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Contents of Potentially Toxic Elements in Forest Soils of the Jizera Mountains Region

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Abstract In central Europe, the region termed 'the Black Triangle' corresponds to an area of concentrated burning of fossil fuels during the period between early 1950s up to the mid 1980s. Although major polluting activities have ceased, effects of atmospheric deposition are still evident. The aims of this study were to determine potentially toxic element (PTE) concentrations in organic and mineral horizons of forest soils in the Jizera Mountains region and to assess the relationship between their distributions and stand factors (altitude, grass cover, forest type). One hundred thirty-eight samples were taken from 98 positions, comprising O, A, E and B horizon samples. Target elements (Cu, Cd, Pb, Zn, Mn) were extracted using 2 M HNO_3 and then analysed by FAAS. Basic statistical parameters were determined, and correlation, *t* test and ANOVA were used to assess the relationship to stand factors. Maps were created using ArcMap 9.1. Two map creation techniques were used—kriging and inverse distance weighting. Highest mean metal contents (except for Mn) were found in organic horizons, with Cu and Pb being particularly concentrated in the upper layers. Strong positive correlation exists between Cd content and altitude, while negative correlation exists for Mn. Cadmium concentration was also significantly higher in areas of grass cover and under spruce canopy as opposed to beech. Surface maps indicated a similar source of pollution with highest concentrations corresponding also to relief and the prevailing wind direction. Overall, mean PTE contents were not excessive. Top enrichment factors proved the anthropogenic origin of Cd, Cu, Pb and Zn, whereas Mn is probably of geologic origin. Analysis of stand factors showed a statistically significant influence of altitude, grass cover and forest type on cadmium concentration in forest soils. Spatial distribution of metals showed a similar pattern for Cd, Cu, Pb and Zn in O horizon soil, which points toward a single source of pollution.

Keywords Forest stand · Forest soils · Atmospheric deposition · Distribution mapping · Heavy metals

1 Introduction

As a consequence of poorly regulated industrial development during the Communist era and extensive coal burning for power generation, the Jizera Mountains region in the Czech Republic is recognised as being one of the most severely affected areas by atmospheric deposition in Europe [16, 27]. Being at the centre of what is referred to as 'the Black Triangle', the Jizera Mountains experienced heavy loads of acid deposition which peaked in the mid-1980s and resulted in the manifestation of several forest degradation processes, including decreasing rate of humification, decrease in quality of soil microbial activity and the formation of aluminium toxic forms in the soil [18, 20, 26]. Irrespective of acid rain, fossil fuel combustion and pyrometallurgic industry are responsible for anthropogenic loads of potentially toxic elements (PTEs), in the form of wet and dry deposition [1]. In areas of known atmospheric pollution due to the aforementioned industrial activities, a positive correlation exists with the increased presence of PTE in soil such as As, Cd, Cr, Cu, Mn, Ni, Pb, Sb and Zn [22]. As part of a spatial interpretation of ambient air quality for the Czech Republic, Hůnová [14] identified the Jizera Mountains as one of the regions exhibiting the poorest air quality in terms of both the ambient air quality and wet atmospheric deposition of pollutants.

When present in excessive concentrations, PTEs exhibit a range of effects, which are detrimental to the growth and reproduction of plants, and have a negative effect on the

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activity of microorganisms, disrupting important biological processes such as nitrogen fixation, adenosine triphosphate production, soil enzyme activity and subsequently microbial biomass production [3]. Additionally, studies have shown that high metal concentrations alter litter decomposition due to impaired biological functions [4, 10], and the distribution of metals is dependent on litter turnover mostly due to the binding affinity between metals and humified substances [8, 30]. Following political revolution at the end of the 1980s, industrial emissions and the associated atmospheric concentrations of harmful products including PTE have shown a gradually declining trend [25]. However, heavy metals and metalloids persist in the environment indefinitely; therefore, their presence and effects should be monitored. Generally, in soils formed under a cool humid climate, the leaching of trace elements downwards through the profiles is greater than their accumulation, unless there is a high input of these elements into the soils [15]. The forest floor is relatively consistent in its composition, with a litter and humic horizon featuring high cation exchange, overlaying a mineral soil which has little mixing with the upper organic layers [27], highlighting the need for vertical analysis of these soils.

A detailed survey of the Jizera Mountain forests was performed by Borůvka et al. [5, 6], with soil sample analysis focusing on Al distribution and forms. The study identified highest available Al_{KCl} contents in organic surface horizons (related to low pH, high S and N and low Ca and Mg). Organically bound aluminium ($Al_{Na4P2O7}$) was more abundant under spruce compared to beech, with increased concentrations in clear cut areas covered with grass. In a recent publication, Harmens et al. [12] present temporal changes of heavy metals from atmospheric deposition in Europe through the use of mosses as bio-indicators, noting the greatest decrease since 1990 in the concentrations of As, Cd, Pb and V.

The main aim of this study was to determine the level of PTE (Cd, Cu, Mn, Pb, Zn) pollution in samples from the organic and mineral horizons of forest soils in the Jizera Mountains region. The distribution of PTE and their relationship to stand factors (vegetation cover type and altitude) were assessed, and surface mapping was used for the visual projection of PTE distribution.

2 Materials and Methods

2.1 Area Description

The Jizera Mountains region is a protected area in the northern Czech Republic. Altitudes of the sampling sites ranged from 400 to 1000 m (Fig. 1a). Mean annual rainfall in the region ranges from 900 to 1600 mm/year (Fig. 1b). Soils were identified as Podzols (Haplic or Entic) and Cambisols (mainly Dystric). Soil units generally correlate to altitude with Cambisols prevailing in lower positions and Podzols in the

