CHEMICAL COMPOSITION, LEVELS, AND I/O RATIOS OF PM_{10} AND $PM_{2.5}$ IN THE LABORATORY NEAR THE COPPER SMELTER IN BOR, SERBIA

by

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Numerous studies have reported that particulate matter (PM) and its specific chemical constituents were linked to the incidence of respiratory diseases and mortality and lung function. The main aim of this study is to determine metallic content in PM_{10} and $PM_{2.5}$ samples taken simultaneously at several locations in the close vicinity of the copper smelter in Bor and the influence of outdoor PM pollution on indoor PM levels. The measurement campaign was conducted, during the non-heating season of 2020. The PM samples were collected at all sampling sites with low-volume samplers (Sven/Leckel LVS3) on quartz fiber filters (Whatman QMA, 47mm). All samples were analyzed by inductively coupled plasma mass spectrometry (ICP MS). In this way, the mass concentrations of four priority elements (As, Cd, Pb, and Ni) in PM samples were identified and quantified. It has been determined that average indoor PM levels in the laboratory were higher than outdoors. A strong correlation was found between PM_{10} and $PM_{2.5}$ particle levels inside the laboratory and in the outdoor air. Also, a very strong correlation was found between the levels of Pb, Ni, As, and Cd determined in PM_{10} and $PM_{2.5}$ samples inside the laboratory and in the outdoor air. This confirms that mentioned elements originate from the same sources located in the copper smelter complex.

Key words*: air quality, monitoring, PM, carcinogenic elements, indoor air*

Introduction

Numerous studies have reported that PM and its specific chemical constituents were linked to the incidence of respiratory diseases and mortality and lung function [1-3]. Transition metals present in PM are able to damage DNA, induce mutations, and initiate carcinogenesis [4, 5]. However, the quantity of every single metal in PM does not depend only on the magnitude of the source, but also on weather conditions, meteorological factors, such as: wind direction and intensity, spread, and dilute or even accumulate metals in breathable air [6]. The

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relationship between pollutant concentrations in the atmospheric environment and meteorological factors has been reported by numerous papers [7-9]. Agency for Toxic Substances and Disease Registry (ATSDR) Priority List places arsenic and lead as the most significant potential threat to human health due to their known toxicity and potential for human exposure [10].

Arsenic is released into the environment from a variety of natural and anthropogenic sources. Typical concentrations of arsenic range from 1-10 ng/m^3 in rural areas and up to 30 ng/m^3 in uncontaminated urban areas [11]. A large part of the As anthropogenic emission comes from the metal processing industry. The anthropogenic emission of As is several times higher than the natural [11].

Average Pb concentrations in the ambient air are typically below 0.15 μg/m^3 at the rural sites and between 0.15 and 0.5 μg/m^3 in the urban ambient air of the EU cities. Lead in the PM originates from heavy industry, coal burning, metallurgical smelters, and traffic [12]. Average Cd levels in the ambient air ranged from 0.2-2.5 ng/m^3 at urban and traffic-related sites, and up to 20 ng/m^3, at industrial sites. The Cd in PM originates from coal and fuel oil combustion processes, the metallurgical industry, and road transport [11]. Average Ni levels in the ambient air ranged from 1.4-13 ng/m^3 in urban and traffic-related areas, and up to 50 ng/m^3 in the areas near industry [11].

The city of Bor is located in a mountainous forest area in the southeastern part of Serbia, in the direction of the border between Romania, Bulgaria, and Serbia. It has a total population of about 50000 inhabitants. The main economic activity comprises mining and metal processing. Bor Municipality area is under the constant influence of air pollution as the consequence of technological processes in the Copper Smelter Complex Bor located close to the urban and residential areas, as shown in fig. 1.

