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# Rapid assessment of soil contamination by potentially toxic metals in the green spaces of Moscow megalopolis using the portable X-ray analyzer

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## Abstract

**Purpose** Anthropogenic influence leads to significant changes in soil properties and functions. Soil contamination by potentially toxic metals is one of the major environmental problems in urban environments. Traditional soil monitoring methods, while accurate, are often costly and labor-intensive, making it challenging to capture the intricate spatial variations of pollutants in urban soils. Proximal sensing based on X-ray fluorescence (XRF) analysis is considered a cost-effective approach for rapid assessment of soil contamination. The assessment accuracy depends on soil properties (e.g., texture, moisture, organic matter content) and detection limits for different elements. The research aimed to test a portable XRF analyzer for the assessment of soil contamination by potentially toxic metals in green zones of Moscow megalopolis.

**Materials and methods** Initially, Olympus Vanta C pXRF was calibrated using artificially contaminated soil mixtures by Ni, Cu, Pb, Zn, and Cd, representing a diversity of urban soils in Moscow. Linear regression was used to compare pXRF results with the ICP-OES method, and regression coefficients were used to set correction factors ( $k$ ) for observed potentially toxic metals based on soil properties. Subsequently, the spatial mapping accuracy of topsoil contamination in three distinct green areas was assessed using pXRF (with and without correction factors) based on ICP-OES reference concentrations.

**Results** The calibrated pXRF showed high accuracy for Pb ( $R^2=0.94$ ,  $b=0.91$ ,  $p<0.05$ ), Cu ( $R^2=0.95$ ,  $b=0.95$ ,  $p<0.05$ ), and Zn ( $R^2=0.95$ ,  $b=1.04$ ,  $p<0.05$ ), moderate accuracy for Ni ( $R^2=0.68$ ,  $b=0.77$ ,  $p<0.05$ ), and limited accuracy for Cd ( $p>0.05$ ) on a typical urban contamination level due to its high detection limit. Spatial variability in soil contamination was determined by comparison to the health thresholds (approximate permissible concentration and pollution indices), and the areas subjected to land-use restrictions were identified based on the regional environmental regulations. When calibrated by correction factors, mapping accuracy based on pXRF approached that of ICP-OES (in the range of 10%) for Ni, Cu, and Pb in major parts of the areas.

**Conclusion** The study revealed that uncorrected pXRF measurements overestimated contamination. When tailored to specific urban soil conditions, pXRF offers a viable, cost-efficient alternative for assessing soil contamination. The developed approach improved the accuracy and reliability of local soil contamination maps by capturing spatial patterns ignored by conventional methods which is essential to optimize costs of soil rehabilitation and sustainable management of urban soils.

**Keywords** Proximal sensing · Potentially toxic metals · Portable X-ray analyzer · Calibration factors · Land-use planning · Soil management

## 1 Introduction

Urbanization coincides with remarkable changes in soil properties and functions (Pickett et al. 2011; Yang and Zhang 2016). One of the most prevalent negative consequences is

contamination by potentially toxic metals (PTMs) (Madrid et al. 2006). The European Commission identifies PTM contamination as a significant threat to soil quality and health (EC 2006; EU 2022). High PTM contents can deplete soil functionality by suppressing microbial activity and limiting plant growth (Vasenev et al. 2013; McBride et al. 2014). In urban areas, the primary concern is the impact of contaminated soils on human health (Bitukova et al. 2000; Poggio et al. 2009). Potentially toxic metals are resistant to both

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chemical and biological degradation, remaining in the top-soil for extended periods after deposition. This persistence makes them a long-term source of health risks for citizens (Mielke 2015; Mielke et al. 2013; Paltseva et al. 2018). Therefore, monitoring and controlling PTM levels in soils is essential for health and environmental protection policies (Semenkov and Koroleva 2019, 2022; Vasenev et al. 2017).

The heterogeneity of urban soils and the variety of contamination sources, such as traffic, industries, and mining, result in high spatial variability of PTM contents. This variability necessitates numerous observation points for accurate contamination assessment (Lado et al. 2008; von Steiger et al. 1996; Zeng et al. 2021). Traditional monitoring approaches, which involve soil sampling from limited locations followed by PTM analysis using wet chemistry, are expensive and time-consuming. These methods capture only general patterns of PTM distribution within city boundaries, such as comparison between administrative districts or functional zones. However, the data collected is often limited in spatial and temporal scope, leading to unreliable assumptions across spatially variable landscapes (Romzaykina et al. 2021; Dietrich et al. 2023). Hence, there is a demand for smaller-scale, high-frequency measurements to characterize spatial variability in PTM contents within specific areas, aiding soil management and urban planning decisions (Levin and Paltseva 2023; Cherkashina and Pellinen 2021; Guo et al. 2020).

Proximal sensing offers a cost-efficient alternative for monitoring soil contamination. The X-ray fluorescence (XRF) method is favorable for mass analysis due to its ease of use and the ability to quickly obtain substantial data on chemical elements in various materials (Paltseva and Cheng 2019; Byers et al. 2019; Han et al. 2021; Martin and Doucette 2007). Despite its widespread use in soil and sediment analysis (Guo et al. 2020; Shokr et al. 2016; Washbourne et al. 2012), the method requires adjustment to mitigate the effects of humidity, inter-elemental interference, and soil sample heterogeneity in terms of texture and organic matter content (Piorek and Lopez-Avila 1998; Zhu et al. 2011). Calibration experiments and confirmatory analyses on a subset of samples, which represent regional soil conditions and a range of PTM contents, should be conducted using inductively coupled plasma atomic emission spectroscopy (ICP-OES) alongside portable XRF (pXRF). This approach is recommended to enhance the accuracy of proximal sensing (Radu and Diamond 2009; Riebe et al. 2019). As a result, spectral datasets and calibration coefficients specific to the region of interest are obtained, facilitating the further implementation of proximal sensing techniques (Martin and Doucette 2007; Tavares et al. 2022). Soil surveys in various locations have successfully utilized calibrated pXRF results for soil contamination assessment and mapping at different spatial scales. These studies also emphasize that calibration

approaches and correction factors should be tailored to the specific PTMs, contamination sources, and soil conditions of new research areas and sites (Bechet et al. 2018; Cheng et al. 2015; Dvornikov et al. 2022; Paltseva et al. 2022).

Moscow is among the largest megalopolises in Europe. Intensive traffic, former and currently operating industries result in considerable soil contamination by PTMs (Kosheleva et al. 2018; Romzaykina et al. 2021; Vlasov et al. 2021). The city government recognizes healthy soils as a vital goal in its environmental management strategy, actively working towards the remediation of contaminated sites (Kulbachevsky 2021). So far, monitoring soil contamination in Moscow is based on the conventional approach, which does not allow considering soil heterogeneity to explore variability in soil contamination. Moscow's soils are notably diverse, comprising a mix of semi-natural and man-altered Retisols and man-made Technosols constructed from a wide array of materials including peat, compost, sand, and dredged sediments (Brianskaia et al. 2020; Ivashchenko et al. 2021). This heterogeneity shall be considered to implement pXRF for the rapid soil contamination assessment that is especially relevant for green spaces, where health risks of PTMs in soil can be very high due to many direct and indirect exposure pathways, e.g., by contacting soil at the playgrounds, lying on the green lawns, or growing food at the community gardens (Massas et al. 2010; Rózański et al. 2018; Vasenev et al. 2017).

Our research aims to explore the applicability of pXRF proximal sensing for rapid contamination assessment in the heterogeneous soils of Moscow's green spaces. Initially, we adjusted the pXRF readings through lab experiments using artificially contaminated soils that represent the variability of soil properties in Moscow. Subsequently, we tested the performance of the adjusted pXRF in three green zones exposed to varying levels of contamination, comparing the results with those obtained through the reference ICP-OES technique. Finally, we analyzed the potential implementation of the assessment outcomes to support decisions in soil and environmental management.

