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Priority Substances in Waters of Albanian Ports

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GC/ECD/FID

Abstract: *This study was to determine the concentration of organochlorine pesticides, polychlorinated biphenyls, polycyclic aromatic hydrocarbons, and BTEX (benzene, toluene, ethylbenzene and o-, m- p-xylenes) in water samples of Adriatic Sea on the Albanian coastline. These pollutants are classified as priority substances because of their stability and toxicity. Stations near the ports of Durres and Vlora were selected for this study. Durres and Vlora ports are the largest ports in Albania which are processing more than 90% of shipping transport for people and commerce in Albania. Intense ship transport and commercial processing are the main reasons for water pollution in port areas. Water currents and new arrivals from Albanian rivers can influence their pollution. Marine water samples from the ports of Durres and Vlora were taken in for the same stations in two periods, May and July 2022. Liquid-liquid extraction (LLE) and hexane as extracting solvents were used to isolate organochlorine pesticides and PCBs by water samples. PAHs were extracted using two steps LLE technique. Firstly, by using dichloromethane and after that hexane as extracting solvent. Organic phases were dried by using Na2SO4 anhidrous. Clean-up procedures were performed by using SPE techniques. After the concentration, the samples were injected in Varian 450 GC equipped with ECD and FID detectors. BTEX was analyzed using HS/SPME technique followed by GC/FID technique. Organochlorine pesticides (mostly their metabolites) were detected almost in all analyzed water for both periods of sampling. The main factors can be their previous use, new arrivals from rivers, and the water currents of the Adriatic Sea. PCBs, PAHs and BTEX were found almost for all analyzed samples. Their presence could be because of anthropogenic factors (intense activity and ship transport) in the port area. The higher level of all pollutants was in July because of the intense ship transport in this period of the year. The higher levels were found inside port areas for both periods. Priority substance levels in water samples of Durres and Vlora ports were higher/comparable with reported levels of them from other stations of the Adriatic Sea (Albania coast).*

1. INTRODUCTION

This study determined priority substance concentrations in water samples for the ports of Durres and Vlora which are the main ports of Albania. These ports are located respectively in the central and south Adriatic Sea, Albanian coastline. Both ports are processed more than 90% of shipping for people and commerce in Albania. The port of Durres is the largest seaport in Albania. It is an artificial basin that is formed between two moles. The Port is located at the north end of the Bay of Durres, located in the central Adriatic Sea. Part of the port of Durres is a fishing harbor that lies at the north end of the East Mole. From 2014, the port ranks as the largest passenger port in Albania and one of the largest passenger ports in the Adriatic Sea. In the north of Durres port (5 km distance) is located the hydrocarbon ports of Porto-Romano.

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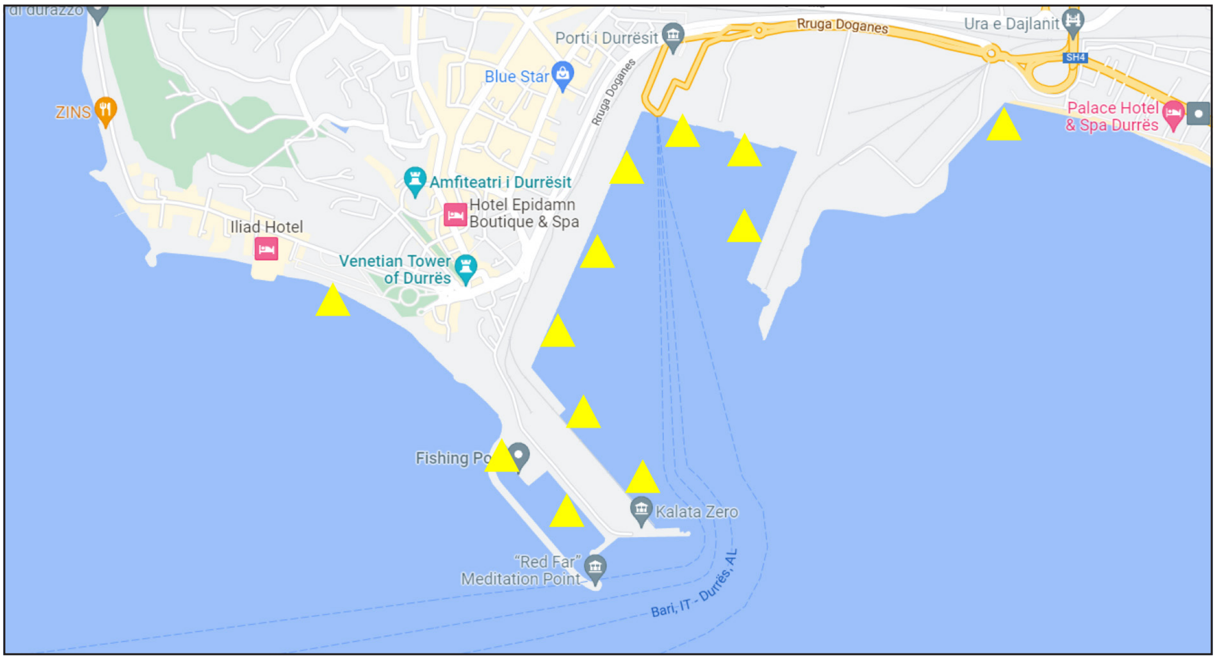
Shortly, it is thought that the port area will be re-conceptualized and expanded as one of the largest ports of the Mediterranean Sea. The port of Vlora is the second port of Albania. It is located in Vlora bay, near the city of Vlora, south Albania. The port is considered part of the Lungomare Master Plan of Vlora. Part of this project is the construction of a yacht port, and some new roads to make the port area most accessible. Until now, Vlora's port is the main function of passengers and commercial shipping. In fact, beside Vlora port in the area of Vlora bay are located other ports such as a petroleum port (Petrolifera near Zverneci), Marina port for Delta Force (near Radhima), Military Base of Orikumi and a fishing harbor (near Zverneci). In the area of Vlora bay operates some tourist ships and many small boats (motorized one) that serve as tourist transportation to visit beautiful areas of Sazani Island and the Karaburuni peninsula. Vlora bay (and Vlora's port) has intense ship transport and except this, the geographical position of the bay favors the concentration of pollutants inside of it. The negative impact of Vjosa and Semani rivermouth that bring new arrivals of pollutants (pesticide, hydrocarbons, detergents, etc.) as well as the internal/external water currents are not excluded as factors that affect pollution of Vlora bay. Vlora's port is affected by this pollution adding the pollution to this area by ships, automobile transport and commercial import/export that operate on it.

