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Some of the characteristic substances in the Devola River, Albania

ABSTRACT

This preliminary study presents first data on organochlorine pesticides (OCPs) and polychlorinated biphenyls (PCBs) concentrations in water and sediment samples of Devolli River. It is located in Southeast Albania, in Korca field, which is the second agricultural area in Albania. Sampling stations water of Devolli River were selected in eight different stations, starting from its stream (Gramozi Mountain) to the end of Korca field, near Banja hydropower (Gramshi region). Sampling of water and sediments were done in the same stations, in December 2018. Pesticides were used in the past in this area for many years mainly for agricultural purposes. Also, basin of Devolli River is affected from industrial activity and urban pollution, too. Water irrigation and rainfall contribute on their transport from soil to underground and surface waters. Organochlorine pesticides and PCB are very stable compounds that can be found in the environment for many years after their application. Liquid-liquid extraction was used for isolation of OCPs and PCBs in water samples while ultrasonic extraction was used for their extraction in sediment samples. The simultaneous analysis of organochlorine pollutants in water samples was performed by gas chromatography technique using electron capture detector (GC/ECD). Rtx-5 capillary column (30 m x 0.33 mm x 0.25 um) was used for separation of 17 organochlorine pesticides according EPA 8081A and 7 PCB markers. Relatively high concentrations of organochlorine pesticides were detected in water and sediment samples of Devolli River compare with other rivers of Albania. Their presence can be due to their previous use in Korca field. Volatile PCBs were found in higher concentration in water samples because of atmospheric deposition. Levels of some individual pesticides in surface water samples of Devolli River were found to be higher than permitted levels according to Albania and EU norms. Responsible authorities should be done continues monitoring in water samples of this river.

***Keywords**: Devolli River; organochlorine pesticides; PCB markers; water analyze; sediment analyze; GC/ECD*

1. INTRODUCTION

The Devolli River is 196 km long. It originates from Gramozi Mountain (Southeast Albania), continues its course near Bilishti city, passes through the Korca Field and then in the mountainous area between Korca and Gramshi region. All water catchment area of the Devoll River lies in Albania teritory. It is composed of a very large number of seasonal streams which are dry in summer and torrential in winter.

The catchment area of the Devoll River is 3,139 km² and the average altitude above sea level is about 960 m. Its average flow is 49.5 m³/s. The river is slope up to the Banja hydropower plant and behind it the river takes characteristics of a calm river. Near Kucova (in central Albania) it joins to the Osumi River to form the Semani River which flows into the Adriatic Sea.

Note that, there is no previous evidence about the concentration of organochlorine pollutants in the water ecosystem of the Devoll River. This preleminary study shown not only their pollution level in Devolli River but indirectly the impact of agricultural activity in Korca field which lies in the South-East of Albania. This area was used and continue to be used for agricultural purposes because is very fertile, especially for cornes, fruits and vegetables. The main parts of these fields are

