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Agricultural Valorization of Urban Sewage Sludge: Short-Term Effects on Trace Elements Contamination in Cultivated Soil

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ABSTRACT

Urban sewage sludge (USS) contains potentially hazardous Trace Elements (TEs), including Zn, Cu, Cr, Ni, Pb, and Cd, as well as Trace Organic Contaminants (TOCs), such as Aromatic Polycyclic Hydrocarbons (PAHs). The accumulation of TEs in agricultural soils increases their uptake by crops, thereby affecting food quality and human health. This study aimed to evaluate the short-term effects of different USS application rates on total TE (TTE) and soil metal pollution in cultivated soil in Boukhalfa, Tizi-Ouzou district, Algeria (4°0′52″E, 36°45′4″N), to prevent soil contamination and associated ecological and health risks. Based on legislative recommendations for their use in agriculture, TTEs and PAHs in the USS and TTEs in the soil samples were analyzed. A completely randomized block design was implemented with USS applied at rates of 15, 30, and 45t.ha^{-1} t USS, implemented in March 2017. One year later, composite soil samples were collected from the 0–20 cm surface layer of each elementary plot (EP). The results showed that the soil was suitable for USS application, with PAHs levels in the USS and TTE concentrations in the soil remaining below the regulatory limits. The increase in TTE concentrations in the amended soils (D3) was 32, 15, 18, 13, and 5% for Zn, Cu, Pb, Cr, and Ni, respectively. The maximum PI (0.32) was recorded on D3. However, all values remained below the regulatory limits. Overall, the short-term application of USS at 15, 30, and 45t. ha^{-1} did not lead to soil contamination by multiple TEs. These findings support the safe use of USS as an organic amendment under controlled conditions with regulated application rates. However, long-term monitoring is essential to determine the potential cumulative effects on soil quality and crop uptake and to develop optimized management strategies for sustainable sludge reuse in agriculture.

INTRODUCTION

Urban sewage sludge is a major by-product of wastewater treatment (WWT) processes (Liang et al. 2022). The annual quantity produced worldwide increased rapidly from 45 million tons of DM in 2017 (Danich et Ozbakkalogh 2022) to approximately 160 million tons of DM in 2023 (Feng et al. 2023). Furthermore, the global management and disposal of this waste around the world is complex and challenging (Uggetti et al. 2010). Its incineration generates air pollution and secondary pollutants (Chen et al. 2020), and landfilling significantly pollutes the surrounding soils (Hadi 2023). However, its agricultural reuse is the most efficient and least restrictive method and a better alternative to landfilling and incineration (Zoghlami et al. 2016, Marzougui et al. 2022). This method of valorization, through its spread on agricultural soils, is favored because of its abundance in organic matter (OM) and nutrients (Cherfouh et al. 2024). In recent years, Algeria has achieved considerable progress in domestic WWT, with the number of WWT plants (WWTPs) increasing from 177 in 2018 to more than 200 in 2021 (MWR 2021). This situation led to an increase in the national annual USS, reaching 105.000 tons of DM, with the Tizi-Ouzou district contributing approximately 1.770 tons of DM (NSO 2024). Indeed, the intensive use of agricultural land, scarcity of organic amendments, and high mineralization of OM have led to a reduction in OM content (Dridi & Toumi 1999). While USS has the potential to provide OM and nutrients (Djafari 2020), this dual value qualifies it as an organic amendment and fertilizer, attracting the attention of farmers (Cherfouh 2019). Its application to soils also has a considerable effect on improving their physical, chemical, and biological properties (Douaer et al. 2021, Cherfouh 2024), enhancing their fertility (Curci et al. 2020, Marin & Rusanescu 2023) and increasing crop yield (Yagmur et al. 2017).

In Algeria, methods used for USS agricultural valorization through direct spreading on croplands are empirical (Cherfouh et al. 2018). They lack prior studies on soil and sludge, the determination of applicable doses, and the monitoring of amended soils. Such practices are still used today because of the lack of regulatory requirements. However, the concentration of heavy metals in soil amended with USS should be periodically monitored to maintain the level of TEs within safe limits, sustain soil quality, and prevent food chain contamination risks (Adyasha et al. 2021). Furthermore, USS is a source of TEs (Zn, Cu, Ni, Pb, Cr, Cd, and Hg) and organic pollutants that are harmful to the environment (Dume et al. 2023). As reported by Agoro et al. (2020), approximately 80%–90% of TEs in wastewater are found in USS. Liu et al. (2013) and Zaragueta et al. (2021) reported that the metallic pollution load of USS is a major obstacle to its spread on agricultural soils because its use entails the transfer of TEs and TOCs to arable lands. Furthermore, the regular application of USS can elevate TTE concentrations in soil to toxic levels, leading to gradual accumulation (Hasnine et al. 2017) and associated health risks (Shamsollahi et al. 2019, Aghanaghad et al. 2025).

