

## BIMODAL DISTRIBUTION OF POTENTIALLY TOXIC ELEMENTS IN URBAN SOILS: THE IMPACT OF PARTICLE SIZE

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*Abstract.* The aim of the study was to ascertain the distribution of potentially toxic elements (PTEs) across different soil particle sizes, particularly on Fluvisol soil type in Novi Sad, Serbia, characterised by a significant proportion of sand as coarser particles. PTEs, such as As, Co, Cr, Cu, Mn, Ni, and Pb were analysed in urban soil near major roads. Bulk samples of urban soil were collected and subsequently subdivided into seven different particle size fractions using the wet sieving technique. Based on PTE concentrations and PTE mass loadings ( $\mu\text{g kg}^{-1}$ ), the research confirmed a bimodal binding of PTEs according to soil particle sizes. The highest contribution was observed in the finest fractions ( $< 50 \mu\text{m}$ ), followed by larger fractions ( $> 500 \mu\text{m}$ ), while medium-sized particles ( $50 - 500 \mu\text{m}$ ) displayed the lowest contribution. Due to the predominant presence of sand in urban soils, it is important to include coarser fractions in risk assessments.

*Key words:* inorganic contaminants, soil fractionation, soil texture, urban environment.

### 1. INTRODUCTION

Potentially toxic elements PTEs (previously referred to as heavy metals), especially those of anthropogenic origin, tend to accumulate in fine urban soil fractions, posing health risks to humans, primarily through the inhalation of resuspended soil particles, ingestion via a hand-to-mouth path, or skin contact. The content of PTEs in specific soil fractions of urban soil or road sediments has been investigated by numerous authors [1,2,3]. The majority of studies have concluded that PTEs tend to bind more to finer particles in the soil (such as clay minerals) because finer particles have a larger surface area per unit mass and a negative charge on their surface [4,5]. Recent studies on potentially toxic elements (PTEs)

in urban soils have primarily focused on calculating contamination factors (CF) and geo-accumulation indexes (I<sub>geo</sub>), analysing their spatial distribution within cities, distinguishing between geogenic and anthropogenic sources of PTEs using PCA statistical tools, and the assessment of various human health risks [6,7,8]. Some studies evaluate the health risks associated with potentially toxic elements (PTEs) found in plants cultivated in urban areas [9,10,11]. Li *et al.* [12] emphasise that the highest bioaccessibility of PTEs is not consistently found in the finest size fraction due to substantial variations in pollutant distribution across different soil types. Kovač *et al.* [13] highlight that the sorption of PTEs is influenced more by soil physicochemical characteristics at different soil depths than by soil type *per se*. Additionally, different elements may demonstrate diverse relationships between bioaccessibility and soil particles [12]. Regarding texture, urban soils generally contain a higher proportion of coarser fractions [14] for two reasons. Significant towns developed over time near major rivers and coastal zones. Such areas originating from fluvial, beach and offshore deposits, are characterized by a higher sand content as their natural parent material [15,7]. Another contributing factor could be the incorporation of artificially made materials such as concrete and building bricks into urban soils [14].

The overview of the textural composition of major cities worldwide based on the literature is shown in Table 1. Urban soil, with its porous structure and limited absorption capacity, cannot efficiently purify water by filtering and absorbing pollutants from surface runoff, posing a potential risk for groundwater contamination. Compaction drastically decreases infiltration rates in sandy soils [16]. Physical degradation, including soil compaction and structure destruction, diminishes the soil's capability to store and infiltrate water, resulting in amplified runoff and heightened pollutant loads in nearby water bodies [14]. Ružičić *et al.* [17] emphasize the importance of researching the percolation of PTEs through soil and unsaturated zones, as well as soil properties that can influence the retention/mobilization of PTEs in alluvial soil.

