DETERMINATION OF ORGANIC POLLUTANT LEVELS IN THE KUNE-VAINI LAGOON COMPLEX

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Abstract

This paper presents the levels of organochlorine pesticides, their residues, polychlorinated biphenyls (PCBs) and polycyclic aromatic hydrocarbons (PAHs) in water samples and sediments from the Kune-Vaini lagoon complex, located near Lezha. This area is known for the great variety of water bodies where can mention the rivers Drin and Mat which have influenced the formation of these lagoons as well as the Adriatic Sea with which they communicate. Water and sediment samples were taken at the same stations inside and outside lagoons in the Adriatic Sea. The samples were taken in March 2023. The liquid-liquid technique was used for the extraction of organic pollutants from the water samples, while ultrasonic extraction was used for their isolation from the sediment samples. Their clean-up procedures were carried out in SPE columns. The qualitative and quantitative analysis of organic pollutants was carried out in the Varian 450 model gas chromatograph, equipped with a μECD and FID detector. Rtx-5 capillary column were used for the separation of organochlorine pollutants and VF-1ms column for PAH hydrocarbon isolation. Organic pollutants were found in all stations of Kune-Vain lagoons. The highest levels of contamination were found in sediments compared to the water samples. The different profile of analyzed pollutants in water and sediments is noticeable due to the new arrivals from agricultural and industrial activities not only from the areas near the lagoons but also from the Drin and Mat rivers. Analysis/monitoring of organic pollutants in water and sediment samples of this important area of the central Adriatic should be continuous.

Key words: *Kune-Vaini lagoons, organochlorine pesticides; PCB; PAH; water analyzes; GC/ECD/FID.*

Përmbledhje

Ky punim paraqet nivelet e pesticideve klororganike, mbetjeve të tyre, poliklorbifenilet (PCB) dhe hidrokarburet policiklike aromatike (PAH) në mostra uji dhe sedimente nga kompleksi i lagunave të Kune-Vainit, Lezhë. Kjo zonë është e njohur për larminë e madhe të trupave ujore ku përmendim lumenjtë Drin dhe Mat të cilët kanë ndikuar në formimin e këtyre lagunave si dhe Detin Adriatik me të cilin ato komunikojnë. Mostrat e ujit dhe sedimenteve janë marrë në të njëjtat stacione në brendësi dhe jashtë lagunave në Detin Adriatik. Mostrat u morrën në Mars 2023. Për ekstraktimin e ndotësve organikë nga mostrat e ujit u përdor teknika lëng-lëng ndërsa ekstraktimi me ultratinguj u përdor për izolimin e tyre nga mostrat e sedimenteve. Procedurat e pastrimit të tyre u realizuan në kollona SPE. Analiza cilësore dhe sasiore e ndotësve organikë u realizua në aparatin e gaz kromatografit model Varian 450, i pajisur me detektor μECD and FID. Për ndarjen e ndotësve klororganikë u përdor kollona kapilare Rtx-5 dhe kollona VF-1ms për ndarjen e hidrokarbureve. Ndotësit organikë u gjetën thuajse në të gjithë stacionet e lagunave të Kune-Vainit. Nivelet më të larta të ndotjes ishin në sedimente krahasuar me ato të ujit. Bie në sy profili i ndryshëm në ujë dhe sedimente kjo për shkak të prurjeve të reja që vijnë nga aktivitetet bujqësore dhe industriale jo vetëm nga zonat afër lagunave por edhe nga lumenjtë Drin dhe Mat. Analiza/monitorimi i ndotësve organikë në mostrat e ujit dhe sedimentit të kësaj zone të rëndësishme të Adriatikut qendror duhet të jetë i vazhdueshëm.

Fjalë kyçe: *Laguna e Kune-Vaini, pesticidet klororganike; PCB; PAH; analizat e ujit; GC/ECD/FID.*

Introduction

Albania is located in the Western Balkan penisula and faced by Ionian Sea and Adriatic Sea. The total coastline length of Albania is 316 km. About 260 km belong to the Adriatic Sea, starting from the Vlora Bay, in the South, to the rivermouth of Buna River, in the North Albania. The entire coastline of our country has a great diversity including sandy and rocky beaches, lagoons, river estuaries, ports, etc. In this study, the area of the Kune-Vaini lagoon complex, was consider. The complex of lagoons lies near the city of Lezha in the Central Adriatic Sea. Lezha represents an important ecological area, where the mountain, fields, rivers, lagoons and the sea constitute a prominent area. The Drin River flows into Lezha, and the Mat River in the South of the city. Both rivers form in their delta the Kune-Vain and Patoku lagoons. Near the rivers as well as near lagoons lie the fertile fields of Zadrima, Torovica and Mati

(Bregu i Matit), which are important for the country because of elevated agricultural activity. In recent years, in the Lezha area, has been an increase in industrial activities and services, especially tourism, which has a significant increase from year to year (Nuro *et al*, 2014).