This study aimed to evaluate the short-term effects of spreading different application rates of USS on TE accumulation and soil pollution in cultivated soil west of the Tizi-Ouzou district, northern Algeria.

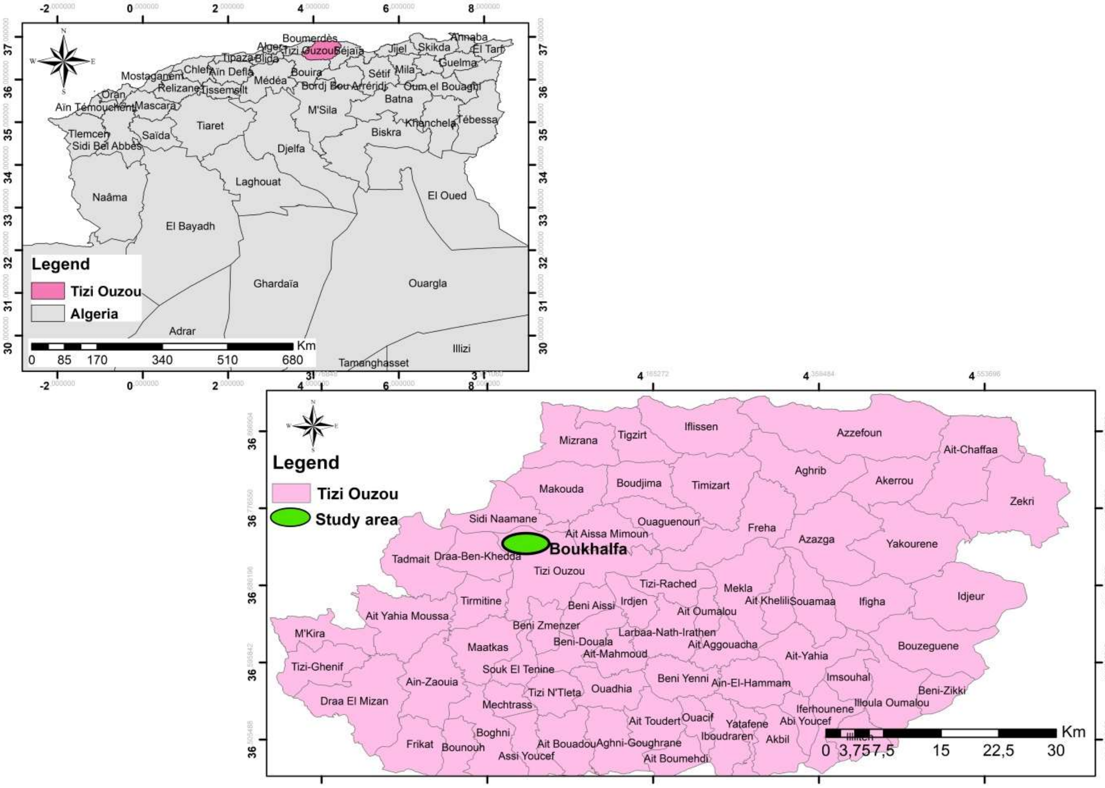


Fig. 1: Location of the study area.

MATERIALS AND METHODS

Study Area

The research was conducted in the Boukhalfa region, located in the Tizi-Ouzou district (4°0′52″E, 36°45′4″N) (Fig. 1). The region's climate is characterized by wet and cold conditions during the winter months and hot and dry conditions during the summer. The USS used in this experiment was collected from a WWTP located in Boukhalfa, west of Tizi-Ouzou. Its capacity is approximately 25.000 population equivalents, corresponding to a total wastewater (TWW) volume of 3.750 m³ per day (NSO 2024).

