Scientific Papers. Series A. Agronomy, Vol. LXVIII, No. 1, 2025
ISSN 2285-5785; ISSN CD-ROM 2285-5793; ISSN Online 2285-5807; ISSN-L 2285-5785

IMPACT OF DIGESTATE AND COMPOST APPLICATIONS ON SOIL CHEMICAL CHARACTERISTICS AND METALS AVAILABILITY IN A CONTAMINATED SOIL

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Abstract

Applying organic amendments such as digestate and compost is increasingly recognized as a sustainable strategy for improving soil quality, particularly in contaminated sites. This study investigates the effects of digestate and compost applications on the chemical characteristics of heavy metals-polluted soil and their influence on metal availability. The research was carried out in the greenhouse conditions, using maize as test plant. The main parameters analyzed were pH, organic carbon (Corg), total nitrogen (Nt), mobile phosphorus (P_{AL}), mobile potassium (K_{AL}), and the bioavailable forms of heavy metals (Pb, Cd, Cu, Mn, Ni, and Zn). Results revealed that both amendments enhanced soil pH, P_{AL}, and K_{AL}, reducing the bioavailability of heavy metals. The compost has proven to have very good performance in decreasing the mobility of heavy metals, mainly Cu, Mn, Ni, and Zn. This study highlights the suitability of these organic amendments for remediating polluted soils and improving soil fertility, offering a sustainable approach to land management in polluted areas.

***Key words**: digestate, compost, soil fertility, contamination/pollution, heavy metals.*

INTRODUCTION

Soil is a vital and limited resource. The economy, the environment, and the society depend on healthy soils, which hold over 20% of the planet's biodiversity, are the largest terrestrial carbon pool, and provide water and habitats for all living organisms (Decaëns et al., 2006; FAO, 2020; EC, 2021).
Veerman et al. (2020) estimated in their report for the European Commission (EC) that approximately 60 to 70% of soils in the European Union (EU) are not healthy, a statistic also detailed in the EC's Impact Assessment Report (EC, 2008).
Healthy soils are essential for achieving climate neutrality; therefore, the EU implemented a new Soil Strategy (EC, 2021), which aims to restore degraded soils and remediate contaminated sites. If effectively implemented, this strategy could significantly improve the health of EU soils. The European Environment Agency (EEA) estimated, in 2021, that around 2.8 million land sites were contaminated in the EU (EEA, 2022). Soil contamination can occur due to human activities or natural causes and involves different contaminants, such as pesticides, ammonia and nitrate, herbicides, petroleum hydrocarbons, and heavy metals (Neethu et al., 2021; Panico et al., 2023). The most frequent contaminants are mineral oils and heavy metals (EEA, 2022).
Heavy metal contamination presents a serious threat since the presence of metals such as lead (Pb), cadmium (Cd), copper (Cu), manganese (Mn), nickel (Ni) and zinc (Zn) can inhibit plant growth and contaminate the food chain, impacting both human and animal health (Tchounwou et al., 2012; Panagos et al., 2013; Jaishankar et al., 2014; Liu et al., 2018; Panico et al., 2023).
Various *in situ* and *ex situ* remediation techniques have been developed to restore heavy metal-contaminated sites, such as surface capping, encapsulation, landfilling, soil flushing, soil washing, electrokinetic extraction,

stabilization, solidification, vitrification, phytoremediation, and bioremediation (Khalid et al., 2017; Liu et al., 2018).

The immobilization of heavy metals in soil involves various physical and chemical processes that reduce their bioavailability and mobility. The main mechanisms by which heavy metals can be immobilized include: i) *precipitation* which is based on increasing soil pH by adding alkaline amendments contributing to the precipitation of heavy metals (Park et al., 2011a; Karalić et al., 2013); ii) *complexation* – organic amendments can form complexes with heavy metals, reducing their mobility and preventing their uptake by plants (Alam et al., 2020); iii) *adsorption* – the physical adsorption of heavy metals onto soil particles can lead to their immobilization (Nartey et al., 2014; Li et al., 2021); and iv) *microbial activity* – through processes such as biosorption and bioaccumulation, certain bacteria and fungi can uptake heavy metals and immobilize them in less harmful forms (Khalid et al., 2017; Azhar et al., 2022).