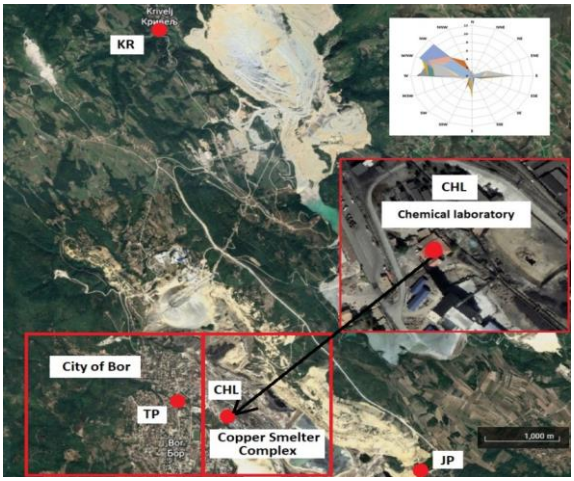

Figure 1. Location of measurement sites (red spots: KR-Krivelj, TP-Town Park, JP - Jugopetrol, and CHL -Chemical Laboratory) relative to the Copper Smelter Complex and urban areas of Bor city

The main aim of this study is to determine selected chemical elements content in PM_{10} and $PM_{2.5}$ as well as to determine the influence of outdoor PM pollution on indoor PM levels. Samples were taken simultaneously in the Chemical Laboratory (CHL), located inside a fenceline of the Copper Smelter Complex Bor (shown in fig. 1), and in outdoor air near the CHL. For this purpose, four priority elements (As, Pb, Ni, and Cd) in PM_{10} and $PM_{2.5}$ are identified and quantified. The measurement campaign was conducted during the no-heating period of 2020.

Materials and methods

Sampling locations and measurements equipment

The measurement campaign was conducted from June 15th, 2020 to July 1st, 2020, and from September 18th to October 12th, 2020. Measuring point CHL is situated inside a

fenceline of the Copper Smelter Complex Bor (44° 04' 29" N, 22° 06' 26" E), Republic of Serbia. Measuring point KR is located in the village of Krivelj, about 6 km north of the copper smelter. The position of this measuring point is downwind in relation to the copper smelter when the winds blow from the south. Near this measuring point is the surface mine Veliki Krivelj, as shown in fig. 1. Measuring point TP is located downwind of the E wind from the copper smelter. This location is about 650 m W from the copper smelter. Measuring point JP is located about 3 km S-SW from the copper smelter. The position of this measuring point is downwind in relation to the copper smelter when the winds blow from the N-NW direction. Near this measuring point (1 km NE) is the city landfill.

The 24-hour PM samples were collected simultaneously (10 a. m.-10 a. m.) with the low-volume samplers (Sven/Leckel LVS3) on quartz fiber filters (Whatman QMA, 47 mm). Before and after exposure, filters were preconditioned and weighed three times following the procedure proposed in SRPS EN 12341:2015 [13]. The loaded filters, after gravimetric measurements, were further prepared for chemical analyses in accordance with the procedure of SRPS EN 14902:2008/AC:2013 [14].

Four LVS3 reference samplers were used in the campaign at the CHL site. Two PM samplers (carrying PM_{10} and $PM_{2.5}$ sampling heads) were placed in the laboratory. The other two PM samplers were placed outside, in the laboratory yard. The laboratory has a volume of 120 m^3, a windows surface of 4 m^2, and only one door. During the campaign, all the windows were usually closed. The laboratory floor is made of concrete. The laboratory has a ventilation system that was in operation during the measurement campaign. The flow rate of all LVS3 samplers (38.3 Lpm) was calibrated using certified flow meters at the beginning of the measurement campaign. At the other measuring sites: KR, TP, and JP, only outdoor PM_{10} concentrations were measured and analyzed. These places represent, in fact, control measurement places that were included to show the difference in the concentrations of the observed elements in PM_{10} samples within the copper smelter complex and in the urban parts of the city of Bor.

