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MORPHOLOGY AND COMPOSITION OF PARTICULATE MATTER IN THE AIR OF URBAN AREA

MORFOLOGIA I SKŁAD PYŁU ZAWIESZONEGO W POWIETRZU OBSZARU MIEJSKIEGO

Abstract: Atmospheric pollution by particulate matter has become increasingly important in recent years, because it was found that these airborne particles have various adverse effects on the environment and human health. To extend the knowledge of such pollutants, detailed characterization of sizes, morphology and chemical composition of individual airborne particles is desired. In the presented research, the air microparticles were taken from discarded fiberglass tape filters which are commonly used in MP101M continuous suspended particulate analysers to determine the mass concentration of the particulate matter. These samples, which were collected in Kutna Hora (Czech Republic), were studied using the methods of scanning electron microscopy and energy-dispersive X-ray spectroscopy. Based on microscopic photographs, various geometrical parameters (Feret's diameter, perimeter, cross-section surface area, circularity, aspect ratio, roundness and solidity) of 237 particles were assessed, while the spectroscopy measurements revealed elemental composition of these particles. Statistical evaluation of the measured data was done using the methods of principal component analysis and compositional data analysis. By these methods, certain relationships among the geometrical parameters and the content of chemical elements in the particles were found. This research also demonstrated that the fiberglass air filters can easily be reused to gain additional information about airborne particles in various places at any time. The character of the particles can also provide information about a possible source of contamination.

Keywords: particulate matter, electron microscopy, morphology, shape, elemental composition, air filters

Introduction

Particulate matter, PM consists of tiny airborne particles of anthropogenic or natural origin which can affect the environment and human health adversely. The degree of

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hazard arising from PM depends especially on the particles' concentration, chemical composition, shapes and sizes [1, 2]. Particles smaller than 10 micrometres in diameter, PM10 can be inhaled into human lower respiratory tract and cause respiratory problems [3]. Particles smaller than 2.5 micrometres. PM2.5 can get to bronchi and develop asthma or chronic bronchitis [4], and particles with a diameter of less than 0.1 micrometre ($PM_{0.1}$) are able to penetrate even into the bloodstream [5] and harm a cardiovascular system, nervous system and other organs. Toxic metals as well as organic carcinogens can be bound to PM and transported over long distances by the air [6, 7]. The smaller the particle is, the longer distance it can overcome. PM in smaller than 1 μm in diameter can stay in the atmosphere for days or even weeks and therefore can be transported over distances up to thousands of kilometres. The coarse particles are deposited more easily, so they usually travel less than 10 km from their source. However, coarse mineral dust may be transported by dust storms for over 1000 km [7]. For these reasons, the knowledge of particle size distribution is useful for assessing the impact of PM on human health and environment.

An important role in dispersion of pollutants in the air play topographical factors (esp. terrain profile and surface roughness) [8], air quality is also related to human activities such as industrial production or road traffic [9]. No wonder that the PM air pollution is a growing problem particularly in cities [10] where significant amounts of emissions are generated.

Recently, various analytical techniques for characterization of air PM have been available. Their comprehensive description is beyond the scope of this paper, nevertheless let us mention at least a few of the commonly used. A common approach is to measure PM by the means of scanning electron microscopy (SEM) in combination with energy-dispersive X-ray spectroscopy (EDS) [11–13]. This allows to perform relatively fast morphological and elemental analysis of single particles, including spectral mapping of the selected particle [14]. Elemental composition can be determined also by proton induced X-ray emission [15], while molecular composition and the structure of both organic and inorganic PM materials is revealed by Raman spectroscopy [16]. Detailed information about the particle surface provide atomic force microscopy [17, 18]. For observing nano-sized particles, transmission electron microscopy is suitable [19].