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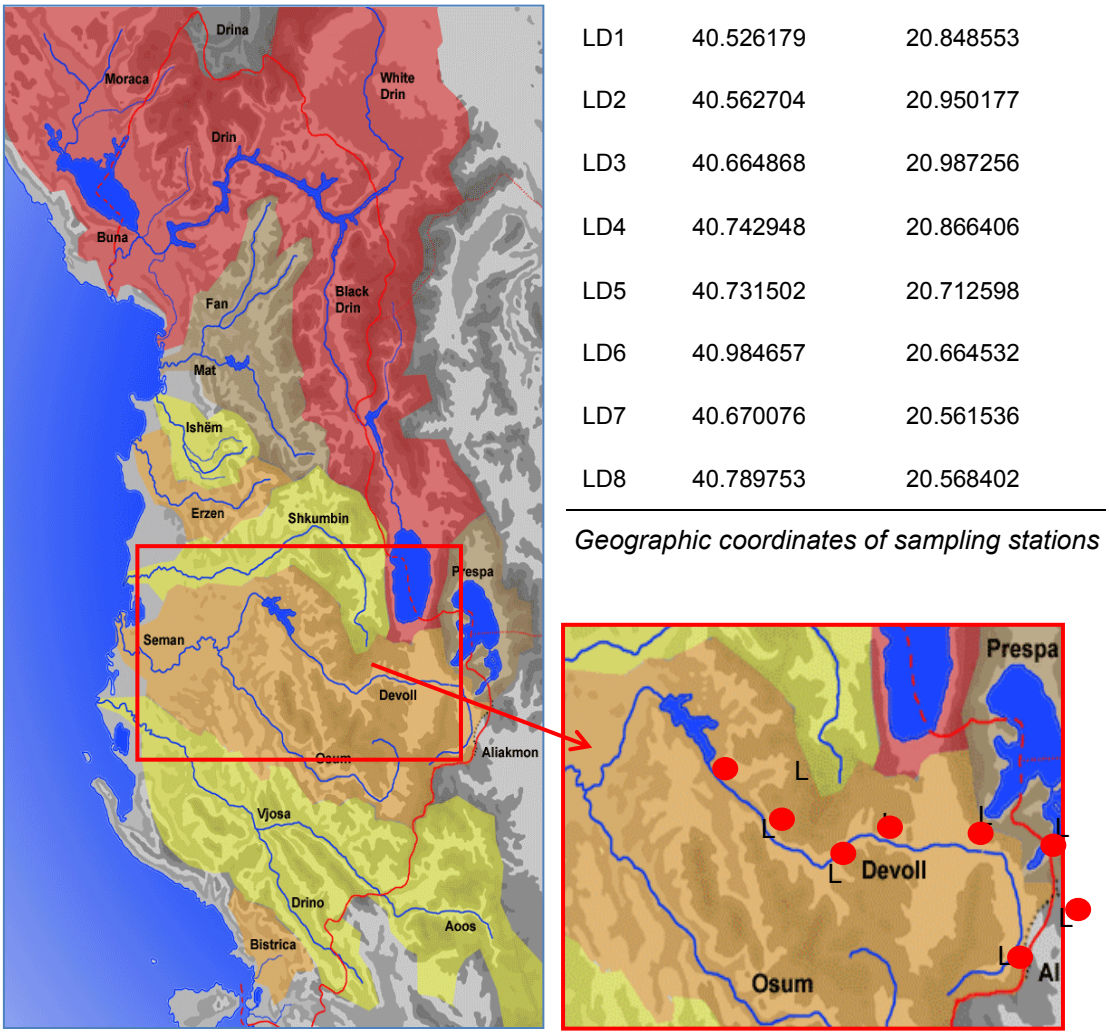
covered by the Devolli River and their branches. In the past the main parts of Korca field have been a wetland. For this reason, firstly, pesticides were used for against mosquitos (malaria vector) and after that for agricultural purposes. Organochlorine pesticides (DDT, Lindane, HCB, etc) were used widely in this area from 1946 to 1990s. Polychlorinated biphenyls (PCBs) were not in use in Albania until 90' but they are reported in many ecosystems of our country because of atmospheric depositions [1-5].

OCPs and PCBs have high stability, high bioaccumulation capacity and the ability to spread out far away of the application site. Generally, these compounds are difficult to degrade especially in the soil or sediment where their speed of degradation is lower. Many chromatographic methods were developed to detect all possible and known organic pollutants in environmental samples. Determination of chlorinated pollutants in surface water and sediment samples of Devolli River was realized by using capillary GC/ECD method.

2. MATERIAL AND METHODS

Sampling of water and sediment samples in Devolli River

Water and sediment samples of Devolli River were taken in eight stations, starting from Gramozi Mountain (where it is located its stream) to Gramshi area (before Devolli Hydropower plant). Stations were the same for both matrices. Sampling was realized in December 2018. The sampling stations are presenrted in Figure 1. Water samples (2.5 l) were taken in Teflon bottles. The sampling method was based on UNEP/MED Wg. 128/2, 1997. Sediments were taken using standard Van Veen grape (10 to 30 cm in depth). Water and sediment samples were tranported and conserved at +4^{o}C prior to their analyze.