One effective, well-documented and tested approach to mitigate soil contamination is the application of organic amendments (Khan et al., 2004; Khalid et al., 2017). These materials improve soil structure and fertility (Sobhi et al., 2024) and play a vital role in immobilizing heavy metals, reducing their availability to plants (Park et al., 2011a; Omokaro et al., 2024). The use of organic amendments (OAs')is increasingly seen as a viable strategy for remediating contaminated soils, aligning with the EU's Soil Strategy to restore degraded lands (EC, 2021).

As is already known, the supply of organic matter contributes to improving soil's physical and chemical properties as well as soil's biological activity (Bolan et al., 2014; Khalid et al., 2017). Additionally, organic matter (OM) acts on the mobility of heavy metals in the soil, thereby reducing their negative impact on soil microorganisms (Venegas et al., 2015; Khalid et al., 2017).

In the 1990s, one of the most polluted sites in Europe was the city of Copșa Mică, and it remains the most polluted city in Romania until present. Two factories, Carbosin, which produced carbon black, and Sometra, a non-ferrous metallurgical smelter, were behind this pollution. Carbosin shut down in 1993, but the smelter is still operational (Vrînceanu et al., 2009; Muntean et al., 2010; Stanescu et al., 2017).

The objective of this study was to evaluate the comparative effects of digestate and compost applications on the chemical characteristics of metal-contaminated soil and their influence on metal availability. The assessment of metal availability was carried out only by using the specific chemical extraction with DTPA - TEA - $CaCl_2$ (Diethylenetriaminepentaacetic acid – Triethanolamine – Calcium chloride solution). The experiment is part of a research work that aims to restore soil health by reducing heavy metal mobility, improving nutrient reserves, and ensuring favorable conditions for plant development in contaminated soils.

The effects of the OAs on important soil indicators, such as soil pH, N, C_{org}, P_{AL}, K_{AL}, and the content of heavy metals (in accessible forms to plants), were evaluated within the experiment.

MATERIALS AND METHODS

Soil and organic amendments' physiochemical characteristics

The soil used for our study was collected from a 0-20 cm depth in an Aluvisol soil type, in an area heavily impacted by heavy metal contamination. This agricultural land is located approximately 1,500 meters from the exhaust chimney of the former Sometra industrial facility in Copşa Mică. The soil samples were air-dried and milled, and the chemical characteristics and heavy metals content were analyzed (Table 1).

The OAs' and the soil used in the experiment were chemically analyzed for their nutrient content (N, P, K, and OM), contaminants (heavy metals), and total salt content (TSC). The results are also presented in Table 1.

The digestate used was produced by the Alexandru Baciu farm's biogas production unit, and the compost was obtained by co-composting sheep manure with pruning lavender residues and residues from the distillation of lavender stalks, as described by Jafri et al. (2024). The soil is characterized by a low nutrient content and a high content of Cd, Pb, and Zn. The Cd, Pb, and Zn content of the soil used in our experiment exceeds the alert thresholds for sensitive use of land imposed by Order No. 756/1997 issued by the Ministry of Waters, Forests and Environmental Protection (3 mg/kg for Cd, 50 mg/kg for Pb, and 300 mg/kg Zn).

It should be noted that the values of C_{org}, OM, and nutrients (P and K) content established for the two types of OA are significantly different.

Table 1. Loss-of-ignition (LOI), chemical characteristics, and heavy metal contents of soil and OAs used in the experiment

Parameter	U.M.*)	Values		
		Soil	Compost	Digestate
pH		6.12	8.03	8.13
DM	%	-	56.03	26.84
LOI	%	-	46.45	51.30
OM	%	-	46.77	89.31
N_t	%	0.12	1.85	2.13
C_{org}	% dm**)	1.135	16.1	38.9
P	% dm	-	1.35	0.58
K	% dm	-	3.88	1.29
P_{AL}	mg/kg	29.5	-	-
K_{AL}	mg/kg	83.0	-	-
Cd	mg/kg	14.8	0.515	0.246
Cu	mg/kg	46.6	27.9	219
Cr	mg/kg		25.6	4.31
Ni	mg/kg	24.5	18.1	4.04
Mn	mg/kg	511	447	138
Pb	mg/kg	339	17.9	0.762
Zn	mg/kg	708	185	99.5