USS and Soil: Sampling and Analysis

Dehydrated secondary USS from the drying bed of a WWTP was selected for the experiment. USS was collected a year after dehydration (September 2016). To assess the suitability of the soil in the study plot for amendment with USS, samples were taken at random from a top depth of 0-20 cm. Both soil and USS samples were air-dried at room temperature, ground, and mixed to obtain composite samples for analysis. One part was sieved to 2 mm following NF ISO 11464 for physicochemical analysis, and the other part of the USS was stored in brown glass bottles at 4°C for the determination of PAHs. To determine the TTEs, sieving was performed

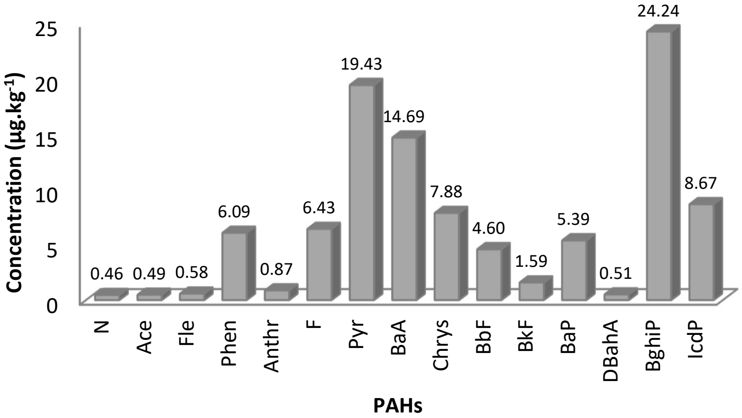


Fig. 2: PAHs concentration in the study USS.

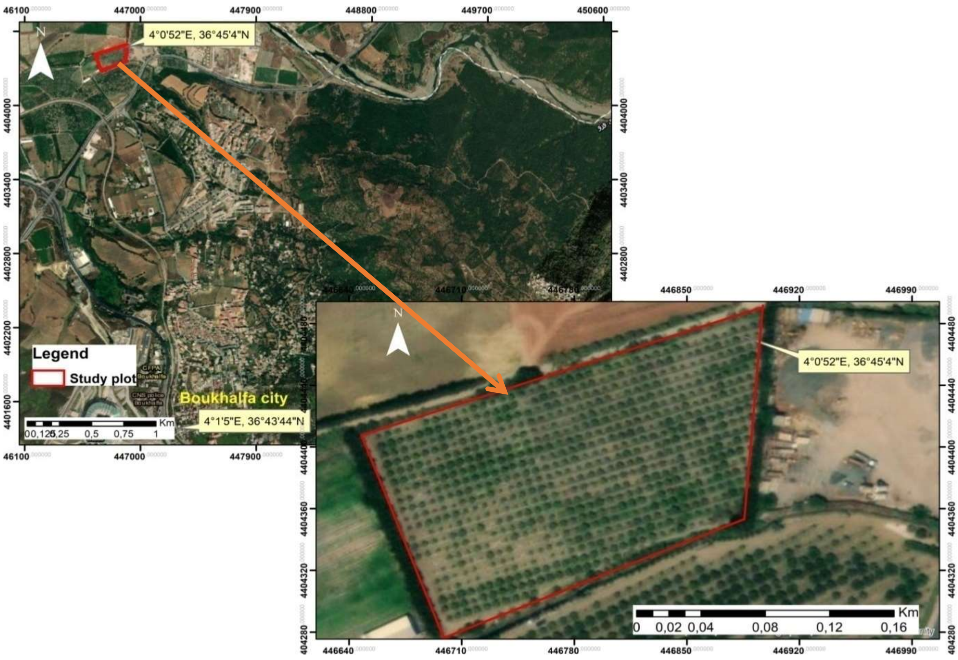


Fig. 3: Location of the farm and study plot.

at 200µm. The main characteristics for USS are: 47% DM, pH 6.6, Kavai 0.092 g.kg^{-1}, Pavai 1.16 g.kg^{-1}, NTK 2.91%, moisture 46%, total polyphenols (TP) 5.18%, EC 3.44 dS/m, $CaCO_3$ 14.2%, OM 52%. The total concentrations of Zn, Cu, Pb, Cr, Ni, and Cd determined by an Atomic Absorption Spectrophotometer (AAS) following the Aqua Regale method were 689 mg.kg^{-1}, 214.3 mg.kg^{-1}, 74.5 mg.kg^{-1}, 37.8 mg.kg^{-1}, 19.1 mg.kg^{-1}, and 0.87 mg.kg^{-1}, respectively. The TTE content and total phenol (TP) content are within the standards of EU legislation, which governs the use of SS for agriculture (EC, 2002). The order of concentration was Zn > Cu > Pb > Cr > Ni > Cd.

