

Original Research

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 Hydrocarbons and Aromatic Polycyclic Hydrocarbons (PAHs). The accumulation of TEs in agricultural soils increases their uptake by crops, which affects food quality and human health. The objective of this study is 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 its use in agriculture, TTEs and PAHs in the USS and TTEs in soil samples were analyzed. A completely randomized block design was implemented with USS applied at rates 15, 30, and 45t.ha⁻¹ of USS, implemented in March 2017. One year later, composite soil samples were collected from the 0-20cm surface layer from each elementary plot (EP). Results showed that the soil was suitable for USS application, with PAHs levels in USS and TTE concentrations in soil remaining below the regulatory limits. Increases in TTEs concentrations in amended soils corresponding to the highest rate (D3) are of 32, 15, 18, 13, and 5% for Zn, Cu, Pb, Cr, and Ni respectively. The maximum PI (0.32) was also recorded at D3. However, all values remained below regulatory limits. Overall, 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 and regulated

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application rates. However, long-term monitoring is essential to determine potential cumulative effects on soil quality, crop uptake, and develop optimized management strategies for sustainable sludge reuse in agriculture.

INTRODUCTION

Urban sewage sludge is a major and inevitable by-product generated by wastewater treatment (WWT) processes (Liang et al. 2022). The annual quantity produced in the world 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 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 its landfill also significantly pollutes the surrounding soils (Hadi 2023). However, its agricultural reuse is the most efficient, least restrictive way, and a better alternative than landfill and incineration (Zoghlami et al. 2016, Marzougui et al. 2022). This method of valorization, through its spreading 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 (MRE 2021). This situation led to an increase in the national annual production of USS, reaching 105.000 tons of DM, with the Tizi-Ouzou district contributing about 1.770 tons of DM (DHW 2024). Indeed, the effect of intensive use of agricultural lands, the scarcity of organic amendments, and the great mineralization of OM lead to a reduction in their OM content (Dridi et 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. E. 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 of the soil and sludge, a determination of applicable doses, and monitoring of amended soils. Such practices are still used today because of the absence of regulatory requirements. However, the concentration of heavy metals in soil amended with USS should be periodically monitored to keep the level of TEs within safe limits and to sustain soil quality and prevent food chain contamination risks (Adyasha et al. 2021). Furthermore, USS is a source of TEs (e.g., 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), about 80% to 90% of TEs contained 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, regular application of USS can elevate TTE concentrations in soil to toxic levels, which can lead to a gradual accumulation (Hasnine et al. 2017) and associated health risks (Shamsollahi et al. 2019, Aghanaghad et al. 2025).

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

2. 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 months. The USS used in this experiment was collected from the WWTP located in Boukhalfa, west of Tizi-Ouzou. Its capacity is about 25.000 population equivalents, corresponding to a total wastewater (TWW) volume of 3.750 m³ per day (ONA 2024).

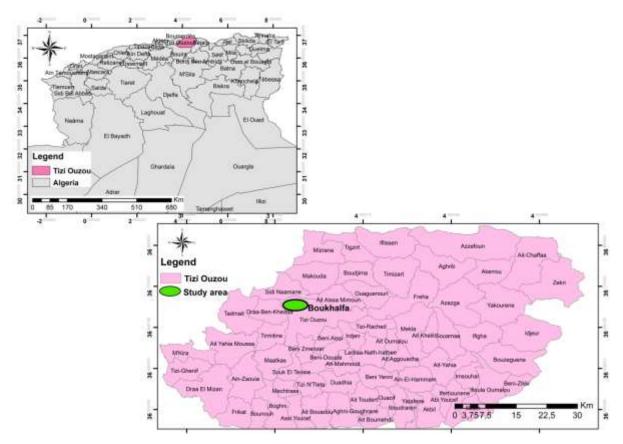


Fig.1: Location of the study area

USS and Soil: Sampling and Analysis

Dehydrated secondary USS from the drying bed of WWTP was selected for the experiment. The USS was collected a year after the dehydration (September 2016). To assess the suitability of the soil on the study plot to be amended with USS, samples were taken at random from a top depth of 0-20 cm. Both soil and USS samples are air dried at room temperature, then ground and mixed to obtain composite samples for analysis. One part is sieved to 2mm following NF ISO 11464 for physicochemical analysis, and the other part of USS is stored in brown glass bottles at 4°C for the determination of PAHs. For the determination of TTEs, the sieving is carried out at 200µm. The main characteristics for USS are: 47% DM, pH 6.6, Kavai 0.32mg.kg⁻¹, Pavai 11.6 mg.kg⁻¹,

