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ORIGINAL PAPER

Assessment of the air quality in an industrial zone using active moss biomonitoring

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Abstract
The study aimed to evaluate the level of air contamination in the area around the Kosogorsky industrial enterprise in Tula, Russia, in the winter of 2021/2022. For the study, *Pleurozium schreberi*, *Sphagnum fallax*, and *Dicranum polysetum* mosses were used for the first time in the 3-month active biomonitoring (moss-bag technique). Heavy metals elements (Mn, Fe, Ni, Cu, Zn, Cd, and Pb) were determined using atomic absorption spectrometry. In addition, mercury concentrations were determined with an AMA254 apparatus. The study's results for Fe, Cu, Zn, Pb, and Hg confirm the statistical significance of the species' effect on the accumulation of these elements. Values of relative accumulation factor (RAF) > 1.00 at selected measurement points indicate that the site is contaminated as a result of industrial activities (mainly Mn and Fe). Wind direction influenced moss contamination at selected measurement points, which was confirmed by cluster analysis. In the future, long-term or different seasons biomonitoring studies should be conducted in this area.

Keywords Air pollution · Heavy metals · Metallurgical plant · Moss-bag · Vitality

Introduction

Studies using mosses as biomonitors of air quality contribute to the indication of trends and changes in pollution over the years and the identification of sources of emissions of elements accumulated by mosses (Godzik 2020; Kapusta and Godzik 2020). Although Poland and its neighboring Central European countries are the most polluted areas in Europe (Godzik 2020), the influence of industry and the increasing share of pollution from urban areas still dominate (Doan Phan et al. 2019; Kapusta and Godzik 2020). This is because moss's chemical composition is specific to land use, and moss uptake increases from agricultural to industrial sites (Di Palma et al. 2017). The period of moss uptake is also essential, as changes in accumulation are observed from season to season (Hussain and Hoque 2022); the effect of the moss collection site (e.g., tree canopy) on metal deposition (Kondo et al. 2022) or selecting the most suitable species to assess elemental deposition at a given site (Cowden and Aherne 2019a; Tien et al. 2020). Long-term studies under the ICP Vegetation program have confirmed the effectiveness of using mosses for biomonitoring in assessing atmospheric deposition of metals. Changes in the concentrations of heavy metals accumulated by these plants can be recorded over the years (Hristozova et al. 2020). Moss is a good passive sampler for airborne contaminants and can provide valuable information on chemical signatures and deposition of metals (Mudge et al. 2019). Moss biomonitoring can be used to efficiently evaluate regional patterns in atmospheric metal deposition associated with industrial emissions sources (Cowden and Aherne 2019b). For example, in two metropolitan areas in the western province of Sri Lanka using the moss biomonitoring method, selected elements were determined: Zn, Cu, Pb, Ni, and Cr, whose origin is traced to petrochemical applications operating in the study area, with mainly Ni and Cr coming from the local petrochemical industry operating around the moss sampling site (Jayalath et al. 2021). Based on metal concentrations in mosses, *Polytrichum commune* and *Pleurozium schreberi,* the impacts of emitters such as a chlor-alkali plant, a glass smelter, two power plants, and a ceramic and porcelain factory were assessed (Zawadzki et al. 2016). Neutron activation analysis and atomic absorption spectrometry allowed the determination of more than 30 elements in samples of *P.*

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schreberi mosses collected in Central Russia, in which the primary sources of air pollution were identified by factor analysis, which included: industry, oil refinery companies, and thermal power plants (Vergel et al. 2020). Research conducted in a neighboring region (the Moscow region), has revealed that the primary contributors to air pollution in the region are industrial activity, thermal power plants, and transportation (Vergel et al. 2019). Additionally, another study has observed a positive trend toward a reduction in contamination in mosses, which is attributed to the COVID-19 pandemic (Yushin et al. 2020; Vergel et al. 2022).

