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SELECTED TRACE ELEMENT CONCENTRATIONS IN AMBIENT AIR AND IN HORSE CHESTNUT LEAVES IN BELGRADE

Article Highlights

- Lead concentration showed a decreasing trend during the entire studied period
- The horse chestnut leaves showed good response to changes in trace element atmospheric concentrations
- Different sources of trace elements in ambient air and in horse chestnut leaves were observed
- During the studied period, traffic was one of the major sources of the measured elements in Belgrade
- The trace element concentrations in PM10 were below the limits of Serbian and EU legislation

Abstract

In this study, airborne particulates (PM10) and leaves of horse chestnut were collected at selected urban sites in Belgrade, Serbia, in years 2006, 2009 and 2012. All samples were analysed for trace element concentrations of As, Cd, Cr, Ni and Pb. It was found that, during the study period, the differences among PM10 mass trace element concentrations were not considerable, and that the measured mass and trace elements concentrations were below the Serbian and EU legislation limits. The highest values of trace element concentrations in leaves were observed in year 2012, with the exception of Pb. Lead concentration had a decreasing trend during the whole studied period, in both PM10 and tree leaves. Since leaded gasoline was banned in 2011, a possible reason could be an increasing number of vehicles using unleaded kind along the previous years. Although trace elements in horse chestnut leaves were accumulated only during the summer season, horse chestnut leaves showed good response to changes in trace element atmospheric concentrations. However, seasonal variability was evident in trace element source apportionment due to the lack of stationary heating system influence. The principal component analysis showed that during the studied period, one of the major sources of the measured elements was fossil fuel combustion.

Keywords: PM10, tree leaves, air pollution, PCA, WeBIOPATR.

Urban environment is heavily impacted by airborne particulates originated from fossil fuel combustion, traffic, industry and other anthropogenic activities. A number of epidemiological studies [1,2] have demonstrated that acute and chronic health effects are related to the inhalable PM10 exposure in the urban environment. PM10 is considered harmful for human health because it can cause respiratory and cardiovascular diseases, and no safe level has been identified. Even at concentrations below current air quality guidelines they pose a health risk [3]. Mortality associated with air pollution is about 15-20% higher in cities with high levels of pollution compared to relatively cleaner cities [4].

Trace elements, attached to PM10, can be toxic and may have dangerous impacts on human health. Lead exposures have developmental and neurobehavioural effects on fetuses, infants and children, and elevate blood pressure in adults. The main emis-

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sion source of lead in the atmosphere for many years has been the use of leaded gasoline in vehicles. Since the lead content in fuels has been regulated in most Europe countries since year 2000 [5], industrial sources and fuel burning activities have assumed a bigger importance in ambient lead production. However, leaded gasoline was still widely used in Serbia throughout the last decades, although with decreasing tendency during last years due to increasing number of the new types of vehicles using the unleaded kind. The consumption of unleaded gasoline was increasing from the beginning of 2000's, but the official ban of leaded gasoline was put into effect at the beginning of 2011. However, the average age of vehicles in Serbia during the studied period was still very high. Of the total vehicles, the number of those older than 10 years in year 2008 in Serbia was over 65%, while only 17% of vehicles was less than 5 years old [6]. Arsenic exposure is associated with increased risk of skin and lung cancer. Cadmium is associated with kidney and bone damage and has also been identified as a potential human carcinogen, causing lung cancer. Nickel is a known carcinogen and also has other non-cancerous effects, e.g. on the endocrine system [4]. The main arsenic-emitting anthropogenic sources are the stationary combustion of fossil fuels (especially coal-burning) and to a lesser extent the metallurgical industry. Cadmium and nickel compounds in particulate matter mainly originate from coal and fuel oil combustion processes, metallurgical industry, and road transport [4]. Although the atmospheric levels of trace elements are usually low, they contribute to the deposition and build-up in soils, sediments and organisms. On the other hand, the absorption rates of some elements, *i.e.*, lead and cadmium, by inhalation are significantly higher (up to 50–60%) than those by ingestion (between 3 and 10%) [7]. Air pollution is only one source of exposure to these metals but their persistence and potential for long-range atmospheric transport means that atmospheric emissions of heavy metals affect even the most remote regions [8]. The assessment of the trace element content in ambient air gives important information for development of risk assessment strategies.

