

Chemical, biological changes and mobility of heavy metals in soil treated with composted tannery sludge and cultivated with corn

Alterações químicas, biológicas e mobilidade de metais pesados no solo tratado com lodo de curtume compostado e cultivado com milho

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Abstract: The increase in industrial waste generation, especially tannery sludge, poses environmental challenges due to its organic matter content and the presence of heavy metals. Composting this waste has been pointed out as an alternative capable of reducing risks and expanding its potential for agricultural use. From this perspective, the study aimed to evaluate how composted tannery sludge (CTS) influences heavy metal levels, as well as the chemical and microbiological attributes of the soil, in addition to the development of maize (*Zea mays* L.) and the dynamics of these metals in different soil fractions. The experiment was conducted in a greenhouse, in pots, under a completely randomized design, using six doses of CTS (0 to 40 Mg ha⁻¹). The analyses covered soil chemical and biological parameters, metal fractionation, and plant biometrics. It was observed that CTS promoted an increase in pH, base saturation, organic matter, and microbial activity, which resulted in greater growth and biomass accumulation of maize. Despite the increase in metal levels, they remained below risk thresholds and showed low mobility, indicating the agronomic feasibility of the residue when managed with proper monitoring.

Keywords: Industrial waste; Soil attributes; Sequential extraction of heavy metals.

Resumo: O aumento da geração de resíduos industriais, em especial o lodo de curtume, impõe desafios ambientais devido ao seu conteúdo de matéria orgânica e à presença de metais pesados. A compostagem desse resíduo tem sido apontada como alternativa capaz de reduzir riscos e ampliar seu potencial de uso agrícola. Nessa perspectiva, o estudo buscou avaliar de que modo o lodo de curtume compostado influencia os teores de metais pesados, os atributos químicos e microbiológicos do solo, além do desenvolvimento da cultura do milho (*Zea mays* L.) e da dinâmica desses metais em diferentes frações do solo. O experimento foi realizado em casa de vegetação, em vasos, sob delineamento inteiramente casualizado, utilizando seis doses de LCC (0 a 40 Mg ha⁻¹). As análises abrangeram parâmetros químicos e biológicos do solo, fracionamento de metais e biometria da planta. Observou-se que o LCC promoveu aumento do pH, da soma de bases, da matéria orgânica e da atividade microbiana, o que se refletiu em maior crescimento e acúmulo de fitomassa do milho. Apesar do acréscimo nos teores de metais, estes permaneceram abaixo dos níveis de risco e apresentaram baixa mobilidade, indicando viabilidade agrônômica do resíduo quando manejado com monitoramento adequado.

Palavras-chave: Resíduo industrial; Atributos do solo; Extração sequencial de metais pesados.

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1. Introduction

Population growth and the intensification of urban and industrial activities have increased the generation of solid wastes, such as sewage sludge, municipal solid waste, and tannery residues. These materials may contain nutrients beneficial to agriculture, but also contaminants of high environmental concern, such as heavy metals and recalcitrant organic compounds (Hariyani *et al.*, 2025). Inadequate disposal of these wastes compromises soil, water, and biota quality, hindering their sustainable use.

Among industrial wastes, tannery sludge stands out due to its high volume and the complexity of its composition, resulting from the use of chromium salts in the tanning process (Kong *et al.*, 2020; Misganaw; Akenaw; Getu, 2024). This residue is rich in organic matter and nutrients, but presents significant levels of heavy metals, especially chromium, which represent both environmental risks and potential for agricultural use (Xu; Wu, 2022).

Composting has been identified as a promising alternative for tannery sludge stabilization, as it promotes the degradation of organic matter, reduces pathogens and phytotoxicity, and generates humic substances capable of complexing heavy metals, thereby decreasing their mobility and bioavailability (Perdigão; Marques; Pereira, 2022).

In tropical soils, characterized by high acidity, low base saturation, and reduced organic matter content, the application of composted residues can correct chemical limitations, increase water retention, and improve soil structure. However, the risk of heavy metal accumulation and their possible entry into the trophic chain requires careful monitoring (Sousa *et al.*, 2022). In addition, the addition of organic compounds may stimulate microbial diversity and activity, but the presence of metals may alter soil community responses, depending on their chemical form and the applied dose (Guo; Liu; Wu, 2022).

Maize (*Zea mays* L.), a crop of high economic importance and high nutritional demand, is sensitive to chemical and biological changes in the soil, constituting an appropriate indicator species to evaluate the agronomic and environmental effects of composted sludge application (Ademe; Guda; Lemma, 2020).

It was hypothesized that composting of tannery sludge reduces heavy metal toxicity, improves soil fertility and microbial activity, and promotes maize development. Thus, the objective was to evaluate the effects of composted tannery sludge application on: (a) heavy metal contents and soil chemical and microbiological attributes; (b) maize crop development; and (c) heavy metal dynamics and their interactions with soil attributes.).

2. Methodology

The soil was collected from a native forest area at Raposa Experimental Farm, belonging to the Federal University of Ceará, located in the municipality of Maracanaú–CE (3°50'66" S; 38°38'38" W), classified as a Red-Yellow Ultisol with sandy texture, according to the Brazilian Soil Classification System (EMBRAPA, 2011).