The method of active biomonitoring is characterized by such advantages as an accurate collection of spatial–temporal data or the simplicity and cost-effectiveness of this method in wide environmental monitoring (Limo et al. 2018). It allows a survey of any site of interest, is independent of species presence/absence, and allows control of some factors, which reduces measurement variability and facilitates data interpretation (Catteau et al. 2022). In active biomonitoring studies, it is imperative to standardize a set of factors that are subject to variation across studies, including but not limited to the location of the exposed samples, the impact of the shape of the biomonitor bag, and the position of the bag (Rogova et al. 2018; García-Seoane et al. 2019; Morales-Casa et al. 2019). Nevertheless, as a result of standardization, this method provides more and more new possibilities and approaches for its use to monitor environmental pollution (Sorrentino et al. 2021). Using the moss-bag method with the moss *Sphagnum junghuhnianum*, metal concentrations in mosses were correlated with traffic intensity (Hu et al. 2018). In contrast, short-term exposure to mosses of the genus *Sphagnum* allowed fast screening for the occurrence of methylated volatile arsines or the predominance of particulate As (Arndt and Planer-Friedrich 2018). For the pilot study in Azerbaijan, elevated elemental concentrations obtained as a result of anthropogenic influences were determined at sites that are characterized by cement production, paint production, and oil and gas production (Madadzada et al. 2019). The moss-bag technique is also used in urban areas (Benítez et al. 2021; Ilieva-Makulec et al. 2021), where the moss exposure successfully allows point identification of pollutants as well as characteristic emitters (Ilieva-Makulec et al. 2021). For the Russian study, moss-bag techniques with the moss *Sphagnum girgensohnii* were used to study the vertical distribution of elements in different types of street canyons in Belgrade and Moscow for 10 weeks of exposure. The results showed that elemental accumulation was higher in deep and regular street canyons compared to alley-type canyons (Goryainova et al. 2016). The same moss species was used to assess air pollution in the Park of Moscow. After a 3-month exposure at the state museum-reserve Tsaritsyno, the study showed an accumulation of most of the 35 analyzed elements, which confirms the good properties for use in assessing urban pollution of this moss species (Shvetsova et al. 2019). Biomonitoring in Russia is overwhelmingly dominated by studies of heavy metal and radionuclide deposition, which have a history of more than 40 years and are also still continuing in other countries within the framework of the UNECE ICP Vegetation program (Frontasyeva 2020; Qarri et al. 2023).

The main purpose of the study is to assess the level of air pollution around one of the industrial enterprises in the Tula region, the Russian Federation (RF). The mines of the Moscow Coal Basin are located in the Tula region (Kachurin et al. 2018). A study of trace elements in the soils of the Tula region showed their increased concentration in the soils of cities with industrial facilities (Arlyapov et al. 2015). In Tula's industrial center, the density of dust deposition, the flux of trace metal aerosols, exceeds the regional background many times (Zolotareva 2003). Recently, there has been a decrease in the frequency of the Moscow–Tula area on the blacklists of the Russian Federal Service for Hydrometeorology and Environmental Monitoring (Klyuev 2019). So far, only passive biomonitoring studies using mosses have been conducted in the region. In the first article, for the first time, the moss biomonitoring technique has been applied to air pollution monitoring in Central Russia (Tula region). The results of the study identified eight factors from the elements studied, which were classified according to their primary source through factor analysis (Ermakova et al. 2004). In the second case, research in the region resulted in the recommendation of moss species suitable for biomonitoring as well as the identification of air pollution as activities of the metallurgical, engineering, and chemical industries. These concentrations were several times higher than in other countries (Gorelova et al. 2016b). Therefore, the use of three moss species, *Pleurozium schreberi, Sphagnum fallax*, and *Dicranum polysetum,* was undertaken: in the assessment of atmospheric aerosol pollution by selected elements around the metallurgical plant in Tula and the effect of the winter period on heavy metal concentrations during 90 days of active biomonitoring exposure.

The research was carried out between November 2021 and February 2022 around Kosogorsky metallurgical plant (Tula region, Russian Federation).