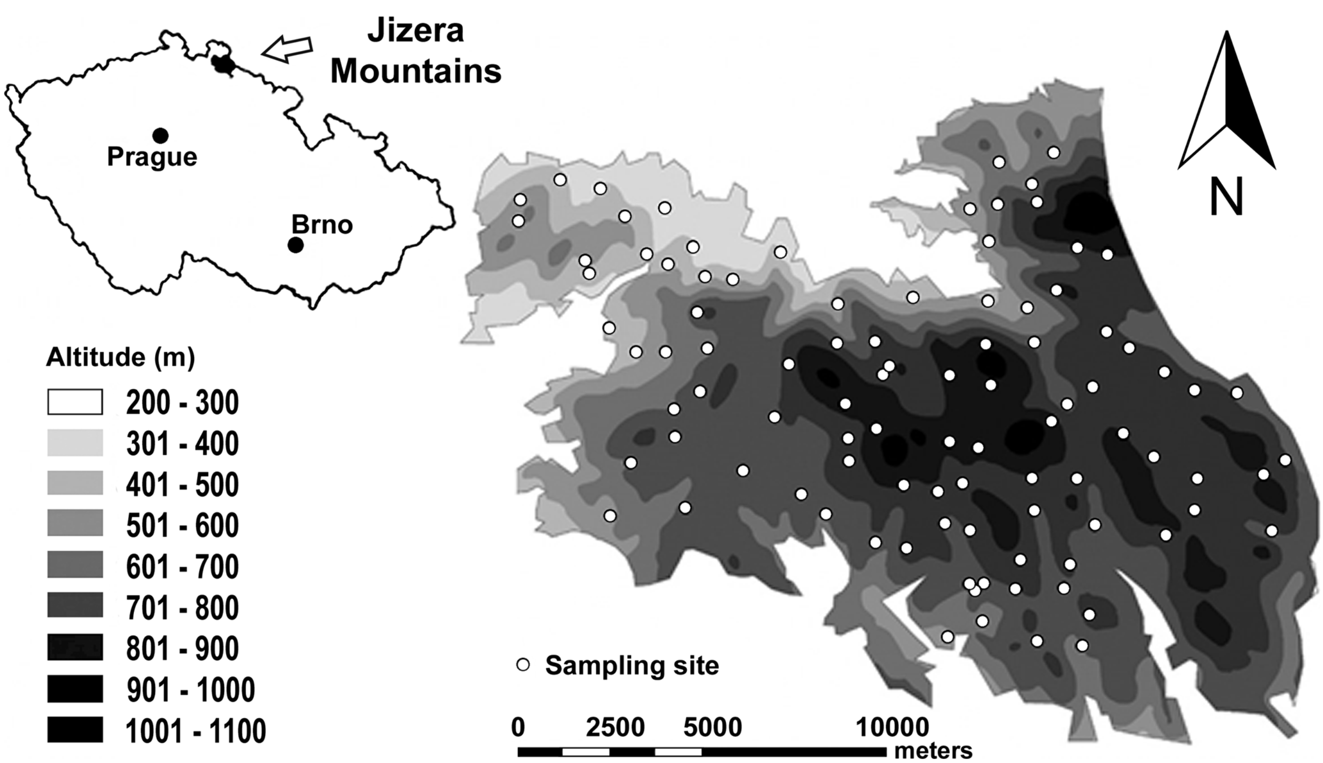
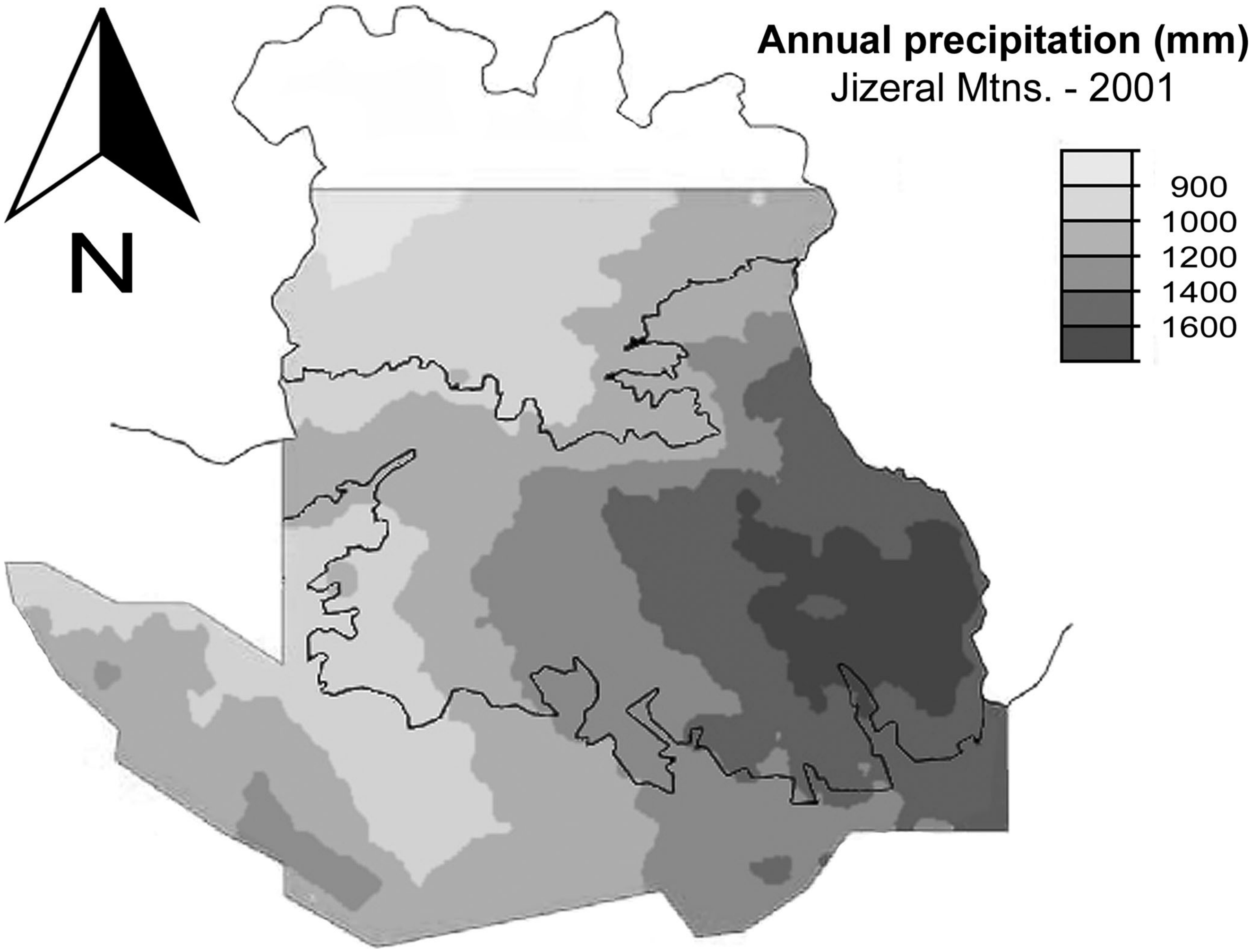


Fig. 1 **a** Sampling region in the Jizera Mountains and corresponding altitude. **b** Mean annual precipitation of the Jizera Mountains region in 2001 [24]. **c** Map of Jizera Mountains region showing tree age and type



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higher positions. All soils were formed on granite bedrock. The prevailing forest type was Norway spruce (*Picea abies*) monoculture, with areas of European beech (*Fagus sylvatica*) forest concentrated in the north-western and northern edge. A large part of the cover in the higher positions consists of open grass area (*Calamagrostis villosa*). Forest stand type and age are illustrated in Fig. 1c.

