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Agricultural use of tannery sludge (TS) may increase risks to soils. Thus, composting is recognized as one of the most suitable alternatives for TS recycling. Field experiments were carried out to evaluate the effects of composted tannery sludge (CTS) on chromium (Cr), cadmium (Cd), nickel (Ni), and lead (Pb) accumulation in soil after 3 years. Soil samples were collected 60 days after CTS application. After 3 years, The CTS increased Cr and Ni content, while Cd and Pb contents decreased. The third year, Cr contents showed linear increases as CTS rates were applied. The application of CTS, after 3 years, in the 2.5, 5, and 10 Mg ha^{-1} increased Cr significantly (140.7%, 159.7%, and 19%, respectively) and Ni (32%, 53%, and 43.8%, respectively) contents in the soil surface layer. This means that consecutive amendments of CTS increase Cr contents in the soil and plants.

Keywords Composting, heavy metals, industrial wastes

Introduction

Annually, tannery industries generate high volume of solid wastes, commonly known as tannery sludge (TS), which is disposed in sanitary sites without specific treatment (Silva et al. 2010). Due the tanning process, TS presents high organic and inorganic contents and significant amounts of hydroxides and carbonates (Selbach et al. 1991). Therefore, the agricultural use of TS may be one alternative for TS recycling (Araújo, Monteiro, and Carvalho 2007). The amendment of TS in the soil may promote plant growth by supplying nutrients, and increase soil pH with consequent reduction in exchangeable aluminum content (Santos et al. 2011). In addition, TS contains high organic matter content and it may improve soil physical and biological properties.

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However, TS contains heavy metals, obtained after tanning process, and they may restrict the use of TS in agricultural due to the environmental risks. Composting TS may be an appropriate alternative for TS recycling before application into the soil as reported by Araújo, Monteiro, and Carvalho (2007). Composting is a biological process of organic residues through the aerobic decomposition (Cai et al. 2007). This process is appropriate to eliminate pathogens (Costa et al. 2005) and to reduce wastes toxicity (Araújo and Monteiro 2006). Several studies were already conducted using urban and industrial wastes composted aiming to verify the effect of applications on the soil and the heavy metals accumulation (Zhang et al. 2000; Wey 2002; Wey and Liu 2005; Araújo and Monteiro 2006; Araújo, Monteiro, and Carvalho 2007). Zhang et al. (2000) evaluated the heavy metals, such as chromium (Cr), cadmium (Cd), and lead (Pb), in the soil and rape and barley plants after amendment of municipal solid waste compost and they found that the compost slightly increased heavy metals concentrations in the soil but did not cause any toxicity to plants. Also, Wei and Liu (2005) evaluated composted sewage sludge, after 3 years, on heavy metals accumulation in soil and plants. Heavy metals, such as copper (Cu) and zinc (Zn), were accumulated in the topsoil and in the barley grains and cabbage leaves (Wei and Liu 2005). Recently, Araújo, Monteiro, and Carvalho (2007) found that composted textile sludge did not promote negative effects on biological nitrogen fixation in soybean and cowpea in a tropical soil.

Therefore, the agricultural use of composted tannery sludge (CTS) implicates in the knowledge of its heavy metals content, after composting, and the behavior of these elements in the soil (Haroun, Idris, and Omar 2007) mainly after successive applications. The objective of this work was to evaluate the effect CTS, after 3 years of consecutive applications, on Cr, Cd, nickel (Ni), and lead (Pb) content in the soil and leaves of cowpea.

Materials and Methods

TS was collected from the wastewater treatment plant of a tannery located at Teresina city, Piauí State, Brazil. The compost was produced with TS a structuring material (sugarcane straw and cattle manure) mixed in the ratio 1:1:3 (sludge:sugarcane:cattle manure). The composting processes were carried out in our research facility using the Beltsville aerated-pile method (USDA 1980) for 85 days. The size of pile was 2 m long by 1 m wide by 1.5 m high. The pile was turned twice during the first and second week and once a week during the rest of the bio-oxidative phase. The bio-oxidative phase of composting was considered finished when the temperature of the pile was stable and near that of the surrounding atmosphere (30 °C). This stage was reached after 55 days of composting, and then the turnings were stopped to allow the compost to mature over a period of 30 days. The temperature increased quickly at the beginning of the process to high thermophilic values (70 °C), which contribute to the hygiene of the end product due to pathogen, weed, and seed reduction.

