Emission of SO_2 and SO_4^{2-} from copper smelter and its influence on the level of total S in soil and moss in Bor, Serbia, and the surroundings

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Abstract

The city of Bor and the surroundings (Eastern Serbia) have been known for exploitation and processing of sulphide copper ores for more than 100 years. Emissions of waste gases and particulate matter rich in heavy metals are characteristic for pyrometallurgical production of copper. Long-term measurement results (2005–2008) indicate an increased sulphur dioxide level in the urban-industrial zone of Bor since it is closest to the copper smelter which is a dominant source of air pollution in the studied area. Average annual sulphur dioxide concentrations at four measuring sites in the urban-industrial zone exceeded the maximum allowable value of 50 μg/m^3. However, the maximum allowable value of the total atmospheric depositions (200 mg/m^2 per day on an annual basis) exceeded only at two of 15 measuring sites in the urban-industrial and rural zone. The highest annual deposition rate of sulphates from deposition was detected in the urban-industrial zone. Since the maximum permitted value for sulphates is not defined by the Serbian regulations, the extent of the pollution cannot be discussed. Since the environment can continuously be polluted through the wet and dry depositions, the biomonitoring by moss was conducted, which revealed significantly higher concentrations of total sulphur in moss in the urban-industrial zone, compared to the background zone. The obtained results confirm the reliability of moss as a bioindicator of ambient pollution. Higher total S concentration in soil samples was noted at the rural site (Ostrelj) located in the close vicinity of two tailing ponds.

Keywords: air pollution, copper smelter, sulphur dioxide, atmospheric depositions.

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The air quality depends mostly on anthropogenic pollution sources, such as transport and industry. Metallurgical production of non-ferrous metals is a significant source of waste gases and particulate matter emitted in the environment. By melting ores with a high content of sulphur, the smelter emits sulphur dioxide (SO_2) in excessive concentrations, which poses threat to the environment and the humans [1,2]. Open pits and tailing ponds from which the coarser particles are distributed by wind represent additional sources of environmental pollution in the vicinity of the metallurgical complexes, consequently they can be rich in hazardous materials [3].

The basic components of biosphere are affected by atmospheric deposition of various pollutants. Atmospheric particles, depending on a size, can be settled by gravity close to the source of pollution, or can be carried by wind to various distances from the source. The most direct consequences of atmospheric deposition, both dry and wet, are acidification of soil and water, and accumulation of heavy metals in the biosphere [3,4].

Biomonitoring is used to monitor a number of pollutants, since it is a reliable way to determine connection between the air pollution in large areas and the temporal and spatial concepts at an affordable price. Information on atmospheric trace elements can be obtained by modelling their atmospheric dispersion and deposition, based on a-priori known emission sources, and by measuring the actual atmospheric occurrences and/or deposition [5]. Moss monitoring technique is an efficient method for the quantitative determination of atmospheric deposition of various pollutants and pollution sources [6–9].

Sulphur is an important nutrient. Plants compensate their need for S, absorbing it from soil in the form of sulphate ions, and partly by foliar absorption from the air. Discarded parts of plants (*e.g.*, leaves and fruits) increase the concentration of S in soil because they are subjected to decomposition and oxidation on the surface of soil. During that process, S is transformed to a form acceptable to plants [10]. However, Sun *et al.* [11] argue that the absorption of S from soil is well regulated and does not cause increased concentrations in plants. When the concentration of S in the soil is sufficient, the ambient air with excessive SO_2 concentration can have a toxic effect on vegetation

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[12]. The natural level of SO_2 in the air (about 5 μg/m^3) is not toxic to plants. However, concentrations of SO_2 which are below the maximum allowable concentrations safe for humans can cause serious damage to plants [10]. According to Jablanović *et al.* [10], the concentrations of SO_2 in the air below 50 μg/m^3 may be useful as a source of the necessary S, while concentrations above 100 μg/m^3 cause adverse effects, even to tolerant plant species.