Anthropogenic sources of water pollution in marine areas, including the Adriatic Sea, are classified into two main groups: punctual and non-punctual sources. Punctual sources include discharges of urban waste (sewage) from cities, villages, residential areas, buisneses and ships, industrial waste from processing (import/export) and their storage, leachate from solid waste disposal sites, etc. Non-punctual sources include new arrivals from agricultural lands (rainfall), atmospheric deposits, sewage pipelines, etc. According to the consequences they cause, polluting substances in the marine environment are divided into several groups: High toxic substances to humans or aquatic flora and fauna (Pb, Hg, Cd, As, cyanide, pesticides, etc.); Hazardous substances for humans, flora and fauna, which cause chronic damage (PAH, chlorophenols and pharmaceutical drugs); Substances that cause an increase in BOD/COD (sewage discharge, 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, particles in suspension, plastic, etc.); and microorganisms that are pathogenic to humans, eg, Salmonella, Cholera, etc. (Wang *et al*, 2009; Borshi *et al*, 2016; Murtaj *et al*, 2013)

Organic pollutants mainly come from domestic sewage (raw or treated), urban runoff, industrial (commercial) and agricultural effluents. Sewage effluent is the largest source of organic materials that are discharged first into natural waters and then into the sea. Organic pollutants in marine ecosystems are found in water, sediments and biota. Generally, their concentrations in water are very small due to their binding with particular matter and sedimentation processes, because of this, their presence in sediment is much higher. This process also can cause their presence time to time in water column. The presence of these organic pollutants have been reported at much higher levels than the aquatic environment and in biota (algae, fish, mussels, etc.). Their concentration increases due to bioaccumulation, bioconcentration and biomagnification processes. The organic pollutants considered in this study are organochlorinated pesticides (OCPs), polychlorinated biphenyls (PCBs) and polycyclic aromatic hydrocarbons (PAHs).

Organochlorine pesticides have been used intensively in our country for about 50 years (1945 - 1992) for agricultural purposes. PCBs are not used in our country, but their presence has been reported in many studies mainly due to atmospheric deposition. PAHs are substances that have a natural background, but their presence comes also from the transportation (automobile, ships, ect), extraction and processing of oil, massive burning of forests, urban waste, etc. These substances present high stability in the environment, therefore they are classified as priority substances. In addition to their high stability and their degradation products, these substances are toxic to the environment, biota and population (Akkanen *et al*, 2005; Nuro *et al*, 2018; Borshi *et al*, 2018; EU 2008).

Material dhe methods

2. 1. Study area and sampling technique

Water and sediment samples were collected at 12 stations in the Kune-Vain lagoon complex. 6 stations were selected for each lagoon (12 stations in total) in which 4 in their interior (K1-K4 and V1-V4) and 2 outside them in the Adriatic Sea (K5, K6 and V5, V6). The samples were taken in March 2023. Water samples were taken in Teflon containers in the amount of 2.5 liter for each station. Water samples were taken according to the recommendations of the ISO 5667-3:2018 method. They were transported and stored at +4^{o}C before their analysis in the laboratory. Sediment samples were taken at the same stations using a Van Veen auger. Firstly, sediment samples were air-dried and then dried in a thermostat at 105°C for 8 hours. They were sieved before their analysis. For the determination of organic pollutants, only the 63 micron fraction was analyzed. The sampling stations for the Kune-Vain lagoons are shown in Figure 1.