## 2 Materials and methods

### 2.1 Research area and case sites

Located on the East European Plain (55° 45' N; 37° 37' E), Moscow megalopolis experiences a moderately continental climate with an average annual temperature of 5.8 °C and precipitation of 600 mm. The average altitude of the city's territory is approximately 180 m, and parent materials are dominated by glacial and alluvial deposits. Natural vegetation belongs to south taiga and mixed-forest zones; however, urban green spaces are dominated by the introduced species

(i.e., linden, poplar, maple, and chestnut). The city's natural zonal Retisols have largely been replaced by semi-natural or entirely artificial urban soils (Gerasimova et al. 2003; Prokof'eva et al. 2011, 2014). Historically an industrial and trade hub, Moscow has seen many of its industrial areas relocated and redeveloped in recent decades (Argenbriht 2013, 2018). Concurrently, vehicle numbers have surged, reaching approximately 4.5 million or 347 cars per 1000 residents (Vlasov et al. 2021; Kulbachevsky, 2019). This traffic together with remaining industries (e.g., metal-working and petrochemical plants at the Eastern district) and landfills at the city outskirts is the main sources of soil contamination by PTM (Kosheleva et al. 2015, 2018; Kulbachevsky 2019). The average level of urban soil contamination in the Moscow metropolitan area is moderate; however, the spatial distribution of soil PTM contents is highly variable depending on the district, functional zone, proximity to city center, main highways, or industrial areas (Romzaykina et al. 2021). As a rule, recreational areas and green spaces are less contaminated compared to neighboring residential or traffic zones; however, high variability in soil PTM contents can be expected between and within green spaces (Bol'shakov and Kakhnovich 2002; Ladoni and Mikhaylova 2020; Plekhanova 2000). To consider this variability, three green areas exposed to different contamination levels were selected as the case sites for the research: the urban forest in Moscow Timiryazev Agricultural Academy (low contamination), RUDN University campus (moderate contamination), and Repin's square in the city center (high contamination) (Fig. 1). Topsoil (0–10 cm) mixed samples were collected from 25 to 30 random points in each area (in total  $n = 83$ ) and delivered to the laboratory for further analysis and experiments.

The soils of the case sites exhibited significant variations in morphological and agrochemical properties, as well as in the anthropogenic stresses they were subjected to. The urban forest of Moscow Timiryazev Agricultural Academy was originally a natural protected area, afterwards open for the public to enhance recreational and educational services. The primary recreational activities reported in this area are sports and nature observation. Despite its offerings, the total number of visitors remains relatively low, with fewer than 5000 visitors per day (Matasov et al. 2023). Consequently, the area is dominated by Retisols, which have not undergone significant modifications due to greening or landscaping. The highway at the edge of the urban forest and the walking pathways inside it are the primary sources of anthropogenic disturbance. These disturbances lead to soil over-compaction and PTM contamination (Ivashchenko et al. 2019; Vasenev et al. 2020).

Both the RUDN University campus and Repin's square are dominated by constructed Technosols. These soils are formed by layering organic substrates, such as peat-sand

mixtures or compost, over technogenic subsoil including sand-gravel layers, dredge materials, or other urban sediments (Shchepeleva et al. 2017; Vasenev et al. 2021). The RUDN University campus primarily provides recreational and educational functions. The area sees a substantial influx of visitors, with numbers reaching up to 10,000 per day. This population primarily consists of students, university staff, and residents of the adjacent district, who use the area for both recreation and transit. The primary sources of PTMs in the soils of the campus are the highway roads along its boundaries and the internal roads and parking lots.

Repin's square, situated in the heart of Moscow, is a hub of activity, serving both recreational and transit purposes. The estimated number of visitors surpasses 20,000 daily. While the surrounding highways are the primary contributors to PTM contamination in the park, additional contamination can be attributed to the intensive management and maintenance of the green spaces (Matasov et al. 2020).

## 2.2 Design of the experiment of pXRF calibration using artificially contaminated soil mixtures to calculate correction factor (k)

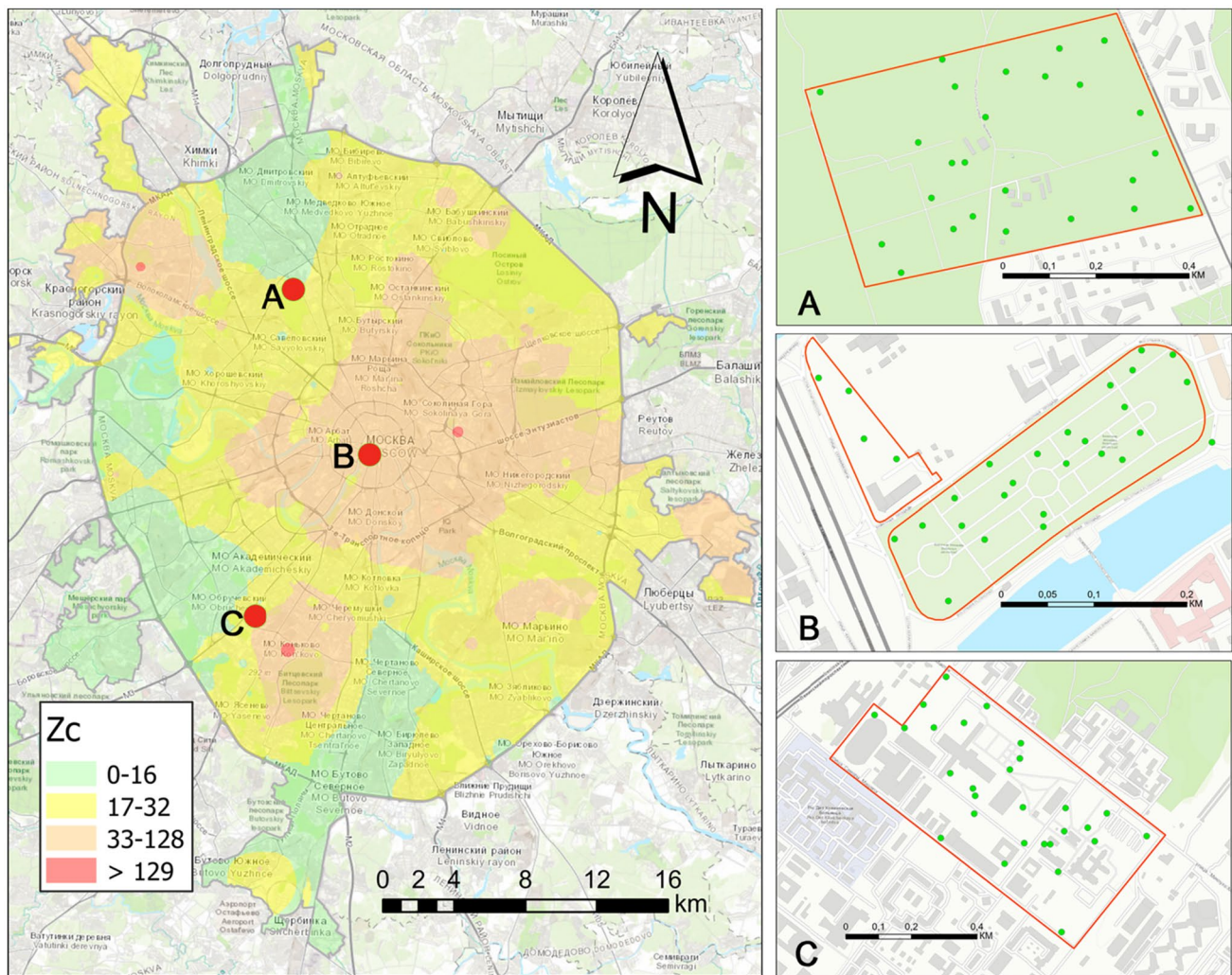
The selection of metals and their contents, as well as the selection of soil mixtures, was done based on a literature review examining the properties of natural and man-made soils in Moscow and other urban and industrial sites (Brianskaia et al. 2020; Ivashchenko et al. 2021; Semenov and Koroleva 2022; Slukovskaya et al. 2019; Tong et al. 2020).

Drawing from prior studies (Paltseva et al. 2022; Radu and Diamond 2009), we selected the following factors known to influence the accuracy of pXRF measurements and subjected them to experimental testing: (i) exposure time, (ii) metal content, and (iii) properties of the soil matrix. To evaluate the first two factors, a preliminary experiment was conducted using aqueous metal solutions (Cu, Zn, Pb, Ni, and Cd) with concentrations ranging from 5 to 200 mg L<sup>-1</sup>. The potentially toxic metal contents were measured at exposure times of 90, 120, and 180 s for the second beam. Notably, the durations of the first and third beams remained constant at 30 s each.

To assess the impact of the soil matrix, we selected three soil substrates with varying pH<sub>water</sub>, soil organic carbon content (SOC, %), and texture: sand (S), loam (L), and valley peat (P).