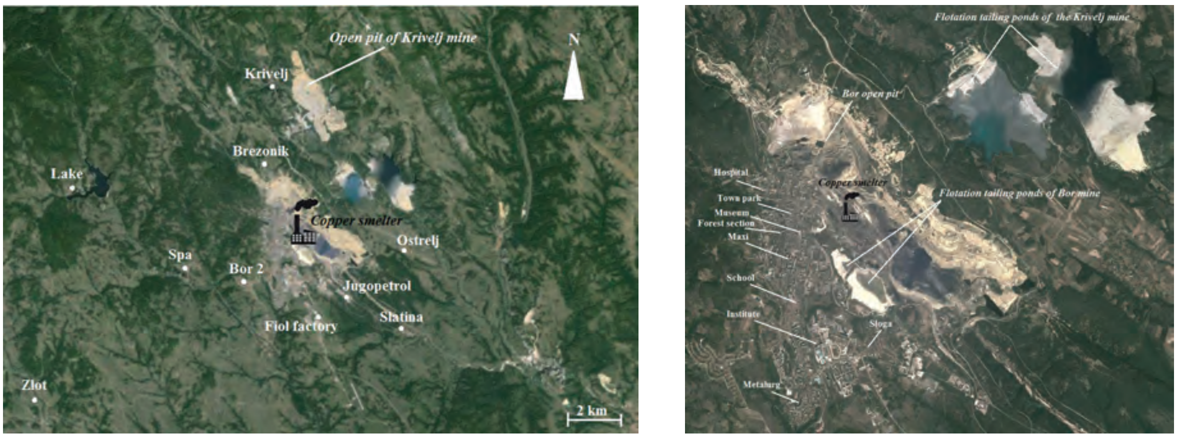
The primary sources of environmental pollution in Bor (Eastern Serbia) are mining and metallurgical processes (pyrometallurgical copper production from the sulphide ores $CuFeS_2$, Cu_2S and CuS). Besides primary emissions of waste gases from the copper smelter, secondary sources of pollution are open pits and tailing ponds situated in close vicinity of the town of Bor and surrounding villages (Figure 1), from which particles rich in sulphates are emitted. The Bor copper mine has started to work in 1903 after the discovery of abundant reserves of copper ores, which were exploited until 1993. In 1906, the first copper smelter started its operations. In the period 1961–1968, a new smelter was built, where sulphide copper ores are melted even today. Smelter waste gases containing on average 3–7% SO_2 are converted to H_2SO_4 in the sulphuric acid plant located within the Mining-Metallurgical Complex Bor. Since the plant provides accepting and processing less than 60% of the gases from the smelter, the rest of the gases are emitted untreated into the atmosphere [13].

The aim of this paper is to present air pollution data on ambient SO_2 concentrations, deposition rate of sulphates (SO_4^{2-}) from atmospheric depositions and total atmospheric depositions in the period of 2005–2008, as well as total S concentrations in soil and moss sampled in the vicinity of the Mining–Metallurgical Complex Bor during 2009.

EXPERIMENTAL

The measuring and sampling sites were selected depending on the dominant source of pollution (copper smelter), prevailing wind directions and the topography of the selected terrain. The studied area (Figure 1) includes four zones, two of which are situated in the close vicinity of the Complex (urban-industrial and rural zone) and the other two are not (background and tourist zone). Ambient SO_2 concentrations were measured at four sites in the urban-industrial zone: Town park, Jugopetrol, Institute and Brezonik. Total atmospheric deposition (TAD) and SO_4^{2-} were determined at 15 sites distributed in the urban-industrial zone (Hospital, Forest section, School, Institute, Sloga, Brezonik, Jugopetrol, Metalurg, Bor 2 and Foil factory), in the rural zone (Slatina, Krivelj and Ostrelj) and the tourist zone (Spa and Lake). Soil sampling sites were distributed in the urban-industrial (Town park and Hospital), the rural (Ostrelj and Slatina) and the background zone (Zlot), while moss was sampled in the urban-industrial (Town park, Institute, Museum and Maxi) and the background zone (Zlot).

