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# Influence of a nickel smelter plant on the mineralogical composition of attic dust in the Tikveš Valley, Republic of Macedonia

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**Abstract** Mineral phases and their content were determined in attic dust samples collected from 27 houses in the Tikveš Valley, Republic of Macedonia. By using quantitative X-ray diffraction, the principal mineral phases were determined to be the serpentinite group (chrysotile, lizardite) and amphibole group of minerals (ribecite, tremolite, actinolite) present in the attic dust samples from this region which are not common constituents of urban dust. Strong correlations existed between these mineral phases in the dust and those in ores processed at a ferronickel smelter plant situated in this region. Spatial distributions of specific mineral phases were made and were consistent with wind directions and predicted deposition (60–70 %) of dust emitted from the metallurgical plant.

**Keywords** Attic dust · Mineralogical composition · Nickel smelter · Tikveš Valley · Republic of Macedonia

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## Introduction

Problems of ecosystem degradation due to pollution are becoming increasingly acute. Significant negative influences include mining works, factories, smelter plants and many other industrial processes, due to their direct venting of metal into the atmosphere. Mines produce large amounts of waste because the used ore and concentrates comprise only a small fraction of the total volume of mined material (Salomons 1995; Dudka and Adriano 1997). This kind of sudden change in the environment exposes the biosphere to a risk of destabilisation, since organisms that developed under conditions of low concentrations of a metal have not developed biochemical pathways capable of detoxifying the metal when it is present at high concentrations. As result of these processes, dust is perpetually introduced into the atmosphere (Sengupta 1993; Repley et al. 1996). Dust is a generic term used to describe fine particles suspended in the atmosphere. Atmospheric particles are tiny particles of solids or liquids suspended in air. These particles vary in size and density. They originate as a result of processes on the surface of the earth and in the atmosphere. They may be either be emitted directly into the atmosphere or formed there by chemical reactions (Finlayson-Pitts and Pitts 2000). Particles may be emitted naturally or have an anthropogenic origin. According to their origin and specific characteristics, particles differ in size, mass, density, morphology, and chemical composition and have various chemical and physical properties (Godish 2004; Baron and Willeke 2005).

The term dust usually comprises street dust and house dust (Culbard et al. 1988; Fergusson and Kim 1991; Fergusson 1992; Dundar and Ozdemir 2005; Ochsenkühn and Ochsenkühn-Petropoulou 2008). However, other types have also been studied in the past. One particular type of house dust is the attic dust studied in this work. It represents

dust deposited in attics abandoned by inhabitants, so that tenant influence is minimised. Attic dust is derived predominantly from external sources such as aerosol deposits and as a result of crop dusting and less from household activities (Šajin 1999, 2003). Attic dust as sampling material has the advantage that its composition remains constant, i.e., chemically unchanged over time, as long as the anthropogenic source of pollution is not changed; otherwise, it changes over time (Žibret and Rokavec 2010; Žibret 2012). Investigations of attic dust chemistry therefore reveal the average historical state of the atmosphere (Šajin 1999, 2000, 2005; Balabanova et al. 2011; Bačeva et al. 2011; Stafilov et al. 2012).

The subject of this study is to analyse the distribution of various mineral phases in attic dust samples as reflection of lithology and anthropogenic influence in the Kavadarci Region, known for its ferronickel industrial activity (Stafilov et al. 2010). In our previous studies, it was shown that the most important source of trace metal deposition is the ferronickel smelter plant situated in this region, contributing to influencing on the presence of higher content of Ni as well as Co, Cr and some other heavy metals in the soil and dust in the city of Kavadarci and its surroundings (Stafilov et al. 2008, 2010; Bačeva et al. 2011; Stafilov et al. 2012). For this reason, the goal of this work was to determine the mineral phases and specific minerals in attic dust samples in Kavadarci and its environs, as well as their spatial distribution, and to assess the size of the area eventually affected by the ferronickel smelter.