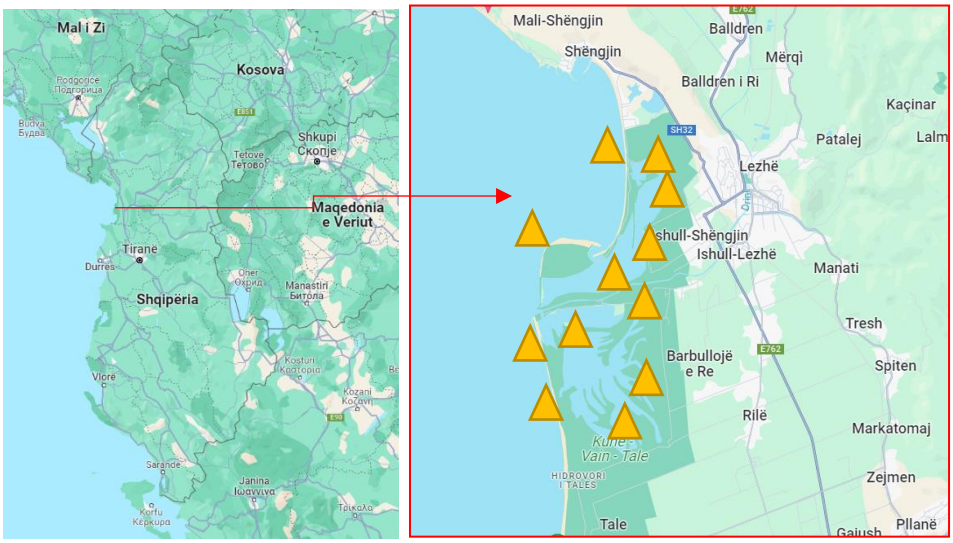

Figure 1. Sampling stations in the complex of Kune-Vaini lagoons

2. 2. Water sample treatment for chlorinated pollutants

Liquid-liquid extraction was used for the isolation of organochlorine pollutants (pesticides, their residues and PCBs) from seawater samples. One liter of water and 2 x 40 ml of n-Hexane as extraction solvent were added to a separatory funnel. After extraction, the organic phase was treated with 5 g of anhydrous Na2SO4 to remove water. Florisil open columns were used for the purification of water samples. 20 ml of n-Hexane/Dichloromethane (4/1) was used to elute the columns. After concentration to 1 ml of n-Hexane, the samples were injected into GC/ECD (Lekkas *et al*, 2004; Vryzas *et al*, 2009; Kostantinon *et al*, 2006; Nuro *et al*, 2014; Borshi *et al*, 2016; Murtaj *et al*, 2014)

2. 3. Sediment sample treatment procedure for OCP and PCB

For the determination of organochlorine pollutants, 5 - 20 g of sediment sample (fraction < 63 microns), dried and pre-sieved, were taken in a 100 ml Erlenmayer where 50 ml of n-Hexane/Dichloromethane (3:1) as extraction solvent. Their extraction was performed in ultrasonic bath for 60 minutes at 30^{o}C. After separation of the organic phase, 5 g of anhydrous sodium sulfate was added to remove water trace. The solvent was evaporated using Kuderna-Danish to 10 ml. Metallic mercury was added to the test tube until the complete

removal of sulfur compounds which cause problems in gas chromatographic analyze. The extract was transferred to a florisil glass column. Elution was performed with 20 ml of n-Hexane/Dichloromethane (4:1) and concentrated in a Kuderna-Danish heater block up to 2 ml. The extract was injected into the gas chromatograph equipped with ECD detector (Nuro *et al*, 2014; Borshi *et al*, 2016; Murtaj *et al*, 2014)

2. 4. Gs chromatography analyze of OCP and PCB

Organochlorine pesticides and PCBs were analyzed simultaneously using Rtx-5 capillary column (30m x 0.25mm x 0.25μm) in a Varian 450 GC gas chromatograph equipped with PTV injector and ECD detector. Helium was used as the carrier gas (1 ml/min) and nitrogen as make-up gas (24 ml/min). Manual injection was done in splitless mode at 300°C. The detected pesticides were: DDT (p,p-DDE, p,p-DDD, p,p-DDT), HCHs (a-, b-, γ- and d-isomers), Heptachlor (Heptachlor and Heptachlorepoxide); Chlordanet (alpha and gamma isomers); Aldrins (Aldrin, Dieldrin, Endrin and their derivatives) and Endosulfans (Endosulfan alfa, Endosulfan beta and Endosulfan sulfate). PCB analysis was based on the determination of seven markers (PCB IUPAC No. 28, 52, 101, 118, 138, 153 and 180). Quantification of pesticides and PCB was based on the external standard method (Nuro *et al*, 2014; Borshi *et al*, 2016; Murtaj *et al*, 2014)

2. 5. Water treatment procedure for PAH analyze

Two-step liquid-liquid extraction (LLE) was used for the extraction of PAHs from seawater samples. One liter of water was treated in a separatory funnel first with 40 ml Dichloromethane (first LLE step) and then with 40 ml n-Hexane (second LLE step). After extraction, the organic phase was dried with 5 g of anhydrous Na2SO4 to remove water. Extracts were concentrated in 1 ml n-Hexane using Kuderna-Danish and then injected into GC/FID for qualitative and quantitative analysis of PAH (Stogiannidis and Laane, 2015; Akkanen *et al*, 2005, Nuro *et al*, 2018; Borshi *et al*, 2018).