Sixteen disturbed subsamples were collected from the 0–20 cm layer using a Dutch auger, which were homogenized to obtain four composite samples. Soil physicochemical analyses were performed according to the methodology described by EMBRAPA (2011), including the following chemical attributes: pH in water, electrical conductivity, exchangeable cations (Ca^{2+} , Mg^{2+} , Na^+ , and K^+), potential acidity ($\text{H} + \text{Al}$), exchangeable aluminum (Al^{3+}), sum of bases, cation exchange capacity, base saturation, aluminum saturation, available phosphorus, organic carbon, total nitrogen, organic matter, and C/N ratio. Physical characterization included particle size distribution (total sand, fine sand, silt, and clay) and soil textural classification (Table 1).

Table 1 – Chemical and physical characterization of the soil in the experimental area of Raposa Farm at a depth of 0–20 cm.

Chemical characteristics		Physical characteristics (g kg ⁻¹)	
pH	4,70	Sand	461,00
CE (dS m ⁻¹)	0,24	Fine sand	318,00
Ca ²⁺ (cmolc dm ⁻³)	0,90	Silt	143,00
Mg ²⁺ (cmolc dm ⁻³)	0,90	Clay	78,00
Na ⁺ (cmolc dm ⁻³)	0,07	Natural clay	24,00
K ⁺ (cmolc dm ⁻³)	0,19	Textural composition	
H + Al (cmolc dm ⁻³)	3,30	Sandy loam	

Al ³⁺ (cmolc dm ⁻³)	1,40
S (cmolc dm ⁻³)	2,10
T (cmolc dm ⁻³)	5,40
V(%)	39,00
M(%)	40,00
PST	1,00
C (g kg ⁻¹)	2,88
N (g kg ⁻¹)	0,27
MO (g kg ⁻¹)	4,97
C/N	11,00
P (g kg ⁻¹)	4,00

Source: Authors (2026).

2.1 Experimental setup and management

The experiment was conducted in two stages: (i) composting of tannery sludge for 110 days in a closed system and (ii) incorporation of the compost into the soil and maize cultivation for 60 days. The pile consisted of tannery sludge, cattle manure, and sugarcane bagasse, in a volumetric proportion of 1:1:3, respectively (Silva, 2012), previously ground to increase the area of microbial activity. The materials were placed in 109 L drums with lateral perforations for aeration and were turned weekly. At the end, samples from four points of each pile were analyzed for physicochemical attributes.

The plots consisted of polyethylene pots containing 10 dm³ of soil, arranged in a completely randomized design with six treatments and four replications (24 units). The treatments were: T0 = 0.0; T1 = 2.0; T2 = 5.0; T3 = 10.0; T4 = 20.0; and T5 = 40.0 Mg ha⁻¹ of CTS. Planting occurred 10 days after CTS incorporation, by direct sowing of hybrid maize AG 1051 (4 seeds/pot). The pots were irrigated with deionized water at 85% of field capacity. After emergence, thinning was performed, maintaining one seedling per pot. Sixty days after sowing, at the beginning of the reproductive phase, the plants were collected and separated into shoots and roots.

2.2 Analysis of heavy metals and chemical attributes of the LCC and of the soil after LCC application

The pseudototal digestion of heavy metals was performed according to method 3051A (Florian; Barnes; Knapp, 1998), using 0.5 g of soil using 0.5 g of soil per sample, analyzed in four analytical replications (quadruplicate), as well as a certified reference material sample (NIST SRM 2709). Samples were treated with 9 mL of HNO₃ and 3 mL of concentrated HCl in Teflon tubes, in a closed microwave oven (1,000 W; preheating 5 min; 175 ± 5 °C for 10 min). The extracts were cooled, filtered (blue band), brought to 50 mL with ultrapure water, and stored in plastic flasks.

Chemical fractionation of metals followed the method proposed by Tessier, Campbell, and Bisson (1979), obtaining the exchangeable, carbonate, oxide, and organic fractions. The determination of manganese (Mn), iron (Fe), zinc (Zn), and copper (Cu) was performed by inductively coupled plasma optical emission spectrometry (ICP-OES), while chromium (Cr), lead (Pb), and nickel (Ni) were determined by atomic absorption spectrometry (AAS). The use of two instrumental techniques was defined according to the detection limits of each method and the expected concentrations of metals in the samples.

Analyses were performed using an ICP-OES spectrometer model ICP-OES model 8300, Perkin Elmer brand, and an atomic absorption spectrometer model Varian 220FS, Varian brand, for the determination of micronutrients and metals, and an atomic absorption spectrometer model Agilent 240FS, Agilent brand, for the determination of K, Na, Ca, Al, and Mg. Analyses of soil chemical attributes, pH, electrical conductivity, total organic carbon, sum of bases, organic matter, calcium, magnesium, potassium, nitrogen, phosphorus, and sodium adsorption ratio, after incorporation of composted tannery sludge, before and after maize cultivation, were performed according to the methodology described by EMBRAPA (2011).

2.3 Plant analyses

Biometric data of the maize crop, such as height and shoot and root dry mass, were obtained 60 days after plant emergence (Figure A–D). For this purpose, the plants were cut at soil level and then washed in running water and rinsed with distilled water in order to avoid the development of saprophytic agents. Subsequently, these samples were placed in paper bags and then subjected to drying in a forced-air circulation oven at controlled temperature (65–70 °C) until constant weight and, finally, weighed to obtain dry mass.