Subsequently, metal solutions were introduced to the air-dried substrates in volumes equivalent to the water volume at water-saturated capacity (WSC). After the addition of the solutions, the substrates were air-dried for 48 h and then analyzed by pXRF. The hygroscopic moisture was then determined (following ignition at 105 °C for 8 h) to adjust the PTM contents in the soil samples. Standard 1 g•L<sup>-1</sup> water solutions of metals (Cu, Ni, Pb, Zn, Cd) were used to prepare





**Fig. 1** Case sites illustrated on the soil contamination map of Moscow, delineated based on the total contamination index ( $Z_c$ ) as per Romzaykina (2021). The sites are as follows: (A) urban forest in Mos-

cow Timiryazev Agricultural Academy (low contamination level), **B** Repin's square in the city center (high contamination level), and (C) RUDN University campus (moderate contamination level)

soil samples with the concentrations 1, 2.5, 10, 25, 100, and 250  $\text{mg}\cdot\text{kg}^{-1}$ . For the preparation of soil samples with metal concentrations 250, 1000, 2500, and 10,000  $\text{mg}\cdot\text{kg}^{-1}$ , salts of nitrates Cu and Ni and 10  $\text{g}\cdot\text{L}^{-1}$  standard water solutions for Pb and Zn were used. A full description of the subsequent of soil samples' preparation is given in Supplementary A. Then, analyzed substrates were subsequently mixed to obtain the following soil mixtures: (i) sand to loam in a 1:1 ratio (SL), (ii) sand to loam to peat in a 1:1:1 ratio (SLP), (iii) sand to peat in a 1:3 ratio (S3P), and (iv) loam to peat in a 1:1 ratio (SP) (all proportions were by volume). These substrates were then re-analyzed using pXRF. For the field samples, PTM contents measured by pXRF were compared to the results of the analysis by ICP-OES.

The correction factor ( $k$ ) was calculated as the slope of the regression line derived from the laboratory experiment for soil mixtures comparing pXRF and ICP-OES values.

### 2.3 Soil analyses

All soil samples underwent grinding, homogenization, air-drying, and sieving through a 2-mm mesh sieve. The screening of PTM contents was conducted using an Olympus Vanta C pXRF analyzer (Olympus, USA). The portable XRF screenings were made in plastic cuvettes made by Olympus and covered with a special film. The "Soil mode" regime of pXRF after precalibration using Standard Olympus 2711A was used.

The ICP-OES analysis was made on PerkinElmer AVIO 200 for air-dried, homogenized, and 0.5-mm ground samples after microwave digestion in an acid mixture (3 mL HNO<sub>3</sub> (puriss. spec., Baum-Lux, Moscow, Russia) + 2 mL HF (puriss. spec., Baum-Lux, Moscow, Russia) + 1 mL HCl (puriss. spec., Baum-Lux, Moscow, Russia) + 5 mL H<sub>2</sub>O per 0.25 g of soil) following the standard M-MVI-80–2008 (2008) method. Multi-Element Calibration Standard 3 and Verification Standard GSO 2499–83 (SDPS-2) were used for QA/QC.

Soil organic carbon content was determined using the Elemental Vario TOC select (Operating instructions 2009); the pH of water extraction was assessed using the potentiometric method in a ratio of 1:5 for mineral soil and mixtures and 1:50 for peat (Vorobyova 1998), and soil texture was determined by a field feeling test (FAO 2006). The soil water-holding capacity (WHC) of these substrates was ascertained by gravimetric method (Schmugge et al. 1980).

## 2.4 Soil contamination assessment and mapping at the case sites

The potentially toxic metal contents of soil samples, collected from the specified research sites (refer to Section. 2.1), were determined using three distinct methods: (i) pXRF screenings, (ii) pXRF screenings adjusted by the estimated correction factors, and (iii) ICP-OES analysis. Soil contamination was assessed for each method based on Russian national standards: Approximate Permissible Concentration Value (APC, as per SR and N 1.2.3685–21 2021, Table 1) and Total Contamination Index (Zc, as outlined by the Ministry of Health of RF 2013); and internationally recognized pollution indices: Pollution Index (PI) and PI<sub>Nemerow</sub> (as described in Cheng et al. 2007; Qingjie et al. 2008; Weissmannová and Pavlovský 2017; Kowalska et al. 2018).

The Pollution Index (PI) for an individual PTM was calculated based on Eq. 1. Pollution levels of PI were determined to be low pollution ( $\leq 1$ ); moderate pollution (1–3), and heavy pollution ( $\geq 3$ ).

$$PI = \frac{C_i}{B} \quad (1)$$

where  $C_i$  is the actual content of the  $i$ th PTM in soil (mg kg<sup>-1</sup>), and  $B$  is the geochemical background contents of PTMs determined according to Kabata-Pendias (2011): Ni (29), Cu (38.9), Zn (70), Pb (27), Cd (0.41), As (0.67), and Mn (488).

The pollution index PI<sub>Nemerow</sub> was calculated based on Eq. 2. Pollution levels of PI<sub>Nemerow</sub> were determined to be clean ( $\leq 0.7$ ); threshold value (0.7–1); light pollution (1–2); moderate pollution (2–3), and heavy pollution ( $\geq 3$ ).

$$PI_{Nemerow} = \sqrt{\frac{(\frac{1}{n} \sum_{i=1}^n PI)^2 + PI_{max}^2}{n}} \quad (2)$$

where PI is the Pollution Index for an individual PTM, PI<sub>max</sub> is the PI value of that PTM, which is the maximum for each individual sample.

The Total Contamination Index (Zc) was calculated based on Eq. 3 with at least six PTMs considered:

$$Zc = \sum KCi - (n - 1) \quad (3)$$

where KCi is the content coefficient of the  $i$ th PTM in soils calculated as a ratio of a PTM content (mg kg<sup>-1</sup>) to the natural reference content (mg kg<sup>-1</sup>), and  $n$  is the number of PTMs. The natural reference contents for PTMs were established in accordance with the national standard CP 11–102-97 (1997), and these values varied based on soil properties. For Retisols with loamy sand and sandy texture, the background content (in mg kg<sup>-1</sup>) was as follows: Ni (6), Cu (8), Zn (28), Pb (6), Cd (0.05), and As (1.5). For Retisols with loamy and clay texture, the background content (in mg kg<sup>-1</sup>) was as follows: Ni (20), Cu (15), Zn (45), Pb (15), Cd (0.12), and As (12.2). Based on the calculated Zc values, soil contamination levels were categorized as follows: permissible level (< 16); moderately hazardous (16–32); highly hazardous (32–128); and extremely hazardous (> 128).

## 2.5 Data analysis and correction factor (k) calculation

Statistical analyses were conducted using R Studio. The normality of the distribution of PTM contents was assessed by the Shapiro–Wilk and Kolmogorov–Smirnov tests. To determine the significance of differences in contamination levels as estimated by pXRF screenings compared to ICP-OES measurements, a paired  $T$ -test was employed. The correlation between pXRF and ICP-OES values was analyzed through linear regression. The accuracy of pXRF was estimated by mean error (ME) and root square mean error (RMSE) based on the validation by ICP-OES measurements. Variations in soil contamination levels across the case sites as well as the effect of multiple factors (e.g., soil mixture type and method

**Table 1** Approximate permissible concentrations (APC) of heavy metals in soil, mg kg<sup>-1</sup>

Soil properties	Ni	Cu	Zn	Pb	Cd	As	Mn
Sand and loamy sand	20	33	55	32	0.5	2	1500
Loam and clay pH < 5.5	40	66	110	65	1.0	5	1500
Loam and clay pH > 5.5	80	132	220	130	2.0	10	1500

of analysis) on the estimated PTM contents were examined using one-way, two-way, and factorial ANOVA with post hoc Tukey's test to identify the homogeneous groups. Levene's test was used to check the equality of dispersions.

Spatial patterns in topsoil contents of Cu, Pb, Ni, and Zn for the three case sites were mapped using the Inverse Distance Weighting (IDW) interpolation approach with cross-validation in the ArcGIS Pro software. The resulting maps were compared regarding the areas exposed to different contamination levels and corresponding land-use restrictions. The three distinct methodologies used to assess PTM contents—ICP-OES, pXRF (without the correction factor), and pXRF  $\times$  k (with the correction factor)—yielded three separate maps for each PTM at every site. Subsequently, the pXRF and pXRF  $\times$  k maps were superimposed onto the ICP-OES map, and the ratio between pXRF (or pXRF  $\times$  k) and ICP-OES was calculated using a raster calculator tool. This computed ratio served as an indicator of map reliability, while its spatial distribution highlighted regions with greater or lesser discrepancies in proximal PTM assessments, reflecting heterogeneity in soil contamination at the sites.

