



Seasonal variation and health risk assessment of organochlorine compounds in urban soils of Novi Sad, Serbia



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HIGHLIGHTS

- Novel method of pressurized liquid extraction for PCBs and OCPs in soil was developed.
- The results revealed that the seasonal variation of PCBs and DDTs was rather stable.
- Health risk assessment suggested that Novi Sad is suitable as residential.
- Profiles of DDTs indicated aged residues.
- OCPs residues and PCBs in soil are not a serious public health threat in Novi Sad.

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ABSTRACT

The aim of this paper was to determine spatial and seasonal variation of 6 indicator polychlorinated biphenyls (PCBs) and 18 organochlorine pesticides (OCPs) in urban soils of Novi Sad, Serbia. Sixty surface soil samples were collected during both winter and summer season, from different types of locations: schools, recreational areas, residential and industrial zones. Samples were treated using the method of accelerated solvent extraction, which allows simultaneous extraction and clean up of PCBs and OCPs. Targeted compounds were analyzed by GC- μ ECD and confirmed by GC-MS. The total concentrations of studied PCBs varied from <LOD to 18.9 ng g⁻¹ (mean 2.84 ng g⁻¹ and median value of 1.21 ng g⁻¹) in winter and from <LOD to 24.6 ng g⁻¹ (mean 3.27 ng g⁻¹ and median value of 1.60 ng g⁻¹) in summer. Regarding the OCPs analyzed, dichlorodiphenyltrichloroethanes (DDTs) were only detected in both seasons. The total concentration of DDTs ranging from <LOD to 86.3 ng g⁻¹ (mean 7.57 ng g⁻¹ and median value of 1.87 ng g⁻¹) in summer and from <LOD to 182 ng g⁻¹ (mean 15.6 ng g⁻¹ and median value of 3.15 ng g⁻¹) in winter. There were no significant difference between seasons for studied PCBs and DDTs. Spatial distribution of PCBs and DDTs were also evaluated using Arc GIS. Additional, principle component analysis was performed to evaluate relationships between PCBs, DDTs and soil characteristics. Positive correlation was observed among p,p'-DDE, p,p'-DDD and organic matter. The total lifetime carcinogenic risk indicated that the lifetime cancer risk was acceptable.

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1. Introduction

Organo-chlorinated compounds (OCCs) such as polychlorinated biphenyls (PCBs) and organochlorine pesticides (OCPs) are semi-volatile, relatively lipophilic and environmentally stable compounds that have been identified in diverse environmental matrices worldwide (Škrbić and Đurisić-Mladenović, 2007; Zhang

et al., 2014). Polychlorinated biphenyls have been extensively used as dielectrics in capacitors and transformers, as plasticizers in paints and joint sealants, and in many further applications because of their extraordinary chemical stability and heat resistance (Glüge et al., 2016). However, these chemical properties also contribute to the persistence of PCBs after their released into the environment (Wu et al., 2011). The manufacture, use, and importation of PCBs have been phased out since 1970s because of their teratogenic, immunogenic, or carcinogenic health effects and persistent nature (Salihoglu et al., 2011). Although concentrated near emission sources, PCBs have become dispersed in the

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environment through long-range atmospheric transportation (Yi-Fan Li et al., 2010). Organochlorinated pesticides were demonstrated as endocrine disruptors and carcinogens (Zhao et al., 2013). OCPs such as dichlorodiphenyltrichloroethanes (DDTs) and hexachlorocyclohexane (HCH) were used in agriculture for increasing food production, in household, gardens, public places and institution due to their efficiency in controlling pests and diseases (Kabir et al., 2015; Zhao et al., 2013). OCPs residues and PCBs have been reported in water, air and soils around the world (Škrbić et al., 2007; Wang et al., 2016; Yu et al., 2014; Zehra et al., 2015).

Soil plays an important role in the global fate and distribution of PCBs and OCPs (Holoubek et al., 2009). Persistent organic compounds (such as PCBs and OCPs) can contaminate surface soil via gaseous exchange mechanisms from the atmosphere, through wet and dry deposition processes and from direct point source inputs (Zhang et al., 2014). Due to their similar physicochemical properties, the fate of PCBs and OCPs is expected to be largely similar in the sediment and soil, but the characteristics of the medium has to be considered (Škrbić et al., 2007). Soil can also act as source of contamination, and therefore it may have deleterious effects on ecosystems and human health (Cachada et al., 2012). Soil is a sink for highly chlorinated PCBs, dichlorodiphenyltrichloroethane (DDT) and for dichlorodiphenyl-dichloroethylene (DDE), with a tendency for deposition during winter and volatilization during summer (Ružičková et al., 2008). Data on the distribution of organic pollutants in soil can help to assess levels of pollution, identify sources of emission and evaluate the environmental health risk (Li et al., 2015). In Serbia, PCBs were mainly used as dielectric fluids in transformers and condensers, which had a PCB composition with penta to hepta-chlorinated congeners (Škrbić and Đurišić-Mladenović, 2007). During the Balkan conflicts of the 1990s, burned or damaged industrial and military targets released large amounts of PCBs into the environment: more than 1000 electric transformer stations that contained PCB oil were damaged (IARC, 2015). Since many of damaged transformers and capacitors were originally filled with PCB mixtures, their leakage into the soils and industrial grounds became the major source of PCB contamination in the region (Klánová et al., 2007; Škrbić and Đurišić-Mladenović, 2007). Despite worldwide restriction by the Stockholm convention (2001) for the usage and production of PCBs and OCPs, these chemicals are still found in various environmental compartments from different parts of the world (Zehra et al., 2015).

