Romanian Journal of Ecology & Environmental Chemistry, 7(2), 2025
https://doi.org/10.21698/rjeec.2025.204

Article

Organochlorine pesticides and polychlorinated biphenyl levels in sediments from Petrolifera and Porto Romano ports in Albania

JONIDA TAHIRAJ*, SONILA SHEHU, ESMERALDA HALO, BLEDAR MURTAJ, ELDA MARKU, AUREL NUR

University of Tirana, Faculty of Natural Sciences, Department of Chemistry, Boulevard "Martyrs of the Nation", Sheshi "Mother Teresa", Tirana, Albania
**Corresponding author: jonida.tahiraj@fshn.edu.al*

Received:	*Reviewed:*	*Accepted:*
15.07.2025	*30.09.2025*	*07.10.2025*

Abstract
Organochlorine pesticides (OCPs) and polychlorinated biphenyls (PCBs) pose significant concerns due to their harmful impacts on both wildlife and human health. This study analyzed the concentrations and distribution patterns of OCPs and PCBs in surface sediments collected from Porto-Romano and Petrolifera ports. The results showed that sediment samples from Porto-Romano contained higher levels of OCPs and PCBs than those from Petrolifera. Notably, Petrolifera Port dominated low-chlorinated PCBs, such as PCB28 and PCB52, whereas Porto-Romano displayed a mixture of both high- and low-chlorinated PCBs. Sediment quality guidelines (SQGs) for aquatic ecosystems were used to assess the potential impact of the detected PCBs and OCPs in sediments from both sites.

Keywords: *OCPs, PCBs, port areas, sediments*

INTRODUCTION

Persistent organic pollutants (POPs) are organic compounds that originate from either natural sources or human activities and resist breakdown through photolytic, chemical, and biological processes [1]. Although the manufacture and application of various persistent organic pollutants, especially organochlorine pesticides (OCs) and polychlorinated biphenyls (PCBs), have been prohibited or limited in numerous countries, these compounds continue to be prevalent. They can be found in even the most remote regions of the globe because of their ability to migrate long distances from their sources, which is attributed to their relatively high Henry's law constants [2,3]. They can migrate to the atmosphere and be transported over long distances in a gaseous state before undergoing dry or wet deposition in marine environments. Therefore, the aquatic environment is a significant reservoir for persistent organic pollutants, allowing these compounds to accumulate in various compartments [1]. Sediments play an essential and dynamic role in marine areas, storing a wide variety of POPs either temporarily or permanently [4]. These compounds enter the ecosystem through direct discharge, atmospheric deposition, and runoff from terrestrial sources. The sources of PCBs are primarily related to their past use in transformers, electrical equipment, industrial activities, and port operations [5]. They can bioaccumulate in fatty tissues of fish and mammals. Numerous studies have reported that sediments from various harbors, bays, and coastal areas contain high levels of PCBs and OCPs due to the combination of their diversified and industrialized economy, small geographic size, and the high economic importance of coastal resources for fisheries and tourism [4,6,7]. Understanding their presence and distribution is crucial for assessing environmental quality. Therefore, this study examines the presence of persistent organic pollutants along the Albanian coastline, specifically in the Petrolifera and Porto Romano areas, by measuring the concentrations of OCPs and PCBs in sediments. It also compares the port levels with those reported in similar studies and assesses sediment quality using a methodology that combines chemical parameters with their associated biological effects [8].

MATERIALS AND METHODS

Study area

Two industrial ports in Albania were chosen as sampling locations: Petrolifera and Porto Romano. Petrolifera is situated in Vlora Bay along the Albanian Adriatic coast. The management of oil, gas, and their by-products occurs at Petrolifera Port, approximately 3.7 km north of Vlora Port. This facility includes an oil jetty designed for tanker discharge operations. Porto Romano is the largest industrial port in Albania, located 6.5 kilometres north of Durrës city. Before 1990, this area was mainly known for its chemical manufacturing plant, which produced agricultural pesticides and chemicals used in leather processing [9]. The operations of this plant have had a significant impact on the environment, resulting in groundwater contamination and posing serious health risks. Oil, gas, and their derivatives are processed at Romano Port, situated about 11 km north of Durrës Port. This facility features an oil jetty capable of handling tankers with a maximum draft of 11 meters for unloading purposes.