Materials and methods

Exposure of three moss species *Pleurozium schreberi* (*Pl*), *Sphagnum fallax* (*Sp*), and *Dicranum polysetum* (*Dp*) as part of active biomonitoring for 3 months during winter (November 2021–February 2022) around Kosogorsky metallurgical plant (Tula, RF). Active biomonitoring studies using mosses have not previously been conducted for this area. The species selected by the authors are commonly used in biomonitoring studies (Benítez et al. 2021; Bertrim and Aherne 2023). The sample exposure site was chosen due to previously unstudied air pollution using the moss-bag technique. In general, biomonitoring studies have been successfully conducted on industrial sites (Culicov et al. 2016; Sergeeva et al. 2021b). Winter was selected for the study because of the higher elemental concentrations reported in the literature at this time compared to the others (Cortis et al. 2016) and because of the influence of the heating season (Rogova et al. 2021; Urošević et al. 2022) or temperature inversion (Barandovski et al. 2012). Moss samples were collected and prepared before exposure following the international guideline ICP Vegetation (ICP Vegetation 2020) at the University of Opole. Before exposure in active biomonitoring, the preparation method affects the initial elemental concentration in mosses, so they were prepared before exposure by an earlier study (Świsłowski et al. 2021). Next, 3 g of mosses were each packed into nylon nets, shipped to Russia, and there exposed in bags. The control sample was not exposed and was left for pre-exposure analyses of element concentrations to assess background pollution. Mosses were hung at a height of about 2.00 m from the ground, and samples were located at seven sites with three bags of each species in every site (nine bags in one site), in general, 63 samples throughout the study area. The total number of samples used in the study is $n = 105$; five subsamples were used from three bags from a given site: 5 subsamples × 7 measurement sites × 3 moss species = 105 samples.

Table 1 The instrumental detection limits (IDL) and instrumental quantification limits (IQL) for the iCE 3500 (mg/L) spectrometer (Thermo Fisher Scientific Inc. 2011)

Element	IDL	IQL
Mn	0.0016	0.020
Fe	0.0043	0.050
Ni	0.0043	0.050
Cu	0.0045	0.033
Zn	0.0033	0.010
Cd	0.0028	0.013
Pb	0.0130	0.070

Table 2 Comparison of measured and certified concentrations in BCR-482 *lichen* (Rajfur et al. 2018)

	BCR-482 *lichen*		AAS ($n = 5$)		*Dev.***
Element	Concentration	Measurement uncertainty	Average	±*SD** of the concentrations	
	[mg/kg d.m.]				[%]
Mn	33.0	0.50	31.7	0.68	−3.90
Fe	804	160	771	154	−4.10
Ni	2.47	0.07	2.16	0.32	−13.0
Cu	7.03	0.19	6.63	0.17	−5.70
Zn	100.6	2.20	95.1	2.30	−5.50
Cd	0.56	0.02	0.53	0.03	−5.30
Pb	40.9	1.40	38.2	1.00	−6.60

*standard deviation

**relative difference between the measured (c_m) and certified (c_c) concentration 100% (c_m-c_c)/c_c

Exposure area

Kosogorsky metallurgical plant (KMP, Tula) is the largest iron works plant in the Tula region and one of the oldest in Central Russia. The plant specializes in producing cast iron foundry, high-carbon ferromanganese, steel castings, pipes, tiles cast iron, spare parts for drilling equipment, and building materials. Consequently, the main pollutants in industrial emissions are iron (Fe), lead (Pb), zinc (Zn), copper (Cu), cadmium (Cd), and sulfur oxide. In the close vicinity to the plant (400 m from KMP), the concentration of manganese (Mn) and cadmium (Cd) in soil exceeds those in the control plot by factors of 16.3 and 92, respectively. The factors for other metals range from 1.6 iron (Fe) to 5.7 lead (Pb) (Gongalsky et al. 2007).

After 3 months of exposure, each moss sample, with a mass of 1.000 ± 0.001-g dry mass (d.m.), was mineralized in a mixture of nitric acid and hydrogen peroxide using a microwave oven (Berghof Company, DE). The mineralization process was carried out at the temperature of 180 °C. Manganese (Mn), iron (Fe), nickel (Ni), copper (Cu), zinc (Zn), cadmium (Cd), and lead (Pb) were determined using an atomic absorption flame spectrometer type iCE 3500 (Thermo Scientific, USA). Concentrations of metals were determined in solution after mineralization filtration and were diluted into volumetric flasks of 25 cm^3. Concentrations of analytes were determined in three analytical replicates. Calibration of the spectrometer was performed with standard solutions (ANALYTIKA Ltd., CZ). The values of the highest concentrations of the models used for calibration (2.00 mg/dm^3 for Cd, 5.00 mg/dm^3 for Ni, Cu, Zn, and Pb, 7.50 mg/dm^3 for Mn, and 10.0 mg/dm^3 for Fe) were approved as linear limits to signal dependence on concentration. The concentration of Hg in the moss samples (0.04 g ± 0.001 g d.m.) was determined with AMA 254 mercury analyzer (Altec Ltd., CZ). Table 1 presents the instrumental detection limits (*IDL*) and instrumental quantification limits (IQL) of the iCE 3500 spectrometer. Table 2 shows the concentrations of heavy metals in certified reference materials BCR-482 *lichen*, produced at the Institute for Reference Materials and Measurements, Belgium.