2. 6. Sediment samples treatment for PAH analyze

For PAH determination, 5-20 g of sediment sample (63 micron fraction) were taken in a 100 ml Erlenmayer where 40 ml of n-Hexane was added as extraction solvent. Their extraction was performed by using ultrasonic bath for 60 minutes at 30^{o}C. After separation of the organic phase, 2 g of anhydrous sodium sulfate was added to remove water. The solvent was evaporated using Kuderna-Danish to 2 ml. The extract was injected into the gas chromatograph

equipped with FID detector (Stogiannidis and Laane, 2015; Akkanen *et al*, 2005, Nuro *et al*, 2018).

2. 7. GC/FID determination of PAH in water and sediment samples

Gas chromatographic analyzes of PAH in water samples were performed with a Varian 450 GC apparatus equipped with a flame ionization detector and a PTV injector. Capillary column VF-1 ms (30m x 0.33mm x 0.25μm) was used for the separation of 13 PAH according to EPA Method 525. Helium was used as carrier gas at 1ml/min. The FID temperature was maintained at 280°C. Nitrogen was used as carrier gas (24 ml/min). Hydrogen and air were the flame detector gases at 30 ml/min and 300 ml/min, respectively. The EPA 525 standard mixture was used for qualitative and quantitative PAH analysis. Acenaphthylene, Fluorene, Phenanthrene, Anthracene, Pyrene, Benzo [a] anthracene, Chrysene, Perylene, Fluoranthene, Benzo [b] fluoranthene, Indeo [1,2,3-cd] pyrene, Dibenzo [a, b] anthracene and Benzo [g, h, i] perylene were determined in water and sediment samples. PAH quantification is based on the external standard method (Nuro *et al*, 2018; Borshi *et al*, 2018).

Results and discussions

In this study were evaluated levels of some priority substances (organochlorine pesticides, PCBs and PAHs) in water and sediment samples of Kune-Vain lagoons. These lagoons are located in the central Adriatic Sea, near Lezha city. Water and sediment samples were taken in March 2023, inside and outside lagoon areas. Organochlorine pesticides, their degradation products and PCB markers were analyzed using the GC/ECD technique while PAH were analyzed by using GC/FID technique. OCP, PCB and PAH compounds are classified as priority substances due to their persistence and toxicity. Monitoring of them in seawater is important not only for the environment, but also for organisms (including humans).

Figure 2 shows the total of organochlorine pesticides in water samples (ppb - ug/l) and sediment samples (ppb - ug/kg) for the Kune-Vaini lagoon complex. The average level of OCP for water samples was 2.33 ppb while for sediment samples 6.84 ppb. The highest level of pesticides for water samples was found in the iner stations of Kune lagoon (K1-K4). This could be related mainly by the impacts of agricultural lands near lagoons and the new arrivals by Drini and Mati rivers. This fact is also reflected in the sediment samples for Kune Lagoon, which also have the most polluted stations where pesticide levels were found from 2.75 to 15.28 ppb. Also, high levels of them have been found

for sediment samples in the iner stations of Vaini Lagoon (V1-V4). This can be related to the water currents inside the lagoon, the depth of the water, the granular particles in the water column, the speed of sedimentation and the influence of Drini River in these stations.

The profile of organochlorine pesticides in Kune-Vaini lagoons was shown in Figure 3. The profile of pesticides in water samples was: a-Chlordane > Dieldrin > Endrin > Endosulfan i > Methoxychlor > Aldrin > Lindane. The presence of these pesticides in water samples can be related mostly to the recent uses of them or their new arrivals from river discharges. The pesticide profile for the sediment samples was different. It was as follow: Aldrin > Endosulfane sulfate > DDE > Methoxychlor > Endrin ketone > a-Chlordane > Heptachlor epoxide. Profile of pesticides in water and sediment samples was influenced by the individual levels of each pollutant in specific stations, the time of their use, degradation processes and new arrivals or recent uses. Note that for all samples at higher levels were found their degradation products, and not the active substances (applied as pesticides). Persistence of individual OCP in the aquatic environment and/or in the sediment samples should be considered.

