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CONTRIBUTION TO MINE TAILINGS TOXICITY ASSESSMENT

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ABSTRACT

Various chemical, biological, and biochemical assays are currently employed to evaluate the state of mine tailing dumps. However, some chemical approaches could overestimate the environmental footprint. This study presents an assessment of the current state of the inactive mine tailing dump area, employing a sequential chemical extraction procedure (SEP) to study heavy metal fractionation and contamination indices, thereby assessing the mobility and potential bioavailability. The studied samples contained elevated concentrations of As, Cu, Pb, and Zn, of which Pb was the most abundant element. The results showed that although high total heavy metal content in soil and mine tailing was observed, the mobility factors rarely exceeded 10 %. Moreover, the values of contamination factors, individual and global, were below 1 and 6, respectively. Thus, the mine tailings and surrounding soils could be classified as sources with low contamination potential. The applied approach could benefit the ongoing efforts toward the sustainable management and conservation of post-mining landscapes.

Keywords: mine tailings, surrounding soils, contamination, toxicity indices.

INTRODUCTION

Mining activity is associated with a wide range of environmental, ecological, health-related, and economic risks. Mine tailings are recognized as a global contaminant due to their complex composition [1]. A major environmental concern derives from the long-term persistence of contamination, which continues to affect the environment long after mining activities have ceased. Environmental risk, associated with mine tailings, encompasses both landscape degradation and the contamination of surrounding water and soil systems. Contaminated environmental sites are typically characterized by increased levels of heavy metal/loids, including arsenic (As), cadmium (Cd), chromium (Cr), lead (Pb), zinc (Zn), and iron (Fe). They are relatively non-biodegradable and are highly toxic even at low concentrations [1]. Prolonged exposure to contaminated water or soil can lead to bioaccumulation of toxic metals in the food chain, resulting in serious health implications. The environmental fate of these tailings, the condition of the surrounding ecosystems, the effectiveness of remediation strategies, and the impact on human health have been the subject of extensive investigation. It is advisable and relevant to have a sustainable remediation strategy, which is crucial for effective environmental monitoring and management. To address the challenges, a comprehensive study on different ecotoxicity approaches and remediation techniques has been conducted.

From a practical point of view, the pollution degree of an environmental area can be estimated by single approaches or by combining different ones. A key objective in recent scientific studies is the evaluation of the bioavailability of toxic heavy metals in contaminated soils and waters. While measurements based mostly on total heavy metal concentrations may overestimate ecological and human health risks, it is essential to quantify the bioavailable fraction to obtain a more accurate risk profile. Notably, even the non-bioavailable fraction of heavy metals can become mobile and bio accessible under favourable environmental conditions, such as changes in pH, redox potential, or microbial activity. It is not a coincidence that one of the most applied procedures for ecological risk assessment is the sequential extraction procedure (SEPs). It intends to simulate different environmental conditions, such as rainwater acidification, oxidation, or reduction [2, 3]. The application of SEPs is noticed on a variety of sample matrices under different experimental conditions. It should be noted that the SEPs are influenced by different factors, such as reagent concentrations, extraction time, mass-to-volume ratio, scaling of the experiment, etc. [3, 4]. Nevertheless, these methods of selective extraction are useful in the exploration of the chemical behaviour of targeted heavy metals and are often applied as an approach for toxicity estimation of numerous environmental matrices, i.e., water, soil, mine tailing, and sludge [1 - 3, 5]. It should be mentioned that different SEPs are available, and attention should be paid when choosing an extraction procedure. As it was mentioned above, all commonly known methodologies are based on approximately the same principles- the differences appear to be due to the various reagent concentrations and combinations of reagents, so the leaching process does not completely match. For that reason, the results interpretation should be carried out unambiguously. Regardless of the confirmed positive aspects of SEPs, it is sometimes necessary to apply additional approaches.