In May 2023, nine surface sediment samples were collected from Porto Romano port, while eight samples were obtained from Petrolifera port using a Van Veen grab. The samples from Porto Romano were designated as SPR stations, whereas those from Petrolifera were labelled as SP stations. Four sampling sites (SP_1, SP_2) and (SP_7, SP_8) were located outside the port area, while the remaining sites (SP_3 to SP_6) were situated within the port area. The sampling methodology was consistent for both Porto Romano and Petrolifera. Maps of the sampling locations are shown in Figure 1.

Station	Latitude	Longitude
SPR-1	41.372743	19.417985
SPR-2	41.374428	19.419772
SPR-3	41.377875	19.421558
SPR-4	41.379292	19.42166
SPR-5	41.380842	19.421669
SPR-6	41.381518	19.42183
SPR-7	41.384041	19.421115
SPR-8	41.387518	19.419688
SPR-9	41.390614	19.414147

Station	Latitude	Longitude
SP-1	40.469099	19.454158
SP-2	40.470265	19.452218
SP-3	40.471354	19.450069
SP-4	40.472675	19.448637
SP-5	40.474151	19.447311
SP-6	40.476171	19.445167
SP-7	40.477415	19.442922
SP-8	40.479202	19.441287

Fig. 1. Location of sediment sampling points at Porto Romano (labelled SPR-1) and Petrolifera (labelled SP-1)

OCPs and PCBs were determined using the procedure described by [6]. The sampling method was based on ISO 5667-3:2018. Briefly, the sediments were air-dried, ground, and passed through a sieve. A 63 µm fraction was used for further analysis. Sediment samples (15 g) were treated with 40 mL of n-hexane/dichloromethane (3:1, v/v), and extraction was performed in an ultrasonic bath for 60 min at 30 °C. After extraction, 2 g of anhydrous sodium sulphate was added to remove any remaining traces of water, and metallic mercury was added to remove any sulphur, mainly due to its high effectiveness at removing elemental sulphur (S_8), which can interfere with electron capture detection (ECD). Moreover, in other analyses, especially under ISO 23646:2022 [10], we will begin to use safer alternatives such as activated copper and sulphur scrubbers, which are increasingly used to reduce mercury hazards. The concentrated extract was purified on a Florisil column. The target fractions were eluted using 20 ml of n-hexane/dichloromethane (4:1, v/v). In addition, the combined eluates

were collected in Kuderna-Danish and evaporated to 2 mL, ready for instrumental analysis. The analytical method used for the determination of organochlorine pesticides (OCPs) in soil samples was based on the procedures outlined in ISO 10382:2002 [11].
The target compounds were analysed simultaneously using a Varian 450 GC gas chromatograph with an ECD detector and an Rtx-5 (30 m long x 0.25 mm i.d. x 0.25 μm) capillary column. Helium was used as the carrier gas at a flow rate of 1 mL/min, and nitrogen as the makeup gas at a flow rate of 24 mL/min. the oven temperature program used in GC-ECD is: initial temperature: 70°C, hold for 2 min; ramp 1: 5 °C/min to 220°C, hold for 5 min; ramp 2: 5 °C/min to 300°C, hold for 12 min; total run time: 65 minutes.
The sediment samples were analysed for the following analytes: PCBs (IUPAC# 28, 52,101, 118, 138, 153, and 180); pesticides (∑DDTs (p,p′-DDT, p,p′-DDE and p,p′-DDD), ∑HCHs (α-HCH, β-HCH, γ-HCH and δ-HCH), ∑Chlordanes (γ-Chlordane, α-Chlordane), ∑Chlors (Heptachlor, Heptachlor Epoxide, Methoxychloroxy), ∑Endosulfans (Endosulfan I, Endosulfan II, Endosulfan sulphate), ∑drins (Aldrin, Dieldrin, Endrin, Endrin Ketone and Endrin aldehyde,) and Mirex.
The chromatograms of the analytes of interest are shown in figures 2 and 3.