### 3 Results

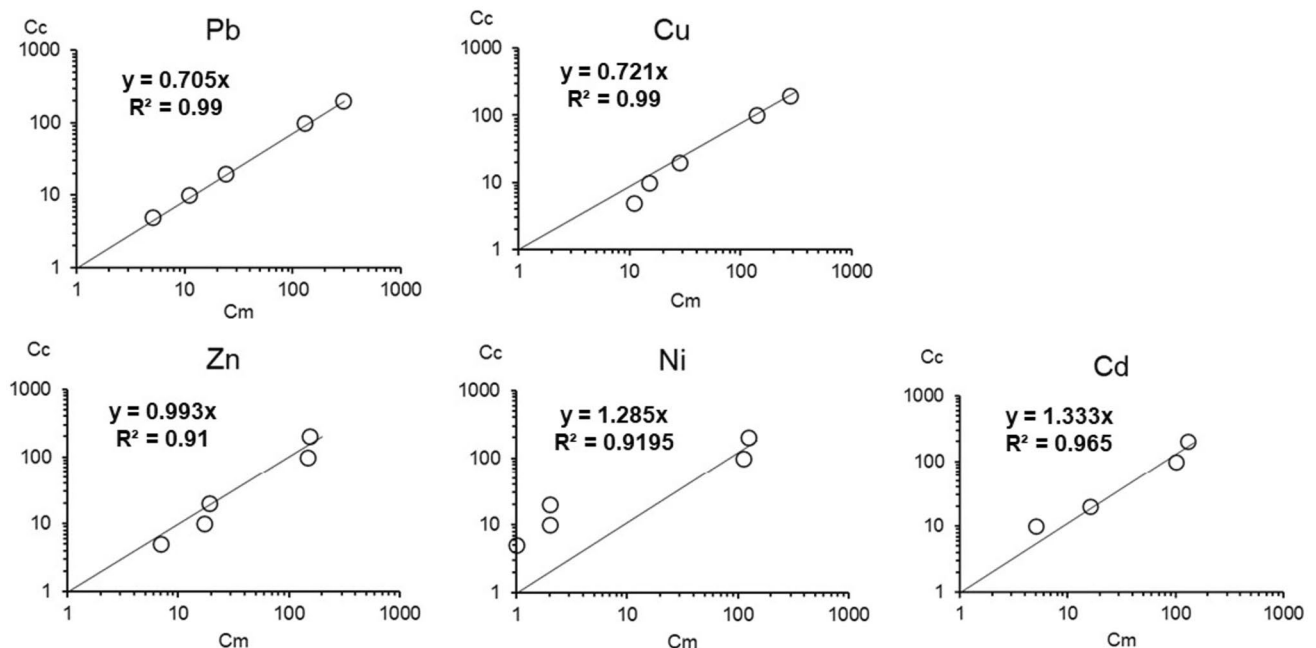
#### 3.1 Calibration experiments and estimating correction factors

Preliminary laboratory experiments using aqueous solutions were conducted to assess the impact of exposure duration and

PTM concentration on the accuracy of pXRF readings. This was done to incorporate these effects into the pXRF screening protocol. The pXRF readings obtained with an exposure time of 120 and 180 s for the second beam were nearly identical. In contrast, readings taken with a 90-s exposure showed significant deviations, especially at lower concentrations. As a result, an exposure time of 120 s for the second beam was established as the standard and subsequently used in pXRF measurements.

The accuracy of pXRF readings varied among the PTMs. Elements with a larger atomic mass, such as Pb, exhibited higher accuracy, while those with a smaller atomic mass, like Ni, showed lower accuracy. The reliability of pXRF results decreased in the sequence: Pb > Cu > Zn > Cd > Ni. The error margins for Pb and Cu were comparable to those of the reference ICP-OES method. However, for Ni, at contents below 200 mg kg<sup>-1</sup>, the error reached 50% or even higher (Fig. 2). Based on the preliminary calibration experiment, pXRF readings were considered reliable for Pb, Cu, Zn, and Cd at contents exceeding 10 mg kg<sup>-1</sup>. For Ni, pXRF results are trustworthy at contents above 200 mg kg<sup>-1</sup>, whereas at the lower contents, the method is more suitable for a qualitative rather than quantitative analysis.

Experiments involving various substrates and soil mixtures were conducted to evaluate the influence of the soil matrix on the accuracy of pXRF readings and to determine the correction factors. The derived correction factors exhibited significant variations between the elements. Additionally, there were notable differences when comparing organic materials (P and S3P) to mineral materials (all other substrates and mixtures) (Table 2).



**Fig. 2** Relationships between pXRF measurements ( $C_m$ ) and calculated contents ( $C_c$ ) of PTMs in aqueous solutions (all contents in mg kg<sup>-1</sup>). The equations display the results of the linear regression analysis, while the black line indicates a 1:1 line



The most consistent results between pXRF and ICP-OES were observed for Pb, Cu, and Ni. For these elements, the error margin remained within 25% for all mineral substrates. The pXRF readings consistently overestimated the ICP-OES values of Pb, Cu, and Ni across all materials, except for the Pb content in SL mixtures, where a minor underestimation was noted. Sand yielded the most accurate pXRF results. In contrast, pXRF readings for organic materials overestimated the ICP-OES values by 25 to 45% for all elements, with the exception of Cd. For Cd, the measurement error in organic materials soared to 100%. Unlike the other elements, the ICP-OES values for Cd were consistently underestimated by the pXRF readings.

### 3.2 Soil properties and contamination assessment at the case sites by pXRF and ICP-OES

The topsoil organic carbon contents in Repin's square were almost two times higher than in other sites. Peat-sand mixtures predominantly constituted the substrate under lawns, while loamy topsoils were more common under trees, shrubs, or near walking paths. The RUDN campus was dominated by loamy soils with relatively low SOC content. The topsoil of the urban forest was primarily sandy loam and had higher median carbon contents with a wider range compared to the soils at the RUDN campus. The topsoil in Repin's square was slightly alkaline, while the topsoil of the RUDN campus ranged from neutral to slightly alkaline. The topsoil of the urban forest was moderately acidic that is typical for the natural Retisols in the region (Fig. 3B).

Based on the ICP-OES measurement, the highest topsoil PTM contents were observed in Repin's square. The patterns identified for the RUDN campus and the urban forest varied depending on the specific PTMs. The urban forest exhibited elevated contents of Cu and Pb, while the soils of the university campus had higher contents of Ni and Cd (Table 3). On average, the topsoil PTM contents in both the campus and the urban forest remained below the APC thresholds (Table 2), whereas the values recorded at Repin's square surpassed the APC thresholds for all PTMs.

When evaluating pollution indices based on seven PTMs, the RUDN campus and urban forests displayed relatively low pollution levels. In stark contrast, the pollution levels in Repin's square were considerably elevated (Table 3). The comprehensive dataset highlighted the variation in urban soil properties, including SOC content, pH, and texture (Fig. 3). It encompassed a broad spectrum of pollution levels, ranging from pristine to highly contaminated soils (Table 3). This diversity provided an ideal backdrop for evaluating the efficacy of the pXRF rapid screening method in assessing soil contamination in Moscow.

The distribution (probability density function) of PTMs, as determined by pXRF readings across all locations, was compared with the ICP-OES measurements. The results, based on both methodologies, exhibited a left-skewed distribution curve (Fig. 4). Consequently, the data was log-normalized for subsequent analysis. According to the paired *t*-test, there was no significant difference between the mean PTM contents derived from the two methods (with a paired *t*-test for each PTM yielding a *p*-value > 0.05). On average, the pXRF results were elevated by 10 to 30% in comparison to the ICP-OES measurements. The most congruent outcomes between the two methods were observed for Pb. The distributions were nearly identical, and the ratio of pXRF to ICP-OES results hovered close to 1. This consistency was maintained across the various substrates and soil mixtures examined at the case sites.

