

## ORGANIC POLLUTANTS IN FIERZA LAKE

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### **Abstract**

*This study presents the levels of some organic pollutants in water and sediments from Fierza Lake. This ecosystem is the largest artificial lake in Albania. It is formed by water flows of White Drin and Black Drin rivers which join together near the Kukes city (Albania territory). Water basin area of this lake lied in huge territory including Kosovo (White Drin River originates near Peja), North Macedonia (Black Drin River originates from Ohrid Lake near Struga) and in Albanian area (East – Northeast). For this reason, Fierza Lake can be affected by urban waste, industrial and agricultural activity. Organochlorine pesticides (OCPs), polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs) and BTEX (Benzene, Toluene, Ethylbenzene and Xylene isomers) were determined in water samples and sediments of Fierza Lake. Water and sediment samples were taken at the same stations (10 stations in total) in May 2024. The liquid-liquid extraction technique was used for the isolation of OCPs, PCBs and PAHs from the water samples, while ultrasonic extraction was used for their extraction from the sediment samples. Head space technique were used for isolation of BTEX in water and sediment samples. The qualitative and quantitative analysis of organic pollutants was performed by using gas chromatography technique, Varian 450 GC, equipped with a  $\mu$ ECD and FID detector. Rtx-5 capillary column were used for the separation of organochlorine pollutants and VF-Ims column for PAH separation. Organic pollutants were found almost in all analyzed samples. Their profile was different between sediment and water samples for the same stations. Their level and profile could be connected with sources of pollution, pollutant properties, new arrivals from rivers (urban waste, agricultural and industrial activities), etc. Analysis of organic*

*pollutants in water and sediment samples of Fierza Lake must be continuous by authorities because this ecosystem can be affected by many factors.*

**Key words:** *Fierza Lake, organic pollutants; water analyzes; GC/ECD/FID.*

### **Përmbledhje**

*Ky studim paraqet nivelet e ndotësve organikë në ujë dhe sedimente nga Liqeni i Fierzës. Ky ekosistem është liqeni artificial me i madh në Shqipëri. Ai është formuar nga prurjet e lumenjve Drin i Bardhë dhe Drin i Zi, të cilët bashkohen pranë qytetit të Kukësit (në territorin Shqiptar). Zona ujëmbledhëse e këtij liqeni shtrihet në një territor të gjerë i cili përfshin Kosovën (Drini i Bardhë e ka burimin pranë Pejës), Maqedoninë e Veriut (Drini i Zi buron nga Liqeni i Ohrit pranë Strugës) dhe Shqipërinë. Ky ekosistem mund të ndikohet nga mbetjet/derdhjet urbane, aktiviteti industrial dhe bujqësor. Pesticidet klororganike (OCPs), poliklorbifenilet (PCB), hidrokarburet policiklike aromatike (PAH) dhe BTEX (Benzen, Toluën, Etilbenzen dhe izomerët e Ksilenit) u analizuan në mostra uji dhe sedimente nga Liqeni i Fierzës. Mostrat e ujit dhe sedimenteve u morrën në të njëjtat stacione (10 stacione në total) në Maj 2024. Për ekstraktimin e OCP, PCB dhe PAH nga mostrat e ujit u përdor teknika lëng-lëng ndërsa ekstraktimi me ultratinguj u përdor për izolimin e tyre nga mostrat e sedimenteve. Teknika "Head space" u përdor për izolimin e BTEX nga mostrat e ujit dhe sedimenteve. Analiza cilësore dhe sasiore e ndotësve organikë u realizua në aparatin e gaz kromatografit model Varian 450, i pajisur me detektor  $\mu$ ECD 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 gjitha mostrat e analizuara. Profili i tyre ishte i ndryshëm për mostrat e sedimenteve dhe të ujit për të njëjtin stacion të marrë në shqyrtim. Nivelet dhe profili i tyre mund të jetë i lidhur me burimet e të ndotjes, vetitë e ndotësve, prurjet e reja që vijnë nga lumenjtë (derdhje urbane, aktivitete bujqësore dhe industriale), etj. Analiza e ndotësve organikë në mostrat e ujit dhe sedimentit të Liqenit të Fierzës duhet të jetë i vazhdueshëm nga autoritetet përgjegjëse sepse ky ekosistem mund të ndikohet nga faktorë të ndryshëm.*

**Fjalë kyçe:** *Liqeni i Fierzës; ndotësit organikë; analizat e ujit; GC/ECD/FID.*

### **Introduction**

Fierza Lake is the main artificial lake in Albania. It was built on the Drin River to produce electricity (hydropower plant) in 1978. The dam of Fierze is on the