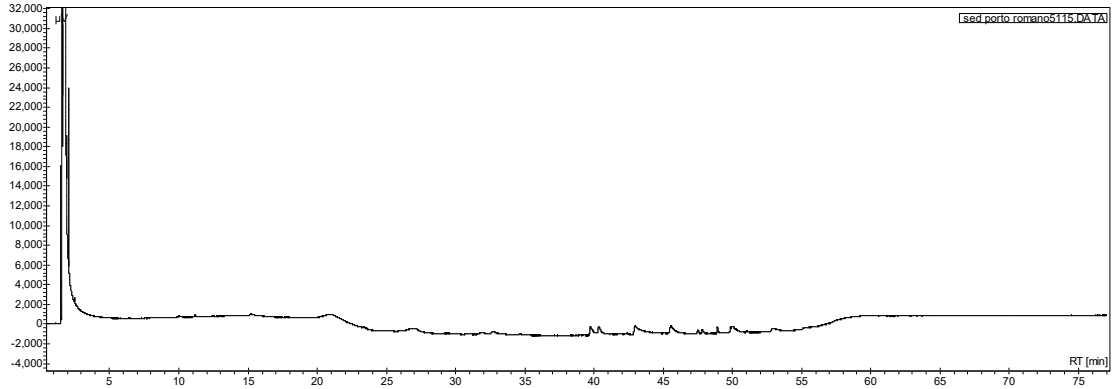

Fig. 2. OCPs and PCBs were analysed simultaneously in sediment samples in the Porto Romano Port

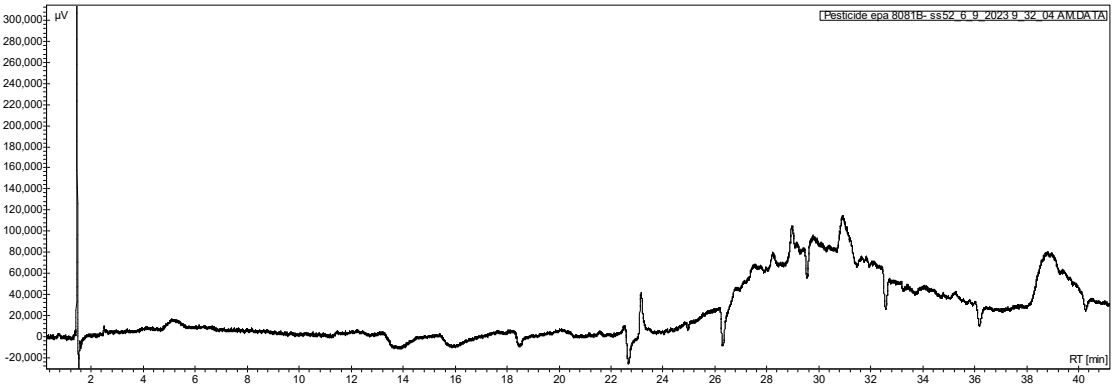

Fig. 3. OCPs and PCBs were analysed simultaneously in sediment samples in the Petrolifera Port

Quantification of the target compounds was performed using an external standard calibration. All data were statistically analyzed using MiniTab 18.