LD1	40.526179	20.848553
LD2	40.562704	20.950177
LD3	40.664868	20.987256
LD4	40.742948	20.866406
LD5	40.731502	20.712598
LD6	40.984657	20.664532
LD7	40.670076	20.561536
LD8	40.789753	20.568402

Geographic coordinates of sampling stations

Figure 1. Sampling stations in Devolli River

Slika 1. Stanice za uzorkovanje u reci Devolli

Samples treatment for pesticide and PCB analyzes

Water samples: Liquid-liquid extraction was used to extract simultaneously organochlorine pesticides and polychlorinated biphenyls from water samples of Devolli River. Water (1000 ml) and n-Hexane (30 ml as extracting solvent) were added in a separatory funnel. After separation, the organic phase was dried with 5 g anhidrous Na_2SO_4 for water removing. A Florisil column was used for final clean-up procedure. 20 ml n-Hexane/Dichloromethane (4/1) was used for their elution. After concentration to 2 ml, the samples were injected in GC/ECD [6-8].

Sediment samples: Ultrasonic bath was used for extraction of OCPs and PCBs from sediment samples of Devolli River. 10 g dry sediments and 50 ml n-Hexane/Dichloromethane (3/1) were added in glass vial (100 ml) with Teflon cap. After extraction, the organic phase was dried with 5 g anhidrous Na_2SO_4 for water removing. Elute was treated with metallic mercury for sulfur removing. A Florisil column was used for the final clean-up step. 20 ml n-Hexane/Dichloromethane (4/1) was used for elution of organochlorine pollutants. After the concentration to 2 ml, the samples were injected in GC/ECD [6-9].

Chromatographic analyzes of pesticides and PCBs

Organochlorine pesticides and PCBs were analyzed simultaneously using capillary column Rtx-5, (30m long x 0.25mm i.d. x 0.25 μm film thicknesses) on a gas chromatograph HP 6890 Series Plus with μECD detector. Helium was used as carrier gas (1ml/min) and nitrogen as make-up gas (24 ml/min). Manual injection was done in splitless mode in 280^0C. The organochlorine pesticides detected were: HCHs (a-, b-, γ- and d-isomers), DDT's-related chemicals (o,p-DDE, p,p-DDE, p,p-DDD, p,p-DDT), Heptachlors (Heptachlor and Heptachlor epoxide), Chlordanes (alfa and gama isomer), Aldrine's (Aldrine, Dieldrin, Endrin and Endrin ketone) and Endosulfane's (I, II and Endosulfane sulphate isomer's). Analysis of PCBs was based on the determination of the seven PCB markers (IUPAC Nr. 28, 52, 101, 118, 138, 153, 180 plus 209 congenier).

Quality assurance procedures included the analyses of certified sample IAEA 435 to determine the method's precision and accuracy [1,6,7,10]. Five calibration points with concentrations of 5, 10, 25, 50, 100 ng/ul were selected for mixture of pesticides and PCB's. Qualitative analyze was based on external standard method.

3. RESULTS AND DISCUSSION

Analyze of organochlorine pesticides, their residues and PCB was realized in water and sediment samples of Devolli River in December 2018. Devolli River passes through Korca Field which is the second agricultural area in Albania. Figure 2 shows total of organochlorine pesticides in water and sediment samples of Devolli River. Pesticides were found in all analyzed samples. This is expectable because they were used in this area for more than 40 years firstly for against malaria vectors and after that for agricultural purposes. Organochlorine pollutants such as pesticides are very stable compounds while they can be found in environment for many years after their application [6,7,9]. The average level of pesticides in water samples was 97.7 ug/l while in sediment samples it was 217.2 ug/kg sediment samples. Presence of pesticides and their degradation products could be related mainly to previous uses of organochlorine pesticides in the Korca field for agricultural purposes. The most polluted stations for water samples were LDW4, LDW5 and LDW6. These stations extend from Proger (start of Korca field) to Maliqi stations (the end of Korca Field). Short-term effect of agricultural processes on Korca Field was shown to be higher in these stations. The most polluted sediment stations was LDS7 which is located at the end of the Korca field due to the concentration of these organic pollutants from the drainage canals of this field (long-term effect). It was observed differences on distribution of pesticides between water and sediments stations (Figure 3).

