd, Cu, Pb, Zn			
					Cd	Cu	Pb	Zn
					-----mg kg ⁻¹ -----			
Hunterdon	0–15	6.92 bct†	69.9 d	16.6 ef	3.79 a	18.2 c	24.3 c	95.3 c
	15–30	6.40 c	35.3 d	8.9 f	3.62 a	18.0 c	23.6 c	95.0 c
Salem	0–15	7.87 a	203 c	90.0 a	1.94 c	54.1 a	23.7 c	207 a
	15–30	7.81 a	186 c	74.0 b	1.66 c	51.8 a	18.0 cd	149 b
Gloucester-E	0–15	5.46 d	80.5 d	29.0 cd	0.81 d	8.7 de	18.2 cd	27.5 f
	15–30	5.66 d	81.5 d	17.9 ef	0.81 d	6.2 e	17.9 cd	26.4 f
Gloucester-H	0–15	5.66 d	50.2 d	14.5 ef	1.58 c	15.4 cd	15.2 d	39.6 ef
	15–30	6.44 c	39.7 d	10.9 f	1.73 c	10.4 c-e	13.7 de	41.0 d-f
Middlesex	0–15	7.08 b	234 bc	25.8 cd	0.76 d	18.5 c	14.8 de	30.7 ef
	15–30	5.47 d	163 c	10.3 f	0.98 d	12.8 c-e	8.6 e	20.4 f
Monmouth	0–15	7.33 ab	402 a	32.5 c	2.42 b	32.8 b	53.9 b	58.3 de
	15–30	7.33 ab	286 b	31.2 c	2.89 b	28.0 b	72.3 a	70.0 cd

† Within columns, treatment means having letters in common are not significantly different at $P \leq 0.05$.

sites. Soil EC in the 0–15 cm depth ranged from 50.2 at the Harrison site to 402 $\mu\text{S cm}^{-1}$ at the Monmouth site, where it declined significantly with increasing soil depth. The Monmouth site was located within the cloverleaf ramp of a major highway and may have been affected by road salt. Additionally, the biosolids applied to New Jersey soils were treated with lime kiln dust and quicklime (CaO) which increased the EC content of the biosolids and appeared to be reflected in the two sites with high application rates in Monmouth & Middlesex sites. Soil Total **Carbon** (TC) concentration for both the surface and subsurface depths was highest at the Salem site. There was a trend of decreased TC concentration with increasing soil depth, which was significant for the Salem, Gloucester-E and Middlesex sites. Averaged over soil depth, soil Cd concentration ranged from 0.81 mg kg^{-1} at the Gloucester-E site to 3.71 mg kg^{-1} at the Hunterdon site. In all cases, soil Cd levels were below the 39 mg kg^{-1} limit that requires notification before Cd-contaminated soil can be released for other uses. The Hunterdon and Monmouth sites had Cd concentrations that were considered safe for residential use (Sonon and Gaskin 2009) and all the elements measured had levels below what could be suitable for home gardens as stipulated under the US EPA 40 **Code of Federal Regulations** (CFR) Part 503.13. Soil Cd concentration was not significantly affected by soil depth. The variations observed in Cd concentration between sites may have been due to variations in the composition of the applied biosolids. According to Silveira, Alleoni, and Guilherme (2003), biosolids obtained from domestic sources have lower levels of heavy metals compared to those with significant contamination from industrial sources. Soil Cu concentration in the surface layer ranged from 8.67 mg kg^{-1} at the Gloucester-E site to 54.1 mg kg^{-1} at the Salem site, a value significantly higher than those of the other sites. There was a trend of decreased soil Cu concentration with increased soil depth, but differences were not significant. Soil Pb concentration, averaged over soil depth, ranged from 11.7 mg kg^{-1} at the Middlesex site to 63.1 mg kg^{-1} at the Monmouth site. The significantly higher Pb concentration at the Monmouth site relative to the other sites may have been due to residual Pb from the use of leaded gasoline, as this site was in a cloverleaf ramp near a major highway. Wuana and Okieimen (2011) stated that Pb from leaded gasoline can elevate Pb levels in soil along roadways. In all cases, Pb levels were below the value (75 mg kg^{-1}) considered safe for residential use (Sonon and Gaskin 2009). Soil Zn concentration in the surface layer ranged from 27.5 mg kg^{-1} at the Gloucester-E site to 207 mg kg^{-1} at the Salem site. The latter value, although significantly higher than those of the other sites, was nevertheless below the release notification concentration limit of 2800 mg kg^{-1} (U.S. Environmental Protection Agency 1993; U.S. Environmental Protection Agency 1995). This was also indicated in a general public safety guideline for home or public gardens where the safe levels were shown under heavy metals and gardens. Except for the Salem site, soil Zn did not vary significantly with soil depth. Averaged over sites, pH, EC, and soil C and Cu concentrations were significantly higher in the 0–15 cm depth compared to the 15–30 cm depth, whereas soil Cd, Pb and Zn concentrations were not significantly affected by soil depth (Table 4).