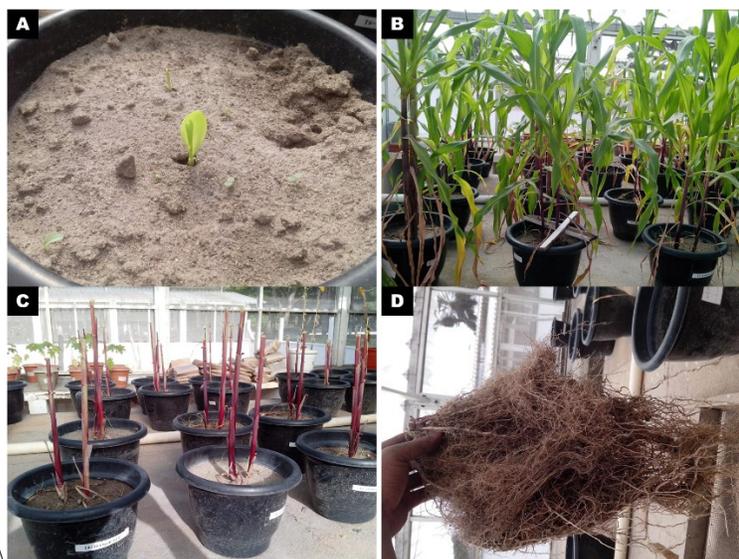


Figure 1 – Sequence of cultivation and fractionation of maize plants (*Zea mays* L.) to obtain the different plant parts. (A) Seedling emergence five days after planting. (B) Plants at 30 days of cultivation in pots, at the moment prior to sampling. (C) Stems after leaf removal. (D) Root system isolated after separation from the stems and leaves..

Source: Authors (2026).

2.4 Statistical analyses

Data were subjected to regression analysis to evaluate the influence of CTS doses on heavy metals, chemical and biological attributes, and maize growth. Pearson correlation ($p < 0.05$) was applied to verify the suitability of variables for Factor Analysis (FA). Subsequently, FA by principal components was performed with normalized data (mean = 0; variance = 1). Factors with eigenvalues > 1.0 were extracted, and Varimax rotation was applied, considering factor loadings ≥ 0.60 as significant.

The database was structured in Microsoft Access® (2024) and Excel® (2024), while analysis of variance and multivariate analyses were performed in RStudio® (2025.05.1+513) and STATISTICA® (v.13.3.721) (Hilbe, 2007; Lee, 2021).

3. Results and discussion

3.1 Chemical analysis of the residue

The results of the chemical analysis of the composted tannery sludge applied to the soil are presented in Table 2 and correspond to the mean \pm standard deviation of four analytical replicates (quadruplicate).

The contents of Cr, Cu, Zn, Ni, Fe, Pb, and Mn were below the soil quality guideline values (VRQs) for the presence of chemical substances, characterizing a condition of non-contamination, in accordance with CONAMA Resolution No. 420/2009. In this sense, the metals present in the residue did not exhibit toxicity levels that would prevent its agricultural use, indicating that composted tannery sludge may be an alternative for improving soil fertility, biota, and physical structure. However, caution is recommended regarding successive applications of the residue, since continuous use may promote gradual metal accumulation in the soil over time (Laik *et al.*, 2025).

Table 2 – Chemical analysis of tannery sludge (TS) and composted tannery sludge (CTS) after 110 days of composting.

Parameters	Limit of quantification	TS Results (mg kg ⁻¹)	CTS Results (mg kg ⁻¹)
Calcium	0,1	1.203,5	1.305,5
TOC	0,1	7.039,5	7.255,6
Lead	1,0	< 1	< 1
Copper	1,0	3,0	2,0
Chromium	1,0	9,0	5,0
Soluble Iron	1,0	11,0	9,0
Phosphorus	0,1	< 1	< 0,1
Magnesium	0,1	2.019,5	1.285,0
Maganese	1,0	4,0	3,0
Nickel	1,0	2,0	2,0
Nitrogen	0,1	274,1	170,6
pH	0,1	7,2	6,8
Potassium	1,0	17,0	24,0
Zinc	1,0	29,0	31,0

TOC – total organic carbon; TS – tannery sludge; CTS – composted tannery sludge. Values correspond to the mean ± standard deviation of four analytical replicates (n = 4). The analyses were performed according to the general guidelines of the Standard Methods for the Examination of Water and Wastewater.

Source: Authors (2026).

High levels of TOC, N, P, Ca, Fe, and Mg favored microbial activity, while the near-neutral pH maintained metals in stable and unavailable forms, a condition essential for maize development (Delibacak; Ongun, 2016). After 60 days of CTS application, significant effects of pH and SB were observed as a function of the rates (Table 3). The 40 Mg ha⁻¹ rate presented the highest values, attributed to carbon mineralization and OH⁻ production, which increased K⁺, Ca²⁺, and Mg²⁺ concentrations (Meena *et al.*, 2019). The dissolution of hydroxides and carbonates originating from the tanning process also contributed to the increase in pH, reducing H⁺ activity and increasing SB and CEC, which limits metal mobility and availability (Berilli *et al.*, 2015; Araujo *et al.*, 2020; Delibacak; Ongun, 2016).

Table 3 – Chemical soil attributes 60 days after the application of composted tannery sludge (CTS) rates.