Figure 4 shown the classes of pesticides for the analyzed samples (water and sediment) from the Kune-Vain lagoon complex. For water samples, their profile was: Aldrins > HCH > Endosulfanes > Chlordanes > Heptachlors > DDT. For the sediment samples, their profile was: Aldrins > Endosulfanes > Chlordanes > DDT > Heptachlors > HCH. The profiles are mainly related to the physico-chemical properties of pesticides such as their stability, solubility, polarity, etc. The time and amount of their use are also factors that influence this profile. The effects of water currents inside and outside the lagoons are factors that can affect directly quantities and distribution of OCP in the lagoon complex.

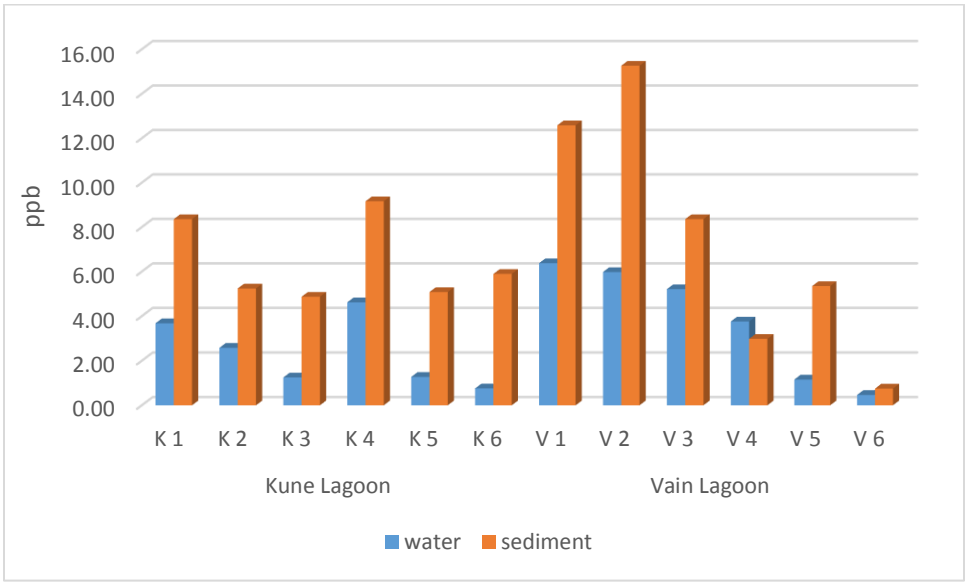

Figure 2. Total of organcohlorine pesticides in Kune-Vaini lagoon complex

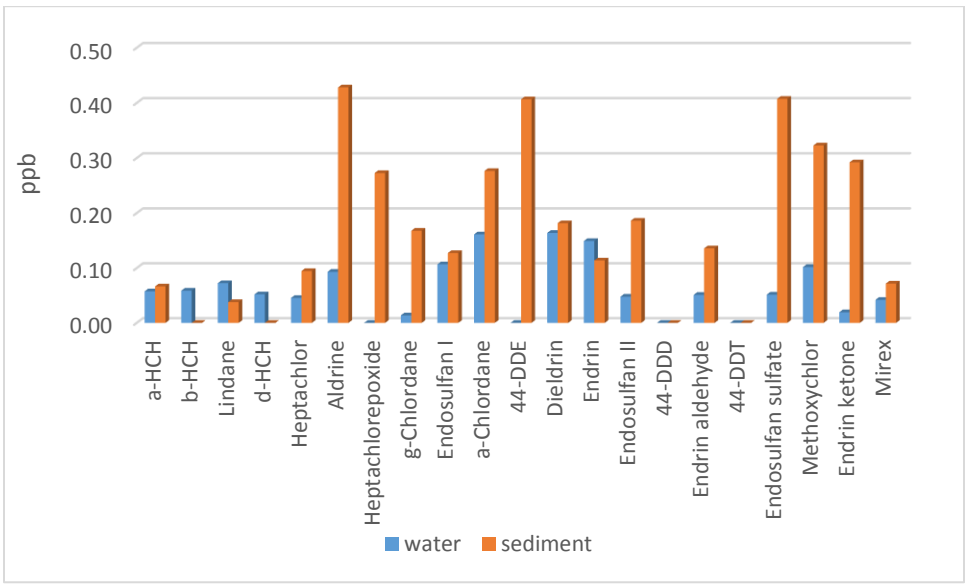

Figure 3. Profile of organochlorine pesticides in Kune-Vaini lagoon complex

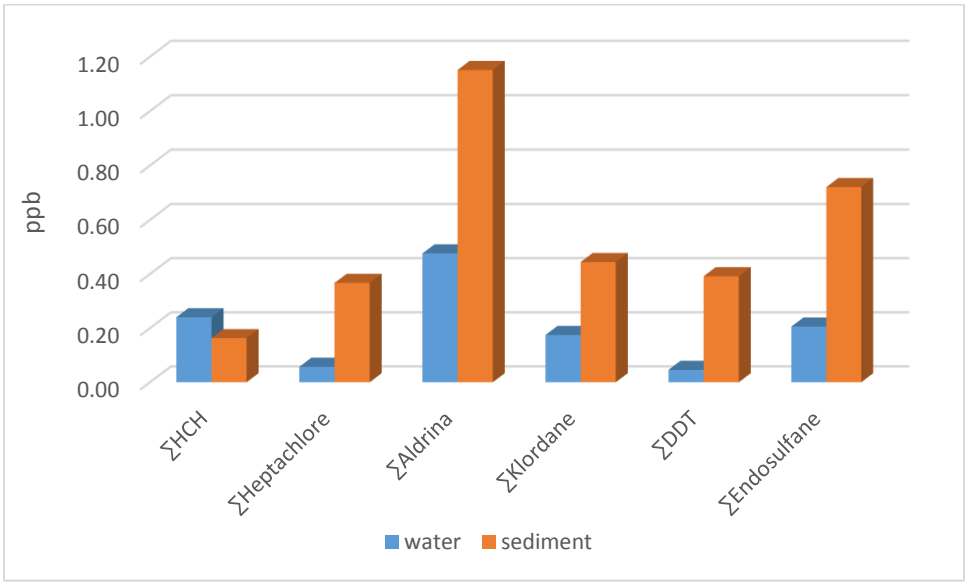

Figure 4. Profile of organochlorine pesticide classes in Kune-Vain lagoons

Figure 5 present the total of PCB markers in water samples (ppb - ug/l) and sediment samples (ppb - ug/kg) analyzed in Kune-Vaini lagoon complex, March 2023. Average contamination level for water samples was 3.78 ppb while for sediment samples 4.58 ppb. PCB level was found in higher level for water samples located in internal stations of both lagoons (K1-K4 and V1-V4). The high levels of them were detected at K2 and V1 stations. Their presence in water samples should be mainly due to atmospheric deposition. Also, PCBs were found at higher level for the sediment samples taken inside of both lagoons. Their presence must be because of terresterial sources near the study stations. Atmospheric deposition and the influence of water currents inside/outside the lagoons are not excluded.

The PCB profile in water and sediment samples was shown in Figure 6. The profile for the water samples was built almost by the presence of PCB 28 and PCB 52 which are representative of volatile PCBs. This fact once again proves the presence of PCB as a result of atmospheric deposits. In addition to volatile PCBs, the presence of heavy PCBs including PCB 180 was detected in the sediment samples. This could be related with punctual sources near these stations. The impact of ship transport and other industrial activities that take place near these stations are the main factors. Other factors that affect PCB

concentrations and profiles are sea currents, new arrivals from the rivers and the physical properties of each analyzed congeners.

