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OCCURRENCE AND LEVELS OF HAZARDOUS ORGANIC COMPOUNDS IN SOILS FROM INDUSTRIAL AREAS

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Abstract

Hazardous organic compounds are organic chemicals characterized by toxicity, persistence, and bioaccumulation potential, as well as their ability to distribute across environmental compartments such as soil, water, and air. This study aims to evaluate the contamination levels of soils from industrial areas with polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), and organochlorine insecticides (OCPs) in order to establish a database for the development of a soil emission inventory. For this purpose, 40 soil samples were collected from industrial sites in the southern region of the country, extracted using hexane-like solvents, and analyzed by gas chromatography (GC) and high-performance liquid chromatography (HPLC). The results indicate that soil concentrations of PCBs ranged between 0.002 mg/kg and 0.326 mg/kg, whereas PAH concentrations ranged from 0.044 mg/kg to 12.022 mg/kg. Among OCPs, lindane and DDE - the main metabolite of DDT - were the most frequently detected OCPs in soil.

Key words*: contamination, OCPs, PAHs, PCBs, soil.*

INTRODUCTION

Polychlorinated biphenyls (PCBs), organochlorine pesticides (OCPs), and polycyclic aromatic hydrocarbons (PAHs) are environmentally persistent organic contaminants that share several physicochemical and toxicological characteristics. These compounds are hydrophobic and lipophilic, exhibiting low water solubility and a strong affinity for organic matter and biological lipids, which promotes their accumulation in soils, sediments, and living organisms (Rissato et al., 2006). Due to their chemical stability and resistance to degradation, they persist in the environment and can undergo long-range atmospheric transport, leading to global distribution. The Stockholm Convention on Persistent Organic Pollutants (POPs) was adopted in 2001 to eliminate or restrict chemicals that are persistent, bioaccumulative, toxic, and capable of long-range environmental transport (UNEP, 2001). Both PCBs and OCPs were included among the initial substances targeted for global control. PCBs were listed in Annex A (Elimination) at the Convention because of their extensive industrial use and widespread environmental contamination. Parties were required to cease production and new uses, identify and label PCB-containing equipment, and ensure environmentally sound management and disposal. The Convention set a goal of eliminating the use of PCBs in equipment by 2025 and achieving environmentally sound waste management by 2028. Several OCPs were also classified as POPs, reflecting decades of evidence linking them to ecological damage and human health risks (Jones & Voogt, 1999). Pesticides such as aldrin, dieldrin, endrin, heptachlor, chlordane, toxaphene and mirex were included in Annex A (Elimination), while DDT was additionally placed in Annex B (Restriction) to allow limited use for malaria vector control under specific condition (Lallas, 2001). PAHs are organic pollutants composed of two or more condensed rings. The US Environmental Protection Agency (USEPA) declared 16 compounds from the PAHs category as priority pollutants in 1983, due to their presence in the environment at high concentrations, their recalcitrant nature, and toxicity (Barathi et al., 2023). Many high-molecular-weight PAHs display similar persistence and bioaccumulative behavior as PCBs and OCPs. All three groups are associated with significant health risks, including carcinogenic, mutagenic, endocrine-disrupting,

and immunotoxic effects, making them important targets for environmental monitoring and risk assessment (La Merrill et al., 2020). These compounds originate primarily from anthropogenic activities, although some natural sources exist. PCBs were intentionally manufactured for use in electrical equipment, hydraulic fluids, plasticizers, and other industrial applications, and their release occurred through production, usage, disposal, and leakage from contaminated materials (Rubin, 1999; Hannah et al., 2022; Sandu et al., 2025). OCPs were widely applied in agriculture and public health programs for pest control, with environmental contamination resulting from large-scale application, improper handling, and long-term persistence in soils (Jones & Voogt, 1999). In contrast, PAHs are mainly formed as by-products of incomplete combustion of organic matter, including fossil fuels, biomass burning, industrial processes, and vehicular emissions; however, they can also originate from natural events such as wildfires and volcanic activity (Ravindra et al., 2008). Overall, industrialization, agricultural practices, and combustion-related processes represent the principal sources of these contaminants in the environment.