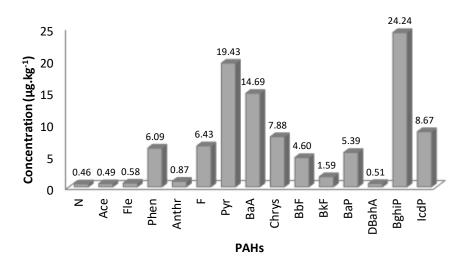
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NTK 1.91%, moisture 46%, total polyphenols (TP) 5.18%, EC 3.44 dS/m, CaCO₃ 14.2%, OM 52%. The total concentrations of Zn, Cu, Pb, Cr, Ni, and Cd determined by an Atomic Absorption Spectrophotometer (AAS) following the Aqua Regale method were 689mg.kg⁻¹, 214.3mg.kg⁻¹, 47.5mg.kg⁻¹, 37.8mg.kg⁻¹, 19.1mg.kg⁻¹, and 0.87mg.kg⁻¹ respectively. The TTE contents and Totals Polyphenol (TP) are within the standards of EU legislation, which governs the use of SS for agriculture (EC, 2002). The concentration order is: Zn > Cu > Pb > Cr > Ni > Cd.

The PAHs were extracted by 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 USS application, 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; CaCO₃ 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 method; 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 an 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). The experimental design was configured using R software. Each plot had a surface area of 25m^2 , and the distance between two plots was 5m to avoid contamination between control and amended plots with different rates of USS (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).



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Fig. 2: PAHs concentration in the study USS

Fig. 3: Location of the farm and study plot

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, due to the low concentrations of TEs and TOCs in the SS, the application rates were increased to 45t.ha⁻¹. The sludge was incorporated into the topsoil to a depth of approximately 20cm through manual mixing within each EP, encompassing an area of 15m² (3mx5m) during the final stage of the agrumes' phenological cycle.



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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 (0–20cm) to evaluate the short-term impact of USS application on TTE contents and soil metallic pollution. According to Campos et al. (2019), in the short term, most of the TEs introduced by USS are retained in the surface layer (0-20cm). The three soil samples were mixed to constitute a composite sample and directly analyzed. The TTEs were determined using the previously mentioned method.

Pollution Index of Soil

The pollution index (PI) is the criterion commonly used to assess soil toxicity. It is 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. The calculation is performed using the following formula:

$$PI = (Cd/2 + Cu/100 + Pb/100 + Cr/150 + Zn/300 + ...)/N$$
 (Chon et al.1998).

Data Analysis

A variety of analytical techniques were employed to assess the impact of USS on soil pollution and to evaluate the differences between application rates. These techniques included a correlation matrix and analysis of variance (ANOVA). Significant differences between means were identified using HSD (Tukey's Honest Significant Difference) test or Games–Howell test. A significance level of 0.05 was used throughout. All statistical analyses were conducted using R software (R Core Team 2024).

Figure 5 summarizes the different steps of the experiment.