## Study area

Among the valleys in Macedonia, Tikveš Valley stands out in particular as a separate geographic entity with its own geomorphological and antropogeographical characteristics (Hristov et al. 1965). With an area of 2,120 km<sup>2</sup>, this valley occupies a substantial part of the territory of the Republic of Macedonia. It is bounded by mountains: to the south by the Mariovo–Magelanian mountains, ranging up to 1,700 m. Mountain heights to the east and west are also quite high. West of the valley is Mount “Borila” at 1,500 m and to the south is “Bali” mountain at 1,400 m and “Karadak” with a height of 750 m. Because of such limitation by mountains, Tikveš Valley is cut from the Vardar River on its north side and on the west side by the Crna River; the Luda Mara River passes through the middle of the valley (Fig. 1).

The geographic position and relief of the Tikveš Valley is an important factor that affects the overall climatic characteristics. The Tikveš Valley is an area where two zonal climates, continental and Mediterranean, intersect and influence the local climate. The influence of the continental climate spreads from the north and continues along the Vardar and Bregalnica Rivers. As a result of its impact, there are brief very cold periods. The Mediterranean climate in

turn flows in from the south and the Aegean Sea into the Vardar River valley, and as a result of its impact, there are beneficial winters with relatively high temperatures.

The mean annual temperature is 13.6 °C. The average winter temperature is 3.2 °C and the absolute minimal temperature is –22 °C. In contrast, the summertime brings high temperatures. The average temperature in July is 24.7 °C with an absolute maximum of 42 °C. Mean annual precipitation is estimated at 477 mm (Lazarevski 1993). The major wind direction is from the north and northwest.

Most of the Tikveš Valley is characterised by small amounts of rainfall and the territory around the city is considered the site with the least rainfall per square metre on the territory of the Republic of Macedonia (in Kavadarci, the average rainfall is 484 mm).

The population in the Tikveš Valley (about 60,000) is engaged mainly in agriculture (cultivation of vegetables and grapes and wine production). About 100 million kg of grapes are generated annually in this valley.

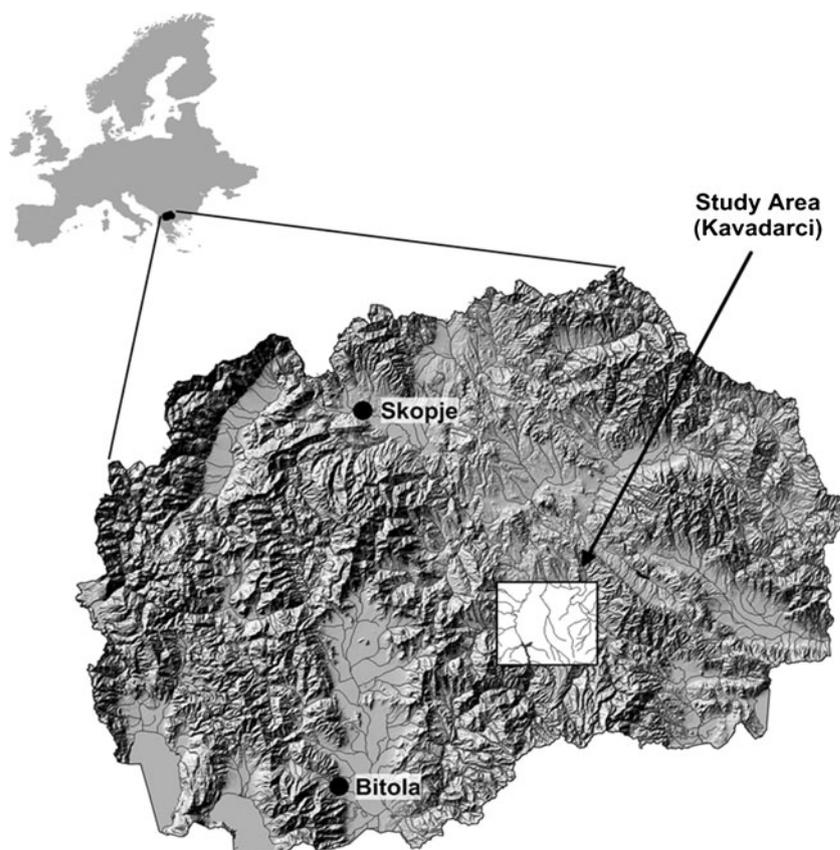
In 1980, a nickel production plant is built in Tikveš Valley which started with the production in 1982 with an annually processing of about 1.5 million tons of latherite types of nickelous ore. Starting in 2005, in total, about 900,000 tonnes of ore come from the Rzanovo mine annually (southern parts of Kozuf Mountain in Tikveš Valley), and since then, the smelter plant has begun processing ore originating from Albania, Turkey and Indonesia. Data concerning the composition (chemical and mineralogical) of these types of ores are presented in several publications (Boev and Ivanova 1998; Boev et al. 2009; Boev and Bermanec 2005). The operation of this plant in any case affects changes in the composition (mineralogical) of urban dust in the Tikveš Valley.