Drin River. Fierza Lake is 70 km length and a maximum area of 7000 ha. Its maximum depth is 128 m and its volume is about 2.7 billion m<sup>3</sup> (Camaj et al, 2024). The waters of the lake are also used for irrigation of agricultural land, fish growth/fishing and recreation for the residents of the area around it. The main water source of the lake is Drin River. This river ( the longest river in Albania), is formed by the union near the Kuksi City of its two branches: the White Drin and the Black Drin. The White Drin originates near Peja, Kosovo, in Radavc, while the Black Drin originates in Struga, North Macedonia, from the northern part of Ohrid Lake. The catchment area of Drin, including its two main branches, has a very large area (11,829 km<sup>2</sup>) which extends to Kosovo, Albania, North Macedonia and Greece (Prespa Lake), therefore the possibility of pollution of Fierza Lake is very high (camaj et al, 2024). The main sources of pollution, first in both branches of Drin River and then in the Fierze Lake, are: liquid/solid urban waste discharges, agriculture, industrial and mining waste, automobilist transport, mechanics, etc. (Camaj et al, 2024; Halo et al, 2023; Nuro et al, 2024).

In this study was determined concentrations of organochlorine pesticides (OCPs), polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs) and BTEX (Benzene, Toluene, Ethylbenzene and Xylene isomers) in water and sediment samples of the Fierza Lake. OCP and PCB have chlorine atoms in their molecules (organochlorine pollutants) while PAH and BTEX are hydrocarbons (polycyclic aromatic and benzene derivatives). These pollutants have high stability, high bioaccumulation capacity, and high toxicity. They can be found far away from the application site because they have the ability to spread out easily in the environment (Guan et al, 2009; Lekkas et al, 2004; Marini and Frapiccini, 2013).

This is the reason why these pollutants are part of the priority substances list. It is recomanded that these pollutants must be continuously monitored in the environment (Halo et al, 2023; Nuro et al, 2024). The main use of pesticides is for agricultural purposes, but their absorption in the soil and further soil leaching cause these compounds to end up in the environment (especially in the water environment). Their punctual sources or intentional/accidental spills do not exclude their presence in the environment. PCBs mixtures are widely used in generators and electrical transformers oils. Also, PCBs have many other uses as plasticizers, in photocopies, etc.

They can be easily formed from burning urban waste or industrial emissions of processes that realized at high temperatures. These are some of the reasons

why these pollutants are so widespread in the environment (Leklas et al, 2014; Li et al, 2006; Nuro et al 2022; Yang et al, 2009). PAH and BTEX are pollutants that have different origins, starting from the natural background, urban spills, mines, mechanical businesses, fuel supply points, emissions from transport, emissions from industry, massive forest fires, waste burning, etc. (Borshi et al, 2018; Nuro et al, 2018; Storelli et al, 2007). Even these pollutants are widespread in nature due to the many opportunities that they have to penetrate the environment. This study aims to bring not only analytical data but also to be an information for institutions, interested persons/associations and residents of the area near Fierze Lake.

## **Material dhe methods**

### **2. 1. Study area and sampling in Fierza Lake**

Water and sediment sampling were realized in May 2024 at 10 different stations of Fierza Lake. Water samples were taken in glass containers in the amount of 2.5 liters for each station. Water samples were transported and stored at +4°C before their analysis at the laboratory. In each station, sediment samples were taken, in a depth of 0.8 - 1.7 m using the Van Veen grab sampler. The sediment samples primary were air dried and after that were dried for 4 hours at 105°C in a thermostat. Sediment samples were ground in a mortar and sieved. In the analyze was taken only the 63 micron fraction. The sampling stations in Fierza Lake were shown at Figure 1.



**Figure 1.** Sampling stations at Fierza Lake

## 2. 2. Treatment of water sample for organochlorine pollutants

Liquid-liquid extraction was used for the isolation of organochlorine pollutants (pesticides, their residues and PCBs) from water 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 dry with 5 g of anhydrous Na<sub>2</sub>SO<sub>4</sub>. A florisil column was used for the clean-up procedure. 20 ml of n-Hexane/Dichloromethane (4/1) was used to elute the column. 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*, 2024; Borshi *et al*, 2016; Murtaj *et al*, 2013)

## 2. 3. Treatment of sediment samples for OCPs and PCBs analyze

For the determination of organochlorine pollutants, 15 - 20 g of sediment sample (fraction < 63 microns), were taken in a 100 ml Erlenmayer where 50 ml of n-Hexane/Dichloromethane (3:1) as extraction solvent were added. Their extraction was performed in ultrasonic bath for 60 minutes at 30°C. After separation of the extract was evaporated using Kuderna-Danish to 10 ml. Metallic mercury was added in a closed test tube until the complete removal of sulfur compounds. The extract was transferred to a florisil glass column. Elution was performed with 20 ml of n-Hexane/Dichloromethane (4:1).

