

Levels of PAHs in soil samples from the vicinity of oil refinery Novi Sad-Serbia

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ABSTRACT

The levels of 16 polycyclic aromatic hydrocarbons (PAHs) from the priority list of the U.S. Environmental Protection Agency were determined in soil samples collected in Novi Sad (the capital of Vojvodina Province, Serbia), a region with chemical industry and oil refinery. Samples were also collected in an urban/residential zone and in a presumably unpolluted site (control sample). The sampling sites were chosen in order to study the influence of the oil refinery on PAH levels. The contents of PAHs were determined by GC/FID. Benzo(a) pyrene known to be carcinogenic formed 2-13% of the sum concentration of the identified PAHs for all investigated samples. Basic physical-chemical properties like organic matter content, pH and water content were determined for each composite soil sample. No significant correlation was observed between these variables and the PAH-load of soils. Data obtained were compared with ones previously found for soils in Novi Sad, as well as with soils collected throughout the world and compared to target values set by the Dutch authorities for unpolluted soil. The total carcinogenic potency for each sampling site was calculated and compared with the reference value obtained on the base of the target concentrations.

Keywords: carcinogenic potency; oil refinery; polycyclic aromatic hydrocarbons; soil; urban zone.

INTRODUCTION

Polycyclic aromatic hydrocarbons (PAHs) are a class of persistent organic pollutants (POPs) produced by the incomplete combustion of organic material, either natural, as with forest fires, or anthropogenic, such as by industrial activities, heating from fossil fuels or in motor vehicle. PAHs have been detected around the world even at sites which are located far from industrial activity such as in the polar regions (Wania & Mackay 1996). As the Arctic is far from all industrial sources, anthropogenic PAHs could only reach it by long-range atmospheric transport (Wilcke 2007).

Atmospheric deposition is the most common source of pollution in soil (Li *et al.* 2001). Previous studies implied that POPs measured in soils correlate with those in the atmosphere, and therefore, soil PAH concentrations are usually considered good indicators of the surrounding pollution and environmental risks (Wild & Jones 1995).

Because of their persistence, low vapor pressures and high octanol/air partition coefficients, PAHs can strongly adsorb to soil organic matter and are likely to be retained for a long time (Wilcke 2007). Consequently, soil is one of the main reservoirs for PAHs in the environment. A number of law-abiding guidelines on soil quality are set to protect human and ecological health in developed countries, but the significance of soil protection is often neglected in developing countries and ones in transition, like Serbia.

To date, there is only limited data on PAH concentrations in the Serbian urban soil (Škrbić *et al.* 2005, Crnković *et al.* 2007). The extent of PAH-burden in Novi Sad soil was previously studied in 2001, covering the whole city area (Škrbić *et al.* 2005). However, there is no systematic long-term study on this subject and, consequently, there is no information available about the extent of soil pollution in the city over the years.

The major objective of this paper is to characterize the spatial distribution of soil PAHs and to compare these data with the previous findings, as well as to study the influence of oil refinery on their levels. The data are compared with those available in the literature for other urban areas worldwide. In addition, the total carcinogenic potency of soil samples was calculated in order to estimate the environmental and health risks.

MATERIALS AND METHODS

Soil collection

Novi Sad is the capital of the northern Serbian province of Vojvodina. It is located in the southern part of Europe, between the 19th and 20th degree of the east longitude and 45th and 46th degree of the north latitude, at the altitude of 72 to 80 m. Novi Sad lies on the left bank of the river Danube, namely on the 1255th km of its flow and on the mouth of one of the magisterial Danube-Tisa-Danube canals into the river Danube. With 15 suburban settlements, the territory of the City of Novi Sad encompasses a surface area of 702,7 km². It is the second largest city in Serbia, after Belgrade, and its large industrial and financial centre represents the most fertile agricultural and economical region in Serbia. One of the biggest

industrial combines in the city is the oil refinery, located 3 km to the north of the city centre along with the thermal power plant, near the suburban settlement of Šangaj. The refinery complex consists of production facilities and storage tanks for crude oil and oil products (mainly gasoline and diesel fuel). During the 1999 NATO bombing of Yugoslavia, the Novi Sad oil refinery was bombarded daily and was severely damaged. Due to the uncontrolled combustion during refinery fires following the bombardments, many highly toxic substances were released, such as some PAHs, which are found to be highly carcinogenic (Škrbić 2008).