In the scientific literature, various remediation strategies appear. The global aim is to reduce the toxic concentrations of heavy metals and to decrease the harmful effects on the environment and human health. The selection of an appropriate remediation technique should be guided by site-specific factors, including the geographical location of the contaminated area, the presence of co-contaminants, previously recorded heavy metal concentrations, and the projected costs of the remediation process. It is known that soil microorganisms can influence organic contaminants, while heavy metal/loids need physical or chemical removal [6].

A few papers proposed a remediation classification based on the physical, chemical, and biological methods applied [7, 8]. According to Ghosh et al., most of the conventional ex-situ remediation techniques require the removal of contaminated soil from the polluted area, followed by appropriate treatment and subsequent restoration. Most often, this approach is expensive and harms the ecosystem. Contrary to this, the in-situ approaches are characterized by reduced influence on the environment. In their scientific review, the authors emphasize techniques such as rhizofiltration, phyto stabilisation, phytoextraction, phytovolatilization, and phytodegradation.

In a recent paper, the classification of different remediation techniques is described in detail. The advantages and limitations of some common approaches are discussed. The focus is on the biological remediation techniques for heavy metal decontamination of soil and water systems using microbes [8].

Another approach for estimating the toxicity of mine tailings, as well as surrounding water and soil samples, is reported by Das et al. [8]. This research is based on a germination assay. As a control plant, a Triticum aestivum is applied. This research demonstrated that seedlings cultivated in these soils accumulate increased levels of heavy metals, suggesting they are useful as biological indicators for estimating contaminant bioavailability. By integrating multiple analytical approaches, such as sequential extraction, chemical characterization, and biological assays, the study contributes to the development of an algorithm for toxicity assessment of contaminated sites. Remediated landfills, despite initial stabilization, may become uncontrolled sources of toxic compounds with unpredictable environmental behaviour. Therefore, understanding the correlation between heavy metal leaching and soil absorption characteristics is critical for evaluating the long-term effectiveness of remediation strategies.

The primary objective of this study is to assess the current state of mine tailings by applying a sequential chemical extraction procedure and contamination indices. The algorithm is designed to support ongoing efforts toward the sustainable management and conservation of post-mining landscapes. This integrative approach facilitates the identification of residual contamination and potential ecological risks.

EXPERIMENTAL

Materials and methods

Soil sampling

The analysed samples were obtained from the mine tailings and surroundings in Tarniţa-Suceava, Romania. The activity of the polymetallic mine was closed in 2006, and some remediation procedures were applied in accordance with well-accepted practices. A detailed description of the area can be found in Ilieva et al. and Necula et al. [9, 10]. Samples were taken from the mine tailings and the surrounding soils as follows: (P1) soil sample taken at 2 m from the river and near the main tailing dump; (P2) soil sample taken 100 m from the tailing dump; (P3) collected from the tailing dump. An arable soil sample (agricultural soil from the Vidin region in Bulgaria) was used for comparison.

Analytical instruments and reagents

All used reagents were of analytical purity grade. All dilutions were made using distilled water. Multi-element standard solution Ultra Scientific, containing 24 elements and arsenic standard solution in 5 % HNO_3, was used for preparing ICP-OES calibration curves. A Prodigy High Dispersion ICP-OES, Teledyne Leeman Labs, USA, equipped with a dual view torch, a cyclonic nebulization chamber, and a concentric nebulizer, was used. At least three replicates were performed to quantify the analytical signal. A set of calibration standards with an appropriate concentration range, depending on the sample concentrations, was prepared. The pH measurements were conducted by a Jenway pH meter with a combined glass electrode BOECO, Germany.

Procedures

The procedures for analysing the soil and tailing dump material for determination of reactive acidity, cation exchange capacity, a five-step sequential extraction procedure, and total heavy metal content are presented in detail in a previous study [9]. For conducting the SEP, five different solutions were prepared: (1) 1 M $Mg(NO_3)_2$; (2) 0.1 M $NH_2OH.HCl$; (3) 0.25 M $NH_2OH.HCl$ + 0.25 M HCl; (4) 0.25 M $(NH_4)_2C_2O_4$ + 0.1M $H_2C_2O_4$ + 0.1 M $C_6H_8O_7$; (5) HNO_3 + HCl (1 : 3).

