LEVELS OF PRIORITY SUBSTANCES IN LEZHE – SHENGJIN, ADRIATIC SEA COASTLINE OF ALBANIA

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ABSTRACT

The Lezhe-Shengjin coastline is characterized by a large variety of water bodies that have a direct impact on the quality of the water in the Adriatic Sea. The Mat and Drin rivers flow into this area, which also contains the Kune and Vain lagoon complex. This region could be affected by urban pollution from the cities of Lezhe and Shengjin, as well as by agricultural and industrial activities, and the influence of the port of Shengjin. These factors motivated us to conduct this preliminary study to determine the concentrations of priority substances in water and sediment samples from the Lezhe-Shengjin coastline. The analyzed compounds were organochlorine pesticides (OCPs), polychlorinated biphenyls (PCBs), and polycyclic aromatic hydrocarbons (PAHs), which are of great importance due to their potential toxicity to aquatic organisms and human health. Although the production and use of these substances have been banned for decades, their presence continues to be reported due to their high persistence in the environment. Water and sediment samples were collected at 12 stations, ranging from Shengjin port to Vain lagoon, in May 2023. Analyses of organochlorine compounds and polycyclic aromatic hydrocarbons were performed using a gas chromatograph (Varian 450 GC) equipped with ECD and FID detectors. Organochlorine pesticides, their degradation products, PCB markers, and PAHs were detected in almost all samples. Their presence may be related to their previous uses due to anthropogenic factors (urban pollution, agricultural and industrial activities), ship transport, etc. Water currents and new inputs from Albanian rivers can be considered important factors impacting their levels and profiles. The levels of these substances in water and sediment samples from the Lezhe-Shengjin coastline were comparable to reported levels from other stations in the Adriatic Sea. However, authorities should increase their attention to continuous monitoring of the levels of these substances in this area.

Keywords: Adriatic Sea, Organochlorine pesticides, PCBs, PAH, GC/ECD/FID

1. INTRODUCTION

Albania is bordered by the Ionian and Adriatic seas and has a coastline of 316 km. About 260 kilometers to the Adriatic Sea, starting from Vlora Bay in the south to the Buna River mouth in the north. The entire coastline of our country has great diversity, including sandy and rocky beaches, lagoons, river estuaries, ports, etc. (Corsi *et al*., 2010; Borshi *et al*., 2016). In this study, the coastline from Lezhe to Shengjin, which lies along the Adriatic Sea, is considered. Lezhe city represents a very important and unique ecological system, where the mountain, the field, the forest, the archaeological and historical monuments, the lagoon, and the sea constitute a prominent community. The Drin River flows into Lezha city, which together with the Mat River forms the Kune-Vain lagoon complex and the Patoku Lagoon. Nearby lie the fertile fields of Zadrima, Torovica, and Mati. Shengjin is located 7 kilometers north of Lezha city. Shengjin offers a pleasant panorama, characterized by a clean sea surrounded by mountains and a beautiful landscape. In recent years, the city has evolved, especially through the development of the construction industry and tourism. Near Shengjin is a port that has been operational since 2009, providing a connection between Shengjin and the city of Bari, Italy. The port is also important for the fishing boats that operate in this area. The port of Shengjin is ranked as the third largest in the country and is the only one in the north. The harbor is protected by long rock walls that extend into the sea (Nuro *et al*., 2014).

Anthropogenic sources of water pollution in the Adriatic area are classified into two main groups: point and non-point sources. Point sources include discharges of urban liquid waste (sewage) from cities and ships, waste from industrial activities and mechanical businesses, import/export activities and their storage, rinsing waters from solid waste disposal sites, etc. (Konstantinou *et al*., 2006; Borshi *et al*., 2016; Nuro *et al*., 2018). Non-point sources include agricultural land drainage water discharges, polluted rain, sewage pipeline leaks, new inputs from rivers, etc. According to the consequences they cause, polluting substances in the marine environment are divided into several groups:substances highly toxic to humans or aquatic flora and fauna (Pb, Hg, Cd, As, cyanides, pesticides, etc.); hazardous substances for humans, flora, and fauna, which cause chronic damage (PAHs, chlorophenols, and many medicinal drugs); substances that cause an increase in BOD/COD (sewage discharges, liquid waste from the food industry and livestock farms, etc.); substances that can cause an increase in the rate of eutrophication of waters (nitrates and phosphates);substances that damage the appearance of waters (oil, detergents, sludge, suspended

particles, plastics, etc.); microorganisms that are pathogenic for humans, e.g., Salmonella, Cholera vibrio, etc. (Froebner *et al*., 2018; Mohamed *et al*., 2011).