To our best knowledge only one study has been carried out in Serbia, Novi Sad in order to determine concentration of PCBs and OCPs in soils for different land use (Škrbić et al., 2007). The main objectives of this paper were to determine the seasonal and spatial variations of PCBs and OCPs in Novi Sad urban soils and to estimate health risks, especially to children, via ingestion, inhalation and skin contact. Results presented in this paper provide the first reliable information for the city of Novi Sad, contributing to awareness on soil contamination. The results are compared with available data all around the world.

2. Materials and methods

2.1. Soil sampling

Sixty composite surface soil samples (30 samples each season) from the same locations were collected during stable weather conditions from 15 different locations in Novi Sad, Serbia, in July 2014 and March 2015, to represent the summer and winter seasons, respectively. At each location, soil samples were taken from two sites, one along the nearest road and the other away from the road (e.g. in the schoolyard) in order to investigate spatial, seasonal and short-range variability of the OCCs occurrence at the chosen

locations. The distribution of sampling locations is shown in Fig. 1. The soil samples were coded to reflect the number of the locations (from 1 to 15), whether they were collected in the vicinity of the road (coded "a") or away from it (coded "b") and also to reflect when they were collected during winter ("w") or summer season ("s").

The sampling locations have been divided according to the different functional area into four groups: school yards (1, 9, 13, 14), recreational areas (2, 3, 4, 5, 8, 10) (e.g. park, river beach), residential areas (6, 7, 12) and industrial zones (11, 15). Detailed description of the sampling locations and sampling methodology is given at Škrbić et al. (2016). Organic matter (OM) was determined for each soil sample measuring sample loss after 2 h at 550 °C as described in Škrbić and Čupić (2004). pH measurements were carried out in deionized water at a soil/water ratio of 1/2.5 with potentiometric glass electrode. Particle size distribution (content of clay, silt and sand) was determined by the sedimentation method (Škrbić and Čupić, 2004).

2.2. Chemicals and materials

The standard solution mixture of 6 indicator PCBs congeners PCB 28, PCB 52, PCB 101, PCB 138, PCB 153 and PCB 180 was purchased from AccuStandard (New Haven, CT, USA). A mixed standard solution of 18 OCPs containing (α -, β -, γ -, δ -HCH, p,p'-DDT, p,p'-DDE, p,p'-DDD, aldrin, dieldrin, endrin, endrin-aldehyde, endrin-keeton, endosulfan I and II, endosulfan-sulfate, heptachlor, heptachlor-epoxide and methoxychlor) in hexane/toluene mixture was purchased from Dr. Ehrenstorfer, Germany. PCB 209 (2,20,3,30,4,40,5,50,6,60-decachlorobiphenyl) and 2,4,5,6-Tetrachloro-*m*-xylene (THX) were obtained from Sigma-Aldrich and used as surrogate standards for PCBs and OCPs determination. *n*-Hexane (Fisher Chemical, UK) and methylene chloride (Fluka, Sigma-Aldrich) were of pesticide residue analysis grade. Silica gel (CarloErba), alumina oxide (Sigma-Aldrich) and diatomaceous earth (Thermo Scientific) were activated in furnace at 350 °C for 4 h and stored in a desiccator prior to use.

2.3. Sample extraction

Accelerated solvent extraction system ASE 350 (Dionex, Thermo Fisher Scientific, USA) was used for the simultaneous extraction and clean-up of 6 indicator PCBs and 18 OCPs in soil samples. It is apparent that OCPs significantly vary in their chemical structure (in contrast to PCBs, which are rather similar), thus extraction conditions that work well for certain OCPs might not necessarily work for others (Hussen et al., 2006). During ASE method development, several parameters such as temperature, extraction time, flush volume, number of extraction cycles, sorbent type and solvents were optimized. In order to gain acceptable recoveries, different ratios of solvent as hexane, dichloromethane and acetone were used. Also different extraction temperatures, extraction times and number of extraction cycles were tested. Method described below gives the best results.

Soil sample (5 g) dispersed with diatomaceous earth (3 g) was loaded into a stainless-steel extraction cell (33 mL) above silica gel (2 g) and alumina oxide (3 g) layer, each separated by cellulose filters. Extraction was performed at 1500 p.s.i., temperature of 100 °C with a flush volume 140% of the extraction cell using *n*-hexane and methylene chloride (1:2, v:v). The extraction cell was purged with a nitrogen stream for 80 s and static extraction time was 5 min in two cycles. A vacuum rotary evaporator (Heidolph, Germany) was used to concentrate the ASE extracts to about 5 mL, and then gentle stream of nitrogen to concentrate the extracts to 0.1 mL. Each composite sample was prepared and analyzed in

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