Chemical analysis

The samples were analyzed by Inductively Coupled Plasma Mass Spectrometry (ICP MS Agilent 7700) [15]. The detection limits for As, Pb, Cd, and Ni determined by the ICP MS method were 0.1 ng/m^3, 0.5 ng/m^3, 0.02 ng/m^3, and 0.7 ng/m^3, respectively. The limit of detection (LOD) assessment was done by calculating the standard deviation of repeated measurements of laboratory blank filter (n = 10) impurities and multiplied by factor three (LOD = 3 × SD, where SD is the standard deviation). The accuracy of the method was determined according to the standard SRPS EN 14902:2008/AC:2013: EN by measuring the Certified Reference Material (CRM Urban dust-NIST 1648) [16] and comparing the determined values with certified values [17]. Recoveries were in the range of 80% to 120% for all measured chemical elements. In this way, the mass concentrations of four trace elements (As, Cd, Pb, Ni) in PM_{10} and $PM_{2.5}$ samples are identified and quantified.

Results and discussion

According to the legislative of the Republic of Serbia [18], the prescribed daily limit value for PM_{10} concentration is 50 μg/m^3 not to be exceeded more than 35 times per calendar year and annual average value for PM_{10} concentration is 40 μg/m^3. It is also the prescribed annual average limit value for $PM_{2.5}$ concentration of 25 μg/m^3. The same regulation prescribes a daily limit for Pb content in PM_{10} of 1000 ng/m^3 and annual average limits for Pb, Cd, Ni, and As contents in PM_{10} of 500 ng/m^3, 5 ng/m^3, 20 ng/m^3, and 6 ng/m^3, respectively.

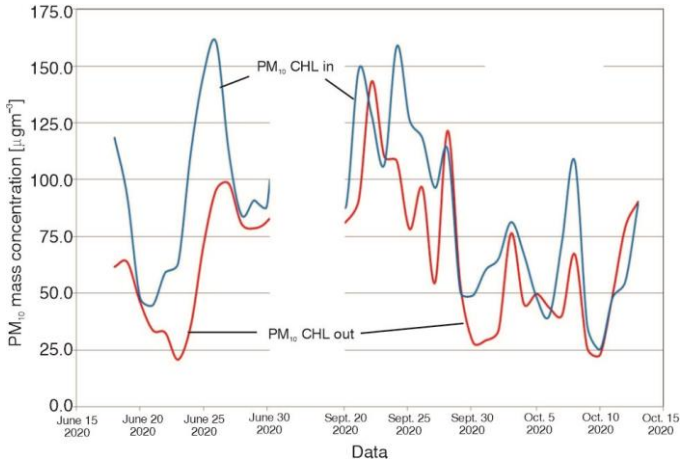

Figure 2. Average daily PM_{10} levels determined at CHL during the measurements campaign

Figure 2 shows a line diagram of indoor and outdoor PM_{10} concentrations determined at CHL during the measurements campaign.

According to data shown in tab. 1, in the studied period, PM_{10} levels at CHL were usually above the prescribed daily limits. In addition, according to the results of indicative measurements [18] obtained in this research, average levels of As, Pb, Ni, and Cd determined in outdoor PM_{10} samples at CHL were higher than prescribed annual target values (As average values were 39 folds higher than the annual target value, while Pb, Cd, and Ni average values were s 2.1, 1.2, and 6.8 folds higher than the annual target values, respectively).

Table 1. Summary statistic of PM_{10} levels and chemical content of As, Pb, Ni, and Cd (standard deviation – SD, coefficient of correlation, *R*) in PM_{10} samples at CHL

	In (n = 36)		Out (n = 36)		I/O ratio		R (in *vs.* out)
	Average	SD	Average	SD	Average	SD	
				[μgm^{-3}]			
PM_{10}	86.4	37.0	64.9	30.8	1.4	0.6	0.727**
				[ngm^{-3}]			
As	232.7	400.8	263.2	480.9	1.2	0.7	0.991**
Pb	1245.8	1457.0	1063.0	1434.6	1.4	0.6	0.974**
Ni	24.6	17.1	23.9	16.8	1.0	0.2	0.929**
Cd	33.6	61.1	34.1	61.9	1.1	0.5	0.986**

**. Correlation is significant at the 0.01 level (2-tailed)

The PM_{10} levels in outdoor air at CHL were higher than the daily limit value during 58.3% of measurement days (21/36). The number of days exceeding the daily limit value for Pb concentration in PM_{10} at CHL in outdoor air was 27.8% (10/36).

