

Article

Sustainable Management Approaches to Heavy Metal Pollution in Arid Soils Using Soil Amendments and Plant-Based Remediation

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Abstract

This study examined the effect of sulfur, ethylenediaminetetraacetic acid (EDTA), olive mill wastewater (OMW), and their mixtures in remediating metal-polluted soils by implementing both leaching trials and a greenhouse experiment with sunflower (*Helianthus annuus*). In the leaching study, soils were subjected to five discharge volumes (V1–V5). EDTA significantly improved metal mobility of Cd (221.4) mg·kg^{−1} in V2, Pb (340.8) mg·kg^{−1} in V3, and Zn (1.01) mg·kg^{−1} in V3, while OMW moderately mobilized Cd and Mn. However, sulfur mitigated leaching by buffering soil pH and metal immobilization. Mixed treatments revealed moderate leaching behavior. EDTA lowered soil pH (5.3) and raised EC (1763) μS/cm, while sulfur maintained stable chemical environments. In the greenhouse experiment, amendments significantly influenced biomass and metal uptake. Sunflower roots accumulated the highest Cd under sulfur (733.5) mg·kg^{−1} and Mn under EDTA (743.3) mg·kg^{−1}. EDTA restricted Cd translocation (TF = 0), while OMW enhanced Cr movement to shoots (TF = 17.6). EDTA also reduced Cd bioavailability, whereas OMW raised Pb and Mn availability. Overall, EDTA improved metal solubility for potential removal and sulfur in stabilized metals, while OMW acted as a moderate mobilizer. Sunflower demonstrated selective metal uptake, indicating its potential in phytoremediation strategies tailored to specific contaminants.

Keywords: phytoremediation; heavy metals; EDTA; sulfur; olive mill wastewater; sunflower; leaching; soil amendments



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1. Introduction

Soil contamination with heavy metals (HMs) has emerged as a critical global environmental problem, specifically as industrial growth, intensive agriculture, mining, metallurgy, extensive farming, and improper waste disposal continue to intensify their accumulations in land ecosystems [1–8]. Many authors confirmed [9,10] that elements such as lead (Pb), copper (Cu), cadmium (Cd), manganese (Mn), zinc (Zn), and nickel (Ni), pose significant hazards, are considered non-degradable, can persist in soil for decades, and tend to accumulate within food chains, leading to prolonged ecological and health concerns. Remediating HMs contamination becomes even more difficult in arid and semi-arid regions due to their persistence, toxicity, and bioaccumulative nature, which hinder the efficacy of

conventional techniques. Among the major human-related contributors to HMs, mining is one of the most significant and pervasive causes of soil contamination globally. During mining operations, large areas of the soil are disturbed, generating substantial amounts of metal-enriched waste that is often kept in open spaces. Degradation can occur widely as a result of these residues seeping into nearby soils and water systems. The Mahad Al-Dahab gold mine in Western Saudi Arabia, a major mining site, has been repeatedly associated with significant contaminations of Pb, Cd, and Cr in the surrounding soils [5,11–13]. Over the years, physicochemical approaches have been employed to address soil pollution. Conventional physiochemical methods, such as soil excavation and replacement, washing, solidification/stabilization, and vitrification, can deliver relatively quick outcomes, but they are frequently expensive, energy-intensive, upset the structure of the soil, and can produce secondary pollution [9,14]. On the other hand, while newer technologies like electrokinetic remediation, nanomaterial amendments, and the use of biochar provide better efficiency and, in certain situations, in situ treatment options, they still have issues with scalability, long-term stability, and possible ecological hazards [15,16].

To address these limitations, eco-friendly strategies such as phytoremediation have gained increasing attention. It has been confirmed that the mobility and chemical form of metals are largely influenced by factors like pH, redox conditions, and the amount of organic matter present [17,18], which affects the performance of plant-based remediation. Numerous studies reported by [11,19] agree that in situ techniques are generally more cost-effective than conventional methods, which promotes interest in integrating phytoremediation with soil amendments that can either lock or immobilize metals, or by enhancing their solubility for plant uptake [20]. A range of amendments has gained widespread recognition for their effectiveness in facilitating the remediation processes. Ethylenediaminetetraacetic acid (EDTA), a well-established chelating agent, is highly effective at binding with metals, thereby facilitating their uptake by plants and improving their phytoavailability [21]. However, its environmental persistence needs to be prevented from further pollution [22]. Sulfur amendments can lower soil pH through microbial oxidation, which promotes immobilization of HMs through precipitation or adsorption processes in addition to their ability to supply essential nutrients for plant growth [12,23,24]. Olive mill wastewater (OMW), a byproduct of olive oil production, contains organic acids, phenolics, and nutrients that can improve soil properties and stimulate microbial activity [25,26]. Its influence on HM mobility varies depending on soil characteristics, sometimes promoting stabilization and increasing its solubility [24,27]. Despite the growing interest in sustainable remediation strategies, the combination effects of the individual amendments, such as sulfur, EDTA, and OMW, remained poorly understood. This study addressed the knowledge gap by investigating the performance of these individual amendments and their combinations in remediating HM-contaminated soils from the Mahad Al-Dahab mining site. This study offers solid insights into the efficacy of amendments for remediating contaminated soils in dry mining regions by introducing an integrated experimental approach involving column leaching and greenhouse trials with sunflower to assess both metal mobility and plant uptake.

2. Materials and Methods

2.1. Study Location and Soil Collection

Mahad Al Dahab is a historic gold-mining district in Western Saudi Arabia about 170 km southeast of Madinah (23.275° N–23.575° N, 40.625° E–40.925° E). This area experiences an arid climate, with average yearly temperature near 28 °C and reaching up to 43 °C in summer months, and receives less than 60 mm of rainfall annually. Roughly 200 kg of surface soil influenced by mining activities was collected and transported to the

soil laboratory at the Advanced Agricultural and Food Technology Institute, Sustainability and Environment Sector, King Abdulaziz City for Science and Technology. The collected soil samples were air-dried, sieved with a 2 mm stainless-steel sieve, and analyzed for their chemical and physical properties.

2.2. Soil Properties

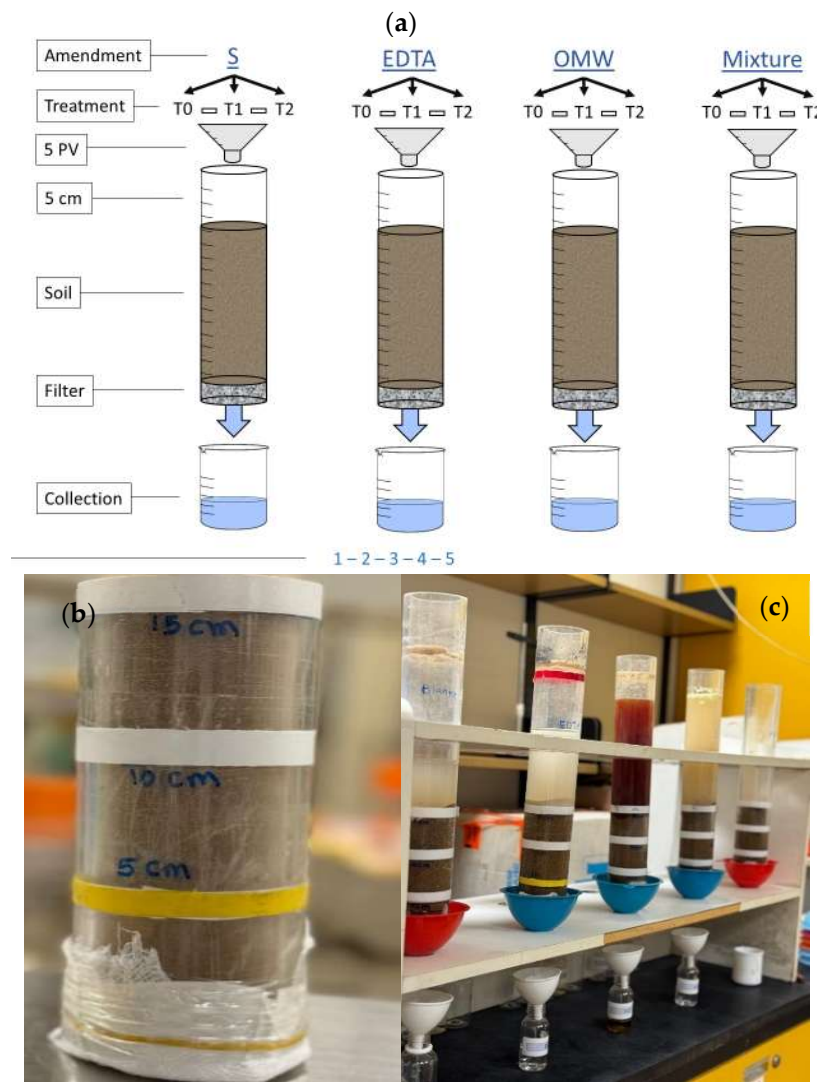
Before applying treatments, essential soil properties were determined prior to experimentation. A 1:5 soil-to-water suspension was prepared to measure soil pH using a pH meter. Electrical conductivity (EC) was determined from saturated paste extracts. The hydrometer method was used to determine soil texture, while calcium carbonate content was determined using calcimeters. Moreover, soil HMs such as Cd, Cr, Cu, Ni, Pb, Zn, and Mn were analyzed using the DTPA-extractable method by Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) [28]. Total concentrations were determined after digestion [29] with acids such as hydrofluoric acid (HF) (40%, for analysis EMSURE[®] ISO, Reag. Ph Eur), sulfuric acid H₂SO₄ (98%, Merck, Darmstadt, Germany), and perchloric acid (HClO₄) (70% (Reag. USP) for analysis, ACS, ISO). Available N, P, and K were measured by the Kjeldahl method, Olsen's method, and flame photometer method, respectively [30].