Chemical analysis of the samples collected during the air quality monitoring program in the Bor area was performed in the Mining and Metallurgy Institute [14]. Ambient SO_2 concentrations were measured with stationary automatic and mobile measuring stations. At the sites, Town park and Jugopetrol, SO_2 concentrations were measured automatically by the UV fluorescent sulphur dioxide analyser (Model AF22M) according to the ISO standard (ISO 10498:2004). The method is based on detecting the characteristic fluorescence radiation emitted by SO_2 molecules. In the presence of a specific wavelength of UV light (214 nm), the SO_2 molecules reach a temporary excited electronic state. The subsequent relaxation produces fluorescence radiation, which is measured by a non-cooled photomultiplier tube. The analyser provides 15-min concentra-



(a) (b)

Figure 1. The map of the studied area: a) Eastern Serbia; b) the town of Bor.

tions of SO_2. The measurement is in the range 0–10000 μg/m^3. At the measuring sites, Institute and Brezonik, SO_2 concentrations were measured by the hydrogen peroxide method, based on the absorption of SO_2 from the air in the reagent containing 0.03 mol/dm^3 H_2O_2 at pH 5. The obtained solution is titrated with a standard solution (0.002 mol/dm^3 NaOH). The lower limit of detection for this method is 25 μg/m^3 [13].

Determination of total atmospheric deposition was conducted by the sedimentation method [15,16]. The method enables determination of liquid and solid fraction from depositions. During the period of 30±2 days, the particles were collected according to their ability to settle under their own weight. The sedimentator, mounted on 1.5 m-high tripods to avoid the collection of dust picked up by wind, consists of a funnel and a dry polyethylene cylindrical container. In the summer period, solution of copper(II) sulphate is added into the sedimentator in order to prevent algae growth. In the laboratory, solid fraction is separated from liquid by filtration. From the liquid fraction SO_4^{2-} concentrations are determined by the turbidimetric method. Total atmospheric depositions represent a sum of soluble and insoluble fraction given in milligrams per square metre per day (mg/m^2 per day).

Soil and moss samples were air dried first for 10 days at room temperature. In order to obtain concentrations on a dry matter basis, the samples were further dried in a dryer for a minimum of 24 h at 50 °C. The dried moss samples were ground in a laboratory mill into fine powder (average particle diameter less than 100 μm). To avoid contamination, the mill was thoroughly cleaned after each grinding. Soil samples were collected from the A-horizon (top soil 10–20 cm in depth) and stored in clean polyethylene bags. The soil samples were sieved through steel sieve, and then ground in the same way as the moss samples. After preparation, soil and moss material were digested according to the U.S. EPA method 3050B as in Piczak *et al.* [17]. Determination of total S concentration in the samples of soil and moss by ICP-AES and the quality control was performed at the Mining and Metallurgy Institute in Bor [18].

RESULTS AND DISCUSSION

Air pollution data

Taking into account the location of the Mining Metallurgical Complex and dominant wind directions, pollutants are spread over the town of Bor and the surrounding area [13] according to patterns given by the wind rose diagrams (Figure 2). From the shown 4-year period, it could be concluded that dominant winds in the studied area are in the West, East and South directions.

The average monthly SO_2 concentrations obtained at the four measuring sites in the urban-industrial zone of Bor in the period from 2005 to 2008 are given in Figure 3.

It can be seen that the highest monthly SO_2 concentration reached 450 μg/m^3 at the measuring site Jugopetrol in February 2005 (Figure 3a). At all the measuring sites during a 4-year period the lowest average SO_2 concentrations were detected at the sites