2.2 Soil Sampling

From an original sample set (e.g. Mládková et al. and Mládková et al. [18, 19]), 138 samples were selected from 98 positions, comprising 23 O horizon (mixture of F and H horizons), 18 A horizon, 27 E horizon and 70 B horizon samples. Samples were taken according to an irregular grid covering the area with an average sampling density of approximately 1 site/2 km^2. With respect to grass cover, 30 % of samples were from fully covered sites, 9 % from partially covered and 61 % from sites with no grass. Samples were air-dried and sieved to 2 mm prior to analysis.

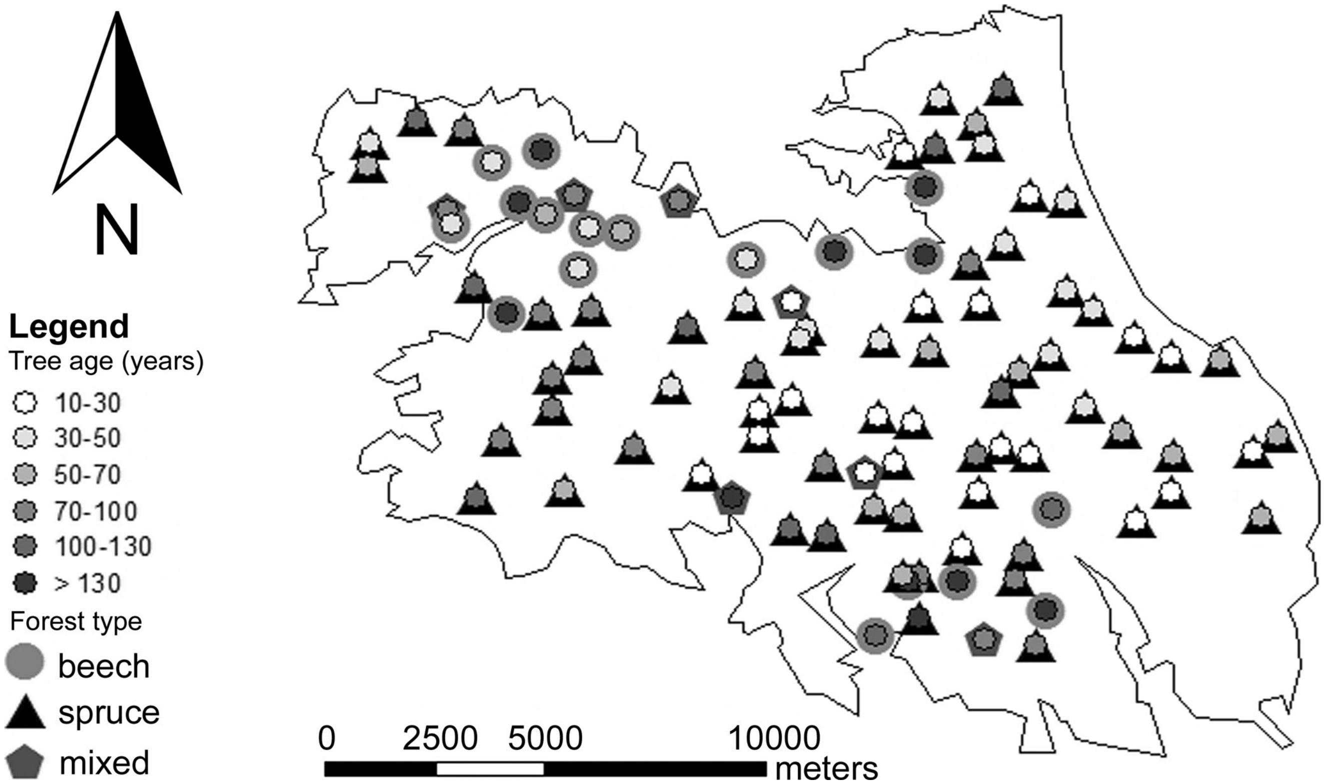
2.3 Analysis Methodology

Concentrations of target elements in soil samples were determined by continuous shaking of 5 g of soil in 50 ml of 2 M HNO_3 for 6 h. Extracts were then filtered (Filtrak 390) and analysed for Cd, Cu, Mn, Pb and Zn by FAAS (Varian 200 with SIPS 20 using Analytika s.r.o standards). Samples and standards were matrix matched, and all analyses were performed in triplicates.

Statgraphics Plus 4.0 was used for the determination of basic statistical parameters, and for assessment of statistical significant differences between variables, correlation, *t* test for independent, normally distributed samples and analysis of variance (ANOVA) were used to assess at the 0.05 significance level, the relationship to stand factors, altitude, forest type and grass cover, respectively.

Top enrichment factor (TEF) was calculated as the ratio between O horizon and B horizon contents of PTE.

Final maps were created using ArcMap 9.1 software. Interpolation was used as a basis of the maps formation. Two



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map creation techniques were used; ordinary kriging was applied in cases where spatial dependence among variables was observed. Inverse distance weighting was used in four cases: Cu and Zn in O horizon and Cu and Mn in A + E horizons. Due to a relatively small number of sampling points, maps show only general spatial trends of values, not detailed spatial distribution. In particular, the values close to the area border have higher uncertainty due to extrapolation. For the purpose of mapping, A and E horizons were combined in some cases, the A horizon in Podzols was either missing or was very shallow, and in other cases, the A horizon showed some eluviation. O and B horizons are presented individually.

3 Results

3.1 Basic Statistical Parameters

Mean and standard deviation were determined for O, A, E and B horizons (Table 1). PTE concentrations were measured in the following ranges: Cd 0.02–1.9, Cu 0.91–29.2, Mn 0.50–481.6, Pb 10.9–326.3 and Zn 3.38–27.2 mg/kg. Highest mean concentrations of PTE were found in O horizons for all elements with the exception of Mn where the highest mean was observed in the B horizon. One-way ANOVA was applied to data to determine the significance of PTE distribution between horizons. Although significant differences among sample means exist for all PTE, the most pronounced is for Cu and Pb with strongest retention in the organic horizon. The probable geologic source of Mn as opposed to anthropogenic input is evident due to higher contents with increased depth. The likely origin of PTE into forest soils is also expressed by the TEF in Table 2.