Extracts were concentrated in Kuderna-Danish up to 2 ml. The extract was injected into the gas chromatograph equipped with ECD detector (Nuro *et al*, 2024; Borshi *et al*, 2016; Murtaj *et al*, 2014)

#### **2. 4. GC/ECD analyze for OCPs and PCBs**

Organochlorine pesticides and PCBs were analyzed simultaneously using Rtx-5 capillary column (30m x 0.25mm x 0.25 $\mu$ m) in a Varian 450 GC gas chromatograph equipped with PTV injector and ECD detector. Nitrogen was used as the carrier (1 ml/min) and make-up gas (24 ml/min). Manual injection was done in splitless mode at 280°C. The analyzed pesticides were: DDT (p,p-DDE, p,p-DDD, p,p-DDT), HCHs ( $\alpha$ -,  $\beta$ -,  $\gamma$ - and  $\delta$ -isomers), Heptachlor (Heptachlor and Heptachlorepoxyde); Chlordanet ( $\alpha$  and  $\gamma$  isomers); Aldrins (Aldrin, Dieldrin, Endrin and their derivatives) and Endosulfans (Endosulfan  $\alpha$ , 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 (Guan *et al*, 2018; Li *et al*, 2009; Lazar *et al*, 2011; Nuro *et al*, 2024; Borshi *et al*, 2016; Murtaj *et al*, 2013; Mohamed *et al*, 2011)

#### **2. 5. Treatment of water samples for PAH analyze**

Two-step liquid-liquid extraction (LLE) was used for the extraction of PAHs from water 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 Na<sub>2</sub>SO<sub>4</sub> 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; Guan *et al*, 2005, Nuro *et al*, 2018; Borshi *et al*, 2018).

#### **2. 6. Treatment of sediment samples for PAH analyze**

For determination of PAH, 15-20 g of sediment sample (63 micron fraction) were taken in a 100 ml Erlenmayer where 50 ml of n-Hexane was added as extraction solvent. Their extraction was performed by using ultrasonic bath for 60 minutes at 30°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 (Froehner, et la, 2018; Stogiannidis and Laane,

2015; Mandić and Vrančić, 2017; Marini and Frapiccini, 2013; Nuro *et al*, 2024).

### **2. 7. GC/FID determination of PAHs 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. Nitrogen was used as carrier (1ml/min) and make-up gas (24 ml/min). Hydrogen and air were the flame detector gases at 30 ml/min and 300 ml/min, respectively. The FID temperature was maintained at 280°C. 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 (Borshi *et al*, 2018; <sup>a</sup>Nuro *et al*, 2018; Stogiannidis and Laane 2015; Yang *et al*, 2019).

### **2. 8. HS-SPME technique for determination of BTEX in water and sediment samples**

Determination of BTEX in water and sediment samples was realized using solid phase micro-extraction in static head space mode (HS/SPME) followed by GC/FID technique. 5 ml of water sample (or 5 g dry sediment) was put in a 10 ml head space vial. 100 µm PDMS (Polydimethyl Siloxane) fiber was used to extract BTEX from water and/or sediment samples. Adsorption process was realized in 50°C (using a water bath) for 30 minutes. Desorption process (20 seconds in 280°C) was realized in the 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 separation of Benzene, Toluene, Ethyl benzene and Xylene isomers. Nitrogen was used as carrier and make-up gas. FID temperature was held at 280°C. A mixture of BTEX was used for their qualitative and quantitative analyze based on external standard method (<sup>b</sup>Nuro *et al*, 2022).

## **Results and discussions**

In this study were performed analysis of organochlorine pesticides (21 individual of pesticides and their residues according to EPA 8081B), PCB markers (7 congeners), PAH (13 most toxic according to EPA 525 Method)

and BTEX in water and sediment samples of Fierza Lake in May 2024. These organic pollutants can be found in this important ecosystem because of industrial and agricultural activity as well as urban pollution in the water basin area of this lake. Note that, these pollutants are very stable compounds and can be found in the environment far away from the application place for many years after their application.