RESULTS AND DISCUSSION

OCP concentrations

The concentrations of OCPs, expressed as the sum of the different pesticides in sediment samples from Petrolifera and Porto Romano ports, are presented in Table 1. OCPs were found at almost all sampling locations in both areas. The concentration of OCPs (ΣOCPs) in sediments from Petrolifera ranged from 9.761 ng g^{-1} to 26.31 ng g^{-1}, with a mean value of 20.31 ng g^{-1}. The levels of total OCPs in the sediments from Porto Romano ranged from 9.438 ng g^{-1} to 109 ng $g^{-1,}$ with a mean value of

55.61 ng g^{-1}. The average of the total OCP levels was lower than those reported by Mehlhorn et al. [12] (470 ± 210 ng g^{-1}). In sediment samples from Petrolifera, the ∑HCH varied from 3.584 to 7.674 ng g^{-1}, ∑DDT from n.d to 3.846 ng g^{-1}, ∑drins from 0.055 ng g^{-1} to 6.49 ng g^{-1}, ∑endosulfans from 0.946 ng g^{-1} to 8.204 ng g^{-1}, ∑chlors from 0.447 ng g^{-1} to 9.59 ng g^{-1}, and Σchlordanes from n.d to 2.12 ng g^{-1}. In sediment samples from Porto Romano port the ∑HCH varied from 2.75 to 51.16 ng g^{-1}, ∑DDT from n.d to 1.168 ng g^{-1}, ∑drins from 0.526 to 24.03 ng g^{-1}, ∑endosulfans from 0.73 to 31.18 ng g^{-1}, ∑chlors from 0.884 to 24.46 ng g^{-1}, and Σchlordanes from n.d to 19.56 ng g^{-1}. Mirex was not detected in either area.

The ∑HCH levels detected in Petrolifera were lower than those in the Porto Romano port, indicating their long-standing presence in the area. Although technical Lindane has been banned in Albania since 1995, remnants of old stockpiles can still be found in Porto Romano. The isomeric profiles of HCHs are shown in Figure 3. δ-HCH was the most dominant isomer in both ports. The HCH levels found in both ports indicate that contamination primarily originates from the historical use of lindane.

Table 1. The mean, minimum, and maximum concentrations of organochlorine pesticides in sediment samples from Petrolifera and Porto Romano ports

Analytes	Petrolifera port (n=8)			Porto Romano port (n=9)		
	Mean	Min	Max	Mean	Min	Max
ΣHCH	6.301	3.584	7.674	21.49	2.75	51.16
Σdrins	4.04	0.055	6.498	8.15	0.526	24.02
Σchlors	4.02	0.447	9.596	6.13	0.884	24.46
Σchlordanes	0.533	n.d	2.12	3.53	n.d	19.56
Σendosulfans	4.368	0.946	8.204	15.64	0.73	31.18
ΣDDTs	1.047	n.d	3.846	0.51	n.d	1.168
ΣOCPs	20.31	9.761	26.306	55.61	9.438	109

where: **ΣHCH** (α-HCH, β-HCH, Lindane, δ-HCH); **Σdrins** (Aldrin, Dieldrin, Endrin, Endrin aldehyde, Endrin ketone); **Σchlors** (Heptachlor, Heptachlor epoxide, Methoxychlor) **Σchlordanes** (α-Chlordane, γ-Chlordane); **Σendosulfans** (Endosulfan I, Endosulfan II, Endosulfan sulfate); **ΣDDTs** (p,p′-DDT, p,p′-DDD, p,p′-DDE).