The PAHs were extracted using the Soxhlet method (NF ISO 15013877), and their determination was accomplished using high-performance liquid chromatography (HPLC). Additionally, their levels were well below the limit standards of EU legislation for agricultural use (Fig. 2). Before the application of USS, a series of physicochemical analyses were conducted on the soil samples. These analyses included the determination of soil particle size using the Robinson pipette method; pH measurement in a 1:5 ratio using a pH meter; electrical conductivity (EC) measurement in a 1:5 ratio using a conductimeter; $CaCO_3$ content determination using a Bernard calcimeter; OM content assessment by loss on ignition; total nitrogen analysis using the Kjeldahl method; bioavailable phosphorus determination using the Olsen method; and CEC and available potassium extraction using ammonium acetate (1N) at pH 7, quantified by Flame Spectrophotometry. The TTEs (Zn, Cu, Cr, Ni, and Pb) were determined using the aforementioned extraction and analytical methods; Cd-T was not quantified as its concentration was below the detection limit (DL).

Experimental Design

The research was conducted at a field site from March 2017 to March 2018 on alluvial soil cultivated with agrumes, located in the Boukhalfa region (Fig. 3). A completely randomized block experimental design was implemented, featuring four replicates and four plots (three doses and one control plot). The experimental design was configured using the R software. Each plot had a surface area of 25m², and the distance between two plots was 5m to avoid contamination between the control and amended plots with different USS rates (Fig. 4). The soil treatment experiment consisted of four control plots that received no amendment (D0) and twelve plots amended with increasing rates at 15 t.ha^{-1} (D1), 30 t.ha^{-1} (D2), and 45 t.ha^{-1} (D3).

The application rates were selected according to the theoretical limits established by European legislation, which suggests a maximum rate of 30t.ha^{-1}of DM (COSTEA, 2022). However, owing to the low concentrations of TEs and TOCs in the SS, the application rates were increased to 45 t.ha^{-1}. The sludge was incorporated into the topsoil to a depth of approximately 20 cm through manual mixing within each EP, encompassing an area of 15 m^2 (3mx5m) during the final stage of the phenological cycle of the agrumes.

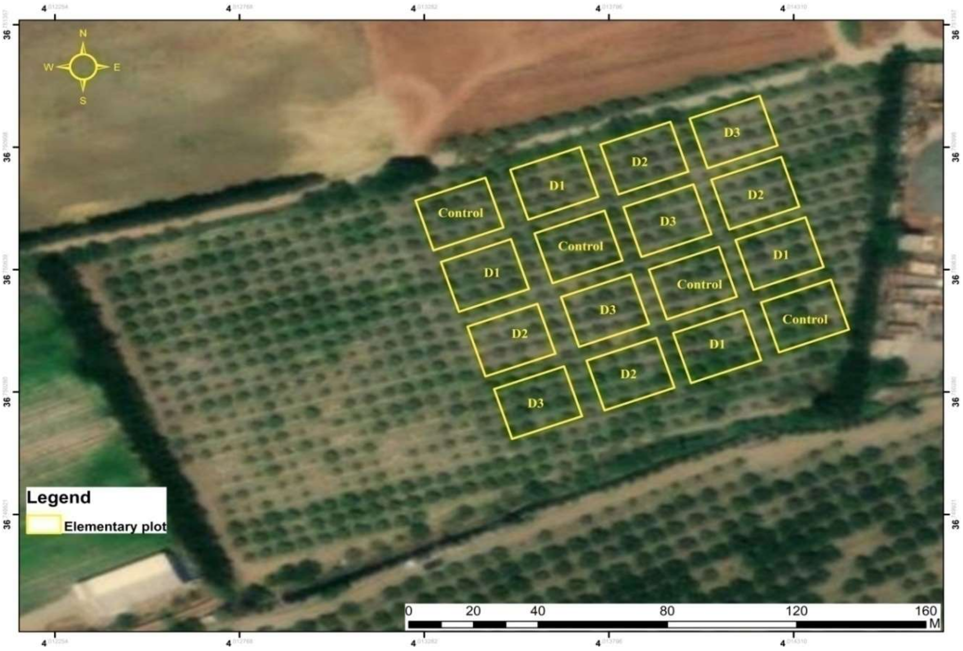


Fig. 4: Experimental design.

Soil Sampling and Analysis after USS Application

In the subsequent year, three soil samples were collected from each EP at the topsoil level (0–20 cm) to evaluate the short-term impact of USS application on TTE content and soil metallic pollution. According to Campos et al. (2019), in the short term, most TEs introduced by USS are retained in the surface layer (0–20 cm). The three soil samples were mixed to form a composite sample and analyzed directly. TTEs were determined using the method described above.