Composted tannery sludge rates (CTS)	pH	EC	SB	K	CEC	SAR	N	P	TOC	SOM
.....Mg ha ⁻¹		(ds/)cmolcdm ⁻³g kg ⁻¹	
o Mg ha ⁻¹	5,7b	0,5d	2,7de	0,07a	2,8a	0,59c	0,8c	3,7f	7,2e	12,4d
2 Mg ha ⁻¹	6,0bc	0,6cd	3,1cd	0,07a	2,9a	0,67bc	1,3b	5,0ef	9,0cd	15,6c
5 Mg ha ⁻¹	6,2ab	0,7c	2,9cd	0,06a	3,0a	0,7ab	1,4b	5,6e	8,9d	15,4c
10 Mg ha ⁻¹	6,2ab	0,6cd	3,2bc	0,07a	3,0a	0,74ab	1,5b	7,7d	10,2bc	17,6b
20 Mg ha ⁻¹	6,3ab	0,7c	3,5ab	0,07a	3,1a	0,76ab	1,6b	10,6c	10,3b	17,8b
40 Mg ha ⁻¹	6,5a	0,8b	3,9a	0,08a	3,2a	0,79a	2,4a	16,9b	12,4a	21,4a

Values followed by the same letter in the column do not differ from each other by the Tukey test at 5% probability ($p \leq 0.05$). pH in water; EC = electrical conductivity (dS m⁻¹); SB = sum of bases (cmolc dm⁻³); K = exchangeable potassium (cmolc dm⁻³); CEC = cation exchange capacity (cmolc dm⁻³); SAR = sodium adsorption ratio; N = total nitrogen (g kg⁻¹); P = available phosphorus (g kg⁻¹); TOC = total organic carbon (g kg⁻¹); SOM = soil organic matter (g kg⁻¹).

Source: Authors (2026).

CTS contributed to increasing SOM and TOC with increasing rates (Table 3), an effect attributed to microbial decomposition of the organic compounds present in the residue (Araujo *et al.*, 2020). The highest N concentration was observed at the 40 Mg ha⁻¹ rate (Table 3). The increase in pH favored bacterial and fungal development, intensifying

organic matter decomposition and N release (Sousa *et al.*, 2017). Furthermore, the low C/N ratio of the residue facilitated N mineralization, making it more available to microbial biomass (Silva *et al.*, 2022).

CTS application also increased P contents, possibly due to OM mineralization or the cumulative effect of the rates (Table 3). However, these values were still below adequate concentrations for crop development, corroborating studies indicating low P concentrations in tannery sludge and the need for external supplementation (Silva *et al.*, 2022).

For K, no significant increase was observed (Table 3), due to the low concentrations in the residue (Haroun; Idris; Omar, 2009) and immobilization in microbial biomass, since this element is restricted to the living organic fraction and is not part of stable organic compounds (Novais *et al.*, 2007).

CTS application promoted a gradual increase in SAR values, especially at higher rates (Table 3); however, these values remained low and within ranges considered safe for agricultural use, not characterizing an immediate risk of sodicity. EC also showed a slight increase with increasing rates, remaining at low levels. Nevertheless, continuous monitoring is recommended in successive applications to avoid salt accumulation in the soil, as reported in other studies (Bilal Hussain *et al.*, 2024; Sousa *et al.*, 2022).

3.2 Heavy metal analysis

Quality control of the analyses was performed using the certified soil sample (NIST SRM 2709 San Joaquin soil) for heavy metals (Stahl *et al.*, 2016). Heavy metal recovery values for pseudototal solubilization ranged from 38 to 78% (Table 4), which are within the admissible range for most metals when compared with recovered values and, especially, with leachate values, 64 to 90% (Paye *et al.*, 2010).

Table 4 – Concentration range and mean content of certified heavy metals (NIST SRM 2709 San Joaquin soil) by the US Department of Commerce used in the analyses by the USEPA (3051 A) method.

Metal / Element	Determined value	Certified value (NIST) ¹	Recovery (determined) ²	Recovered by leachate ³	Leachate-based recovery ⁴
 mg kg ⁻¹		%	%	%
Mn	419,70	538 ± 17,00	78	87	90
Cr	49,70	130 ± 4	38	61	62
Fe	21470,70	35000 ± 1100	61	86	71
Ni	50,40	88 ± 5,00	57	89	64
Pb	11,60	19,8 ± 0,5	59	69	84
Zn	78,70	106 ± 3,00	74	94	79
Cu	24,16	34,6 ± 0,7	70	92	76

(1) NIST: National Institute of Standards and Technology. (2) % Recovery (determined) = (determined value/certified value) × 100. (3) % Recovery by leachate = (leachate median (NIST)/certified value) × 100. (4) % Recovery (determined) leachate-based = (determined recovery/leachate recovery) × 100. ND = values not determined by NIST (2002).

Source: Authors (2026).

In Figure 2, the effects of CTS rates on heavy metal contents in the soil after 60 days are shown. Regression indicated a significant effect of the residue, with increases in Cu (8.0–16.3 mg kg⁻¹) and Cr (4.6–25.9 mg kg⁻¹) at the 40 Mg ha⁻¹ rate (Figure 1A). This increase results from tanning salts released during decomposition, although the OM in the residue may immobilize metals via stable complexes with OH⁻ and COOH⁻ or insoluble organometallic compounds, reducing mobility and phytotoxicity (Gao *et al.*, 2023). Elevated pH and carbonates also restrict Cr, except in soils rich in MnO₂, where oxidation and increased mobility may occur (Dhal *et al.*, 2013; Wang *et al.*, 2021).