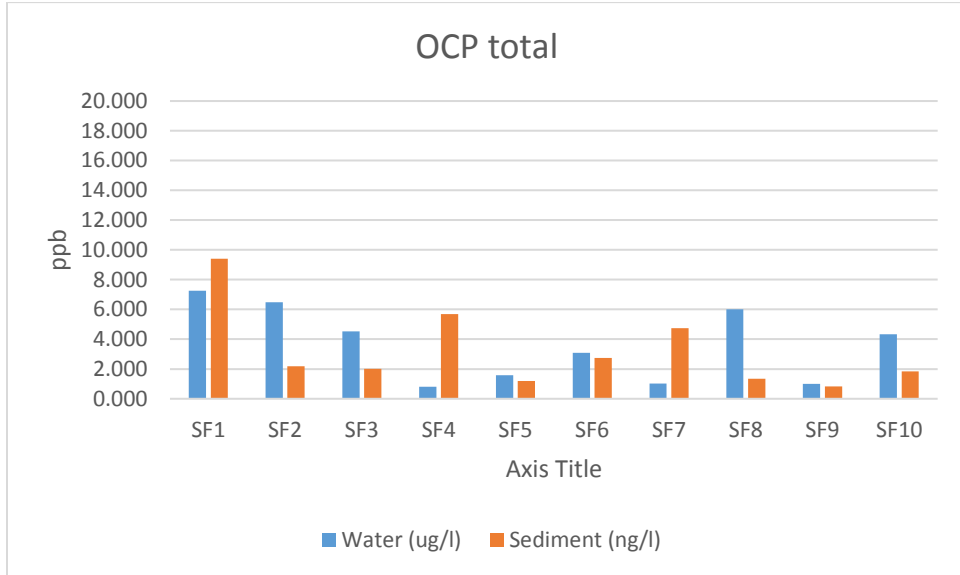
Organochlorine pesticides and their residues were found in all analyzed water and sediment samples of Fierza Lake (Figure 2). Their concentrations (average values) in water samples were 3.61 ppb (or ug/L) and 3.20 ppb (or ug/kg dry sediment) in sediment samples. The highest level of pesticides in the water were found in station SF1 (near the city of Kuksi) with 9.41 ppb, followed by their levels in stations SF4 (5.68 ppb) and SF7 (4.75 ppb). The lowest levels of them were for stations SF5, SF8 and SF9 where the total was  $\leq 1$  ppb. For sediment samples, the levels were in the range between  $\leq 1$  ppb (at stations SF4, SF7 and SF9) to 7.25 ppb (at station SF1).

The highest levels of pesticides in the water samples should be related to their recent use for agricultural purposes. Their presence may also be related with water irrigation of agricultural lands and their transfer first to channels, river water and then to the lake. Profile of pesticides in water samples (Figure 3) was as follows: Endosulfan II, Aldrin, Endrin, Endosulfan I, Endrin aldehyde and d-HCH; while their profile in sediment samples was: DDE, Endrin ketone, Endosulfane II, Endosulfane I and a-HCH. Figure 4 shows the profile of OCP classes in Fierza Lake. Aldrines and Endosulfanes were found in higher concentration than other pesticides. These OCPs can be used recently in agricultural areas in the catchment area of the lake. Their presence may also be a consequence of any punctual sources (accidental/intentional spillage) or from soil leaching from agricultural lands.

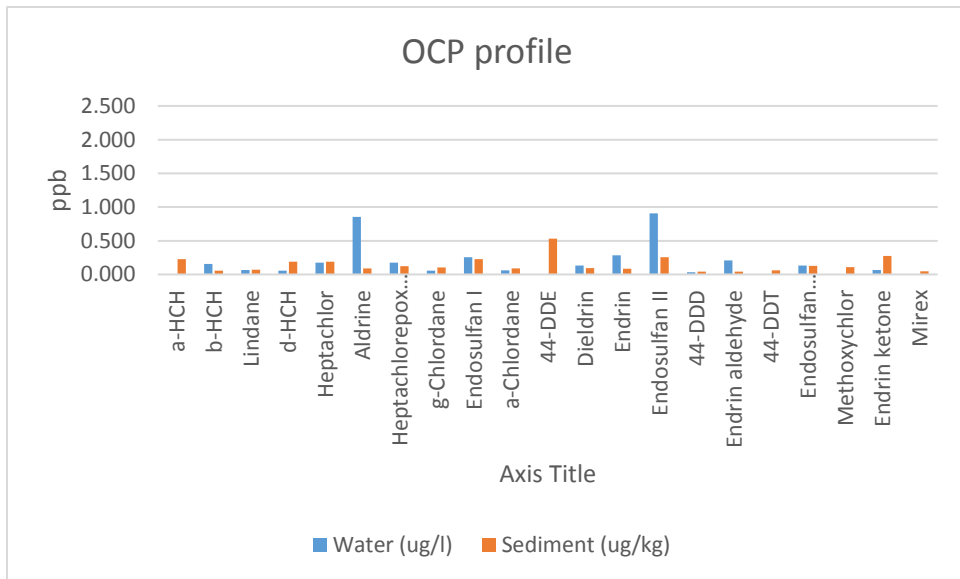
Also, their presence has been reported as an impurity in pesticides that are allowed to be used for agricultural purposes (Nuro et al, 2016). In the sediment samples were detected almost all pesticides, but the highest levels were for DDT, Endosulfan, Aldrin and HCH. The observed differences in the total and profile of pesticides for water and sediment samples should be related to the type of pesticides used by the farmers of these areas, to the time of their use and/or arrival in the lake and also, to the physico-chemical properties of each analyzed individual. Pesticide concentration for water samples did not exceed the standards set by Directive 2008/105/EC for surface waters. These levels can also be considered lower/comparable to the analyzed surface water in our