In the past decades, biomonitoring of air quality using plants has been widely used to detect and monitor trace and other element atmospheric contamination [9,10]. Although mosses are usually used in biomonitoring studies, as they are recognized as the most appropriate biomonitors of atmospheric trace elements pollution, they are often absent in urban areas. In such situations, trees can act as biomonitors of air pollution and they have been studied in search of a suitable biomonitor. Tree leaves are very efficient at trapping atmospheric particles, and they have a role in reducing the level of fine atmospheric particulates [11]. Leaves of various tree species have been studied in biomonitoring surveys in urban areas in order to find sensitive species that gave a good response to changes in bulk atmospheric pollution. Previous studies in the Belgrade urban area have shown that horse chestnut leaves could be a valuable tool for monitoring of trace elements in the atmosphere [12-14].

The objective of the present work was: *i*) determination of temporal variability of PM10 mass concentrations; *ii*) preliminary assessment of selected trace element content in ambient air by horse chestnut tree leaf samples collected in years 2006, 2009 and 2012. Another objective was to assess the reliability of passive biomonitoring in the evaluation of natural and anthropogenic sources or airborne particles. The main cause of an element concentration increase is accumulation of atmospheric particles at the surface of leaves. The magnitude of the accumulation depends on the concentration of trace elements in particles and also on the biological ability of the tree leaves to bioaccumulate the element. Therefore, assessing the concentrations of trace elements in ambient air and in tree leaves, in parallel, could give an insight in biomonitoring as an additional method to classical monitoring techniques.

METHODOLOGY

Sampling and sample preparation

The site selection was based on our previous studies on biomonitoring of trace elements [13,15], as well as on available data concerning potential traffic "hotspots" in the Belgrade urban area (Figure 1). The emphasis was on the highly traffic-exposed locations, close to the big junctions or main routes for vehicles. All parks and monitoring sites are located in the centre of Belgrade with characteristically intense and slow moving traffic (>50,000 vehicles per day). Other possible influences include individual heating units, distributed in all parts of Belgrade, thermal power plant "TENT" in Obrenovac, located *cca.* 30 km southwest from Belgrade and the oil refinery in Pančevo, located *cca.* 20 km in a northeast direction. Geographical coordinates of the sampling sites are shown in Figure 1.

The sampling and selection of the elements were performed in accordance with the Serbian air quality legislative for air quality monitoring [16,17], and the two air quality directives in force in EU