On day 85, 20 subsamples were randomly collected from compost to produce a composite sample. The chemical characteristics of CTS were determined by the Environmental Protection Agency (EPA) 3051 method (USEPA 1986) and are shown in Table 1. Bioavailable heavy metals content were determined by EPA method 3050B (USEPA 1986).

The experiment was carried out under field conditions at the "Long-Term Experimental Field" from Agricultural Science Center, Teresina, Piauí state (05° 05′ S; 42° 48′ W, 75 m). The regional climate is dry tropical (Köppen), and it is characterized by two distinct seasons: rainy summer and dry winter, with annual average temperatures of 30 °C and rainfall of 1,200 mm. The rainy season extends from January to April when

Table 1
Chemical properties of composted tannery sludge (CTS)

Properties	CTS 2009	CTS 2010	CTS 2011	Limits of heavy metals permitted[a]
pH	7.8	7.2	7.5	
C (g kg^{-1})	187.5	195.3	201.2	
N (g kg^{-1})	12.8	13.9	15.1	
P (g kg^{-1})	4.02	3.83	4.91	
K (g kg^{-1})	3.25	3.51	2.90	
Ca (g kg^{-1})	95.33	84.28	121.18	
Mg (g kg^{-1})	6.80	5.71	7.21	
S (g kg^{-1})	9.39	8.43	10.20	
Cu (mg kg^{-1})	17.80	19.51	16.38	1500
Zn (mg kg^{-1})	141.67	128.31	127.81	2800
Ni (mg kg^{-1})	21.92	28.61	23.26	420
Cd (mg kg^{-1})	2.87	3.93	1.93	39
Cr (mg kg^{-1})	2,255	2,581	1,943	1000
Pb (mg kg^{-1})	42.67	38.54	40.31	300

[a]CONAMA (2006).

90% of total annual rainfall occurs. The soil is classified as oxisol soil (clay, 10%; silt, 28%; sand, 62% at a depth of 0–20 cm). The average values of chemical properties in the 0–20 cm depth, at the first year, are presented in Table 2.

The experiments were conducted in 2009 and 2010 with six treatments: 0 (without TSC application), 5, 10, 20, and 40 t ha^{-1} of CTS (dry basis) and nitrogen (N) phosphorus (P) potassium (K) fertilizer. The NPK treatment was composed of 80, 60, and 40 kg ha^{-1} N, phosphorus pentoxide (P_2O_5) and potassium oxide (K_2O) (urea, super single phosphate, and potassium chloride, respectively). In 2011, CTS rates were reduced for 2.5, 5, 10, and 20 t ha^{-1} of CTS (dry basis). Thus, the accumulated rates were 0, 12.5, 25, 50, and 100 t ha^{-1}. The experiment was arranged in a completely randomized design with four

Table 2
Soil chemical properties (0–20 cm) at the first year of CTS application

Treatment (t ha^{-1})	pH	SOM (g kg^{-1})	P (mg kg^{1})	K (mg kg^{1})	Ca ($cmol_c$ kg^{-1})	Mg ($cmol_c$ kg^{-1})	Cr (mg kg^{-1})	Cd (mg kg^{-1})	Ni (mg kg^{-1})	Pb (mg kg^{-1})
0	6.7	7.27	3.57	43.0	0.93	0.28	2.69	0.07	0.42	2.15
2.5	7.3	6.30	1.94	35.2	1.04	0.22	4.86	0.08	0.50	2.36
5	7.8	6.30	1.13	82.1	1.03	0.17	6.43	0.17	0.41	2.71
10	7.7	7.30	4.33	35.2	1.06	0.17	17.3	0.17	0.31	2.35
20	7.8	6.50	1.87	78.2	1.04	0.16	30.3	0.18	0.54	2.84

SOM, soil organic matter.

replications. The plots were marked out (20 m^2 each and 12 m^2 of useful area for soil and plant sampling) including rows spaced 1.0 m apart.