The concentration of ∑DDT in sediment samples from Petrolifera port was composed of 16% p,p′-DDT, 72% p,p′-DDD, and 12 % p,p′-DDE, whereas in the sediment samples from Porto Romano was composed of 21% p,p′-DDT, 16% p,p′-DDD, and 63 % p,p′-DDE. This is consistent with the expected degradation pattern of DDT. The low concentration of p,p′-DDT suggests an old input of technical DDT, which, according to the WHO, contains ~ 77% p,p′-DDT, 15% o,p′-DDT, and negligible percentages of its metabolites p,p′-DDD (0.3%) and p,p'-DDE (4%) [13]. The low proportion of p,p′-DDT measured in sediments suggests an old input of technical DDT in both environments. As mentioned in other studies, to distinguish between current and historical usage of DDT, we calculated the ratio of p,p′-DDT to the sum of (p,p′-DDD+ p,p′-DDE) [14]. Hiller et al. [15] used a ratio of 0.4 to distinguish between recent and historical DDT inputs. Sediments from both areas were characterized by DDT/(DDD + DDE) ratios that did not exceed 0.4, indicating the historical use of DDT and the long-term degradation process. The study areas can still be impacted by agricultural practices in inland and coastal regions through water and sediment transport along the shoreline, as well as through atmospheric deposition and surface runoff.

Aldrin was detected in 75% of the samples from the Petrolifera port and in 33% of the samples from Porto Romano, whereas dieldrin was detected in all sediment samples in both areas. Its presence in lower abundance compared to dieldrin indicates the same findings reported by Mehlhorn et al.[12] regarding the long-term degradation of aldrin into a more stable and persistent dieldrin metabolite. The lower concentrations of dieldrin compared to other metabolites (endrin, endrin ketone, and endrin aldehyde) suggest their historical use in both areas. Many researchers have used the heptachlor/heptachlor epoxide ratio to distinguish between recent and past applications of heptachlor. In the Petrolifera port, the heptachlor/heptachlor epoxide ratio in four samples was greater than one,

suggesting possible recent use. This contrasts with findings from Porto Romano, which indicate historical use of heptachlor.

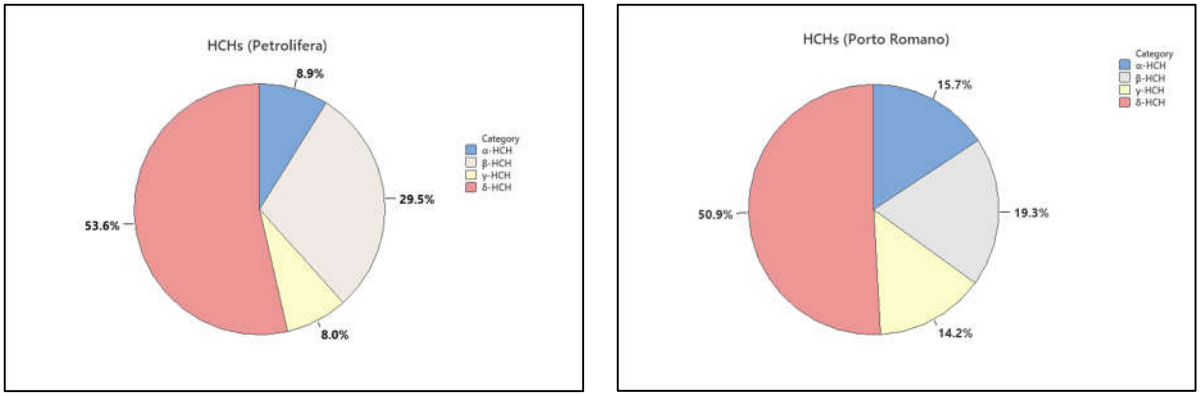

Fig. 4. Isomeric profile of HCHs in (a) Petrolifera port and (b) in Porto Romano port

In terms of distribution, our results exhibited slight variation in ∑HCHs, ∑drins, ∑chlors, ∑chlordanes, and ∑endosulfans in sediments from Petrolifera compared to their distribution found in sediment samples from Porto Romano. This distribution is expected due to the presence of Lindane and the use of OCPs. Different concentrations of pollutants can be attributed to different degradation behaviors and weathering of compounds.