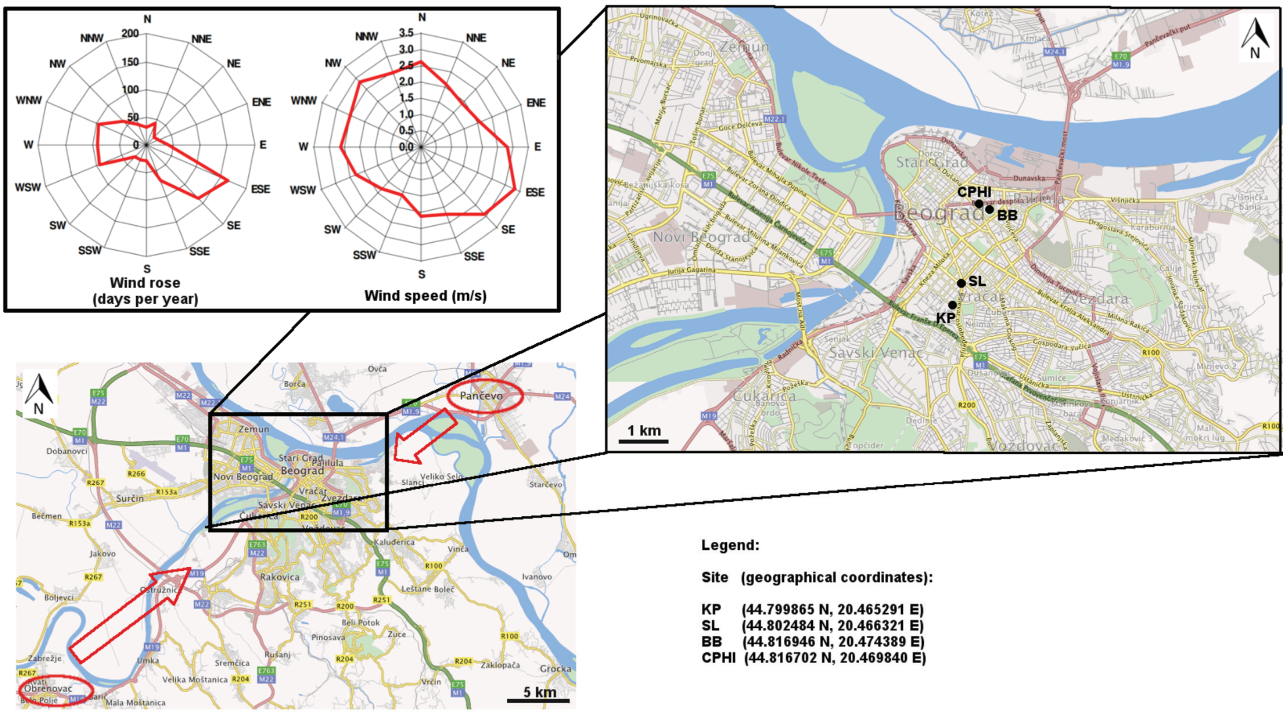

Figure 1. Map of the study area with geographical coordinates - PM10 sampling sites (CPHI - City Public Health Institute, SL - Slavija square), and horse chestnut tree leaves sampling sites (BB - Botanička bašta, KP - Karađorđev park).

[18,19]. The samples, PM10 and leaves, were collected from sites exposed to high traffic influence in the Belgrade city centre. The PM10 sampling was done using two automatic monitoring stations for continuous atmospheric particulate monitoring [20] close to the City Public Health Institute, and at square Slavija, the busiest roundabout in Belgrade. The sampling was done as regulated by the Serbian legislative directives [17], on a daily basis, continuously during the whole year, and the sampling time was 24 h. The trace element content in PM10 was assessed by ICP-MS, using EPA method O-3.5 [21] and analysed for concentrations of As, Cd, Cr, Ni and Pb.

The leaves of horse chestnut (*Aesculus hippocastanum* L.) were sampled from two representative parks (Karađorđev park and Botanička bašta) situated also in the Belgrade city center, and close to the previously mentioned automatic monitoring stations. Leaves were cut with stainless scissors at about 2 m height above ground and taken by hand, wearing polyethylene gloves. Five subsamples (10-15 fully developed leaves per tree) of approximately the same age were collected per species per site. Samples were taken from trees growing in different parts of the parks and leaves were collected from all sides of the crown without directional preference to give a good "average" composition at the site. The sampling was held during the multi-year period, in years 2006, 2009 and 2012. Leaves were collected once a year, at the end of vegetation cycle, in September. Leaf samples were briefly washed (for 3-5 s) with double-distilled water before further analysis, in order to assess only accumulated and strongly adhered particles on the leaf surface. Microwave digestion of approximately 0.4 g leaf sample (dry weight) was performed with 3 mL of 65% HNO_3 (Suprapur, Merck) and 2 ml of 30% H_2O_2 (Suprapur, Merck). The concentrations of As, Cd, Cr, Ni and Pb in leaves were measured from the digested samples by ICP-MS using an Agilent 7500ce mass spectrometer equipped with an Octopole Reaction System (ORS). Quality control was performed using the standard reference material lichen-336 (IAEA).

Data analysis

Principal component analysis (PCA) is a frequently used multivariate statistical method for the assessment of relationships among measured parameters and it was used in various studies on trace elements in PM10 and leaf samples and the identification of their pollution sources [22,23]. In this study, Varimax rotated PCA was used to assess the effects of sources to PM10 and leaves trace elements concentration. The analysis was performed using SPSS 12 software [24].

RESULTS AND DISCUSSION

PM10 concentration and trace element ambient air concentration

Temporal trend of the PM10 mass concentration in the Belgrade urban area, obtained in this study, is presented in Figure 2. The small decrease of PM10 mass concentration was observed during the first years of the study (2006 to 2009), and with another increase in 2012. However, the differences among the amount of particulates during the period of observation were not considerable and could be within the common environmental variation.

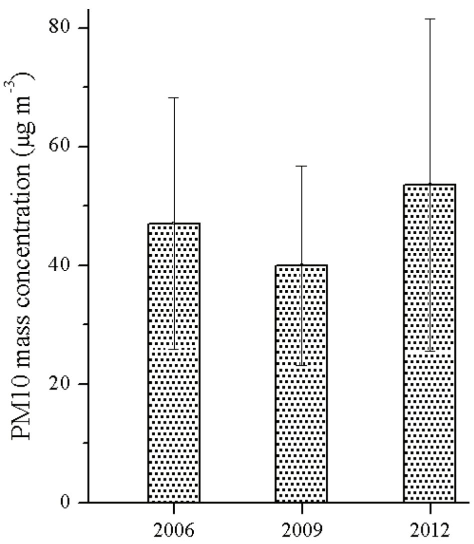

Figure 2. Temporal trend of mean PM10 mass concentration of the years 2006, 2009 and 2012.