This study aims to evaluate the contamination levels of soils from industrial areas with these compounds, providing baseline data that may contribute to the future development of a soil emission inventory supporting the implementation of Regulation (EU) 2019/1021 regarding persistent organic pollutants. The originality of this study consists in providing an up-to-date picture of soil contamination correlated with the current profile of industrial activity in southern Romania.

MATERIALS AND METHODS

40 topsoil samples (0-20 cm) were collected in spring of 2024 from areas potentially affected by industrial activities, as follows (Table 1):

- Pitești (P1-P5) and Mioveni (P8-P11)

In the vicinity of the city was located one of Romania's largest oil refineries, which may have contributed to the local environmental contamination through the historical use of PCB-containing equipment and the release of PAHs from oil processing. Additionally, historical agricultural practices in the surrounding area may have left residues of OCPs in soils. Mioveni also hosts industrial facilities. PCBs could originate from older industrial and electrical equipment, including facilities associated with automotive manufacturing. PAHs could be released through vehicle emissions, local industrial activity, and fuel combustion.

Table 1. Sample locations of the soil samples

Sample code	Sampling location	Latitude (N)	Longitude (E)
P1	Pitești 1	44°48'05"	24°56'19"
P2	Pitești 2	44°48'01"	24°56'15"
P3	Pitești 3	44°49'01"	24°55'08"
P4	Pitești 4	44°49'06"	24°56'10"
P5	Pitești 5	44°50'20"	24°52'58"
P6	Ploiești 1	44°53'01"	26°05'55"
P7	Ploiești 2	44°56'32"	26°04'15"
P8	Mioveni 1	44°57'11"	24°56'20"
P9	Mioveni 2	44°57'10"	24°56'28"
P10	Mioveni 3	44°57'01"	24°56'38"
P11	Mioveni 5	44°56'49"	24°55'33"
P12	Câmpulung 1	45°18'03"	25°04'02"
P13	Câmpulung 2	45°17'39"	25°03'48"
P14	Râmnicu Vâlcea 1	45°02'33"	24°18'06"
P15	Râmnicu Vâlcea 2	45°03'10"	24°18'43"
P16	Râmnicu Vâlcea 3	45°02'21"	24°17'52"
P17	Râmnicu Vâlcea 4	45°02'58"	24°18'46"
P18	Slatina 1	44°24'46"	24°23'36"
P19	Slatina 2	44°24'47"	24°24'02"
P20	Slatina 3	44°24'53"	24°24'28"
P21	Slatina 4	44°24'47"	24°24'22"
P22	Slatina 5	44°24'54"	24°23'51"
P23	Slatina 6	44°25'17"	24°39'59"
P24	Slatina 7	44°26'06"	24°23'19"
P25	Slatina 8	44°26'17"	24°23'13"
P26	Slatina 9	44°26'38"	24°22'44"
P27	Slatina 10	44°26'41"	24°22'50"
P28	Brașov 1	45°40'51"	25°36'52"
P29	Brașov 2	45°39'55"	25°35'30"
P30	București 1	44°26'01"	26°11'05"
P31	București 2	44°25'38"	26°10'42"
P32	București 3	44°24'28"	26°11'28"
P33	București 4	44°24'19"	26°11'29"
P34	București 5	44°22'03"	26°08'38"
P35	București 6	44°21'36"	26°08'58"
P36	București 7	44°21'56"	26°05'44"
P37	Chitila	44°29'37"	26°00'09"
P38	Popești Leordeni	44°21'52"	26°11'40"
P39	Jilava	44°20'44"	26°05'00"
P40	Domnești	44°24'26"	25°58'01"

- Ploiești (P6, P7)

Soil contamination with PCBs in the vicinity of the refinery is mainly associated with facilities and equipment that were in operation before the regulatory phase-out of PCBs. In addition, PAHs may be released into the environment through incomplete combustion processes related to petroleum refining activities, fuel combustion, and vehicle emissions

- Câmpulung, Râmnicu Vâlcea, Brașov (P12-P17, P28, P29)

Soil samples were collected from three former industrial sites: an industrial park in Câmpulung, an area in the vicinity of an industrial platform in Râmnicu Vâlcea and Brașov.