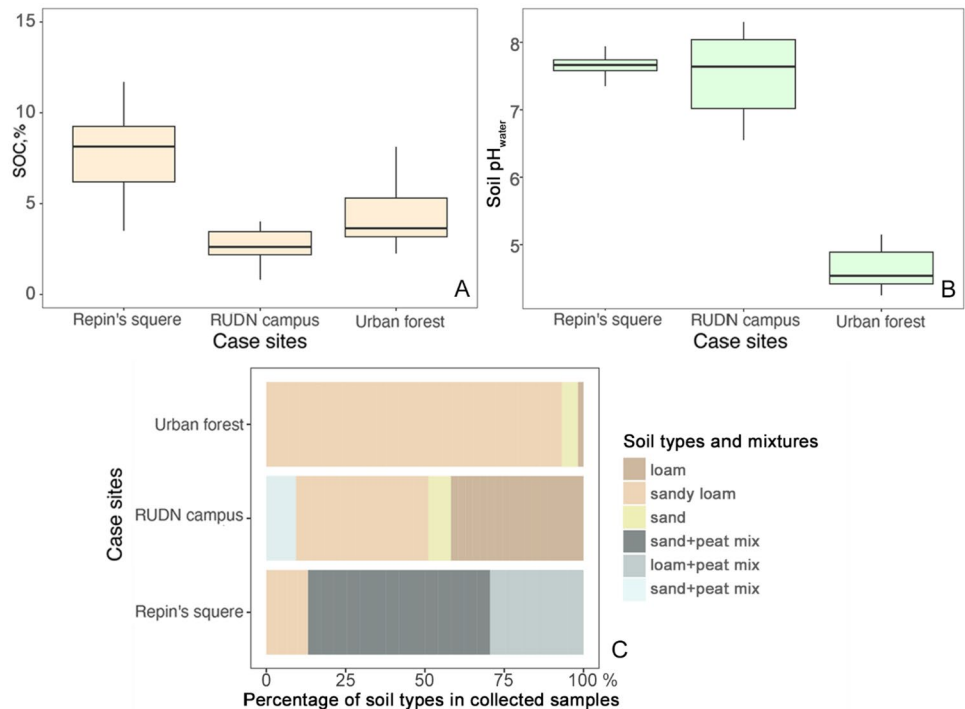
For Cu and Zn, the pXRF readings overestimated the ICP-OES values by 10 to 25%. Similarly to the laboratory experiments, the most significant overestimation of Zn content was observed in organic substrates. However, for Cu, the influence of the soil matrix on the accuracy of pXRF readings was not statistically significant (as determined by ANOVA with a *p*-value > 0.05). The most pronounced discrepancies were evident for Ni and Cd. For these elements, pXRF considerably underestimated the ICP-OES values, especially at lower contents (< 50 mg kg<sup>-1</sup> for Ni and < 5 mg kg<sup>-1</sup> for Cd). This reduced accuracy of pXRF readings at minimal contents is evident from the visual disparities in the distributions. While the ICP-OES distributions exhibited two distinct peaks, these were absent in the pXRF distribution curve. Specifically, for

**Table 2** Correction factors (*k*) for PTM contents in different soil matrixes based on the laboratory calibration experiments (italic, 0.90 > *k* > 1.10; bold, 0.75 > *k* > 0.89; bold italic, *k* < 0.75 or *k* > 1.10)

Soil mixtures			Pb	Cu	Zn	Ni	Cd
Index	SOC (%)	pH <sub>water</sub>					
S	0.09	5.2	<i>0.96</i>	<b>0.76</b>	<i>0.95</i>	<b>0.79</b>	<i>1.07</i>
L	3.46	7.3	<b>0.87</b>	<b>0.89</b>	<b>0.84</b>	<i>0.98</i>	<b>0.66</b>
P	33.37	6.5	<i>0.75</i>	<i>0.56</i>	<i>0.65</i>	<b>0.66</b>	<b>2.06</b>
SL	1.48	6.2	<i>1.06</i>	<b>0.88</b>	<i>0.69</i>	<i>0.93</i>	<b>0.88</b>
LP	10.43	7.0	<b>0.88</b>	<b>0.81</b>	<b>0.77</b>	<i>0.95</i>	<b>0.82</b>
SLP	5.02	6.7	<i>0.94</i>	<b>0.87</b>	<i>0.71</i>	<b>0.87</b>	<i>0.93</i>
S3P	13.05	6.2	<i>0.97</i>	<b>0.68</b>	<b>0.64</b>	<b>0.83</b>	<b>1.45</b>



**Fig. 3** The boxplots (median and interquartile range) of topsoil SOC contents (A) and pH<sub>water</sub> values (B), and the composition of soil mixtures present (C) at the case study sites



Cd content, the ratio between ICP-OES and pXRF results was as much as twofold, reinforcing the notion that pXRF is not suitable for quantifying low contents of Cd at least in the studied urban soils, where the average Cd contents were comparable to the detection limit (Fig. 4).

Given that the primary discrepancies between the pXRF readings and ICP-OES results for the soils of the case sites mirrored those observed in the calibration experiments, the previously estimated correction factors were applied to adjust the PTM results derived from pXRF. As a result, the accuracy improved significantly. The determination coefficient ( $R^2$ ) for the regression

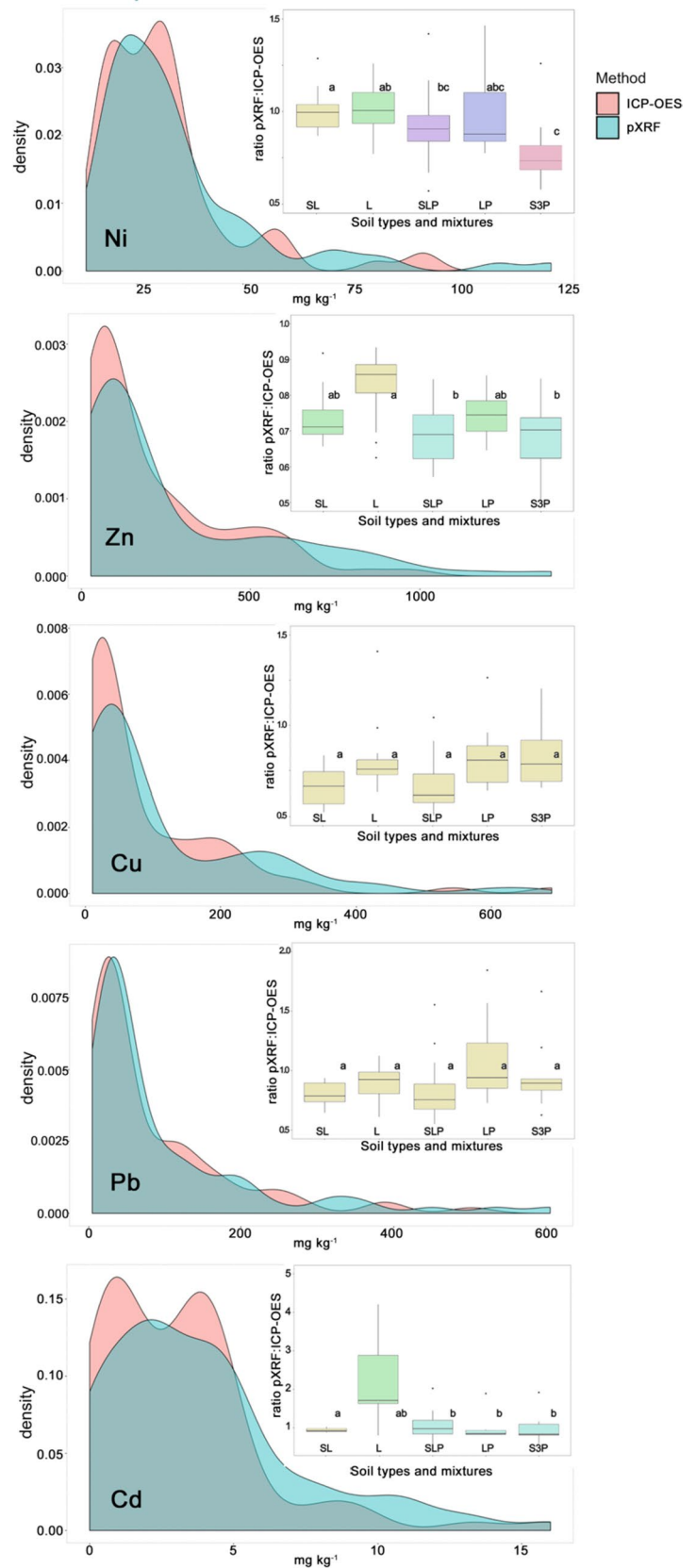
between ICP-OES and adjusted pXRF readings were 0.68, 0.93, 0.94, and 0.95 for Ni, Pb, Zn, and Cu correspondingly and the regression coefficients ( $b$ ) approached 1.0 (Fig. 5). Both ME and RMSE decreased substantially for Ni, Cu, and Zn. The following changes in ME and RMSE were obtained by implementation of the correction factors for Ni, Cu, and Zn correspondingly: ME (without/with regression factor)— $-9/-2$ ,  $-55/-13$ , and  $-176/27$ , for RMSE (without/with regression factor)— $12/10$ ,  $46/29$ , and  $127/56$ . For Pb, the errors before and after correction factor implementation were comparable and stayed below 20% of the mean. For Cd though, the errors were

**Table 3** Potentially toxic metal (PTM) contents are presented as mean  $\pm$  standard deviation in  $\text{mg kg}^{-1}$ , derived from ICP-OES analyses. The letters indicate homogeneous groups based on ANOVA post hoc Tuckey test. The formatting in the table delineates their relation to the APC (approximate permissible concentration) thresh-

olds from Table 1: italic for exceeding I APC, bold for surpassing II APC, and bold italic for surpassing III APC. The table also features soil pollution indexes, with italic indicating low, bold denoting moderate to high, and bold italic indicating extremely high levels of contamination