2.2.1. Greenhouse Trial

A pot experiment was carried out in a controlled environment at King Saud University using a completely randomized design with three replicates per treatment. Sulfur was applied at 1 or 2 t/ha, OMW at 5% or 10% (*v/w*), EDTA at 25 or 50 mmol/kg either individually or in combination at their higher rates (2 t ha⁻¹ sulfur, 10% OMW, and 50 mmol kg⁻¹ EDTA). Control pots of the control contained unamended soil. All pots received an equal amount of contaminated soil (5 kg). Six sunflower seeds (*Helianthus annuus*) were sown per pot, and thinned to three seedlings after two weeks. Sunflower was chosen for its well-established tolerance to heavy metals and uptake potential. Greenhouse conditions were maintained at an average temperature of 28 °C, 14 h light/10 h dark, and 60% relative humidity. Plant parameters such as shoot height and weight, root length and weight, as well as both fresh and dry weights, were measured after six weeks using a calibrated scale. Shoots and roots were dried in an electrical oven for 48 h at 72 °C. Thereafter, dried plant tissues were finely ground and digested using sulfuric acids and hydrogen peroxide (H₂O₂) (30%, Sigma-Aldrich, St. Louis, MO, USA) prior to ICP-OES analysis. Post-harvest soil samples were homogenized, air-dried, sieved (2 mm), and analyzed for pH, EC, nutrients, and metal concentrations as per the earlier methods.

2.2.2. Column Leaching Experiment

Prior to the greenhouse pot experiment, a leaching trial was established to validate the suitability of the selected soil amendments (Scheme 1). The trial was performed using acrylic columns (2.5 cm diameter, 15 cm length) marked with graduated centimeters to enable precise sampling at different depths. Each column was packed with 553 g of contaminated soil, and the base of the column was fitted with filter paper to facilitate drainage. This study used five treatments: (1) control with 440 mL of tap water, (2) EDTA solution at 50 mmol N (440 mL), (3) sulfur at 5 g total (1 g per pour) with 440 mL water, (4) OMW at 440 mL, and (5) a mixture of 1.5 g sulfur, 146 mL water, 146 mL EDTA, and 146 mL OMW. Each treatment was applied in five equal volumes, and chelates were collected sequentially five times, following the leaching sequence. Columns were sectioned into three depths (0–5 cm, 5–10 cm, and 10–15 cm) along the column. Samples from each depth were analyzed for pH, EC, and both available and total HMs.



Scheme 1. (a) diagram showing the entire column experiment process; (b) column showing the three depths (5, 10, and 15) cm; (c) treatment added to the columns and receiving the leached solutions.

2.3. Calculation of the Bioaccumulation (BF) and Translocation Factors (TF)

(a) Bioaccumulation factor (BF):

BF, which indicates the plant's ability to accumulate HMs from soil into its body, was computed as the proportion between the average HMs concentration in the soil and the average HMs content in the plant shoot. This study followed the methods of [28,31–33], in which the bioaccumulation factor (**BF**) was calculated as the ratio of HM concentration in shoots to that in soil.

$$\text{BF} = \frac{C_1}{C_2} \quad (1)$$

where C_1 stands for the average concentration of a metal in a plant, and C_2 refers to the average concentration of the metal in soil.

(b) Translocation factor (TF):

TF refers to the ratio of metal concentration in the roots to that in the shoots, and is used to calculate the ability of a plant to transfer metals from the roots to the shoots using the following equation [34,35]:

$$\text{TF} = \frac{M_1}{M_2}$$

where M_1 and M_2 represent the mean of metal concentrations in the shoots and roots, respectively.

2.4. Statistical Analysis

This study used Statistix (V.8.1) and OriginPro (V. 2025) software to analyze the collected data of both greenhouse and column leaching experiments. Descriptive statistics, such as means and standard errors, were calculated for all measured variables. This study applied one-way or two-way ANOVA to test treatment effects on HMs concentrations, pH, EC, biomass, **BF**, and **TF**. Post-hoc separation of means employed the least significant difference (LSD) test and was used in both experiments for post-hoc comparisons. Statistical significance was tested at $p < 0.05$.

3. Results

3.1. Soil Physical and Chemical Characteristics

The physicochemical analysis of the soil used in this study showed the soil's average pH of 8.4 (Table 1), indicating an alkaline condition, potentially affecting the availability of nutrients and HMs contents [1]. This soil had lower salinity Ec (83) Ms/cm and a dominant sandy texture (70%), complemented by 20% silt and 10% clay. This soil contains 5.5% of calcium carbonate, which is considered slightly calcareous, and this can influence its pH and nutrient availability. The soil is notably low in N (0.06%) and phosphorus (0.064%), which are both essential for healthy plant growth and development. However, K levels are moderate (0.9%). The total concentrations of HMs in soil were as follows: Cd (53.6 mg kg⁻¹), Pb (84.9 mg kg⁻¹), Cu (47.1 mg kg⁻¹), Ni (53.1 mg kg⁻¹), Zn (106.4 mg kg⁻¹), and Mn (1168 mg kg⁻¹), as shown in Table 1.

Table 1. Physical and chemical properties of the soil used in this study.

Parameter	Mean
pH	8.4 ± 0.31
Ec Ms/cm	83 ± 4.2
Silt %	20
Clay %	10
Sand %	70
Caco3	5.5 ± 0.07
TN %	0.06 ± 0.003
TP %	0.064 ± 0.001
TK %	0.9 ± 0.003
Cd mg g ⁻¹	53.6 ± 0.9
Cr mg kg ⁻¹	94.5 ± 0.3
Cu mg kg ⁻¹	47.1 ± 0.53
Ni mg kg ⁻¹	53.1 ± 0.36
Pb mg kg ⁻¹	84.9 ± 0.08
Zn mg kg ⁻¹	106.4 ± 0.04
Mn mg kg ⁻¹	1168 ± 0.11

3.2. Some of the Essential Chemical Properties of OMWW

The analysis of OMWW showed that this liquid material is characterized by low pH (4.47 ± 0.06), high EC (14.2 ± 0.10), and high concentration of phenols (Table 2). It contains a significant amount of nitrogen (N), phosphorus (P), and potassium (K).

Table 2. Chemical characteristics of olive mill wastewater (OMW).