The most important sources of PM emission in urban areas are considered to be the incomplete combustion of fossil fuels, friction of mechanical components such as brakes and tyre rubber, increasing proportion of diesel, high weight and four-wheel drive vehicles, traffic, together with industrial and domestic combustion processes [25]. However, another source that may exert a considerable influence on ambient level and composition of PM10 is mineral dust, which exhibits high geographical variations.

The mean concentration of PM10 in year 2006 was 47 μg m^{-3}, while in 2012 the concentration was 54 μg m^{-3}, which exceeded the European Union limit value of 50 μg m^{-3} (daily limit that should not to be exceeded more than 35 days in a year) [19]. The proposed limit value for the annual average concentration is 40 μg m^{-3} and it was exceeded in years 2006 and 2012, while in the year 2009 the annual average concentration was 40 μg m^{-3}. However, in the Serbian legislation the same values of daily and average annual limits are proposed as in the European Union, with the obligation to be met by January 2016 [17]. WHO Air Quality Guidelines for PM10 is 20 μg m^{-3} (annual average), which is more stringent than corresponding standards in the EU legislation; it refers to target values rather than limit values, since no threshold for PM below which any damage to health is observed, has been identified [3]. Therefore PM10 levels proposed by WHO Air Quality Guidelines were exceeded in all studied years.

During the last decades various measures and air quality standards have been implemented in Europe's law in order to reduce the concentration of atmospheric PM and its impact on human health and the environment. As a result, the emissions of PM and their precursors have continuously declined in EU [26], as well as ambient PM10 concentrations. Despite of these positive trends, the air quality standards for PM10 are still regularly exceeded in various European regions [27].

Although, as mentioned before, the differences in PM10 concentration during the observation period were not considerable and could be within the common environmental variation, trace element ambient concentration did not show this trend (Figure 3).

The concentration of Cr had the highest value in the year 2012. Compounds of Cr (VI) are well known to be associated with toxic and carcinogenic effects on the bronchial tree. Therefore, the tracking of chromium ambient concentrations is a matter of great importance, because of its connection with severe health impairments [7]. However, in this study, the obtained results represent total element content, as proposed by the Serbian legislative for air quality monitoring [16,17]. On the other hand, the lowest values of Cd and Pb concentrations were reported for 2012. The European Union has set annual limits for Pb (500 ng m^{-3}) [19], and target values for As (6 ng m^{-3}), Cd (5 ng m^{-3}) and Ni (20 ng m^{-3}), measured as contents in PM10 [18]. The values specified are maximum annual averages, which countries are obliged to meet by 2013, except for the limit value for lead which was to be met by 2005. As for the Serbian legislation of trace element content in PM10, annual limit of Pb is to be met by January 2016, while target values for As, Cd and Ni are assessed from the January 2012. That is, data from the year 2012 will be the first to be used for determining compliance with the target values in the 3- to 5-year periods, depending on the needs of the monitoring [17]. The annual limit for Pb was not exceeded during the studied period, with its concentration being lower than 150 ng m^{-3}. Generally, the