An important part of atmospheric PM research is collecting the samples. In these days, samplers with various types of air filters are available for airborne PM collection, however, the sampling processes are considered as complex, because they require appropriate equipment and sampling techniques [20]. On the territory of the Czech Republic, continuous sampling of PM2.5 or PM10 is carried out in 127 measuring stations operated by the Czech Hydrometeorological Institute (CHMI). This PM monitoring serves to obtain information about mass concentrations of these particles in the atmosphere and to check if the concentration limits are not exceeded. The mass concentration of PM10 and PM_2.5 is measured automatically by MP101M continuous suspended particulate analysers [21] which use so called radiometric method. The measurement process looks like this: air from a surrounding atmosphere is blown through a fiberglass filter tape for 24 hours, then the tape is irradiated by beta radiation and its transmittance is evaluated. From this, the mass of particles trapped in the tape is calculated which allows to determine the PM mass concentration in the blown air. Subsequently, the filter tape is shifted, so that a new, clean section of tape is prepared for the next 24-hour measurement. This is repeated until the whole tape is consumed. The used filter tapes are then thrown away as useless. Figure 1 shows the principal scheme of MP101M analyser.

In this paper we demonstrate how it is possible to reuse these discarded filters for gaining more detailed information about air pollutants by performing single particle analyses of PM sizes, morphology and elemental composition. For this purpose, discarded filter tapes from a measuring station located in Kutna Hora (a town in the Central Bohemian Region of the Czech Republic with approximately 20 000 inhabitants) were utilized. The PM samples were collected during five days of measurement by a MP101M device. For the subsequent analyses, the methods of SEM and EDS were applied. The objective was to determine elemental composition of the particles, examine their structural parameters and find possible relationships among these characteristics. Another aim was to find whether the discarded filters from MP101M analysers are suitable for such analyses.

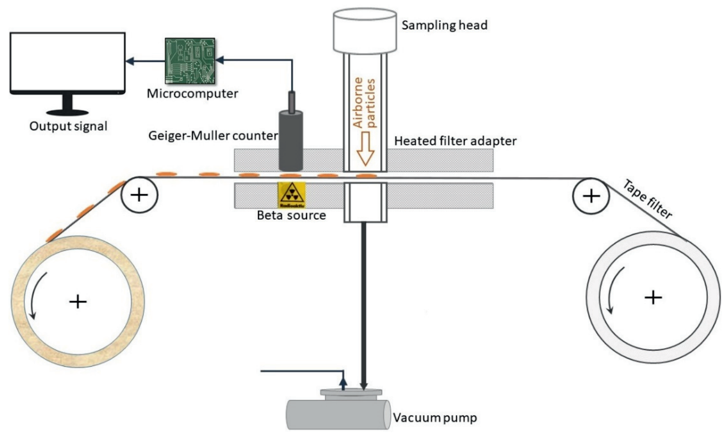

Fig. 1. The principle of MP101M analyser

Material and methods

The studied airborne microparticles were collected in a residential area of Kutna Hora, the measurement station (see Fig. 2a) is located on the lawn right next to the intersection of two narrow roads with sparse traffic. Besides the mentioned roads, there are also other possible sources of air pollutants in the vicinity of the measurement station. Since the station is surrounded by family houses which use local heating, local households can contribute significantly to the imissions. Several other contributors to local air pollution are located in the town Kutna Hora; these are small companies that burn fossil fuels and biomass. Another possible source of PM is a coal power plant in Chvaletice which is situated 16 kilometres northeast from the measurement station.

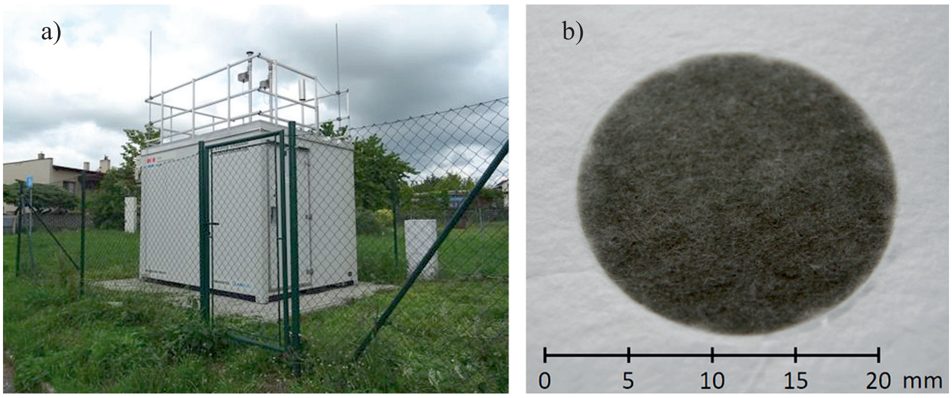

Fig. 2. a) Automatic imission monitoring station in Kutna Hora [24] and b) used fiberglass filter taken from MP101M particulate analyser after 24 hours of atmospheric air suction