For mercury, the instrument's limits of detection (IDL) and quantification (IQL) are 0.003 ng (0.03 µg Hg/dm^3) and 0.01 ng (0.1 µg Hg/dm^3) in the sample, respectively. In order to calibrate the apparatus, standards from ANALYTIKA Ltd. (CZ) were used.

Basic descriptive statistical measures minimum, maximum, and median mean with standard deviation (*SD*) and coefficient of variation (*CV*) for the concentrations of determined elements in moss were calculated (De Agostini et al. 2020; Vergel et al. 2022). The RAF—relative accumulation factor was used to determine increases in concentrations of the analytes in the exposed mosses samples (Zinicovscaia et al. 2018). Statistica (ver. 13.3) and Microsoft Excel 2016 software were used to process and present the data. Shapiro–Wilk's test was used to check data normality. Student's *t*-test and Mann–Whitney *U*-test were used to assess the significance of differences in elemental concentrations between moss species.

Results and discussion

As a first step in the analysis of the results, differences in the significance of heavy metal concentrations in different species of mosses were evaluated regardless of the site of exposure— the results are shown in Table 3.

Table 3 Statistical significance between species in relation to the elements analyzed

Student's *t*-test				
Element	Species comparison	*t*	df	*p*
Zn	Pl vs Sp	24.4	63	**0.000**
	Pl vs Dp	12.2	64	**0.000**
	Sp vs Dp	−11.6	63	**0.000**
Cd	Pl vs Sp	−0.159	24	0.875
	Pl vs Dp	−0.795	23	0.435
	Sp vs Dp	−0.631	25	0.534
Mann–Whitney U-test				
Element	Species comparison	*U*	*Z*	*p*
Mn	Pl vs Sp	482	0.597	0.550
	Pl vs Dp	527	0.224	0.822
	Sp vs Dp	495	−0.426	0.670
Fe	Pl vs Sp	320	2.72	**0.006**
	Pl vs Dp	380	−2.11	**0.035**
	Sp vs Dp	238	−3.80	**0.000**
Cu	Pl vs Sp	3.00	6.88	**0.000**
	Pl vs Dp	515	0.378	0.705
	Sp vs Dp	5.00	−6.86	**0.000**
Pb	Pl vs Sp	346	2.06	**0.039**
	Pl vs Dp	483	−0.172	0.864
	Sp vs Dp	335	−2.38	**0.017**
Hg	Pl vs Sp	85.0	5.81	**0.000**
	Pl vs Dp	189	−4.55	**0.000**
	Sp vs Dp	7.00	−6.83	**0.000**
Ni	Pl vs Sp	75.0	2.38	**0.017**
	Pl vs Dp	109	1.22	0.221
	Sp vs Dp	122	−0.758	0.449

p values in bold are statistically significant

The results of statistical tests showed that only the accumulation of elements: Cd and Mn was not significant for the exposed moss species in the study area. For the other metals, differences between the species used in the exposure were significant. The distribution of elemental concentrations along with basic statistical parameters is shown in Fig. 1 and Table 1 in Supplementary Information (SI), respectively. In the next step, the determined *relative accumulation factor* (RAF) values were analyzed as shown in Fig. 2.

Figure 2 shows the RAFs of concentrations of two selected elements, manganese and iron, for which the most and largest increases were determined at all measurement points (other elements are included in Table 2 SI). RAF values also show that there was a significant increase in nickel values above RAF = 1.00 at points 2 and 6 for *P. schreberi* and at point 2 for *D. polysetum*. On the other hand, slight increments: RAF > 0.500 were determined at measurement point 1 for *P. schreberi* and *S. fallax*. Slight increments were also determined for nickel in *S. fallax*. Considering the RAF values, the mosses can be ranked Sp > Pl > Dp in terms of the frequency of significant metal increments in the mosses during exposure. Cluster analysis between metal concentration with species on seven sites reveals clusters with varying linkage distances. The results obtained by mosses are significantly different in different places. The results indicate a different relationship between measuring points (Fig. 3).