Characteristics	Value
pH	4.47 ± 0.06
EC dS/m	14.2 ± 0.10
Phenols mg/L	5370 ± 3.21
Total N mg/L	845 ± 0.05
NH ₄ -N mg/L	32.5 ± 0.04
Nitrate-N mg/L	<1.13
Total P mg/L	515 ± 0.07
K mg/L	3690 ± 2.56

3.3. Heavy Metal Leaching Across Pour Volumes

Cd leaching from untreated soil was negligible in V1 (Figure 1A) and peaked at V2 (20.91 mg·kg⁻¹) (Figure 1B). EDTA treatment-initiated Cd mobilization from V1 (88.5) mg·kg⁻¹, peaking at V2 (221.4) mg·kg⁻¹, and declining thereafter through V3 (155.5) mg·kg⁻¹ (Figure 1C), V4 (57.41) mg·kg⁻¹ (Figure 1D), and V5 (34.28) mg·kg⁻¹ (Figure 1E). Additionally, Cd release after OMW treatment applied to soil was moderate in V1 (47.8) mg·kg⁻¹ and higher in V3 (171.6) mg·kg⁻¹. Sulfur application led to early Cd detection in V1 (38.6) mg·kg⁻¹ and V2 (54.3) mg·kg⁻¹, with no subsequent leaching. The mixture treatment started with 58.8 mg·kg⁻¹ in V1 and peaked in V3 (175.3 mg·kg⁻¹), suggesting an initial release phase followed by stabilization. However, Cu was not detectable in the control. The EDTA amendment significantly increased Cu leaching, starting at V1 0.05 mg·kg⁻¹ in V1 and peaking at 2.8 mg·kg⁻¹ in V3, with elevated levels persisting in V4 and V5. OMW treatment resulted in minimal Cu release, beginning at 0.01 mg·kg⁻¹ in V1 and rising to 0.06 mg·kg⁻¹ in V3. Sulfur-treated soil showed no Cu detection. The mixture led to moderate Cu leaching, initiating at 0.15 mg·kg⁻¹ in V1 and peaking at V3 (0.24) mg·kg⁻¹. Manganese (Mn) was absent in untreated soil. EDTA treatment resulted in Mn release from V1 (2.42) mg·kg⁻¹, increasing to 5.19 mg·kg⁻¹ at V3. OMW released Mn early in V1 (11.8) mg·kg⁻¹, which peaked at V3 (49.03) mg·kg⁻¹ and remained high in later pours. Sulfur released only minimal Mn in V1 (0.67) mg·kg⁻¹. The mixture treatment showed Mn leaching of 6.12 mg·kg⁻¹ in V1, increasing to the highest value of 51.98 mg·kg⁻¹ at V3. Nickel (Ni) mobility in untreated soil was limited to a low peak at V2 (0.13) mg·kg⁻¹, with no detection in V1. EDTA treatment began at 0.05 mg·kg⁻¹ in V1 and peaked at 0.17 mg·kg⁻¹ in V2. OMW released 0.09 mg·kg⁻¹ in V1, reaching a maximum at V3 (0.29) mg·kg⁻¹. Pb was not found in the untreated soil. The most dramatic leaching was observed with EDTA, which released 101.7 mg·kg⁻¹ in the first leach (V1) and peaked at 340.8 mg·kg⁻¹ in V3. The sulfur treatment was effective at limiting Pb mobility, showing no detection in V1 and very low levels after V2. OMW treatment released smaller amounts, from 2.3 mg·kg⁻¹ in V1 to 7.05 mg·kg⁻¹ in V3. The mixture treatment had an initial reading of 0.08 mg·kg⁻¹ in V1 and a peak of 0.12 mg·kg⁻¹ in V3. The mixture mobilized 143.00 mg·kg⁻¹ in V1 and peaked at 389.00 mg·kg⁻¹ in V3, suggesting enhanced early transport. Zinc (Zn) leaching was undetectable in untreated soil. EDTA introduced Zn leaching at 0.52 mg·kg⁻¹ in V1 and peaked at 1.01 mg·kg⁻¹ in V3. OMW treatment began with 0.02 mg·kg⁻¹ in V1 and peaked at 0.09 mg·kg⁻¹ in V3. Sulfur showed no Zn leaching throughout. Mixture treatment revealed that Pb levels were 0.35 mg·kg⁻¹ in V1 and increased to 0.93 mg·kg⁻¹ in V3. Across all the collected volumes, Pb steadily showed the highest leaching concentrations, with particularly prominent peaks in V2 and V3. This pattern indicates that Pb was a mobile metal or at least strongly held within the column system or retained by the soil. However, HMs of Cd, Cu, and Zn showed low levels throughout the experiment, indicating an effective immobilization, or were present

in limited quantities. While Mn and Ni revealed moderate leaching patterns, their concentrations progressively increased in later pour volumes (V4 and V5), suggesting delayed mobilization. The result of the ANOVA (one-way) showed that the variations in metal concentrations across various treatments were highly significant ($p < 0.001$) for each metal at each pour volume.

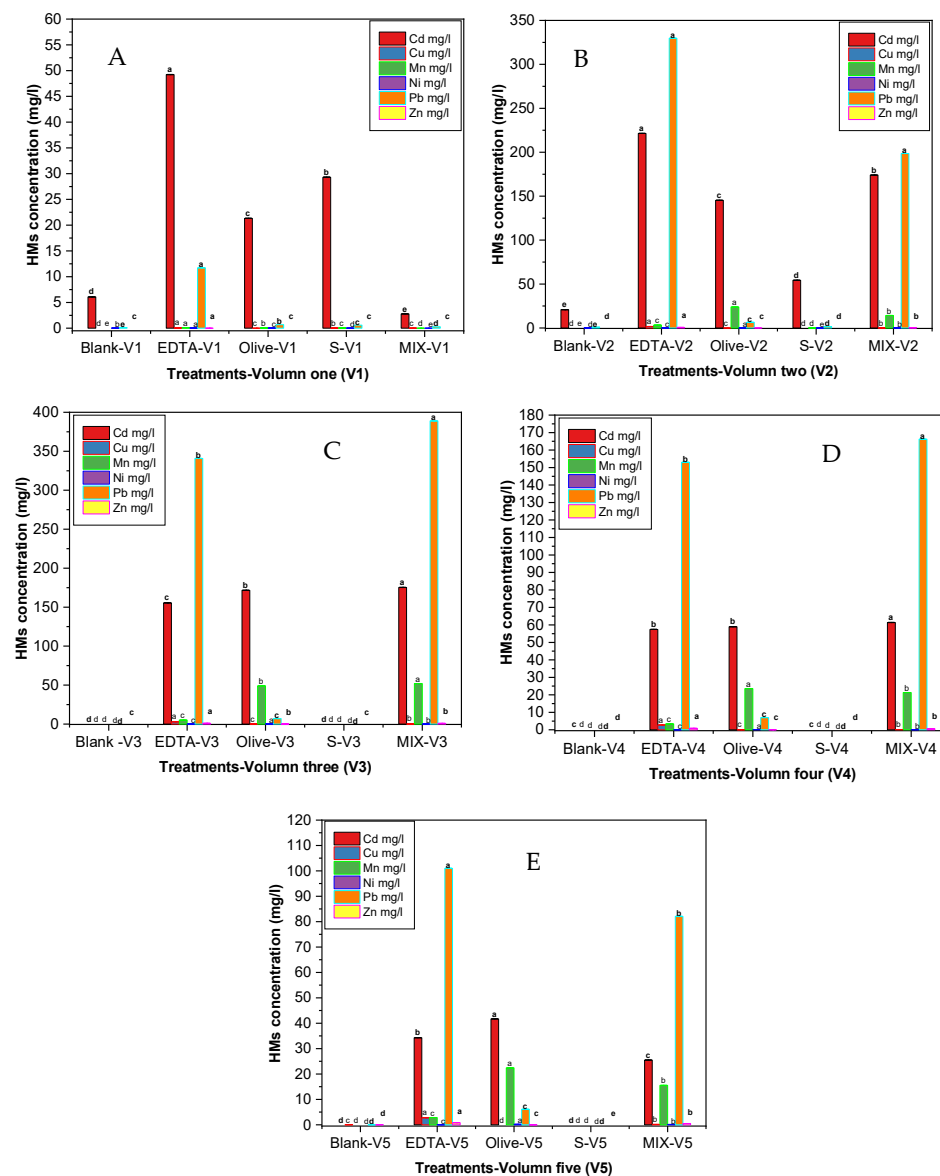


Figure 1. HMs concentrations, as influenced by different treatments at five pour volumes (V1). (A–E) for (V5). Panel (A–E) represents the same elements (Cd, Cu, Mn, Ni, Pb, and Zn). Means with the same letters are not significantly different from each other according to the LSD test ($p < 0.05$).

