Sources of Magnetic Particles from Air Pollution in Mountainous Area

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Abstract

Measurement of magnetic susceptibility of topsoil represents a very useful tool for the detection of atmospherically deposited magnetic particles. The samples of forest soils from the Moravian-Silesian Beskydy Mountains (the Czech Republic) were used for identification of emissions sources of particles with magnetic ordering. Magnetic susceptibility was measured at bulk samples of grain size class under 2 mm. Microanalysis using electron microscope with an energy dispersive X-ray spectrometer was used for the determination of particles in the magnetic fraction. The samples of dust from the sintering plant of the Iron Works in the town of Třinec were analysed. The values of magnetic susceptibility of forest soils in the Třinec region are increased. In the relatively near vicinity of the industrial area, the average value for Javorový Mount was 7.90×10^{-6} m³/kg, for Ostrý Mount it was 6.69×10^{-6} m³/kg. It was proved that they were higher than the average values from the Beskydy Mountains (4.64×10^{-6} m³/kg). The concentrations of lead and magnetic susceptibility in soils showed significant correlation dependence (r_s = 0.85). The iron and steel industry represent the primary source of the pollution load in forest soils of the studied area. Statistically, significant dependences between the organic matter content and the lead and zinc concentrations as well as between the magnetic susceptibility values and the iron concentrations in forest soils were found. It was confirmed that the airborne particles are deposited on vegetation and accumulated in the organic horizon of forest soils.

Keywords: magnetic susceptibility, microanalysis, soil pollution, atmospheric deposition, emissions from metallurgy

Introduction

Magnetic properties of particulate matter (PM) are increasingly used for identification of contamination sources in various environmental materials (Cervi et al., 2014). Dust particles with a relatively large proportion of magnetic particles can be released into the environment by anthropogenic processes (combustion of coal and biomass, metallurgical processes, recycling of solid waste, transport). The concentrations of magnetic particles are increased in the uppermost layers of soils in industrial regions and in the vicinity of industrial sources (Lu et al., 2016). Technogenic magnetic particles (TMP) differ from magnetic particles originated by natural processes mostly by concentrations of risk elements. Particles formed during high-temperature combustion processes have characteristic spherical shape. Particles in emissions from transport and iron metallurgy form irregular non-spherical aggregates (Lu et al., 2016). Magnetic properties of soils are influenced by the mineralogical composition of soils and petrographic type of bedrock. The concentration of magnetic minerals (mostly magnetite) in soils can be expressed more simply as magnetic susceptibility (Magiera et al., 2006a). Magnetic properties of soils are manifestation of magnetite, maghemite and minerals of the spinel group: magnesioferrite (Matysek et al., 2008). The highest concentration of anthropogenic ferromagnetic particles is usually found in humus horizon of forest soils (Kapička et al., 2003). Lead has high concentrations in forest soils in the vicinity of the town of Třinec (37–841 mg/kg, median 211 mg/kg). It was found the statistically significant coefficient of correlation between magnetic susceptibility of forest soils and Pb concentration, r = 0.68 (Matysek et al., 2008). Concentrations of Pb reach values from 30 to 80 mg/kg in humus horizon of forest soils. Soils in relatively high altitudes above sea level contain concentrations of lead comparable with soils in the close neighbourhood of iron works. This trend is observable in the mountainous area south of the town of Třinec (Matysek et al., 2008). The town of Třinec (Figure 1) is located in the eastern part of the Moravian-Silesian Region (the Czech Republic). Metallurgical plant (Iron Works Třinec) situated in the town produces 2000 kilotons of iron and approximately 2600 kilotons of steel per year. It is reflected by the air quality in the region. The aim of this work is the determination of the origin of magnetic particles in forest soils of Třinec region and proving their relationship with high concentrations of Pb in soils.

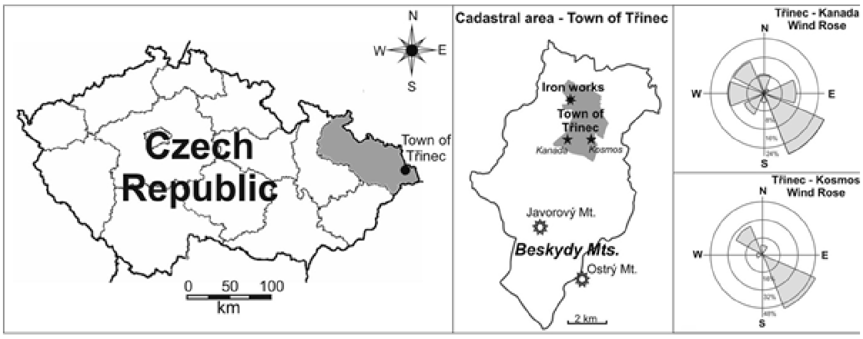
Materials and methods

The study area is located in the vicinity of Třinec iron works: Mount Javorový (1044 metres a.s.l., distance of 7.5 km) and Mount Ostrý (1032 m a.s.l., distance of 10 km).