It is possible to distinguish two main groups of points, which occur regardless of the species of moss and element. This indicates that the type of pollutant is closely related to the particular measurement site (Fig. 3). These data are consistent with the previously discussed results on the increments of metal concentrations at individual measurement points, as well as with the prevailing wind rose at the study site. The prevailing winds at the site are W, WSW, and SW (Meteoblue 2022), which, with the emissions of local industry, affect the pollution of the mosses exposed at points 1, 2, and 6 (compare with the map of Fig. 1). Sources of pollution at points 3, 5, 4, and 7 may be diffuse low emissions and traffic.

As mentioned in the Introduction, the moss-bag technique finds its application in the assessment of air pollution in urban areas (Culicov et al. 2016), but monitoring in different seasons and regions should be taken into account (Mao et al. 2022). For moss transplants, *Hylocomium splendens* significant correlations between element concentrations in mosses and bulk deposition (Cd, Ni, Pb, and Zn) were found for industrial sites (Kosior et al. 2018). The literature indicates that it is Cd, Ni, and Pb contamination that is typical for industrial sites (Varela et al. 2018). So far, the Tula site has only been surveyed by passive biomonitoring using different moss species. The site has been contaminated by elements

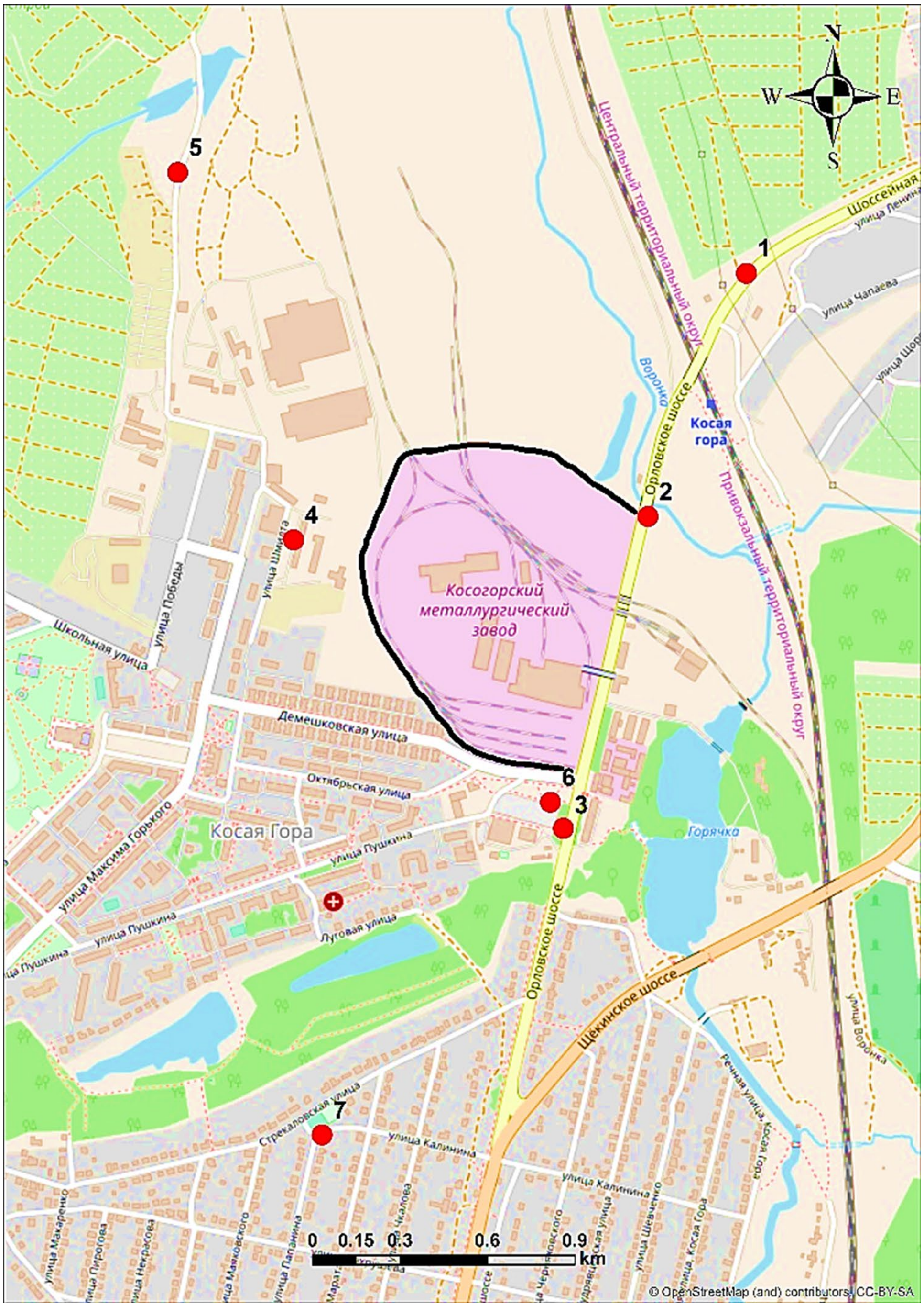