- Slatina

From Slatina, an important industrial center in southern Romania with a long history of manufacturing and heavy industry, were collected 10 soil samples (P18-P27) situated in industrial area.

- Bucharest and Ilfov county

The sampling points were located near industrial platforms (P 30, P31, P34, P35), pharmaceutical manufacturing facilities (P32, P33), tire and rubber material factories (P 38, P39), two concrete mixing plants (P36, P37) and an electrical transformer station (P40) (Figure 1).



Figure 1. A sampling point located near electrical transformers

Samples were collected in March when air temperatures averaged 15°C. Each sample consisted of a composite of five subsamples collected from the four corners and the center of a 5 × 5 m² plot. Representative soil samples weighing approximately 500 g were stored in glass containers, protected from light, and refrigerated until analysis.

PCBs analysis in soil was performed in accordance with SR EN 17322:2020, PAHs analysis followed SR EN 17503:2022, and OCPs determination was carried out according to ISO 23646:2022. Accordingly, in compliance with these standards for the extraction of PCBs, OCPs, and PAHs from soil, an automated Soxhlet extractor was employed. Ten grams of air- dried soil samples were extracted for 2 hours using a 1:1 mixture of hexane:acetone. The extract is then washed with water for chromatography to remove acetone, passed over anhydrous sodium sulfate to remove water and evaporated to the appropriate volume. The PCBs determination (seven isomers indicated by Order 756/1997): PCB 28, PCB 52, PCB 101, PCB 118, PCB 138, PCB 153, PCB 180 and the OCPs determination (α, β, γ, δ HCH, pp'-DDE, pp'-DDD and pp'-DDT) were performed on an Agilent gas chromatograph (GC) equipped with an electron capture detector (ECD), operated at 300 °C and a capillary column (30 m x 0.32 mm x 0.25 mm) with a nonpolar stationary phase (DB-5) (Preda et al., 2025).

The determination of PAHs was carried out using a Knauer ultrahigh-performance liquid chromatograph (UHPLC) equipped with an UV-VIS detection at a wavelength of 254 nm. Order 756/1997 stipulates the quantification of the following priority PAHs: anthracene, benzo(a)anthracene, benzo[b]fluoranthene, benzo[k]fluoranthene, benzo[a]pyrene, benzo[ghi]perylene, chrysene, fluoranthene, indeno(1,2,3)pyren, naphthalene, phenanthrene, pyren. The pair of compounds benzo[ghi]perylene and indeno(1,2,3)pyrene cannot be resolved using this type of detector. The separation of the remaining compounds was carried out on a C18 column (2.1 mm × 100 mm, 1.8 µm) maintained at 40°C. The mobile phase consisted of water and acetonitrile, applied under gradient elution conditions (Preda et al., 2025).

For calibration of the PCBs, OCPs and PAHs were used mixed standard solutions (PCB Calibration Mixture 10 µg/ml, PAH-Mix 100 µg/ml, Organochlorine Pesticide Mix 1000 µg/ml). To minimize contamination, all glassware was rinsed with solvents. To guarantee the representativeness and reliability of the results obtained, QA/QC procedures

included duplicate samples, blanks, and the analysis of a certified reference material.
The limit of quantification in soil is 0.001 mg/kg for each PCB and OCP isomers and 0.01 mg/kg for each PAH compounds. All the results are expressed as dry matter.