Despite the proven affinity of PTEs to bind with the finest particles, it is noteworthy that the coarser sediment fractions exhibit a certain environmental risk due to their significantly greater mass and presence in urban soils. Previous investigations predominantly concentrated on evaluating the risk assessment of PTEs in the finer particle-size fraction of urban soil from the human health perspective. However, it is crucial to consider the larger particles as well, as PTEs bound to them end up in the urban environment and act as diffusive pollutants from the perspective of urban environment quality. Based on prior findings of elevated PTE concentrations in Novi Sad city, Serbia, in the vicinity of busy roads [18,15], this study aims to understand the distribution of PTEs in urban soil across different particle sizes, particularly in Fluvisol soil type, characterized by a significant

proportion of coarser sandy particles.

Table 1

Mechanical composition of the surface layer of soil in major cities worldwide

City	Country	Sand (%)	Silt (%)	Clay (%)	Texture class	
Bangkok	The Kingdom of Thailand	48.8±27.2	25.5±12.6	25.4±17.0	-	[19]
Belgrade	Republic of Serbia	20.0 – 53.3	20.3 – 49.9	26.4 – 30.0	-	[11]
Hong Kong Island	Special Administrative Region of the People's Republic of China	74.0	18.7	7.2	Loamy sand or sandy loam	[20]
Ljubljana	Republic of Slovenia	36.0±13.0	47.2±10.0	16.8±5.2	-	[21]
Łódź	Republic of Poland	n.a.*	n.a.	n.a.	Sand was a dominant textural class	[22]
Naples (central district)	The Italian Republic	71.1±3.79	22.8±1.14	6.1±0.24	-	[23]
Novi Sad	Republic of Serbia	76.3±9.0	15.3±5.1	8.4±4.4	70 % of samples are classified as sandy loam	[15]
Salzburg	Republic of Austria	43.8 – 58.6	34.8 – 48.3	6.6 – 7.9	-	[11]
Seville	Kingdom of Spain	35.3±15.4	38.9±11.8	25.8±9.4	-	[21]
Thessaloniki	Hellenic Republic	62.2 – 63.1	25.6 – 27.9	9.9 – 11.3	-	[11]
Torino	The Italian Republic	71.4±10.6	18.5±7.9	10.2±3.5	-	[21]
Volos seaside port	Hellenic Republic	57±6.7	19±7.5	n.a.	Sandy loam to clayey	[7]
Zagreb and its surroundings, Fluvisols	Republic of Croatia	25.85±17.25	60.94±16.35	13.22±7.24	-	[24]

## 2. MATERIALS AND METHODS

### 2.1. STUDY AREA AND SOIL SAMPLING

This study was conducted on four locations within the urban zone of Novi Sad, the second largest city in Serbia, located (45° 15' N; 19° 50' E). The chosen locations exhibited heightened levels of lead based on prior monitoring [18,15]. Elevated lead content at locations 1, 3, and 4 is associated with traffic, whereas at location 2, it stems from the activities of a small lead-acid battery plant. The area studied in Novi Sad is entirely covered by Holocene alluvial deposits formed by the fluvial activity of the Danube River. The autochthonous (indigenous) soil type in the city is classified as Fluvisol according to the soil map [25] and WRB classification [26]. Composite samples, comprising an average of 10 to 15 individual samples at a 0-10 cm depth, were collected using a soil probe over an area of approximately 50 m<sup>2</sup>. The research was conducted on four bulk soil samples, divided into seven different particle size fractions, making a total of 32 analysed samples (4 bulk + 28 fractions).

### 2.2. SOIL TEXTURE AND BASIC SOIL PROPERTIES

Particle size distribution was determined in the < 2 mm fraction by the pipette method [27]. The size fractions were defined as clay (< 2 µm), silt (2-20 µm), fine sand (20-200 µm) and coarse sand (200-2000 µm). The pH value in 1:2.5 (v/v) soil suspension in 1 M KCl was determined by potentiometric method [28]. The free CaCO<sub>3</sub> content was determined by the volumetric method [29]. Organic matter content was measured using the Tyurin method [30]. Total organic carbon (TOC) was determined by dry combustion [31].