Organochlorine pesticides, polychlorinated biphenyls, polycyclic aromatic hydrocarbons and benzene are classified as Persistent Organic Pollutants (POP) because they are persistent for many years after their application (Shayler et al., 2009). Great concern was caused by chlorinated compounds, which proved to be extremely persistent in the environment and after that in the food chain (Penttila & Siivinen, 1996; WHO & FAO 1983; Wilhelm et al., 2002). For more than 50 years (after the Second World War to 90') organochlorine pesticides were used widely in Albania for agricultural purposes. The main agricultural areas are located in the western of the country near the Adriatic Sea. The use of pesticides in Albania after 1990 decreased rapidly due to migration and the immigration of the population. PCBs were not in use in Albania until 90'. They can be found only in some electrical transformers that were used in the early 1990s, but they were reported in many water ecosystems of our country because of their atmospheric depositions. PAHs and BTEX are pollutants generated by automobile transport, extracting/processing of the oil industry, coal mine, and other industries. These hydrocarbons could be found in marine water because of ship transport or some accidental spills of hydrocarbons. Forest burning and their natural background make them very often in the environment. These pollutants have high stability, high bioaccumulation capacity, and the ability to spread out far away from the application site. Generally, these compounds are difficult to degrade and can persist for many years in particular in water ecosystems (Corsi et al., 2010; Nuro et al., 2012).

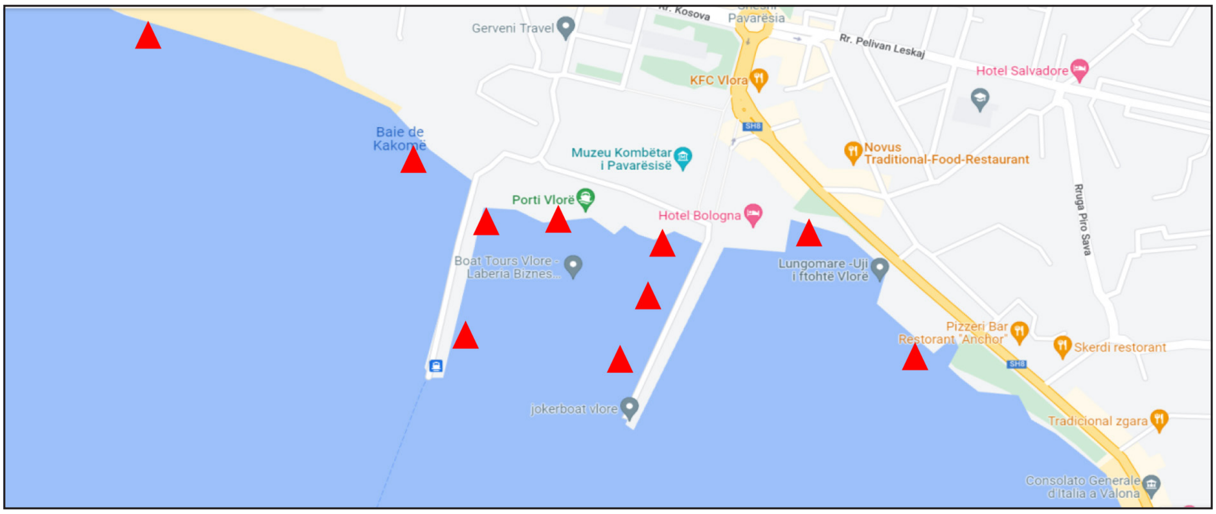
2. MATERIAL AND METHODS

2.1. Water Sampling in the Adriatic Sea

Water samples were taken in 12 different stations of the port of Durres (8 inside and 4 outside its area) and 10 stations of Vlora's port (6 inside and 4 outside port area) for two periods May and July 2022. Sampling periods were chosen for these months because May is a normal working period for both ports while July is the intense period (two to three times higher than the normal period). The sampling stations for Durres and Vlora ports are presented in Figure 1. A quantity of 2,5 litre of marine water was taken from each station in Teflon bottles. The sampling method was based on ISO 5667-3:2018. Water samples were transported and conserved at +4°C before their analysis.



a) Port of Durres sampling site, D1 (left) – D12 (right)



b) Port of Vlora sampling site, V1 (left) – V2 (right)

Figure 1. Sampling stations in the port of Durres and port of Vlora, May and July 2022.