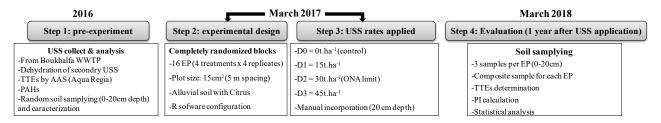


Fig. 5: diagram of experimental steps

3. RESULTS AND DISCUSSION

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

Physico-chemical characteristics of the soil in the study plot before amendment reveal several key findings. Firstly, the soil's texture is classified as sandy loam, which indicates its suitability for amendment with USS due to its average alkaline pH value of 8.2. This pH value prevents the dynamic and solubilization of TE (Dewangana et al. 2023). Secondly, the EC of the soil is very low, with a value of 0.14dS.m⁻¹, and the mean percentage of CaCO₃ is 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

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 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 was not determined due to its low concentration. The order of TTEs in the soil is: Zn > Cu > Cr > Ni > Pb. According to EU regulation, the soil selected for this study is appropriate for the application of USS.

3.2. Effect of USS Application on TTEs Concentrations

One year after the USS application, soil concentrations of TTEs studied increased proportionally with applied rates, exhibiting a rate-dependent response (D3 > D2 > D1 > D0). The findings of this study are consistent with those reported by Zaragüeta et al. (2021). This concordance can be attributed to the fact that more than 70% to 90% of metals present in wastewater are transferred to primary and secondary sludges (Feng et al. 2023). It is noteworthy that 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, thereby aligning with the findings reported by Suhadolc et al. (2010) and Abdul Khaliq et al. (2017). The limited contamination is presumably attributable to the elevated levels of CaCO₃ and pH in the soil, which restrict the release of TEs (Zaragüeta et al. 2021, Uddin et al. 2021). The presence of the TTEs in control soils may be attributed to pedo-geochemical, atmospheric, or agricultural origins, including manure, fertilizers, pesticides, and other inputs (Sallau et al. 2017). Variations in the percentage increase of TTEs could be explained by differences in their initial concentrations in the USS and by distinct geochemical cycles of each element.

3.2.1. 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 Cu-T contents among the amended soils (Table 1). The Post Hoc Comparison Test indicated significant differences between control soils and those amended with rates D1, D2, and D3, with P-values below the 0.05 significance level. On the other hand, no significant differences were obtained between the D1-D2, D1-D3 and D2-D3 amended soil groups (Table 2). Notably, despite the rates of increase recorded, Cu-T concentrations remain below the standards established by EU legislation, which is about 100mg.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 rates groups: a (D3), 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 indicates that the rates used, even at a maximum of 45t.kg⁻¹ at short time, remains within the prescribed safety parameters, thereby negating any potential risk of soil contamination by Cu.

3.2.2. 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.

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ANOVA revealed statistically significant differences in Zn-T between rates (p<0.05) (Table 1). Post-Hot Comparison (Tukey) shows significant differences between D3-D0, D3-D1, D3-D2 and D2-D0 (Table 2). The results revealed three distinct and heterogeneous rates 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 remain 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 applied 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). The 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 to 6kg ha⁻¹year⁻¹ (Ouaggio et al. 2003).

3.2.3. 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%, respectively for soil amended with D1, D2, and D3. Despite the increasing concentrations, levels remained below the standard limits for agricultural soil. As indicated in Table 1, the results of ANOVA demonstrated that there was a significant difference in Pb-T content between the different rates. Further Post Hoc Comparison (Tukey) revealed a significant difference in Pb-T content between D0-D3. There was no significant difference 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 CaCO₃ levels in the soil under investigation have been shown to promote the formation of insoluble lead compounds, thereby impeding its mobility (Zaragüeta et al. 2021).

3.2.4. 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 various rates (Table 1). Subsequent Post Hoc Comparison (Tukey) revealed a significant difference 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 those 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 the 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 the potential for soil contamination are dependent upon its oxidation state. The hexavalent form of Cr is more mobile than the trivalent form. Additionally, the soil type also has a significant impact on Cr mobility. Generally, higher oxidation levels are observed in clay soil compared to sandy soil. Furthermore, the presence of lime has been demonstrated to have a significant impact

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on the availability of Cr (Adrian 1991, Rigueiro-Rodríguez et al. 2011). The findings thus obtained provide a possible explanation for the low risk of contamination observed in the studied soil, crops, and groundwater.