## Basic characteristics of the ferronickel smelter plant

The factory was built in the period of 1976–1982 with pyrometallurgical installations for the processing of latherite nickelous ores, with a yearly capacity of 2 million tons of ore. The average chemical composition of the ore processed in the smelter plant is presented in Table 1 (Boev and Bermanec 2005). The smelter plant produces about 16,000 t of nickel in the form of ferronickel with a Ni content of 25 to 40 %.

Based on studies of mineral associations as well as the major mineral phases, the major nickel-bearing minerals in the ores include magnetite, hematite, clinocllore, talc, sepiolite, magnesioriebeckite, lizardite, antigorite, actinolite, tremolite, chrysotile, dolomite, phlogopite, stilpnomelane, muscovite, quartz, albite, pyrite, maghemite, pirotine, digenite and millerite. Only five of the mentioned minerals are constantly present, including magnetite, hematite, clinocllore, talc and magnesioriebeckite (Boev et al. 2009).

**Fig. 1** Location of the investigated region of Kavadarci and its environs



A simplified scheme of technological process is as follows: the ore comes by transport belt from Rzanovo mine (as well as by rail or truck transportation from the port of Thessaloniki, Greece, for ore originating from mines in Indonesia or from Albania or Turkey). The ore is then processed by crushing, grinding and homogenization. During these processes, a

**Table 1** The average content of some heavy metals in ore from mines from different countries processed in the ferronickel smelter plant (Boev and Bermanec 2005) and of the attic dust samples from the Kavadarci Region (Stafilov et al. 2012) (given in milligrams per kilogram)

Element	Ore from different countries			Attic dust
	Rzanovo, Macedonia	Albania	Turkey	
As	16	21	560	18
Co	160	415	605	18
Cr	3,650	5,100	2,440	140
Cu	19	81	36	50
Mn	1,840	3,200	4,100	510
Ni	4,200	8,800	9,950	230
Pb	20	29.8	27	180
Sb	45	51	9.5	2.4
V	90	164	65	110
Zn	52	199	251	350

certain amount of dust is generated due to the structure of the ore and its very fine grinding. From this plant, the milled and homogenized ore is either inserted into the pelletization plant or goes directly to another section for prereduction (rotary kilns). In these plants, a certain amount of dust is also generated and emitted into the atmosphere, despite the built-in filtering equipment and chimneys. Then, the prereduced material is inserted into the electric furnaces where the material is melted, and after additional reduction, the liquid ferronickel is produced. The ferronickel is then refined into the refining plant, and finally, the granulated ferronickel is produced.