The samples of surface soils (0-5 cm) from representative areas were collected during one day with stable weather conditions in June 2007 by using a stainless steel corer. Five sampling sites were chosen in Šangaj near the Oil Refinery Novi Sad. Samples were collected in the local school backyard (three composite samples, nos. 1-3) and along the local road next to the refinery (two composite samples, nos. 4 and 5). In addition, samples were taken in an urban/residential zone (park-school backyard in the downtown of the city of Novi Sad, sample no. 7) and in a presumably unpolluted site, an arable zone far from direct anthropogenic PAH sources (control sample no. 6). Each sampling point was represented by a rectangular area of 20×50 m with four individual samples (about 250 g).

The collected samples were stored in prewashed glass bottles and immediately transported to the laboratory. The individual samples from each sampling site were mixed together and sieved through a steel mesh (~2 mm) to remove large particles and organic debris, creating seven composite samples. The obtained composite samples were stored frozen until analysis and then dried in a desiccator for 24 hours prior to analysis.

Soil characterization

Basic physical-chemical properties were determined for each composite soil sample. pH measurements were carried out in deionized water (50 ml), after stirring the dried sample portion of 20 g for an hour. The organic matter content was determined as a weight loss by weighing the sample before and after heating at 500°C for 2 hours (Škrbić *et al.* 2005). Water content in the homogenized soil sample was obtained by oven drying at 105°C to the constant mass.

Chemicals

Organic solvents (for residue analysis), anhydrous sodium sulphate and silica gel 60 (70-230 mesh) were purchased from Promochem (Wesel, Germany).

Anhydrous sodium sulphate and silica gel were used after heating at 130°C for 4 h. Distilled water used in the extraction procedure was extracted with cyclohexane. Whatman No. 40 filter paper of 110 mm in diameter from Whatman (Springfield Mill, Maidstone, Kent, UK) was cyclohexane washed prior to use. Sixteen PAHs standard mixture (EPA Method 610) was purchased from Supelco (Bellefonte, PA, USA).

Analytical procedure

Laboratory glassware used in the procedure was washed with detergent, rinsed properly with distilled water and acetone and heated at 110°C for 24 hours. During the procedure, the glassware was wrapped with aluminium foil to prevent PAHs degradation by light.

PAHs determination from soil samples was based on US EPA Standard Methods 3550B, 3630C and 8100 (EPA, 1986). The samples (10 g) were mixed with anhydrous sodium sulphate (30 g), extracted twice with 50 ml dichloromethane (DCM) (each extraction step lasted for 20 minutes) in the ultrasonic apparatus and then filtered throughout filter paper. The obtained extract was concentrated to 1 ml by a rotary evaporator (Laborota 4000, Heidolph Instruments, Germany) and transferred to a column packed with 8 g activated silica gel pre-soaked in cyclohexane and pre-eluted with cyclohexane. The complete transfer of the extract was accomplished by washing the vessel with 2 ml cyclohexane. The first fraction eluted with 15 ml cyclohexane was discarded, whereas the second fraction containing PAHs was eluted with 55 ml of cyclohexane/DCM mixture (7:3, v/v). The eluate was concentrated to about 1 ml in a rotary evaporator, then evaporated in the gentle stream of nitrogen to near dryness. Prior to analysis the internal standard solution of 1,3,5-triphenylbenzene was added and the extract was redissolved in a mixture of DCM and methanol (1:1, v/v). The final volume of the extract was set to be 0.1 ml.

Analysis of the eluted fraction was carried out using DANI 1000 gas chromatograph (GC) equipped with a flame ionization detector (FID). GC/FID analysis was performed on RTX 5 capillary column coated with 5% diphenyl - 95% dimethylpolysiloxane (30 m x 0.25 mm I. D. x 0.25 μ m film thickness). The temperature programme was as follows: initial temperature 50°C held for 4 minutes, increased at a rate of 25°C/min to 200°C, then increased at a rate of 4°C/min to 310°C and held for 4 minutes. The injection port and FID temperatures were kept at 280°C and 300°C, respectively. Helium was used as the carrier gas at a flow of 2 ml/min. Aliquot of 1 μ l of the prepared sample was

injected into the GC system in a splitless mode with a purge time of 4 minutes.

The PAHs identification was performed by a comparison of their retention time with those of standard mixture. The quantitative analysis was done by the internal standard method. Five calibration standards with concentration evenly distributed through the working range were prepared.

The detection limits of the applied method for individual PAH compounds were about 0.01 $\mu\text{g/g}$. Quality control consisted of an analysis of blank, spiked and duplicate samples. Blank samples were treated and analyzed with the same method as the actual samples. Blank analysis showed no interference peaks with the individual PAH compounds analysis.