In the measurement station, the PM10 particles were trapped into fiberglass filters (shown in Fig. 2b) during five days of collection. Subsequently, the particles were shaken out of the filters onto a conductive carbon tape and observed by a scanning electron microscope FlexSEM 1000 (Hitachi). The microscope was working in secondary electrons mode, the used accelerating voltage was 15 kV. Examples of observed particles are given in Figure 3.

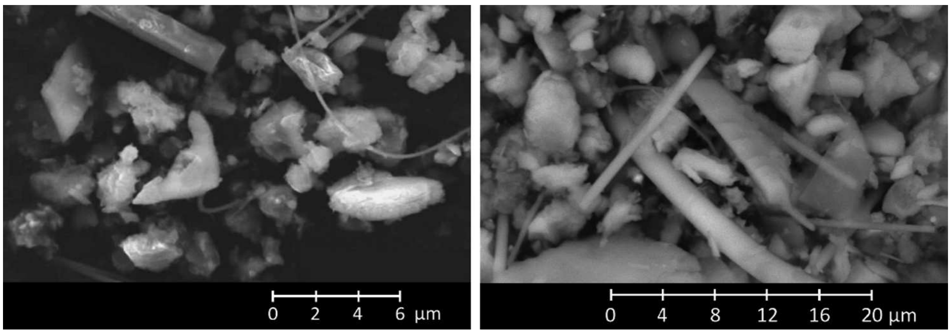

Fig. 3. SEM images of air microparticles obtained from air filters

Elemental composition of 237 individual microparticles was measured by using EDS detector from Oxford Instruments (detector type: SDD – Silicon Drift Detector, detection area: 30 mm^2, energy resolution: 137 eV at Mn K_α line). This detector is able to measure chemical elements from boron to uranium.

Further, SEM images of these particles were analysed in ImageJ software [22] so that for each particle the following geometrical characteristics were determined: Feret's diameter, area, circularity, aspect ratio, roundness and solidity. Feret's diameter (F_d) is the longest distance between any two points along the imaged particle's boundary. Circularity *cr* is defined as follows:

$$cr = 4\pi \cdot \frac{\text{area}}{\text{perimeter}^2} \tag{1}$$

with values ranging from 0 (extremely elongated particle) to 1 (perfectly circular particle). Aspect ratio, *AR* has the meaning of the ratio of minor axis to minor axis of the particle's fitted ellipse:

$$AR = \frac{\text{fitted ellipse major axis}}{\text{fitted ellipse minor axis}} \tag{2}$$

Roundness *ro* is a quantity related to the circularity, it can be calculated according to the following formula:

$$ro = 4 \cdot \frac{\text{area}}{\pi \cdot (\text{fitted ellipse major axis})^2} \tag{3}$$

Solidity *so* expresses how much the particle is compact, it is defined in equation:

$$so = \frac{\text{area}}{\text{convex area}} \tag{4}$$

where the convex area (convex hull) can be thought of as an area of rubber band wrapped tightly around the imaged particle's boundary. More detailed description of all these quantities can be found in [23].

Statistical methods

In the data analysis, statistical methods were used. Distributions of the determined geometrical parameters were assessed using their graphical representations in histograms. Covariabilities among the variables were estimated using the Principal Component Analysis (PCA) method [25], the data describing the geometrical parameters of particulate matter were normalised prior to the computations.