The sampling of soils and their processing was performed according to the Technical Standard ISO 19258. Thirty soil sampling sites were located at the slopes of Javorový Mt. and Ostrý Mt., samples were taken from the depth of 5 to 10 cm (after removing the detritus - uppermost horizon). The orientation of sampling was selected according to the prevailing directions of wind flowing in the region (NE, NW, SE), the height interval of sampling was 100 m of altitude. Sampling

Fig. 1. The Czech Republic, the Moravian-Silesian Region shaded. Study area in the vicinity of the town of Třinec. Prevailing directions of air flowing

Rys. 1. Republika Czeska, Region Morawsko-Śląski. Miejsce poboru próbek w pobliżu miasta Trzyniec. Dominujące kierunki przepływu powietrza.



Tab. 1. The mean values of mass magnetic susceptibility (×10^{-6} m³/kg) with standard deviations obtained for altitudes from 500 to 1000 m a.s.l

Tab. 1. Średnie wartości podatności magnetycznej (× 10^{-6} m³/kg) dla wysokości od 500 do 1000 m nad poziomem morza.

Locality	500 m a.s.l.	600 m a.s.l.	700 m a.s.l.	800 m a.s.l.	900 m a.s.l.	1000 m a.s.l.	All samples
Javorový	2.48±1.26	9.96±0.90	10.62±6.41	10.46±2.77	5.98±5.29	-	7.90±4.72
Ostrý	-	5.14±2.44	5.96±1.88	4.17±1.73	7.19±4.40	10.97±3.11	6.69±3.45

sites of forest soils at both localities were located mostly in deciduous and mixed forests. Homogenized samples with the grain size of less than 2 mm were used for analyses. The determination of chemical composition of soils – concentrations of risk elements was performed by portable energy dispersive X-ray fluorescence spectrometer Innov-X DELTA Professional (InnovX Systems, U.S.A.). The mass magnetic susceptibility (χ) of the soil samples (fraction with grain size below 2 mm) was measured using the Bartington MS2 instrument and MS2B dual sensor at two frequencies: 0.465 kHz (low frequency) and 4.65 kHz (high frequency). Magnetic particles from soils were separated by hand magnet. The point chemical analysis of particles was performed by scanning electron microscope (SEM) FEI Quanta 650 FEG with energy dispersive analyzer EDAX. Mineralogical phase analysis of dust from Iron Works Třinec was performed by the method of X-ray diffraction (diffractometer Bruker Advance D8). The statistical analysis and the correlation analysis at the 0.5 level of significance were performed using the statistical software OriginPro 8.5.

Results and discussion

The study area (Javorový Mt. and Ostrý Mt.) is located in the area of flysch zone of West Carpathians. Flysch sediments are formed by characteristic intercalations of clay and sand rocks. In the region of Moravian-Silesian Beskydy Mts., volcanic rocks of teschenite association occur with minerals like magnetite and goethite. The mass magnetic susceptibility of flysch rocks (sandstones, siltstones, and shales) is relatively very low, approximately 7×10^{-8} m^3/kg. These values vary widely and can be increased by the presence of siderite or glauconite in the rocks (Matysek et al., 2008). The research conducted by various authors in the regions under the influence of industrial emission indicates the anthropogenic character of the magnetic susceptibility, with the values higher than 60×10^{-8} m^3/kg in the forest topsoil (Chlupáčová et al., 2010). The χ value of soils can vary depending on the region (lithology), emission sources (i.e., power industry, metallurgy) and distance from this emission sources (Łukasik et al., 2016). Accumulation of ferromagnetic mineral phases occurs mostly in the surface layer of forest soils in the O horizon (Strzyszcz and Magiera, 1998). The mean value of mass magnetic susceptibility for the area of Javorový Mt. is 7.90 ± 4.72×10^{-6} m^3/kg and Ostrý Mt. 6.69 ± 3.45×10^{-6} m^3/kg (Table 1). These values are higher than the average value determined in the Moravian-Silesian Beskydy Mts. – 4.64 ± 4.41×10^{-6} m^3/kg (Matysek et al., 2008), and pronouncedly higher than the values of χ for selected mountain areas in Poland, e.g. Lasocki Ridge, eastern Karkonosze Mts. with the average value of 82.0 ± 2.05×10^{-8} m^3/kg, and Jazwina (Sleza Massif) 120.2 ± 96.38×10^{-8} m^3/kg (Magiera et al., 2006b). Table 1 presents the mean values of magnetic susceptibility and standard deviations determined in the altitude from 500 to 1000 m a.s.l. The highest values were found in the area of Javorový Mt. for altitude from 700 to 800 m a.s.l., for Ostrý Mt. in the altitude of 1000 m a.s.l. Figure 2 displays box-plot, which shows the increase of the values of magnetic susceptibility with the increasing altitude, what is in accordance with results published by Matysek et al. (2008) for the area of Moravian-Silesian Beskydy Mts.