Calculation of contamination indices

The individual and global contamination indices (ICI and GCI, respectively) were calculated based on the sequential extraction data following the approach proposed by Necula et al. [10].

Individual contamination factors (ICF) and global contamination factors were calculated following the equations Eq.(1) and Eq. (2):

$$\mathrm{ICF} = \frac{F_1+F_2+F_3+F_4}{F_5} \qquad (1)$$

$$\mathrm{GCF} = \Sigma\mathrm{ICF} \qquad (2)$$

The thresholds proposed by Necula et al. and Matabane et al. [10, 11] were applied to the results of the sequential procedure to classify the heavy metals according to their contamination potential.

Mobility factors (MF) were calculated based on Eq. (3) and the strategy presented in Zhao et al. [12]:

$$\mathrm{MF} = \frac{F_1}{F_1+F_2+F_3+F_4+F_5} \times 100 \qquad (3)$$

where: F1, F2, F3, F4, and F5 are the concentrations of heavy metal in each sequential extraction solution. As proposed by the authors, the MF above 10 % denotes an element that exhibits high mobility under the sequential extraction conditions and could be leached under environmental conditions.

RESULTS AND DISCUSSION

Sample characteristics

The chemical composition and some physicochemical characteristics of the studied samples are summarized in Table 1. It should be noted that the low pH of the aqueous slurry of the tailing material and soils collected near the tailing dump signifies the enhancement of the solubility and mobility of heavy metals. The cation exchange capacity (CEC) of samples P1 and P2 was relatively low (4.18 and 2.20 cmol kg^{-1}, respectively). Sample P3 (tailing material) showed a high CEC, possibly due to the fine particle size and increased reactivity after the flotation processing of the ore. The high level of exchangeable aluminium indicated the potential for increased release of protons (H^+) into aqueous solution. Thus, enhancing the metal solubility by lowering the pH upon Al^{3+} hydrolysis. The total heavy metal concentrations were quantified as the

Table 1. Sample composition and characteristics.

Properties		P1	P2	P3	4 (control sample)
pH (H_2O)		3.39	3.20	1.38	8.72
pH (1M KCl)		3.42	3.20	1.29	7.58
Total heavy metal content	(g kg^{-1})	2.13	2.34	1.73	0.18
Fe	(g kg^{-1})	401 ± 1	426 ± 1	43 6± 1	34 ± 0.4
Mn		0.7 ± 0.1	0.8 ± 0.1	0.6 ± 0.1	0.8 ± 0.1
CEC (cmol kg^{-1})		4.18	2.20	15.9	23.2
Na^+	(mg kg^{-1})	69 ± 1	81 ± 1	82 ± 1	83 ± 1
K^+		17 ± 0.4	19 ± 0.4	234 ± 1	201 ± 1
Ca^{2+}		702 ± 1	208 ± 1	2769 ± 12	4001 ± 11
Mg^{2+}		44 ± 0.6	53 ± 0.7	18 ± 0.2	276 ± 1
Al^{3+}		14 ± 0.3	59 ± 0.3	97 ± 0.3	0.5 ± 0.1

cumulative content of Cu, Pb, Zn, Cr, Mn, Cd, Ag, and Ni following the aqua regia digestion procedure and ICP-OES measurement. The following order was observed: P2 (0.23 %) > P1 (0.21 %) > P3 (0.17 %) > P4 (0.02 %). All three samples showed heavy metal levels significantly exceeding limit values of Cd (3 mg kg^{-1}), Cu (140 mg kg^{-1}), Ni (75 mg kg^{-1}), Pb (300 mg kg^{-1}), and Zn (300 mg kg^{-1}), according to Council Directive (86/278/EEC) (as amended) classifying the respective sites as contaminated. In contrast, the Bulgarian control soil sample (P4) demonstrated the lowest total heavy metal content. The provided limits are for agricultural soils and are not directly applicable to the strongly acidic soils (pH 1.38 - 3.39), as pH significantly increases the bioavailable concentration of these heavy metals. Higher acidity mobilizes metals, meaning they become more soluble and thus more toxic and available for uptake by plants [13, 14], even at concentrations below the Directive's thresholds.