The I/O ratio of PM concentration is often used to justify the presence of indoor sources (I/O>1) or infiltration of ambient air (I/O≤1). PM_{10} and Pb, As, Cd and Ni contained in PM_{10} average daily I/O ratios were 1.4, 1.4, 1.2, 1.1 and 1.0, respectively, tab. 1. From total of 36 days daily I/O ratios were higher than 1 during 27 days for PM_{10} and Pb, in 20 day for As and Ni, while for 16 for Cd.

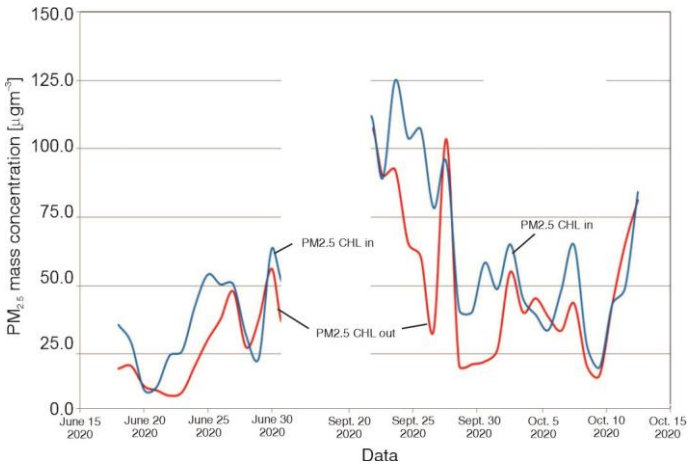

Figure 3. Average daily $PM_{2.5}$ levels determined at CHL during the measurements campaign

Strong correlations ($0.8 > R > 0.6$) between indoor and outdoor PM_{10} concentrations were found. A very strong correlation ($R > 0.8$) between indoor and outdoor concentrations of As, Pb, Ni, and Cd determined in PM_{10} samples at CHL was found. Such values of R indicate that most of these elements determined in PM_{10} samples originate from the same source. Figure 3 shows a line diagram of indoor and outdoor $PM_{2.5}$ concentrations at CHL determined during the measurements campaign.

The $PM_{2.5}$ average level in indoor air at CHL was more than twice higher than the annual average limit value, while it was 1.67 times higher than in outdoor air at CHL, tab. 2.

For $PM_{2.5}$ and Pb, As, Cd, and Ni in $PM_{2.5}$ average daily I/O ratios were 1.4, 1.4, 1.3, 1.1, and 1.0, respectively, tab. 2. From total of 36 days average daily I/O ratios were higher than 1 in 28 days for $PM_{2.5}$, 29 day for Pb, 22 days for As, and 18 day from 36 for Ni.

Strong correlations between indoor and outdoor $PM_{2.5}$ concentrations were found. A very strong correlation between indoor and outdoor concentrations of As, Pb, Ni, and Cd determined in $PM_{2.5}$ samples was found. Such values of R indicate that most of these elements determined in $PM_{2.5}$ samples originate from the same source.

Table 2. Summary statistic of $PM_{2.5}$ levels and content of As, Pb, Ni, and Cd (standard deviation – SD, coefficient of correlation, *R*) in $PM_{2.5}$ samples at CHL

	in (n = 36)		out (n =3 6)		I/O ratio		R
	Average	SD	Average	SD	Average	SD	(in *vs.* out)
[μgm^{-3}]							
$PM_{2.5}$	53.6	28.9	41.8	26.4	1.4	0.5	0.851**
[ngm^{-3}]							
As	211.8	374.8	213.5	401.4	1.3	0.9	0.994**
Pb	1104.0	1415.6	919.2	1284.1	1.4	0.5	0.979**
Ni	21.1	15.5	21.5	16.5	1.0	0.2	0.888**
Cd	31.9	58.8	31.1	59.8	1.1	0.4	0.994**

**. Correlation is significant at the 0.01 level (2-tailed)

Average daily ratios for $PM_{2.5}/PM_{10}$ in indoor and outdoor air at CHL were very similar (0.7), tab. 3. This is in good correlation with the typical values of this ratio for urban

parts of the city of Bor (0.5-0.6) [19], and the fact that at the majority of European stations this ratio is around 0.65 (range from 0.42 to 0.82) [20]. Also, the same conclusion applies to the ratios of the content of the observed elements in the $PM_{2.5}$ fraction and in the PM_{10} fraction in the indoor and outdoor air around CHL.