TEFs were calculated for the studied PTE (Table 2). TEF values greater than 1 indicate accumulation of PTE in the upper horizon, whereas a TEF value less than 1 generally indicates geological origin or the possibility of leaching from top soil. High TEF values were observed for Cu and Pb, which reflects the binding affinity that these elements have towards organic matter [2]. TEF values >1 were observed for Cd and Zn. However, Mn has a value less than 1, indicating that this element either originates from parent material or is highly mobile.

3.2 Spatial Relationship with Stand Factors

3.2.1 Altitude

A highly significant positive correlation between altitude and Cd concentration was identified in the O horizon and, to a lesser extent, in the B horizon, while a significant negative correlation was determined for Mn in almost all horizons

Table 1 Basic statistical parameters of PTE in soil horizons

Horizon	Parameter	Cd	Cu	Mn	Pb	Zn
O (23)	Mean (mg/kg)	0.82	15.2	37.1	163	14.6
	Standard deviation	0.44	5.09	74.5	52.9	3.62
A (18)	Mean (mg/kg)	0.34	4.75	12.3	52.7	8.44
	Standard deviation	0.19	3.04	8.60	16.0	2.81
E (27)	Mean (mg/kg)	0.26	4.04	15.1	32.6	6.25
	Standard deviation	0.21	2.23	47.9	14.7	1.72
B (70)	Mean (mg/kg)	0.49	3.13	81.4	38.2	13.6
	Standard deviation	0.15	1.85	83.1	19.3	4.46
F ratio of residual square mean	25.0	102	8.66	132	33.2	
P value of significance	<0.001	<0.001	<0.001	<0.001	<0.001	

Value in parenthesis refers to number of samples

(Table 3). Other elements showed mostly weak and non-significant dependencies on altitude with the exception of Cu in the B horizon.

3.2.2 Grass Cover

ANOVA and multiple range tests were applied to individual data sets by horizons for comparing forest vegetation zones. In the O horizon, a statistically significant difference among the variables was determined at the 95 % confidence level for Cd (Table 4), with mean values decreasing as follows: full grass cover (1.26 mg/kg) > partial grass cover (1.01 mg/kg) > no grass cover (0.58 mg/kg). Cu, Mn, Pb and Zn did not show any statistically significant difference with respect to grass cover, and no significant difference between grass cover categories was determined in mineral horizons.

3.2.3 Forest Type

t Test was used to determine differences among PTE contents under spruce and beech forest (Table 5). Given that *P* value is <0.05, there is a significant difference between the two types of mono-species forest at the 95 % confidence level in the case of Cd. Although not statistically significant, there was also an observed higher mean content of Pb and Cu under spruce forest compared to beech, whereas Zn and Mn were higher under beech.

Table 2 Top enrichment factor (mean values)

	Cd	Cu	Mn	Pb	Zn
TEF	1.46	7.80	0.69	5.53	1.20

3.3 Map Projection

PTE spatial distribution maps are given in Fig. 2. The maps showed a similar distribution of Cd, Cu, Pb and Zn in the O horizon, indicating a common source of pollution between these elements. Such similarity was not observed for the lower horizons. Maximum O horizon contents of Cd, Cu, Pb and Zn were found in the central western half of the region, corresponding to the relief and main windward direction. Kriging errors are given in Fig. 2d for Cd in all horizons. The errors were similar for all studied PTEs, so for the purpose of simplicity of presentation, only Cd is shown.

The spatial distributions of TEF (calculated as the ratio between PTE concentrations in O and B horizons) are illustrated in Fig. 3. No significant common pattern was observed for TEF value distribution or relationships between particular elements.

4 Discussion

A broad study concerning the distribution of PTE in forest floor humus in the Czech Republic was carried out by Suchara and Sucharová [27] only a decade ago. Humus samples taken from the Jizera Mountains contained elevated levels of Al,

Table 3 Correlation coefficients between PTE content and altitude in different horizons

Horizon	Cd	Cu	Mn	Pb	Zn
O	0.67***	−0.01	−0.45*	0.33	0.05
A	−0.39	0.06	0.07	−0.09	−0.08
E	0.10	0.32	−0.52**	0.09	0.03
B	0.25*	0.37**	−0.43***	0.06	−0.31*

*,**,***Significant at 5, 1 and 0.01 probability level, respectively

Table 4 Multiple range test and ANOVA for grass cover effects (O horizon)

Parameter	Cd	Cu	Mn	Pb	Zn
Full grass cover (mg/kg)	1.26 b	13.76	7.92	191.53	16.32
Partial grass cover (mg/kg)	1.01ab	17.56	7.98	148.88	12.14
No grass cover (mg/kg)	0.58 a	15.54	55.89	150.71	14.16
F ratio of residual square mean	10.32	0.48	1.10	1.46	1.33
P value of significance	0.001	0.627	0.353	0.255	0.287

Within the columns, values followed by the same letter are not significantly different at $P \leq 0.05$

most likely present as $Al(OH)_3$ which is dispersed throughout the water phase of coal [21]. As, Cr and Mo occurred from the combustion of lignite containing arsenopyrite, Cr- and Mo-bearing compounds, mostly in the north-western Bohemia region [27] and V from chemical and oil industries. Considerably lower concentrations of Cd, Cu, Pb and Zn were observed in the forests of northern Czech Republic within the Black Triangle. Contamination with Cd and Cu generally corresponds with the operation of steel and engineering industries with increased soil concentrations found largely in the northern Slovak Republic. Some increased humus concentrations of Cu may be attributed to the combustion of lignite. Lead contamination in the Czech Republic correlates most strongly with smelting and Pb processing operations, found mostly in southern Bohemia, particularly in Přibram. Hot spots of Zn generally correlate with steel and engineering industries and processing of polymetallic ores, which were concentrated in central and south-west Bohemia [27] as opposed to in the Black Triangle area, which was impacted mostly by coal burning. A similar trend of PTE occurrence was observed in our research whereby the studied elements did not prove to be particularly high when compared to Czech guidelines concerning PTE contents in agricultural soils [17] (no guidelines have yet been published for forest soils of the Czech Republic). Following are the maximum aforementioned elements in the O horizon as a percentage of the Czech agricultural guideline value (AGV) for comparison: Cd (191 %), Cu (58.3 %), Pb (407 %) and Zn (21.7 %). Only Cd and Pb exceed the AGV for a 2 M HNO_3 extraction. However, these high values do not necessarily have to present a danger, as the nature of the forest floor with respect to soil organic matter content and sorption ability is incomparable to the top soil of arable land.