### 2.3. SOIL FRACTIONATION BY WET SIEVING

The separation of soil particles into seven fractions was performed using the wet sieving method [32,33]. The initial stage involved separating water-resistant structural soil aggregates by immersing 100 g soil samples in deionised water. Subsequently, the samples were sieved through a series of mesh sizes:

1000 µm, 500 µm, 250 µm, 125 µm, 50 µm and 25 µm.

The number of sieve immersions in deionised water gradually decreased with the reduction in fraction size, ranging from 25 to 15. Specific particle sizes (coarseness intervals) were presented as a percentage of the total mass of aggregates.

#### 2.4. LASER-BASED DETERMINATION OF PARTICLE SIZE DISTRIBUTION

Particle size distributions of soil samples were also determined by the laser diffraction method using a Malvern Mastersizer 2000 particle size. Mie light scattering theory, integrated into the instrument's analytical procedure, assumes particles spherical shape. In consequence, the results obtained for particle size are equivalent to sphere diameters.

#### 2.5. DETERMINATION OF PTE CONCENTRATIONS

Element analysis was conducted in four bulk samples and in each of their seven fractions, resulting in a total of 32 samples. The total concentrations of As, Co, Cr, Cu, Mn, Ni, Pb and Zn were analysed after microwave digestion of soil samples in concentrated HNO<sub>3</sub> and H<sub>2</sub>O<sub>2</sub> (5 HNO<sub>3</sub>:1 H<sub>2</sub>O<sub>2</sub>, and 1:50 solid:solution ratio) by stepwise heating up to 180°C using a Milestone ETHOS1 for 55 min. The concentration of the elements in prepared samples was determined by ICP-OES (Vista Pro-Axial, Varian) by the U.S. EPA Method 200.7:2001 [34]. Quality assurance and quality control (QA/QC) assessments were conducted using the certified reference materials IRMM ERM-CC141 (Loam soil) and BCR 142R (Light sandy soil). The obtained limit of detection (LOD) ranged from 0.01 (Ni) to 0.05 (Zn) mg L<sup>-1</sup>, while the method detection limit (MDL) was 1.0 mg kg<sup>-1</sup> for all elements, providing adequate sensitivity for the analysis. In this study, the percentage of recovery ranged from 91 to 117 %, which provided adequate analytical accuracy and precision.

#### 2.6. PTE MASS LOADINGS

The mass loading of elements across various particle size fractions is an important contamination index [3]. Loading combines PTE concentrations, on a grain size basis, with data on the mass distribution. Then, this index reveals the percentage of PTE present in each fraction. PTE loadings on a grain size fraction basis ( $GSF_{Load}$ ) are calculated according to the following equation [35]:

$$GSF_{Load} = 100 \cdot \left( \frac{X_i \cdot GS_i}{\sum_{i=1}^7 X_i \cdot GS_i} \right) \quad (1)$$

where  $X_i$  is the element concentration in a determined grain size fraction in mg kg<sup>-1</sup>, with seven classes per sample, and  $GS_i$  - is the mass percentage of that fraction in the sample, which has limits of 0 - 100 %. The summation of  $GSF_{Load}$  values for an individual sample is 100 %.