PTM Index	Urban forest ( $n = 25$ )		RUDN campus ( $n = 28$ )		Repin's square ( $n = 30$ )	
	PTM ( $\text{mg kg}^{-1}$ )	PI	PTM ( $\text{mg kg}^{-1}$ )	PI	PTM ( $\text{mg kg}^{-1}$ )	PI
As	$2.3 \pm 5.1^b$	$2.9 \pm 7.1$	$4.7 \pm 5.7^b$	<b><math>6.9 \pm 8.6</math></b>	$12.5 \pm 3.6^a$	<b><math>19.9 \pm 4.5</math></b>
Mn	$644.2 \pm 413.0^a$	$1.4 \pm 0.8$	$641.3 \pm 241.4^{ab}$	$1.3 \pm 0.5$	$509.3 \pm 185.2^b$	$1.0 \pm 0.4$
Ni	$22.0 \pm 9.0^c$	$0.8 \pm 0.3$	$32.7 \pm 13.5^b$	$1.1 \pm 0.5$	$38.7 \pm 18.6^a$	$1.3 \pm 0.6$
Cu	$63.8 \pm 132.0^b$	$1.7 \pm 3.4$	$33.1 \pm 18.7^b$	$0.8 \pm 0.5$	<b><math>171.7 \pm 112.4^a</math></b>	<b><math>4.4 \pm 2.9</math></b>
Zn	$107.1 \pm 139.6^b$	$1.6 \pm 2.0$	<b><math>129.3 \pm 99.7^b</math></b>	$1.8 \pm 1.4$	<b><math>389.3 \pm 234.6^a</math></b>	<b><math>5.6 \pm 3.3</math></b>
Pb	$56.6 \pm 61.9^b$	$2.1 \pm 2.3$	$28.0 \pm 17.3^b$	$1.0 \pm 0.6$	<b><math>165.7 \pm 115.3^a</math></b>	<b><math>6.1 \pm 4.3</math></b>
Cd	$0.7 \pm 0.8^b$	$2.0 \pm 3.1$	$3.7 \pm 0.4^a$	<b><math>8.9 \pm 1.2</math></b>	$4.7 \pm 4.1$	<b><math>10.1 \pm 9.9</math></b>
Zc	$11.3 \pm 17.7$		<b><math>39.2 \pm 10.9</math></b>		<b><math>73.0 \pm 42.4</math></b>	
PI <sub>Nemerow</sub>	<b><math>2.1 \pm 2.5</math></b>		<b><math>4.9 \pm 2.5</math></b>		<b><math>8.5 \pm 2.6</math></b>	

**Fig. 4** The density curves of PTM contents measured in  $\text{mg kg}^{-1}$ , with the ICP-OES method represented in pink and the pXRF method in blue ( $p > 0.05$ ). The accompanying boxplots (median and interquartile range) detail the ratios between the ICP-OES and pXRF measurements for different soil mixtures as described in Sect. 2.2

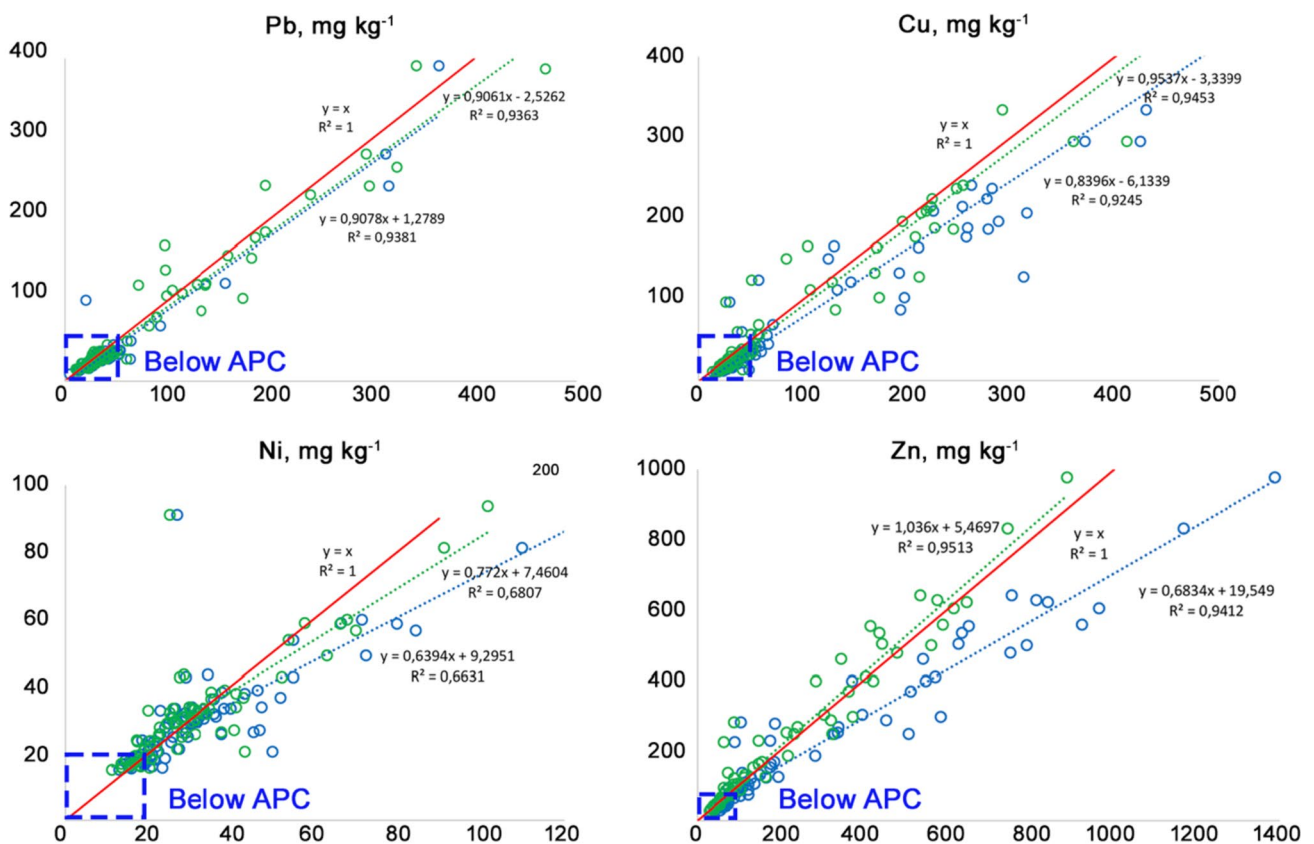


40 to 70% of the mean and did not decrease after adjustment. Given that both laboratory and field experiments consistently highlighted the limited accuracy of pXRF for Cd detection, this PTM was subsequently omitted from further soil contamination assessments and mapping at the case sites.