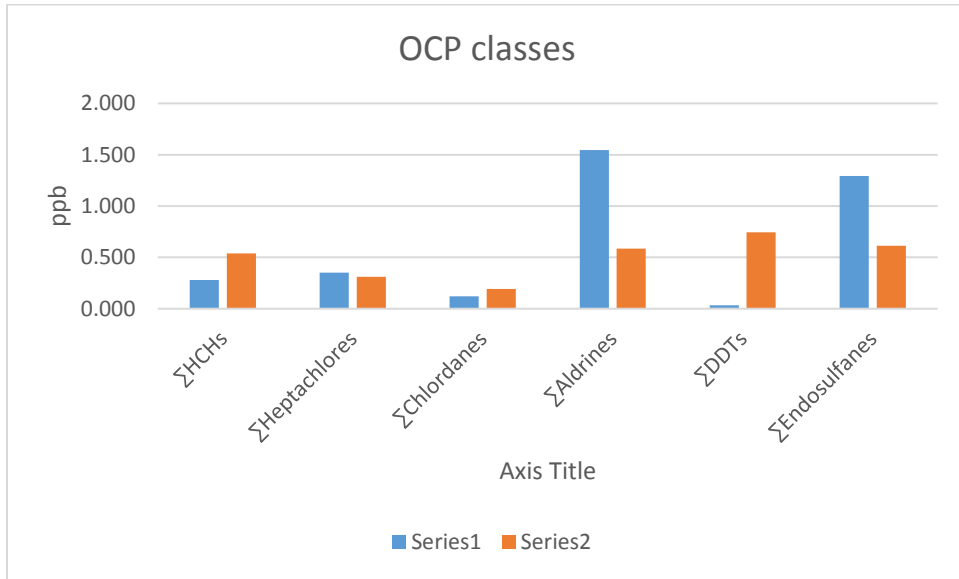
country in recent years (Borshi et al, 2016; Camaj et al, 2024; Halo et al, 2023; Myrtaj et al 2013; <sup>a</sup>Nuro et al, 2022; Nuro et al, 2024).



**Figure 2.** Total of pesticides in water and sediment samples of Fierza Lake



**Figure 3.** Profile of pesticides and their degradation products in water and sediment samples of Fierza Lake



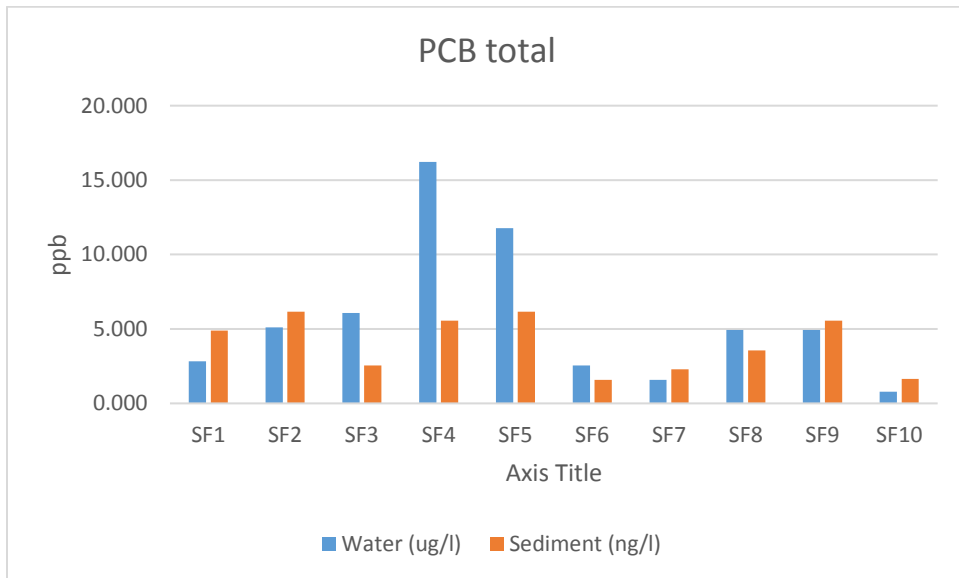
**Figure 4.** Classes of pesticides in water and sediment samples of Fierza Lake

PCBs were found almost in all analyzed samples (water and sediment) of Fierza Lake (Figure 5). Their concentrations in water samples ranged from  $\leq 1$  ppb (or ug/l) in SF10 and SF7 stations to 16.23 ppb in SF4 station. Also, for SF5 station, total of PCB was found in higher concentration than others stations (11.25 ppb). Also, stations SF4 and SF5 were most polluted for sediment samples with 6.04 ppb. Stations SF6, SF7 and SF10 had the lowest PCB levels at about 2.0 ppb. Note, that for the same stations, PCB levels were 2-3 times higher than OCPs. PCB presence could be because of some mechanical businesses located in the water basin area (including cities and surroundings of Peja, Kukes, Diber, etc.) discharge their wastes directly to the rivers and/or effluents of Fierza Lake. Also, repairs of electrical transformations and/or generators used in Fierza hydropower could be an important factor for their presence.