The concentrations of chemical elements in the examined material were determined too. The variables describing concentrations have some specific properties: they are limited to the range between 0 and 1 (on an appropriate scale) and are not independent on each other. To avoid misinterpretation of calculation results and false conclusions, constrained data that show the composition of the totals require application of appropriate statistical methods. Compositional data are not independent on each other, because if the content of one component increases, the others have to decrease. These characteristics of the variables exclude the application of standard statistical techniques to the raw data, the standard methods are typically intended for data analysis that ranges from $-\infty$ to $+\infty$. Another problem is the influence of the selection of variables used in calculations on their results. Such an effect is called a subcompositional incoherence [26–29].

The analysis of the data requires the consideration of problems in an appropriate sample space. For compositional variables such a space is simplex [30, 31].

For the above reasons, the methods developed for analysis of compositional data (CoDA) were used in the statistical calculations.

The PCA method was used to analyse the relationship between concentrations [25, 32, 33]. Before the calculations, the concentrations of elements in individual particles were transformed by clr (centred log ratio) transformation [26]:

$$\mathrm{clr}(\mathbf{c}) = \ln\left[\frac{c_1}{\mathrm{g_m}(\mathbf{c})}, \quad \frac{c_2}{\mathrm{g_m}(\mathbf{c})}, \ldots, \quad \frac{c_D}{\mathrm{g_m}(\mathbf{c})}\right]$$

where:

$$\mathrm{g_m}(\mathbf{c}) = \left(\prod_{i=1}^{D} c_i\right)^{1/D}$$

and $\mathbf{c}$ is the vector of concentrations.

This transformation allows to calculate the distance between the points representing the compositions of individual particles in the Euclidean space in conformance with the distances in the simplex space.

Finally, the statistical evaluation of both the geometrical characteristics and EDS results was performed using R language with packages *compositions* and *Hmisc* [34–36].

Results and discussion

Structural parameters

In Fig. 4, histograms of the size and shape descriptors' values are shown. Distributions of these parameters tend to be unimodal. Variables *AR*, *area* and F_d are positively skewed, whereas for circularity and solidity a trend to a negatively skewed distribution was observed. The results for circularity and *AR* indicate elongated shapes of the vast majority of the observed microparticles. The solidity values suggest rough to intermediately rough surface of the particles.

Most of the parameters are correlated. A strong correlation (0.92) was observed for the Feret's diameter and area, also solidity and circularity are correlated strongly (0.90). An intermediate correlation was calculated between Feret's diameter and circularity (–0.58) as well as between *AR* and circularity (–0.50) and between roundness and circularity (0.49). The values of other correlations are lower than 0.38, correlations between area and *AR* and between area and roundness are statistically insignificant.

In Fig. 4 histograms of distribution of the parameters determined are shown.