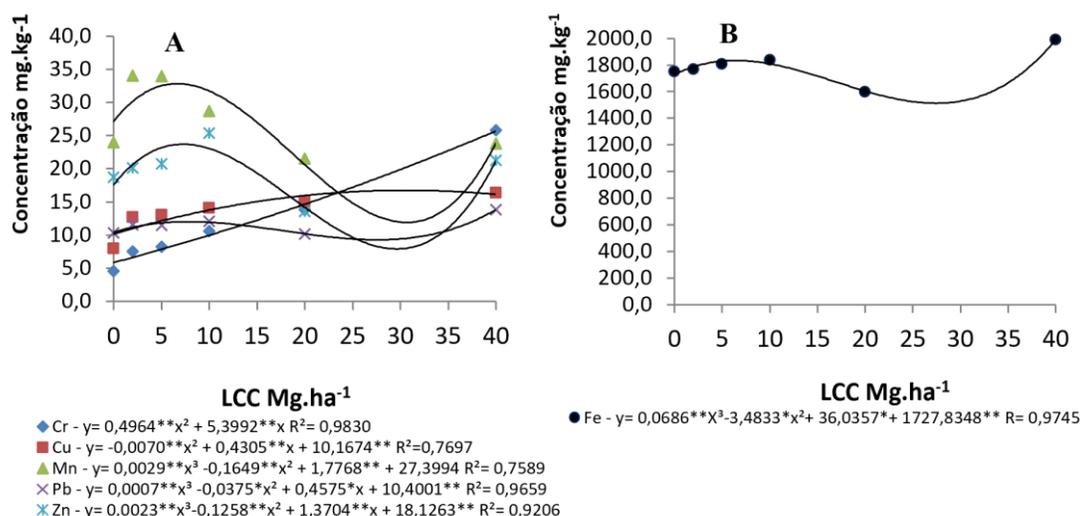
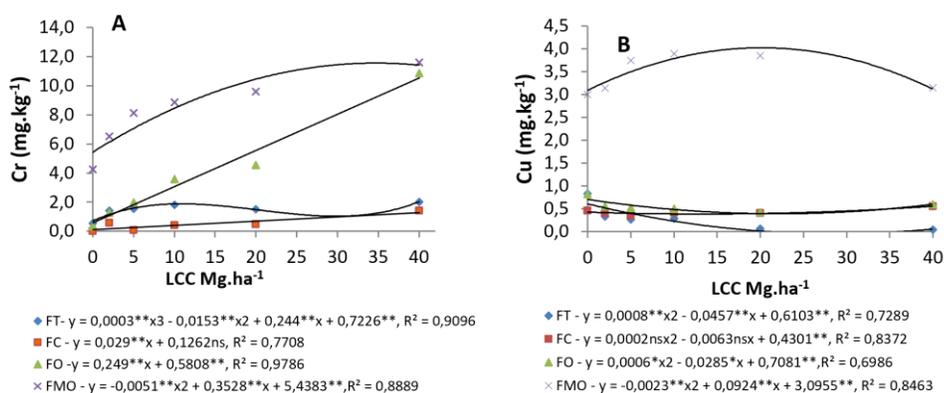


Figure 2 – Concentrations of semi-total metal contents 60 days after the application of composted tannery sludge (CTS). Source: Authors (2026).

CTS increased Mn, Zn, and Pb at the 2 and 5 Mg ha⁻¹ rates, but reduced them at 20 Mg ha⁻¹ (Figure 1A). Variations are associated with OM decomposition, which increases availability, and leaching, which reduces contents (Hashem *et al.*, 2021). At 40 Mg ha⁻¹, Mn, Zn, Pb (Figure 2A), and Fe (Figure 2B) increased, probably because they are associated with active/passive OM, reducing availability in soil solution (Gao *et al.*, 2023).

After 60 days, sequential fractionation showed that Cr was mainly concentrated in the organic fraction, but also increased in the oxide fraction with higher CTS rates (Figure 3A). At 40 Mg ha⁻¹, mean increases of 60% (organic) and 90% (oxide) compared to the control were observed, indicating complexation by organic acids at pH 5.0–8.0, maintaining Cr stable and poorly soluble (Fendorf, 1995; Xu *et al.*, 2020).