### 3.3 Mapping soil contamination

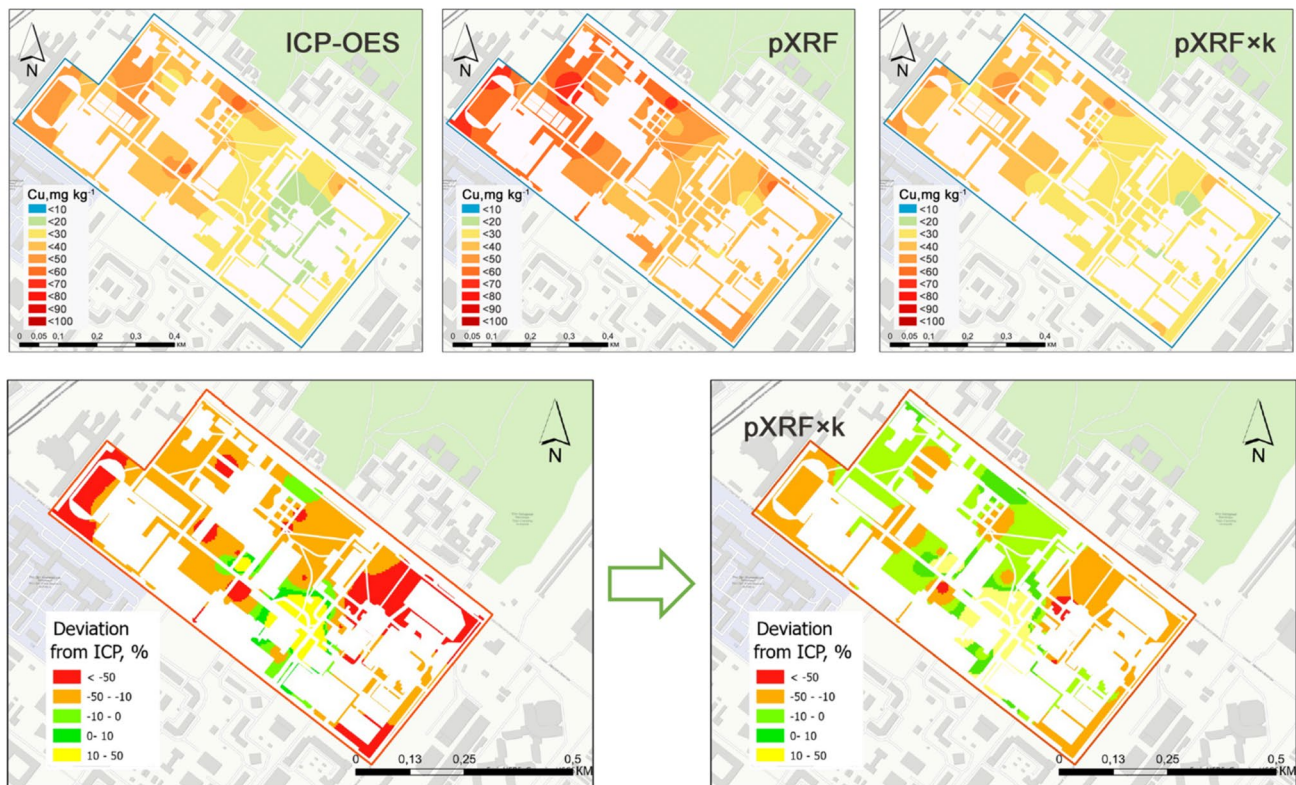
The implementation of correction factors allowed to increase in the reliability of the contamination maps interpolated from the point measurements, primarily by a more relevant reflection of the spatial patterns in contaminated and non-contaminated areas. The reliability of the maps was analyzed based on the deviation of the pXRF and pXRF  $\times$  k maps from the ICP-OES-based maps considered as a reference. For all PTM, the application of the correction factor substantially reduced areas with significant ( $> 30\%$ ) deviations on the PTM maps of the case sites. The fraction of the areas considered as non-deviated ( $\pm 10\%$  from ICP-OES values) increased from 2 to 6 times depending on the PTM and research site.

For instance, on the Cu map of the RUDN campus, the fraction of the areas with significant deviation reduced from almost 40% for the pXRF-based map to less than 5% for the pXRF  $\times$  k-based map (Fig. 6). Spatial patterns in these deviations coincided with different vegetation patches or soil properties. The deviation hotspot was observed in the area where peat-sand mixtures were used to reconstruct lawns. This suggests that high SOC content in these areas likely interfered with Cu detection by pXRF. This phenomenon was also observed in earlier laboratory experiments. The fraction of non-deviated areas for Cu, Pb, and Ni reached 50 to 70% that was substantially higher compared to the non-adjusted pXRF map. In Repins' square, located in the city center where PTM levels were significantly higher compared to the RUDN campus, the reduction in deviation was even more pronounced—the share of the non-deviated areas increased two times for Pb and Ni, 3 times for Cu, and almost 13 times for Zn. In the urban forest, the improvement of Zn and Ni maps was substantial and the share of non-deviated area on the adjusted maps of these PTM reached correspondingly 64 and 93%. However, for Cu and Ni, the application of the



**Fig. 5** The regression between the pXRF ( $x$ ) and ICP-OES ( $y$ ) analyses for the total soil dataset ( $n=83$ ) collected at the case sites, both with (green) and without (blue) application of the correction factor.

The red line indicates a 1:1 line, while the blue box highlights values below the APC(I) threshold



**Fig. 6** Spatial distribution of deviations in proximal assessments of Cu content from the ICP-OES reference, comparing pXRF without correction factors (left) and with correction factors (right)

correction coefficient did not allow to reach more than 25% of the non-deviated areas, which was the lowest result among all the sites (Table 4).

## 4 Discussion

### 4.1 Soil contamination by PTM in Moscow megapolis

Soil contamination by PTMs is a common environmental challenge faced by major cities, and the metropolis of Moscow is no exception. The median PTM contents we observed were higher than those in the suburbs (as reported by Romzaykina et al. 2021; Demina et al. 2022) and align with findings from previous studies conducted in Moscow (Nikiforova and Kosheleva 2007; Kasimov et al. 2016;

Nikolaeva et al. 2017). The pronounced spatial variability in PTMs is a characteristic trend in urban soil surveys (Ajmone-Marsan and Biasoli 2010) and can typically be attributed to the presence of multiple contamination sources. In Moscow, motor vehicles and industrial activities have historically been identified as the primary contributors to PTM accumulation in soils. A study of long-term dynamics in soil PTM contents by Kosheleva and Nikiforova (2016) highlighted a decline in Pb levels over the past decade, compared with a marked rise in Cd contents during the same timeframe. The gradual transition from leaded to unleaded gasoline, coupled with soil reclamation practices, has contributed to the reduction of Pb contamination. Conversely, the extensive use of mineral and organic fertilizers, as well as sewage sludge for urban greening, has led to an increase in Cd input into Moscow's urban soils. Elevated contents of Zn and Cu primarily

**Table 4** Comparison of site area percentages where pXRF measurements are within  $\pm 10\%$  of ICP-OES values, before and after applying the correction factor (pXRF  $\times$  k)

Object/method	Cu		Pb		Ni		Zn	
	pXRF	pXRF $\times$ k	pXRF	pXRF $\times$ k	pXRF	pXRF $\times$ k	pXRF	pXRF $\times$ k
Urban forest	21.4	25.7	3.3	11.3	83.0	92.8	0.0	63.6
RUDN campus	12.1	47.3	68.7	77.3	47.6	68.5	38.6	31.4
Repin's square	10.8	39.7	29.6	57.5	15.2	28.1	4.8	65.7



originate from car components, tire wear, and oil lubricants (Kasimov et al. 2016; Kosheleva et al. 2018). Given this, it's unsurprising that the soils of Repin's square, situated near a major highway, exhibited higher contamination levels of these elements compared to the other study sites.

Despite the contamination levels being significantly lower than those observed in other large cities such as New York City (Cheng et al. 2015), London (Kelly et al. 1996), Manila (Pfeiffer 1988), and Quezon City (Navarrete et al. 2017), the potential health risks remain considerable. The existing civil engineering and health standards, most of which were formulated in the 1980s and 1990s (e.g., RF GR -87 2008; CP 11–102-97 1997), mandate wet chemistry as the sole method for assessing soil contamination by PTM. The high labor and time demand of this method hinder the detailed assessment of spatial heterogeneity at site levels, which is critical for making informed soil management and land-use decisions. Currently, Moscow's environmental agency conducts soil PTM assessments in fewer than a thousand locations annually, a number insufficient given the city's vast and varied landscape (Kulbachevsky 2019, 2021). In this context, the exploration of pXRF screening as a low-cost alternative holds significant relevance, offering a promising avenue for more comprehensive soil contamination assessments in Moscow.

#### 4.2 Limitations of pXRF for PTM screening in urban soils

The pXRF instruments have gained popularity as a tool for assessing PTM contamination in soil, with various instruments being utilized in studies worldwide (Radu et al. 2013; Borges et al. 2020; Xia et al. 2022). The choice of instrument is based on specific study tasks, such as the range of elements, matrix, detection limits, and costs. From a variety of available instruments (e.g., Niton XL3t, Bruker Tracer III-V, or Innov-X Alpha PXRF0), we selected Olympus Vanta C shown by previous studies to be a trade-off considering the precision of the "Soil mode" and costs (Yang et al., 2015; Islam et al. 2018; McLaren et al. 2012; Urrutia-Goyes et al. 2017; Zhu and Weindorf 2011; Radu et al. 2013; Guo et al. 2020). Calibrating pXRF instruments is essential for accurately measuring PTM contents in soil samples, and various calibration methods have been explored. Some studies used certified reference materials or soil standards for calibration (Bispo et al. 2021), while others employed regression models (Wan et al. 2020) or a combination of external and internal calibration (Jeong et al. 2021). We develop correction factors based on regression between pXRF readings and ICP-OES measurements calculated for different PTMs and soil matrixes (substrates and soil mixture).