RESULTS AND DISCUSSIONS

PCBs content in soil

The total concentration of PCB compounds ranged from 0.002 mg/kg to 0.326 mg/kg. The highest content exceeds the alert threshold of 0.25 mg/kg, it was observed at sampling point P33, collected from the former site of a chemical manufacturing company specializing in the production of solvents, thinners, and other industrial chemical products (Figure 2). Schuhmacher et al. (2004) considered that in a chemical plant, PCBs may have been generated unintentionally as by-products of industrial processes.
20% of the analyzed samples showed normal values of concentration (<0.01 mg/kg), 77% had concentrations between the upper limit of the normal value and the alert threshold, and the remaining 3% exceeded the alert threshold.
Regarding the contribution of the seven isomers to the total PCB content, it can be observed that, at most sampling points, highly chlorinated compounds (PCB 138, PCB 153, and PCB 180) are predominant (Figure 3). Numerous studies have documented the presence of highly chlorinated PCBs such as PCB 138, PCB 153 and PCB 180 in soils from industrialized or urbanized areas, reflecting their persistence and historical contamination patterns (Nadal et al., 2007; Li et al., 2023; Sandu et al., 2025). For example, Li et al. (2023) reported hexachlorinated and heptachlorinated congeners among the dominant PCB profiles in soils around an industrial park. There are several sampling points (P8, P15, P29, P30, P35, and P36) where, in contrast, low-chlorinated compound, specifically PCB 28 and PCB 52, are predominant. The investigated sites are located in Mioveni, within the industrial area of Brașov and Râmnicu Vâlcea, on the industrial platform in Bucharest, and in areas situated in the vicinity of tire and rubber manufacturing facilities. Thus, in point P15, collected from a chemical plant from Râmnicu Vâlcea, the major omologue is PCB 52 (41%), the tetra-chlorinated isomer. In this point the PCB 52 concentration (0.034 mg/kg) exceeds the intervention limit (0.01 mg/kg).

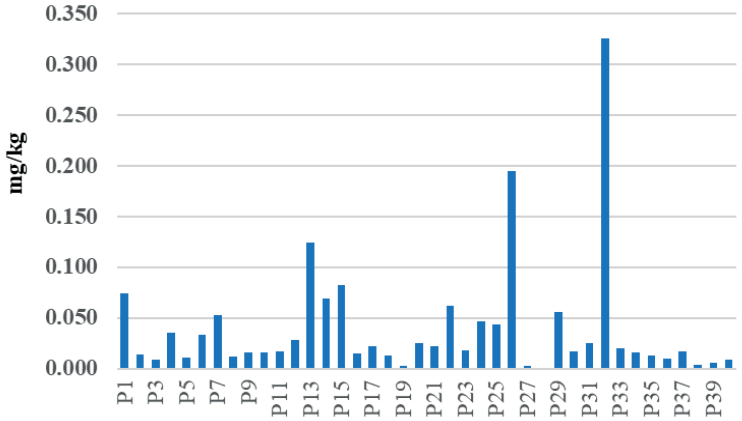


Figure 2. The total content of PCBs in soil samples collected from industrial areas

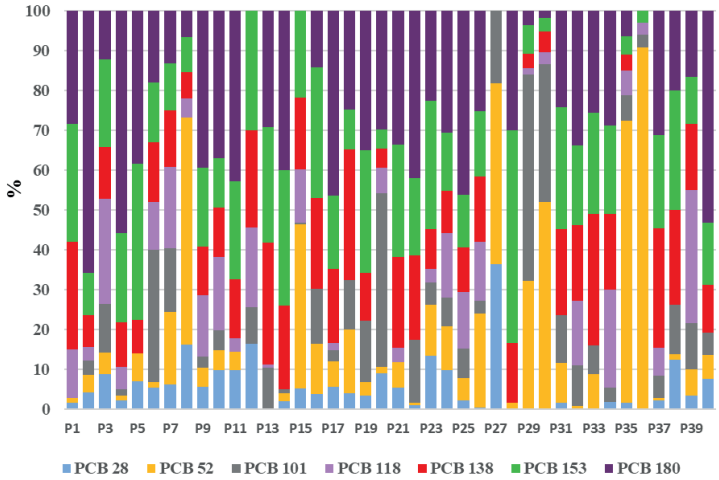


Figure 3. The contribution of the isomers to the total content of PCBs

Similar findings were reported by Zhang et al. (2014), who found that tetra-CBs (31%) and tri-CBs (19%) were the dominant PCB homologs in soils collected from an industrial area. The predominance of low-chlorinated congeners (PCB 28 and PCB 52) at several sampling sites may indicate the influence of local industrial emissions. Tri- and tetra-chlorinated PCBs are frequently associated with unintentional industrial releases and urban/industrial activities, including metal processing and the manufacture or use of polymer- and rubber-based materials (Sandu et al., 2025). Their presence may also be caused by dechlorination of higher chlorinated PCBs which break down over time in the environment (Tolefe et al., 2025). Salihoglu et al. (2011) have shown that intense industrial activity is the main source of high levels of PCB contamination in soil.