Pollution Index of Soil

The pollution index (PI) is a commonly used criterion for assessing soil toxicity. It was calculated by taking the ratio of the concentration of each TTE (mg.kg^{-1}) in the soil to its respective reference value, and then averaging the sum of these ratios across all studied elements. Calculations were performed using the following formula:

$$PI = (Cd/2 + Cu/100 + Pb/100 + Cr/150 + Zn/300 + ...)/N$$

(Chon et al.1998).

Data Analysis

Various analytical techniques were employed to assess the impact of USS on soil pollution and to evaluate the differences between application rates. These techniques include a correlation matrix and analysis of variance (ANOVA). Significant differences between means were identified using Tukey's Honest Significant Difference (HSD) or Games-Howell tests. The significance level was set at 0.05. All statistical analyses were conducted using R software (R Core Team 2024). Fig. 5 summarizes the different steps of this experiment.

RESULTS AND DISCUSSION

The Characteristics of the Studied Soil and its Suitability for Amendment with USS

The physicochemical characteristics of the soil in the study plot before amendment revealed several key findings. First, the soil texture was classified as sandy loam, which indicates its suitability for amendment with USS owing to its average alkaline pH value of 8.2. This pH value prevents the dynamic and solubilization of TE (Dewangana et al. 2023). Second, the EC of the soil was very low, with a value of 0.14dS.m^{-1}, and the mean percentage of $CaCO_3$ was 8%. The soil's concentration of fertilizing elements and CEC qualifies it as having low chemical fertility. The levels of available phosphorus range from approximately 3.5mg.kg^{-1}, nitrogen is around 0.2%, exchangeable potassium is 33cmol(+).kg^{-1}, the CEC is about 16cmol(+).kg^{-1}, and the OM is about 1.2%. TTEs are within the standard limits of agricultural soils (NF U44-041 AFNOR); their concentrations are 86.9mg.kg^{-1}, 35.1mg.kg^{-1}, 33.3mg.kg^{-1}, 28.7mg.kg^{-1}, and 12.5mg.kg^{-1} for Zn, Cu, Cr, Ni, and Pb, respectively. Conversely, the total Cd concentration was not determined because of its low concentration. The order of TTEs in the soil was Zn > Cu > Cr > Ni > Pb. According to EU regulations, the soil selected for this study was appropriate for the application of USS.

Effect of USS Application on TTEs Concentrations

One year after the USS application, the soil concentrations of TTEs increased proportionally with the applied rates, exhibiting a rate-dependent response (D3 > D2 > D1 > D0). The findings of this study are consistent with those of Zaragüeta et al. (2021). This concordance can be attributed to the fact that 70–90% of metals present in wastewater are transferred to primary and secondary sludges (Feng et al. 2023). Notably, the observed concentrations of Cu-T, Zn-T, Cr-T, Pb-T, and Ni-T in the soil samples remained well below the regulatory limits set by the EU, aligning with the findings of Suhadolc et al. (2010) and Abdul Khaliq et al. (2017). The limited contamination is presumably attributable to the elevated levels of $CaCO_3$ and pH in the soil, which restrict the release of TEs (Zaragüeta et al. 2021, Uddin et al. 2025). The presence of TTEs in control soils may be attributed to pedo-geochemical, atmospheric, or agricultural origins, including manure, fertilizers, pesticides, and other inputs (Sallau et al. 2016). Variations in the percentage increase in TTEs can be explained by differences in their initial concentrations in the USS and the distinct geochemical cycles of each element.

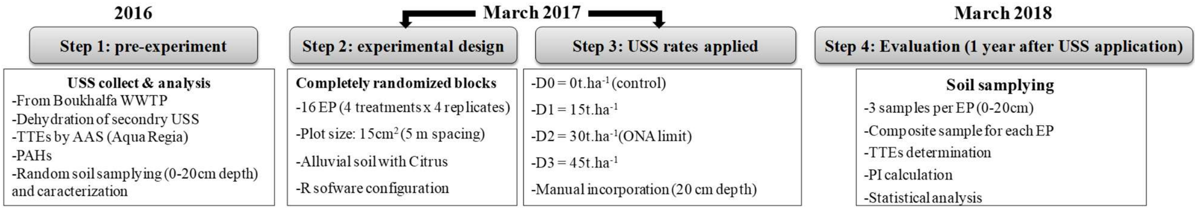


Fig. 5: diagram of experimental steps.