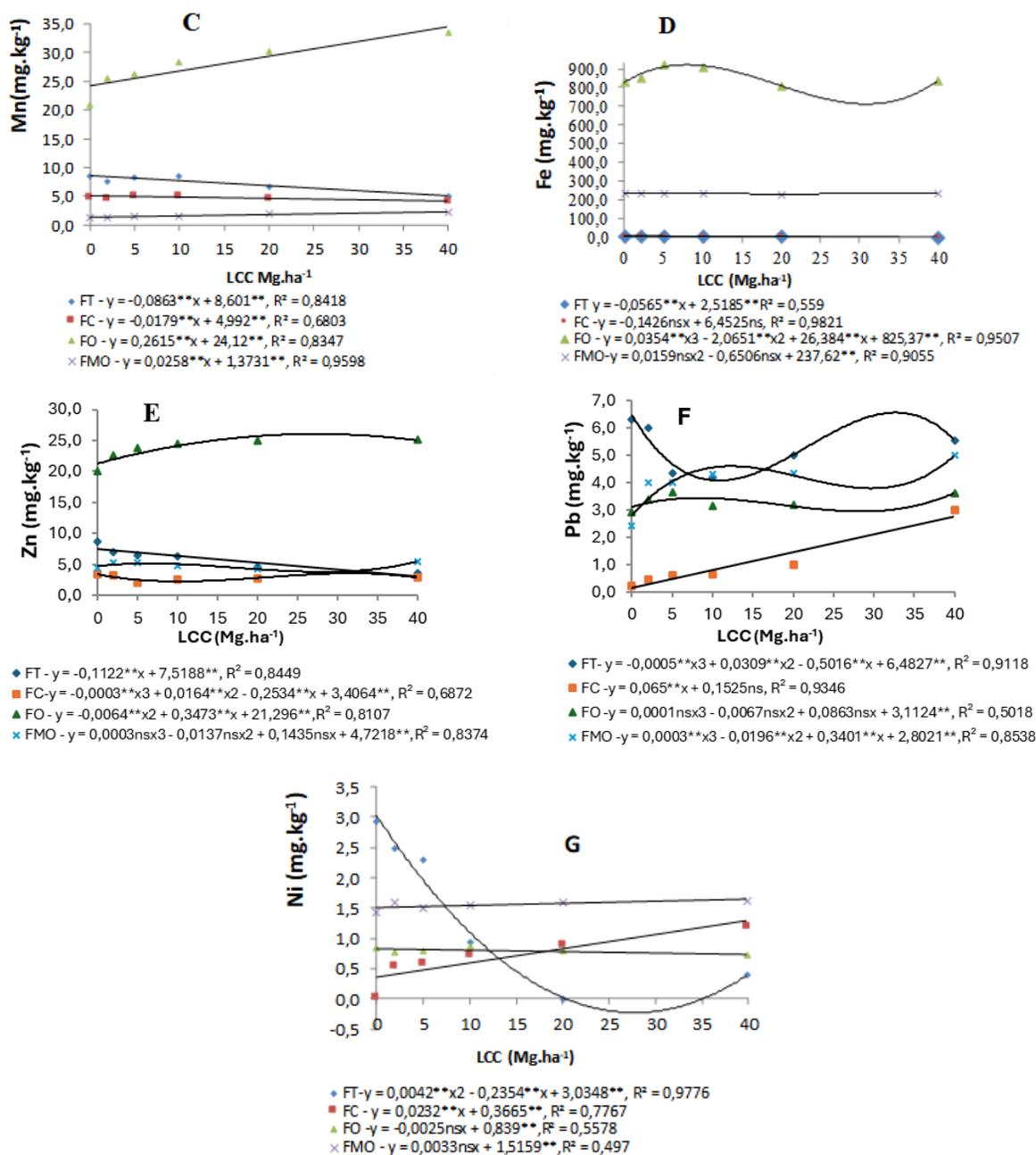


Figure 3 – Cr, Cu, Mn, Fe, and Zn contents in the soil fractions: exchangeable (F1), carbonate-bound (F2), oxide-bound (F3), and organic matter-bound (F4).

Source: Authors (2026).

Cu was concentrated in the organic fraction (Figure 3B), evidencing its affinity for humic substances (Dede *et al.*, 2023). Low pH increases its availability due to greater binding with OH⁻ and COO⁻, regulated by clay and CEC (Liu *et al.*, 2016). The low contribution in exchangeable, carbonate, and oxide fractions results from competition for adsorption sites and the strong binding of Cu to OM during composting (Escobar *et al.*, 2024). However, soluble complexes may increase the mobile fraction depending on OM quality (Arenas-Lago *et al.*, 2014).

Mn, Fe, and Zn were concentrated in the oxide fraction (Figures 3C–E), indicating low solubility and lower risk of toxicity (Dede *et al.*, 2023; CONAMA, 2009). Burt *et al.* (2003) also reported that Mn and Fe derive from natural minerals, not only from anthropogenic activity. Low concentrations in exchangeable and organic fractions reflect the instability of formed complexes. Zn, due to its affinity with Fe and Mn (Wydro *et al.*, 2021), was concentrated in oxides. However, values were below contamination limits (20–110 mg kg⁻¹; mean 90 mg kg⁻¹), since CTS contained only 31 mg kg⁻¹ (Table 2).

CTS also increased Pb and Ni in the carbonate fraction but had little influence on other fractions (Figures 3F and G), except for exchangeable Ni, which decreased to zero at 20 Mg ha⁻¹, due to increased pH and OM, intensifying competition for adsorption sites (Santos *et al.*, 2014). Despite this, the presence of Pb and Ni in carbonate fractions is concerning, as acidification may increase their mobility and toxicity. The low contribution in oxide and organic fractions is due to the low affinity of these metals (Kashem; Singh; Kawai, 2006).

3.3 Analysis of soil biological attributes

After 60 days of treatment application, regression analysis showed that residue addition significantly increased both soil microbial biomass carbon (MBC) and soil biological respiration (SBR), contributing to increased soil biota (Figure 4).