### 3. RESULTS AND DISCUSSION

#### 3.1. CHEMICAL COMPOSITION AND SOIL TEXTURE OF BULK SOIL SAMPLES

The pH values detected within the unfractionated bulk samples depict a limited variance, ranging from 7.29 to 7.66, characterising the soils as slightly alkaline (Table 2), which is common for urban soils [14,7]. The organic matter content consistently trends among three samples (ranging from 3.2 to 3.6 % - classified as moderately humic soils), while one sample exhibited less than 3 % (classified as slightly humic soil). The carbonate content varied widely, ranging from 3.8 % to 12.7 %. Sample 1 is categorised as slightly carbonate, whereas the remaining three samples are classified as highly carbonate soils (Table 2). The described basic chemical properties of the soil generally result in a reduced availability of PTEs for soil biota - mainly higher plant, due to the alkaline reaction, higher carbonate content, and increased organic matter content. However, human hand-to-mouth bioaccessibility may not adhere to the same principles as the bioavailability of PTEs to plants. Li *et al.* [12] emphasise that certain studies indicate that elevated pH levels affect lower bioavailability of PTEs, while research by Liu *et al.* [36] demonstrates that an increase in soil organic matter content could potentially elevate Pb bioaccessibility, attributed to the existence of Pb-humic complexes in gastrointestinal fluids.

Due to limited quantities of certain fractions after sieving, basic chemical properties and total organic carbon (TOC) were assessed only in the first two samples per fraction. pH generally decreases with larger particle sizes, correlating with carbonate content in sample 2, while organic matter is more prevalent in larger particles in sample 1.

*Table 2*

Chemical properties and soil texture of bulk samples

Sample	pH (KCl)	CaCO <sub>3</sub> (%)	OM (%)	Clay < 2 (µm)	Silt 2 - 20 (µm)	Fine sand 20 - 200 (µm)	Coarse sand 200 - 2000 (µm)
1	7.29	3.80	3.58	16.5	21.8	51.1	10.5
2	7.61	8.45	2.72	9.1	13.2	44.7	33.1
3	7.39	6.33	3.23	10.2	14.7	48.0	27.1
4	7.66	12.67	3.30	3.6	8.3	42.6	45.5
Mean	7.49	7.81	3.21	9.85	14.50	46.6	29.05
± SD	±0.18	±3.75	±0.36	±5.29	±5.58	±3.73	±14.55

OM - Organic matter

Based on the soil texture of the initial bulk samples, it is evident that they primarily consist of elevated levels of fine sand followed by coarse sand (Table 2), while the sum of these two large fractions amounts to as much as 62 to 88 % of sample composition. This aligns to the characteristics of the Fluvisol soil type located in the observed area [25,26,37].

### 3.2. PTE CONTENTS IN BULK SOIL SAMPLES

Based on the total content of PTEs in bulk samples (Table 3), it has been shown that they exceed the background value for unpolluted agricultural soils analysed in the investigated region [38], as is commonly observed in urban soils worldwide. Regarding the prescribed limit values (LV), exceedances have been identified for the Cu, and Zn concentrations, and slightly for the content of Co but these elements remain below the remediation values. Pb concentrations are notably higher than limit values for samples 3 and 4 (230.1 mg kg<sup>-1</sup> and 318.7 mg kg<sup>-1</sup>, respectively). Such significant pollution is attributed to long-term contamination from the past use of lead as a fuel additive [15]. The remediation value exceeded for Pb (999 mg kg<sup>-1</sup>) at location in the proximity of a small lead-acid battery plant where sample 2 was taken.

Table 3

PTE concentrations (mg kg<sup>-1</sup>) in bulk samples

Sample	As	Co	Cr	Cu	Mn	Ni	Pb	Zn
(mg kg <sup>-1</sup> )								
1	9.5	<b>11.2</b>	39.6	25.3	531.4	33.2	<b>85.5</b>	94.7
2	5.8	6.2	28.1	<b>40.0</b>	417.7	32.4	<b>999.1</b>	125.2
3	6.9	7.9	35.5	31.3	414.6	29.5	<b>230.1</b>	134.1
4	6.1	6.1	44.8	<b>86.7</b>	328.6	34.2	<b>318.7</b>	<b>162.7</b>
Mean	7.1	7.8	37.0	<b>45.9</b>	423.1	32.3	<b>408.4</b>	129.2
± SD	±1.7	±2.4	±7.0	±27.9	±83.2	±2.0	±405.4	±28.0
<sup>a</sup> BV			30.0	17.1		14.8	17.2	60.3
<sup>b</sup> LV	29.0	9.0	100.0	36.0	/	35.0	85.0	140.0
<sup>c</sup> RV	55.0	240.0	380.0	190.0	/	210.0	530.0	720.0