Organic pollutants come mainly from urban wastewater, industrial/mechanical activities, ship and automobile transport, as well as agricultural activities. Urban wastewater is the largest source of organic materials that are discharged first into natural waters and then into the sea (Nuro *et al*., 2014). Organic pollutants in marine ecosystems are found in water, sediment, and biota. Their concentrations in water are very small due to sedimentation processes; therefore, their presence is much higher in sediment. These substances in sediment have a longer degradation period. Additionally, organic pollutants have been reported at higher levels in biota (algae, fish, mussels, etc.). Their concentration increases due to the processes of bioaccumulation, bioconcentration, and biomagnification. The organic pollutants analyzed in this study were organochlorine pesticides (OCPs), polychlorinated biphenyls (PCBs), and polycyclic aromatic hydrocarbons (PAHs). Organochlorine pesticides were used intensively in our country for more than 50 years (1945–1992) for agricultural purposes (EU, 2007). PCBs are not used in our country, but their presence has been reported in many studies, mainly due to atmospheric deposits. The presence of PAHs comes mainly from the transportation, extraction, and processing of oil, the massive burning of forests, urban waste, etc. PAHs also have a natural background. OCPs, PCBs, and PAHs are classified as priority substances based on the EU Directive of 2008 in the field of water policy because of their persistence and toxicity (Nuro *et al*., 2018; Corsi *et al*., 2010; Borshi *et al*., 2018).

2. MATERIALS AND METHODS

2.1. Study station and sampling techniques

Water and sediment samples were collected at 12 stations, ranging from Shengjin port to Vain beach, in May 2023. Water samples were taken in Teflon containers, with 2.5 liters collected from each station. The samples were collected according to the ISO 5667-14:2016 methodology. They were transported and kept at +4°C before analytical analysis in the laboratory. Sediment samples were taken at the same stations using a Van Veen grab. Sediment samples were air-dried and sieved before analysis, with a fraction of 63 micrometers considered for the analyses of organic pollutants. The sampling stations for the Lezhe-Shengjin coastline are shown in Figure 1.

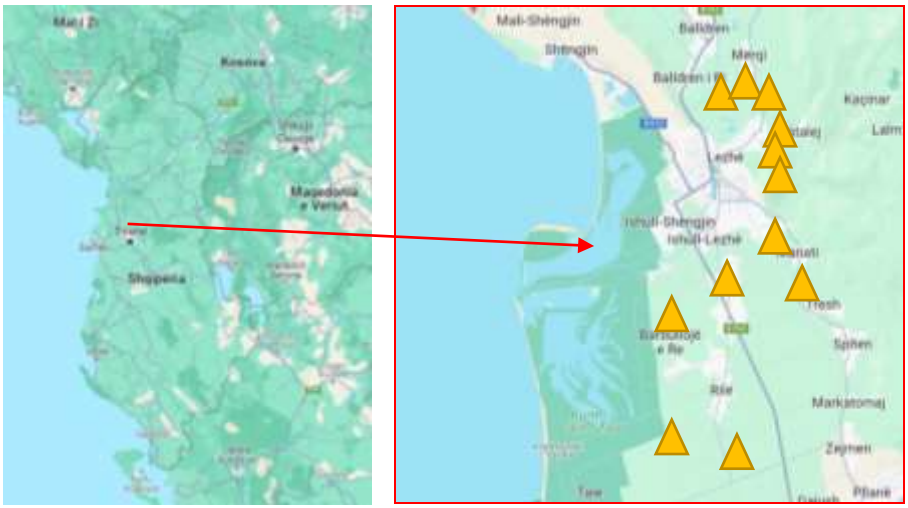

Fig.1: Sampling station in Lezhe - Shengjin coastline, Central Adriatic Sea, May 2023.