*) U.M. – unit of measurement
**) dm – dry matter

Soil and OAs' chemical analysis

The soil and OAs' samples underwent air-drying and grinded and were then passed through a 2 mm sieve prior to chemical analysis. The soil and OAs' physicochemical characteristics and heavy metal content were analyzed using ICPA methodologies (1981, 1983, 1986). The pH was measured potentiometrically using a pH meter in a 1:2.5 w/v soil and 0.5:2.5 w/v OAs' and deionized water extract. The analysis of dry matter (DM), loss on ignition (LOI), and organic matter (OM) was performed using the ICPA methodology (1983). Total nitrogen (N_t) was determined using a Vario MACRO Cube Elemental Analyser. P_{AL} and K_{AL} were determined in ammonium lactate acetate extraction. For P_{AL}, a colorimetric method was used, while K_{AL} was determined using flame atomic emission spectrometry (STAS 7184 19-82) based on the Egner-Riehm method (Tănase et al., 2022). C_{org} content was determined by the Walkley-Black titrimetric method, and nitrate nitrogen (NO_3^--N) was determined potentiometrically according to the ICPA methodology (1983).

The pseudo-total concentrations of metals were determined in the soil samples collected before the application of treatments using atomic absorption spectrometry after extraction by the aqua regia-microwave digestion method. Microwave digestion was performed using the Ethos Easy Microwave Digestion System, with 10 mL of aqua regia (7.5 mL HCl and 2.5 mL HNO_3) at 140°C for 30 min. The method was developed according to SR ISO 11466:1999. The same method was used for the analysis of the pseudo-total metal concentrations in the OAs' (Vrînceanu et al., 2019).

DTPA-extractable heavy metals (Pb, Cd, Cu, Mn, Ni, and Zn) were obtained by extracting 10g of soil with 20 ml of an extraction solution (0.05 M DTPA, 0.01 M $CaCl_2$, and 0.1 M tetraethylammonium, buffered to pH 7.3) following the SR ISO 14870:2002 and SR ISO 11047:1998 protocols. The extracted samples were then analyzed for available heavy metal content using a GBC Avanta 932AA flame atomic absorption spectrometer (Vrînceanu et al., 2017).

All laboratory analyses were conducted in triplicate, and the reported results represent the averages of these three replicates.

Pot experiment

A pot experiment was conducted in the greenhouse of the National Research and Development Institute for Soil Science, Agrochemistry and Environmental Protection – ICPA Bucharest, Romania. The soil was thoroughly mixed with 20 g, 40 g, and 80 g (equivalent to 15 t/ha, 30 t/ha, and 60 t/ha) of each OA, resulting in six treatments. The experiment was carried out using a randomized design with three replicates for each treatment. Additionally, two control pots were made, resulting in a total of twenty pots. Felix hybrid of maize (*Zea mays* L.) from the National Research and Development Institute for Agriculture Fundulea was used as a test plant.

The soil used for sowing was watered before planting; after that, 10 seeds were planted per pot. The plants were watered regularly to maintain the soil moisture content.

After successful germination, the plants were thinned out, with five plants remaining per pot.

At the end of the testing period, all plants were harvested, and soil samples were taken from each pot for further analysis.

Statistical analysis

Analysis of variance (ANOVA) was performed for statistical analysis, followed by Tukey`s range test (honesty significant difference). The variability in the collected data was indicated by the standard deviation ($p < 0.05$).

RESULTS AND DISCUSSIONS

The effects of OAs application on pH, N_t, N-NO_3, C_{org}, P_{AL}, K_{AL}, and TSC

Organic amendments typically influence various soil parameters, such as pH, Nt, N-NO_3, Corg, P_{AL}, K_{AL}, and TSC.

pH

Organic amendments can alter soil pH, which in turn can enhance the immobilization of heavy metals, as it is a factor governing the behavior of heavy metals in the soil-plant system (Hossain et al., 2010; Uchimiya et al., 2010; Park et al., 2011b). The pH of the soil from our experiment changed regardless of the dose or type of OAs applied. Significant increases in pH values were noted due to the increase in the quantity of OAs' regardless of its type, starting with the application of an equivalent amount of 30 t/ha. However, the application of compost, regardless of the applied dose, resulted in a significant increase in pH values compared to digestate (Figure 1). (De Vries & Breeuwsma, 1987; Kingery et al., 1994; Zaller & Köpke, 2004; van Herwijnen et al., 2006; Chang et al., 2007; Das et al., 2023).