The heavy metal content in the studied samples, obtained after acid digestion and ICP-OES measurement, is presented in Table 2. Notably, sample P4 was the only sample to contain detectable levels of chromium (12 mg kg^{-1}) and showed the highest manganese concentration among all studied samples.

Fractional distribution of heavy metals in soil samples

The applied procedure enabled detailed characterization of heavy metal partitioning across various geochemical soil fractions [15]. This distribution contributed to understanding both metal bioavailability and associated toxicity. The obtained data are presented in Fig. 1.

Copper (Cu) was predominantly concentrated in the residual fraction of all analysed soil samples, indicating low bioavailability. Specifically, Cu content in the residual fraction was estimated at 97 % (P1), 98 % (P2), and 69 % (P3). In contrast, only trace amounts of Cu were observed in the exchangeable and organic/manganese oxide-associated fractions - less than 1 % for samples P1 and P2, and 13 % in P3.

The amorphous iron oxides (fraction 3) retained minor copper concentrations - 2 % (P1), 1 % (P2), and 5 % (P3), indicating a significant affinity between copper and iron oxides. This observation aligns with previous findings [16].

Lead (Pb) was the second most abundant heavy metal in the analysed samples, with the highest concentrations detected in the residue dump material (Fig. 1; sample P3). Sequential extraction data revealed that 26 % of the total Pb content was associated with amorphous iron oxides during the third extraction step. In contrast, only 2 % of lead was recovered from the fraction targeting crystalline iron oxides. Collectively, this indicates that 28 % of the total lead was bound to iron oxide components. An additional 6 % of Pb was distributed between the exchangeable fraction and the manganese oxide-bound fraction (F1+ F2), highlighting limited mobility under certain conditions. The residual fraction

Table 2. Heavy metal content in samples P1- P4.

Heavy metal Sample	As	Cu	Pb	Zn	Cd	Ag	Mn	Ni	Cr
	mg kg^{-1}								
P1	748 ± 1.1	1322 ± 3	1267 ± 3	803 ± 1	8 ± 0.2	< 0.5	688 ± 3	< 1	< 0.5
P2	848 ± 1.1	1412 ± 3	1552 ± 3	817 ± 1	52 ± 0.2	53 ± 0.04	757 ± 3	< 1	< 0.5
P3	935 ± 1.1	1222 ± 3	4093 ± 3	498 ± 1	5 ± 0.2	< 0.5	633 ± 3	< 1	< 0.5
P4	2 ± 0.3	60 ± 0.3	57 ± 0.4	105 ± 1	< 0.5	< 0.5	782 ± 3	< 1	12 ± 0.2