2.2. Treatment of water samples for pesticide and PCB analyses

Liquid-liquid extraction was used for the extraction of organochlorine pesticides and polychlorinated biphenyls from marine water samples. One liter of water and 40 ml of n-hexane as the extracting solvent were added to a separatory funnel. After extraction, the organic phase was dried with 5 g of anhydrous Na2SO4 to remove water. A Florisil column was used for sample clean-up. Twenty milliliters of n-hexane/dichloromethane (4/1) was used for elution. After concentrating the samples to 1 ml of hexane, they were injected into a GC/ECD (Lekkas *et al*., 2004; Vryzas *et al*., 2009; Nuro *et al*., 2012).

2.3. Treatment of sediment samples for analysis of chlorinated pollutants

For the determination of organochlorine pollutants, 5 g of sediment samples (fraction < 63 microns) were taken, dried, pre-sieved, and placed in a 100 ml beaker. Fifty milliliters of n-hexane/dichloromethane (3:1) were added as the extraction solvent. Extraction was carried out in an ultrasonic bath for 60 minutes at 30 °C. After separation of the organic phase, 2 g of anhydrous sodium sulfate was added to remove traces of water. The solvent was evaporated using a Kuderna-Danish apparatus to 10 ml. Metallic mercury was added to the test tube until the complete removal of sulfur compounds, which are typically found in sediment samples and hinder gas chromatographic analysis. The extract was carefully transferred to an open glass column (10 cm x 0.8 cm i.d.) packed with Florisil. Elution was performed with 20 ml of n-hexane/dichloromethane (4:1) and collected in a

Kuderna-Danish flask, where it was evaporated to near dryness (2 ml). The concentrated extract was then injected into a gas chromatograph equipped with an ECD detector (Murtaj *et al*., 2013; Nuro *et al*., 2018).

2.4. Gas chromatography analysis of pesticides and PCBs

Organochlorine pesticides and PCBs were analyzed simultaneously using capillary columns of 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 an ECD detector. Helium was used as a carrier gas (1 ml/min), while nitrogen was used as a 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); Chlordanes (alpha and gamma isomers); Aldrin's (Aldrine, Dieldrine, and Endrin) and Endosulfanes (Endosulfan alpha, Endosulfan beta and Endosulfan sulfat). The analysis of PCBs was based on the determination of the seven PCB markers (IUPAC Nos. 28, 52, 101, 118, 138, 153, and 180). The quantification of OCPs and PCBs was based on an external standard method. Standard mixtures of organochlorine pesticides (21 individuals) and 7 PCB markers with concentrations of 1ppb, 2 ppb, 5ppb, 10ppb, 25ppb and 100ppb were used to built calibration curves and method evaluation. Recovery for OCPs varied from 81.5% (Dieldrin) to 114% (DDT), while for PCB recovery varied from 92% (PCB 180) to 110% (PCB 118). The LOD for individual pesticides varied from 0.03 ppb (DDT) to 0.07 ppb (Heptachlor) while for PCBs the values varied from 0.04 - 0.05 ppb (Lekkas *et al*., 2004; Kostantinou *et al*, 2006; Vryzas *et al*., 2009; Nuro *et al*., 2012; Borshi *et al*, 2016).

2.5. Treatment of water samples for PAH analyses

Two-step liquid-liquid extraction (LLE) was used for extracting PAHs from marine water samples. One liter of water with first 40 ml of dichloromethane (first step LLE) and then 40 ml of 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 injected in GC/FID for qualification or quantification of PAHs (Lekkas *et al*, 2004; Wang *et al*, 2009; Nuro *et al*, 2014).

2.6. Treatment of sediment samples for PAH analyzes

Only the 63-micron fractions were taken into analysis. For the determination of chlorine-organic pollutants, 5 g of sediment was taken into a 100 ml Erlenmayer, where 50 ml of n-hexane was added as an extraction solvent. Their extraction was carried out in an ultrasonic bath for 30 minutes

at 30 °C. After separation of the organic phase, 2 g of anhydrous sodium sulfate was added to remove traces of water. The solvent was evaporated using Kuderna-Danish to 2 ml. The extract was injected into the gas chromatograph equipped with an FID detector (Akkanen *et al*, 2005; Nuro *et al*, 2018).