3.4. pH and Electrical Conductivity (EC)

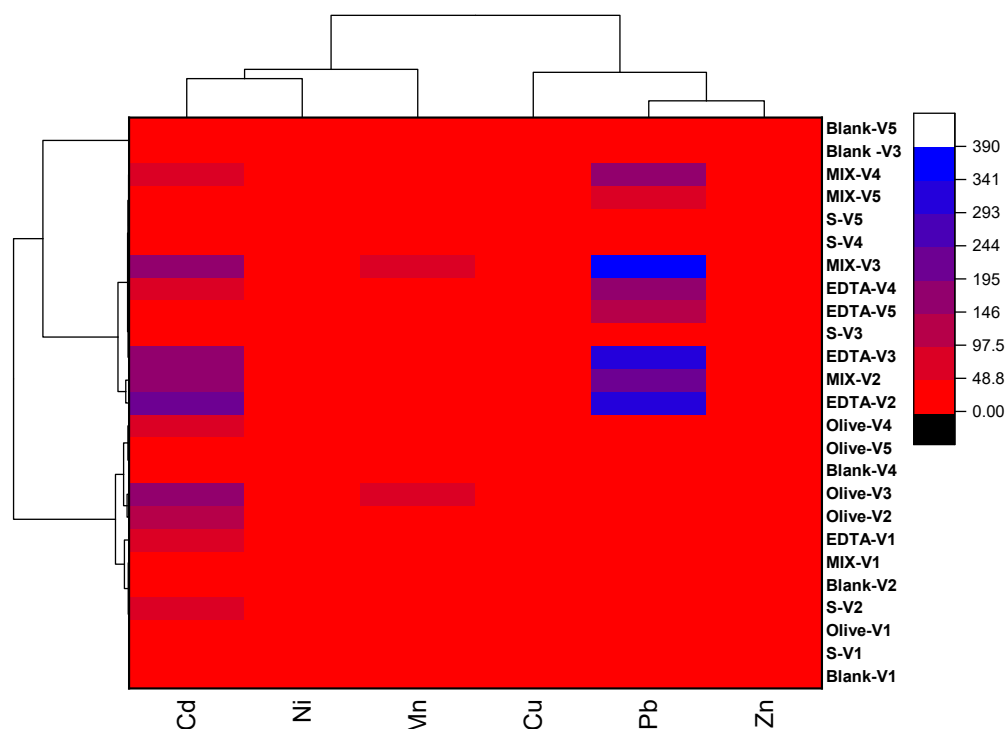
Table 3 showed that the pH remained nearly constant (7.6–7.8), and EC levels were low in the untreated soil, ranging from 107 to 140 $\text{mS}\cdot\text{cm}^{-1}$ (Table 3). In the treated soil with EDTA, pH significantly reduced from 7.7 at 5 cm depth to 5.3 at 15 cm depth, and dramatically increased EC values from 190 to 1763 $\text{mS}\cdot\text{cm}^{-1}$. This suggests the mobilization of metal ions and dissolved salts. OMW treatments produced a slight pH reduction (7.0 to 6.9) with high EC across depths (672–935) $\text{mS}\cdot\text{cm}^{-1}$. Sulfur treatments resulted in stable pH values (7.6–7.7) and low EC (120–146) $\text{mS}\cdot\text{cm}^{-1}$. However, the mixture treatment exhibited a modest pH drop (7.4 to 7.6) and EC increase from 390 to 554 $\text{mS}\cdot\text{cm}^{-1}$ (Table 3).

Table 3. Soil pH and Ec at three different depths of column.

Treatment	pH			EC (ms/cm)		
	5 cm	10 cm	15 cm	5 cm	10 cm	15 cm
Untreated soil	7.6	7.8	7.6	140	107	111
EDTA	7.7	7	5.3	190	320	1763
Olive	7	6.8	6.9	850	672	935
Sulfur	7.6	7.7	7.7	138	120	146
Mixture	7.4	7.3	7.6	390	402	554

3.5. Metal Distribution Patterns and Leaching Trends Across Sequential Pour Volumes in Amended Soil Columns

The distribution of the targeted metals (Cd, Mn, Cu, Pb, and Zn) across the column leaching experiment is shown in the heatmap below (Figure 2). The sequential pour volumes (V1 through V5) represent the progressive stages of metal elution. As a result, most metal combinations showed consistently low levels. Pb concentration, however, showed a markedly different pattern, which highlights its elevated presence compared to the other HMs.

**Figure 2.** Heatmap showing the effect of soil remediation amendments on heavy metal leaching across sequential pour volumes (V1–V5) in a column experiment.

In the initial pour (V1), Pb levels remained minimal across all treatments (mix, sulfur, EDTA, and olive). This implies strong initial sorption or the delayed mobilization of bound fractions. This was noticed in the sample mixture (V3 and V4), sulfur (V4), EDTA (V3 and V4), and OMW (V4), where the highest concentrations were observed. Control or untreated soil showed negligible concentrations, which highlights the role of the amendments in facilitating Pb release. Consequently, EDTA and mixture treatments are more effective in promoting Pb elution than sulfur and OMW.

4. Plant Response to Phytoremediation

4.1. Dry Matter

For the Sunflower crop (Figure 3), control (S0) resulted in a DM of 1.5 g/pot. Sulfur treatments at 1 ton/ha and 2 ton/ha achieved DM values of 1.9 g/pot and 2.1 g/pot, respectively. EDTA treatments at 25 mmol/kg and 50 mmol/kg had DM of 2.3 g/pot and 2.6 g/pot, respectively, which were the highest among all treatments. OMW at 5% and 10% showed DM values of 2.1 g/pot and 1.9 g/pot, respectively. Combined treatments (low and high) had DM values of 2.2 g/pot and 1.7 g/pot, respectively.

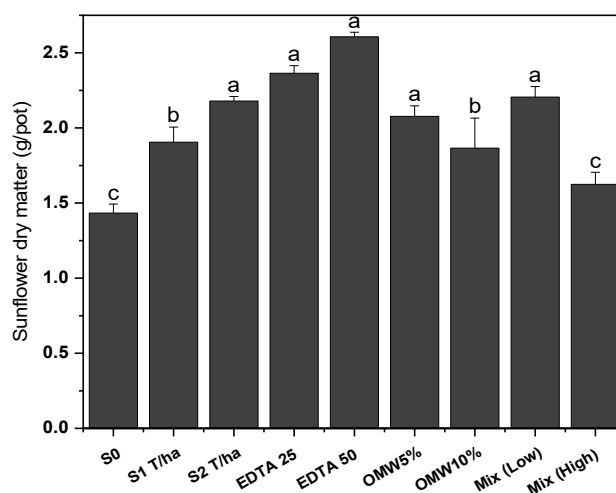


Figure 3. Effect of sulfur, OMW, and EDTA amendments on the dry matter of sunflower plants. Means with the same letters are not significantly different from each other according to the LSD test ($p < 0.05$).

4.2. Total Heavy Metals (HMSs) Content in Sunflower Shoots

Sunflower shoots in the untreated soil (S0) had low levels of Cr (3.5 mg kg^{-1}) (Figure 4a), Cu (0.73 mg kg^{-1}) (Figure 4b), Ni (0.58 mg kg^{-1}) (Figure 4c), and Zn (0.25 mg kg^{-1}) (Figure 4d), and intermediate concentrations of Cd (114.5 mg kg^{-1}) (Figure 4e), Mn (20 mg kg^{-1}) (Figure 4f), and Pb (32 mg kg^{-1}) (Figure 4g). While Cu (0.83 mg kg^{-1}) and Ni (0.68 mg kg^{-1}) remained low, sulfur application at 1 T ha^{-1} dramatically decreased Cd to 58.25 mg kg^{-1} and increased Mn (25.8 mg kg^{-1}) and Pb (73.25 mg kg^{-1}). With no change in Ni and considerable Zn (3.75 mg kg^{-1}), Cd decreased further to 14.75 mg kg^{-1} ; Cr increased to 7.75 mg kg^{-1} , and Cu increased to 1.1 mg kg^{-1} at 2 T ha^{-1} .

Cd uptake was significantly mitigated by EDTA treatments; the lowest content was at 50 mmol kg^{-1} (0.58 mg kg^{-1}). While Cu remained in the $1.05\text{--}0.95 \text{ mg kg}^{-1}$ range, EDTA 25 mmol kg^{-1} also produced low Mn (12.5 mg kg^{-1}) and Cr (0.75 mg kg^{-1}). With low contents of Cd (22.25 mg kg^{-1}), OMW at 5% significantly increased Cr (263.8 mg kg^{-1}), Mn ($138.25 \text{ mg kg}^{-1}$), Ni (202.8 mg kg^{-1}), and Cu (10.25 mg kg^{-1}). OMW 10% produced intermediate amounts of Cd (55.5 mg kg^{-1}), Cu (2.5 mg kg^{-1}), and the highest levels of Pb (97 mg kg^{-1}) and Zn (34.5 mg kg^{-1}). Treatment of mixture (low) decreased Cd (9.5 mg kg^{-1}) and raised Cr (123 mg kg^{-1}) and Ni (92 mg kg^{-1}), while Cu was at 2.5 mg kg^{-1} . Mn (38.3 mg kg^{-1}), Pb (16.25 mg kg^{-1}), Zn (27.8 mg kg^{-1}), and Cu (2.8 mg kg^{-1}) were all raised with mix (high), whereas Cr (53.3 mg kg^{-1}) and Cd (12.8 mg kg^{-1}) were slightly increased.