Table 5 *t* test for PTE content and forest type (O horizon)

Parameter	Cd	Cu	Mn	Pb	Zn
Beech (mg/kg)	0.53	14.78	108.80	139.68	16.65
Spruce (mg/kg)	1.02	15.90	10.76	178.93	14.14
Independent *t*	−3.42	−0.42	1.84	−1.49	1.35
P value of significance	0.003	0.680	0.125	0.155	0.193

The differentiation between geological enrichment and anthropogenic pollution in weakly polluted soil samples from northern and north-eastern Czech Republic was studied by Borůvka et al. [7]; their results suggest that Cu and Zn are mainly of geological origin and that Pb and Cd are a considerable contribution of anthropogenic pollution. In this study, the TEF identified higher proportions of all PTE in the O horizon compared to predominantly mineral subsoil, with the exception of Mn which is likely to be originating from parent material. Map projection shows the greatest occurrence of Mn in all horizons in the north-western portion of the studied area (Fig. 2c). Geologic composition of the northern part of the studied locality includes orthogneiss, which has a high variable content of MnO (0.01 to 0.27 %), and phonolite of the Paleogene-Neogene age with 0.20 % MnO. Homogenous porphyritic granites in the southern part contain manganese-bearing compounds to a lesser extent of 0.04–0.09 % [9]. When forest types were compared, the mean value for Mn in beech forest which was considerably higher than in spruce can be attributed to two aspects: geological origin and/or deeper root systems of beech, which can transport Mn up from the subsoil. Hernandez et al. [13] investigated the heavy metal distribution in 11 selected forest sites in France with different types of atmospheric deposition. The abundance of heavy metals measured in these soils decreased as follows: Zn > Pb > Cu > Cd. In the case of Cu and Zn, some surface soil horizons presented enrichment factors ranging between 2 and 4, whereas Pb and Cd enrichment factors were far higher in surface horizons of forest soils, which were expected to be derived from long range transportation of anthropogenic atmospheric pollutants. In this study, we identified the highest TEF in the case of Cu (7.8) and Pb (5.5). As organic matter is one of the most influential factors controlling the movement of Cu and Pb, it is logical that TEF is highest for these elements in the forest floor where there is a great deal of surface humic material. This hypothesis was confirmed, to some extent, in the Polish Karkonosze National Park, a mountainous forest region, which neighbours our studied Jizera Mountains region. Szopka et al. [29] identified lead concentrations in upper (0–10 cm) forest floor layers in the range of 19–248 mg/kg. Calculated pools of Pb correlated strongly with organic matter; however, spatial distributions were

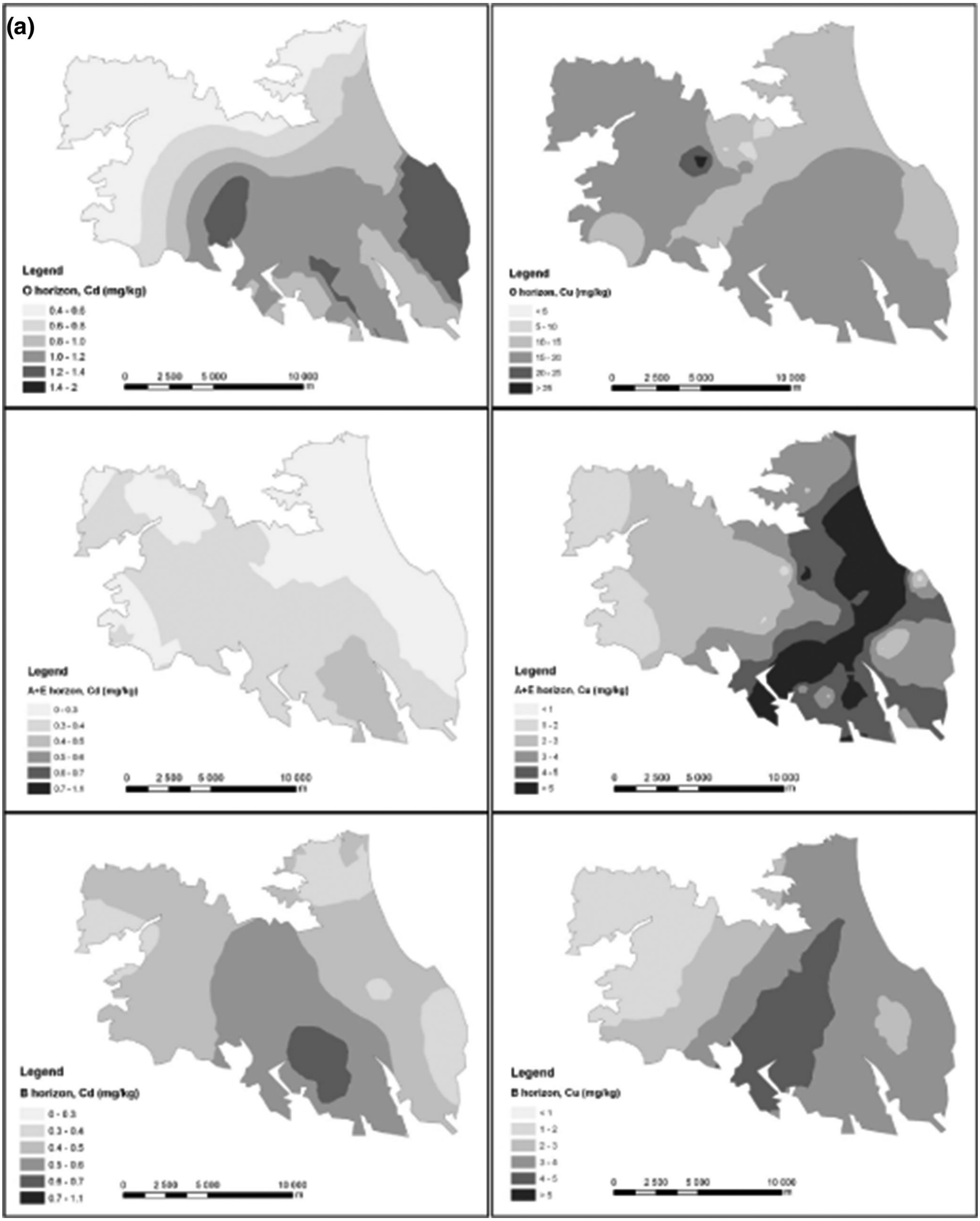
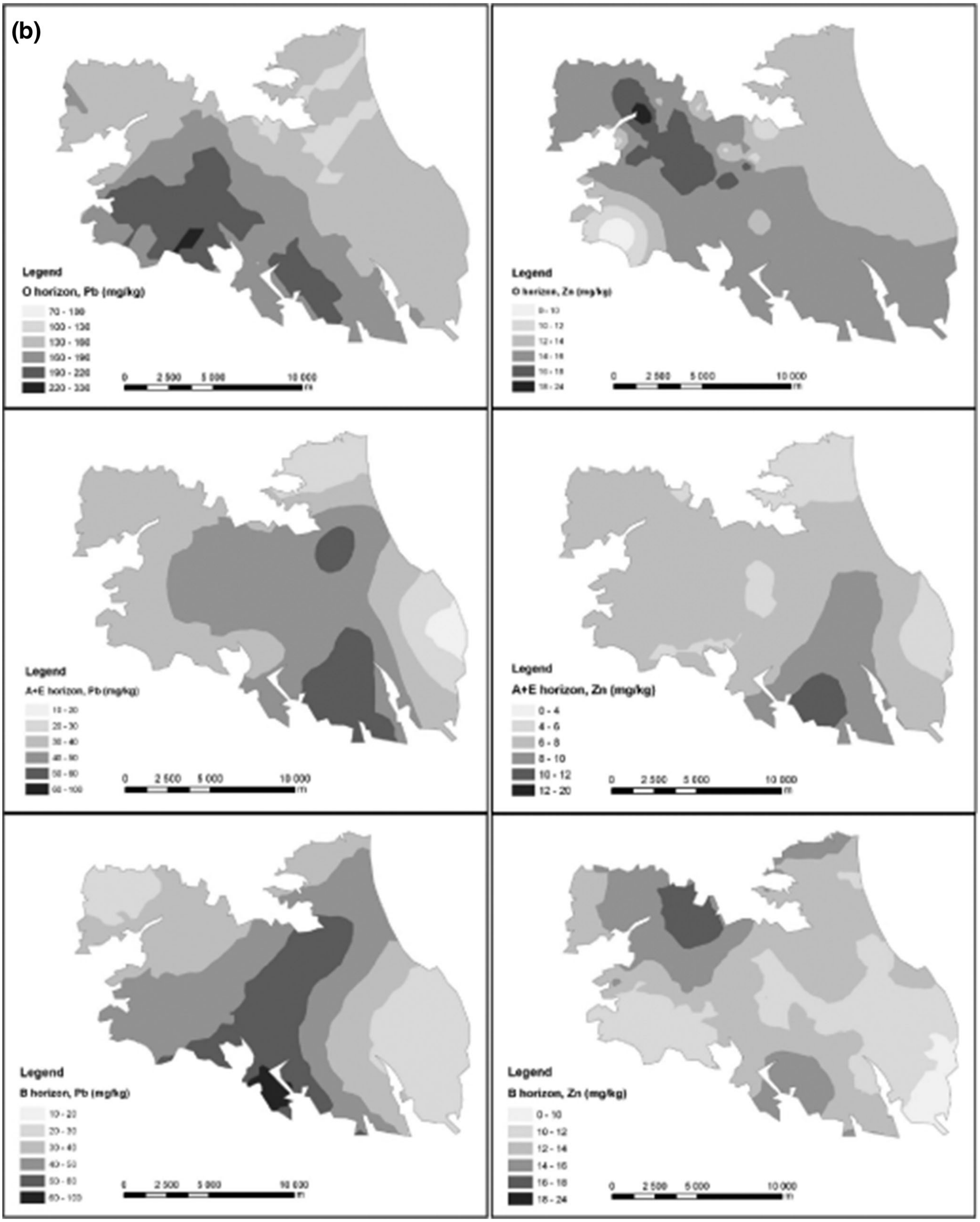