PAHs content in soil

The total concentration of polycyclic aromatic hydrocarbons (PAHs) in soils collected from the industrial areas ranged from 0.044 mg/kg to 12.022 mg/kg, with a mean value of 0.636 mg/kg. According to Order 756/1997, the normal values for total PAH concentrations in soil must be below 0.1 mg/kg. These results indicate that 10.91% of the analyzed soil samples were within the normal concentration range, 87.20% exceeded the normal threshold but remained below the alert level, and 1.89% surpassed the alert threshold of 7.5 mg/kg. The highest value (12.022 mg/kg) was recorded in the soil sample collected from Slatina industrial area (P32). Elevated concentrations were also recorded at sampling points P39 (1.505 mg/kg), P28 (1.342 mg/kg), and P35 (1.336 mg/kg); however, at these points, the alert threshold for sensitive land uses of 7.5 mg/kg was not reached. Similar results were reported by Wang et al. (2010) who found PAH concentrations ranging between 0.031 mg/kg and 1.475 mg/kg in soil collected from an industrial city. Regarding the individual compounds it was observed that the highest average concentrations in soil corresponded to chrysene, benzo[b]fluoranthene, and benzo[a]anthracene (Figure 4). These substances belong to the category of high molecular weight polycyclic aromatic hydrocarbons (HMW PAHs), containing more than four aromatic rings per molecule. Chrysene and benzo[b]fluoranthene exceeded the alert threshold for sensitive land uses of 2 mg/kg (2.210 mg/kg and 2.874 mg/kg, respectively), while benzo[a]anthracene was very close to this threshold (1.890 mg/kg). These concentrations were measured in soil sample P32 collected in the vicinity of a facility engaged in the production of graphite electrodes, refractory materials, and other coal-derived products - industrial activities that involve high-temperature processing and the use of petroleum-derived feedstocks. Among the low molecular weight compounds (LMW PAHs, with fewer than four aromatic rings per

molecule), the highest average soil concentration, 0.064 mg/kg, was observed for phenanthrene - which is also the most thermodynamically stable.

Examining the compound profile, it can be observed in Figure 4 that the decreasing order of concentrations in the studied soils is as follows: chrysene > benzo[b]fluoranthene > benzo[a]anthracene > phenanthrene > fluorene > benzo[a]pyrene > benzo[k]fluoranthene > anthracene > fluorene > naphthalene.

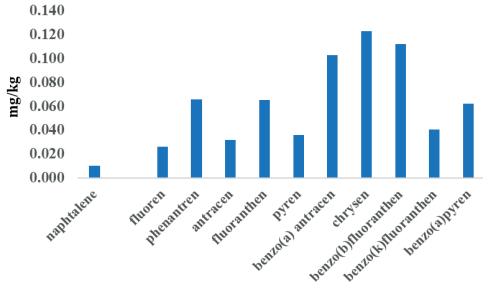


Figure 4. Average concentration of the individual PAH compounds in soils collected from industrial areas

It was observed that low molecular weight (LMW) hydrocarbons have a lower total concentration than high molecular weight (HMW) hydrocarbons, which may indicate a pyrogenic source (combustion-related processes). The ∑LMW/∑HMW PAH ratio can be used to distinguish petrogenic from pyrogenic sources (Zhang et al., 2008). Values of this ratio below 1 indicate pyrogenic sources, while values above 1 suggest petrogenic sources. Petrogenic sources include crude oil and petroleum-derived products such as kerosene, gasoline, lubricating oils, and asphalt. Pyrogenic sources result from combustion processes, including the burning of fossil fuels (coal, wood, oil) or biomass (forests, grasslands, crop residues).