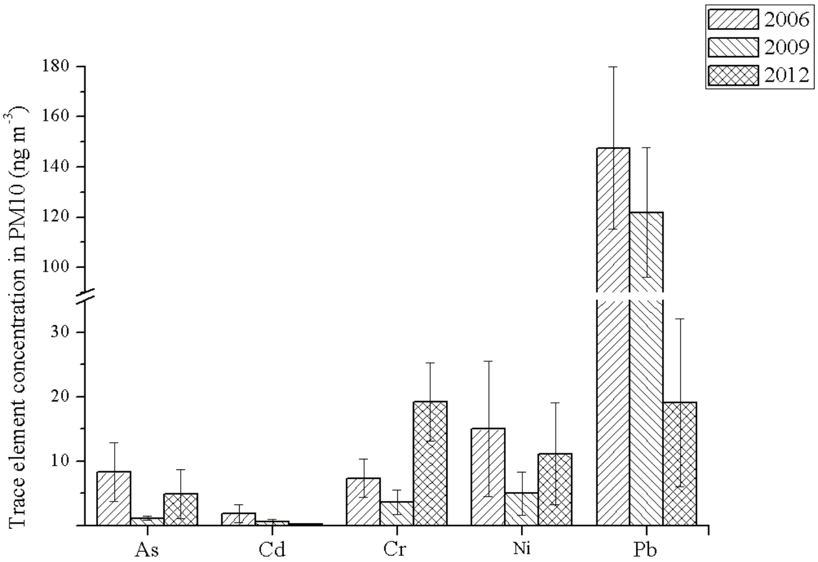

Figure 3. Annual mean trace element ambient concentrations of the years 2006, 2009 and 2012.

concentrations of measured trace elements were below the limits of EU legislation. The only exceedance of these values was the mean annual concentration of As in the year 2006 (8.3 ng m^{-3}). However, it should be noted that the determination of the exceedance of assessment thresholds requires 5-year monitoring periods. In case of As, the only exceedance was in the year 2006, therefore its average 5-year concentration was not above the target value. As from the observed results and with a similar future trend in trace element concentrations, it is not expected to encounter major problems in meeting air quality requirements concerning levels of trace elements in ambient air in the Belgrade urban area.

Comparison of the study results with other studies showed elevated concentration of Ni, but also lower concentration of As, Cd, and Cr [28-30]. Nickel mainly originates from combustion of fossil fuels or traffic vehicle exhaust as an additive in fuels, although its relatively high geological origin in Belgrade was confirmed [31]. Lead was the most abundant element in the samples, since it was still widely used as additive in gasoline, but with a strongly declining trend during the studied period as a result of increased use of unleaded gasoline. However, lead content in ambient air is still higher than it was reported in some studies, since in most of the EU countries leaded gasoline was put out from the use from January 2000, although many EU member states began even earlier [5]. Brake linings have been demonstrated to result in the release of Cd, Cr and Ni [32], while various vehicle electronic components and batteries of cars that contain Cd have been reported as a potential source of it [33].

PM10 total element content during the year 2012 is presented in two seasons, April-September and October-March (Table 1), since the period of particulate deposition on leaf surfaces lasts from April to September during the vegetation cycle of deciduous trees. This year is also interesting from the aspect of phasing out of leaded gasoline from the beginning of 2011. The high concentrations of Cr and Ni in 2012 were due to their high emission in the winter period (October-March, Table 1), probably due to the use of fossil fuels for heating.

Trace elements in leaf samples

Trace element concentrations in leaf samples presented the particle deposition and accumulation during the period of the vegetation cycle, from April to September (Figure 4). Following the decrease of trace elements concentrations in the year 2009, there was another increase for most of the elements in the year 2012, except in the case of lead, which had a continuous decline during the studied period. However, the concentration of As did not show a decrease in the year 2009. Also, the highest values of trace element concentrations were measured in leaves sampled in September 2012, with the exception of lead, which had a decreasing trend during the whole studied period. However, at the beginning of the studied period, the most abundant element in the leaves was Pb. As it was reported previously [34], even though the concentration of Pb is strongly declining in the past years, lead is still present in the environment.

Temporal decrease of Pb concentrations in leaves as a consequence of decreasing use of leaded

Table 1. Seasonal trace element concentrations in PM10 in 2012 (μg g^{-1}; RSD are presented in %)

Sample	Value	As	Cd	Cr	Ni	Pb
Annual	Mean	108	4.7	923	624	491
	SD	53	4.3	1557	1392	284
	RSD	49.07	91.49	168.69	223.08	57.84
Apr-Sept	Mean	75	4.3	544	209	430
	SD	30	3	82	58	133
	RSD	40	69.77	15.07	27.75	30.93
	Min	17	0.05	163	61	127
	Max	331	59	976	461	1732
Oct-Mar	Mean	141	5.2	1302	1038	551
	SD	52	3	2263	1962	398
	RSD	36.88	57.69	173.81	189.02	72.23
	Min	8.6	0.04	111	60	75
	Max	934	58	12822	14567	4357

gasoline was also reported for other plant species [35,36]. Notwithstanding, the accumulation of this element had higher values in all studied years than the "reference plant" value (1 μg g^{-1}, given by Markert) [37]. However, at the end of the studied period, in the year 2012, the Pb concentration had lower values than Cr and Ni. The main sources of Ni and Cr are the stationary sources of fossil fuel combustion, mostly active during heating season [38]. On the other hand, these elements also could originate from almost all traffic-related sources (brake linings, tire wear and tear, combustion of gasoline) [5]. However, elevated level of Ni concentration could indicate possible resuspension of soil. When compared with the "reference plant" values for other elements, concentration of As also showed higher values during the whole studied period (0.01 μg g^{-1}), while concentration of Cd never exceeded the values of the "reference plant" (0.05 μg g^{-1}).