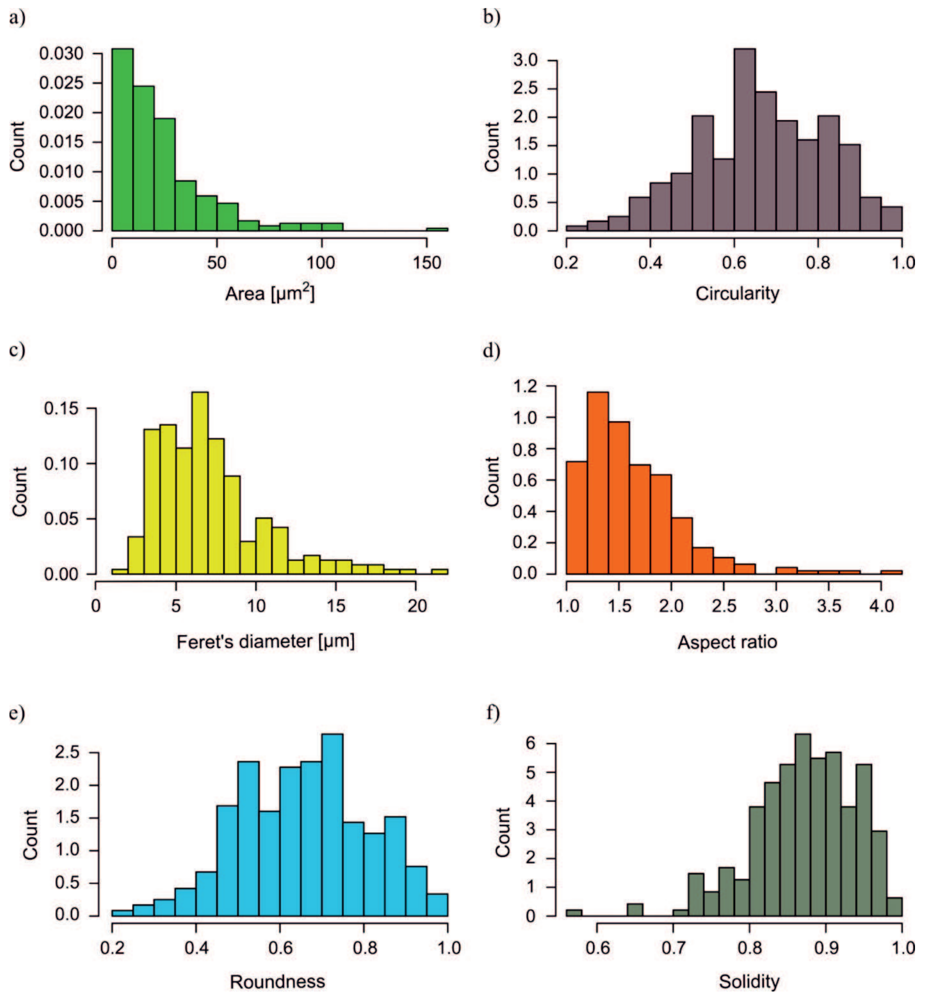

Fig. 4. Histograms of structural parameters distribution: a) area, b) circularity, c) Feret's diameter, d) aspect ratio, e) roundness, f) solidity

The PCA method was applied to all the mentioned size and shape descriptors for multidimensional data examination. The results of PCA can be summarized as follows:

– Variance related to the first principal component (PC1) constitutes 56 % of the total variance, the share of PC2 variance in total is 25 % and for PC3 the due value is 16 %. Thus, the first three principal components jointly include about 97 % of the total variance.

– The PC1 component is composed of all parameters with similar shares, i.e. the contributions of all variables are similar.

– In PC2, the contribution of solidity and circularity is insignificant.

– In PC3, solidity and circularity are the main components.

– Correlation analysis revealed certain relationships between variables, as presented in Figure 5. No trends of forming groups of points were observed.

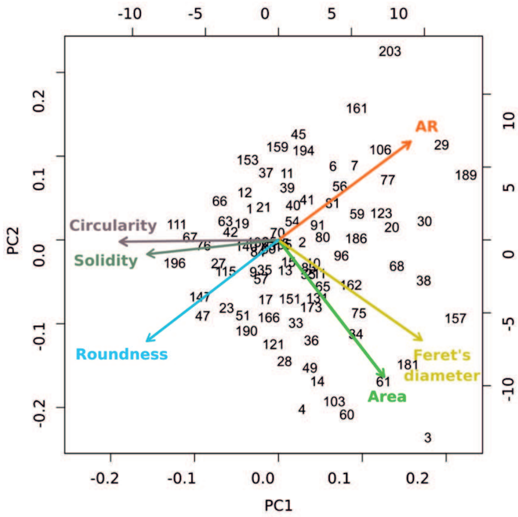

Fig. 5. Variables factor map of the PM structural parameters

Elemental composition

For elemental composition analysis, only those chemical elements were selected for which at least 50 % of results were higher than the detection limit (i.e. whose median of concentration was greater than zero). Distributions of the selected elements' concentrations are shown in Fig. 6. The main components on the particles' surface are C and O, their distributions are either multimodal or uniform. Distributions of Fe, Ca, Si, Al, Na and K are positively skewed.