The samples fortified at two levels (1 $\mu\text{g/g}$ and 20 $\mu\text{g/g}$) were prepared by adding a known volume of 16 EPA PAH standard solution and then analysed as previously described. The recoveries for PAHs were in the range of 69-90% (relative standard deviation (RSD) less than 12%). The obtained data were not adjusted on the basis of these recoveries.

RESULTS AND DISCUSSION

The basic physical and chemical properties of the composite soil samples through the investigated sites are listed in Table 1. The concentrations of PAHs in soil samples are presented in Table 2. Results are given as a mean value of duplicate analysis for each composite sample and each sampling site.

Table 1. Analysis of organic matter, water content and pH of investigated soil samples from Novi Sad (June 2007)

Soil characteristic	Sample no.						
	1	2	3	4	5	6	7
Loss of ignition, %	12.3	24.7	25.5	21.0	6.30	16.2	9.87
Water content, %	14.1	20.6	13.6	6.65	5.72	13.1	10.5
pH	6.6	6.7	6.7	7.2	7.2	7.8	7.7

Table 2. Content of polycyclic aromatic hydrocarbons ($\mu\text{g/g dw}$) in soil samples from Novi Sad (June 2007)

Dutch target values ($\mu\text{g/g}$) for unpolluted soil (VROM, 1994), benzo(a)pyrene equivalent factors (BPEF) and total carcinogenic potency (TCP) of each analyzed sample ($\mu\text{g BaPeq/g}$)

Compound	Sample no.							BPEF		
	1	2	3	4	5	6	7	Dutch target value	*	**
Naphthalene	1.13	0.97	12.5	1.26	1.36	nd	nd			
Acenaphthylene	2.18	3.01	8.83	2.13	1.11	nd	nd			
Acenaphthene	0.54	1.20	2.36	1.09	0.42	nd	nd			
Phenanthrene	0.85	1.20	3.62	1.26	0.78	0.05	0.06	0.045	0.00064	
Anthracene	0.30	0.29	1.54	0.44	0.08	0.10	0.07	0.050		
Fluorene	0.07	nd	0.57	0.18	0.57	nd	nd			
Fluoranthene	0.12	0.21	4.03	0.85	0.10	nd	0.10	0.020		
Pyrene	0.05	0.07	0.31	0.12	0.10	nd	0.11			
Chryzene	0.09	0.13	0.58	0.10	0.08	nd	0.12	0.020	0.026	
Benz(a)anthracene	0.05	0.06	0.37	0.10	0.03	0.05	0.11	0.020	0.014	0.006
Benzo(b)fluoranthene	0.70	0.69	1.72	0.51	0.27	0.06	0.08	0.11	0.076	
Benzo(k)fluoranthene	0.85	0.22	0.99	0.19	0.52	0.08	0.07	0.025	0.037	0.076
Benzo(a)pyrene	0.63	0.41	0.81	0.24	0.39	0.09	0.09	0.025	1	1

Compound	Sample no.							BPEF		
	1	2	3	4	5	6	7	Dutch target value	*	**
Dibenzo(ah)anthracene	1.34	1.93	5.27	1.48	0.33	0.14	0.06		0.89	
Benzo(ghi)perylene	0.13	0.06	2.47	0.24	0.05	nd	0.05	0.020	0.012	
Indeno(1,2,3-cd)pyrene	0.14	0.21	1.00	0.35	0.05	0.10	0.06	0.025	0.067	0.080
∑PAHs	9.17	10.7	46.9	10.5	6.25	0.67	0.98			
TCP1***	1.94	2.24	5.78	1.63	0.73	0.22	0.16	0.029****		
TCP2***	0.76	0.50	1.10	0.32	0.46	0.11	0.11	0.029****		

nd-not detected;

* according MOE 1997;

** according Zaponni et al. 1993;

*** TCP1 and TCP2 are calculated by applying BPEFs reported by MOE (1997) and Zaponni et al. (1993), respectively;

**** reference values of TCP calculated according the Dutch target values and BPEFs reported by MOE (1997) and Zaponni et al. (1993), respectively