A decreasing tendency of lead concentration in ambient air and in leaf samples was evident even before the official ban of leaded gasoline. However, the concentration of Pb after the 7-year period was about 7 times lower in ambient air and almost 3 times lower in the leaf samples then at the beginning of the study period, indicating that during the examined period, one of most significant sources of this element was traffic and also the presence of lead in the environment due to past emissions. Some authors suggested that, after the ban on the use of lead

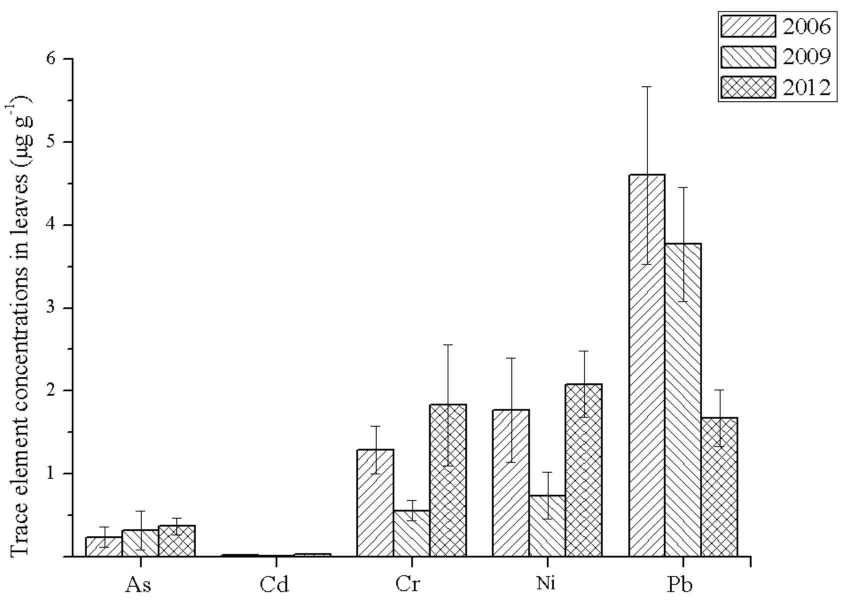

Figure 4. Temporal trend of trace element concentrations in horse chestnut leaves of the years 2006, 2009 and 2012.

additives in gasoline, the contribution of lead from traffic fuel combustion is low [39]; therefore, other sources of lead become more prominent [40].

Based on our previous studies and the current research, it can be concluded that plant tissue can give a better insight of results in terms of sensibility and reproducibility. The degree of trace elements content in plant tissue is proportional to urbanization, industrial activity and density of traffic [41]. The element content in tree leaves may originate from soil by uptake *via* roots or from atmospheric deposition on the leaf surfaces. However, the topsoil in the Belgrade urban area is slightly alkaline (pH 8) [15], which decreases bioavailability of trace metals and favours foliar uptake [9].

As it was suggested from the previous studies [12-15], horse chestnut showed a good response as bioindicator of trace element air pollution, as it proved to be good in reflecting changes in atmospheric lead pollution. Trees could be effective indicators of the impact of pollution sources, because of their ability to accumulate trace elements. Also, the effect observed is a time-averaged result, which will be more reliable than that obtained from direct determination of the pollutant concentrations in air for a short period. However, direct comparison of trace element concentrations in PM10 and in leaf samples could not be done since the processes of accumulation and deposition of particles are different in case of PM10 and leaves. In PM10 automatic sampling, the flow of air is passed through the filter while a natural deposition occurs on the leaf surfaces. The PM10 sampling was done continuously during the whole year, on a daily basis, while tree leaves were sampled in September, at the end of the vegetation cycle. Nevertheless, the trace element concentrations trend in 2012, from April to September was Cr>Pb>Ni>As>Cd (Table 1), while in leaf samples from the same year was Ni>Cr>Pb>>As>Cd. The similar trends were observed except the elevated level of Ni in leaves, which could indicate possible resuspension of soil, as its relatively high geological origin in Belgrade was confirmed [42].