To get a more comprehensive perspective of PM elemental composition, the Compositional Data Analysis (CoDA) methods were used [28, 29]. Prior to actual calculations, the raw content of elements in the particles was recalculated to concentrations in subcomposition comprising only the elements considered. In this way the influence of undetermined components on relationships between the elements studied is avoided. In the analysis, the calculation of linear correlation coefficient was skipped. The reason is that it has been shown that in compositional data covariances and variance of a component are not independent, leading to a spurious correlation effect [37].

The PCA was performed too, using the data transformed by the centred log-ratio (clr) function. The results of PCA can be summarized as follows:

– Variance related to the first principal component (PC1) constitutes 33 % of the total variance, the share of PC2 variance in total is 23 % and for PC3 the due value is 19 %.

– The first three principal components jointly include only about 75 % of the total variance. In the data analysis, the first three principal components have to be studied.

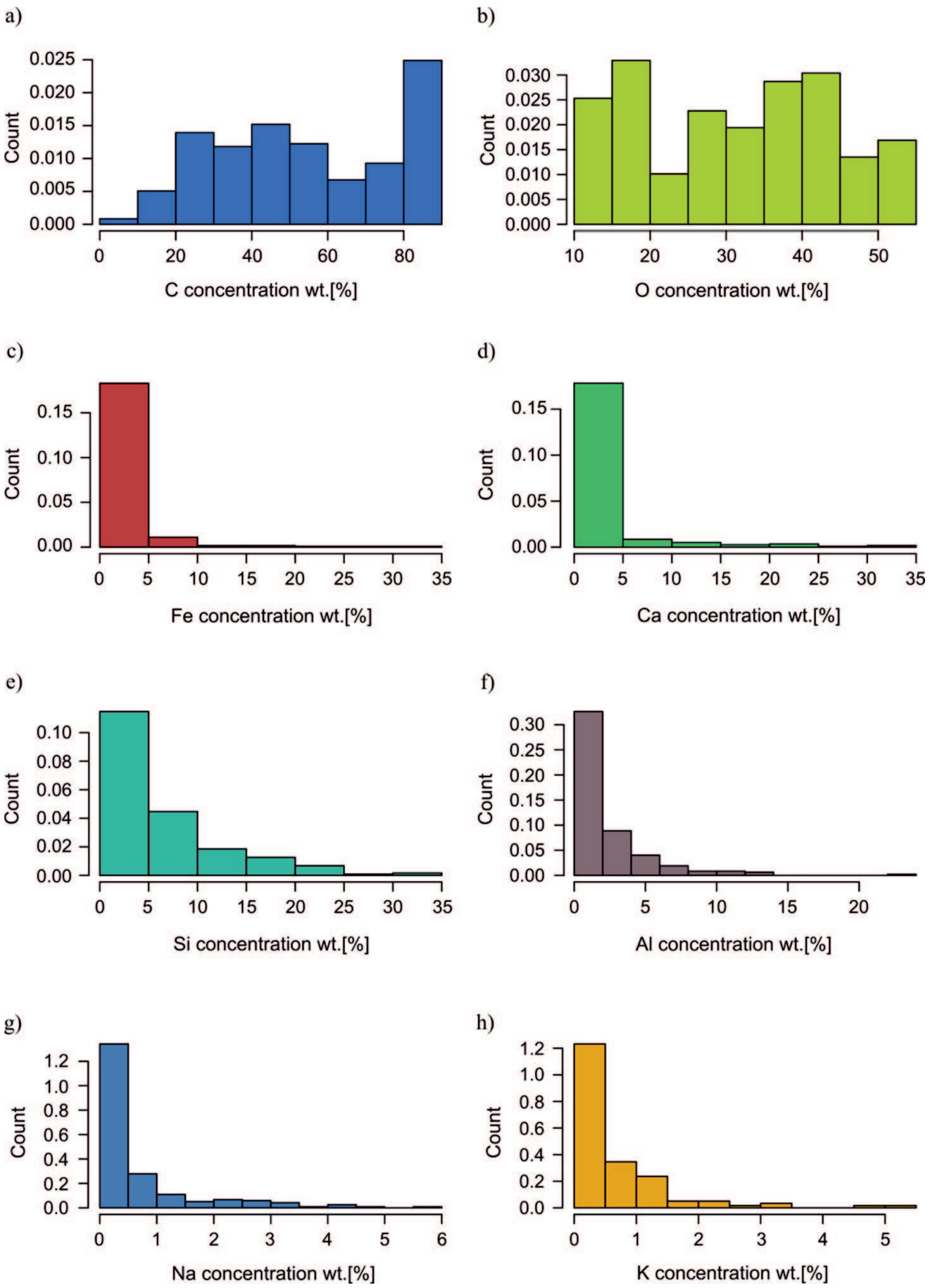

Fig. 6. Histograms of elements' concentrations in the microparticles a) C, b) O, c) Fe, d) Ca, e) Si, f) Al, g) Na and h) K