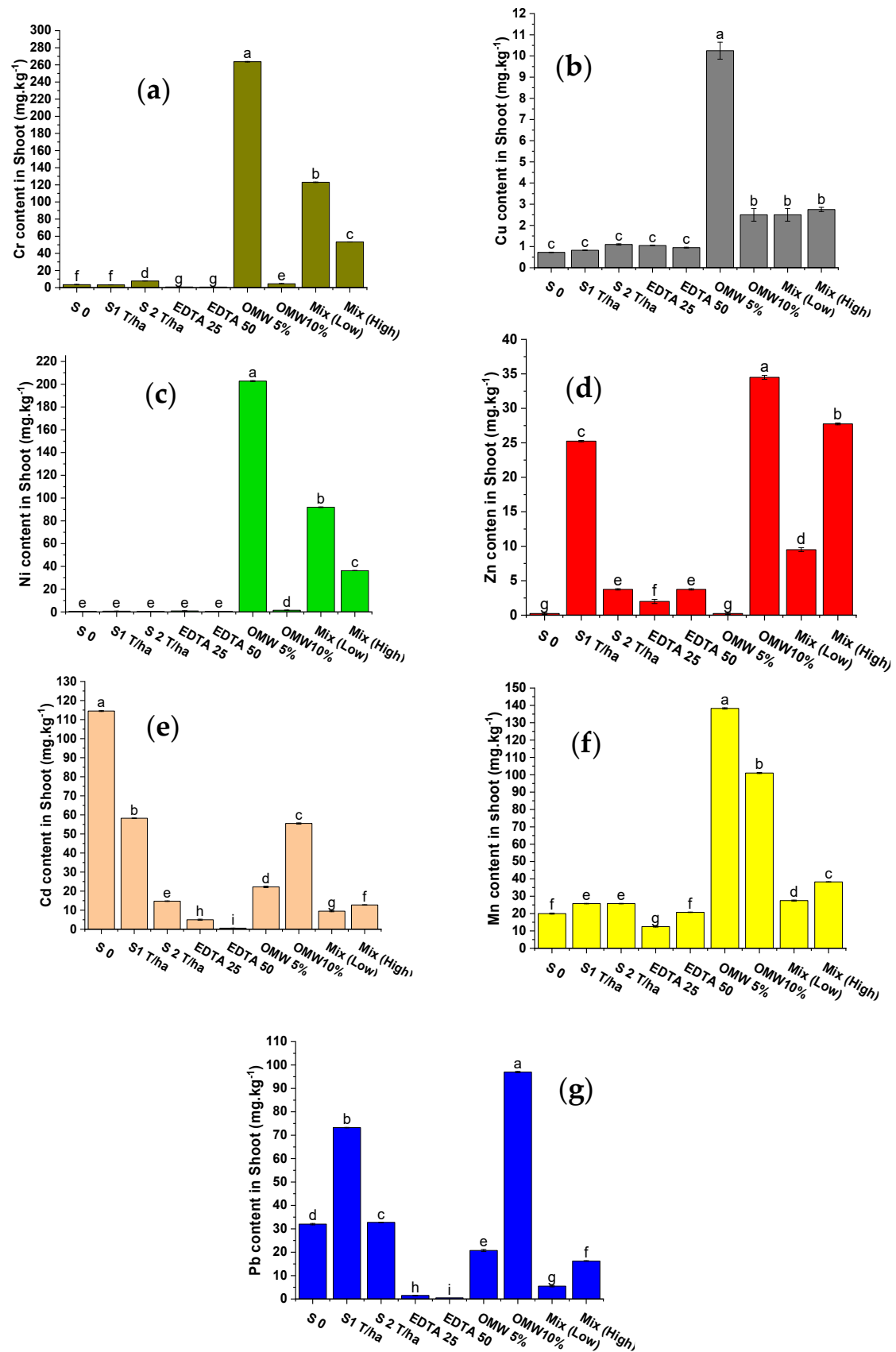


Figure 4. Effect of sulfur, OMW, and EDTA soil amendments on HMSs uptake by the shoots of sunflower, (a) represents total uptake of Cr, (b) Cu, (c) Ni, (d) Zn, (e) Cd, (f) Mn, and (g) Pb. Means with the same letters are not significantly different from each other according to the LSD test ($p < 0.05$).

4.3. Total Heavy Metals (HMSs) in Sunflower Roots

For sunflower roots (Figure 5a,b), the untreated control (S0) showed relatively high levels of Cd (553 mg kg^{-1}), Mn (301.8 mg kg^{-1}), and Pb (472.8 mg kg^{-1}). Sulfur application at 1 ton/ha significantly increased Cd uptake to 733.5 mg kg^{-1} , while reducing Cr to 3 mg kg^{-1} . At 2 ton/ha, Cd levels decreased to 146.5 mg kg^{-1} . EDTA treatment, especially at 25 mmol kg^{-1} , resulted in a decrease in Cd (114.5 mg kg^{-1}) and Mn (165.5 mg kg^{-1}) levels, while Ni uptake remained moderate (17.5 mg kg^{-1}). At 50 mmol/kg of EDTA, Mn uptake increased significantly to 743.3 mg kg^{-1} . These findings showed that OMW treatment at 5% Pb led to a moderate Cd (278.5 mg kg^{-1}) level, while the Mn was at (471.5 mg kg^{-1}) level. However, OMW at the 10% level revealed that Cd declined to 236.3 mg kg^{-1} , and Mn also reduced to 221.2 mg kg^{-1} . The low rate mixture showed moderate Cd (234.7 mg kg^{-1}) and Mn (314.8 mg kg^{-1}) levels. The high-rate mixture led to a significant increase in Ni uptake (100.2 mg kg^{-1}), moderate Cd (258 mg kg^{-1}), and Mn (296 mg kg^{-1}) levels.

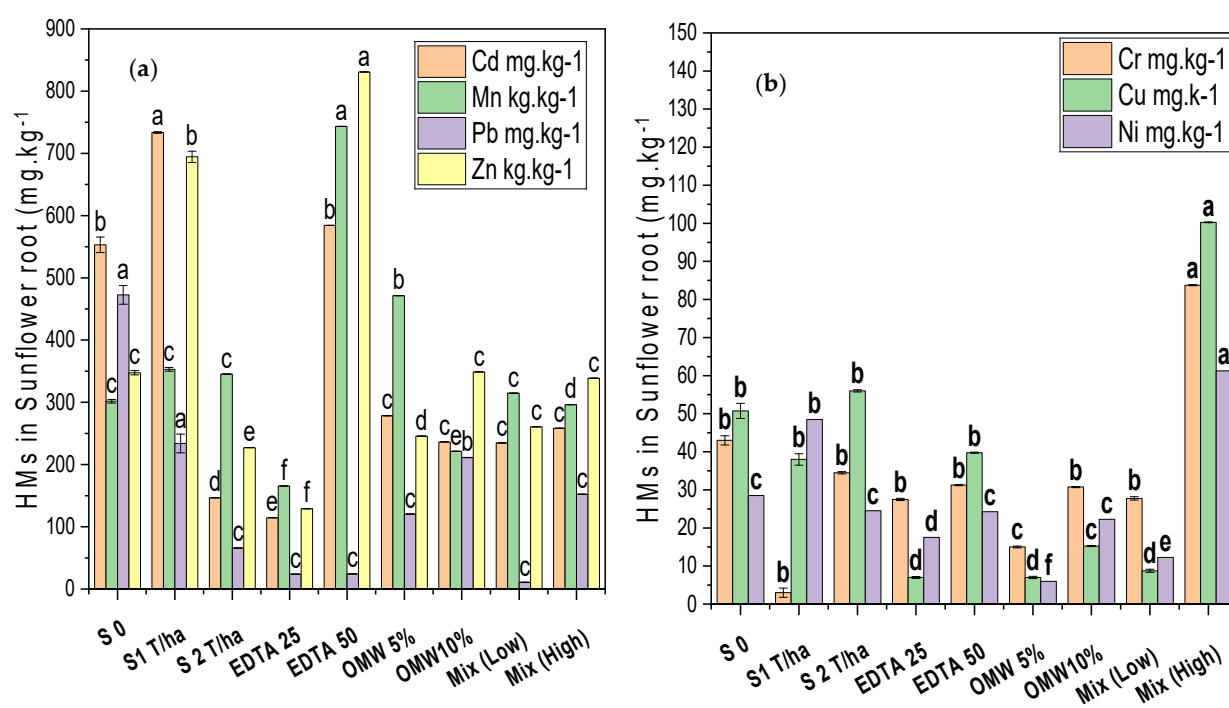


Figure 5. Effect of sulfur, OMW, and EDTA soil amendments on HMS uptake by roots of sunflower, panel (a) represents total HMSs of Cd, Mn, Pb, and Zn, while panel (b) represents Cr, Cu, and Ni. Means with the same letters are not significantly different from each other according to the LSD test ($p < 0.05$).