N_t, N-NO_3, and C_{org}

The immobilization of heavy metals can be influenced by N_t, C_{org}, and N-NO_3. Their interactions facilitate microbial activities, enhance the formation of stable complexes, and contribute to the improvement of soil structure. As detailed by Liu et al. (2021), these elements facilitate metal immobilization through multiple mechanisms, including chemical stabilization, wherein heavy metals are transformed into less soluble and less toxic forms, and physical encapsulation, whereby heavy metals are sequestered within a low-permeability matrix (Liu et al., 2021).

No significant variations in soil nitrogen content were observed in our experiment, regardless of the amendment type (Figure 1). Consistent with findings from similar studies, nitrogen derived from OAs' is not readily available to plants, with only 5–15% of the N_t input from compost becoming plant-accessible (Reimer et al., 2023). The application of digestate and compost exerted comparable effects on the soil's C_{org} content as those recorded for N_t content. As illustrated in Figure 1, there were no significant differences between the compost and digestate treatments, regardless of the application rate. However, comparison to the unfertilized control, a significant increase in soil nitrogen content was recorded in the treatment corresponding to an OA application rate equivalent to 60 t/ha.

The N mineralization rate in the soil fertilized with OAs is reflected in the N-NO_3 content. In this experiment, an increase in the N-NO_3 content values was noted up to the application of quantities equivalent to the dose of 30 t/ha. After applying the equivalent amount of 60 t/ha of organic fertilizer, both in terms of compost and digestate, a decrease of N-NO_3 content was observed to values similar to those determined in the control (Figure 1).

P_{AL} and K_{AL}

Analyzing the variation in the P_{AL} and K_{AL} contents of the soil treated with the two types of fertilizer, a sharp increase in the values of these nutrients is observed due to the increase in the amounts of compost (Figure 1). Moreover, a significant increase in P_{AL} content values is noted after applying compost (regardless of the applied dose), approximately 2.6 times higher than the value obtained after applying digestate. The significant increases in the values of the P_{AL} content compared to the control are mainly due to the P input from the compost. It is noted that when quantities of compost equivalent to 60 t/ha were applied, the increase compared to the control was over 3.7 times.

In the case of K_{AL}, applying compost resulted in significant increases compared to digestate application, regardless of the applied dose. A sharp increase in the values of the K_{AL} content, compared to the control, is observed even from the application of the first dose of compost (Figure 1).

The application of OAs' generally enhances the availability of P and K in soils. The combined effect of P_{AL} and K_{AL} can enhance the immobilization of heavy metals. Phosphorus aids in transforming metals into insoluble complexes, while potassium supports plant health and influences soil chemistry. Together, they enhance microbial activity, improve soil structure, and create a conducive environment for effective heavy metal immobilization, resulting in the formation of stable metal-organic complexes that significantly lower metal mobility (Radziemska et al., 2020). Different studies have shown that high phosphorus levels can effectively bind heavy metals to soil organic matter, reducing their availability (Ruby et al., 1994; Hettiarachchi et al., 2000; Basta et al., 2001; Cao et al., 2002; Maenpaa et al., 2002; Borlan et al., 2003; Zwonitzer et al., 2003; Amanullah Mahar et al., 2015; Seshadri et al., 2017; Andrunik et al., 2020), and mobilize heavy metals, such as Pb, Cd (Chen et al., 2024), and Zn (Andrunik et al., 2020) into less soluble forms a phenomenon also observed in our experiment (Figure 2).