Principal component analysis

The principal components analysis (PCA) model approach was performed to identify and quantify the trace element source components in air and in horse chestnut leaves. In case of both PM10 samples and horse chestnut leaves, 5 elements (As, Cd, Cr, Ni and Pb) measured during 2002 – 2006 were included in the PCA. Horse chestnut leaves were collected at Botanička bašta site, while PM10 samples were collected at the automatic station close to the City Public Health Institute, in the close proximity of Botanička bašta. The factor loading matrix after Varimax rotation and the contribution of each component are presented in Figures 5 and 6. The system extracted the principal component with eigenvalue greater than 1.

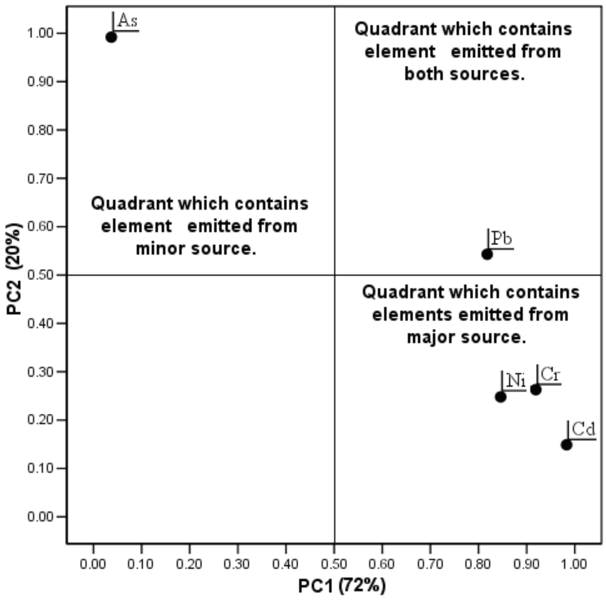

Figure 5. Principal component analysis for trace elements in ambient air from year 2002 to 2006.

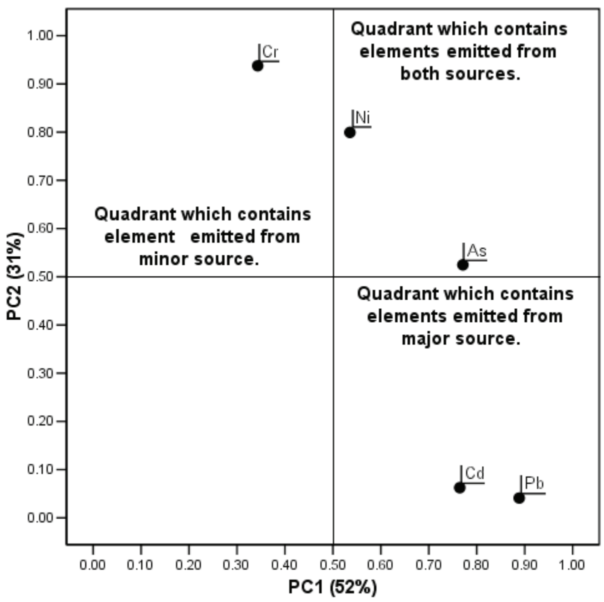

Figure 6. Principal component analysis for trace elements in horse chestnut leaves from Botanička bašta, from year 2002 to 2006.

PM10 samples

The PCA of trace elements in ambient air is presented in Figure 5. Two major components were identified and explained a sum of 92% of the overall variance in the data set in ambient air for period 2002–2006. The first component was associated with Cd, Ni, Pb, and Cr, and it explained 72% of the total variance. This component probably represented fossil fuel combustion, originating from stationary sources and traffic-related emission. Continuing use of leaded gasoline could be pointed out as a main source of Pb, although resuspension of previously contaminated soil and dust could not be neglected. It was pointed out recently [40], that combustion of the lubricant oil could act as a potential source of trace elements, in addition to fuel combustion. The second component had high loadings of As and moderate loadings of Pb, which could be associated with coal combustion. This component was accounting to the 20% of the total variance.