2.7. 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. A 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 the EPA 525 Method. Helium was used as a carrier gas at 1 ml/min. The 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 at 30 ml/min and 300 ml/min, respectively. The EPA 525 Standard Mixture was used for qualitative and quantitative analyses 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. The quantification of PAHs was based on an external standard method. EPA 525 standard mixture was used to buitt calibration curves and method evaluation. Recovery for PAH varied from 79.2% (Dibenzo [g,h,i] anthracene) to 118.5%. (Anthracene). The LOD for individual PAH varied from 0.03 ppm (Anthracene) to 0.06 ppm (Benzo [k] fluoranthrene) (Lekkas *et al*., 2004; Wang *et al*., 2009; Nuro *et al*., 2014; Stogiannidis *and* Laane, 2015).

3. RESULTS AND DISCUSSIONS

In this study, levels of some priority substances (organochlorine pesticides, PCBs, and PAHs) were evaluated in seawater samples from the Lezhe-Shengjin coastline located in the Adriatic Sea, Albanian coastline. Samples were taken in May 2023. Organochlorine pesticides, their degradation products, and PCB markers were analyzed using GC/ECD techniques. Polycyclic aromatic were analyzed using GC/FID techniques. OCPs, PCBs, and PAHs are classified as priority substances because of their stability and toxicity. Analyzes of priority substances in seawater is important not only for the environment but also for organisms (including humans) because of their persistence and toxicity.

Figure 2 shows the total amount of organochlorine pesticides in water samples (ppb - ug/l) and sediment samples (ppb - ug/kg) for the Lezhe-Shengjin coastline stations. The average level of contamination for water

samples was 1.45 ppb, while for sediment samples it was 3.84 ppb. The highest levels of pesticides in water samples were found in Kune 1 and Vain 1 samples, respectively, with 4.62 and 3.76 ppb. The high levels in these stations are mainly related to the stronger impacts that come as a result of the agricultural activity near Kune-Vaini lagoons. New arrivals of pesticides by Drin and Mat rivers could be another important factor that influence their presence. Also, this fact was reflected in the sediment samples for Kune Lagoon, which were the most polluted stations, where pesticide levels ranged from 5.08 to 9.12 ppb. High levels of OCPs were found in Shengjin samples. This should be mainly related by water currents of the Adriatic Sea, which are oriented from the north (Corsi et al 2012; Nuro et al 2014).

The profile of organochlorine pesticides is given in Figure 3. The profile of pesticides in water was: Dieldrin > a-Chlordane > Endrin > Endosulfan I > Methoxychlor. The presence of these pesticides in water at higher levels is connected mainly with their recent use and/or new arrivals from Drin and Mat rivers. OCP profile for sediment samples was different. It was: Aldrin > Endosulfane sulfate > DDE > Methoxychlor > Endrin ketone > a-Chlordane > Heptachlor epoxide. It should be said that the profile of pesticides in water and sediment samples is influenced by the individual levels of each pollutant at specific stations. Degradation processes of OCPs, their physical-chemical properties, new arrivals and water currents could be main factors. It was noted that at higher levels were found the degradation products (Heptachlor epoxide, DDE, Endrin ketone, etc), not active substances that were applied as pesticides.

Figure 4 shows the classes of pesticides for water and sediment samples of Lezhe-Shengjin coastline. For water samples, their profile was as follows: Aldrines > DDTs > HCHs > Endosulfanes > Chlordanes > Heptachlores; while for sediment samples, their profile was: Aldrines > DDTs > Endosulfanes > Chlordanes > Heptachlores > HCHs. These profiles could be mainly related to the physico-chemical properties of pesticides, such as stability, solubility, polarity, etc. The time and amount of their use are also factors that influence their profile.