Virginia

At the two Virginia study sites, soil samples were collected in 2017 from plots of two agronomic experiments. At Location I, treatments were applied for 4 years (2000, 2002–2004) and consisted of biosolids compost (BC), biosolids compost + fertilizer (BCF), and a fertilizer control. At Location II, five rates of biosolids, including a zero-biosolids fertilized control, were applied in 1996.

Table 4. Soil pH, electrical conductivity, and concentrations of C, Cd, Cu, Pb and Zn in soils collected from biosolids-amended fields in New Jersey, averaged over soil depth.

Depth cm	pH	EC $\mu\text{S cm}^{-1}$	C gkg^{-1}	Cd	Cu	Pb	Zn
				----- mg kg^{-1} -----			
0–15	6.74 a†	139 a	30.6 a	2.05 a	22 a	21 a	77 a
15–30	6.39 b	105 b	20.6 b	2.08 a	19 b	19 a	68 a

† Within columns, treatment means having letters in common are not significantly different at $P \leq 0.05$.

Location I

Apart from the 15–30 cm depth of the BCF treatment, there was no significant difference in soil pH between the control and biosolids-amended treatments at Location I (Table 5). Soil pH also did not change with increasing soil depth. This might be due to the decomposition and cycling or use of the amendments. At each soil depth, soil EC did not differ significantly between the biosolids and control treatments, but EC decreased significantly with increasing depth for all treatments. At each soil depth, soil C concentration in the biosolids-amended treatments was greater than or equal to those of the control. Spargo, Evanylo, and Alley (2006) observed higher levels of soil C in these same plots in 2004. This decline in soil C concentration over time may have resulted from loss of C due to limited return of biomass to the soil from the types of crops grown at this site (Evanylo and Sherony 2002) as well as normal C mineralization. The Soil C concentration was slightly higher at the 0–15 cm depth when compared to the 15–30 cm depth and compares to the research results of (Li and Evanylo 2013). Soil Cd levels at this site were below the instrument detection limit, so no Cd data is presented. Soil Cu, Pb and Zn concentrations at this site were not significantly influenced by biosolids application. Averaged over soil depth, soil pH, EC and C differed significantly from the control only in the BCF treatment, which had higher values of all three variables (Table 6).

Location II

At Location II, soil pH at the 0–15 cm depth did not differ significantly between the biosolids-amended and control treatments, and neither was the soil pH significantly affected by soil depth (Table 7). At each soil depth, soil EC in the biosolids treatments was greater than or equal to the control value. Except at the highest biosolids rate (7xBS), soil EC decreased with increased soil depth. This was not considered an important variable to logically predict the behavior of biosolids long term application effects. This is because EC is very temporal, typically elevated in the spring/summer shortly after application of soluble fertilizer and reduced in late winter/early spring following winter leaching rainfall. Soil C concentration decreased with increased soil depth for all treatments. Soil Cd levels at this site were below the instrument detection limit, so no Cd data is presented. At the 0–15 cm depth, there was a trend of lower soil Cu concentration in the biosolids treatments compared to the control, but at the lower soil depth there were no significant differences among treatments. There was a trend of decreased soil Cu with increased depth. Soil Pb concentration at both depths did not differ significantly between the biosolids and control treatments. Soil Pb was significantly affected by soil depth only in the 3xBS treatment, where it was higher at the lower depth. Soil Zn concentration at each soil depth was not significantly affected by biosolids addition but it decreased with increased soil depth in all treatments. Averaged over soil depth, biosolids addition at all rates did not significantly affect soil pH and EC (Table 8). Soil C concentration in the biosolids treatments differed from the control only in the 5xBS treatment, which had a slightly higher value. Soil Cu concentration was lower in the biosolids treatments compared to the control. Soil Pb concentration was not significantly influenced by biosolids application. Soil Zn concentration in the biosolids treatments was less than or equal to the

Table 5. Soil pH, electrical conductivity, and concentrations of C, Cu, Pb and Zn in soils collected from biosolids-amended fields at Location I in Virginia.

Treatment	Depth	pH	EC μScm^{-1}	C g kg^{-1}	Cu	Pb	Zn
					----- mg kg ⁻¹ -----		
Control	0–15	5.93 b†	112 a	19.4 b	36.4 a	8.81 a	44.6 a
	15–30	5.91 b	62 c	12.8 c	34.6 a	8.61 a	44.5 a
BC	0–15	6.21 ab	105 b	20.1 b	36.1 a	8.45 a	49.7 a
	15–30	6.14 ab	60 c	19.4 b	35.3 a	8.17 a	44.5 a
BCF	0–15	6.26 ab	113 a	27.8 a	39.7 a	9.53 a	46.2 a
	15–30	6.36 a	75 c	12.9 c	35.3 a	8.73 a	44.5 a

† Within columns, treatment means having letters in common are not significantly different at $P \leq 0.05$.