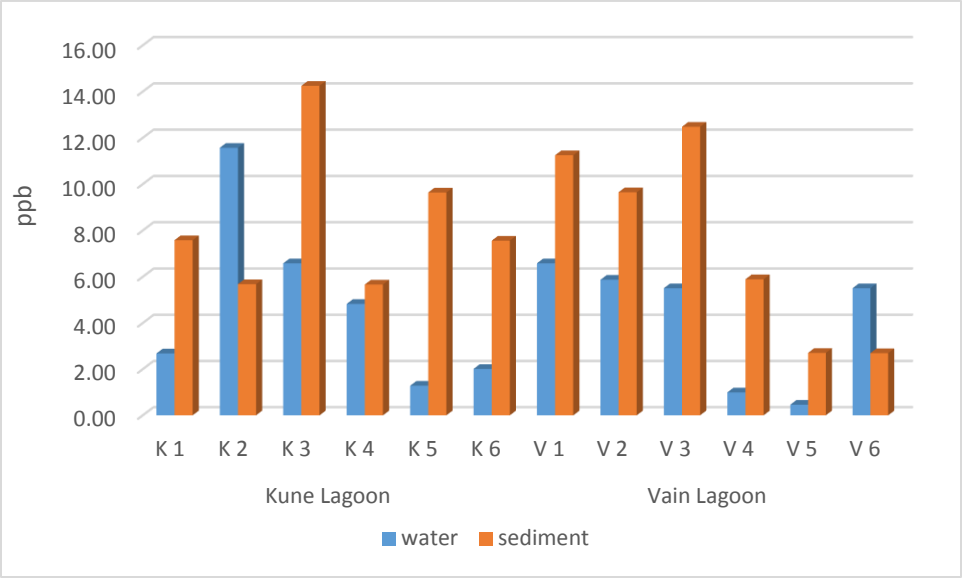

Figure 5. Total of PCB markers in Kune-Vaini lagoon complex, Mars 2023

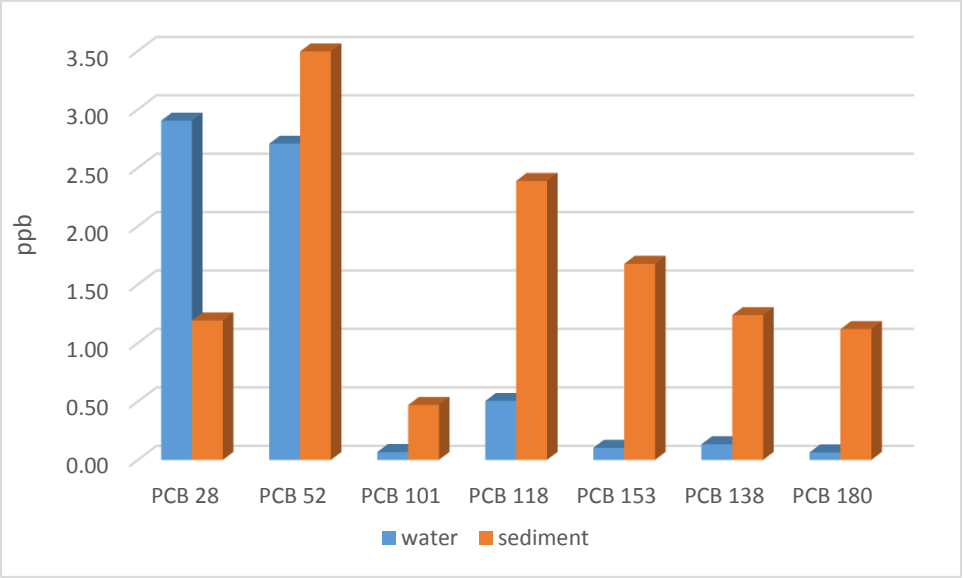

Figure 6. Profile of PCBs in Kune-Vain lagoons, March 2023

Figure 7 shown the total of PAH in water samples (ppm - mg/l) and sediment samples (ppm - mg/kg) for the Kune-Vain lagoon complex. The average level of PAH contamination in water samples was 0.41 ppm, while for sediment samples it was 2.35 ppm. The highest level of PAH for water samples was found in V1 station with 1.12 ppm. Their presence in water samples should be mainly due to marine transport or hydrocarbon spillages (acidents) at these stations. PAHs were found at high levels for sediment samples at V6 and K6 stations. Their presence must be mainly a consequence of marine transport and punctual sources and/or discharges of these pollutants near the study stations.

The PAH profile was shown in Figure 8. Their profile for the 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 PAH profile in the sediment samples was: Fluorene > Phenanthrene > Benzo[a]anthracene > Pyrene > Chrysene > Perylene > Acenftylene. These hydrocarbons are both natural and antropogenic origin. The influence of transport, pyrogenic processes, natural origin, water currents and their physico-chemical properties could be the main factors of PAH presence.