2.2. Treatment of Water Samples for Pesticide and Pcb Analyzes

Liquid-liquid extraction was used for the extraction of organochlorine pesticide and polychlorinated biphenyls from marine water samples. One liter of water and 50 ml n-hexane as extracting solvent were added in a separatory funnel. After extraction, the organic phase was dried with 5 g of anhydrous Na_2SO_4 for water removal. A florisil column was used for the sample clean-up. 20 ml n-hexane/dichloromethane (4/1) was used for elution. After concentration to 1 ml hexane, the samples were injected in GC/ECD (Lekkas et al., 2004; Vryzas et al., 2009; Nuro et al., 2012).

2.3. Gas Chromatography Analysis of Pesticides and PCBs

Organochlorine pesticides and PCBs were analyzed simultaneously using capillary column type Rtx-5 (30 m long x 0.25 mm i.d. x 0.25 µm film thicknesses) on a gas chromatograph Varian 450

GC with ECD detector. Helium was used as carrier gas (1 ml/min) while nitrogen as make-up gas (24 ml/min). The manual injection was done in splitless mode at 280°C. The organochlorine pesticides detected were DDT-related chemicals (o,p-DDE, p,p-DDE, p,p-DDD, p,p-DDT), HCHs (a-, b-, γ- and d-isomers), Heptachlor's (Heptachlor and Heptachlorepoxide); Aldrin's (Aldrine, Dieldrine and Endrin) and Endosulfanes (Endosulfan alpha, Endosulfan beta and Endosulfan sulfat). Analysis of PCBs was based on the determination of the seven PCB markers (IUPAC Nr. 28, 52, 101, 118, 138, 153 and 180). Quantification of OCPs and PCBs was based on an external standard method (Vryzas et al., 2009; Lekkas et al., 2004; Nuro et al., 2014).

2.4. Treatment of Water Samples for PAH Analyzes

Two steps liquid-liquid extraction (LLE) was used for extracting PAHs from marine water samples. One liter of water with firstly 30 ml dichloromethane (first step LLE) and after that 30 ml hexane (second step LLE) as extracting solvent was added in a separator funnel. After extraction, the organic phase was dried with 5 g of anhydrous Na_2SO_4 for water removal. Extracts were concentrated to 1 ml hexane using Kuderna-Danish and then were injected in GC/FID for qualification/quantification of PAHs (Nuro et al., 2014; Wang et al., 2009; Lekkas et al., 2004).

2.5. GC/FID Determination of PAH in Water Samples

Gas chromatographic analyses of PAHs in water samples were realized with a Varian 450 GC instrument equipped with a flame ionization detector and PTV injector. VF-1 ms capillary column (30 m x 0.33 mm x 0.25 μm) was used for the qualification and quantification of 13 PAHs according to EPA 525 Method. Helium was used as carrier gas with 1 ml/min. FID temperature was held at 280°C. Nitrogen was used as the make-up gas (25 ml/min). Hydrogen and air were flame detector gases with 30 ml/min and 300 ml/min, respectively. EPA 525 Standard Mixture was used for qualitative and quantitative analysis of PAHs. Acenaphthylene, Fluorene, Phenanthrene, Anthracene, Pyrene, Benzo [a] anthracene, Chrysene, Perilene, Benzo [b] fluoranthene, Benzo [k] fluoranthene, Indeo [1,2,3-cd] pyrene, Dibenzo [a, b] anthracene and Benzo [g, h, i] perylene were determined in seawater samples. Quantification of PAHs was based on an external standard method (Nuro et al., 2014; Wang et al., 2009; Lekkas et al., 2004).

2.6. HS-SPME Technique for Determination of BTEX in Water Samples

Determination of BTEX in water samples was realized using solid phase micro-extraction in static headspace mode (HS/SPME) followed by GC/FID technique. 5 ml of water sample was put in a 10 ml headspace vial. 100 um PDMS fiber was used to extract BTEX from water samples. The adsorption process was realized at 50°C (using a water bath) for 45 minutes. The desorption process (30 seconds in 280°C) was realized in a PTV injector (HS mode was selected) of a Varian 450 GC instrument. VF-1 ms capillary column (30 m x 0.33 mm x 0.25 μm) was used for the separation of Benzene, Toluene, Ethyl benzene and Xylene isomers. Helium was used as carrier gas with 1 ml/min. FID temperature was held at 280°C. Nitrogen was used as the make-up gas (25 ml/min). BTEX Mixture was used for their qualitative and quantitative analysis based on an external standard method (Nuro et al., 2014).