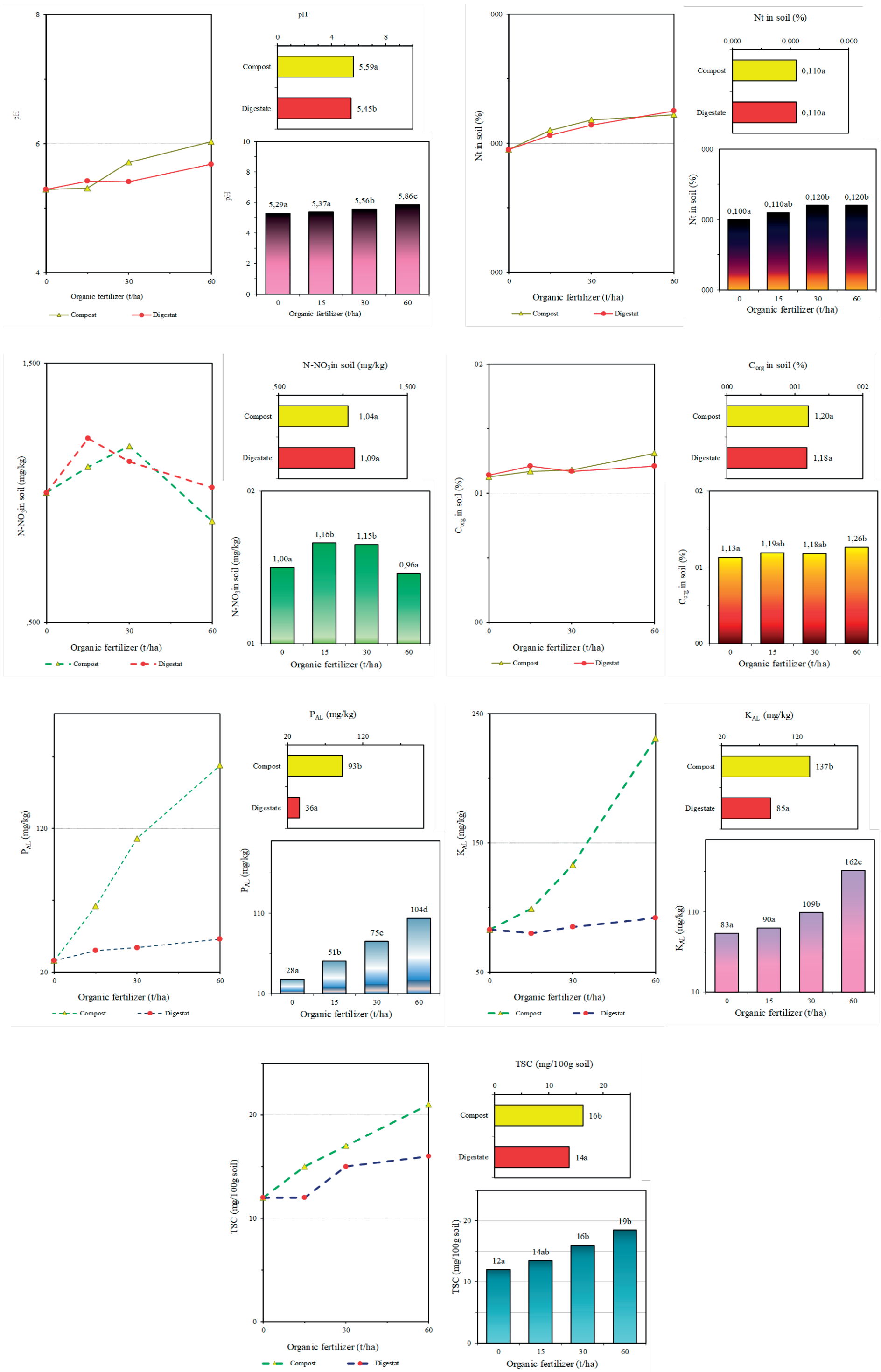
The application of P and K has been shown to influence soil pH, where the resulting increase in pH from phosphorus and potassium amendments tends to enhance the precipitation of heavy metals (Liu et al., 2021; Ou et al., 2018), in agreement with the observations of the present study.

Total salt content

Compost and digestate application can also introduce significant amounts of salts into soils, limiting plant development. Therefore, it is important to monitor this indicator in the case of long-term application of OAs, such as compost or digestate. Regarding the data obtained through this experiment, it was observed that, in the case of application of compost, TSC increased 0.8 times compared to digestate (Figure 1). Regardless of the type of OA used, increasing the applied dose led to raised values of TSC compared to the control, starting with applying a quantity equivalent to the dose of 30 t/ha.

The effects of OAs' application on heavy metals

When compost is applied over a long period of time, the issue of accumulation of potentially toxic elements such as Zn, Ni, and Cu may arise. While plant uptake of these contaminants is not frequently measured when compost is applied, their accumulation in the soil is a risk often associated with compost application (Amlinger et al., 2003; Weissengruber et al., 2018).



*Values followed by the same letter (a, b, c) do not differ significantly from each other (Tukey multiple comparison method - significance threshold α=0.05).