<sup>a</sup> BV - Background value, element concentrations for unpolluted agricultural soils analysed in the investigated region [38]

<sup>b</sup> LV - Limit value, exceeding requires additional research and monitoring [39] (OG 30/2018 and OG 64/2019)

<sup>c</sup> RV - Remediation value, exceedingly more than 25 m<sup>3</sup> of soil volume requires remediation to be conducted (OG 30/2018 and OG 64/2019) [39.]

### 3.3. SOIL PARTICLE MASS AND SIZE DISTRIBUTIONS

Figure 1 shows cumulative undersize PSD curves. The percentage of particles smaller than 100  $\mu\text{m}$  is similar in all samples. A clear difference between sample 1 and other investigated samples (2, 3, 4) is evident for particles with equivalent sphere diameters above 100  $\mu\text{m}$ . The mass percentage of different fractions obtained by wet sieving of bulk samples (1-4) is given in Table 4. The results revealed that particle mass distribution among the seven grain size fractions is similar for samples 2, 3 and 4. The highest mass fraction was obtained for the fractions of 50-125, 125-250, and 250-500  $\mu\text{m}$ , which account for 65 – 75 % of the total sample mass. These results are in accordance with previous research of Luo *et al.* [20], and Ljung *et al.* [4], where the majority of the total mass is composed of the fractions within 50-500  $\mu\text{m}$ . The lowest mass fraction for these samples was recorded for the two finest (< 25; 25-125  $\mu\text{m}$ ) and two coarsest (500-1000; >1000  $\mu\text{m}$ ) fractions.

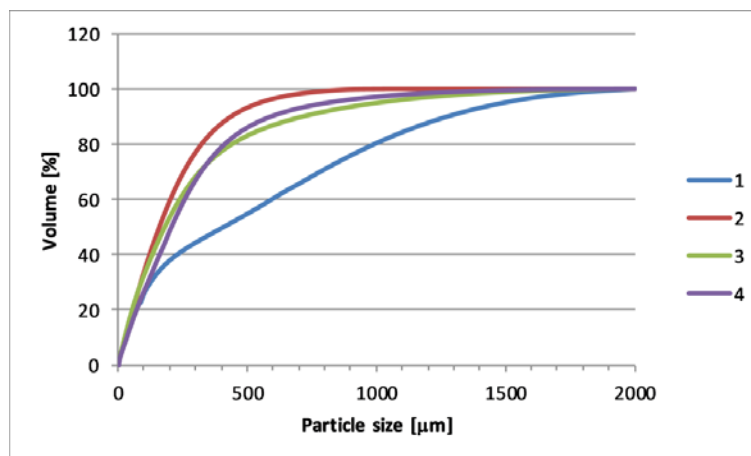


Fig. 1 - Cumulative undersize PSD curves

In the case of sample 1, the particle size distribution differs from the other three samples, as the fraction < 25  $\mu\text{m}$  has a particular percentage, amounting to 15 %. The highest value was recorded for the fraction of 50 - 125  $\mu\text{m}$  (approximately 20 %), while the mass percentage of the remaining fractions are quite uniform, within the range of 10 – 15 %. By comparing the data in Table 2, it is evident that a higher clay content and a lower proportion of coarse sand characterise sample 1. The mass percentage of individual fractions in the samples was calculated regarding the volume-based PSD results. The calculation results showed a good agreement with the measured mass fractions for all samples, as can be seen in Figure 2 (for sample 2).