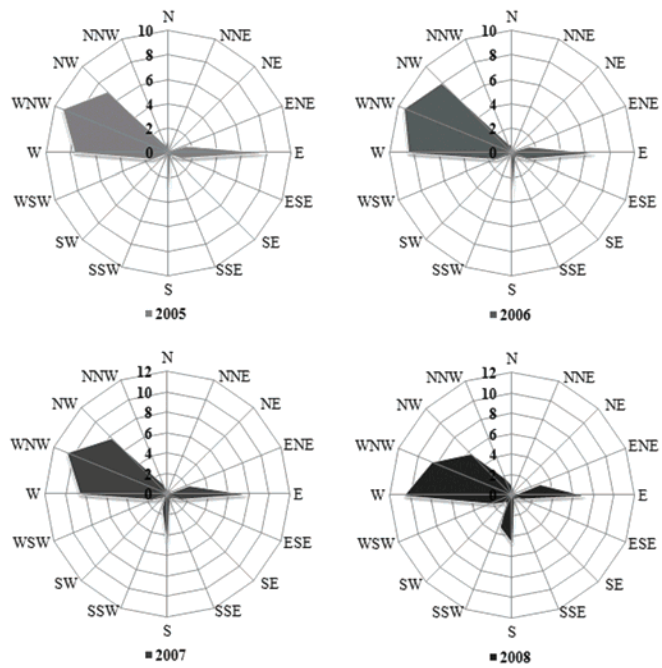


Figure 2. Wind rose diagram (%) for the study area in the period 2005–2008.