High concentrations of lead in soils were found for the area of Javorový Mt. (311 ± 168 mg/kg) and Ostrý Mt. (316 ± 213 mg/kg). Sucharova et al. (2011) report significant variations between the minimal and maximal concentration (19 and 1863 mg/kg) for forest soils in the Czech Republic, with the median value of 78 mg/kg. The maximal concentration of Pb was described particularly from the eastern part of the republic. The high concentrations of Pb in forest soils in the Karkonosze National Park (soil layers 0–10 cm and 10–20 cm) in the ranges 19–248 and 4–196 mg/kg respectively were published by Szopka et al. (2013). From results listed in Table 2 it follows that concentrations of elements in organic horizon of forest soils in the area of Javorový Mt. and Ostrý Mt. are comparable with the exception of manganese. In the area of Ostrý Mt. and Javorový Mt., concentrations of Fe in forest soils are approximately 5× higher than the median value for forest soils in the Czech

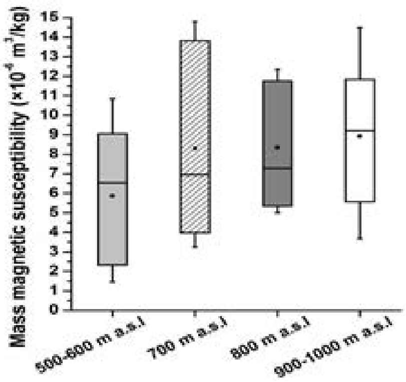


Fig. 2. Box plot – Mass magnetic susceptibility of soils in the altitude from 500 to 1000 m a.s.l

Rys. 2. Podatność magnetyczna gleb na wysokości od 500 do 1000 m npm

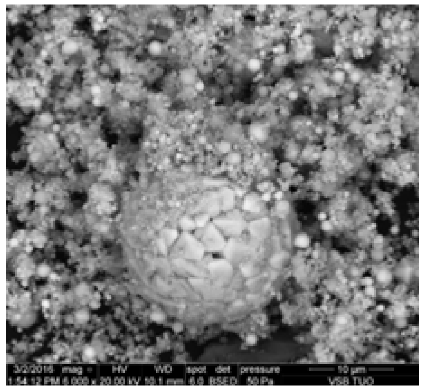


Fig. 3. Electron microphotograph of the spherical particle of magnetite in dust from the steel mill

Rys. 3. Mikrofotografia elektronowa sferycznej cząstki magnetytu w pyle z huty

Tab. 2. Concentrations of elements and content of organic matter in soil samples from the Beskydy Mts. and forest soil from the whole area of the Czech Republic [Explanations: S.D. – Standard deviation, OM – Organic matter, FS – Median of concentrations in organic horizon of forest soils]

Tab. 2. Stężenia pierwiastków i zawartości materii organicznej w próbkach gleby z Beskidów. i gleby leśne z całego obszaru Republiki Czeskiej [Wyjaśnienia: S.D. – Odchylenie standardowe, OM – Materia organiczna, FS – Mediana stężeń w organicznym horyzoncie gleb leśnych]