Figure 1. Effects of compost and digestate application on pH, N_t, N-NO_3, C_{org}, P_{AL}, K_{AL}, and TSC

Cadmium

Cd is the seventh most toxic metal that can remain in the soil for several decades (Jaishankar et al., 2014). Being an extremely mobile element in the soil-plant system, the risk of translocation is high. It is essential to limit its transfer from the soil solution to crop plants by acting on the soil factors that govern the behavior of Cd in the soil. The supply of OM enhanced the retention of the metal in the soil's adsorbent complex, reducing the mobile fraction of Cd in the soil, that is, the fraction accessible to plants. Our experimental data indicate a decrease in the extractable Cd content. There are no significant differences due to the type of OAs (Figure 2). Previous studies have indicated that the available concentration of Cd in soil increases with decreasing soil pH (Li et al., 2014; Yu et al., 2016; Hou et al., 2019), which supports our findings.

Zinc

Zn is considered an essential element for plant development, which serves as a structural element for certain enzymes and is involved in numerous physiological processes (Gupta & Kalra, 2006; Ciobanu, 2019). Zn ions can have both beneficial and toxic effects on plant cells. The main factor influencing the distribution of zinc in the soil is pH, which essentially affects the mobility of Zn ions. As soil pH decreases, the solubility and absorption of Zn increase, thereby raising the potential for phytotoxicity. (Ciobanu, 2019). Compared to the control, the values of available Zn content in soil decrease significantly after applying an OA quantity equivalent to 60 t/ha.

Copper

Cu is an essential element for plant development, but its presence in the culture substrate can cause phytotoxicity. Experimental data reveals significant differences in terms of extractable Cu content in DTPA between the compost and digestate variants. The application of compost produced a significant decrease in the extractable Cu content compared to digestate, regardless of the applied dose (Figure 2). Data from the literature indicate that Cu toxicity occurs at relatively high Cu concentrations, which vary depending on the plant species (Barbosa et al., 2013; Alobaidi et al., 2015; Shun et al., 2018).

Manganese

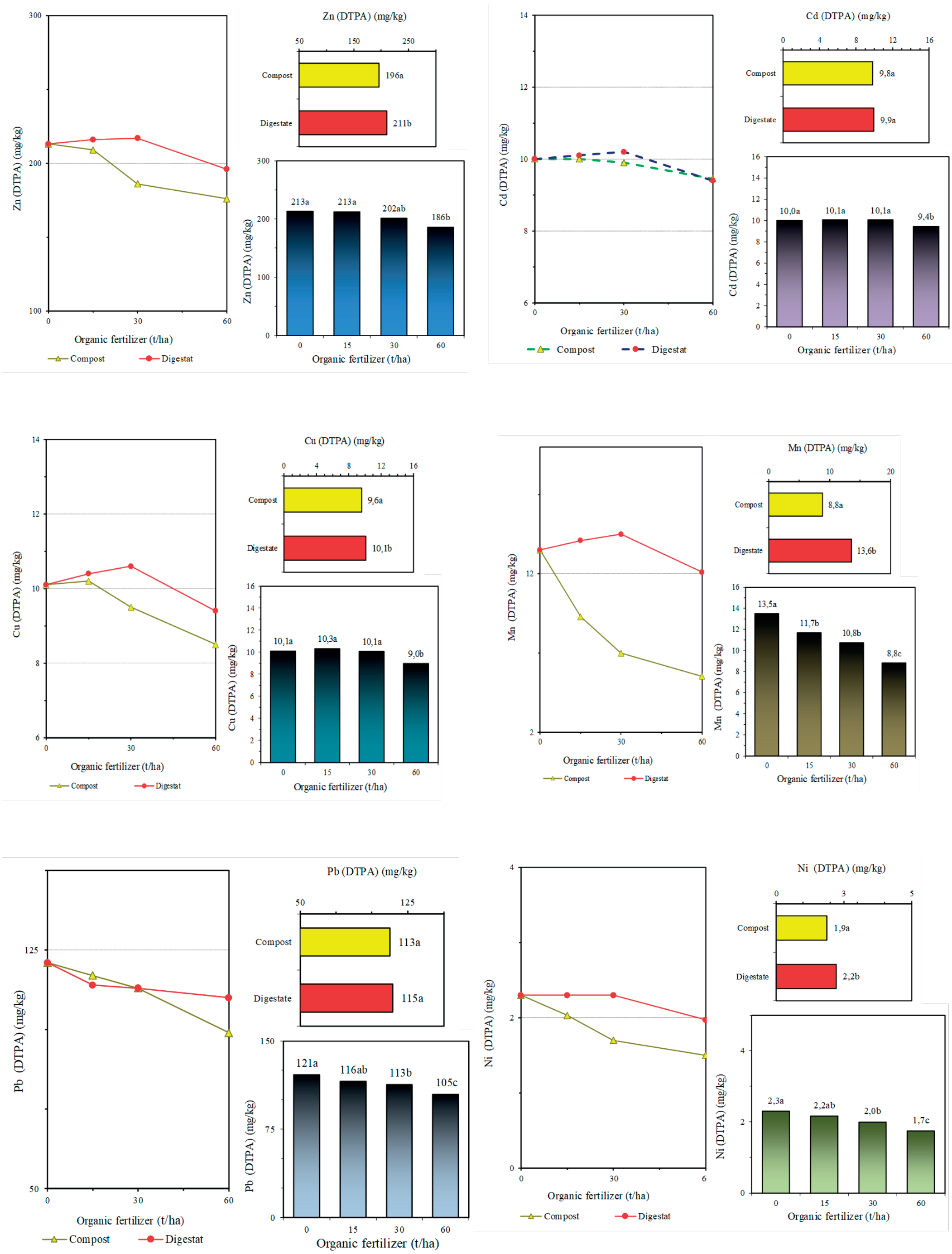
Mn is also an essential element for plant development. The soil material used in the experiment does not contain excessive Mn; however, its high solubility leads to the accumulation of high amounts of Mn in the soil solution under acidic conditions.