Fig. 1 Locations of measuring points; the red dots indicate moss exposure sites, and the black line marks the area of the Kosogorsky metallurgical plant. 1—Pervomayskiy Shosseynaya Street [1 km from KMP]; 2—Orlovskoye road, a point on the Voronka river bridge [0,5 km from KMP]; 3—green areas/park at the textile house [0,2 km from KMP]; 4—green area next to an industrial equipment company/machinery park [0,5 km from KMP]; 5—forest area [1,2 km from KMP]; 6—green areas/park at the textile house [0,1 km from KMP]; and 7—residential area/playground [1,0 km from KMP]

of natural origin (e.g., Zn) from obvious anthropogenic sources: the iron–vanadium industry (i.e., Fe) (Shvetsova et al. 2019) and other industries and general urban activities (e.g., Mn, Zn, Cu, and Pb) (Ermakova et al. 2004). The same elements were determined in soil and invertebrates in the vicinity of a metallurgic factory near Tula. Most metal contamination was confined to the immediate surroundings and did not extend beyond a few kilometers (Straalen et al. 2001). All these elements were determined in this study as part of active moss biomonitoring. In the second publication, the concentrations of some elements have increased over the decade, and their labeled concentrations are dangerous to the health and even life of people living in the area (Gorelova et al. 2016a). Three-month experiment is a too short time, performed only during the winter, to come to such conclusions. However, the impact of winter and home heating on air pollution should also be emphasized. Studies conducted in Serbia indicate similar levels of pollution by selected elements (Vuković et al. 2015). However, the determination of selected heavy metals in all moss species indicates air