– In a scree plot (Fig. 7a), the indication of the most important principal components is ambiguous.

– In PC1, the main components are Ca, Na, and in less extent, Si and C.

– In PC2, Fe and Na dominate, but the contributions of Ca and Al are also significant.

– The PC3 comprises C, Al and Si, the contributions of Fe and O are remarkable too.

– The component with the lowest variability, PC7, comprises O, C and Si. This component probably represents low variability in the concentrations of carbonates and silicates in the PM.

– Among the pairs of elements' concentrations, only Al and Si are related to each other in some extent. This can be a result of high aluminosilicate content in the PM. Because aluminosilicates are abundant in the Earth's crust, it can be expected that such particles originate from soil.

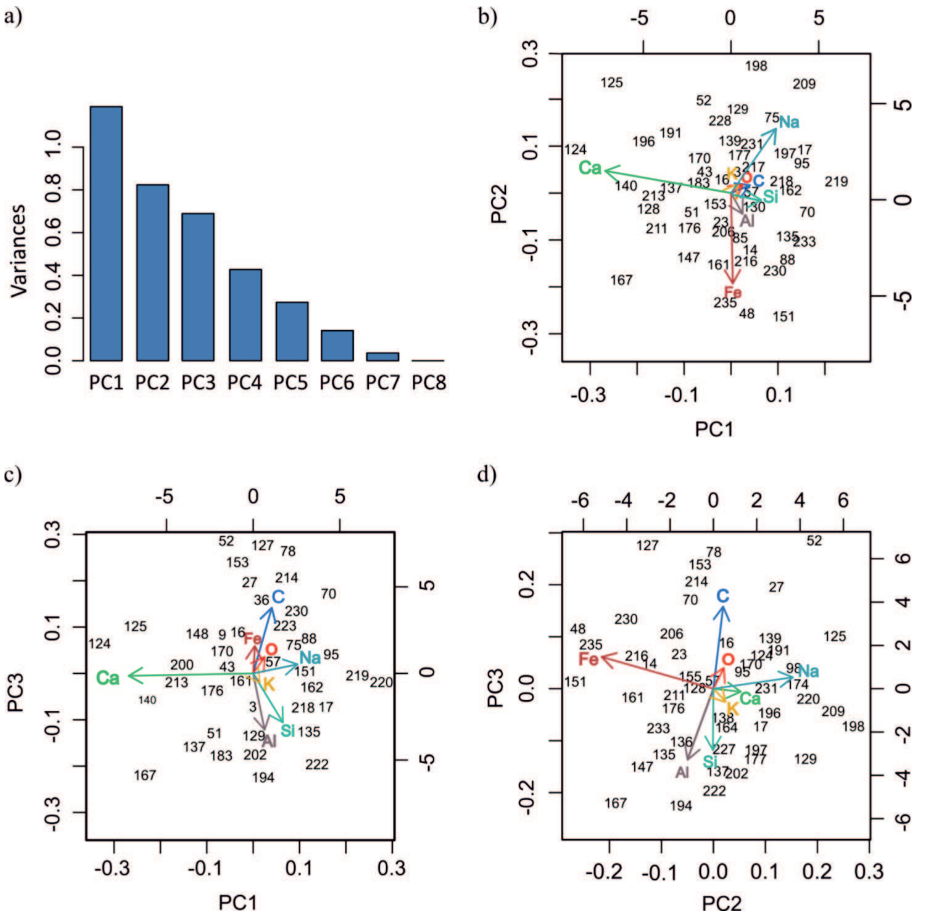

Fig. 7. Scree plot a) of the principal components' variances and b–d) PC1-PC3 variables factor maps for elements' concentration

Factor maps of PC1-PC3 variables are presented in Figure 7b–d. In addition to the above-mentioned chemical elements, the EDS analysis revealed also small concentrations of Mg, S, Ti, P, Cl, Br, Zn, Cu and Cr in some particles.