The \sum PAHs value refers to the sum of identified PAH components in the investigated samples, which ranged from 0.67 $\mu\text{g/g}$ in the control sample to 46.93 $\mu\text{g/g}$ in the schoolyard near the oil refinery. Mean \sum PAH value and standard deviation for all samples were 12.17 $\mu\text{g/g}$ and 15.89 $\mu\text{g/g}$, respectively, with a median value of 9.17 $\mu\text{g/g}$. PAH profiles of analyzed samples are shown in Fig. 1. Of the PAHs detected, low-molecular weight PAHs, including 2- and 3-ring PAHs, contributed significantly to the \sum PAH concentration in samples near the oil refinery (nos. 1 - 5), while their contributions were much less in the control sample (no. 6) and in the one from the urban zone (no. 7), in which higher-molecular weight PAHs (from 4- to 6-ring compounds) dominated. Wilcke (2007) hypothesized that there are two distinct PAH soil patterns: a background and a human-made one. A large part of the composition of PAH mixtures in soils can be explained as simple mixing of these two major source groups. The background signal is dominated by natural PAHs produced biologically or released by vegetation fires or volcanic exhalations, including also ubiquitous atmospherically distributed anthropogenic emissions. The other source pattern originates from the combustion of fossil fuels dominated by the presence of a number of high-molecular weight PAHs (Wilcke 2007) irrespective of the different fuels and combustion conditions. Furthermore, soils which are contaminated by spills of unburnt petroleum might also be characterized by high concentrations of low-molecular weight PAHs, particularly naphthalene and phenanthrene (Aislabie *et al.* 1999). Thus, it could be concluded that the main source of PAHs detected in soils 6 and 7 were fossil fuel combustion processes. As the lower molecular weight PAHs are more volatile than the higher molecular weight PAHs, higher loadings of low-molecular PAHs in the vicinity of the oil refinery could be explained by the volatilization from the accidental spillage or leaks of oil from storage tanks and transportation lines, which contributed to the localized soil loadings of PAHs.

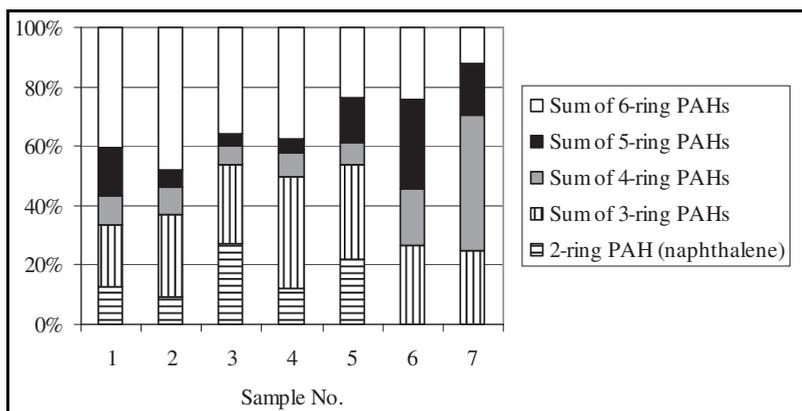


Fig. 1 PAHs profile of analyzed soil samples

In only one sample in the vicinity of the refinery, the determined concentrations was higher than the intervention value for soil sanitation set by the Dutch government of 40 μg PAH/g (VROM 1994, van Brummelen *et al.* 1996). Nevertheless, all identified compounds were detected in the quantities higher than the target values (0.020-0.050 $\mu\text{g/g}$) regulated by the Dutch government for unpolluted soil (see Table 2).

The total carcinogenic potency (TCP) of each sampling site and the reference one obtained using the Dutch target values for unpolluted soil are also presented in Table 2. Comparison of the estimated values revealed that all sites had the carcinogenic potency above the reference one. Benzo(a)pyrene, known to be carcinogenic, was identified in all samples and was above the Dutch target level of 0.025 $\mu\text{g/g}$. It formed 2-13% of $\sum\text{PAH}$ in investigated samples.

Previously determined PAH contents in surface soils of Novi Sad (Škrbić *et al.* 2005, see Table 3) coincided with the levels found in the urban zone and the control sample. The loads in the samples near the refinery were much higher. Considering the reported background PAH concentration in Bulgaria of 2-22 $\mu\text{g/kg}$ (Atanassov 2001) and the suggested level of endogenous total PAHs concentrations in soils of 1-10 $\mu\text{g/kg}$ (Edwards 1983), it could be concluded that the soils of Novi Sad were contaminated above the natural and background level. On the other hand, a comparison of the present data with those reported in other parts of the world (Table 3) revealed that our values were within the range of the published ones.