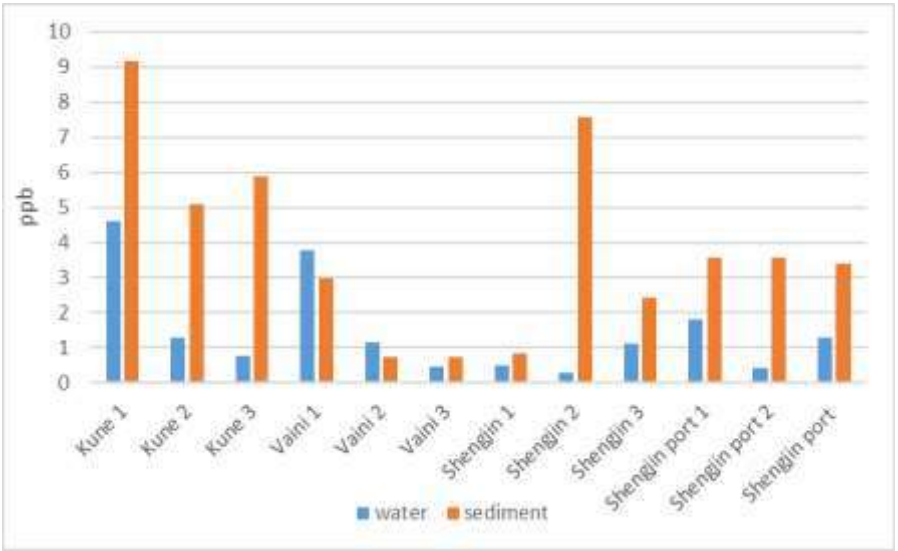

Fig. 2: Total of Organochlorine Pesticides on the Lezhe-Shengjin Coastline, Adriatic Sea, May 2023.

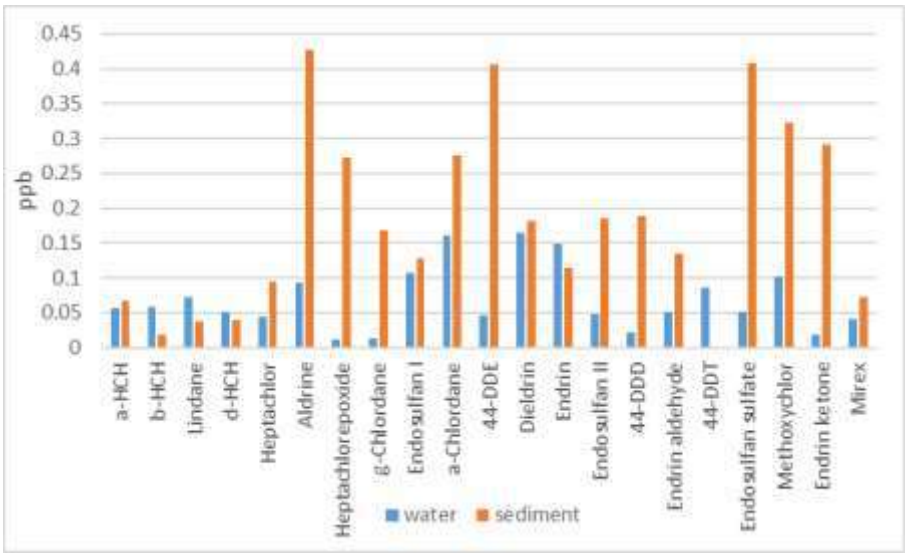

Fig.3: Profile of Organochlorine Pesticides on the Lezhe-Shengjin Coastline, Adriatic Sea, May 2023.