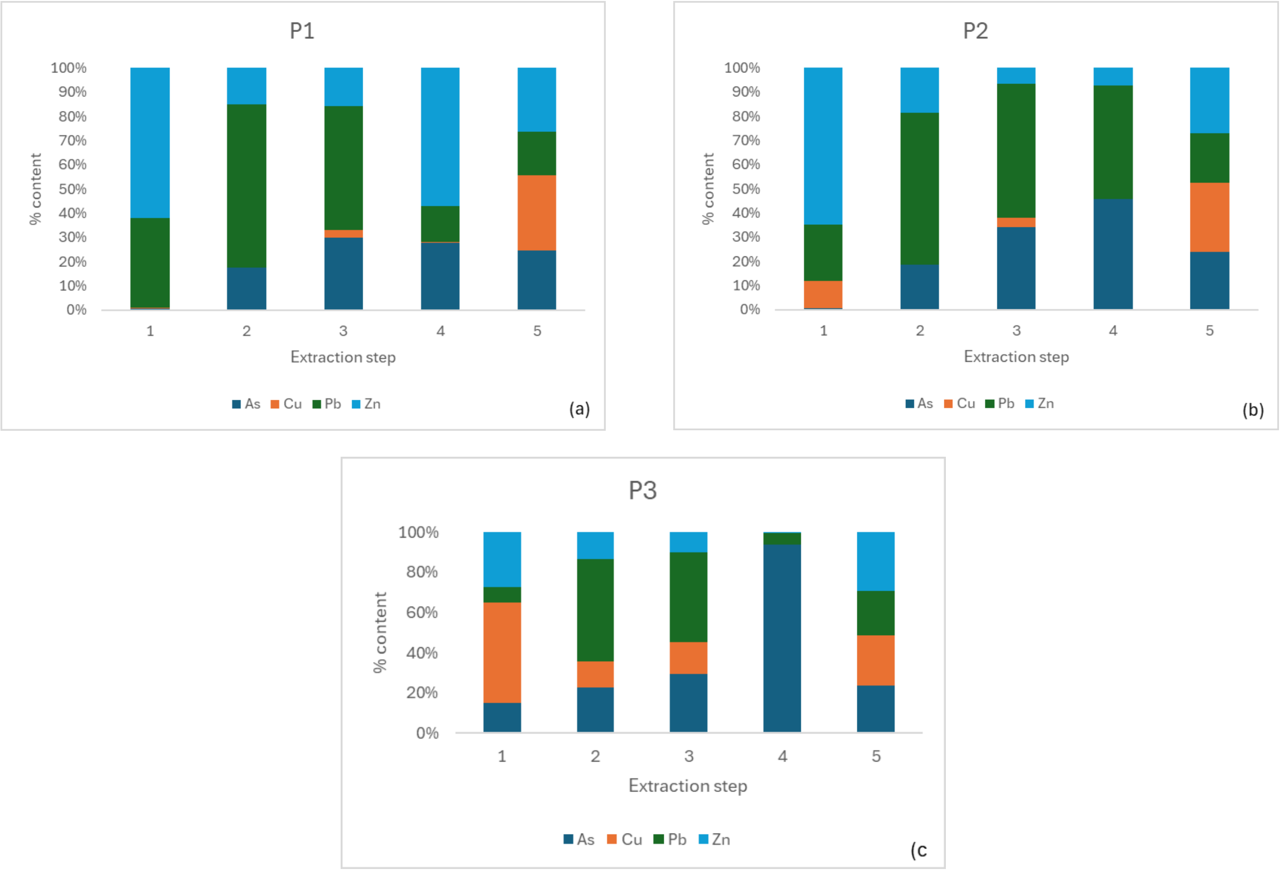

Fig. 1. Percentage fractionation of As, Cu, Pb, and Zn in the five-step sequential extraction procedure in Samples P1 (a), P2 (b) and P3 (c).

accounted for the majority of Pb content, with 67 % of total lead sequestered in forms considered relatively stable and less bioavailable.

All analyzed samples contained elevated concentrations of zinc (Zn), ranging from 456 to 884 mg kg^{-1}. The sequential extraction procedure revealed that the highest quantity of zinc was recovered during step 3, corresponding to the amorphous iron oxide-bound fraction. Specifically, 9 % of the total Zn in sample P1, 2 % in sample P2, and 3 % in sample P3 were associated with this phase. These results suggest that Zn tends to preferentially bind with amorphous Fe oxides over other geochemical fractions. Sample P3 is an exception that could be related to the low pH = 1.29 of the soil [17]. Although the other two are also considered acidic, they follow the established trend of 10 % in the third extraction step reported by several research groups [18 - 20].

Arsenic (As) was detected in all examined soil samples, with concentrations in descending order as follows: P2 > P3 > P1 (Fig. 1). Sequential extraction revealed that only a small fraction of arsenic (approximately 2 %) was recovered during the initial two stages in samples P1 and P2, indicating limited availability in exchangeable and easily reducible phases. Nevertheless, total arsenic concentrations exceeded the threshold value of 40 mg kg^{-1} dry soil [16], classifying all samples as significantly contaminated.

A substantial portion of exchangeable heavy metals was associated with amorphous iron oxides, with the majority partitioned into the residual fraction. The fractionation pattern showed both the potential mobility and bioavailability of these contaminants under favourable environmental conditions.