Copper

Cu-T displayed the second-highest concentration among analyzed TTEs, following Zn-T, with concentrations ranging from 34.97mg.kg^{-1} in control soils (D0) to 41.07mg.kg^{-1} under the highest application rate (D3), with an increase of 3%, 4%, and 15% for D1, D2, and D3 treatments, respectively. ANOVA revealed significant differences in the Cu-T content among the amended soils (Table 1). The Post Hoc Comparison Test indicated significant differences between the control soils and those amended with D1, D2, and D3, with P-values below the 0.05 significance level. However, no significant differences were observed between the D1-D2, D1-D3, and D2-D3 amended soil groups (Table 2). Notably, despite the recorded rates of increase, Cu-T concentrations remained below the standards established by EU legislation, which is approximately 100 mg. kg^{-1} in agricultural soils (Fig. 6a), indicating that even at maximum application rates, Cu-T did not approach the maximum allowable amounts for agricultural soil. The results revealed two distinct and heterogeneous rate groups: a (D3) and b (D0-D1-D2), as illustrated in Fig. 6a. The results obtained are consistent with those of Eid et al. (2018) and Zaragüeta et al. (2021) and indicate that the rates used, even at a maximum of 45t.kg^{-1} at a short time, remain within the prescribed safety parameters, thereby negating any potential risk of soil contamination by Cu.

Zinc

Among the elements studied, Zn-T exhibited the highest levels in the studied soil, ranging from 84.9mg.kg^{-1} (D0) to 124.2mg. kg^{-1} (D3), with respective increases of 12%, 22%, and 32% for D1, D2, and D3, respectively. ANOVA revealed statistically significant differences in Zn-T between the rates ($p<0.05$) (Table 1). Post-Hot Comparison (Tukey) showed significant differences between D3-D0, D3-D1, D3-D2, and D2-D0 (Table 2). The results revealed three distinct and heterogeneous rate groups: a (D3), b (D1-D2), and c (D0), as illustrated in Fig. 6b. It is important to emphasize that, despite the rates of increase recorded, Zn-T concentrations remained below the standards established by Loué (1993), which suggests that Zn-T concentration vary from 10 to 300mg.kg^{-1} in agricultural soil. The significant enrichment observed in the soil with increasing USS rates can be related to the high concentration of Zn in USS studied (689mg.kg^{-1}). The results of the present study corroborate those of Eid et al. (2018) and Zaragüeta et al. (2021). These findings suggest that there is no risk of soil contamination by Zn. However, it is important to recognize the sensitivity of citrus to Zn deficiency. The recommended Zn application rate for citrus cultivation ranges from 4-6 kg ha^{-1}year^{-1} (Quaggio et al. 2003).