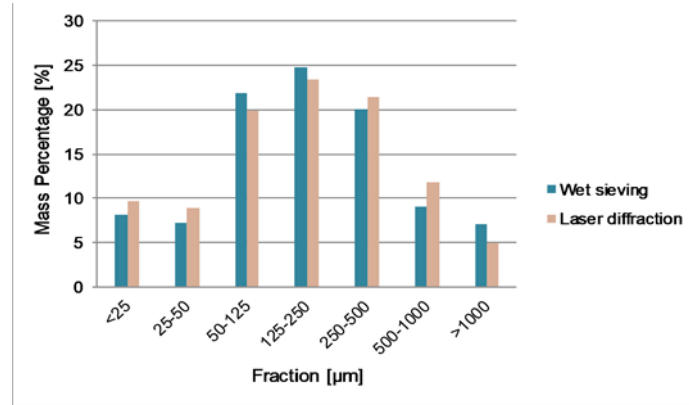


Fig. 2 - Mass percentage of fractions in sample 2

Table 4

Mass percentage of seven fractions in soil samples

Sample	Fractions (µm)	Mass percentage (%)	Sample	Fractions (µm)	Mass percentage (%)
1	< 25	14.60	3	< 25	8.70
	25-50	10.03		25-50	7.70
	50-125	20.69		50-125	21.80
	125-250	11.58		125-250	24.80
	250-500	12.42		250-500	20.00
	500-1000	12.93		500-1000	8.90
	>1000	16.24		>1000	6.70
Sample	Fractions (µm)	Mass percentage (%)	Sample	Fractions (µm)	Mass percentage (%)
2	< 25	11.52	4	< 25	5.41
	25-50	8.94		25-50	6.18
	50-125	15.83		50-125	20.24
	125-250	26.46		125-250	25.50
	250-500	24.96		250-500	30.20
	500-1000	7.36		500-1000	7.50
	>1000	4.05		>1000	3.81

#### 3.4. POTENTIALLY TOXIC ELEMENT CONCENTRATIONS IN PARTICLE SIZE FRACTIONS

The distribution of PTEs within different fractions is illustrated in Figure 3. In general, concentrations of elements in these fractions are higher than those in the bulk samples indicating the heterogeneous distribution of their concentrations across various soil fractions. Qin *et al.* [40] and Gong *et al.* [41] found that the element concentrations generally increased with the decrease of particle size. Luo *et al.* [20] reported higher concentrations of PTEs in urban soil fractions in Hong Kong, specifically within the clay fraction ( $< 2 \mu\text{m}$ ), fine silt (2-10  $\mu\text{m}$ ), and fine sand (50-100  $\mu\text{m}$ ), as opposed to the bulk samples. The study found that elements had a strong affinity for the finest soil fraction, with significantly greater concentrations of all measured PTEs in the  $<25 \mu\text{m}$  fraction. Concentrations of observed PTEs decrease with the increase in particle size up to fraction 125-250  $\mu\text{m}$ . The concentrations of PTEs rise to start from fraction 250-500  $\mu\text{m}$  but remain below the values determined for the finest fraction, with minor deviations (Figure 3).

Notably, a bimodal distribution is observed for all elements, with significant concentrations identified in larger fractions. In addition to the accretion of PTEs within the smallest fractions ( $< 50 \mu\text{m}$ ), an augmented concentration is notably evident within the coarse sand fraction ( $> 500 \mu\text{m}$ ). Certainly, in the soil fraction  $> 250 \mu\text{m}$  exists a noteworthy quantity of PTEs that deserves further attention from the perspective of risk to the urban environment. This outcome aligns with the literature and is explained by the increase in organic matter content within the larger particle fraction [42], as previously mentioned in section 3.1. This phenomenon can be attributed to the accumulation of PTEs in fine and coarse aggregates, influenced by various complex factors. These factors include the initial adsorption of new inputs of PTEs by the coarse aggregates, and the presence of microaggregates embedded within the coarse particles [43]. Similar findings of higher concentrations of PTEs in the largest soil fraction were reported by Martínez-Martínez *et al.* [44] based on soil investigations near a mine in the Murcia region, Spain. From an ecological perspective, it is crucial to emphasise that PTE concentrations determined in examined soil samples are minimal in fractions representing the highest mass proportion.