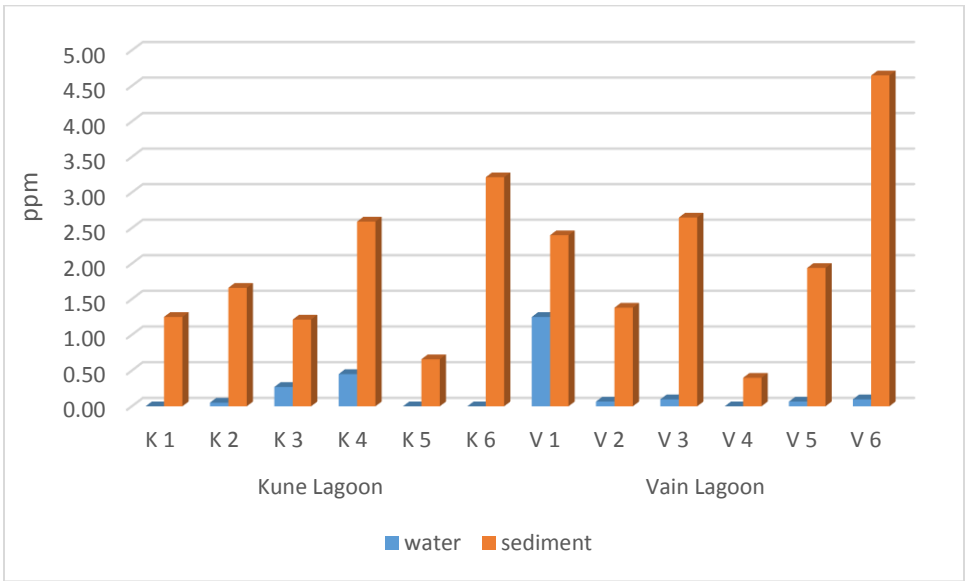

Figure 7. Total of PAH in Kune-Vaini lagoon, Mars 2023

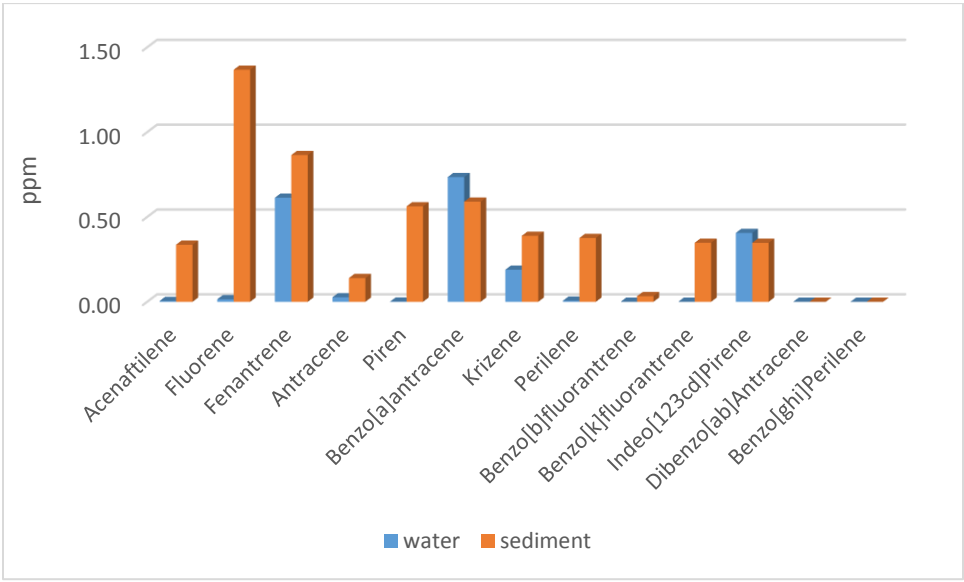

Figure 8. Profile of PAHs in Kune-Vain lagoons, March 2023

Conclusions

In this study, were evaluated the levels of organochlorine pesticides, PCBs and PAHs in water and sediment samples from Kune-Vaini lagoons, in the central Adriatic Sea. These compounds are part of priority substances list due to their persistence and toxicity. Organochlorine pesticides were detected in all analyzed stations. The level of OCP was greater in the sediment samples. This must be related mainly to the previous uses of these compounds, their degradation processes and new arrivals from rivers and water currents. Water and sediment samples inside the lagoons were most polluted. The profile of pesticides in water and sediment samples was influenced by the individual levels of each pesticide at specific stations. Degradation products were found in higher level confront to active substances applied as pesticides.

The presence of PCBs in water samples should be mainly due to atmospheric deposition because of detection of volatile PCB in high level. PCBs were found at higher levels for sediment samples. Their presence must be consequence of punctual sources of these pollutants near the study stations.

Atmospheric deposits and the influence of water currents could be the main pollution factors. Ship transport and industrial activities near these stations have a direct impact in PCB pollution. The presence of PAH must be mainly a consequence of ship transport and punctual sources of these pollutants near the study stations. The pollution level for pesticides, PCBs and PAHs in Kune-Vain lagoons was lower/comparable with previous works carried out in different areas of the Adriatic Sea (Nuro *et al*, 2014; Nuro *et al*, 2018; Borshi *et al*, 2016; Borshi *et al*, 2018; Murtaj *et al*, 2014). The presence of priority substances in the water and sediment samples of Kune-Vain lagoons should be an incentive for the authorities to have a continuous control of their levels in these areas and in the Adriatic Sea.

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