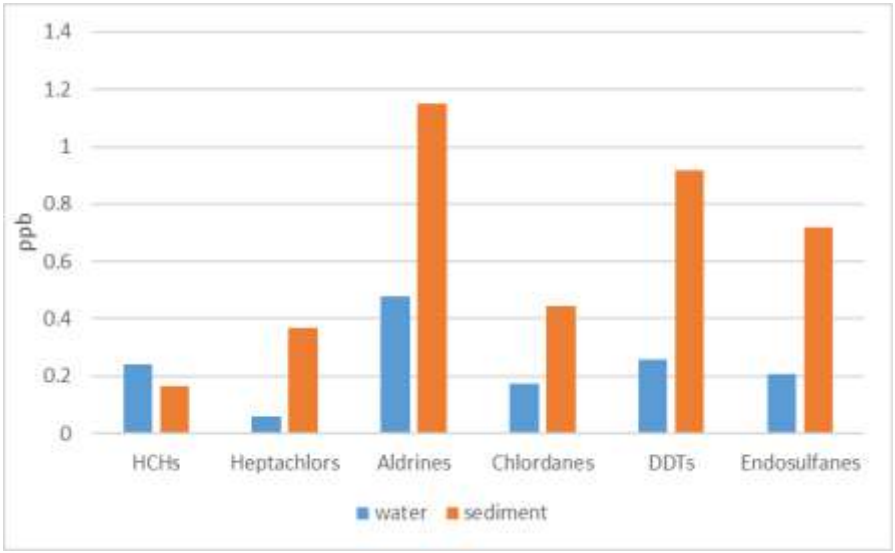

Fig. 4: Classes of pesticides on the Lezhe-Shengjin coastline, Adriatic Sea, May 2023.

Figure 5 shows the total PCB in water samples (ppb - ug/l) and sediment samples (ppb - ug/kg) for the stations along the Lezhe-Shengjin coastline in May 2023. The average level of PCB markers for the water samples was 3.08 ppb, while for the sediment samples it was 7.58 ppb. The highest level of PCBs in water samples was found in Shengjin samples. Even in some stations in Kune 1 and Vain 3, high levels of them were detected. Their presence in water samples should be mainly due to atmospheric depositions. Also, PCBs were found at a higher level in the sediment samples at both Shengjin port stations, Shengjin 3 station, and Kune samples. Their presence must be mainly a result of punctual discharges (oil discharges by mechanical buisnesses, ship remont/defects, etc.) near the study stations. Intense atmospheric depositions, new arrivals from the rivers and the influence of water currents of Adriatic Sea can influence on the PCB levels in the sediment samples.

The profile of PCBs is given in Figure 6. Their profile for the water samples was built almost by the presence of PCB 28 and PCB 52, which are representatives of volatile PCBs. This fact once again proves the presence of PCBs as a result of atmospheric deposits (Nuro *et al* 2014). In the sediment samples, in addition to volatile PCBs, the presence of heavy PCBs, including PCB 180, was detected. This is related to their point sources near these stations. The impact of ship and automobilist transport and other industrial activities (mainly mechanical buisnesses) that perform near these stations are the main factors. Other factors could be water currents and the physical

properties of each of the analyzed compounds (Corsi *et al* 2010, Nuro *et al* 2014).

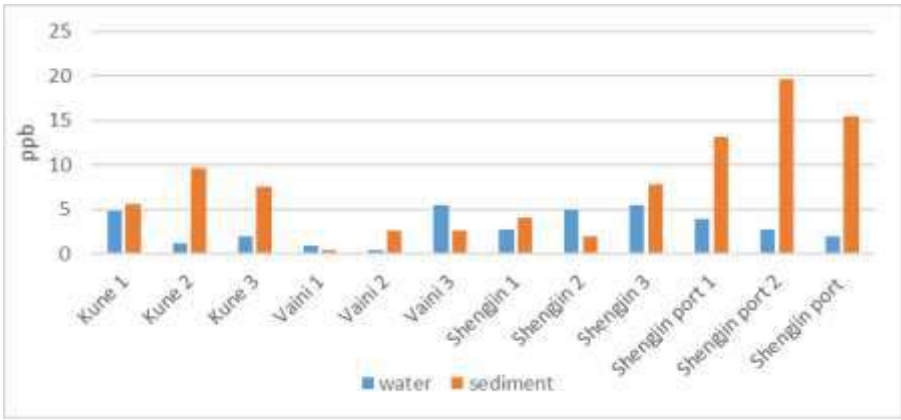

Fig. 5. Total of PCBs in the Lezhe-Shengjin coastline, Adriatic Sea, May 2023.