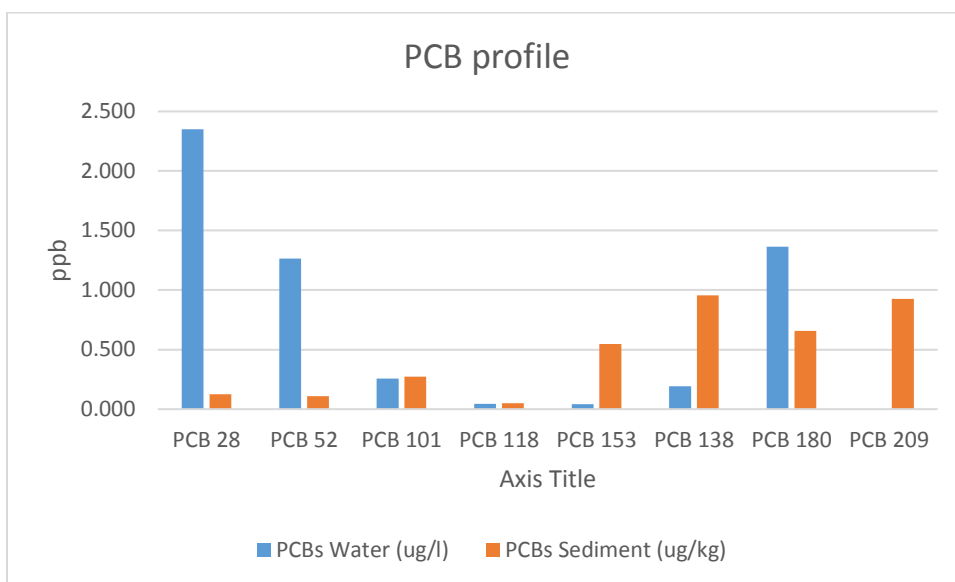
The profile of PCB markers (Figure 6) in water was: PCB 28 > PCB 180 > PCB 52. The presence of volatile PCB in high levels (PCB 28 and PCB 52) was noted. Volatile PCBs can have terrestrial and/or atmospheric origin, while heavy PCBs (PCB 180) only have terrestrial origin. PCB levels in water samples were lower than 50 ppb, which is the maximum level defined in Directive 2008/105/EC. Even the levels of PCB markers in the water of Lake

Fierze had levels comparable to the levels reported by works of recent years for Albanian waterbodies (Camaj et al, 2024; Halo et al, 2023; Myrtaj et al 2013; <sup>a</sup>Nuro et al, 2022; Nuro et al, 2024).

The profile of PCBs in sediment samples was very different from the water samples. A higher level of heavy PCBs was observed, which could be related to their presence in this area from terrestrial sources. This fact, should be brought to the attention of the authorities that take care of the preservation of the environment in this area.