Relationship between structural parameters and elemental composition in the PM

Only a weak, but statistically significant at $p = 0.021$, relationship between Feret's diameter and silicon concentration (c_{Si}) in the PM was found. The multiple R-squared coefficient was 0.064. The corresponding linear equation is $c_{\mathrm{Si}} = b_0 + b_1 \cdot F_{\mathrm{d}}$, where the b_0 value is 0.37 with a standard error $SE_{b0} = 0.14$ and $b_1 = 0.040$ with $SE_{b1} = 0.017$. For other relationships between the structural parameters and element's concentration only statistically insignificant correlation coefficients were obtained.

Conclusion

The methods of SEM and EDS were applied to study structural characteristics and elemental composition of 237 airborne particles. A subsequent statistical evaluation of the data revealed only a weak relationship between Feret's diameter and silicon concentration, no other statistically significant linear relationships between the geometrical parameters and chemical composition of the particles were found. Regarding the geometrical parameters themselves, most of them are correlated. As for the chemical composition, only the concentrations of Al and Si in the particles are related to each other in some extent.

In this research it was also shown how discarded air filters can be reused to gain additional information about PM particles, whose atmospheric concentrations are continuously monitored in many air quality monitoring stations on the territory of the Czech Republic. The introduced analyses can therefore be used to study the character of PM particles in various places at any time. This can be useful especially when aźsuspicion of environmental contamination by e.g. toxic metals is somewhere. The character of PM particles can also suggest a possible source of air contamination in a certain area.

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Abstrakt: Zanieczyszczenie powietrza przez pył zawieszony (PM) staje się w ostatnich latach coraz bardziej znaczące. Stwierdzono, że unoszące się w powietrzu cząsteczki niosą różne niekorzystne skutki dla środowiska i zdrowia ludzkiego. Aby poszerzyć wiedzę na temat takich zanieczyszczeń, pożądana jest szczegółowa charakterystyka rozmiarów, morfologii i składu chemicznego poszczególnych cząstek zawieszonych w powietrzu. W prezentowanych badaniach mikrocząsteczki powietrza zostały pobrane z filtrów taśmowych z włókna szklanego, które są powszechnie stosowane w analizatorach cząstek stałych MP101M. Próbki zostały zebrane w Kutnej Horze (Republika Czeska), były badane metodami skaningowej mikroskopii elektronowej i spektroskopii rentgenowskiej z rozpraszaniem energii. Na podstawie zdjęć mikroskopowych oceniono różne parametry geometryczne (m.in. średnicę Fereta, obwód, powierzchnię przekroju poprzecznego, krągłość, współczynnik kształtu) 237 cząstek, a pomiary spektroskopowe pozwoliły na określenie składu pierwiastkowego tych cząstek. Statystyczną ocenę otrzymanych wyników wykonano metodami analizy głównych składowych i metodami analizy zmiennych złożonych. Stwierdzono istnienie pewnych zależności pomiędzy parametrami geometrycznymi a zawartością pierwiastków chemicznych w cząstkach. Badania te wykazały również, że filtry powietrza z włókna szklanego mogą być z łatwością ponownie wykorzystane do uzyskania dodatkowych informacji o cząstkach zawieszonych w powietrzu w różnych miejscach w dowolnym czasie. Charakter cząsteczek może również dostarczyć informacji o możliwym źródle zanieczyszczeń.

Słowa kluczowe: pył zawieszony, mikroskopia elektronowa, morfologia, kształt, skład pierwiastkowy, filtry powietrza