In water samples were found mainly pesticides that are used recent years or that come from soil leaching while in sediments were found pesticides and their residues from their previous use. These facts reflect on the profile of organochlorine pesticides in water and sediments of Devolli River (Figure 4). Their profile in water samples was: Endosulfan I > Endosulfan II > Endosulfan sulfate > Aldrin > gama-Chlordane while the profile in sediment samples was: DDTs > alpha-Chlordane > Endosulfan sulfate > Endosulfane II > gamma-Chlordane > Endrin. Figure 5 shown the classes of organochlorine pesticides analyzed in this study. Their profile in water samples was: Endosulfane's > Aldrines > Chlordanes > HCHs > Heptachlores > DDTs while the profile in sediment samples was: DDTs > Endosulfane's > Chlordane's > Aldrines > Heptachlores > HCHs. It should be noted that

pesticide profiles (individual and based on their classes) were dominated by the same pesticides in both graphs but with differences in their concentrations between water and sediments. This is related to previous uses of these pesticides in agro-agricultural areas near the Devoll River. Degradation processes and their physical-chemical stability influence in their profile. Also, new arrivals of pesticides and their residues from soil (agricultural areas) through drainage channels finish to the river (water and sediments). Levels of pesticides found in water samples of Devolli River were relatively higher compared to those found in previous studies for other Albanian rivers [3-5].

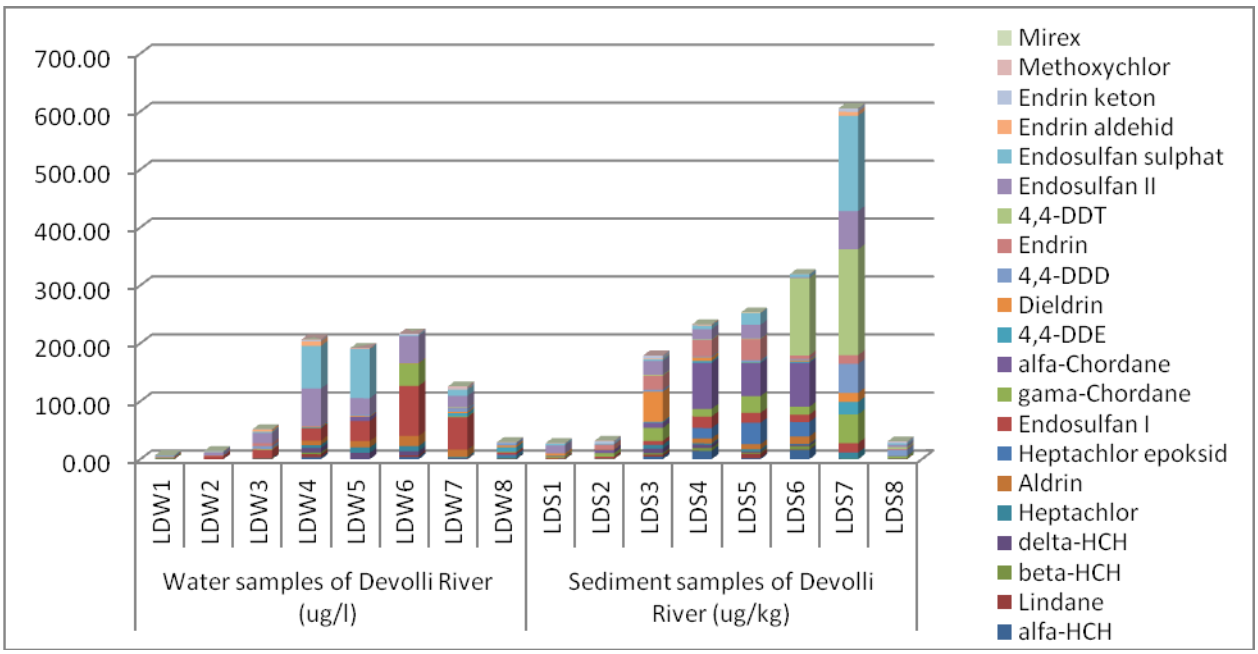


Figure 2. Total of organochlorine pesticides in water and sediment samples of Devolli River