3. RESULTS AND DISCUSSION

In this study were analyzed organochlorinated pesticides, PCBs, PAHs and BTEX were in marine water samples from the ports of Durres and Vlora, Adriatic Sea. These are the main ports of Albania for passengers and commercial shipping. Samples were taken in May and July 2022. Organochlorine pesticides, their degradation products and PCB markers were analyzed using GC/ECD techniques. The analysis of pesticides is based on the EPA 8081B standard, in which Lindane and its isomers, Heptachlors, Aldrins, Chlordanes, DDTs and Endosulfanes are analyzed. Analysis of PCBs in water samples was based on determination of seven PCB markers. Polycyclic aromatic and volatile hydrocarbons were analyzed using GC/FID techniques. OCPs, PCBs, PAHs and BTEX were classified as priority substances because of their stability and toxicity.

Organochlorine pesticides were detected in all water samples analyzed from the ports of Durres and Vlora, for both periods (May and July 2022). The higher average value of pesticides was found in Vlora port (July 2022) with 8.2 ng/l. Vlora samples were the most polluted for both periods. The presence of pesticides in the marine water of the Adriatic Sea must be due to their previous uses in agricultural areas near the ports, river discharges (new arrival because of soil rinsing), water currents, punctual sources near the port, etc. The total of pesticides for each station was higher for water samples in July 2022 (Figure 2). Agricultural activity and new arrivals from the rivers could be the main factors. Momentum values are not excluded. It was noted that levels of pesticides for Vlora stations were higher than for Durres because of the geographic position of Vlora Bay. It favors the concentration of pollutants inside the bay. The most polluted stations for Durres were D11 in May and D10 station in July while for Vlora were V4 in May and V6 in July (Figure 3). All these stations are inside the ports of Durres and Vlora. Semi-closed areas of ports influence their concentration. Distribution and profile of organochlorine pesticides (Figure 4) were found to be different for the ports of Durres and Vlora, for both periods (May and July 2022). Their presence is based mainly on the individual concentration of some pesticides. Endrine ketone was found higher for both ports in May but Endosulfane I, Heptachlor and d-HCH were found higher in July. This fact could be connected mainly with the momentum values of these pollutants in water samples. OCP levels in water samples of Albanian ports were comparable with the reported data in previous studies on the Adriatic Sea, Albanian part (Murtaj et al., 2014; Como et al., 2013, Nuro et al., 2017).

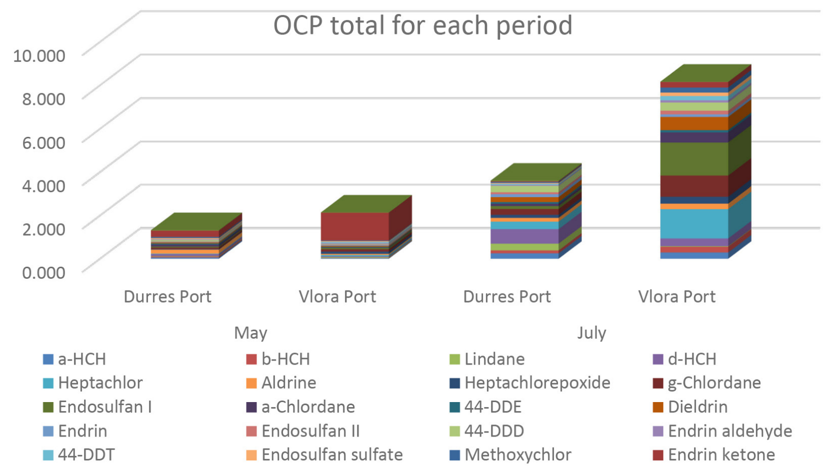


Figure 2. Total of organochlorine pesticides (ng/l) for both periods (May and July 2022) in water samples of Durres and Vlora ports, Adriatic Sea

Source: Own research

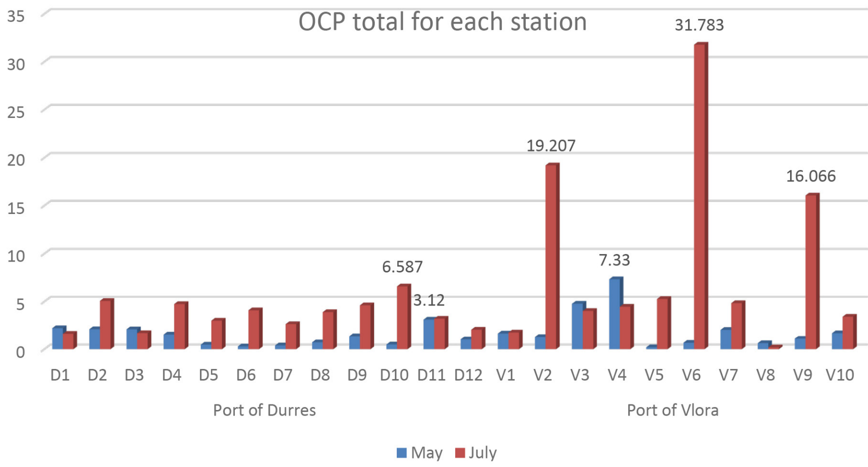


Figure 3. Total of pesticides (ng/l) for each station of Durres and Vlora ports
Source: Own research