Locality	Fe	Mn	Zn	Pb	Cr	Cd	Cu	Zr	As	OM	Reference
	(mg/kg)									(wt.%)	
Beskydy Mts. mean	27588	464	178	226	81	-	19	91	30	-	*Matysek et al.,2008*
S.D.	*16351*	*495*	*92*	*101*	*32*	-	*16*	*42*	*12*		
Javorový Mt. mean	38363	660	303	311	71	1.6	58	161	52	67	*This study*
S.D.	*11050*	*344*	*106*	*168*	*17*	*0.44*	*27*	*53*	*22*	*14*	
Ostrý Mt. mean	36313	463	300	316	73	1.2	53	193	50	65	
S.D.	*9677*	*157*	*141*	*213*	*19*	*0.4*	*27*	*63*	*18*	*14*	
FS-Czech Republic	7200	392	63	78	22	0.56	23			81	*Sucharova et al.,2011*

Republic, for Pb 4×, for Cr 3×, and for Mn and Cu two times higher (Sucharova et al., 2011). Relationships between individual elements were studied using Spearman correlation coefficients with respect to identification of contamination sources. Statistically significant correlation coefficient was proved for Fe × Mn, Pb, Zn, As, Cu, Cr in forest soils in the areas of Ostrý Mt. and Javorový Mt. An enrichment factor (EF) was selected for expression of contamination degree of forest soils. It was calculated as a ratio: (concentration of risk element/concentration of Zr) in the sample of analysed soil/(concentration of element/concentration of Zr) in the lithogenic background. The geochemical background was determined as concentrations of elements in topsoil (Salminen, 2014). Zirconium was selected as a typical lithophile element. In the air deposits from the iron works in Třinec, it was present below the limit of detection of the analytical method. The highest value of enrichment factor was found for Pb, Zn, Cu, and As. The enrichment factor in forest soils was higher in the area of Javorový Mt. than in the area of Ostrý Mt. for all studied elements (Fig.4). The highest enrichment factor was determined for soils in both areas of summits of Ostrý Mt. and Javorový Mt. High levels of precipitation are strongly correlated with heavy metal deposition and seem to be the main source of heavy metal deposition at higher altitudes (Zechmeister, 1995). The highest concentrations of Pb in the summit areas are not in accordance with the results of Szopka et al. (2013), who reported concentrations of Pb to be significantly higher in the lowest altitudinal zone (500–750 m a.s.l.) compared to the highest zone (1250–1380 m a.s.l.), what can be explained by seeder-feeder effect and horizontal transport of pollutants.

Correlation analysis proved the statistically significant relationship between magnetic susceptibility and risk elements Cu, Fe, As, Pb, Zn together with organic soil component. At the same time, significant inverse proportionality was determined between magnetic susceptibility and lithogenic elements Si, Al, Ti, and Zr (Table 3). The statistically significant relationships were also found between concentrations of Pb, Zn and the content of the organic component in soils. The correlation coefficient for the content of organic matter in soils and Pb (Javorový Mt.) reached the value $r_s = 0.89^*$. Similarly, for the content of organic matter in soil and Zn, it was $r_s = 0.70^*$. The similar relationship for Pb was reported by Szopka et al (2013). Statistically significant correlation relationships were also found for the summit of Ostrý Mt. between the content of the organic component of soils and Pb ($r_s = 0.80^*$) as well as for Zn $r_s = 0.73^*$. The concentrations of Pb, Zn, and Fe in soils have relationships with the content of organic matter in soils. Metals are primarily caught by vegetation, which is the source of organic matter in humus horizon. The development of humus horizon is influenced by density and character of wood vegetation.

Chemical and mineralogical analyses of dust from the iron works in Třinec were performed in order to determine the origin of magnetic particles in soil samples. The results of mineralogical analysis of crystalline phase are presented in Table 4.

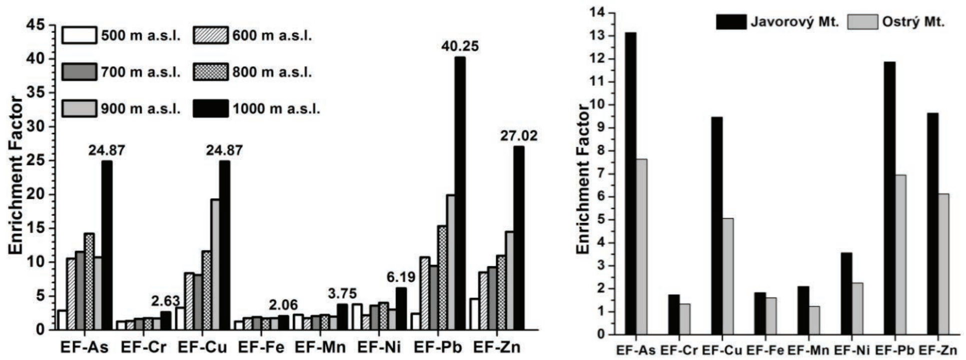


Fig. 4. Left: Relationship between the altitude above sea level and EF. Right: Median of enrichment factor EF for soils from Javorový Mt. and Ostrý Mt.
Rys. 4. Po lewej: Związek między wysokością nad poziomem morza a EF. Po prawej: Mediana współczynnika wzbogacenia EF dla gleb z Javorový Mt. i Ostrý Mt.