In each year, CTS was applied 10 days before cowpea (*Vigna unguiculata*) sowing. It was spread on the soil surface with incorporation into the 20 cm layer with a harrow. The mineral fertilization was applied with cowpea sowing. N fertilization was divided and the side dressing was applied 30 days after sowing. Cowpea was grown at a density of 5 plants m^{-1} (about 62,000 plants ha^{-1}). Soil sampling was done 60 days after CTS application. Soil samples were collected at the 0–20 and 20–40 cm depth, using five subsamples per plot. The soil samples were dried, sieved (2.0 mm), and stored before analyses. Soil pH (1:2.5 soil:water), P, K, and magnesium (Mg) were evaluated according with Tedesco, Gianello, and Bissani (1995). Soil organic C (SOC) was determined by the Walkley–Black method (Walkley and Black 1934). Chromium, Cd, Ni, and Pb content were analyzed according to EPA 3050B method (USEPA 1996) after soil digestion with nitric acid (HNO_3), hydrochloric acid (HCl), and hydrogen peroxide (H_2O_2). The soil extracts were analyzed for Cr, Cd, Ni, and Pb by atomic absorption spectrophotometry. The leaves were collected in 10 plants inside the plots. Chromium, Cd, Ni, and Pb analyses were done according methods described in USEPA (1986).

The data were submitted to the analysis of variance (ANOVA) and the means were compared by the Student's test (5% level) and regression analyses. All the statistical analyses were performed with the SPSS (SPSS, Inc., Chicago, IL, USA) (version 15.0) software package.

Results and Discussion

The chemical properties of CTS used annually indicate the stability in your composition (Table 1). The C/N ratio of CTS ($\leq$ 25) corresponded to that of a matured compost (CONAMA 2006). The high values of CTS pH and calcium (Ca) content are related to hydroxides and carbonates used during tanning process (Selbach et al. 1991). The organic matter from animal leather contributes for high organic matter content in the CTS. The CTS showed high Cd, Pb, Cr, and Ni content (Table 1) and, except for Cr, these metals content are below the maximum limits established by CONAMA (2006). The concentration of Cr were 1.9–2.5 higher than the upper limits for Cr by CONAMA (2006) (Table 1). However, as observed in Table 2, soil pH increased above 7.0 as CTS rates increased and, when soil pH is alkaline, heavy metals stay inert in the soil under forms with low mobility (Hayes and Traina 1998). Specially for Cr, in pH values above 5.0, Cr is in the insoluble form of chromium hydroxide [$Cr(OH)_3$] (Aquino Neto and Camargo 2000) reducing the toxic potential. In addition, as the CTS pH is alkaline, Cr is found in the trivalent form (Cr^{3+}), which is more stable and has a low solubility and mobility (Alcântara and Camargo 2001).

The analyses of heavy metals in the soil (0–20 cm) at the first and after 3 years of CTS application showed significant increases in the Cr and Ni content (Table 3). The Cr content increased more than 100% with application of 2.5 and 5 Mg ha^{-1} of CTS, while Ni content showed lower increment than Cr after CTS application. This increase of Cr content in the soil is due to high Cr concentration in CTS. The Cd and Pb contents slightly decreased after 3 years of CTS application (Table 3). This probably occurred due to low content of these elements in CTS and the soil pH as reported by Costa et al. (2007).

Data in Table 3 shows that the metal concentrations (Cd, Ni, and Pb) were lower than the upper limits for soils (EU 1986) that is 4, 74, and 41 mg kg^{-1} soil. The upper limit

Table 3
Cr, Cd, Ni, and Pb in soil (mg kg^{-1}) at the first and after 3 years of CTS application

	CTS application		
CTS (t ha^{-1})	First year	After 3 years	Difference %
	Cr		
2.5	4.86 ± 1.2	11.7 ± 1.9	+140.7
5	6.43 ± 1.5	16.7 ± 3.5	+159.7
10	17.3 ± 3.4	20.6 ± 3.9	+19.0
20	30.3 ± 6.8	32.7 ± 7.2	+7.9
	Cd		
2.5	0.08 ± 0.01	0.05 ± 0.01	−60.0
5	0.17 ± 0.08	0.07 ± 0.02	−58.8
10	0.17 ± 0.06	0.06 ± 0.01	−64.7
20	0.18 ± 0.09	0.06 ± 0.01	−66.7
	Ni		
2.5	0.50 ± 0.09	0.66 ± 0.1	+32.0
5	0.41 ± 0.06	0.63 ± 0.09	+53.0
10	0.45 ± 0.08	0.65 ± 0.1	+43.8
20	0.54 ± 0.09	0.62 ± 0.08	+14.8
	Pb		
2.5	2.36 ± 0.8	1.81 ± 0.7	−23.3
5	2.71 ± 1.0	1.94 ± 0.9	−28.4
10	2.35 ± 1.1	1.91 ± 0.6	−18.7
20	2.84 ± 0.8	2.15 ± 1.0	−24.2

for Cr is not given by EU (1986). However, the lower upper limits concentration allowed in agricultural soils treated with sewage sludge is 250 mg kg^{-1} recommended by the EPA (Harrison, McBride, and Bouldin 1999). The Cr concentration is also lower than the upper limits in agricultural soils amended with sewage sludge.