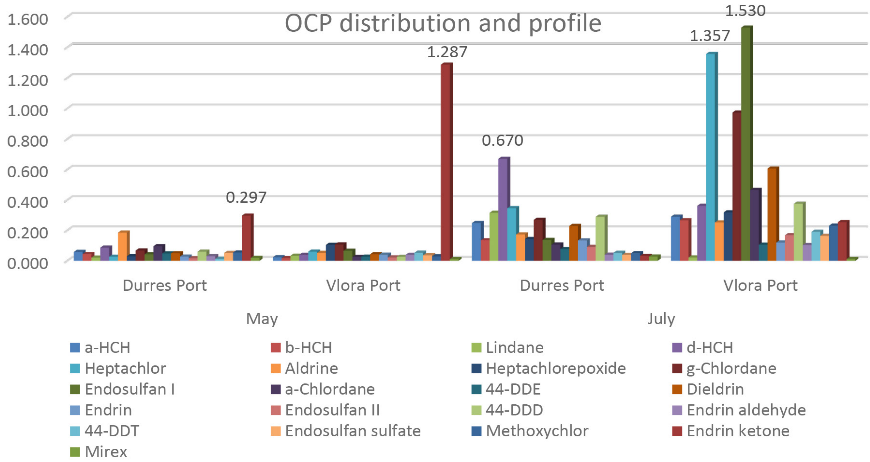


Figure 4. Distribution and profile of pesticides (ng/l) in water samples of Durres and Vlora ports
Source: Own research

PCB markers were detected in all water samples analyzed from the ports of Durres and Vlora, for both periods (May and July 2022). Also for PCBs, the higher average value was found in the port of Vlora (July 2022) at 5.4 ng/l (Figure 5). Again, Vlora samples were the most polluted for both periods. Their presence can be related to the elevated industrial activity near the ports of Durres and Vlora. Punctual sources and momentum values are not excluded. Again, the total of PCBs for each station was found in higher concentrations for water samples in July 2022. Also, levels of pesticides for Vlora stations were higher than Durres, because of the geographic position of Vlora Bay (Figure 6). It favors the concentration of pollutants inside the bay. The most polluted stations for Durres were D1 in May and D1 station in July while for Vlora were V2 in May and V7 in July. D1 and V2 stations are located outside the port areas while D11 and V7 stations are inside the ports of Durres and Vlora. Terrestrial sources and industrial activity are the main reasons for PAC's presence. The distribution and profile of PCB markers (Figure 7) were found to be different for the ports of Durres and Vlora and for both periods (May and July 2022). Their presence is based mainly on the individual concentration of PCB congeners. PCB 52 was found higher for both ports in May and PCB118 was found higher in July. This fact could be connected with different pollution origins of PCBs in these areas of the Adriatic Sea. It's a combination of atmospheric deposition of volatile congeners (PCB 28 and PCB 52) and terrestrial sources of heavy PCBs (PCB

118 – PCB 180) that are the main factors. Water currents and momentum values can influence their profile. PCB levels in water samples of Durres and Vlora ports were higher/comparable with the reported data in previous studies on the Adriatic Sea, Albanian part (Murtaj et al., 2014; Como et al., 2013, Nuro et al., 2017).

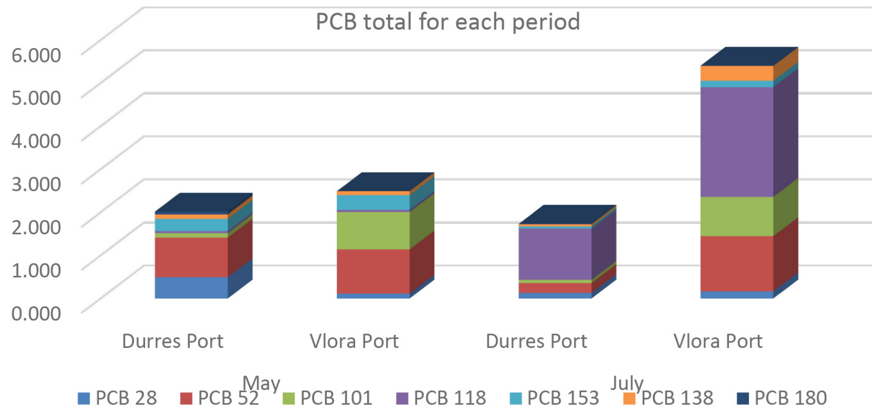


Figure 5. Total of PCB markers (ng/l) for each period of sampling (May and July 2022) in Durres and Vlora ports