Environmental indices

Mobility factors calculated from sequential extraction data according to Zhao et al. are presented in Table 3 [12].

The values of mobility factors allowed the classification of the elements according to their mobility and potential for leaching in the environment under certain conditions, as follows:

Copper and zinc were found to have a high potential for mobility in sample P3 tailing material. A concentration above the threshold of 10 % was observed in the $MgNO_3$ fraction (F1) [12]. Fraction 1 presented the exchangeable fraction of heavy metals along with the metal ions in soil solution that could be easily released in the environment [5]. The samples P1 and P2 showed low values of mobility factors of Cu and Zn (Table 3) that indicated the fixation of Cu and Zn in the sample matrix, even though elevated total concentrations were found (Table 2).

Pb and As showed values of MF above 10 % in the fraction F3 in all the studied samples. Fraction 3 (0.1 M $NH_2OH.HCl$ + 0.25 M HCl) presented the heavy metals adsorbed on amorphous Fe oxides. According to Arenas-Lago et al. [6], in the presence of copper and low pH, the typically stable Fe and Mn oxides could release heavy metals into the environment [5, 22]. However, it should be noted that, in contrast to Pb, As was found to be the most mobile in samples P1 and P2, although their total concentrations were lower than that in the tailing sample P3.

The same pattern of mobility of studied heavy metals in different matrices of the soils and tailing material samples indicated that the surrounding area of the tailing dump is possibly contaminated by the tailing dust during windy periods.

Table 4 presents the individual and global contamination index obtained from sequential extraction data. The results showed low contamination potential of all studied samples (ICF < 1 and GCF < 6) according to

Table 3. Mobility factors for heavy metals in soil and mine tailing material based on sequential extraction data.

Mobility Factors, %					
Sample	Fraction	As	Cu	Pb	Zn
P1	F1	0.0	0.0	1.7	2.9
	F2	2.8	0.0	10.9	2.4
	F3	17.6	1.7	29.9	9.2
	F4	0.8	0.0	0.4	1.6
P2	F1	0.0	0.4	0.9	2.4
	F2	1.5	0.0	4.5	1.5
	F3	12.0	1.3	19.2	2.4
	F4	4.6	0.0	4.8	0.7
P3	F1	5.6	20.1	3.8	11.0
	F2	8.6	5.5	2.6	5.5
	F3	8.4	5.0	17.1	3.2
	F4	18.2	0.0	1.5	0.0

Table 4. Individual contamination index (ICF) and global contamination index (GCF) of studied heavy metals in soils and mine tailing material.

Sample	ICF				GCF
	As	Cu	Pb	Zn	
P1	0.27	0.02	0.75	0.19	1.23
P2	0.22	0.02	0.42	0.08	0.74
P3	0.69	0.44	0.34	0.25	1.71

the classification proposed by Necula et al. and Matabane et al. [10, 11]. The obtained low contamination index supported the findings of Drochiou et al. of the viability of endemic species in the area near the tailing dump [23]. Although the high total content of heavy metals in mine tailing, the ICF and GCF had low values. Limited mobility and contamination potential of tailing material could be explained partially by the solidification of mine tailings in the post-mining period.

CONCLUSIONS

The results in this study contribute to the efforts to increase the level of reliability of environmental footprint estimation. The applied methodology included a five-step sequential extraction procedure and assessing the environmental contamination potential via mobility factors and contamination indices. It should be noted that the chemically based methodology, although allowing modelling of different environmental conditions throughout the sequential extraction procedure, still considers only chemical and physical factors. To completely assess the environmental footprint, the methodology should be expanded by applying bioindicators and following the accumulation of contaminants. The proposed algorithm could be a decision-support tool for environmental monitoring, remediation planning, and sustainable land management.

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Authors' contributions

L.A.: Investigation, Methodology, Writing Original Draft, Review, Editing, Data curation, Project administration; D.I.: Conceptualization, Methodology, Investigation, Data curation; A. S.: Conceptualization, Methodology, Editing.

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