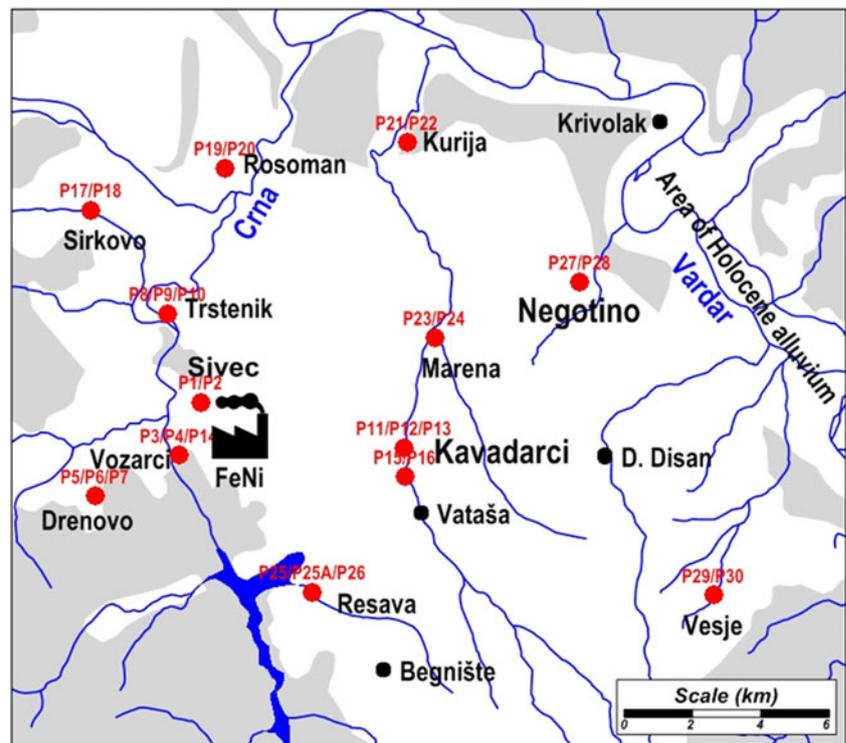
Based on these processes, it can be concluded that during processing of the nickel ore, a certain amount of dust is generated and emitted into the air in the Tikveš region. Legal norms existing in the Republic of Macedonia specify  $50 \mu\text{g m}^{-3}$  or less. It must be noted that the emission of dust observed from the factory for the production of nickel in fact exceeds this limit (Bačeva et al. 2011, 2012; Stafilov et al. 2012).

## Materials and methods

### Sampling

Attic dust was sampled from 27 old rural houses from 13 settlements in the Tikveš Valley in 2008 (Fig. 2). The houses

**Fig. 2** Attic dust sample locations



were of similar age (constructed after 1982, the year the ferronickel smelter plant was built). In every settlement, attic dust was collected from two or three houses on different sites. The collection of attic dust samples was performed according to the adopted protocol (Šajn 2003; Balabanova et al. 2011, Bačeva et al. 2011; Stafilov et al. 2012). Attic dust was collected with a plastic brush in polyethylene bags. The surface layer from the attic timber was discarded away and the finest dust was collected. The fraction of attic dust smaller than 0.125 mm in size was prepared by sieving.

#### Instrumentation

The mineralogical content of the collected dust samples was determined using an X-ray Siemens D 500 equipped with an automated computer and a Cu-monochromatic lamp working at 40 kV and 30 mA. Quantitative analysis of the mineral phases present was performed using the DIFRAC-11 software package and program support by Evaluation (EVAL) and Identification Diffraction Reflex (IDR).

The values given for the quantitative composition of the analysed samples represent an average of three replicates. For QA and QC of the measurements, referent materials and standards from various mineralogical compositions were used: BDS 17385/96 (standard for ore and ore concentrates for X-ray diffraction quantitative phase analysis), ST SEV 3534-82 (SpS-quartz sand), ST SEV 2981-81 (KN-2, limestone), ST SEV 2980-81 (MpA-copper ore) and USZ 47-2008 (granite

“MGT-1”). In several cases, standard addition method was applied by using some of the aforementioned RM and satisfactory values for the recoveries were obtained.

Differential thermal measurements were performed using the Q-1500-D instrument produced by MOM, Hungary, under the following conditions: sampling mass of 500 mg, DTA sensitivity of 250  $\mu$ V, DTG sensitivity of 500  $\mu$ V, heating rate of 10  $^{\circ}$ C/min, temperature interval of measurement of 15–20 to 1,000  $^{\circ}$ C and furnace ambient air without turbulences.