In this study, the relatively low levels of OCPs in sediment samples indicate a significant decline due to the long-term ban on these insecticides in Albania. OCPs in aquatic environments mainly originate from pesticide use in agriculture, runoff from farmland soils, and industrial wastewater discharge. Pesticides may still be present in environmental compartments in some regions of the country, either because of historic use or long-range transport from a single source.

Additionally, the concentrations of OCPs found in this study were compared with those from other studies. The sediment OCP levels measured in both areas were lower than those reported in Richards Bay Harbour. Total OCP concentrations at Petrolifera Port were lower than in other studies, but Porto Romano had higher levels than Jieshi Bay.

Table 2. Levels of some OCPs in sediments from different areas

Locations	∑OCP ng g^{-1}	∑HCH ng g^{-1}	∑DDT ng g^{-1}	∑drins ng g^{-1}	∑endosulfans ng g^{-1}	∑chlor ng g^{-1}	References
Richards Bay Harbour	135÷1020	35÷230	12÷350	43÷270	21÷260	18÷280	[16]
Jieshi Bay13	17.75÷ 38.98	14.64÷ 30.53	3.11÷8.45				[17]
Petrolifera Port	9.761÷ 26.31	3.584÷ 7.674	n.d÷3.846	0.055÷ 6.498	0.946÷8.024	0.447÷ 9.596	This study
Porto Romano Port	9.438÷ 109	2.75÷ 51.16	n.d÷1.168	0.526÷ 24.02	0.73÷31.18	0.884÷ 24.46	This study

n.d. not detected

PCB concentration

Seven individual congeners (PCB 28, 52, 101, 118, 138, 153, and 180) were measured in each sediment sample collected from both areas. The analysed PCBs were also categorized as tri-CBs (PCB 28), tetra-CBs (PCB 52), penta-CBs (PCB 118), hexa-CBs (PCB 153), and hepta-CBs (PCB 180). The concentrations of Σ_7PCBs in surface sediment samples from Petrolifera Port ranged from 5.792 to 35.9 ng g^{-1}, with a median of 15.08 ng g^{-1} and a mean of 15.08 ng g^{-1}. In Porto Romano Port, the sum of Σ_7PCBs ranged from 4.64 to 25.15 ng g^{-1}, with a median of 14.69 and a mean of 15.32 ng g^{-1}. The highest concentration of Σ_7PCBs was observed at Petrolifera Port. The levels found

in this study were similar to those reported in Izmir Bay, Turkey, as shown in Table 3. The PCB levels in the two ports are lower than those reported from Southern Italy, Volturno River, Portuguese Coastal area, Portugal, Egyptian Coast, Egypt, Tripoli Harbor, Lebanon, but higher than those in the Port Elizabeth Harbour, South Africa. The high level of $\sum_7$PCBs at Porto Romano port may be due to the emission of PCBs from paints, old equipment, or materials used in some vessels, especially during ship maintenance activities. It could also be linked to the use of capacitors and transformers in the industrial area nearby. Indeed, other studies report that PCBs can originate from shipping and industrial activities [18]. Regular dredging operations may influence the deposition of PCBs in shipping channels, which can be exacerbated by river runoff.

Table 3. Levels of PCBs in sediments from coastal areas worldwide

Locations	**$\sum$PCBs (Range) ng g^{-1}**	**References**
Volturno River, Southern Italy	$\sum_{32}$ PCBs (4.37÷64.4)	[19]
Portuguese Coastal area, Portugal	$\sum_7$ PCBs (0.3÷466.8)	[20]
Egyptian Coast, Egypt	$\sum_{96}$ PCBs (2.29÷377)	[21]
Izmir Bay, Turkey	$\sum_{20}$PCBs (0.21÷31.95)	[22]
Tripoli Harbor, Lebanon	$\sum_{28}$PCBs (17.81÷301.95)	[18]
Port Elizabeth Harbour,South Africa	$\sum$PCBs (0.56÷2.35)	[23]
Porto Romano port	$\sum_7$ PCBs (4.64÷25.15)	This study
Petrolifera Port	$\sum_7$ PCBs (5.792÷35.9)	This study