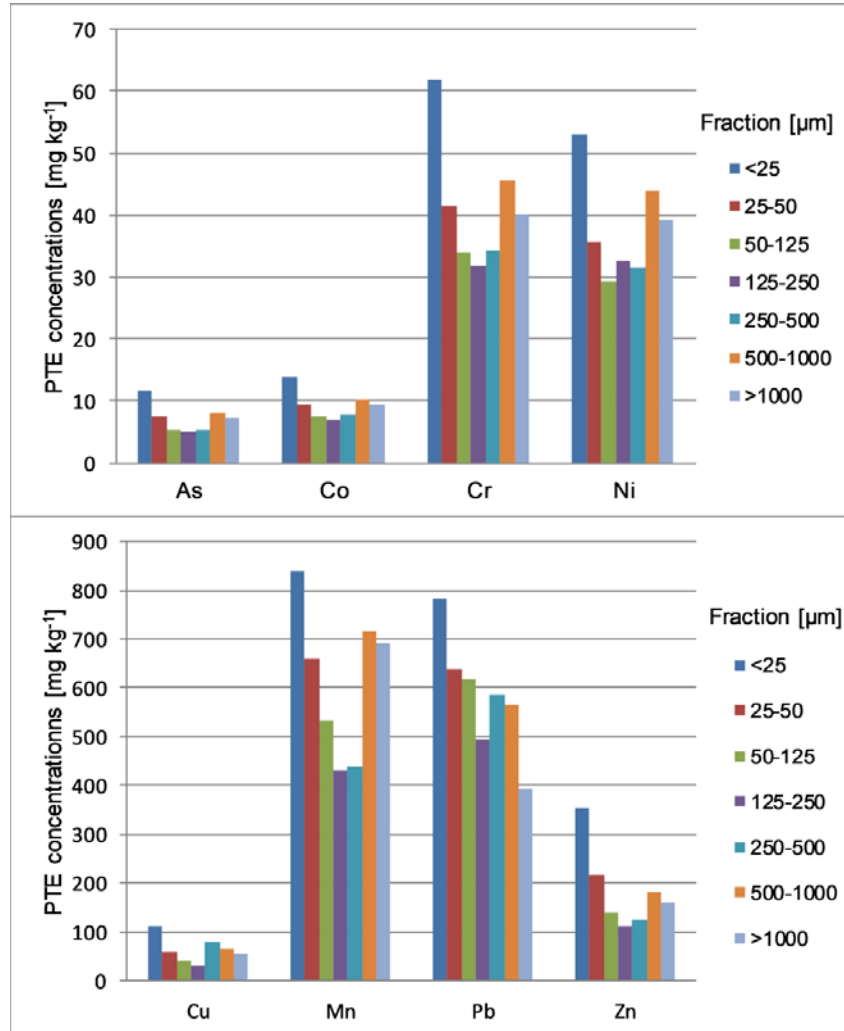


Fig. 3 - Mean values of total PTEs in soil fraction

### 3.5. INDICATORS OF ELEMENTS' PREFERENTIAL BINDING IN SOIL FRACTIONS

Based on the mass loadings ( $GSF_{Load}$ ) of potentially toxic elements, the level of PTE contamination in soil samples across various fractions was assessed. The mean values of  $GSF_{Load}$  are graphically represented in Figure 4. The calculated values of PTE loading ( $GSF_{Load}$ ) vary among individual fractions. When the PTE concentration in a specific fraction is significant and its mass percentage is low, the overall contribution of this fraction to the total element concentration in the “bulk”