#### Generation of maps

The maps with spatial distribution of the analysed minerals were prepared using the Inverse Distance Weighting method for map generation (Geographic Information System (2012) software ESRI<sup>®</sup> ArcGis<sup>™</sup> 9.2 and its extension Geostatistical Analyst). Spatial distribution maps were constructed according to the coordinates and sample content of each specific sample.

## Results and discussion

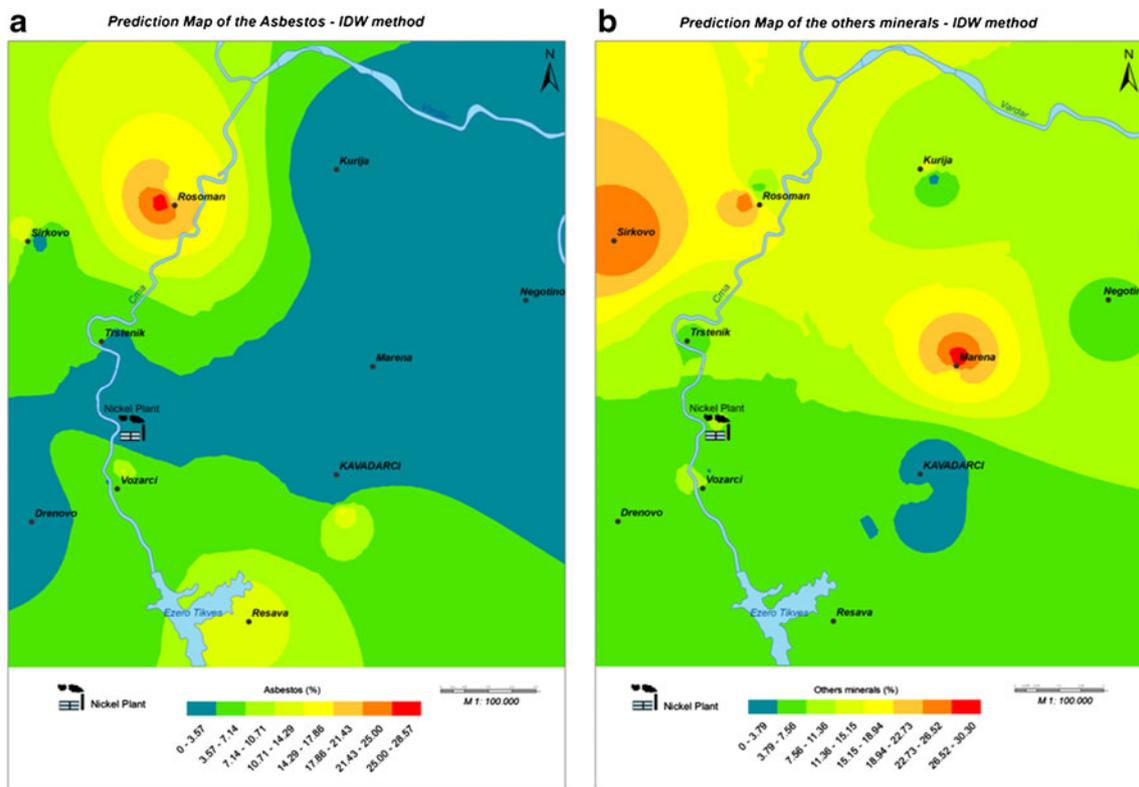
By applying X-ray diffraction, the minerals present in the collected dust samples were determined. For those minerals which are sensitive to X-ray diffraction, the quantitative analysis of the mineral phases present was performed using the software package DIFRAC-11 with program support from EVAL and IDR. The results of the quantitative mineralogical