Source: Own research

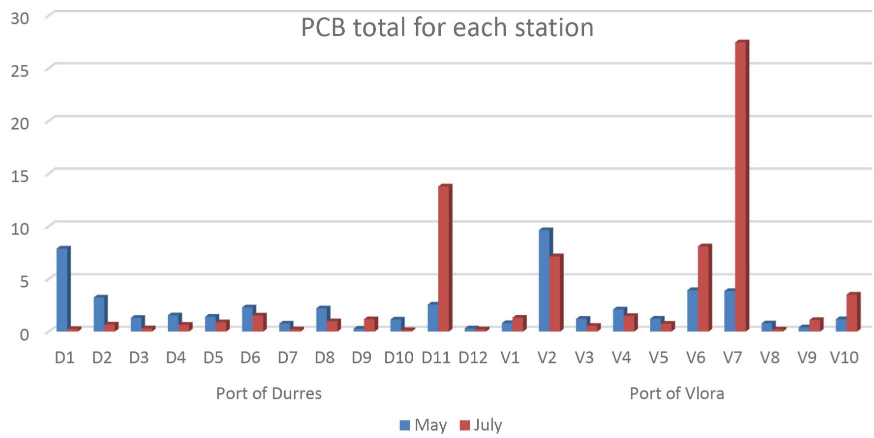


Figure 6. PCB markers (ng/l) for each station of Durres and Vlora ports

Source: Own research

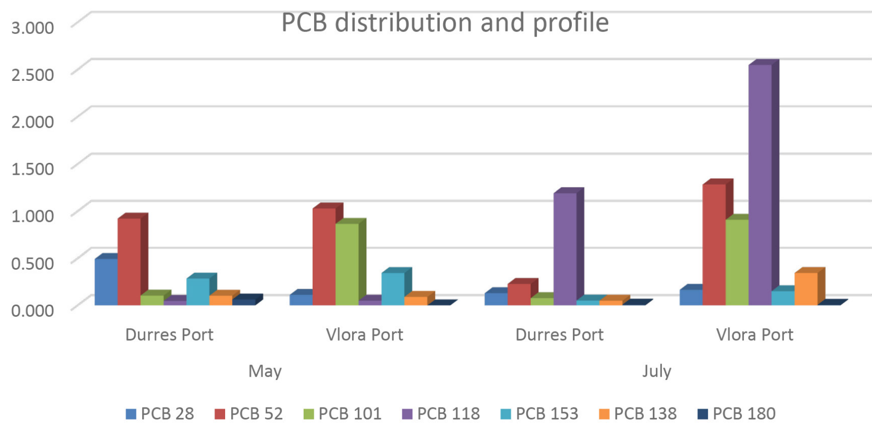


Figure 7. Distribution and profile of PCBs (ng/l) in water samples of Durres and Vlora ports

Source: Own research

Polycyclic aromatic hydrocarbons were detected in all water samples analyzed from the ports of Durres and Vlora, for both periods (May and July 2022). The higher average value of PAHs was found in the port of Durres (1.2 ug/l) in May and Vlora port in July 2022 (5.5 ug/l). PAH levels in water samples found in July were higher in July. Their presence could be because of elevated ship transport in this period 2-3 times higher than normal. Automobilist transport and any possible accident spillage could be another source of PAH pollution in marine water samples of Durres and Vlora ports. The total of PAHs for each station was higher for water samples of July 2022 (Figure 8). The presence of PAHs is mainly because of the intense ship and automobilist transport in this period. Momentum values are not excluded. Again, levels of PAHs for Vlora stations were higher than Durres because Vlora Bay favors the concentration of pollutants inside the bay as well as in Vlora port. The most polluted stations for Durres were D8 in May and D3 station in July while for Vlora were V8 in May and V4 in July (Figure 9). All these stations are inside the ports of Durres and Vlora. Ship transport and semi-closed areas of ports influence their concentration. Distribution and profile of PAHs (Figure 10) were found to be different for the ports of Durres and Vlora in May but it was almost the same for both ports in July. Their presence is based mainly on the individual concentration of some PAHs. Fluorene and Acenaftilene were found higher in May while Benzo[k]fluoranthrene and Anthracene were at a higher level in July respectively for Durres and Vlora samples. This fact could be connected mainly with the momentum values of these pollutants in water samples. PAH levels in seawater samples of Albanian ports were in the same range/higher than the reported levels for other stations of the Adriatic Sea, Albania (Magi et al., 2002; Marini & Frapiccini, 2013; Mandić & Vrančić, 2017). The concentration of PAH individuals (Anthracene) was higher than the permitted level according to Albanian and EU norms (Directive 2008/105/EC).