The profiles of the homologous PCBs are shown in Figure 4. At the Petrolifera port, PCB28, PCB52, and PCB138 were the most abundant congeners, making up 89% of the total PCBs. In the sediment from Porto Romano port, the three most abundant congeners were PCB153, PCB28, and PCB138, accounting for 62% of the total PCBs. Low-chlorinated PCBs (PCB28 and PCB52) dominated the homolog distribution at Petrolifera port, while at Porto Romano port, the distribution was dominated by both high- and low-chlorinated PCBs. The homolog profile at the Petrolifera port aligns with findings from other studies [24, 255]. Since PCB use has been banned for many years, the main factors influencing PCB distribution are atmospheric deposition and surface runoff. Lower chlorinated PCBs are more mobile, tend to accumulate in the atmosphere, and are removed by precipitation, entering the water environment via river runoff and sediment sinking. Other studies suggest that higher chlorinated PCBs may result from microbial dichlorination in anaerobic sediments.

Elevated PCB concentrations observed in some sites within the inner areas of these two ports may be linked to intensive human activities. Recently, these areas have seen significant growth in tourism, aquaculture, and maritime activities. PCB 118 was the most prevalent component because of its resistance to degradation, which aligns with findings from other studies [3, 26]. Low-chlorinated PCBs found in these sediments are likely due to their tendency to accumulate in the atmosphere. The tri- and penta-PCBs have been widely used in transformers, capacitors, and as paint additives [27]. Therefore, discharges from old electrical equipment and non-point sources, such as atmospheric deposition and surface runoff, influence the distribution of PCBs [25]. These compounds, with 2 to 5 chlorine atoms, can be washed out by rain and enter water systems through runoff, eventually settling in sediments [24, 25]. Moreover, higher-chlorinated PCBs may undergo reductive dichlorination under anaerobic conditions over time, resulting in the formation of lower-chlorinated congeners.

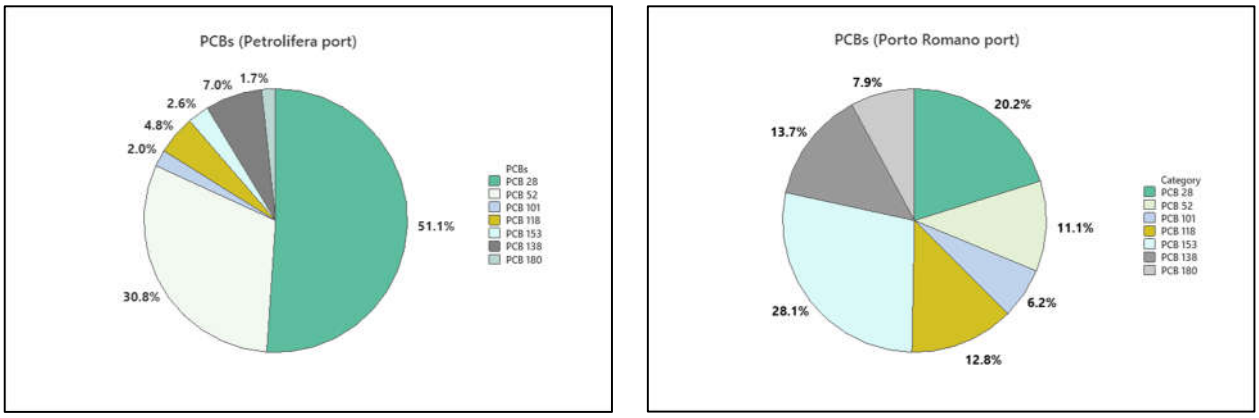

Fig. 4. The profiles of the homologous PCBs in each area

Ecological risk assessment

PCBs and OCPs are toxic to aquatic organisms, and the potential ecotoxicological risks they pose have raised significant concern in estuarine and coastal areas [28]. Currently, in Albania, there are no guidelines available for POPs in sediments. In this study, the preliminary evaluation of potential toxicological effects was conducted using the Sediment Quality Guidelines (SQGs) proposed by the National Oceanic and Atmospheric Administration (NOAA) [29].