content analyses of the 27 attic dust samples are presented in Table S1 of the Electronic Supporting Information. A comparison of the mineralogical content determined in the analysed attic dust samples (quartz, muscovite, Mg–Fe clinocllore, calcite and plagioclase; serpentine minerals (lizardite, chrysotile); dolomite, microcline, cumingtonite, tremolite, actinolite, beidellite, brucite, kaolinite, crocidolite, ferro-glaucophane, riebeckite, talc, diaspore, montmorillite, enstatite, grosular and halloysite (provided in Table S1 of the Electronic Supporting Information); and the minerals present in the ore processed in the ferronickel smelter plant (magnetite, hematite, Mg–Fe clinocllore, talc, sepiolite, riebeckite, lizardite, antigorite, actinolite, tremolite, chrysotile, dolomite, phlogopite, stilpnomelane, muscovite, quartz, albite, pyrite, maghemite, pirotine, digenite, millerite and kaolinite)) shows that almost all the minerals (about 90 %) present in ore material are represented in the attic dust samples as well.

The determined mineral content is in agreement with that detected in the polluted area by analysis of heavy metals present in the processed ore presented in our previously published data (Bačeva et al. 2011; Stafilov et al. 2012). Namely, the ferronickel smelter plant uses ore that contains between 1 and 2.5 % Ni, about 0.05 % Co, 1–3 % Cr, etc. (Data are provided in Table S2 of the Electronic Supporting Information). Therefore, we expect these elements to have significantly higher contents in samples of attic dust

compared to other elements. As can be seen from the data presented in Table 1, the average value of Ni in samples of attic dust taken from the Kavadarci area is  $230 \text{ mgkg}^{-1}$ , while the median value for the content of Ni in soil from this region is  $72 \text{ mgkg}^{-1}$  (Stafilov et al. 2010). However, the range of values shows a high content of nickel in samples taken from the vicinity of the ferronickel smelter plant, ranging from 89 to  $1200 \text{ mgkg}^{-1}$  (Data are provided in Table S3 of the Electronic Supporting Information). Cobalt and chromium results show significantly higher contents in the samples of attic dust taken from the Kavadarci area as well (Table 1). The average value for Co in attic dust samples taken from the studied area is  $18 \text{ mgkg}^{-1}$  (ranging from 10 to  $52 \text{ mgkg}^{-1}$ ), and the median value for Cr is  $140 \text{ mgkg}^{-1}$  (ranges from 72 to  $510 \text{ mgkg}^{-1}$ ) (Data are provided in Table S3 of the Electronic Supporting Information). However, their content in the soils of this region is lower, and the median value for Co is  $15 \text{ mgkg}^{-1}$  and for Cr is  $50 \text{ mgkg}^{-1}$  (Stafilov et al. 2010).

It should be mentioned that the mineralogical content of attic dust depends on several conditions: the current erosion of the rocks and soil (Sterk and Goossens 2007), the traffic intensity in the urban settlements, wind intensity, the presence of the industries generating dust, etc. The quantity of dust in the air as well as its mineralogical content significantly affect the populations' health (Ostro 1994). On the