Fig. 2 Interpolated maps of **a** PTE (Cd *left*, Cu *right*), **b** PTE (Pb *left*, Zn *right*) and **c** Mn distribution in different soil horizons of the Jizera Mountains region. **d** Kriging errors for Cd in O, AE and B horizons



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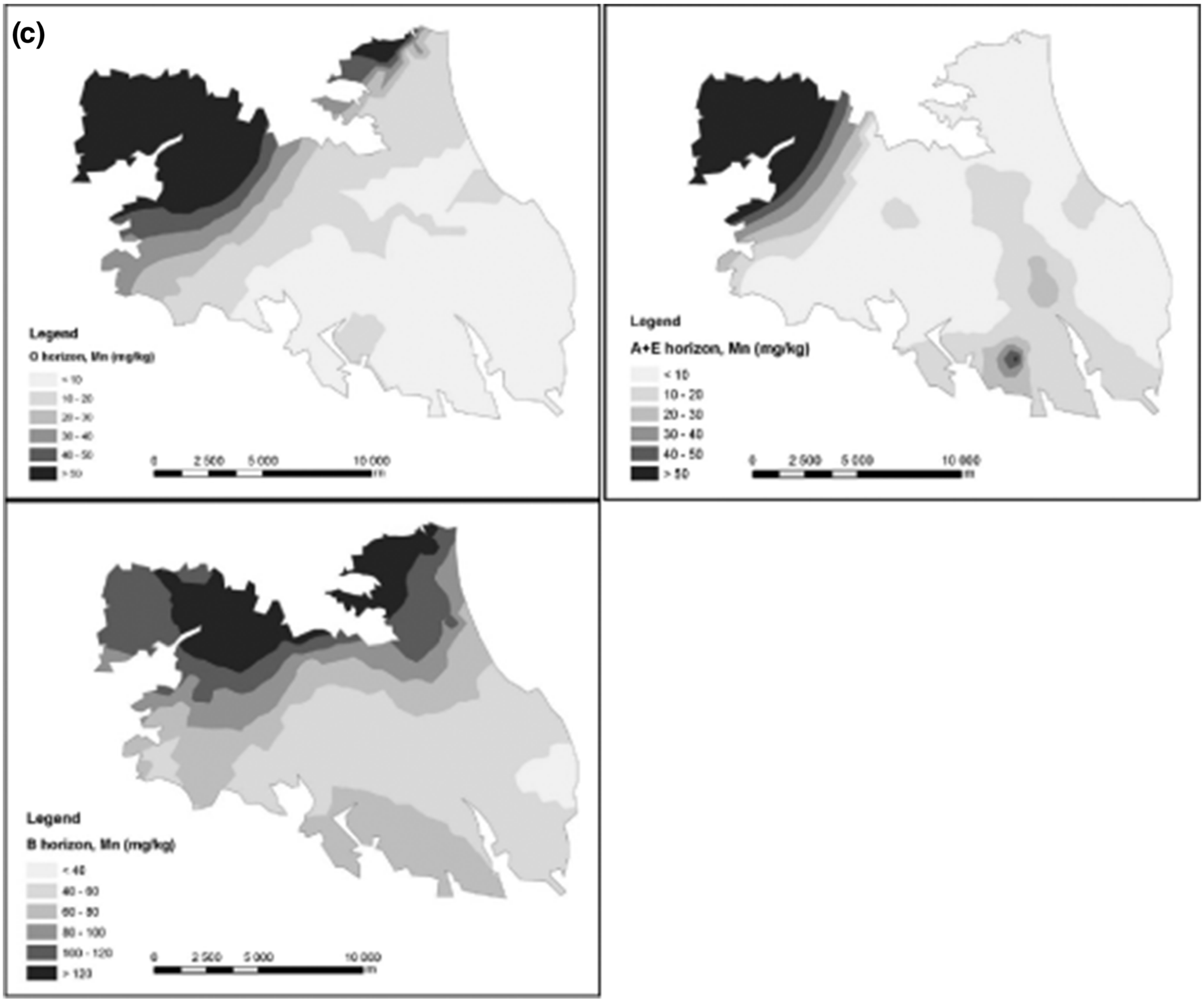