The comparison of PM_{10} levels and content of selected elements in PM_{10} samples in the city of Bor during the measurements campaign

In order to get a wider picture of the spatial distribution of air pollution by PM_{10} particles and the content of PM_{10} samples, additional data have been analyzed from the measurement sites that belong to the local air quality monitoring network KR, TP, and JP, fig. 1. Table 3 shows comparative results for PM_{10} outdoor samples from the available measuring sites in the local air quality monitoring network in the city of Bor during the measurement campaign. The characteristics of the listed measurement points in tab. 4 are provided in Mining and Metallurgy Institute Bor annual reports about quality of ambient air in Bor [21].

Table 3. Summary statistic of indoor and outdoor ratios of $PM_{2.5}/PM_{10}$ levels and ratios of content of As, Pb, Ni, and Cd in $PM_{2.5}$ *vs.* content of As, Pb, Ni, and Cd in PM_{10} at CHL

Ratio	$PM_{2.5}$ in/ PM_{10} in	Pb $PM_{2.5}$ in/ Pb PM_{10} in	Cd $PM_{2.5}$ in/ Cd PM_{10} in	Ni $PM_{2.5}$ in/ Ni PM_{10} in	As $PM_{2.5}$ in/ As PM_{10} in
Min	0.3	0.2	0.1	0.6	0.4
Max	0.9	1.0	1.0	1.0	1.0
Average	0.7	0.7	0.8	0.8	0.8
SD	0.2	0.2	0.2	0.1	0.2

Ratio	$PM_{2.5}$ out/ PM_{10} out	Pb $PM_{2.5}$ out/ Pb PM_{10} out	Cd $PM_{2.5}$ out/ Cd PM_{10} out	Ni $PM_{2.5}$ out/ Ni PM_{10} out	As $PM_{2.5}$ out/ As PM_{10} out
Min	0.3	0.3	0.3	0.6	0.4
Max	0.9	0.9	0.9	1.0	0.9
Average	0.7	0.8	0.8	0.9	0.8
SD	0.2	0.1	0.1	0.1	0.1

Table 4. Average daily outdoor PM_{10} levels and content of As, Pb, Ni, and Cd at CHL, KR, TP, and JP during the measurements campaign

	CHL out	KR out	TP out	JP out
PM_{10} ($\mu g/m^3$)	64.9	23.1	28.3	26.4
As (ng/m^3)	263.2	3.9	15.3	76.2
Pb (ng/m^3)	1063.0	4.3	63.5	383.5
Ni (ng/m^3)	24.0	5.4	11.0	21.9
Cd (ng/m^3)	35.5	0.1	1.5	9.2

There was almost no precipitation during the measurement campaign (1 rainy day, 18 mm/m^2), and the average wind speed was 1.3 m/s. Such meteorological conditions do not favor the removal of air pollution, but on the contrary, accelerate the deposition of PM_{10} particles near the source of PM pollution. This is the main reason that according to data shown in

Table 4, average levels of PM_{10} in the wider area around the copper smelter were more than two folds lower than inside the Copper Smelter Complex fenceline.

The PM_{10} levels as well as the levels of the selected elements in PM_{10} are the lowest at the measuring site KR concerning the other measuring sites as it is shown in tab. 4. This is because this measuring site, compared with the other measuring sites, is the furthest from the copper smelter (as shown in fig. 1), as the main source of heavy metals in the PM.

Measuring sites TP and JP are at the dominant wind directions relative to the copper smelter (especially JP as shown in fig. 1), also those measuring points are closer to the copper smelter in comparison with the measuring point KR, so that, the levels of the selected elements in PM_{10} are higher from those levels recorded at the measuring point KR. Pearson correlation coefficients between the outdoor PM_{10} levels and levels of selected chemical elements at CHL and the other measuring sites are presented in tab. 5.