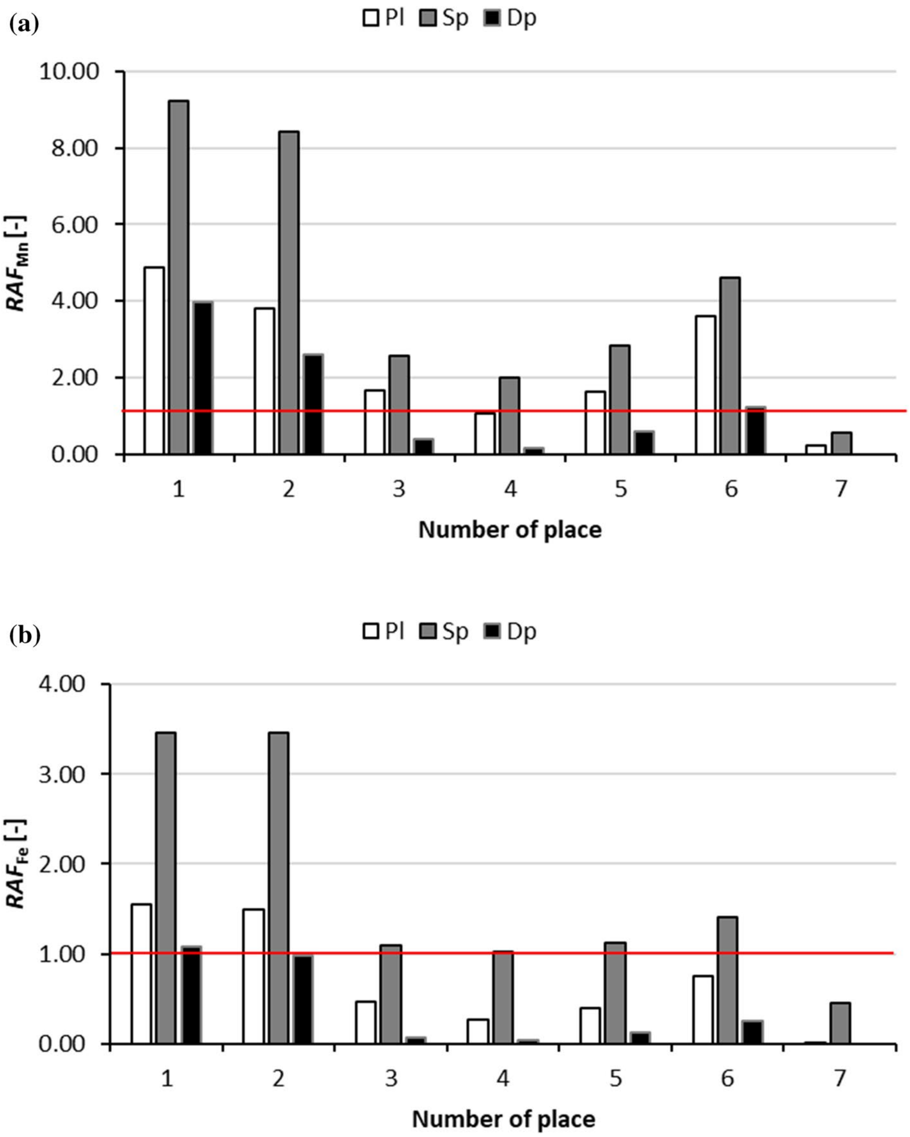

Fig. 2 RAF values for **a** manganese and **b** iron at 7 measurement points in the analyzed species; Pl—*Pleurozium schreberi*, Sp—*Sphagnum fallax*, and Dp—*Dicranum polysetum*. The red line shows the value of RAF = 1.00, indicating a significant increase in analyte above this value

pollution due to active human activities in the area. Due to the location of the measurement site in the immediate vicinity, the area studied is Moscow, about 200 km from Tula. Ten-week exposure to *Sphagnum girgensohnii* made it possible to assess the pollution, where the mosses accumulated the most pollution in open space with traffic, which was also attributed as the source of enrichment of the mosses in a particular elements (Goryainova et al. 2016). A 3-month study in the same city (but in the park of the state museum reserve "Tsaritsyno") with the same indicator species showed low RAF values (Shvetsova et al. 2019). This proves that moss is a sensitive biomonitor in urban areas, but land use significantly affects the obtained result of the concentration of elements accumulated by mosses during exposure.

The species used in biomonitoring studies are significant because, for example, mosses of the genus *Dicranum* are known to be good accumulators of mercury (Demková et al. 2020). That was confirmed in this study, where the highest concentration of Hg was recorded in the moss *D. polysetum*, as well as confirmed by its relatively poor accumulation of metals compared to other species (Demková et al. 2019). Therefore, it is essential to use several moss species during biomonitoring for the possibility of comparison and selection of the best accumulating species (Motyka et al. 2011; Suoranta et al. 2016; Zawadzki et al. 2016). This is also indicated by this study (Table 3), as well as an earlier report where *S. fallax* was the best accumulator during winter (Świsłowski et al. 2022). The use of *P. schreberi* in active biomonitoring in other studies is common (Demková et al. 2018; Ilieva-Makulec et al. 2021) and confirms its applicability in this type of research. Compared to the previous works mentioned above, despite exposure in an industrial area, concentrations in the mosses we exposed are lower. This may be influenced by the canopy—the flushing