**Fig. 3** Spatial distribution of asbestos minerals (a) and other various minerals (b)

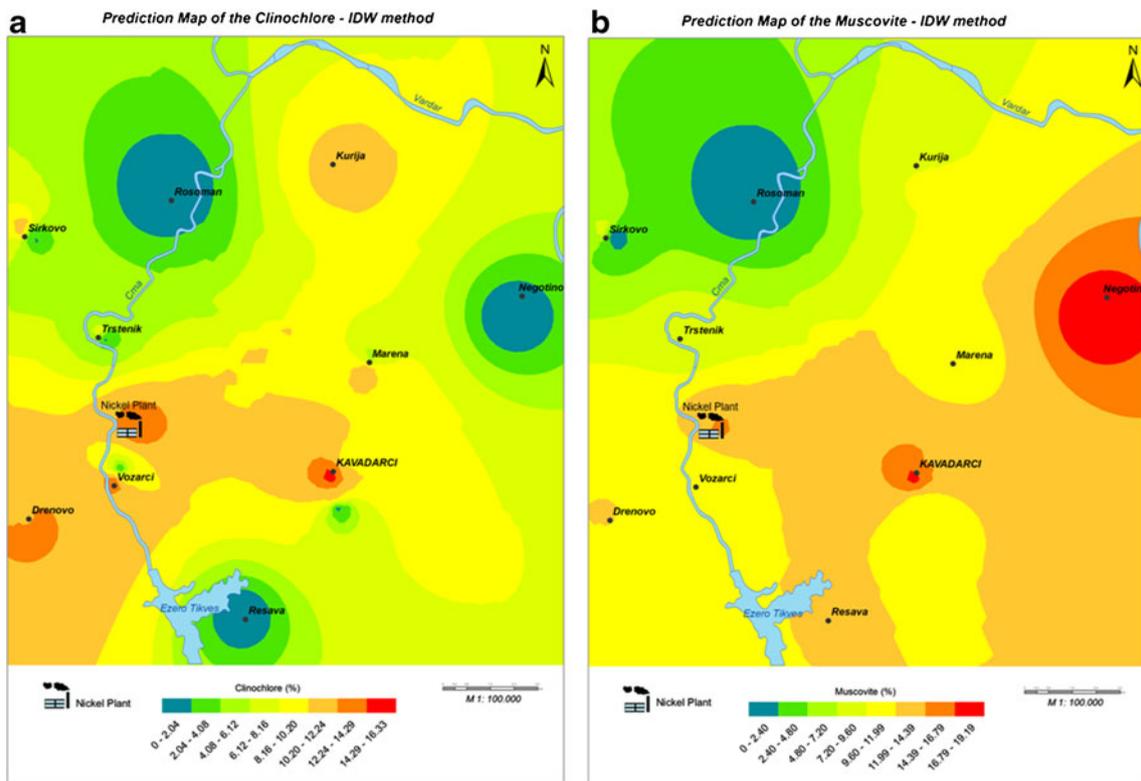


Fig. 4 Spatial distribution of clinochlore (a) and muscovite (b)

basis of the obtained results (Data are provided in Table S1 and S2 the Electronic Supporting Information) and from the distribution maps, it can be concluded that the spatial distribution of minerals shows that minerals from the serpentine group of minerals (chrysotile, crocidolite and other serpentine minerals (Fig. 3a)) have higher content in the area along the

Crna River and the largest contents are in the region of the village of Rosoman. It should be mentioned that the group of serpentine minerals is characterised by its fibrous morphology (Whittaker and Zussman 1971), and as a consequence, they are milled to micrometre dimensions during the crushing process. Such milled serpentine minerals are retained in the air for