Each compound is classified by using two guideline values, known as ERL, the effect range low, which represents the concentration below, (the adverse effects are rarely observed), and the ERM, effect range median (above which adverse toxicological effects would frequently occur. Moderate toxicological incidence is expected at concentrations between the ERL and ERM values. These SQGs have been widely deployed in several studies [19]. Summarized PCB and OCP concentrations in sediments collected from both areas, as well as ERL and ERM values calculated using sediment quality guidelines, are given in the Table 4. In addition, the percentages of samples that exceed the ERL and ERM values are presented.

Table 4. Comparison of individual OCP concentrations and the sum of PCBs measured in surface sediments from both areas, with sediment quality guidelines proposed by NOAA [29]

			Petrolifera port			**Porto Romano port**		
Compounds	ERL	ERM	<ERL %	ERL-ERM %	>ERM %	<ERL %	ERL-ERM %	>ERM %
Lindane	0.32	1	37.5	50	12.5	11	33	56
p,p'-DDT	1	7	100			100		
p,p'-DDD	2	20	87.5	12.5		100		
p,p'-DDE	2.2	27	100			100		
ΣDDTs	1.6	41	75	25		100		
Dieldrin	0.02	8	12.5	87.5			100	
Endrin	0.02	45	37.5	62.5		11	89	
Σchlors	0.5	6	12.5	62.5	25		67	33
ΣPCBs [b]	22.7	180	75	25		67	33	

ERL = effects range-low; ERM = effects range-median. Corresponding sediment limits are given in brackets: ERL (22.7), ERM (180) b Long and Morgan, 1990.

The percentage of samples that exceeded the ERM values for Lindane (56%) and Σchlors (33%) is significantly higher in Porto Romano than in Petrolifera, indicating a high probability of adverse toxicological effects. The concentrations of p,p'-DDT and p,p'-DDE in sediments were significantly lower than the ERL values. The sum of DDT concentrations indicates that only 25% of the samples exceeded the values of 1.6 ng g^{-1}, presenting a moderate effect. Endrin and Dieldrin values in sediment samples from Porto Romano showed higher percentages of samples with moderate toxicological effects.

For sediments from Petrolifera and Port Romano ports, 25% and 33% of the samples, respectively, had PCB concentrations above the ERL value, signalling a notable ecological risk related to the exposure of organisms to the concentrations of Σ_7 PCBs in these sediments.
Therefore, most of the samples indicate a low to moderate likelihood of adverse toxicological effects, except for lindane, and Σchlors could have a significant negative impact on the community; further monitoring of these compounds is recommended.

CONCLUSIONS

This study investigates the levels of OCPs and PCBs in the coastal sediments of Albania, focusing on the ports of Porto Romano and Petrolifera. The combined effect of port activities and industrial processes may act as sources of POPs, affecting harbour sediment quality. Overall, pollutant levels in the inner bay were higher than in the outer bay, indicating the influence of industrial emissions and harbour activities. The PCB concentrations in sediments from both ports showed a dominance of low-chlorinated PCB congeners. The ecological risk assessment indicated a low to moderate potential risk to organisms exposed to OCPs and PCBs in these sediments. Further monitoring is recommended to better understand contamination in both areas.

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Citation: Tahiraj, J., Shehu, S., Halo, E., Murtaj, B., Marku, E., Nuro, A., Organochlorine pesticides and polychlorinated biphenyl levels in sediments from Petrolifera and Porto Romano ports in Albania, *Rom. J. Ecol. Environ. Chem*., **2025**, 7, no.2, pp. 34÷42.