Tab. 3. The values of correlation coefficients for mass magnetic susceptibility and risk elements together with organic matter (n=30) for the Beskydy Mts. [Explanations: OM - Organic matter, *statistically significant relationships]
Tab. 3. Wartości współczynników korelacji dla masowej podatności magnetycznej i elementów ryzyka wraz z materią organiczną (n = 30) dla Beskidów. [Objaśnienia: OM – materia organiczna, *relacje istotne statystycznie]

Si	Al	Fe	K	S	Ti	Mn	Cu	Zn	As	Zr	Pb	OM
-0.79*	-0.59*	0.96*	-0.80*	0.81*	-0.73*	0.62*	0.91*	0.90*	0.93*	-0.73*	0.85*	0.71*

Tab. 4. The main mineral phases in dust from the iron works Třinec (wt.%) [Explanations of dust origin: AE – sintering plant, electrostatic separators. AB – sintering plant, bag filters. B – steel mill. C – secondary dust removal of oxygen-convertor steel mill. D – desulphurization of pig iron.]
Tab. 4. Główne fazy mineralne w pyłach z Huty Żelaza Třinec (% wag.) [Objaśnienia pochodzenia pyłu: AE – spiekalnia, separatory elektrostatyczne. AB – spiekalnia, filtry workowe. B – huta stali. C – wtórne usuwanie pyłu w stalowni konwertorowej. D – odsiarczanie surówki.]

Mineral	AE	AB	B	C	D
Sylvite - KCl	26.33	27.28			
Halite - NaCl	11.54	20.89			
Wuestite - FeO			3.34	0.45	
Magnetite - Fe_3O_4	19.66	1.87	31.1	49.26	42.19
Hematite - Fe_2O_3	23.07		2.11	29.81	18.85
Laurionite - PbCl(OH)	1.65				
Laurelite - $Pb_7F_{12}Cl_2$	0.98				
Penfieldite - $Pb_2Cl_3(OH)$	1.35				
Litharge - PbO		0.54			
Portlandite - $Ca(OH)_2$		32.11			
CaOHCl		5.34			
Calcite - $CaCO_3$		7.57			
Zincite - ZnO				3.77	
Cr-Mn-Fe spinel			53.1		
Gehlenite - $Ca_2Al(Si,Al)_2O_7$				4.88	4.64
Graphite - C			1.92	5.59	11.52
Magnetic susceptibility $\times 10^{-6}$ m^3/kg	7.78	1.45	18.18	1.81	9.47

Iron oxides (magnetite and hematite) form an essential part of dust AE, B, C, and D, where their average content is 55% of the crystalline phase. An example of spherical magnetite particle is in Figure 3. Chlorides (NaCl and KCl) are also important, and they can form up to 48% of the crystalline phase. In dust from the sintering plant, lead minerals were identified (laurelite, laurionite, and penfieldite), chlorides and hydroxichlorides of lead. In dust from oxygen-convertor steel mill, zincite (ZnO) was identified. Important mineral in dust from steel mill is Cr-Mn-Fe spinel. The average chemical composition of particles from dust determined by SEM+EDAX is presented in Table 5.

Dust AE contained particles with high Pb concentrations in the range from 4.1 to 39.1%. Dust B from steel mill has mostly Fe-Cr-Mn spinel as a major component in the crystalline phase. Dust C from oxygen-convertor steel mill contains relatively larger particles of size around 5 μm, with 1.96 to 3.42% of Zn. These particles are probably formed by hematite which contains 69.94% of iron and 30.06% of oxygen. Particles with relatively higher content of Zn (10–15%) are formed by magnetite. The high concentration of Zn in magnetite is possible due to the substitution of Fe2+and Zn2+ with very similar ion radii in magnetite ($Fe^{2+}Fe_2^{3+}O_4$), where Zn has ion radius of size 0.060 and Fe^{2+} 0.063 nm (Hansson et al., 2004). Iron oxide particles from dust D contain only minimal amounts of Zn admixture (0.93–3.36%), in less than 50% of analysed particles. From results of chemical composition of particles it follows that for particles from ore sintering plant (AE) there is typical element association of Fe × Pb, for particles from steel mill is typical association of Fe × Cr

Tab. 5. Chemical analyses of particles (average of 15 particles) by SEM +EDAX (wt.%)
Tab. 5. Analizy chemiczne cząstek (średnio 15 cząstek) metodą SEM + EDAX (% wag.)