Lead

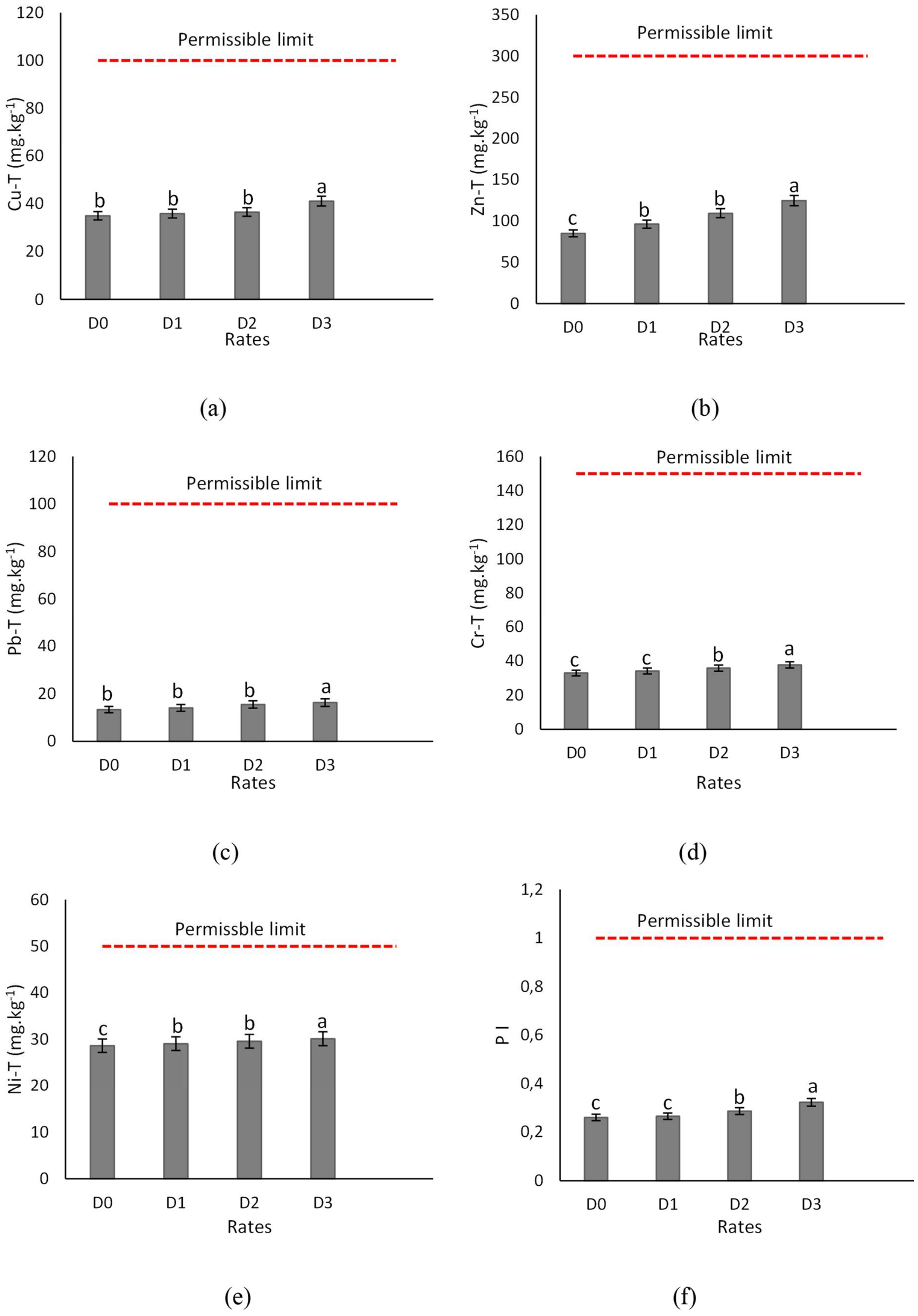
Pb-T concentrations ranged from 15.8mg.kg^{-1} (D0) to 17.5mg.kg^{-1} (D3). The rates of increase were 5%, 14%, and 18% for soils amended with D1, D2, and D3, respectively. Despite the increasing concentrations, the levels remained below the standard limits for agricultural soil. As indicated in Table 1, the ANOVA results demonstrated a significant difference in Pb-T content between the different rates. Further post-hoc comparison (Tukey) revealed a significant difference in the Pb-T content between D0-D3. No significant difference was observed between the remaining rates (Table 2). The findings delineated two distinct and heterogeneous groups: b (D0-D1-D2) and a (D3), as illustrated in Fig. 6c. The elevated pH and $CaCO_3$ levels in the soil under investigation have been shown to promote the formation of insoluble Pb compounds, thereby impeding its mobility (Zaragüeta et al. 2021).

Chromium

Cr-T concentrations in the studied soil ranged from 32.9mg.kg^{-1} in the control EP to 37.7mg.kg^{-1} under the highest sludge application rate (D3), with respective increases of 3%, 8%, and 13% for D1, D2, and D3. ANOVA showed substantial variation in Cr-T content among the different rates (Table 1). Subsequent post-hoc comparisons (Tukey) revealed significant differences between D0-D2, D0-D3, D1-D2, D1-D3 and D2-D3 (Table 2). However, no significant differences were observed between the soils amended with D1 and the control. The findings delineated three distinct heterogeneous rate groups: c (D0-D1), b (D2), and a (D3), as illustrated in Fig. 6d. Despite this statistically significant accumulation, all Cr concentrations remained substantially below 150mg.kg^{-1} regulatory limit for agricultural soils, confirming that current application rates do not pose a risk of contamination. The mobility of Cr in the environment and its potential for soil contamination depend on its oxidation state. The hexavalent form of Cr is more mobile than its trivalent form. Additionally, soil type has a significant impact on Cr mobility. Generally, higher oxidation levels were observed in clay soil than in sandy soil. Furthermore, lime has been demonstrated to have a significant impact on the availability of Cr (Adrian 1991, Rigueiro-Rodríguez et al. 2011). The findings provide a possible explanation for the low risk of contamination observed in the studied soil, crops, and groundwater.

Nickel

Ni-T concentrations ranged from 28.6mg.kg^{-1} (D0) to 30.1mg.kg^{-1} (D3), with a respective increase of 1.5, 3, and 5%, for soils amended with D1, D2, and D3. The ANOVA



(a) (b)

(c) (d)

(e) (f)

Fig. 6: Total contents of TE and PI in the soil control and those amended with different rates of USS

Table 1: Comparison of means (ANOVA).