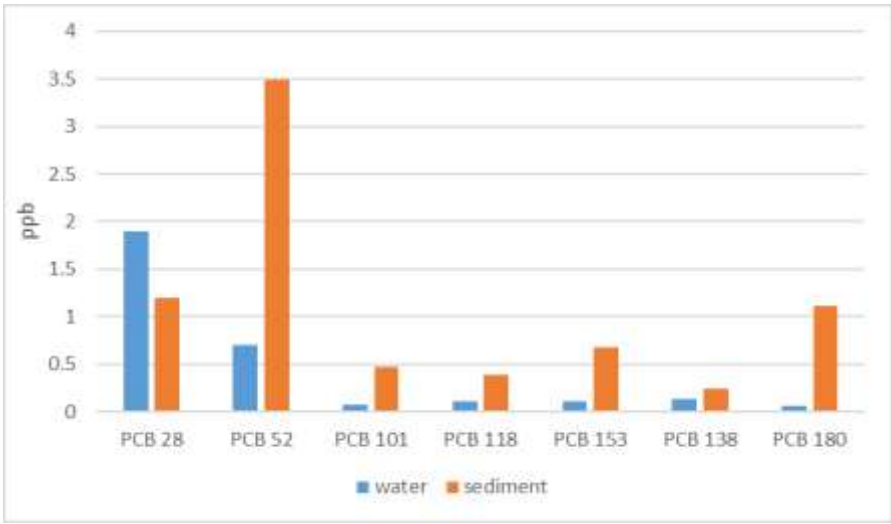

Fig. 6. Profile of PCBs on the Lezhe-Shengjin Coastline, Adriatic Sea, May 2023

Figure 7 shows the total PAHs in water samples (ppm - mg/l) and sediment samples (ppm - mg/kg) from Kune Lagoon, Lezhe to Shengjin port. The average pollution level for water samples was 2.00 ppm, while for sediment samples it was 5.35 ppm. The highest level of PAH for water samples was found in Shengjin 1 port samples with more than 20 ppm. Their presence in water samples should be mainly due to marine transport or

hydrocarbon spills at these stations. PAHs were found at a high level in sediment samples at Shengjin port stations (Shengjin port 2 and Shengjin port 3). Their presence must be mainly a result of marine transport and punctual discharges of these pollutants near the study stations.

The profile of PAHs is given in Figure 8. Their profile for water samples was constructed as follows: Benzo[a]anthracene > Phenanthrene > Indeo[123cd] pyrene. This profile is mainly related to hydrocarbons that are obtained at high temperatures (pyrogenic), which means that their origin must be a consequence of transport or other processes at high temperatures. The profile of PAHs in the sediment samples was: Fluorene > Phenanthrene > Benzo[a]anthracene > Pyrene > Chrysene > Perylene > Acenftilene. These hydrocarbons are mostly of natural origin. The influence of transport and their physico-chemical properties is not excluded, mainly the polarity of these molecules.

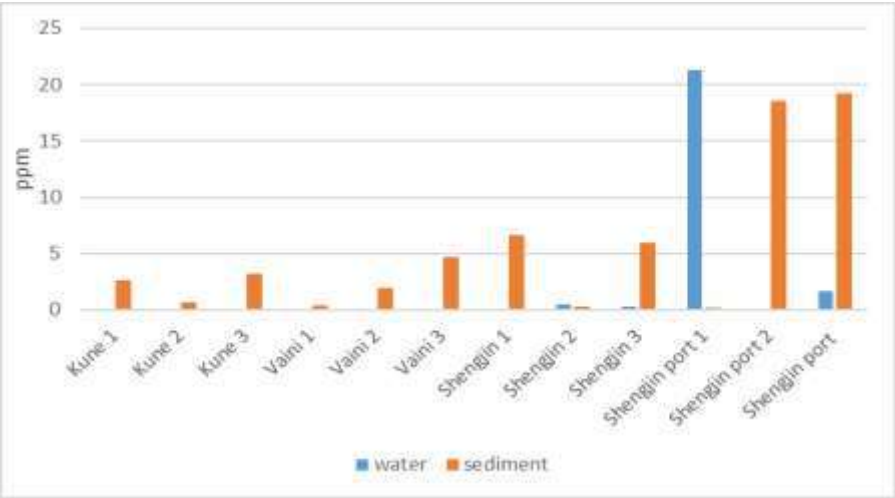

Fig. 7: Total of PAHs in the Lezhe-Shengjin coastline, Adriatic Sea, May 2023.