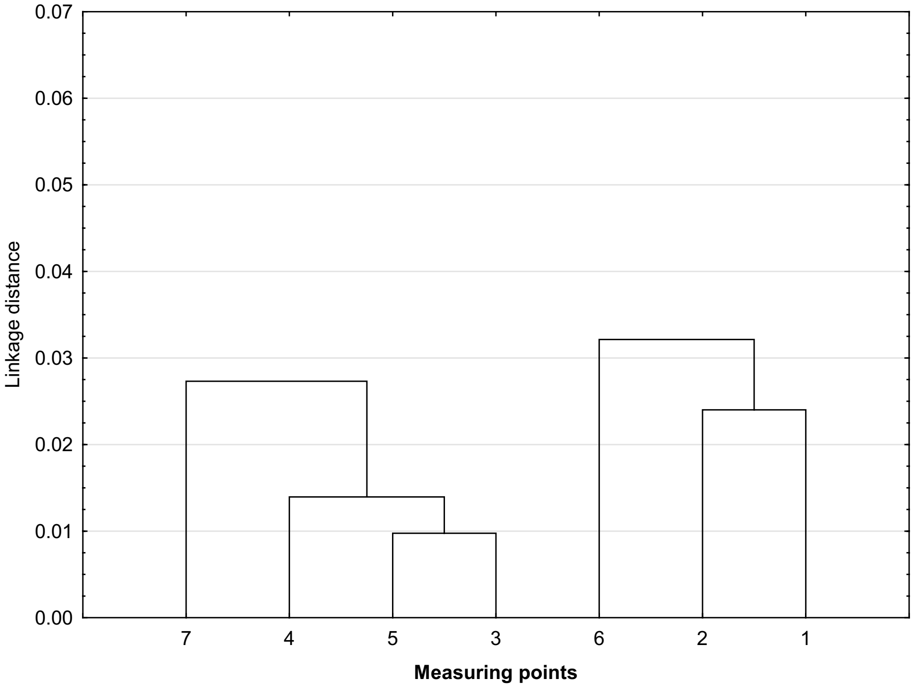

Fig. 3 Cluster analysis of metal concentrations in three moss species at all points (weighted average linkage, 1-r Pearson's distance)

of elements from the mosses under the precipitation, snow (Salo et al. 2016), or the long-time of storing/transporting the samples which may have a negative effect on the concentration of elements (Dołęgowska and Migaszewski 2020).

Biomonitoring studies in similar industrial areas confirm that obtaining high RAF values is related to the source of pollution, which is heavy industry (Sergeeva et al. 2021b). The analysis of rare earth elements and the values obtained by the authors only confirm the air pollution of this region as a result of parks, steelworks, and power station activities (Sergeeva et al. 2021a). For this study area, the highest RAF values were observed for Mn and Fe of those elements considered (Fig. 2). The level of concentrations of these metals in mosses is directly related to industrial activities (Molina-Villalba et al. 2015; Mulyana et al. 2015; Ruiz-Azcona et al. 2021). Point source identification of contamination is, therefore, possible (Messager et al. 2021). For this, the use of a suitable type of moss-bag (Morales-Casa et al. 2019) works well in the moss-bag technique. Flat and rotating (García-Seoane et al. 2019), they allow the bag to be appropriately oriented to the wind, which results in the highest increases in concentrations of selected elements being determined in samples exposed on the leeward side (Świsłowski et al. 2017). Taking this aspect into account (taking into account the frequency and direction of the wind) is crucial for the proper evaluation of point sources of pollution (Lucadamo et al. 2019). Our case confirmed the influence of local industry emissions on the high pollution of mosses at points on the leeward side (Fig. 1 compares with Fig. 3). This is quite well illustrated by these results, where we observe clustering of points depending on the specific source of pollution as influenced by wind direction (Fig. 3).

Conclusion

The moss-bag technique was first used around one of the industrial enterprises in Tula, RF. The results of the conducted study of 90-day exposure to mosses during the winter period indicate the contamination of the study area with selected heavy metals. The source of pollution is mainly active industry (Mn and Fe), but also linear sources of pollution (vehicle traffic and emissions on the railroads and roads/streets). In the future, it would be advisable to continue research on atmospheric aerosol quality using mosses over a long term with different periods to study seasonal changes and trends and to select the optimum biomonitor of atmospheric aerosol pollution. The effect of elements on the health of people living around this industrial area should be evaluated.

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Author contributions PŚ helped in conceptualization, methodology, formal analysis, investigation, measurements, writing—original draft, and visualization. MR helped in conceptualization, methodology, measurements, validation, writing—review and editing, supervision, and project administration.

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Data availability The data that support the findings of this study are available on request from the corresponding author.

Declarations

Conflict of interest The authors have no relevant financial or non-financial interests to disclose.

Consent for publication Not applicable.

Ethics approval and consent to participate Not applicable.

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