	Zn	Cu	Pb	Cr	Ni	PI
p-value	<0.001***	0.001**	0.032*	<0.001***	0.001**	<0.001***

*Significance threshold $\alpha < 0.05$, ** significance threshold $\alpha < 0.01$, *** significance threshold $\alpha < 0.001$

Table 2: Post Hoc Comparisons (p-value)

	Cu	Zn	Pb	Cr	Ni	PI
D0 x D1	0.012*					
D0 x D2	0.002**	0.001**		0 .001**	0.015*	0.009**
D0 x D3	0.037*	<0.001***	0.036*	<0.001***	<0.001***	<0.001***
D1 x D2				0.038*		0.039*
D1 x D3		<0.001***		<0.001***	0.007**	<0.001***
D2 x D3		0.027*		0.020*		0.035*

*Significance threshold $\alpha < 0.05$, ** significance threshold $\alpha < 0.01$, *** significance threshold $\alpha < 0.001$

results showed statistically significant differences in Ni-T between the rates (Table 1). The post-hoc Tukey Test (Tukey) revealed significant differences between D0-D2, D0-D3, and D1-D3 (Table 2). However, no significant differences between D1-D0, D1-D2, and D2-D3. The findings delineated three distinct and heterogeneous groups: c (D0), b (D1-D2), and a (D3), as illustrated in Fig. 6e. Despite this increase, the soil concentrations of this element are below the standards established by EU legislation for agricultural soil. According to Mamindy et al. (2013) and Alves (2014), the increase in Ni-T content may be attributed to the applied rate.

Pollution Index and Soil Contamination

The results of the average PI calculated for all EPs, control, and amended with USS are shown in Fig. 6f. The findings indicate that the PI increased with the rates applied from 0.25 (D0) to 0.32 (D3). However, it is important to note that all values remained below the standard limit established by the EU (2002). The ANOVA test showed significant differences between the rates applied (Table 1). Post-hoc Test (Tukey) revealed significant differences between D0-D2, D0-D3, D1-D2, D1-D3, and D2-D3. However, there was no significant difference between D0-D1 (Table 2). The findings delineated three distinct and heterogeneous groups: c (D0-D1), b (D2), and a (D3), as illustrated in Fig. 6f. The application of sludge to soil at rates of 15, 30, and 45t.ha^{-1} was not associated with the risk of contamination by multiple metallic TEs. PI is used to assess the overall toxicity of contaminated soil (Armel et al. 2022). The PI data obtained in this study can be related to the low metallic composition of the USS and the low rates applied. These results are similar to those of Adyasha et al. (2021) and Ye et al. (2020).

Table 3: Correlation Matrix.

	Cu-T	Zn-T	Pb-T	Cr-T	Ni-T
Zn-T	0.85**				
Pb-T	0.66**	0.68**			
Cr-T	0.87**	0.91**	0.57*		
Ni-T	0.76**	0.84**	0.49*	0.86**	

TTEs-PI Correlations

According to the correlation matrix in Table 3, all the TTEs were highly and positively correlated with each other. The highest correlation was observed between Cr-T and Zn-T, while the lowest was observed between Pb-T and Ni-T. Furthermore, all TTEs exhibited a strong positive correlation with PI, which may be attributed to the initial concentration of TTEs in the soil before amendment with USS. These correlations are consistent with the sequence of concentrations observed for these elements in the soil, which follows the order: Zn > Cu > Cr > Ni > Pb. Our results are similar to those of Shomar et al. (2013).

CONCLUSIONS

The results showed that the studied soil is suitable for amendment with USS. USS analysis revealed that USS is rich in OM, with low concentrations of TTEs and PAHs, indicating good agronomic and environmental quality and enabling its application to agricultural soils without any risk of pollution. Short-term experimentation revealed that the total concentrations of Zn, Cu, Pb, Cr, and Ni increased significantly with increasing application rates. Despite this increase, the concentrations remained below the limit values for agricultural soil. The highest content was obtained for Zn, whereas the lowest was obtained for Ni. The PI was below the standard limit, confirming that the soil was not polluted and posed no risk of toxicity to crops. It is important to note that Cd was not detected because of its low concentration in the applied sludge (0.87mg.kg^{-1}). These results confirm that USS can be used in the short term at a dose of up to

45t.ha^{-1} without any risk of soil degradation through pollution or the accumulation of TTEs and without any risk of crop toxicity. However, continued application may lead to the accumulation. This study suggests that reasonable rates of USS application on croplands do not cause the accumulation of TTEs. Further long-term studies are required to confirm the safe use of USS as a natural soil improver and to explore its potential application in arid and hyper-arid regions where OM and moisture are extremely low to improve soil quality and agricultural production.

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