In this study, the ∑LMW/∑HMW ratio ranged from 0.046 to 2.102. These values indicate the presence of pyrogenic sources in approximately 94% of the analyzed soil samples. Ratios above 1 were observed in samples collected from the Pitești refinery area (all samples), in one sample from the pipe manufacturer area in Slatina, and in the soil sample from the pharmaceutical factory in Bucharest.

Organochlorine insecticides in soil

OCPs have been widely used in agriculture on a global scale for several decades. Montory et al. (2017) showed that various organochlorine compounds, including hexachlorocyclohexanes (HCHs) and dichlorodiphenyltrichloroethanes (DDTs), remain widely present in the environment due to their persistence, despite their production and use having been banned in developed countries during the 1970s and 1980s. In Romania OCPs have been banned since 1985 (Dirtu et al., 2006). However, in our country too, studies show the presence of these residues in the soil (Ene et al., 2012; Tarcau et al.; 2013).

The total concentration of HCHs in collected soil samples ranged between 0.011 mg/kg and 0.291 mg/kg with an average concentration of 0.085 mg/kg. Lindane is present in 84% of the analyzed samples, although in concentrations around normal values (< 0.001). A particular case is observed for the samples collected near the Râmnicu Vâlcea plant (P14-P17), which are contaminated with all four HCH isomers. In this industrial area, the β-HCH isomer exceeds the alert threshold for less sensitive sites (0.15 mg/kg), as defined by Order 756/1997, and lindane reaches and even exceeds the alert threshold for sensitive uses of 0.02 mg/kg (Figure 5).

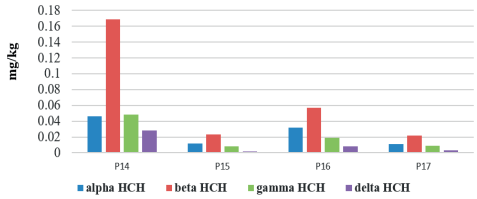


Figure 5. HCH isomer ratio in soil samples collected from Râmnicu Vâlcea

The concentrations of the other HCH isomers (α- and δ-HCH) exceed the upper limit of the normal range (< 0.002 mg/kg and < 0.001 mg/kg, respectively) but remain below their respective alert thresholds (0.1 mg/kg and 0.005 mg/kg, respectively). As illustrated in Figure 5, β-HCH exhibits the highest concentration in this area. Since β-HCH is the most persistent isomer, its predominance relative to the other isomers suggests that the contamination is of historical

origin (Wohrnschimmel et al., 2012). Regarding the total content of DDTs, the mean value of concentration is 0.015 mg/kg, ten times lower than the upper limit of normal values (<0.15 mg/kg). Similar observations were made by Tarcau et al. (2013), who reported that p,p′-DDE and p,p′-DDT were the major contributors to sum DDTs accounting for 54% and 38% of the total concentration, respectively.

CONCLUSIONS

Regarding PCB contamination, 20% of the analyzed soil samples exhibited normal value of concentrations (<0.01 mg/kg), while 77% showed levels between the normal value and the alert threshold, and 3% exceeded the alert threshold. The highest concentration (0.326 mg/kg) was recorded in a soil sample collected from the former site of a chemical manufacturing facility producing solvents, thinners, and other industrial chemicals.

For PAHs, 10.91% of the analyzed soil samples were within the normal range, 87.20% exceeded the normal level but remained below the alert threshold, and 1.89% surpassed the alert level (7.5 mg/kg). The maximum concentration (12.022 mg/kg) was observed in a soil sample collected from the Slatina industrial area. Chrysene, benzo[b]fluoranthene, and benzo[a]anthracene were the dominant PAH compounds detected.

Lindane and p,p′-DDE were the predominant organochlorine pesticide residues in the analyzed soils. The predominance of β-HCH in soils collected from the Râmnicu Vâlcea industrial platform suggests legacy contamination associated with historical industrial activities.

The distribution and the concentrations of PCBs, PAHs, and OCPs observed in this study confirm the strong influence of historical and current industrial activities and provide valuable input for the development of a soil emission inventory under Regulation (EU) 2019/1021.

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