Table 3. PAHs concentrations in soil throughout the world

Location, soil origin	Number of PAHs included in the total content	Soil depth, cm	Range of $\sum\text{PAHs}$ or mean value*, $\mu\text{g/g}$	Reference
Welsh, UK, urban soil	14	0-5	0.119-54.50	Jones <i>et al.</i> , 1989
Brno, Czech Republic, urban soil	16		0.175-45.75	Strnad <i>et al.</i> , 1994
Basel, Switzerland, urban soil	16	0-20	11.10-11.90	Niederer, 1995
Estonia, urban soil	12	0-10	0.011-153.00	Trapido <i>et al.</i> , 1999
Tokushima, Japan, urban soil	13	0-5	0.08-1.64	Yang, 2002

Location, soil origin	Number of PAHs included in the total content	Soil depth, cm	Range of \sum PAHs or mean value*, $\mu\text{g/g}$	Reference
New Orleans, USA, urban area suburban	16	0-2.5	0.906-7.285 0.527-3.753	Mielke <i>et al.</i> , 2004
Tarragona County, Spain industrial area residential area	16	0-3	0.166; 1.002 0.736	Nadal <i>et al.</i> , 2004
Novi Sad, Serbia, urban soil	16	0-5	0.307-1.452	Škrbić <i>et al.</i> , 2005
Hong Kong, urban soil	16	0-10	0.169	Zhang <i>et al.</i> , 2006
Nepal, urban soil	16	0-5	0.136-6.273	Aichner <i>et al.</i> , 2007

*Values are given as in the original paper

In order to get a rough estimate of PAH-soil characteristic correlation in the Novi Sad region, Pearson's correlation between analyzed geochemical factors and soil \sum PAH concentrations was applied. No statistically significant correlation was observed between the used variables applying the criteria value of probability $p < 0.05$. However, the correlation coefficient between \sum PAH contents and LOI values (0.624), as well as between \sum PAH contents and pH (-0.586), could be considered as negligible. This is in accordance with the well known fact that organic matter of soil is responsible for the binding of PAHs, while lower values of pH favours the accumulation of semi-volatile organic pollutants (Carter and Suffet 1982).

CONCLUSION

Sixteen priority EPA polycyclic aromatic hydrocarbons (PAHs) were determined in composite soil samples from seven sites throughout the area of Novi Sad (Vojvodina Province, Serbia). The highest total contents of PAHs were determined for soil samples in the vicinity of the oil refinery, in which the PAH-loads were up to 47 times higher than in the sample from the urban zone; the maximum PAHs concentration found was higher than the intervention value for soil sanitation set by the Dutch authorities.

Low-molecular weight PAHs were predominant at the sites close to the

refinery, while high-molecular weight compounds contributed mostly to the total PAH content in the urban soil and the control sample.

Carcinogenic potency of PAH mixtures found in the investigated soils were above the reference one estimated on the base of the Dutch target values for unpolluted soil.

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مستويات الهيدروكربونات العطرية المتعددة الحلقات في عينات التربة من مناطق قريبة من مصفاة للنفط في نوفي ساد - صربيا

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خلاصة

لقد تم تعيين عدد 16 مستويات من الهيدروكربونات العطرية المتعددة الحلقات من قائمة الأولويات من وكالة حماية البيئة الأمريكية التي تم جمعها في نوفي ساد (مقاطعة فويفودينا - صربيا)، وهي منطقة للصناعات الكيماوية ومعامل تكرير النفط.

كما تم جمع عينات من المناطق الحضرية السكنية ومن موقع يفترض أنه غير ملوثاً (عينة مرجعية). وقد تم اختيار مواقع أخذ العينات من أجل دراسة تأثير مصفاة تكرير النفط على مستويات (PAHs) والتي تم تعيينها بواسطة GC/FID ووجد أن بنزو بيرين التي تعتبر مادة مسرطنة قد شكلت 2-13٪ من مجموع التراكيز المحددة التي تم تعريفها لجميع العينات التي تم فحصها. كما تم تعيين الخصائص الطبيعية - الكيماوية الأساسية من مثل محتوى المادة العضوية، الأس الهيدروجيني والمحتوى المائي لكل من عينات التربة المركبة. ولم يلاحظ أي ارتباط ذو أهمية بين هذه المتغيرات وكمية (PAHs) في التربة. لقد تمت مقارنة البيانات التي تم الحصول عليها مع عينات التربة التي سبق أن وجدت في التربة في نوفي ساد، والعينات التي تم جمعها من جميع أنحاء العالم، مع القيم المستهدفة والتي تم وضعها من قبل السلطات الهولندية للتربة الغير ملوثة. وتم حساب ومقارنة مجموع إمكانية المادة المسرطنة لكل مواقع العينات مع القيمة المرجعية التي حصل عليها على أساس التراكيز المستهدفة.