sample will be relatively small. PTE loading is lowest for fractions 25-50, 500-1000, and > 1000  $\mu\text{m}$ , approximately 8 - 12 % for all elements, respectively, resulting in a combined contribution of these three fractions to the total element concentrations at around 30 %. The element contribution is uniform for fractions < 25, 50-125, and 125-250  $\mu\text{m}$ , ranging between 15-20 %. The highest  $GSF_{\text{Load}}$  for all elements is obtained for the fraction 250-500  $\mu\text{m}$  (17- 28 %). As previously mentioned, despite the high concentration values within the < 25  $\mu\text{m}$  fraction, contribution to the overall PTE concentrations in the bulk sample is not pronounced due to the small mass percentage of that fraction. Based on this part of the research, it can be concluded that the 25-500 fraction exhibits a significant mass percentage in the distribution of PTEs among fractions. In assessing environmental risks, attention should be paid not only to the finest particles < 25  $\mu\text{m}$  but also to coarser soil fractions.

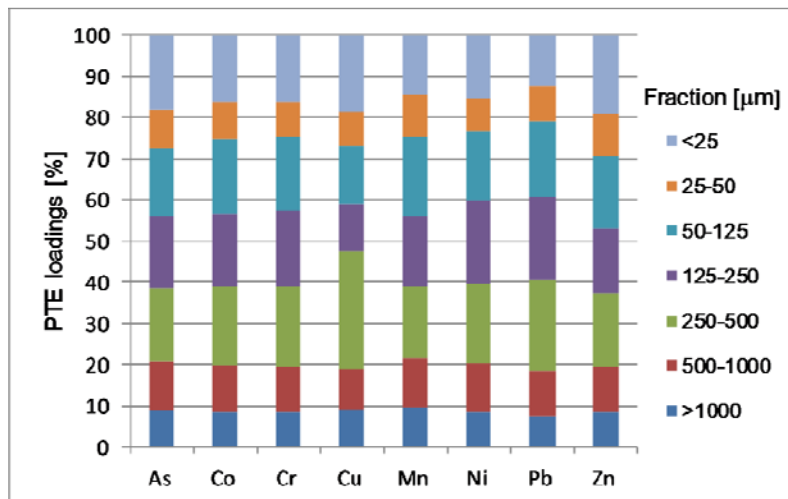


Fig. 4 - Mean PTE loadings ( $GSF_{\text{Load}}$ ) across various soil fractions

#### 4. CONCLUSION

The evaluated bulk soil samples showed characteristics of carbonate soils, featuring a slightly alkaline pH and substantial organic matter content, predominantly comprising fine and coarse sand fractions. The total Cu, Pb, and Zn concentrations in bulk samples exceeded the prescribed limit values. A sample near a small lead-acid battery manufacturer exhibited an exceptionally high Pb concentration.

Based on the analysis of PTE concentrations across varying soil particle sizes, the results confirm the presence of the highest concentration in the finest soil fraction < 25  $\mu\text{m}$ , significantly exceeding those observed in bulk soil samples. Notable, a bimodal distribution pattern is observed for all elements. In addition to the accumulation of PTEs within the smallest fractions (< 50  $\mu\text{m}$ ), a substantial concentration is also notable within the coarse sand fraction (> 500  $\mu\text{m}$ ). Furthermore, based on the calculated PTE loadings ( $GSF_{Load}$ ), the 25-500  $\mu\text{m}$  fraction emerges as a substantial contribution of PTEs among the various fractions. The findings of this study underscore a bimodal pattern of PTEs binding, predominantly within the finest particles and subsequently within the coarsest soil particles. This highlights the need to incorporate coarser particles into the risk assessment framework for urban environments. Moreover, a pressing need exists to conduct additional investigations into the fate and pathways of PTE loads linked to larger soil particles. Coarser particles pose a higher ecological risk due to their greater mass and the transfer of persistent PTEs across urban settings via diverse pathways, acting as diffuse pollutants. Therefore, it is essential to better understand the transport and destination of PTE loads to mitigate the environmental impact of these hazardous substances.

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