Table 5. Correlation coefficients between the outdoor PM_{10} levels and levels of selected chemical elements in PM_{10} samples at CHL and at the other measuring sites (KR, TP, and JP)

	KR PM_{10}	TP PM_{10}	JP PM_{10}
CHL PM_{10}	0.522**	0.621**	0.422*
	KR As	TP As	JP As
CHL As	0.393*	0.845**	0.811**
	KR Pb	TP Pb	JP Pb
CHL Pb	0.508**	0.706**	0.693**
	KR Ni	TP Ni	JP Ni
CHL Ni	–0.263	–0.009	–0.145
	KR Cd	TP Cd	JP Cd
CHL Cd	0.406*	0.825**	0.698**

**. Correlation is significant at the 0.01 level (2-tailed).
*. Correlation is significant at the 0.05 level (2-tailed)

A strong correlation ($0.8 > r > 0.6$) were found between CHL PM_{10} and TP PM_{10}. A moderate correlation ($0.6 > r > 0.4$) was found between CHL PM_{10} and KR PM_{10} and between CHL PM_{10} and JP PM_{10}. Such values of the correlation coefficient indicate that a significant part of the PM_{10} particles at each observed measuring site originates from the same source.

A very strong correlation was found between CHL As and TP As and CHL As and JP As. On the contrary, a weak ($0.4 > r > 0.2$) correlation was determined between CHL As and KR As.

Such values of the correlation coefficient indicate that As detected in PM_{10} samples at CHL have the same origin as As detected in PM_{10} at TP and JP mostly because those measuring points are located on the dominant wind directions relative to the copper smelter, whilst measuring point KR is not at the dominant wind direction relative to the copper smelter.

A strong correlation was found between CHL Pb and TP Pb and CHL Pb and JP Pb and a moderate correlation was determined between CHL Pb and KR Pb. Such values of the correlation coefficient indicate that a significant part of Pb detected in PM_{10} at CHL has the same origin as Pb detected in PM_{10} samples at other measuring sites around the copper smelter.

A very weak negative correlation between CHL Ni and Ni levels at other measurement sites was determined. Such values of the correlation coefficient indicate that most of the Ni determined in PM_{10} samples at remote sites has no common origin as Ni in PM_{10} samples at the CHL site.

A very strong correlation was found between CHL Cd and TP Cd. A strong correlation was found between CHL Cd and CHL JP. On the contrary, a moderate correlation was determined between CHL Cd and KR Cd. Such values of the correlation coefficient indicate that a significant part of Cd detected in PM_{10} samples at CHL has the same origin as Cd detected in PM_{10} samples at TP and JP. The main reason for such distribution of Cd is that TP and JP sites are located in the dominant wind direction relative to the copper smelter, whilst

measurement site KR is not in the dominant wind direction relative to the copper smelter and is at the longest distance from the smelter in the comparison with the TP and JP sites.

Conclusions

The content of suspended particles of the PM_{10} and $PM_{2.5}$ fractions inside and outside the CHL shows that a significant part of the air pollution from the external environment reaches the laboratory. Of particular concern is that levels of PM_{10} and most of the selected elements detected in PM_{10} samples are several times higher near point sources in the smelter than at a distance of a few hundred meters far from the copper smelter fenceline. These results indicate that a significant part of PM particle emissions, enriched with heavy metals and other carcinogenic elements are not included in the waste gas purification systems in the copper smelter. For this reason, the exposure of copper smelter workers to PM pollution is many times higher than the exposure of the population in urban parts of the city of Bor.

The constant air pollution with As, Cd, Ni, and Pb in PM_{10} particles, sometimes in concentrations even several tens of times higher than the target annual concentration values prescribed for these elements in PM_{10} requires urgent actions to reduce anthropogenic emission of suspended particles in Bor, eg. coverage of all waste gases from the copper smelter by purification systems.

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