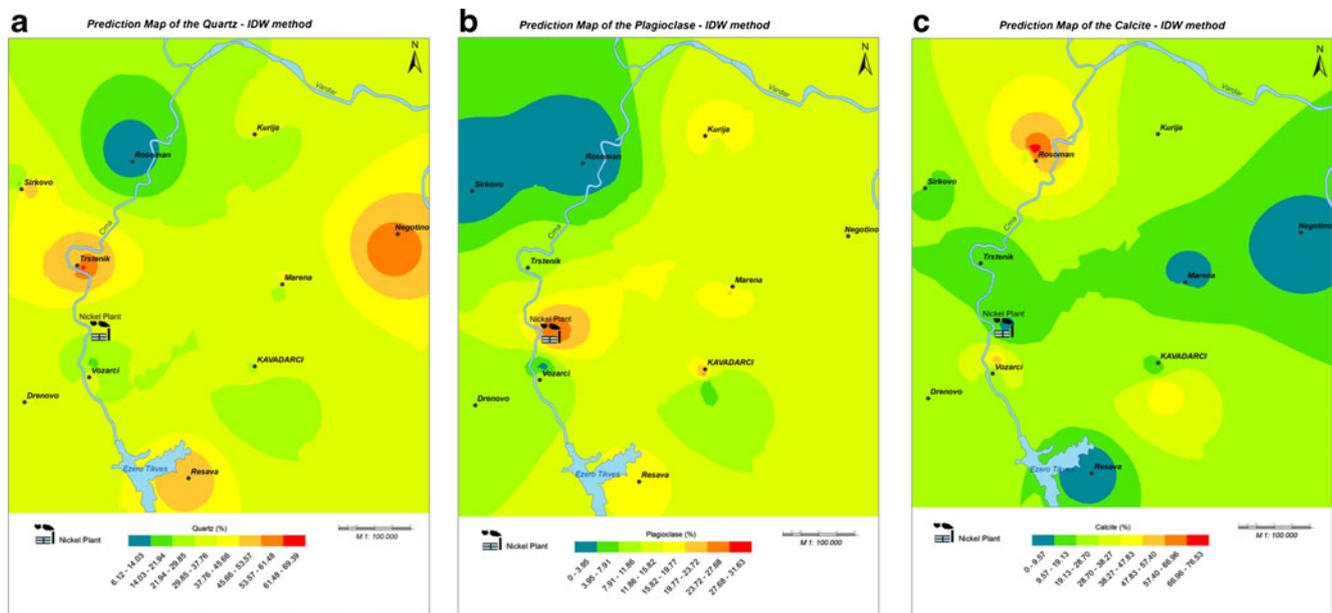


Fig. 5 Spatial distribution of quartz (a), plagioclase (b) and calcite (c)

a long time even during period of relatively high humidity and could be transported very easily along the direction of wind in the area (Lazarevski 1993).

The spatial distribution of minerals from another groups of minerals (dolomite, Fe–Mg amphibole, beidellite, tremolite, actinolite, microcline, brucite, kaolinite, fero-glaucophane, albite, talc, etc.) showed a specific nimbus of scattering (Fig. 3b), which in most cases coincided with the direction of movement of air masses along the Crna River. It can also be concluded that the city of Kavadarci is relatively well protected by a small hill Ljubas which by virtue of its north–south placement behaves like a natural dam against dissemination of dust from the ferronickel factory.

The spatial distribution of minerals of the clinocllore and muscovite (Fig. 4) type is very interesting, due to their relatively high presence in the ores that are processed in the ferronickel smelter plant. From Fig. 4, it can be concluded that these minerals are spread in a direction perpendicular to the main direction of movement of air masses (along the Crna River) and that their highest concentrations are in the region of the plant itself and the cities of Kavadarci and Negotino. The explanation for this phenomenon must be sought in the structure of these minerals (Bailey 1984) (leaf structure) that allows for greater retention in the air, so during certain periods of the year when the wind changes direction, they are scattered in the manner as shown in Fig. 4.

The spatial distribution of quartz (Fig. 5a) shows the appearance of several peaks that are not correlated with the main source of dust of anthropogenic origin (ferronickel production plant). This spatial distribution is basically explained by literature data on mineralogical composition of urban dust (Falkovich et al. 2001). The spatial distribution of plagioclase (Fig. 5b), which is quite common in the ores that are processed in the ferronickel plant, is localized in the vicinity of the plant.

The mineral calcite appears frequently as an integral part of the urban dust (Kalderon-Asael et al. 2009; Pandis et al. 1995). The spatial distribution in the Tikveš Valley shows the maximum content of calcite in the region of the village of Rosoman (Fig. 5c). This increased content of calcite in urban dust around Rosoman could be explained by the influence of the ground geology (Stafilov et al. 2010) and by the fact that a large quarry is located near Rosoman for the production of carbonate fractions from which large amounts of dust (with enormous amounts of calcite) are generated.

## Conclusion

Mineralogical investigation of 27 attic dust samples collected in the Tikveš Valley in the Republic of Macedonia showed that the mineralogical dust composition differs

greatly from the usual mineralogical composition of urban dust. It was determined that the serpentinite (chrysotile, lizardite) and amphibole group of minerals (ribecite, tremolite, actinolite) are present in the attic dust samples from this region which are not common constituents of urban dust. The fact that these minerals are present in the ore processed in the ferronickel smelter plant situated in this area and the high content of Ni in the analysed dust samples confirms that the source of the present pollution is the metallurgical plant

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