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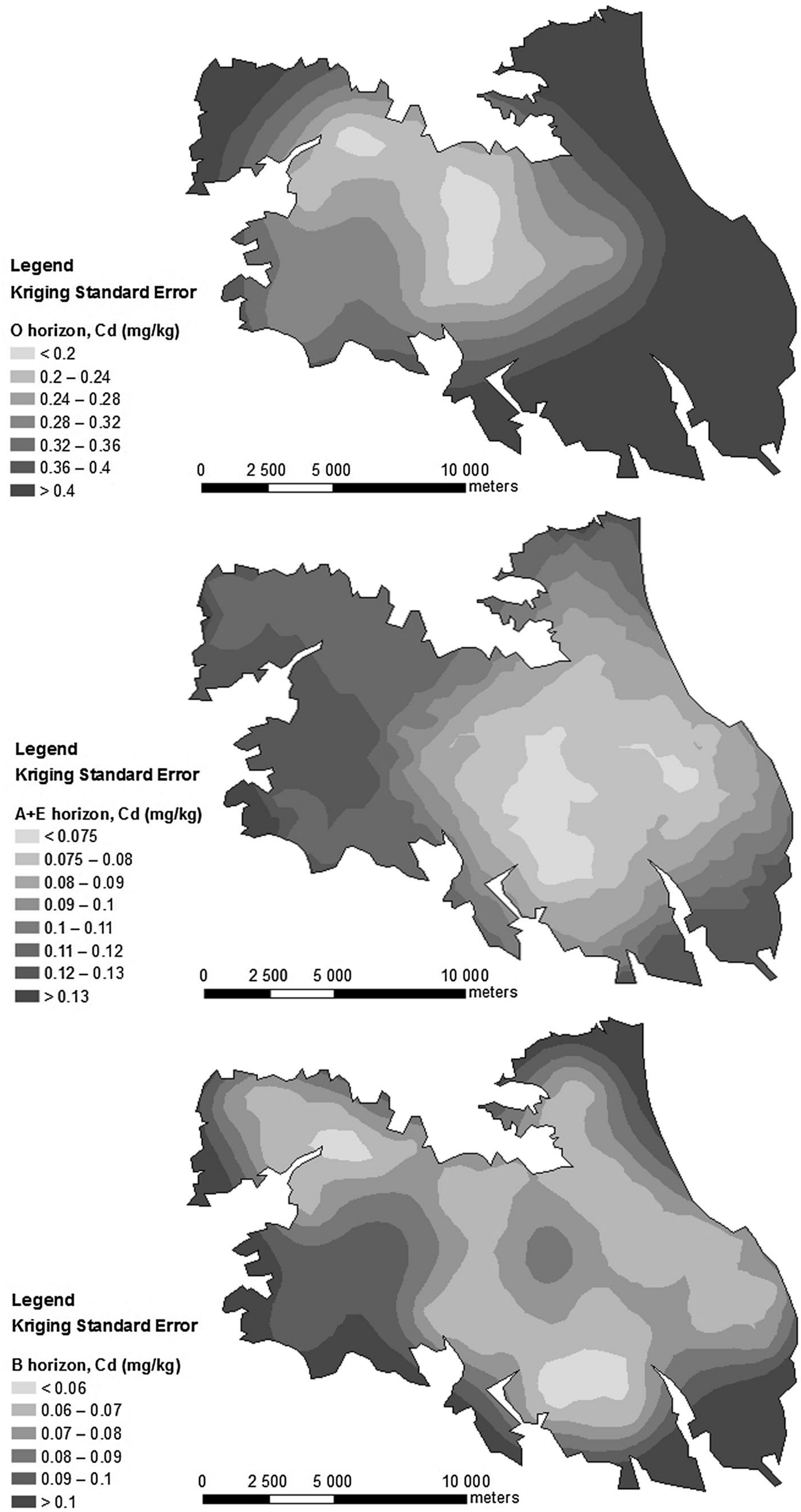
highly variable and, for this reason, were difficult to attribute to other factors such as altitude.

Shparyk and Parpan [23] studied the Ukrainian Carpathians which are characterised by high air pollution caused by emissions from numerous industries. Risk metals were identified as entering the forest system via wet and dry deposition, becoming fixed in soil and accumulating in leaves, needles and mosses. In the Jizera Mountains region, a significant positive correlation between altitude and O horizon Cd concentration was identified. Lead content in the O horizon also increased with altitude, although the trend is not statistically significant and the relationship may be indirect. At higher altitude, there is an increase in precipitation; therefore, there exists a greater input of PTE via wet deposition. The Jizera Mountains region is characterised by overall high annual precipitation (900–1,600 mm); although highest inputs of rainfall occur at the central and central eastern portion of the sampled area (Fig. 1b), this data does not reflect individual peaks of higher altitude. It was noted, previously concerning the map projection, that greater concentrations of PTE in upper horizons generally occurred in the western portion of the study area. Szopka et al. [29] and Suchara and Sucharová [28] made similar observations that the westerly wind is the reason for greatest deposition of atmospheric pollutants on the western side of the mountains which act as a barrier to many airborne pollutants. There are other considerations that can account for varying concentrations with altitude that were not factored into this study, such as proximity to pollution sources, soil pH and slope; therefore, interpretation of data should consider other such variables.