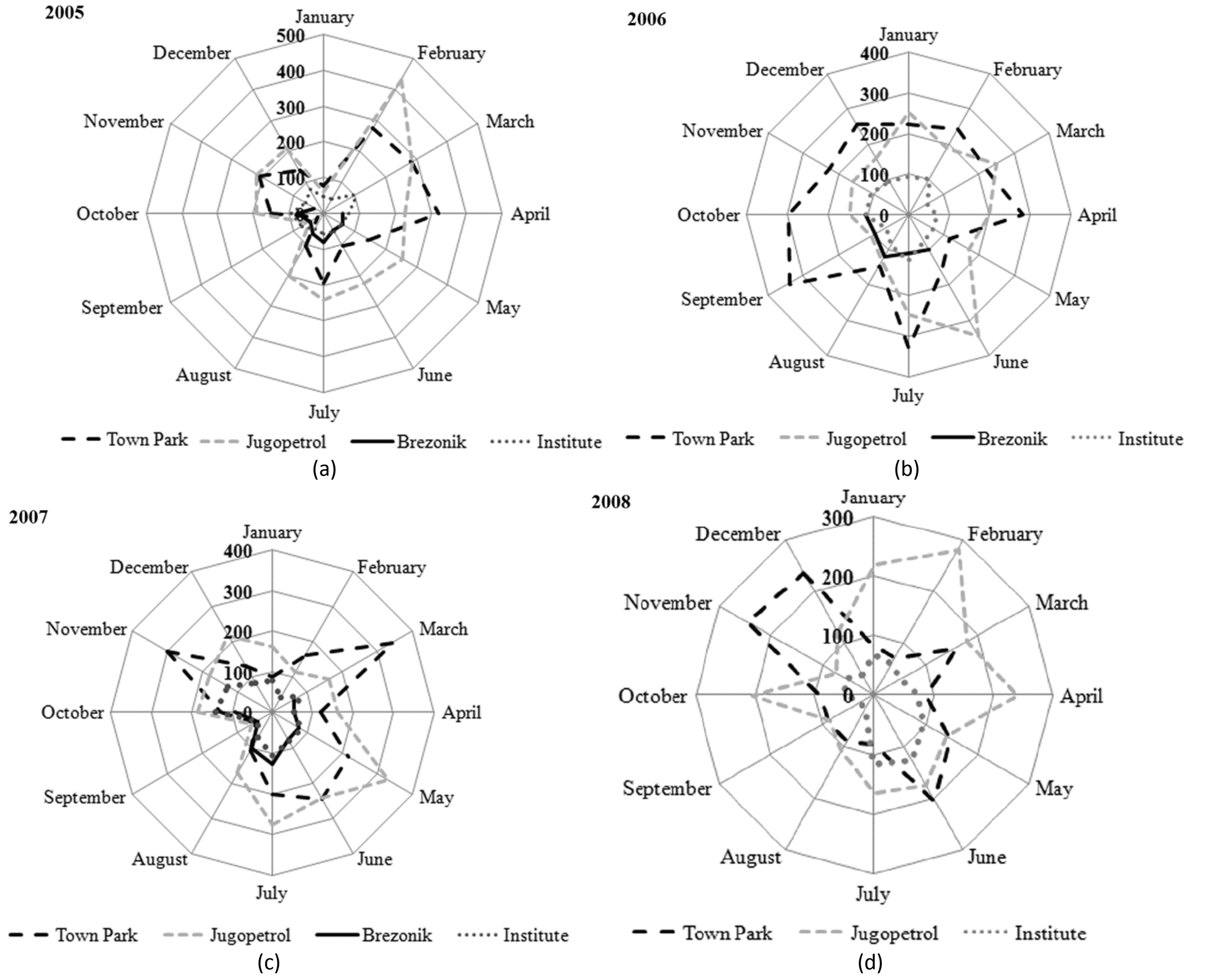


Figure 3. The average monthly SO_2 concentrations (μg/m^3) at the measuring sites Town park, Jugopetrol, Brezonik and Institute for a) 2005; b) 2006; c) 2007; d) 2008.

Institute and Brezonik, and the highest were at the sites Town park and Jugopetrol. The episodes of extremely high concentrations were probably a consequence of irregularities during the operation of the sulphuric acid plant when all the waste gases from the smelter were discharged without treatment into the atmosphere of Bor, as well as unfavourable winds (Figure 2). By comparing the average annual SO_2 concentrations (Table 1) with the current air pollution standard in Serbia [19], it can be said that the inhabitants of Bor live in the endangered environment, since only one annual SO_2 concentration (Institute, 2005) of all the measuring sites was within the maximum allowable value. Šerbula *et al.* [13] showed that ambient SO_2 concentrations mostly followed the anode copper production of the Complex. However, in some cases, the trend was not present and increasing copper production was accompanied by decreasing SO_2 concentrations and vice versa, which was a result of frequent stoppages of the copper smelter and limited capacities of the sulphuric acid plant.

Table 1. The average annual SO_2 concentrations (μg/m^3) in the urban-industrial zone of Bor in the period 2005–2008; maximum allowable concentration for residential areas according to the Serbian Regulation No. RS 75/10 [19]; 50 μg/m^3

Measuring site	2005	2006	2007	2008[a]
Town park	169	238	175	112
Jugopetrol	215	199	189	177
Institute	49	86	82	71
Brezonik	58	104	91	–

[a]Measurements for the period January–September

The Table 2 shows the average annual deposition rates of total atmospheric depositions and SO_4^{2-}. Based on the annual deposition rates of the TADs, it can be said that the maximum allowable value (MAV) given by the Serbian Regulation [19] exceeded only at the measuring sites Slatina (during 2007 and 2008) and Forest section (during 2007). Also, at the site School, during the course of 2008, deposition rate was 199.1 mg/m^2 per day, which is close to the MAV. In general, the

Table 2. The average annual deposition rates (mg/m² per day) of total atmospheric depositions (TAD) and sulphates from soluble fraction at 15 measuring sites in Bor and the surroundings for period 2005–2008; values above the maximum allowable value (MAV) shown in bold