The compost application resulted in a significant decrease in soluble Mn content compared to the application of digestate, regardless of the applied dose. The reduced mobility of Mn in the soil solution, caused by the increased OM content from the application of organic amendments, is evidenced by a significant decrease in the DTPA-extractable Mn content as the dose of applied OAs increases (Figure 2). The lowest value was determined in the variants with the maximum dose of organic amendment (60 t/ha, regardless of the type of amendment).

Lead and Nickel

Pb and Ni are elements of no importance in plant development, unlike other metals, such as Zn, Cu, and Mn. Their presence in high quantities causes toxicity to plants, suppressing the overall growth (Jaishankar et al., 2014; Najeeb et al., 2017), inhibiting the iron uptake (Yongsheng et al., 2011; Jaishankar et al., 2014), and reducing the fertile potential of the soil. The application of organic amendment, regardless of the type, resulted in a significant reduction in mobile Ni content and mobile Pb content (Figure 2), consistent with the findings of Katoh et al. (2016). There are significant differences between the effects produced by digestate and those produced by compost, the application of compost leading to a more pronounced decrease of the mobile Ni contents.

In the case of Pb, although the application of organic amendments decreased the metal's mobility in the soil, the values of extractable Pb contents from the soil were still high.



*Values followed by the same letter (a, b, c) do not differ significantly from each other
(Tukey multiple comparison method - significance threshold α=0.05).

Figure 2. Effects of compost and digestate application on accessible heavy metal (Zn, Cd, Cu, Mn, Pb, Ni) contents (extractable in DTPA - TEA - $CaCl_2$)

CONCLUSIONS

Both compost and digestate used in our study, have emerged as important amendments for remediating contaminated soils and enhancing their chemical properties. The study demonstrates that these amendments can notably improve various soil parameters, including pH, C_{org}, N_t, and the availability of key nutrients and heavy metals. The research shows that both digestate and compost can enhance soil pH and nutrient retention, thereby positively contributing to plant growth and soil health. Specifically, enhancing soil pH can increase nutrient availability, regardless of the type of organic amendment used. Despite the benefits, it is essential to consider the potential risks associated with the long-term application of compost, particularly regarding the accumulation of potentially toxic elements in the soil. The beneficial role of OAs' in immobilizing heavy metals adds complexity to soil management strategies. The ability of these amendments to improve soil fertility and reduce the bioavailability of harmful metals is to be considered in addressing soil contamination issues. Regular assessments of nutrient dynamics and heavy metal concentrations in treated soils are important for sustainable agricultural practices. Additionally, integrating these findings into broader soil conservation policies will help mitigate the adverse effects of industrial activities contributing to soil degradation.

ACKNOWLEDGEMENTS

This research work was financed from Project ADER 18.1.1 Project: Development of solutions to restore soil health in conditions of maintaining environmental sustainability by using organic fertilizers, Funder Romanian Ministry of Agriculture and Rural Development.

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