**Figure 5.** Total of PCBs in water and sediments samples

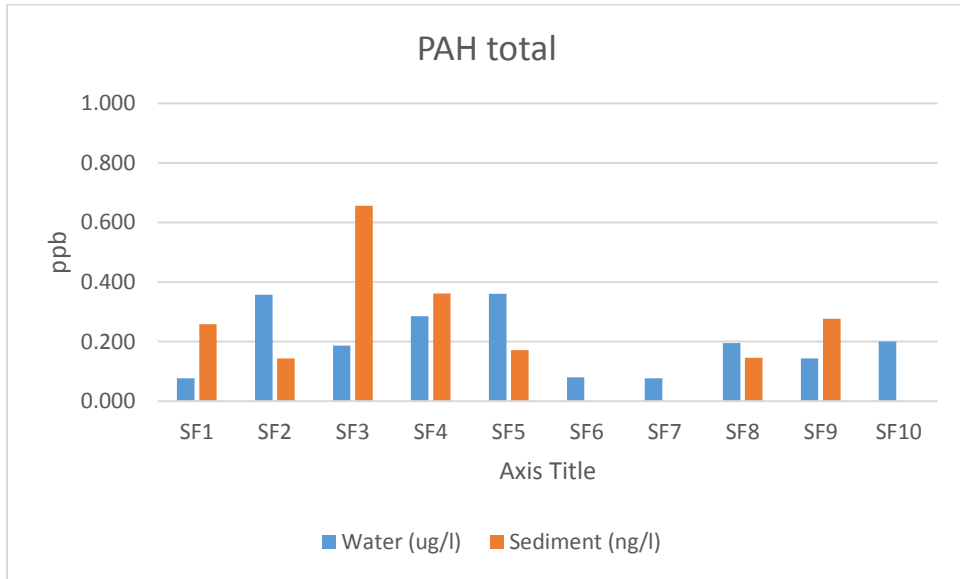


**Figure 6.** Profile of PCB markers in water and sediment samples

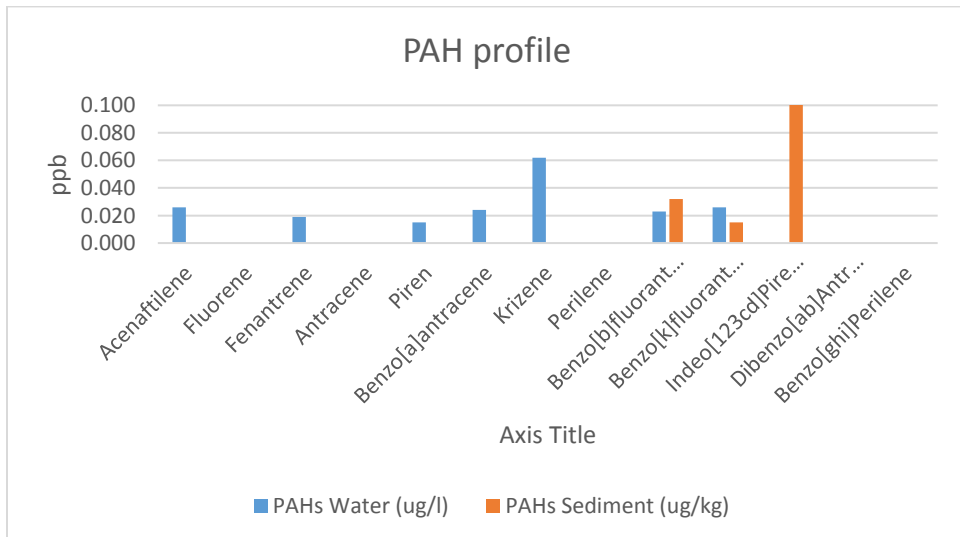
Figure 7 shows the total of PAH levels in water and sediment samples of Fierza Lake. PAHs were found in all water samples and around 70% of sediment samples. Their mean concentration in water samples was 0.2 ppb (or ug/l) while in sediment samples was 0.24 ppb (or ug/kg). Their concentrations in water samples ranged from  $\leq 0.1$  ppb (SF1, SF6 and SF7) to 0.36 ppb (SF2 and SF5). PAH were not detected in sediment samples of SF6, SF7 and SF10 stations, while their maximum for this sample type were found in SF3 station (0.66ppb). Their presence in water and sediment samples could be because of automobile transport, gasoil distributors near the lake and discharges of some mechanical businesses in the water basin area of the lake.

Their presence at a higher level in water should be related to their recent arrivals and their low solubility in water (PAHs and hydrocarbons generally are placed in the form of a film on the surface of the water). In the water samples, PAH with small molecular mass (non-pyrogenic origin) and PAH with large molecular mass (pyrogenic origin) were detected (Figure 8). Their main sources should be from the burning of forests/urban waste, automobile transport or industrial processes that take place at high temperatures. The individual levels for each of the analyzed PAHs do not exceed the maximum level determined by Directive 2008/105/EC. These levels were

lower/comparable with other surface waters in Albania (Borshi et al, 2018; Camaj et al, 2024; Halo et al, 2023; <sup>a</sup>Nuro et al, 2022; Nuro et al, 2024). PAHs of pyrogenic origin were found at higher level in the sediment samples. This fact could be related to their physico-chemical properties, mainly their solubility in water, stability and the ability to bind to the sediments.



**Figure 7.** Total of PAH in water and sediment samples



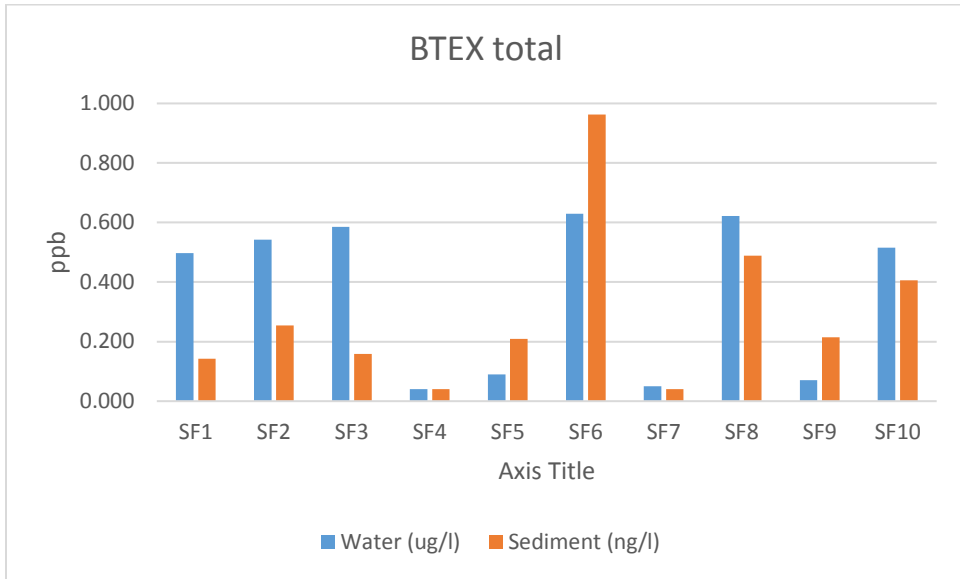
**Figure 8.** Profile of PAH in water and sediment samples

Total of BTEX in water and sediment samples of Fierza Lake was shown in Figure 9. Benzene and its derivatives were found in all water samples and around 70% of sediment samples. Benzene and its derivatives were found for all analyzed samples. Their mean concentration in water samples was 0.36 ppb (or ug/l) while in sediment samples was 0.29 ppb (or ug/kg). Their concentrations in water samples ranged from  $\leq 0.1$  ppb (SF4 and SF7) to around 0.6 ppb (SF2, SF3, SF6, SF8 and SF10).