Measuring site	2005		2006		2007		2008[a]	
	TAD	SO_4^{2-}	TAD	SO_4^{2-}	TAD	SO_4^{2-}	TAD	SO_4^{2-}
Institute	38.1	6.2	113.4	21.5	117.1	14.6	81.7	12.8
Jugopetrol	49.3	10.1	106.7	33.7	137.8	7.8	102.7	11.3
Brezonik	57.3	5.9	116.7	19.9	178.3	15.8	173.8	13.6
Hospital	64.1	7.4	171.0	49.0	187.6	16.9	193.3	39.9
Forest section	70.0	7.2	171.0	38.6	**212.5**	15.0	171.9	22.8
School	48.0	6.5	124.1	27.0	188.5	21.9	199.1	20.6
Metalurg	30.4	3.2	99.9	24.7	116.8	12.2	89.4	13.6
Spa	28.9	2.6	78.6	15.1	74.8	5.6	119.1	17.7
Foil factory	39.7	6.1	117.8	30.3	97.0	4.6	89.8	13.5
Bor 2	35.2	3.3	95.2	22.2	153.1	42.1	105.4	13.5
Lake	35.2	3.1	91.2	20.8	153.4	18.4	117.7	9.5
Slatina	49.6	6.5	141.1	30.3	**240.6**	17.2	**211.3**	18.0
Krivelj	50.6	5.6	123.2	16.1	125.0	12.3	100.1	7.5
Ostrelj	55.5	10.2	139.2	23.9	110.1	3.6	162.6	12.2
Sloga	44.7	8.9	145.7	28.3	144.3	9.4	171.4	16.5
MAV	200	–	200	–	200	–	200	–

[a]Measurements for the period January–September

lowest deposition rates at all the measuring sites were observed in 2005, compared to the rest of the studied period.

The highest deposition rate of sulphates from atmospheric deposition was noted at the measuring sites in the close vicinity of the Complex and in the prevailing wind directions. Also, in 2006, the average SO_4^{2-} deposition rate was higher compared to the values in 2005 and 2007. Considering that the copper smelter is a dominant source of air pollution in the studied area, increased anode copper production could cause a higher level of ambient pollution. During 2005, 2006, 2007 and 2008 the average anode copper production in the Mining–Metallurgy Complex was 3539.5, 3897.5, 3257.5 and 3346.8 t, respectively. However, the maximum allowable value for sulphates from atmospheric deposition is not defined by the Serbian Regulation [19] or by the European Environmental Agency.

In the study of Moreno-Grau *et al.* [15] where the sources of air pollution are mining and metallurgical activities, the deposition rates of the TADs on an annual base were: 787.5 (detected in the urban-industrial zone), 331.3 (urban zone), 386.4 mg/m² per day (intermediate zone). Compared to these values, the TADs deposition rates in the Bor area were a few times lower.

In Figure 4, box plots summarize the average

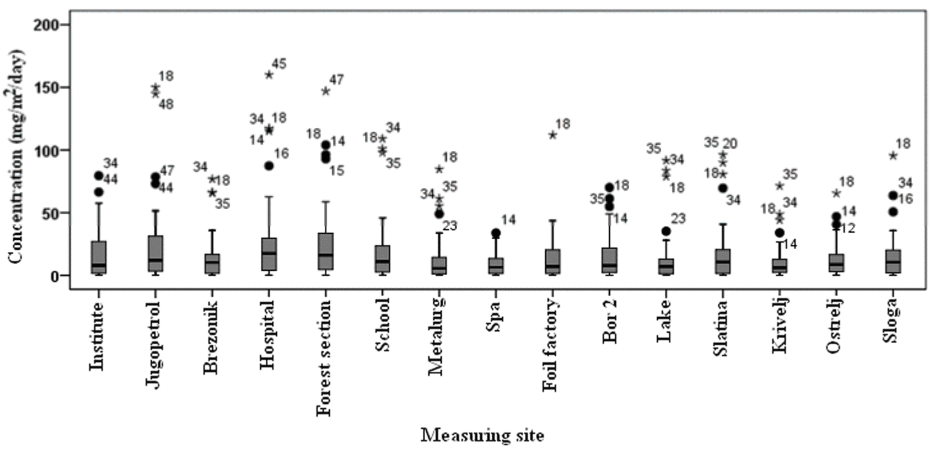


Figure 4. Box plots based on the average monthly sulphate deposition rates at 15 measuring sites in Bor and surroundings in the period 2005–2008 (numbers represent months of 4-year period, N = 48).