The heavy metals accumulation in the leaves of cowpea, at the first and after 3 years of CTS application, showed significant increases in the Cr contents (Table 4), while that Cd, Ni, and Pb levels did not increase after CTS application. The increases of Cr in cowpea leaves were higher than 100% and these results suggest that the Cr was translocated from roots to leaves of cowpea, although Cr is, usually, retained in the plant root system (Dudka et al. 1991; Piotrowska, Dutka, and Gałczyńska 1991). According to Castilhos et al. (2001), the increase in the Cr concentration in the plant root promoted an increase of Cr in the leaves of soybean.

At the third year of CTS application, Cr contents in the soil showed linear increases at 0–20 cm depth (Figure 1), while that at 20–40 cm depth were not found increases in Cr content. In the case of Cd, Ni, and Pb, there were no changes in their concentration in both depths. The results showed that the Cr concentrated in the soil surface. Similar findings were found by Oliveira (2008) and Merlino et al. (2010) in tropical soils after 9 and 11 years of sewage sludge applications. These authors reported that Cr was insoluble or adsorbed to mineral soil surface.

Table 4
Cr, Cd, Ni, and Pb (mg kg^{-1}) in leaves of cowpea at the first and after 3 years of CTS application

	CTS application		
CTS (t ha^{-1})	First year	After 3 years	Difference %
	Cr		
2.5	1.12 ± 0.6	2.26 ± 0.9	+101.7
5	2.40 ± 1.0	5.05 ± 1.6	+110.4
10	3.20 ± 1.4	8.78 ± 2.1	+174.3
20	3.83 ± 1.2	7.55 ± 2.4	+97.1
	Cd		
2.5	0.53 ± 0.09	0.64 ± 0.1	+20.7
5	0.53 ± 0.1	0.65 ± 0.1	+22.6
10	0.54 ± 0.09	0.69 ± 0.08	+27.7
20	0.51 ± 0.07	0.63 ± 0.09	+23.5
	Ni		
2.5	1.83 ± 0.5	1.66 ± 0.6	−9.2
5	1.96 ± 0.7	1.82 ± 0.5	−7.1
10	2.06 ± 0.7	1.88 ± 0.3	−8.7
20	2.14 ± 1.0	1.92 ± 0.8	−10.2
	Pb		
2.5	3.36 ± 0.9	3.49 ± 1.0	+3.8
5	3.51 ± 1.0	3.67 ± 1.2	+4.5
10	3.55 ± 0.9	3.67 ± 0.9	+3.4
20	3.43 ± 1.1	3.55 ± 1.0	+3.5

The results found at the third year of CTS amendment showed that Cr concentration in the leaves increased as CTS rates were increased (Figure 2). According to equation of regression, the maximum content of Cr in the leaves was found with amendment of 14.6 Mg ha^{-1} CTS. These values of Cr content in the leaves of cowpea remained within the range considered as normal for levels of this element in plants, which ranges from 0.03 to 14 mg kg^{-1} (Kabata-Pendias and Pendias 2001).

These increases in Cr contents in the leaves of cowpea are associated with the increase in CTS rates amended to the soil. Our findings are in agreement with Gonçalves et al. (2013) who observed an increase in Cr content in cowpea leaves after application of CTS as a direct response for increase in CTS rates.

The contents of Cd, Ni, and Pb in the leaves of cowpea did not increase with the increases in CTS rates. It occurred due to low Ni, Cd, and Pb concentrations in CTS. These results are in accordance with Souza et al. (2005), which did not find significant effects of TS on Ni, Cd, and Pb concentrations in maize leaves. Similarly, Ferreira et al. (2003), after use of TS rich in Pb, also found low Pb content in leaves of soybean with values below 4 mg kg^{-1}. However, the average of values of Ni, Cd, and Pb found in cowpea shoot were 1.74, 0.65, and 3.61 mg kg^{-1}, respectively, and they are within the normal range for plant tissue proposed by Adriano (1986) for Ni and Kabata-Pendias and Pendias (2001) for Cd and Pb.