4.4. Available Heavy Metals (HMSs) as Extracted by DTPA Following Sunflower Growth

Available HMSs in soil followed by sunflower growth are described in Figure 6a–f. Cd (Figure 6a) availability was highest in the control (S0) at 10.36 mg kg^{-1} . EDTA treatments generally reduced Cd availability, with EDTA 50 mmol kg^{-1} showing the lowest available Cd at 3.4 mg kg^{-1} . Mn (Figure 6b) availability was highest with OMW 5% at 6.06 mg kg^{-1} , while EDTA 50 mmol kg^{-1} showed lower Mn availability at 12.9 mg kg^{-1} . Pb (Figure 6c) availability was highly increased by OMW 10% to 54.2 mg kg^{-1} . Zn (Figure 6d) availability rose to 1.72 mg kg^{-1} with EDTA 50 mmol/kg treatment. Cu (Figure 6e) was most available under EDTA 50 mmol kg^{-1} (0.85 mg kg^{-1}), followed by mix (low) (0.24 mg kg^{-1}). The lowest Cu availability occurred in OMW 5% (0.078 mg kg^{-1}). Ni (Figure 6f) availability was highest under S-2 T/ha (0.58 mg kg^{-1}), followed by EDTA 50 mmol kg^{-1} (0.48 mg kg^{-1}).

The lowest Ni availability was found in the mix (high) (0.015) mg kg⁻¹ and OMW 10% (0.022) mg kg⁻¹.

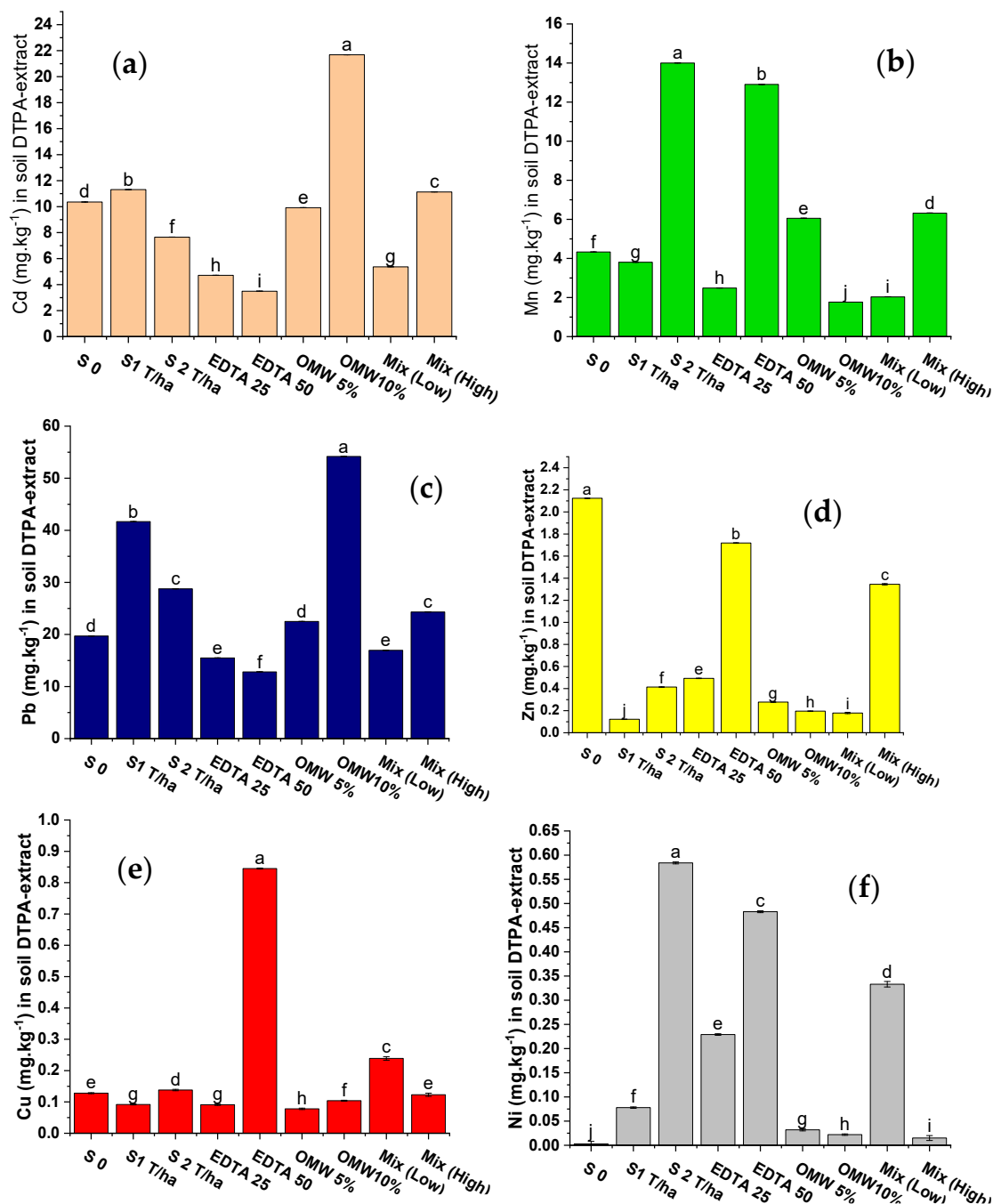


Figure 6. DTPA-extracted HMs from soil after harvesting. Panel (a) represents HM of Cd, (b) Mn, (c) Pb, (d) Zn, (e) Cu, and (f) Ni. Means with the same letters are not significantly different from each other, according to the LSD test ($p < 0.05$).

4.5. Effects on TF and BF

4.5.1. TF Calculation

TF shows the extent to which HMs are transported from the roots to the shoots of the sunflower plants (Table 4). The results revealed varying values of translocation, depending on the treatment and the metal. Consequently, OMW treatment at 5% level displayed high values of TF for various metals, for instance, Cr (17.58), Cu (1.46), and Ni (33.79), suggesting significant mobility of these metals from the roots to the shoots (Table 4). In contrast, EDTA

50 treatment showed low TF for most metals, with Cd showing TF at 0. Treatment of S0 showed that Cd had a TF of 0.21, while EDTA 50 showed a TF of 0.

Table 4. Translocation factor (TF) of (Cd, Cr, Mn, Cu, Ni, Pb, and Zn) in sunflower shoot. Means with the same letters are not significantly different from each other according to the LSD test ($p < 0.05$).

TRT	Cd	Cr	Mn	Cu	Ni	Pb	Zn
SO	0.21 ± 0.03 ^b	0.08 ± 0.002 ^{fg}	0.07 ± 0.001 ^f	0.01 ± 0.001 ^e	0.02 ± 0.005 ^d	0.07 ± 0.008 ^g	0 ± 0 ^c
S1 T/ha	0.08 ± 0.005 ^d	1.06 ± 0.02 ^c	0.07 ± 0.002 ^{ef}	0.02 ± 0.008 ^e	0.01 ± 0.003	0.31 ± 0.003 ^d	0.04 ± 0.009 ^{bc}
S2 T/ha	0.1 ± 0.011 ^c	0.22 ± 0.003 ^e	0.07 ± 0.008 ^f	0.02 ± 0 ^e	0.02 ± 0.003 ^e	0.5 ± 0.006 ^b	0.02 ± 0.006 ^c
EDTA 25	0.04 ± 0.008 ^e	0.03 ± 0.003 ^g	0.08 ± 0.005 ^{ef}	0.15 ± 0.005 ^c	0.05 ± 0.001 ^e	0.06 ± 0.006 ^g	0.02 ± 0.05 ^a
EDTA 50	0 ± 0 ^f	0.02 ± 0.008 ^g	0.03 ± 0.001 ^g	0.02 ± 0.001 ^e	0.02 ± 0.008 ^e	0.02 ± 0.008 ^h	0 ± 0 ^c
OMW 5%	0.24 ± 0.001 ^d	17.58 ± 0.08 ^a	0.29 ± 0.003 ^b	1.46 ± 0.01 ^a	33.79 ± 0.06 ^a	0.17 ± 0.003 ^e	0 ± 0 ^c
OMW 10%	0.08 ± 0.001 ^a	0.15 ± 0.003 ^{ef}	0.46 ± 0.003 ^a	0.16 ± 0.005 ^c	0.07 ± 0.001 ^e	0.46 ± 0.003 ^c	0.1 ± 0.008 ^a
Mix (Low)	0.04 ± 0.006 ^e	4.43 ± 0.008 ^b	0.09 ± 0.006 ^d	0.29 ± 0.002 ^b	7.51 ± 0.06 ^b	0.51 ± 0.009 ^a	0.04 ± 0.009 ^{bc}
Mix (High)	0.05 ± 0.005 ^e	0.64 ± 0.003 ^d	0.13 ± 0.003 ^c	0.03 ± 0.002 ^d	0.59 ± 0.003 ^c	0.11 ± 0.006 ^f	0.001 ± 0.001 ^{ab}
LSD	0.01	0.08	0.06	0.01	0.07	0.01	0.04

4.5.2. BF Calculation

OMW treatment at 5% level resulted in higher BF values for various HMs (Table 5). This represents an increased concentration of these metals by the sunflower plants. This is particularly evident for metals like Cr (2.6), Cu (0.19), and Ni (3.95). EDTA treatment showed minor BF values, which suggests that the accumulation of metals by the plants was significantly reduced. As a result, EDTA at 50 showed a BF of 0.02 for Cd, significantly lower than the control (S0) with a BF of 2.14. These results underscore the effect of these amendments on the overall accumulation of HMs in sunflower biomass.