monthly SO_4^{2-} deposition rates at 15 measuring sites in the studied area. The upper and lower lines of the "boxes" represent lower and upper quartiles (25 and 75% of the data set, interquartile range), while middle line is called median and it represents the middle of the data set (50%). The T-bars that extend from the boxes are called whiskers and represent minimum and maximum values of the data set, excluding outliers. Outliers are the values that are ≥1.5 times greater than the interquartile range (marked with a dot) or ≥3 times greater than the interquartile range (marked with asterisks – extreme values). A few extreme values were detected at the sites Hospital (during September 2008), Forest section (November 2008) and Jugopetrol (June 2006 and December 2008). The measuring sites Hospital and Forest section are located in less than 1 km from the copper smelter, while the Jugopetrol site is situated in the dominant West wind direction. At the measuring site Spa the lowest level of pollution with sulphates is observed in the presented 4-year period.

Biomonitoring data

Concentrations of total S in soil and moss at the sampling sites in the urban-industrial, rural and background zone are given in Tables 3 and 4, respectively. The maximum concentration of S in soil was detected in the rural area at the sampling site Ostrelj (>1200 µg/g), which is probably a consequence of proximity of Bor and Krivelj tailing ponds and unfavourable winds.

Table 3. Average total S concentrations (µg/g of dry weight) in soil samples at the sampling sites in three zones

Urban-industrial		Rural		Background
Town park	Hospital	Ostrelj	Slatina	Zlot
848.7	687.5	1230.3	817.0	561.5

Table 4. Average total S concentrations (µg/g of dry weight) in moss samples at the sampling sites in two zones

Urban-industrial				Background
Town park	Museum	Maxi	Institute	Zlot
7363.0	7009.0	7196.0	3674.0	1482.0

The concentration of total S in moss, sampled in the urban-industrial zone is significantly higher than the concentrations in the background zone and at the Institute site which is not in the direction of the dominant winds. According to Sucharova and Suchara [6], a typical S content in mosses is in the range of 1200–1400 µg/g, which is far lower than the presented concentrations in the urban-industrial zone of Bor. Only the concentration obtained at the sampling site Zlot (the background zone) is within the typical range for sulphur concentrations [6]. The other obtained results are in agreement with Sucharova and Suchara [6], showing that the moss is an efficient bioindicator of ambient pollution by sulphur dioxide. Availability of moss was reduced in the studied area, but obviously certain types of mosses can survive a high level of air pollution, contrary to conclusions of Reimann *et al.* [20].

CONCLUSION

The main air pollution problems in the studied area of Bor and its surroundings originate from copper smelter, open pits and tailing ponds. Waste gases (which contain 3–7% SO_2) and atmospheric depositions (rich in sulphates and heavy metals) are continuously emitted in the atmosphere from these sources. The results presented in the paper are an outcome of a 4-year program (from 2005 to 2008) of air quality monitoring in Bor, which was conducted by the Mining and Metallurgy Institute, combined with biomonitoring data. The measuring sites of ambient SO_2 concentration are distributed in urban-industrial zone, while total atmospheric deposition and SO_4^{2-} sites are distributed in three zones: urban-industrial, rural and tourist, with the emphasis on the urban-industrial zone since it is closest to the copper smelter. Biomonitoring by moss was conducted at four sampling sites in the urban-industrial zone and in the background, while soil samples were taken from three zones. Although the measuring and sampling sites do not overlap completely due to different monitoring networks, measuring methods and availability of samples, some conclusions can be made from the obtained data sets. According to the average 4-year concentrations of SO_2 and deposition rates of the TADs, the level of pollution is decreasing from the Town park (*i.e.*, Forest section for TADs is the closest to the SO_2 site Town park) and Jugopetrol, towards Brezonik and Institute, which can be an evidence of the same pollution source of gaseous and particulate pollution. Higher level of pollution at these sites had an obvious influence on the total S concentrations in moss samples. Moss sampling sites Museum and Maxi are close to the measuring site Town park, where total S concentrations exceeded 7000 µg/g. Similar to the air pollution data, at the sampling site Institute total S concentration was lower. Significantly higher concentrations of total S in mosses from the urban-industrial zone compared to the background indicate its anthropogenic origin and a good response of site-available moss to the airborne pollution. The maximum concentration of total S in soil was detected in the rural area (Ostrelj), which is probably a consequence of proximity of Bor and Krivelj tailing ponds and unfavourable winds. In general, higher concentrations of the monitored polluting substances were noted at the sites closer to the copper smelter and/or at the prevailing wind directions.