BC = biosolids compost, BCF = biosolids compost + fertilizer.

Table 6. Soil pH, electrical conductivity, and concentrations of C, Cu, Pb and Zn in soils collected from biosolids-amended fields at Location I in Virginia, averaged over soil depth.

Treatment	pH	EC μScm^{-1}	C g kg^{-1}	Cu	Pb	Zn
				----- mg kg^{-1} -----		
Control	5.92 b†	87.5 b	16.1 b	35.5 a	8.71 a	44.6 a
BC	6.18 ab	82.5 b	15.9 b	35.7 a	8.31 a	45.3 a
BCF	6.31 a	105 a	20.4 a	37.7 a	9.13 a	47.1 a

† Within columns, treatment means having letters in common are not significantly different at $P \leq 0.05$.

BC = biosolids compost, BCF = biosolids compost + fertilizer.

Table 7. Soil pH, electrical conductivity, and concentrations of C, Cu, Pb and Zn in soils collected from biosolids-amended fields at Location II in Virginia.

Treatment	Depth cm	pH	EC $\mu\text{S/cm}$	C g kg^{-1}	Cu	Pb	Zn
					----- mg kg^{-1} -----		
FC	0–15	5.93 a-c†	85 b-d	13.8 a	12.4 a	16.8 ab	62.7 a
FC	15–30	6.15 ab	67 ef	7.1 de	8.0 b-e	17.2 ab	50.2 b
1xBs	0–15	5.93 a-c	88 ab	14.2 a	9.3 bc	16.2 ab	64.7 a
1xBs	15–30	5.96 ab	67 ef	8.2 cd	7.1 de	18.8 ab	46.6 b
3xBs	0–15	6.13 ab	95 a	14.0 a	8.9 b-d	15.6 b	51.8 a
3xBs	15–30	6.19 a	63 f	8.5 c	6.9 de	21.9 a	41.8 b
5xBs	0–15	5.89 b-d	87 a	14.1 a	7.1 de	15.8 b	61.0 a
5xBs	15–30	5.99 b-d	63 f	9.2 c	6.4 e	20.1 ab	40.7 b
7xBs	0–15	5.73 cd	77 c-e	12.2 b	10.3 ab	16.1 ab	65.4 a
7xBs	15–30	5.62 d	75 de	6.90 e	7.7 c-d	18.8 ab	46.8 b

† Within columns, treatment means having letters in common are not significantly different at $P \leq 0.05$.

FC = fertilized control, BS = biosolids.

control value. These results might signify the mineralization as well as the nutrient and biogeochemical cycling of these nutrients and metals.

Conclusions

Soil pH and EC values in biosolids-amended soils from two regions in the United States varied between locations and sites but were within the normal ranges for crop production. The levels of residual carbon and heavy metals remaining in the studied soils also varied with locations and application rates. The metal levels range from 0.76 to 3.79, 8.7 to 54.1, 15.2 to 53.9, and 26 to 207 mg kg^{-1} for Cd, Cu, Pb and Zn respectively, as well as having carbon levels ranging from 14.5 to 90.0 g kg^{-1} . The carbon levels were not consistent, as different sites received different levels of biosolids, but all the carbon levels were within the allowable limits. Generally, several accounts of biosolids research in different locations nationally, have a consistent conclusion of trace metals concentration and in some cases, possible downward movement in relation to the duration and levels

Table 8. Soil pH, electrical conductivity, and concentrations of C, Cu, Pb and Zn in soils collected from biosolids-amended fields at Location II in Virginia, averaged over soil depth.

Treatments	pH	EC $\mu\text{S/cm}$	C g kg^{-1}	Cu	Pb	Zn
				----- mg kg^{-1} -----		
FC	6.04 ab†	76.2 a	10.4 bc	10.1 a	17.0 a	56.0 a
1xBs	5.94 b	78.0 a	11.2 ab	8.4 bc	17.5 a	55.7 a
3xBs	6.16 a	79.6 a	11.3 ab	7.9 c	18.7 a	50.8 b
5xBs	5.89 a	75.0 a	11.7 a	6.8 d	17.9 a	51.0 b
7xBs	5.68 a	75.8 a	9.6 c	9.0 b	18.3 a	56.1 a

† Within columns, treatment means having letters in common are not significantly different at $P \leq 0.05$.

FC = fertilized control, BS = biosolids.

of applications, but do not necessarily affect their use because the metal concentrations are all below the standards in 40 CFR Part 503. Therefore, the concentration of metals in soils amended with biosolids will primarily depend on the rate and duration of applications as well as the type of crop/plant that would facilitate the biogeochemical cycling and turnover within that site.

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