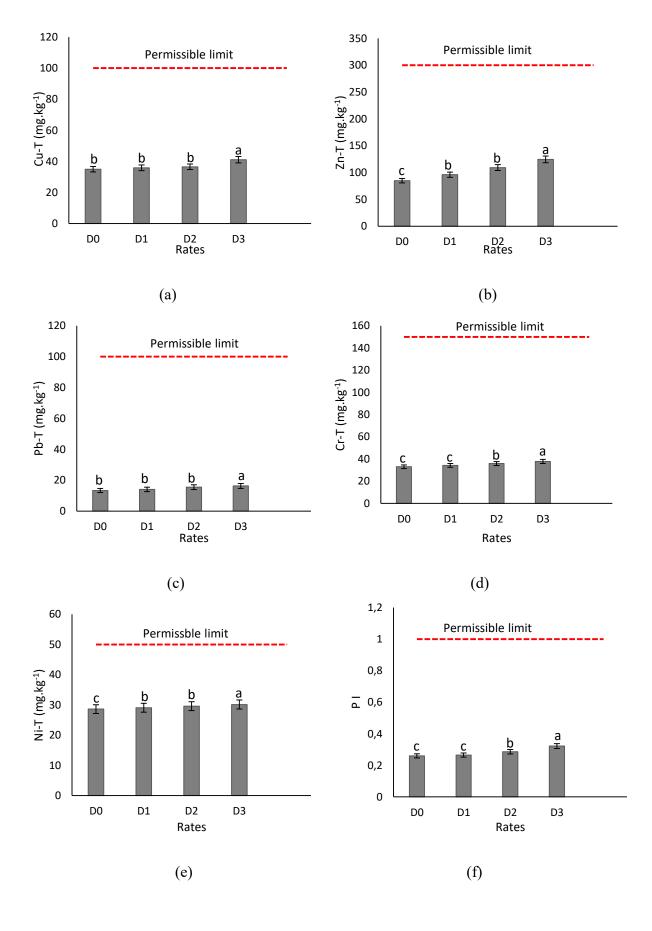
3.2.5. Nickel

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 results showed statistically significant differences in Ni-T between rates (Table 1). The Post Hoc Test (Tukey) revealed significant differences between D0-D2, D0-D3, and D1-D3 (Table 2). However, there were 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 soils. According to Mamindy et al. (2013) and Alves (2014), the increase in Ni-T content may be attributed to the applied rates.

3.3. Pollution Index and Soil Contamination

The results of the average PI calculated for all EPs control and amended with USS are presented 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 EU (2002). The application of the ANOVA test showed significant differences between 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), illustrated in Fig. 6f. The application of sludge to the soil at rates of 15, 30, and 45t.ha⁻¹ is not associated with the risk of contamination by multiple metallic TEs. PI is used to assess the overall toxicity of a contaminated soil (Armel et al. 2022). The PI data obtained in this study can be related to the low metallic composition of USS and the low rates applied. These results are similar to those obtained by Adyasha et al. (2021) and Ye et al. (2020).

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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 α < 0.05, ** significance threshold α < 0.01, *** significance threshold α < 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 α < 0.05, ** significance threshold α < 0.01, *** significance threshold α < 0.001

3.4. TTEs - PI Correlations

According to the correlation matrix in Table 3, all 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 present 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 reported by Shomar et al. (2013).

Table 3: Correlation Matrix

Cu-T	Zn-T	Pb-T	Cr-T	Ni-T

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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**

4. CONCLUSIONS

Results showed that the studied soil is suitable for amendment with USS. The results of the USS analysis revealed that it 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 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 soils. The highest content was obtained for Zn, while the lowest was for Ni. The PI was below standard limits, confirming that the soil is not polluted and poses no risk of toxicity to crops. It is important to note that Cd was not detected due to 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 of TEs. This study suggests that reasonable rates of USS application on croplands do not cause accumulation of TTEs. Further long-term studies are required to confirm the safe use of USS as a natural soil improver, as well as to explore its potential application in arid and hyper-arid regions where OM and moisture are extremely low, for improving soil quality and agricultural production.

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Conflicts of Interest: "The authors declare no conflicts of interest."

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