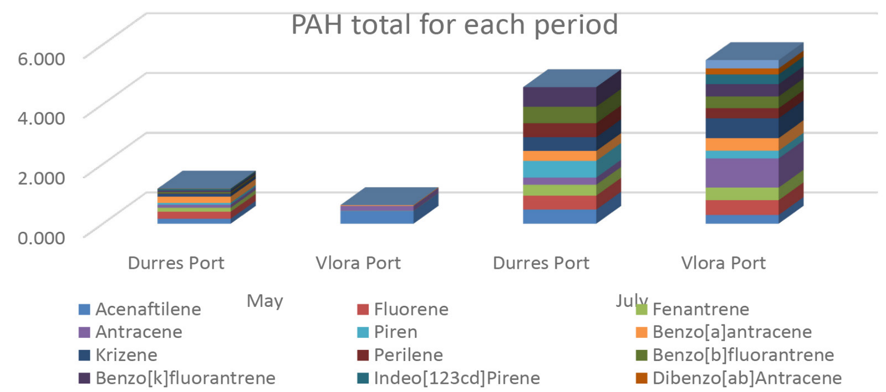


Figure 8. Total of PAHs in water samples of Durres and Vlora ports for both periods (May and July 2022)

Source: Own research

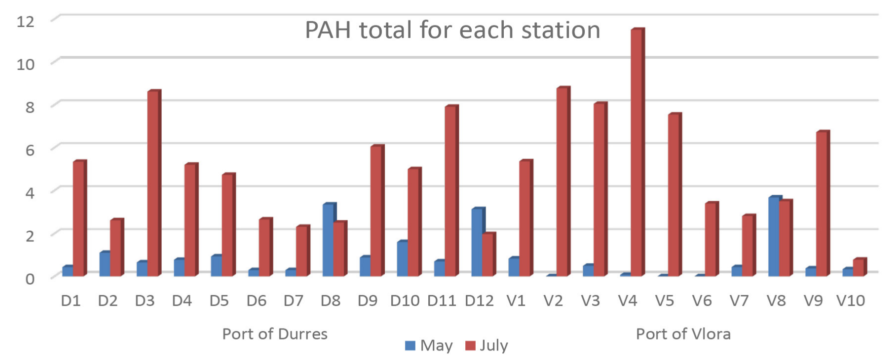


Figure 9. Total of PAH for each station of Durres and Vlora ports

Source: Own research

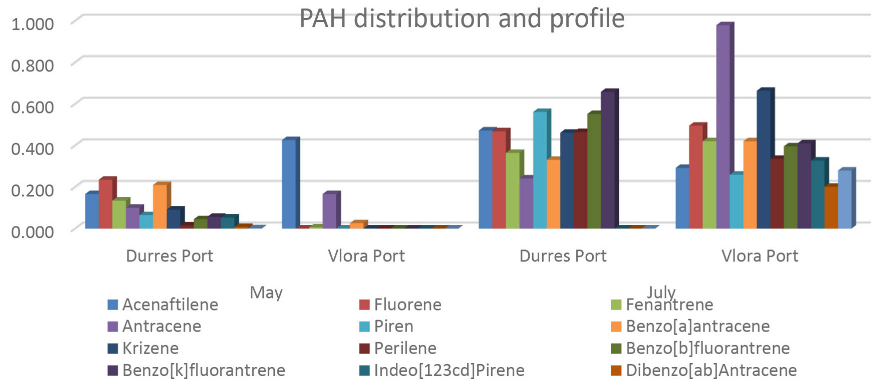


Figure 10. Distribution and profile of PAHs in water samples of Durres and Vlora ports
Source: Own research

BTEX was detected in all water samples analyzed from the ports of Durres and Vlora, for both periods (May and July 2022). The higher average value of BTEX was found for Durres port (July 2022) with 2.7 ug/l. Durres samples were most polluted for both periods. The presence of BTEX could be because of elevated ship transport, automobilist transport and any possible accident spillage of hydrocarbons near the areas of Durres and Vlora ports. Momentum values of BTEX are not excluded. The total BTEX for each station was higher for water samples in July 2022 (Figure 11). The presence of PAHs is mainly because of the intense ship and automobilists transport in this period. Momentum values are not excluded. BTEX levels in Durres stations were higher than in Vlora because of the elevated ship transport in the port of Durres. The most polluted stations for Durres were D9 in May and D3 station in July while for Vlora were V8 in May and V4 in July (Figure 12). All these stations are inside the ports of Durres and Vlora. Ship transport and semi-closed areas of ports influence their concentration. The distribution and profile of BTEX (Figure 13) were found to be different for the ports of Durres and Vlora and for both periods (May and July 2022). Their presence is based mainly on the individual concentration of volatile hydrocarbons. In the May sampling period: Benzene was found higher in the port of Durres while Ethylbenzene was in the port of Vlora. In July: Toluene was higher in Durres while Benzene in Vlora samples. BTEX origin could be the same (ship transport, automobile transport, hydrocarbon spillage, etc.) but momentum values of these pollutants influence their level in water samples. Benzene concentrations were found in higher/comparable concentrations than reported levels for other stations of the Adriatic Sea, Albania (Nuro et al., 2014). Benzene level was higher than the permitted level according to Albanian and EU norms (Directive 2008/105/EC).