Table 5. Bioaccumulation factor (BF) of (Cd, Cr, Mn, Cu, Ni, Pb, and Zn) in sunflower shoot. Means with the same letters are not significantly different from each other according to the LSD test ($p < 0.05$).

TRT	Cd	Cr	Mn	Cu	Ni	Pb	Zn
SO	2.14 ± 0.008 ^a	0.04 ± 0.005 ^{de}	0.02 ± 0.002 ^d	0.02 ± 0.003 ^e	0.01 ± 0 ^d	0.38 ± 0.006 ^c	0 ± 0 ^f
S1 T/ha	1.55 ± 0.04 ^b	0.03 ± 0.008 ^d	0.02 ± 0.009 ^d	0.02 ± 0.001 ^e	0.01 ± 0.005 ^d	0.57 ± 0.009 ^b	0.23 ± 0.009 ^c
S2 T/ha	1.43 ± 0.009 ^g	0.09 ± 0.001 ^{de}	0.03 ± 0.003 ^c	0.03 ± 0.005 ^{cd}	0.01 ± 0 ^d	0.41 ± 0.006 ^c	0.03 ± 0.003 ^e
EDTA 25	0.65 ± 0.01 ^e	0.01 ± 0.003 ^e	0.01 ± 0.003 ^e	0.03 ± 0.003 ^{de}	0.02 ± 0.003 ^d	0.02 ± 0.003 ^f	0 ± 0 ^e
EDTA 50	0.02 ± 0.003 ^h	0.01 ± 0.001 ^e	0.02 ± 0.005 ^d	0.02 ± 0.001 ^e	0.01 ± 0.001 ^d	0.01 ± 0 ^f	0.02 ± 0.001 ^e
OMW 5%	0.7 ± 0.001 ^h	2.6 ± 0.008 ^a	0.11 ± 0.009 ^a	0.19 ± 0.006 ^a	3.95 ± 0.03 ^a	0.32 ± 0.006 ^d	0 ± 0 ^e
OMW 10%	1.15 ± 0.001 ^c	0.05 ± 0.003 ^{de}	0.09 ± 0.003 ^b	0.06 ± 0.006 ^{bc}	0.03 ± 0.006 ^d	0.73 ± 0.006 ^a	0.39 ± 0.06 ^a
Mix (Low)	0.83 ± 0.008 ^d	1.3 ± 0.006 ^b	0.03 ± 0.001 ^c	0.06 ± 0.003 ^{bc}	1.67 ± 0.009 ^b	0.11 ± 0.001 ^e	0.01 ± 0.003 ^d
Mix (High)	1.49 ± 0.003 ^f	0.59 ± 0.01 ^c	0.03 ± 0 ^c	0.05 ± 0.003 ^{bc}	0.74 ± 0.01 ^c	0.34 ± 0.01 ^d	0.25 ± 0.01 ^b
LSD	0.02	0.14	0.008	0.01	0.03	0.02	0.01

5. Discussion

5.1. Column Experiment

The column leaching experiment obviously emphasized the contrasting effects of soil amendments on HMs mobility. Cd showed the biggest response to EDTA treatment, with peak leaching detected at V2 (221.40) mg·kg^{−1} and V3 (155.50) mg·kg^{−1}. This agreed with EDTA's known chelation behavior, forming constant complexes that improve Cd solubility and vertical transport [36,37]. The decline in concentrations after V3 reflects a reduction in bioavailable Cd in surface layers.

Moreover, OMW also facilitated Cd leaching, peaking at V3 (171.6) mg·kg^{−1}; this can be due to the existence of organic acids and phenolic compounds in OMW, which promote metal solubilization [38,39]. In contrast, sulfur treatment considerably decreased Cd mobility, with Cd detected only in early pour volumes of V1 and V2 and peaking at 54.3 mg·kg^{−1}. Acidification from sulfur likely triggered Cd precipitation and enhanced its sorption into soil particles [23,40]. The combined application (mixture) exhibited notable

Cd leaching at V3 ($175.30 \text{ mg}\cdot\text{kg}^{-1}$), suggesting a balance between mobilization by EDTA and immobilization by sulfur.

Copper (Cu) was largely immobile under most treatments. However, EDTA significantly increased Cu leaching, with a peak value at V3 ($2.80 \text{ mg}\cdot\text{kg}^{-1}$), confirming its effectiveness in Cu chelation [22].

Treatments involving both OMW and sulfur revealed insignificant release of Cu. This is due to the fact that Cu tends to form consistent bonds with metal–oxide compounds and organic matter, which prevents it from dissolving into the solution [39,41]. In addition, the mixture treatment showed a moderate leaching ($0.24 \text{ mg}\cdot\text{kg}^{-1}$) at V3. This is probably due to the formation of intricate compounds by sulfur.

Mn leaching was most pronounced in the OMW treatment, reaching $49.03 \text{ mg}\cdot\text{kg}^{-1}$ at V3. This indicates that Mn becomes more soluble due to the presence of organic acids, which either reduce Mn oxides or compete for adsorption sites [39,42]. EDTA caused moderate Mn mobilization ($5.19 \text{ mg}\cdot\text{kg}^{-1}$ at V3), whereas sulfur significantly suppressed Mn movement, indicating potential redox-mediated immobilization [6,23]. The mixture treatment exhibited the highest Mn leaching at V3 ($51.98 \text{ mg}\cdot\text{kg}^{-1}$), suggesting additive or synergistic effects between chelation and acidification.

Ni showed consistently low mobility across the targeted treatments. In untreated soil, a slight increase in V2 ($0.13 \text{ mg}\cdot\text{kg}^{-1}$) was noticed. EDTA application relatively improved Ni leaching ($0.17 \text{ mg}\cdot\text{kg}^{-1}$) at V2, while OMW showed greater release ($0.29 \text{ mg}\cdot\text{kg}^{-1}$) at V3, probably through complexation with organic ingredients.

These findings showed that sulfur treatment resulted in a negligible influence on Ni mobility and was not detected beyond V2. This supports its ability to alleviate conversion metals as investigated by the research by [23,43]. Moreover, the mixture treatment resulted in a moderate rate of leaching at V3 ($0.12 \text{ mg}\cdot\text{kg}^{-1}$). Furthermore, Pb showed a similar pattern to Cd, with EDTA resulting in the highest leaching ($340.8 \text{ mg}\cdot\text{kg}^{-1}$ at V3). This can be attributed to EDTA's strong tendency to bind with Pb under neutral conditions. This is consistent with various studies by [6,44]. OMW promoted moderate Pb release ($7 \text{ mg}\cdot\text{kg}^{-1}$) at V3, which is likely due to the fact that the complexation is weak with dissolved organic matter, as agreed with those in [38]. However, sulfur was effective in reducing Pb leaching beyond a certain point (V2). According to [23,40], sulfur can potentially work to immobilize Pb through participation or binding it within the soil particles. Mixture treatment showed higher Pb concentration peaking at $389 \text{ mg}\cdot\text{kg}^{-1}$ at V3. This indicates an initial effective release of Pb followed by potential stabilization.