Among the five tested PTMs, the pXRF estimates for Cu, Zn, Ni, and Pb contents proved to be reliable and

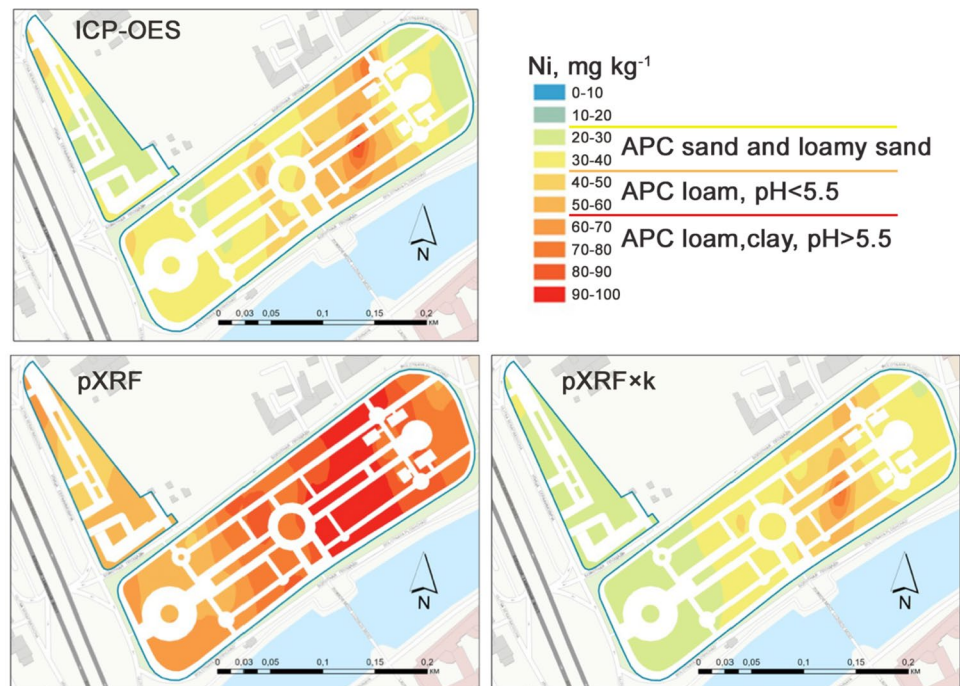
accurate, whereas the accuracy of Cd measurements was notably lower. A plausible explanation for this is that the Cd content in a significant portion of the total sample set was below  $10 \text{ mg kg}^{-1}$ , a value similar to the pXRF detection limit. A similar observation was made in a recent urban soil survey conducted in New England, USA (Sirkovich et al. 2023). In that study, Cd contents ranged between 0.3 and  $3.0 \text{ mg kg}^{-1}$ , which remained below health thresholds. In our research, the Cd contents exhibited a similar range. However, the health thresholds applied in Moscow are considerably more stringent. Given that Cd ranks among the primary pollutants in Moscow's urban soils, relying solely on pXRF could result in overlooking Cd-contaminated sites, thereby posing potential health risks.

The accuracy achieved for other elements was influenced by the properties of the soil matrix, primarily soil texture and organic carbon content, as well as by the masking effect of other elements, e.g., iron (Górka-Kostrubiec et al. 2023). The most accurate measurements were observed for sandy and loamy sandy soils with SOC content of less than 5%. In contrast, PTM contents in peat and peat-sand mixtures were notably overestimated by pXRF. The masking effect of soil organic matter has been well-documented in previous studies (Kalnicky and Singhvi 2001; Paltseva et al. 2022). This effect can be mitigated through additional sample processing, such as the removal of organic matter following the loss on ignition protocol (Ravansari and Lemke 2018). Topsoil in Moscow's green areas often contains imported organic materials, such as peat and composts, and boasts high SOC stocks (Ivashchenko et al. 2019; Vasenev et al. 2021). Given this, the removal of organic matter is advisable as a preprocessing step in the pXRF potentially toxic metal measurement protocol.

#### 4.3 Perspectives on rapid soil contamination screening in urban planning, soil, and environmental management

While pXRF soil contamination screening has its limitations, its straightforward application has made it a sought-after tool for routine tasks in the development and upkeep of urban green spaces, including soil quality control in playgrounds, allotment gardens, and urban farms (Paltseva et al. 2020; Răcușan Ghircoiaș et al. 2023). The method is particularly useful for detailed mapping and identifying "hot spots" at a local scale (Bechet et al. 2018). It facilitates discerning areas that may necessitate cultivation restrictions, thereby serving as an effective tool for soil management (Levin and Paltseva 2023). For instance, Venvik and Boogaard (2020) utilized pXRF to pinpoint soil contamination by Pb, Zn, and Cu from stormwater, subsequently offering recommendations for maintaining sustainable urban drainage systems in the Netherlands. Landes et al. (2019) developed a field

**Fig. 7** Illustration of the varying levels of soil contamination by Ni at Repin's square, along with the corresponding land-use restrictions. The data is based on pXRF screenings, showcasing results with and without the application of correction factors



procedure to screen soil for hazardous Pb contents, which is intended for use by the general public. They utilized a pXRF analyzer to measure Pb contents in glycine extract solutions derived from soil samples, allowing for a continuous scale measurement of Pb levels.

The improvement in mapping PTM obtained for the case sites in our research has a strong practical outcome for land-use planning and soil management since the approach allows a more accurate identification and delineation of the contaminated and clean areas within urban sites. For example, the map of Ni content at the Repin's square based on pXRF shows that almost 70% of topsoils in the area are contaminated (Ni contents are above APC) and shall be removed or remediated. The corrected ( $\text{pXRF} \times k$ ) map reflects a higher variability of soil contamination levels, where contaminated topsoils are concentrated in a more disturbed area at the North-East part of the park dominated by lawns and cover about 20% of the area, whereas 40% of the area at the West part of the park dominated by trees and shrubs has clean soil and can be used without restrictions (Fig. 7). Therefore, a more accurate contamination screening considering soil heterogeneity supports taking relevant decisions on land use and reduces reclamation expenses by up to 50,000 USD compared to a scenario where the entire park area is treated as contaminated per existing soil monitoring guidelines (GR-514 2004).

Moreover, pXRF screening stands as a cost-effective strategy to elevate public awareness regarding soil contamination. This approach aligns with the growing recognition of community-level stewardship and the importance of network

building and information exchange in urban soil management, as highlighted by Schwarz et al. 2016. Such stewardship, bolstered by educational resources, enables communities to better understand and value the benefits of soils in urban areas (Pouyat et al. 2020). Schools, Living Labs, and Non-Government Organizations can leverage pXRF tools for environmental consultancy, or encourage individuals to bring soil samples to labs for rapid contamination testing (e.g., Alabama A&M and Auburn Universities Extension, Delta Urban Soils Laboratory 2013, Cornell University, Macquarie University's VegeSafe). The "Map my environment" 2013 project is a testament to the potential of such initiatives, fostering informed public opinion and enhancing the sense of security regarding soil conditions in urban settings (<https://www.mapmyenvironment.com>, Taylor et al. 2021). By facilitating access to information on soil contamination, pXRF screening can significantly influence public perception and foster a sense of security in urban planning and development policies.

## 5 Conclusion

This study prominently highlights the potential of portable X-ray fluorescence (pXRF) as a robust tool for assessing urban soil contamination. When optimized with essential preprocessing steps such as drying, grinding, and homogenization, and considering the soil matrix properties, pXRF demonstrates enhanced accuracy and reliability. This is

especially vital in urban environments, where soil heterogeneity can significantly impact the detection and assessment of contamination. The need for rapid, accurate, and cost-effective soil testing methods is more pressing than ever, and pXRF's integration into soil contamination assessment meets this need efficiently, making it accessible to a wide range of stakeholders.

The growing momentum of citizen science projects in urban environmental monitoring is noteworthy, as these initiatives empower community members to actively engage in data collection and analysis. Utilizing pXRF in such projects enhances public engagement and awareness about soil contamination issues, fostering a more informed and involved community. This participatory approach can be a key to promoting sustainable urban development and supporting improving the quality of life in urban settings.

Future research should aim to refine the detection capabilities of pXRF, particularly for elements like cadmium where current limitations exist. Expanding the scope of the study to encompass a wider array of soil types and integrating pXRF data with GIS for advanced spatial analysis will offer more profound insights into the patterns of urban soil contamination. Moreover, nurturing community engagement through citizen science initiatives and evaluating the long-term effects of soil contamination on public health and urban policy are essential steps toward a holistic approach to urban environmental management. By enabling broader community participation in environmental monitoring and management, pXRF not only serves as a practical tool for soil assessment but also plays a pivotal role in shaping environmentally conscious urban communities.

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## Declarations

**Conflict of interest** The authors declare no competing interests.

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