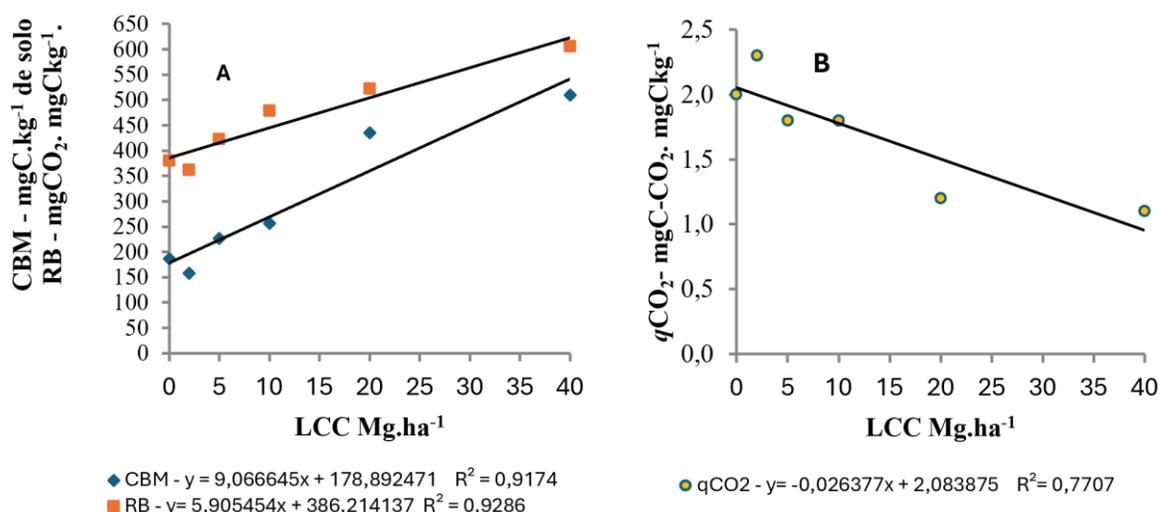


Figure 4 – Soil biological indicators 60 days after the application of composted tannery sludge (CTS) at the following residue rates: 0, 2, 5, 10, 20, and 40 Mg ha⁻¹. MBC – soil microbial biomass carbon; SBR – soil biological respiration; qCO₂ – soil metabolic quotient.

Source: Authors (2026).

After CTS application, stimulation of microbial biomass growth was observed (Figure 4A), due to the input of readily available C and N, improving nutritional conditions for microorganisms and favoring their growth. Similar results were reported by Lin *et al.* (2022), who associated increased MBC with organic matter mineralization and residue persistence in soil.

At the 2 Mg ha⁻¹ rate, MBC and SBR were reduced compared to the control, but values increased from 5 Mg ha⁻¹ onward (Figure 4A). Increased OM favors metal complexation, reducing availability and allowing greater microbial activity in residue decomposition (Souza; Miranda; Oliveira, 2009).

The metabolic quotient (qCO₂) decreased with increasing CTS rates (Figure 4B), indicating absence of adverse effects after 60 days and maintenance of microbial efficiency in OM cycling. According to Lin *et al.* (2022), low qCO₂ values reflect lower stress on the microbial community and absence of toxicity.

3.4 Biometric analysis of the corn plant

Different CTS concentrations significantly contributed to maize crop development, both in height and shoot and root dry mass (Figures 5A and 5B).

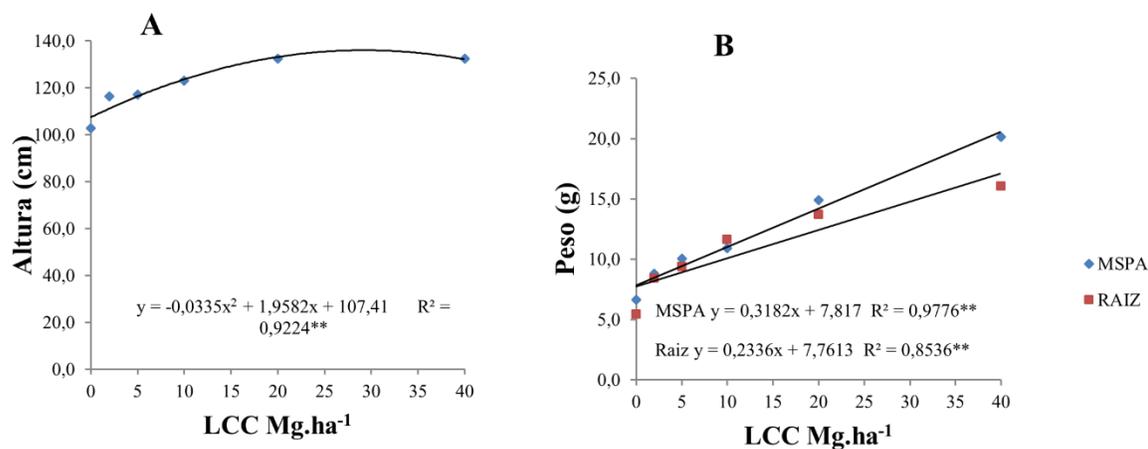


Figure 5 – Development of the maize crop 60 days after the application of composted tannery sludge (CTS). Source: Authors (2026).

Regression analysis indicated that, on average, maize height was higher at 20 Mg ha⁻¹ (132.4 cm) and 40 Mg ha⁻¹ (132.3 cm) (Figure 5), being statistically similar and superior to other treatments, demonstrating the benefit of high CTS rates for maize development.

For shoot and root dry mass (Figure 5B), a significant increase was observed with increasing CTS rates, with 40 Mg ha⁻¹ promoting the highest values for these characteristics. Improved soil conditions, both chemical and biological, provided by increased OM favored better nutrient cycling and increased activity of degrading microorganisms, making essential elements available for maize development (Elsalam *et al.*, 2021). Therefore, improved maize development can be attributed to greater N stabilization in the compost, promoting slow availability of this element, preventing losses and increasing absorption efficiency by the crop, directly reflecting in higher dry matter production (Sousa *et al.*, 2018).

Even with increased heavy metals as a function of increasing CTS rates, none of the studied metals were toxic and did not influence maize plant biometrics, remaining within permitted limits for agricultural use, allowing acceptable and safe application. In view of this, these results demonstrate the potential of the residue as a fertilizer for maize, mainly due to the presence of N, which is the main element involved in the synthesis of organic compounds of plant structure and is responsible for plant growth characteristics (Aline *et al.*, 2016).