Zn leaching was most pronounced at EDTA treatment, reaching its highest level at V3 ($1.01 \text{ mg}\cdot\text{kg}^{-1}$). This is because Zn exhibits a moderate binding affinity for EDTA chelation, though typically lower than for Cd or Pb [6,45]. OMW and sulfur treatments induced minimal Zn leaching, with maximum values of $0.09 \text{ mg}\cdot\text{kg}^{-1}$ and non-detectable levels, respectively. The mixture resulted in moderate Zn release at V3 ($0.93 \text{ mg}\cdot\text{kg}^{-1}$). The consistent and statistically significant differences observed across pour volumes (ANOVA, $p < 0.001$) confirm that metal behavior varies markedly with elution stage, highlighting metal-specific mobility and the evolving influence of amendments over time. HMs of both Mn and Ni are more noticeable in the last volumes, which suggests either delayed mobilization or a buildup of their leaching over time. These patterns underline the significance of the extended monitoring times in remediation.

Our findings showed that pH and EC displayed stable values in the control soil, with a pH range between 7.6 and 7.8, indicating that the environment is nearly neutral to slightly alkaline, while EC showed lower values ($107\text{--}140 \mu\text{S}\cdot\text{cm}^{-1}$). This suggests minimal ionic movement within the control soil, as reported by [6,23]. Treatment of EDTA revealed a gradual reduction in pH with depth from 7.7 at 5 cm depth to 5.3 at 15 cm depth, while the

EC increased from 190 to 1763 $\mu\text{S}\cdot\text{cm}^{-1}$. This is in agreement with the findings by [6,36], who stated that EDTA plays an important role in acidification and ion mobilization.

Additionally, OMW treatment produced a slight reduction in pH (7–6.9) and maintained higher EC values (672–935) $\mu\text{S}\cdot\text{cm}^{-1}$. This can be attributed to the dissolved organic and inorganic solutes. Conversely, the treated columns with sulfur exhibited stable pH (7.6–7.7) and low EC (120–146 $\mu\text{S}\cdot\text{cm}^{-1}$), indicating minimal chemical alteration, whereas the mixture treatment resulted in modest reduction (7.4 to 7.6), while EC increases (390–554 $\mu\text{S}\cdot\text{cm}^{-1}$), reflecting a tempered response in comparison to other treatments.

5.2. Greenhouse Experiment

5.2.1. Dry Matter of Maize, Mustard, and Sunflower Plants

This study's examination of plant growth responses to soil amendments revealed distinct patterns among maize, mustard, and sunflower. The control DM yield of sunflower (1.5 g/pot) established a baseline for comparison. Sunflower's positive correlation between sulfur application and DM indicates a higher sulfur requirement or tolerance under these conditions. The consistent DM enhancement with EDTA highlights its potential to improve nutrient availability through chelation of micronutrients (e.g., Fe, Zn, Mn), which may have indirectly improved the biomass production and photosynthesis [6]. These moderate improvements observed in the mixture treatments suggest that a balanced approach for soil amendment could lead to optimal plant growth. This can moderate the extremes of single treatments by balancing the mechanisms of mobilization and stabilization, for instance, complexation by EDTA and organic acids (OMW) versus immobilization through the induced precipitation by sulfur. This approach can reduce the hazard associated with the large application rates of individual amendments.

5.2.2. Total Heavy Metals in Sunflower Shoots

The study of HMs accumulation in shoots showed that amendment type, application rate, and plant species interact in a complex way. Consequently, the low application rate of sulfur (1 ton/ha) led to increased Cd in the sunflower shoots. These results are in line with the findings by [46], who reported that sulfur promotes soil acidification and enhances Cd bioavailability by desorbing it from soil profiles. The sunflower plant showed a particularly unique response, where Cd levels have increased with low sulfur application, which may also promote rizosphere acidification through microbial sulfur oxidation. EDTA treatments, however, showed lower Cd uptake by the plant shoot, while it increased its solubility in soil. This can be explained by the formation of stable EDTA–Cd complexes that are too large or negatively charged to cross root membranes efficiently. The sunflower plant may also possess a distinct response to absorb and detoxify Cd compared to other plant species.

5.2.3. Total Heavy Metals in Sunflower Roots

Sunflower also demonstrated species-specific trends in metal uptake. Sulfur at 1 t/ha raised Cd to 733.5 $\text{mg}\cdot\text{kg}^{-1}$ in roots, most likely due to the increased solubility from acidification, while at 2 t/ha, Cd from root declined to 146.5 $\text{mg}\cdot\text{kg}^{-1}$, most likely as a result of competitive interactions at root surfaces that restrict absorption or precipitation brought on by the over-acidification [46].

EDTA at 25 $\text{mmol}\cdot\text{kg}^{-1}$ lowered Cd and Mn uptake, whereas the 50 $\text{mmol}\cdot\text{kg}^{-1}$ treatment increased Mn (743.3) $\text{mg}\cdot\text{kg}^{-1}$. This implies that stronger complexes with Mn may be formed via selective chelation at higher rates or concentrations, improving its solubility and availability for absorption. Conversely, excessive chelation for Cd may decrease root absorption because of the complex's low membrane permeability or competition with necessary ions [47]. Treatment of OMW showed moderate uptake at 5% and a slight reduction at 10%. This can be because organic matter-binding sites are saturated or because mobility

is restricted by the development of organometallic complexes. Mixture treatments at a high rate significantly improved Ni uptake ($100.2 \text{ mg} \cdot \text{kg}^{-1}$). This is probably because of the synergistic actions of chelators like EDTA and organic ligands in OMW, which mobilise Ni through ligand exchange and competitive adsorption [37].

5.2.4. Available Heavy Metals (DTPA-Extract) in Soils Following Plant Growth

The findings of this study revealed that Cd availability in untreated soil peaked at $(10.4) \text{ mg} \cdot \text{kg}^{-1}$ and declined with EDTA treatments at 50 mmol/kg with $(3.5) \text{ mg} \cdot \text{kg}^{-1}$. This is consistent with the theory that DTPA, which targets weakly bound or free ionic forms, can extract metals that are less readily bound in EDTA complexes. Successful chelation, as well as possible immobilization in stable complexes that plants cannot access, is reflected in the reduced extractability. Mn availability in sunflowers grown under the treatment of OMW 5% showed the highest value $(6.06) \text{ mg} \cdot \text{kg}^{-1}$, suggesting that Mn may have been released into the solution as a result of OMW's organic acids, reducing Mn oxides, or outcompeting Mn for soil binding sites. The fact that Ni availability was low in all treatments lends credence to the theory that Ni either adsorbs to soil colloids strongly or forms less soluble complexes, particularly in neutral to slightly acidic environments [48]. These findings emphasize the need to carefully manage sulfur and EDTA application rates in order to optimize metal availability for plant uptake without endangering excessive mobilization and possible leaching.

5.2.5. Changes in TF and BF

The findings of this study confirmed that **TF** and **BF** factors provide valuable insights into the efficiency with which plants uptake metals from the soil and translocate them within their tissues. The high **BF** (2.6) and **TF** (17.6) for Cr under OMW treatment indicate strong uptake and efficient root-to-shoot transport. This could be because Cr and organic acids in OMW generate soluble complexes that promote xylem mobility and root absorption.

In contrast, EDTA application showed very low **BF** ($0.02) \text{ mg} \cdot \text{kg}^{-1}$ and **TF** (0) for Cd in sunflower, despite increasing Cd solubility in soil. This, again, emphasises how low membrane permeability or incompatibility with root transport pathways may cause EDTA-Cd complexes to persist in the rhizosphere. These results are consistent with research demonstrating that EDTA can decrease plant absorption efficiency by complexation shielding effects even when it increases overall extractable metal concentrations [21,36,48,49].

6. Conclusions

The findings of this study discovered how treatments of EDTA, OMW, and sulfur and their mixtures influenced HMs in soil and their absorption by sunflower tissues. Consequently, EDTA is the most effective treatment that mobilizes HMs like Cd, Pb, and Cu, increasing their presence in the soil. However, it surprisingly decreased Cd uptake in shoots, regardless of producing more Cd available in the soil. This indicates that EDTA has a complex effect on how plants absorb HMs. This study also showed that sulfur played a double role: at lower rates, it acidified the soil, leading to increased Cd in sunflower shoots, while at higher rates, it facilitated a decrease in Pb and Mn leaching, probably by making these metals bind or form solids in the soil.

OMW notably and significantly improved the uptake and movement of Cr in sunflower, suggesting its ability to manage specific metals. Moreover, the mixture treatments led to consistent consequences in metal behavior. However, in some cases, integrated treatments outperformed individual amendments, most likely due to competition or antagonistic interactions that altered metal availability or uptake. This highlights the complexity

of interactions between amendments and the importance of selecting suitable combinations. In conclusion, these findings underscore the necessity of applying targeted remediation strategies, in which a clear understanding of the specific interactions between amendments and plants is crucial for effectively managing HMs contamination.

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