Slika 2. Ukupno organohlornih pesticida u uzorcima vode i sedimenta reke Devolli

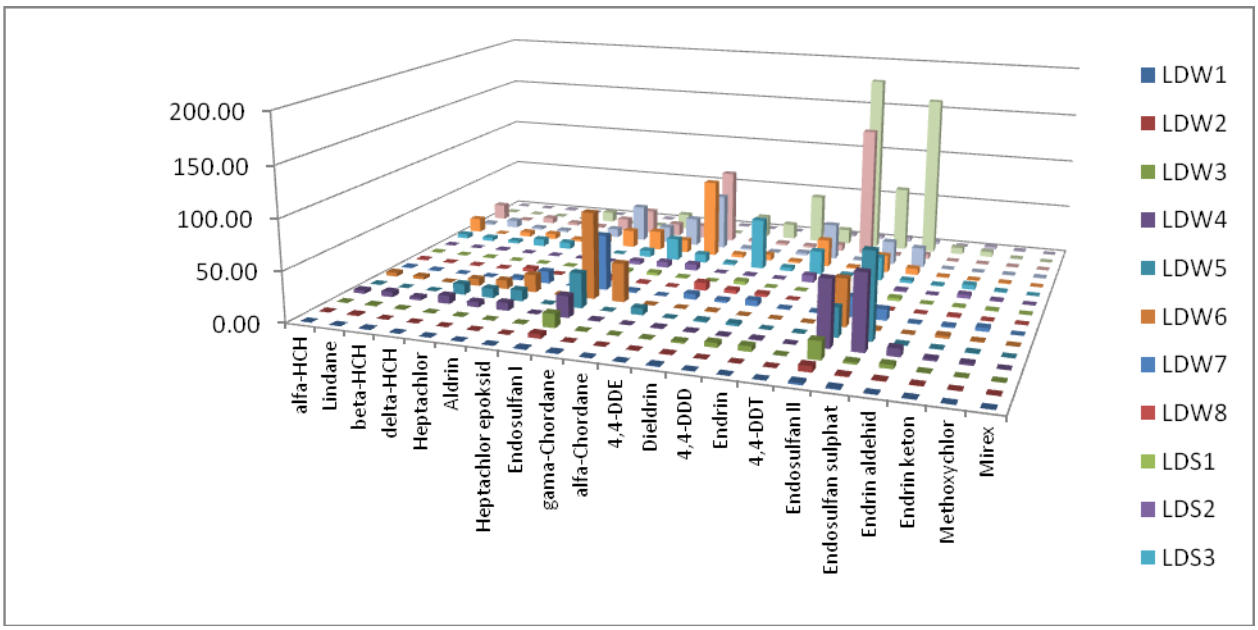


Figure 3. Distribution of organochlorine pesticides in water and sediment samples of Devolli River

Slika 3. Distribucija organohlornih pesticida u uzorcima vode i sedimenta reke Devolli