3.5 Multivariate statistics

The suitability of the data matrix for multivariate analysis was evaluated based on correlations among variables, considering soil chemical attributes (pH, TOC, SOM, SB, CEC, EC, and SAR), heavy metal contents (Cu, Cr, Mn, Fe, Zn, and Pb), and biological attributes (SBR, MBC, and qCO₂), obtained in soil samples after CTS application. Correlations were determined by Pearson’s coefficient, applied to continuous quantitative variables with approximately normal distribution ($p < 0.05$), whose complete matrix is presented as supplementary material.

Subsequently, data were subjected to Principal Component Analysis (PCA), using eigenvalues greater than 1 as the extraction criterion. PCA identified three principal components, which jointly explained 83.66% of total data variance (Table 5), evidencing patterns of association among soil chemical and biological attributes and metal contents.

Table 5 – Factor axes for soil chemical and biological attributes and heavy metal contents for soil samples after the application of CTS, with their respective factor loadings, eigenvalues, total variance, and cumulative variance.

Variable	Component ⁽¹⁾		
	1	2	3

	Factor loadings ⁽²⁾		
Cr	-0,51	0,82	-0,07
Cu	0,07	0,95	-0,12
Mn	0,13	-0,52	-0,72
Fe	-0,41	-0,41	-0,67
Pb	-0,56	0,17	-0,55
Zn	0,39	0,48	-0,63
pH	0,29	0,92	-0,16
COT	-0,95	0,03	-0,06
CTC	-0,45	-0,27	-0,42
MOS	-0,96	-0,03	-0,07
SB	-0,29	0,89	-0,09
CE	-0,84	-0,46	-0,08
RAS	-0,85	0,09	-0,11
RBS	-0,93	0,15	0,08
BMS	-0,93	0,13	0,29
qCO ₂	0,84	-0,12	-0,44
Eigenvalues	7,18	4,25	1,96
Total variance (%)	44,85	26,57	12,25
Cumulative variance (%)	44,85	71,42	83,66

(1) Factor axes obtained by the Varimax method. (2) Factor loadings ≥ 0.60 were considered significant for interpretation purposes.

Source: Authors (2026).

The component matrix showed that PC1 positively correlated SOM, TOC, SBR, MBC, SAR, and EC, and negatively correlated qCO₂, explaining 44.85% of variance. This relationship indicates that microbiological attributes are influenced by soil fertility (Wang *et al.*, 2024). Organic residue addition increased OM, stimulating microbial activity, nutrient release, and fertility (Rashid *et al.*, 2018). The low correlation with metals is due to their low concentration in CTS or to the effect of exchangeable cations (Na⁺, Ca²⁺, Mg²⁺), which saturate OM charges and reduce metal adsorption (Zanello; Melo; Wowk, 2009). The negative correlation with qCO₂ reflects increased SBR and MBC and low metal contents, resulting in lower microbial stress.

PC2 grouped Cu, Cr, pH, and SB, responsible for 26.57% of variance. The association is linked to the effect of pH on charge release and greater retention of metallic cations. Increased pH increases SB by deprotonation of acidic groups in OM and OH⁻ groups in clay minerals, forming insoluble compounds with metals and favoring complexation (Kabata-Pendias, 2010). Thus, CTS increases OM and pH (Araujo *et al.*, 2020), reducing Cr and Cu availability in soil and their uptake by maize.

PC3 showed high loadings for Mn, Fe, Zn, and Pb, explaining 12.25% of variance. Although the input of these metals by CTS is low, the correlation results from geochemical affinity and association with Fe and Mn oxides/hydroxides (Goldschmidt, 1958; Dede *et al.*, 2023). Thus, Zn and Pb mobilized by the residue may subsequently be incorporated into these minerals. In the long term, continuous CTS use may result in accumulation of these metals in the soil.

4. Final considerations

The addition of CTS to the soil contributes to an increase in heavy metal contents; however, their concentrations remain below the maximum values permitted for agricultural use, as defined by CONAMA. Therefore, it can be used in agriculture

without short-term restrictions, provided that caution is taken with successive applications due to the possibility of increasing metal contents in the soil over the long term.

The metals Cr, Cu, and Ni generally showed greater association with the soil organic fraction, suggesting lower immediate bioavailability in the soil solution due to the formation of stable complexes with organic matter. Mn, Fe, and Zn exhibited greater association with the oxide fraction, a behavior widely reported in the literature and related to the strong affinity of these elements for Fe and Mn oxides and hydroxides, reducing their mobility under normal pH conditions.

Although lead (Pb) is considered geochemically low in mobility in soils, the results indicated its distribution in multiple fractions, including organic, oxide, and, to a lesser extent, the exchangeable fraction. This behavior does not characterize high intrinsic mobility, but rather reflects Pb sensitivity to soil conditions, especially variations in pH, organic matter content, and cation exchange capacity, as observed in similar studies.

The correlations observed between metal contents and soil chemical and biological attributes were mostly of low magnitude, indicating the absence of strong linear relationships among these variables under the evaluated conditions. This inference was based on correlation coefficients and patterns identified by Principal Component Analysis. It is noteworthy that more robust statistical analyses, such as redundancy analysis (RDA), could further deepen the understanding of these relationships in future studies.

Overall, the results do not allow the application of CTS to be classified dichotomously as either “safe use” or “risky use.” Rather, what emerges is a zone of agronomic feasibility, conditioned on the use of appropriate rates, temporal monitoring of soil attributes, and integrated interpretation of chemical, biological, and productive aspects. Under these conditions, CTS showed potential to improve soil quality and maize development, with no evidence of toxicity during the evaluated period.

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