	Fe	Mn	Cr	Zn	Pb	Cu	Si	O	Al	Mg	Ca	K	Na	Cl
AE	15.11		0.15		11.73	2.13	4.27	7.29	0.99	0.94	2.51	15.53	7.69	30.86
AB	0.51				6.60	1.38		11.97	0.67	2.54	31.57	8.47	4.39	25.26
B	37.56	12.79	5.74				4.03	29.12		5.52	6.54	0.48		
C	58.36	1.34		9.31			0.71	20.83	0.52	0.41	1.52	0.53		
D	50.57			2.25				21.06	2.78		6.05			

× Pb, and for particles from desulphurization of pig iron (D) as well as from oxygen-convertor steel mill it is association of Fe × Zn. Analysis of particles from dust and mineralogical analysis of dust confirmed that lead-bearing phases form separate minerals.

Conclusions

Mass magnetic susceptibility provides the whole series of statistically significant linear correlations with Fe, Zn, Pb, Cu, and As. Metals in forest soils are bound to iron oxide particles and the organic component of soils. Metals in forest soils in the areas of Javorový Mt. and Ostrý Mt. can be arranged according to the decreasing concentrations in the following way: Fe > Mn > Zn > Pb > Cr > Cu > As > Cd. Lead is neither part of the structure of magnetite nor minerals of the spinel group. It forms in dust from the iron works separate minerals. Together with magnetite, it forms mixed crystalline aggregates. Magnetic susceptibility has the statistically significant relationship with the content of the organic component in soils ($r_s = 0.71$*) which documents catching of these particles by vegetation and their subsequent deposition into humus horizon. From the value of correlation coefficient ($r_s = 0.85$) between magnetic susceptibility and concentration of Pb in soils, it follows that emissions from the sintering plant represent the main source of lead. The enrichment factor proved increased deposition load of forest soils (Pb, Zn) in the areas with the higher altitude above sea level. Lead had the highest enrichment factor.

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Źródła cząstek magnetycznych z zanieczyszczeń powietrza w obszarach górskich

Pomiar podatności magnetycznej wierzchniej warstwy gleby stanowi bardzo przydatne narzędzie do wykrywania osadzonych z atmosfery cząstek magnetycznych. Próbki gleb leśnych z Beskidu Morawsko-Śląskiego (Czechy) wykorzystano do identyfikacji źródeł emisji cząstek o właściwościach magnetycznych. Podatność magnetyczną mierzono w próbkach zbiorczych w klasie ziarnowej poniżej 2 mm. Mikroanaliza z użyciem mikroskopu elektronowego ze spektrometrem dyspersyjnym energii rentgenowskiej została użyta do oznaczenia frakcji magnetycznej cząstek. Przeanalizowano próbki pyłu ze spiekalni Huty Żelaza w Trzyńcu. Stwierdzono wzrost wartość podatności magnetycznej gleb leśnych w regionie Trzyńca. W stosunkowo bliskim sąsiedztwie obszaru przemysłowego Javorový Mount średnia wartość wynosiła $7{,}90 \times 10^{-6}$ m³/kg, w przypadku Ostrý Mount $6{,}69 \times 10^{-6}$ m³/kg. Wykazano, że były one wyższe niż średnie wartości dla Beskidów ($4{,}64 \times 10^{-6}$ m³/kg). Zawartość ołowiu i podatność magnetyczna w glebach wykazały istotną korelację ($r_s = 0{,}85$). Hutnictwo żelaza i stali stanowi główne źródło ładunku zanieczyszczeń w glebach leśnych badanego obszaru. Stwierdzono statystycznie istotne zależności między zawartością materii organicznej a stężeniem ołowiu i cynku, a także między wartościami podatności magnetycznej a stężeniami żelaza w glebach leśnych. Potwierdzono, że zawieszone w powietrzu cząstki osadzają się na roślinności i gromadzą się w organicznym horyzoncie gleb leśnych.

Słowa kluczowe: podatność magnetyczna, mikroanaliza, zanieczyszczenie gleby, osadzanie atmosferyczne, emisje z hutnictwa