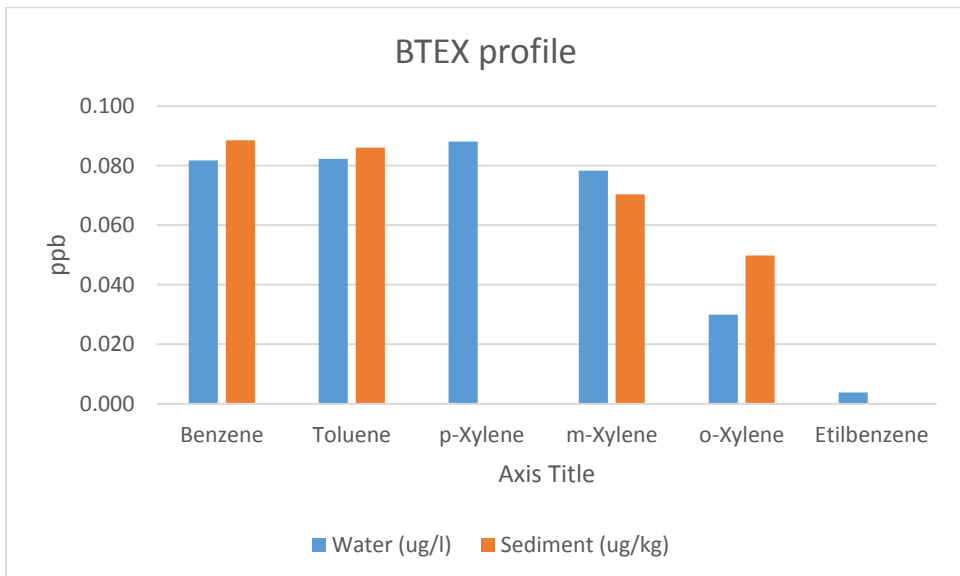
Minimum of BTEX in sediment samples were in SF4 and SF7 stations (lower than 0.05 ppb) while their maximum was in SF6 station (0.96ppb). Their presence in water and sediment samples could be for the same factors as PAHs (PAH and BTEX have almost the same origin). Some important factors of their presence in Fierza Lake could be: automobilist transport, gasoil distributors, discharges of wastes by mechanical businesses, burning of forests/urban waste, etc.

The levels of Benzene and Toluene in all water samples do not exceed the level determined by Directive 2008/105/EC. Also, these levels were comparable to reported data of recent years for surface waters in Albania (Camaj et al, 2024; 2022; <sup>b</sup>Nuro et al, 2022; Nuro et al, 2024). Their presence at a higher level in analyzed water samples is related to their low solubility in water (Also, BTEX form a film thickens in water surface).

The profile of BTEX (Figure 10) in water samples was: Benzene > Toluene > p-Xylene > m-Xylene > o-Xylene > Ethylbenzene. The profile was similar for the sediment samples with the exception of p-Xylene and Ethylbenzene which were not detected. This similarity in their profiles is also related to their same origin in this ecosystem.



**Figure 9.** Total of BTEX in water and sediment samples



**Figure 10.** Profile of BTEX in water and sediment samples

## Conclusions

The objective of this study was evidence of some most toxic persistent pollutants in water and sediment samples of Fierza Lake. Determinations of organochlorine pesticides, their residues, PCB markers, PAHs and BTEX were analyzed based on EU protocols for respective matrices followed by GC/ECD/FID technique. OCPs were found almost in all analyzed samples of Fierza Lake. Presence of pesticides could be because of their previous use for agricultural purposes in the water basin area of this lake. Aldrins and Endosulfanes were the most frequent classes of pesticides.

Their presence may be the result of recent uses or possible punctual sources. PCBs were found in all analyzed samples sometimes 2-3 times higher than OCPs because of their atmospheric deposition origin and/or terrestrial sources (especially function/defect/remont of transformers and generators of Fierza hydropower plant). Also, PAH and BTEX were found in all analyzed samples. Their presence could be because of automobile transport, mechanical businesses, industrial processes, burning of forests/urban wastes, etc. the levels of organic pollutants in this ecosystem were comparable to the levels reported in recent years for surface waters in Albania.

Although the presence of organic pollutants was in levels lower than the determined limits according EU and Albanian norms, their presence should encourage the responsible institutions for continuous analysis of water, sediments and biota of this important ecosystem.

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