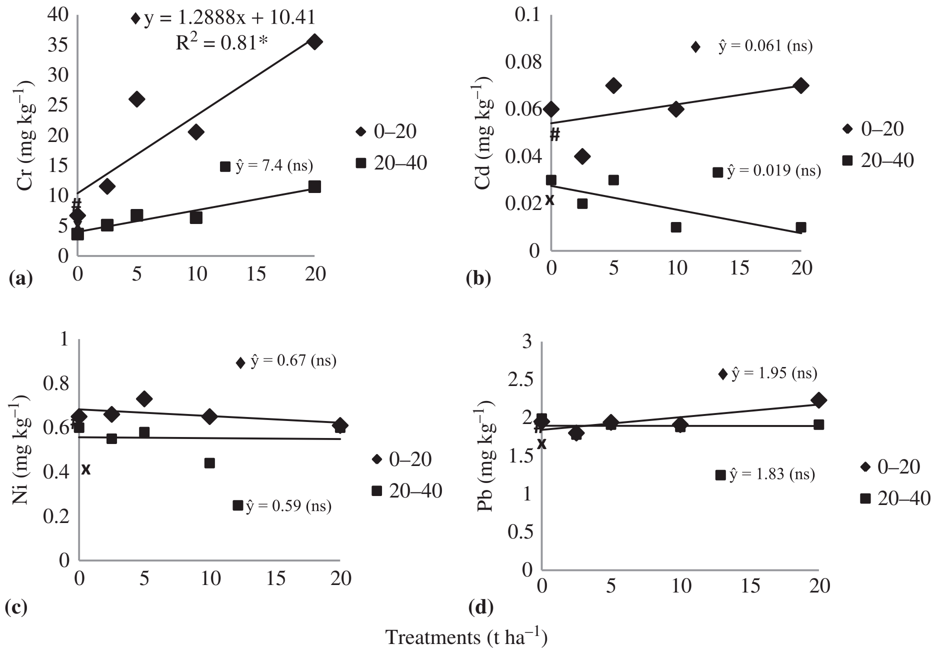

Figure 1. Cr (a), Cd (b), Ni (c), and Pb (d) contents in soil at the third year of CTS application. **#**—NPK (0–20 cm); **x**—NPK (20–40 cm). *5% significance; ns, non-significant.

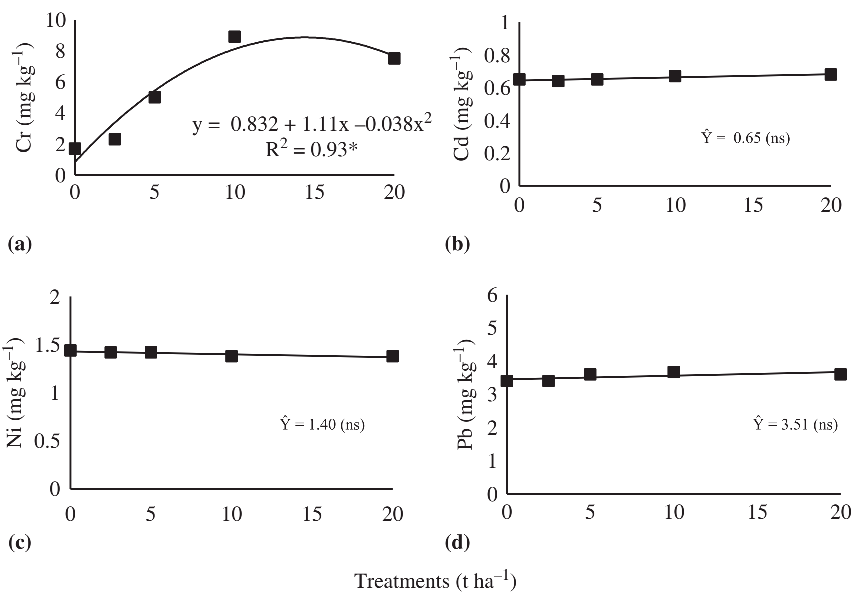

Figure 2. Cr (a), Cd (b), Ni (c), and Pb (d) concentration in cowpea leaves at the third year of CTS amendment. *5% significance; ns, non-significant.

Conclusions

The application of CTS, after 3 years, in the 2.5, 5, and 10 Mg ha^{-1} increased significantly Cr (140.7%, 159.7%, and 19%, respectively) and Ni (32%, 53%, and 43.8%, respectively) contents in the soil surface layer. After 3 years of CTS application, the Cr content increased more than 100% in the leaves. At the third year, Cr concentration in soil and leaves increased linearly as CTS rates increased. It means that consecutive amendments of CTS increase Cr contents in the soil and plants.

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