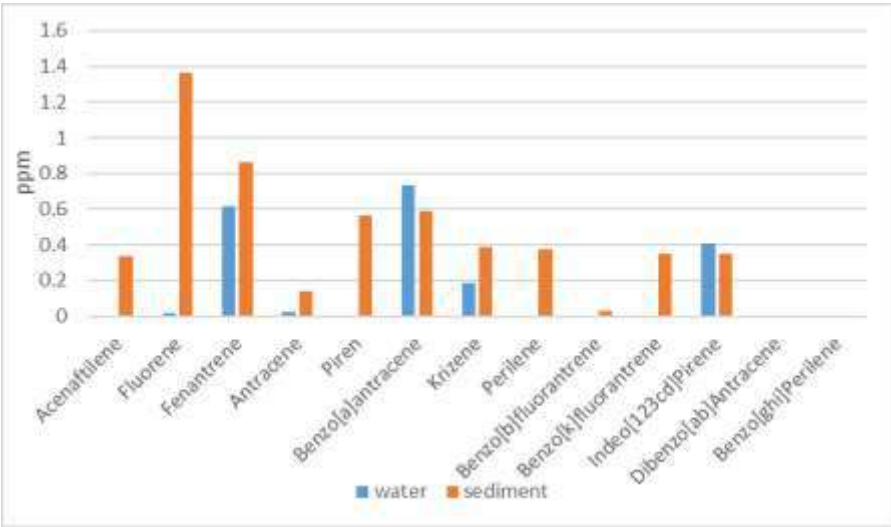

Fig. 8: Profile of PAHs in the Lezhe-Shengjin coastline, Adriatic Sea, May 2023.

4. CONCLUSIONS

In this study, levels of organochlorine pesticides, PCBs, and PAHs were evaluated in seawater samples from the Lezhe–Shengjin coastline located in the Adriatic Sea. These compounds are part of the priority substances list because of their elevated stability and toxicity. The Lezhe-Shengjin coastline is characterized by a large variety of water bodies, including the Drin and Mat rivermouths, the complex of Kune and Vain lagoons, the beaches of Lezhe and Shengjin, and the port of Shengjin, which have a direct impact on the quality of the Adriatic Sea's water.

Organochlorine pesticides were detected in all stations analyzed. The average level of pollution was about three times higher in the sediment samples. This should be related to the previous uses of these compounds. The highest level of pesticides in water samples was found in the samples taken inside the lagoons of Kune and Vain because of the strong impacts that come as a result of the agricultural activities near these lagoons. New arrivals from Drin River and Mat River can concentrate faster in the sediments. Profile of pesticides was influenced by the individual levels of analyzed pollutants. It was noted the presence of degradation products rather than the active substances because of their previous use. The highest level of PCBs in water samples was found in Shengjin samples and in some stations in Kune and Vain lagoons. Their presence in water samples should be mainly due to atmospheric depositions. Also, PCBs were found at higher level in sediment samples. The profile of PCBs in water samples was built by volatile

congeniers while in the sediment samples, heavy PCBs were detected. PAHs were found at higher level in the Shengjin port stations (seawater and sediment samples) due to marine transport and/or hydrocarbon spillages near these stations. Their presence must be mainly a result of marine transport and punctual discharges of these pollutants near the study stations.

OCP, PCB and PAH pollutants were found in lower concentrations than the levels recommended by the Albanian norms and the Directive 2008/105/EC for surface waters. The levels of OCPs, PCBs and PAHs in the water and sediment samples of Lezhe – Shengjin coastline were similar to the levels reported in previous works for the water of the Adriatic Sea (Nuro et al 2014). The presence of priority substances in the water and sediment samples of the Lezhe-Shengjin area should be an incentive for the authorities to have continuous control of their levels in the Adriatic Sea.

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