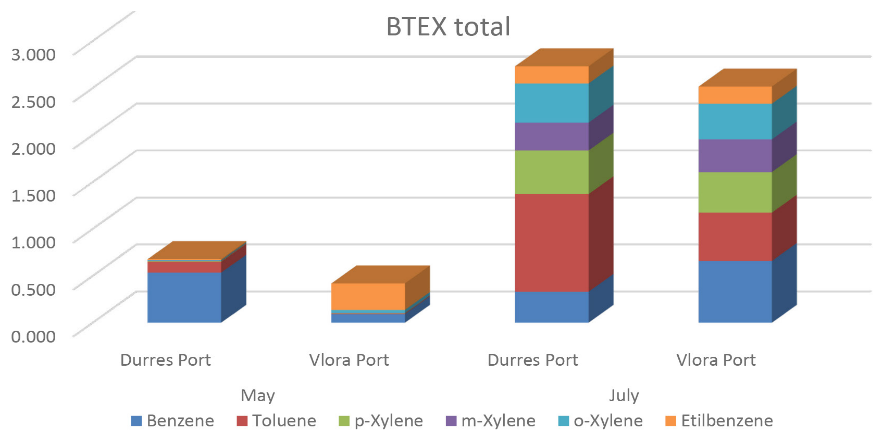


Figure 11. Total of BTEX in water samples of Durres and Vlora ports
Source: Own research

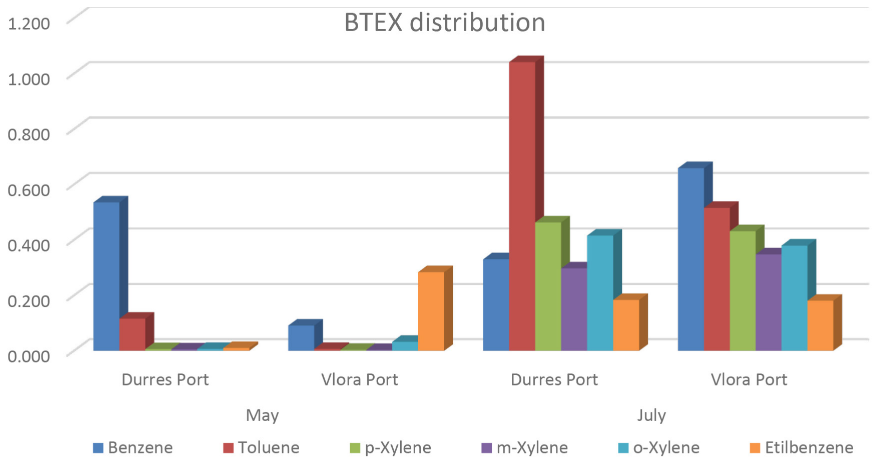


Figure 12. Total of PAHs in water samples of Durres and Vlora ports
Source: Own research

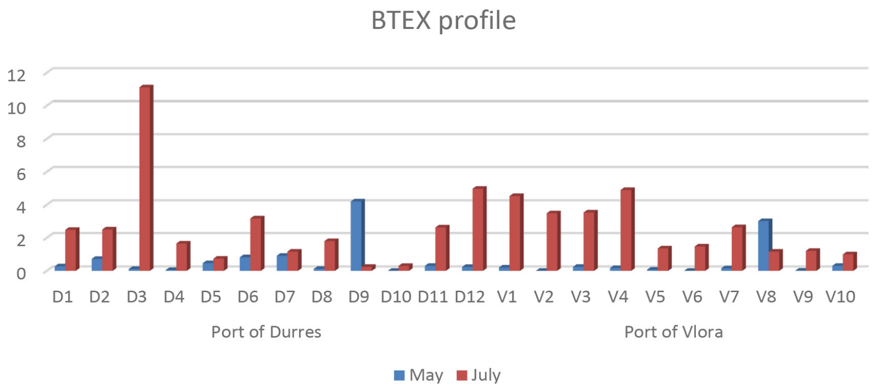


Figure 13. Profile of BTEX in water samples of Durres and Vlora ports
Source: Own research

4. CONCLUSION

Organic pollutants (Pesticides, PCBs, PAHs and BTEX) were found in all water samples of Durres and Vlora ports for both periods, May and July 2022. The higher levels for all pollutants were found in July because of the intense activity in this period (2-3 times higher than normal). Organochlorine pollutants (OCPs and PCBs) were found at a higher level for the samples of the Vlora port. It was noted the presence of degradation products of pesticides at higher levels compared to their active products. This fact is connected with the previous use of pesticides in Albania and their degradation process. PCBs volatile were found at high levels for all seawater samples. Their presence could be because of their atmospheric deposition. In some water samples, heavy PCBs were detected. This could be connected with terrestrial. Momentum values of them are not excluded. The presence of PAHs and BTEX could be because of elevated ship transport, automobilistic transport, and any possible accident spillage of hydrocarbons near the areas of Durres and Vlora ports. Concentrations of some individuals (Endrin ketone, Heptachlor, PCB52, PCB 118, Acenaftilene and Benzene) were found relatively in higher concentrations than others. Punctual sources and water currents can affect this. Generally, priority substance concentrations in the water of Durres and Vlora ports were lower than permitted levels for surface waters according to EU Directive 2013/39 and Albanian norms. Exclude was for some individual pollutants (Acenaftilene and Benzene were found at a higher level than the permitted level). Monitoring of organic pollutants in the water of Albanian ports and the Adriatic Sea should be continuous because of their presence.

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