ANOVA statistics showed a significant trend of decreasing soil Cd concentration with decreasing grass cover. Reasons for this occurrence have been proposed by [11]; grass occurs mainly on open areas which occurred as a result of damage to trees, mostly due to acid deposition. As this was the place of most severe deposition, a greater share of PTE occurs in these



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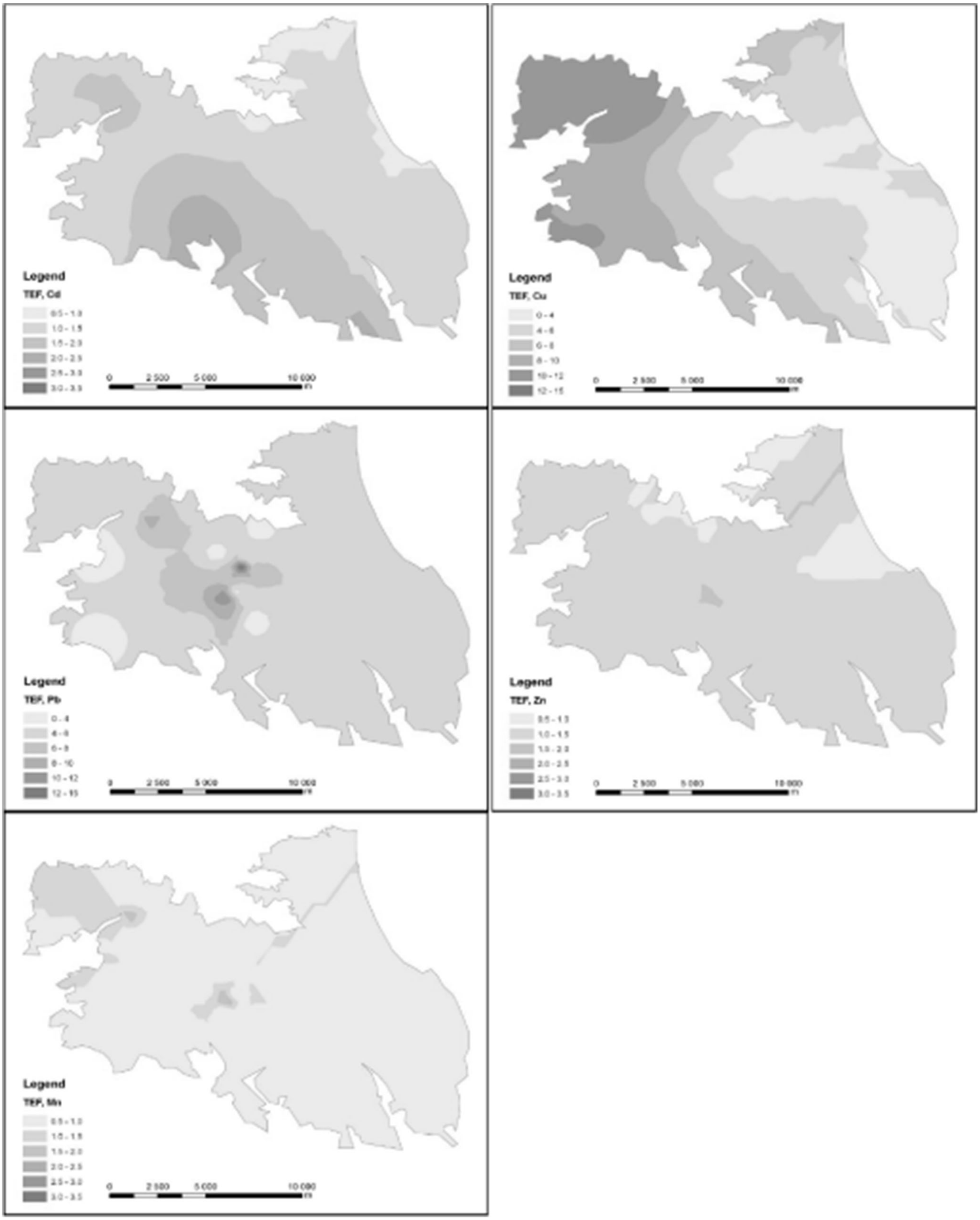

Fig. 3 Top enrichment factor of PTE

zones. Another reason for greater occurrence of PTE in the areas populated by grass can be partly attributed to the fact that these areas were limed in response to the high deposition of acidic compounds [5], which increases the retention of cations at the surface. However, this phenomenon requires further research, as liming could also increase the rate of mineralisation of soil organic matter, consequently reducing the number of sorption sites for PTE in soils. The effect of grass on PTE distribution is principally indirect, as the occurrence of grass only reflects the deposition and consequent management practices that followed. A map was created for tree age data (Fig. 1c). Very young forest is on areas which were afforested in the 1970s and 1980s, the period of highest atmospheric deposition. Lower interception of dry atmospheric deposition during this period can be expected on sites where there were new trees planted at that time; i.e. where the forest is 30–50 years old now. Correlation analysis was made between tree age and PTE content, but no significant trends were shown in any of the horizons. However, there was a significant difference between the two types of monoculture forest, with higher contents of Cd being present under spruce. Likewise, Pb and Cu were found to be more abundant under spruce forest, although this was not statistically proven. Higher PTE accumulation under spruce can be explained by the fact that (1) spruce has a higher specific surface on its needles as opposed to beech and (2) beech is a deciduous tree species. Thus, spruce exhibits increased year-round interception via rain, fog and winter rime and therefore a stronger input of particulate matter to soil. Despite the observed trends, we must consider that there are many variables concerned with such large-scale sampling and more detailed analysis is required to relate stand factors in detail.

5 Conclusions

The aim of the study was to determine the level of forest soil pollution in the Jizera Mountains, an area strongly affected by atmospheric deposition. Compared to AGVs, mean values of PTEs do not appear to be excessive. The only mean value that surpassed the guideline value was lead in the O horizon (two times the guideline limit). TEF proved the anthropogenic origin of cadmium, copper, lead and zinc, whereas manganese appears to be of geologic origin. An analysis of stand factors showed a statistically significant influence of altitude, grass cover and forest type (spruce vs. beech) on cadmium concentration in forest soils. Maps with spatial distribution and TEFs of potentially toxic elements were created. Maps generated for spatial distribution of potentially toxic elements showed a similar pattern for Cd, Cu, Pb and Zn distribution in O horizon soil, which points towards a single source of pollution.

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