It should be noted that during 2015 a new smelter, based on the autogenous flash smelting technology, should be operational within the Mining– Metallurgy Complex. The obtained data provides vital documentation of the current state of the environment in order to assess the efficiency of the planned technical measures to reduce pollutant emissions in the future in the studied area.

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IZVOD

EMISIJA SO_2 I SO_4^{2-} IZ TOPIONICE BAKRA I NJIHOV UTICAJ NA NIVO UKUPNOG S U ZEMLJIŠTU I MAHOVINI U BORU I OKOLINI

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(Naučni rad)

Područje Bora i okoline poznati su po eksploataciji i preradi sulfidnih ruda bakra više od jednog veka. Tokom dugogodišnje proizvodnje bakra emitovane su u atmosferu velike količine otpadnih gasova i čvrstih čestica sa visokim sadržajem teških metala (karakteristično za pirometaluršku proizvodnju bakra) što ima negativan uticaj na celokupnu životnu sredinu. U radu su predstavljeni podaci o zagađenju vazduha za period 2005–2008. godina, i to: ambijentalne koncentracije sumpor-dioksida (SO_2) na četiri merna mesta u urbano-industrijskoj zoni, brzine taloženja ukupnih taložnih materija (UTM) i sulfata (SO_4^{2-}) iz rastvornih materija na 15 mernih mesta u urbano-industrijskoj, ruralnoj i turističkoj zoni. Takođe, prikazane su koncentracije ukupnog sumpora (S) u uzorcima zemljišta i mahovine prikupljenih u septembru 2009. godine na mestima u urbano-industrijskoj, ruralnoj i kontrolnoj zoni ispitivanog područja. Izabrana merna mesta i mesta uzorkovanja nalaze se na pravcima dominantnih vetrova i na različitim udaljenostima od glavnog izvora zagađenja – topionice bakra. Rezultati dugoročnih merenja zagađenja vazduha ukazuju na povećani nivo sumpor-dioksida u Boru u odnosu na vrednost maksimalno dozvoljene koncentracije SO_2 u naseljenim područjima definisane pravilnikom. Prosečne godišnje koncentracije SO_2, izuzev na mernom mestu Institut, tokom ispitivanog perioda višestruko su premašivale maksimalno dozvoljenu vrednost (50 $\mu g/m^3$), što ukazuje na visok stepen zagađenja životne sredine. Maksimalno dozvoljene vrednosti za brzinu taloženja ukupnih taložnih materija na godišnjem nivou bile su prekoračene samo na dva od 15 mernih mesta (Šumska sekcija u urbano-industrijskoj zoni i Slatina u ruralnoj). Najviša prosečna godišnja brzina taloženja sulfata iz ukupnih taložnih materija detektovana je u urbano-industrijskoj zoni. Maksimalno dozvoljena vrednost za ove materije nije definisana pravilnikom, pa se ne može sa sigurnošću tvrditi o zagađenju životne sredine sulfatima, ali je očigledno povećano taloženje na mestima u blizini rudarsko–metalurškog kompleksa. Veće koncentracije ukupnog sumpora u uzorcima zemljišta detektovane su na mestu uzorkovanja Oštrelj u ruralnoj zoni, što je najverovatnije posledica blizine flotacijskih jalovišta borskog i kriveljskog rudnika. Značajno veće koncentracije ukupnog sumpora u uzorcima mahovine u urbano-industrijskoj zoni (preko 7000 mg/kg) u poređenju sa kontrolnom zonom (1482 mg/kg) potvrđuju da je mahovina dobar bioindikator zagađenja vazduha sumpor-dioksidom.

Ključne reči: Zagađenje vazduha • Topionica bakra • Sumpor-dioksid • Atmosferska depozicija