Horse chestnut leaf samples

The PCA of trace element concentrations in leaf samples of horse chestnut was presented in Figure 6. Two components were identified, explaining a sum of 83% of the total variances in leaf samples. Component 1 showed high loadings of Ni, Pb, As, and Cd, and the sum of the 52% of the total variance, could be attributed to vehicle exhaust and other fossil fuel combustion. On the other hand, component 2 was characterized with high loadings of Cr and Ni, and moderate loading of As, accounting for the 31% of the total variance. These elements could be associated with soil and dust resuspension. Cr and Ni have earlier been shown to derive from almost all traffic-related sources (brake linings, tire wear and tear, combustion of lubricant oil), they could be classified as non-source-specific traffic-related metals, mainly resulting from resuspension [5].

Tree leaves of deciduous plants, as horse chestnut, are able to accumulate trace elements during their vegetation period, from April until September. Therefore, the influence of stationary fossil fuel sources (*e.g.*, heating units), which are generally dominant during the winter season, is excluded. On the other hand, monitoring of PM10 is carried out during the whole year and here it is presented in average annual values. Even though the temporal trends of trace element concentrations in ambient air and in horse chestnut leaves had showed similarity, the identified major sources of trace elements were different, indicating possible seasonal variability in their concentrations. It appears that in large urban areas, like Belgrade, vehicle-related and fossil fuel emission sources assume greater importance than industrial sources for the determination of trace element concentration levels in atmospheric particles. Also, traffic as a source of pollution is present during the whole year, unlike stationary fossil fuel combustion, which gains importance mainly during the heating season. However, the source apportionment study can be more accurate with the incorporation of a larger number of trace elements, which could be an important step in the future for an integrated characterization of particulate matter pollution in the area.

CONCLUSIONS

During the study of PM10 mass concentrations and trace element ambient air concentrations, as well as trace element concentrations in tree leaves (*A. hippocastanum*) from the Belgrade city centre, it has been shown that the differences among mass and ambient air element concentrations during the study period were not considerable. The concentrations of measured trace elements in PM10 were below the limits of Serbian and EU legislation. The horse chestnut leaves showed good response to changes in atmospheric trace element pollution, with the similar trend in trace element concentrations as in the ambient air. The highest values of trace element concentrations in horse chestnut leaves were in the year 2012, with the exception of Pb concentration. Lead concentration showed a decreasing trend during the whole studied period, as the result of reduced use of leaded gasoline during the studied period. However, the assessment of source apportionment indicated different sources of trace elements in ambient air and in horse chestnut leaves as a result of different accumulating period in two types of samples. It seems that, during the studied period, fossil fuel combustion was one of the major sources of the measured elements in Belgrade urban area.

Acknowledgments

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KONCENTRACIJE ODABRANIH ELEMENATA U TRAGOVIMA U VAZDUHU I U LISTOVIMA DIVLJEG KESTENA U BEOGRADU

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NAUČNI RAD

U ovom radu određivane su koncentracije elemenata u tragovima, As, Cd, Cr, Ni i Pb u česticama iz vazduha (PM10) i listovima divljeg kestena sakupljenim na odabranim lokacijama u centru Beograda, tokom 2006., 2009., i 2012. godine. Utvrđeno je da razlike u koncentracijama elemenata u tragovima u uzorcima PM10 nisu bile značajne, i da su, tokom posmatranog perioda, bile ispod dozvoljenih granica propisanih od strane Republike Srbije i Evropske Unije. Najviše koncentracije ispitivanih elemenata u listovima su zabeležene u 2012. godini, osim u slučaju olova. Koncentracija olova je imala opadajući trend, i u česticama i u listovima, tokom celog ispitivanog perioda, verovatno zbog povećanja broja vozila koja koriste bezolovni benzin, s obzirom na to da je upotreba olovnog benzina zabranjena početkom 2011. godine. Iako se elementi u tragovima u listovima divljeg kestena akumuliraju samo tokom letnjeg perioda, koncentracije ispitivanih elemenata u listovima su bile u korelaciji sa promenama koncentracija elemenata u vazduhu. Ipak, uočljiva je sezonska varijacija koncentracija elemenata usled nedostatka uticaja stacionarnog grejanja tokom leta. Analiza glavnih komponenti je pokazala da je, tokom posmatranog perioda, jedan od glavnih izvora ispitivanih elemenata bilo sagorevanje fosilnih goriva.

Ključne reči: PM10, lišće drveća, zagađenje vazduha, PCA, WeBIOPATR.