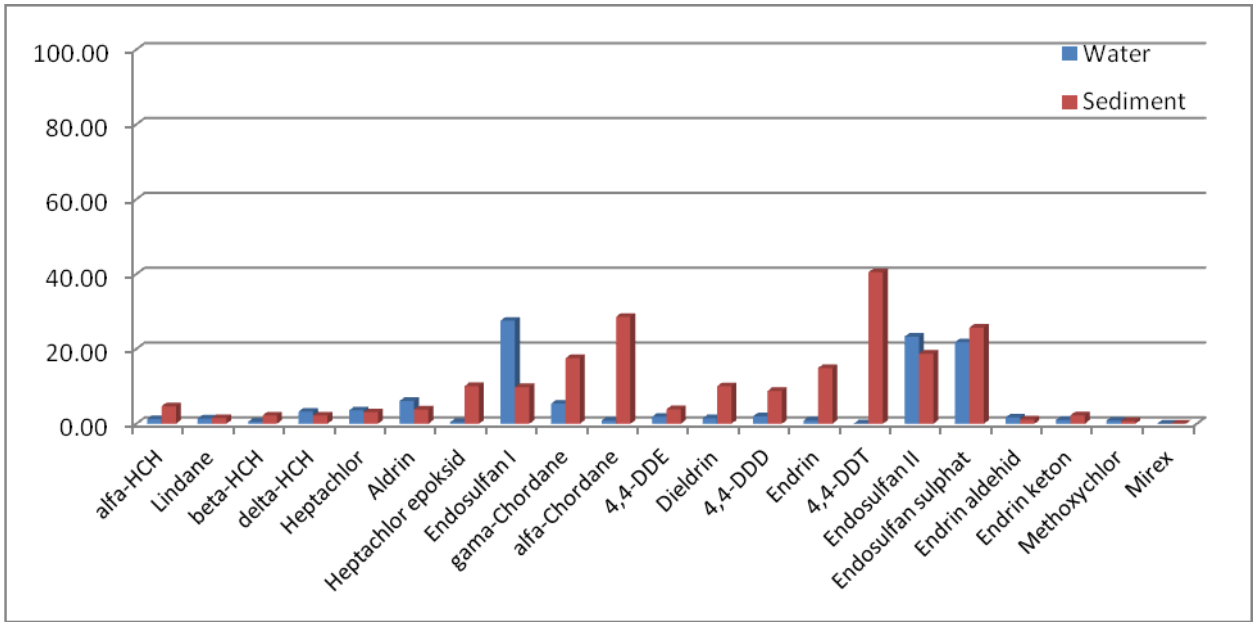


Figure 4. Profile of organochlorine pesticides in water and sediment samples

Slika 4. Profil organohlornih pesticida u uzorcima vode i sedimenta

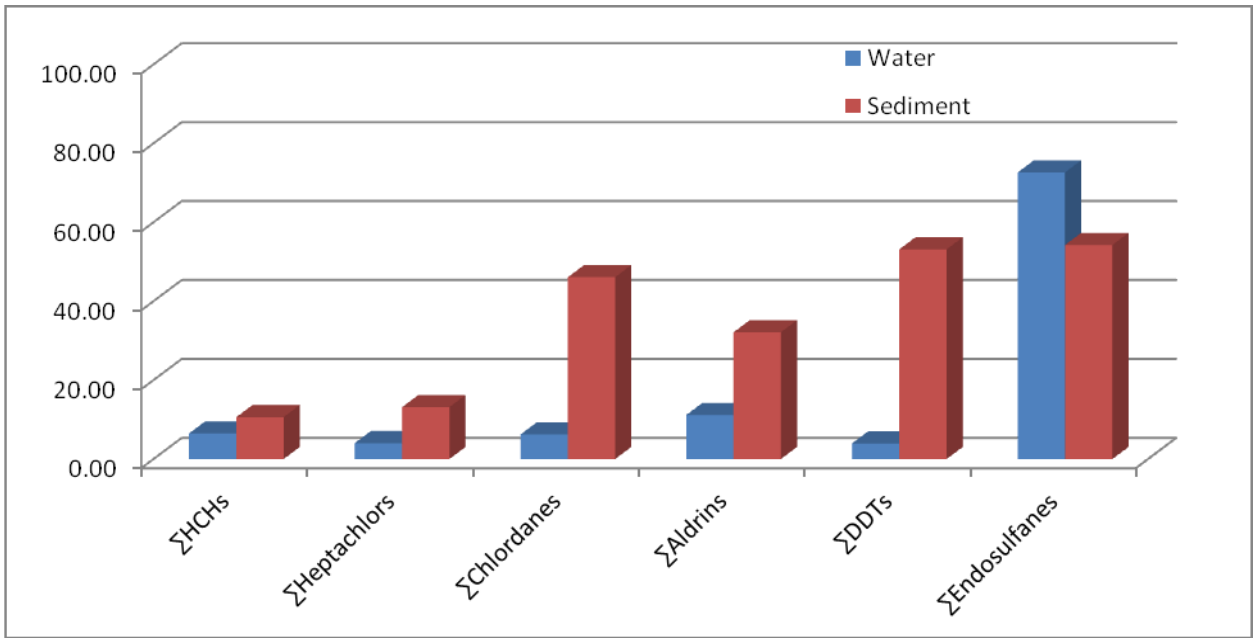


Figure 5. Profile of the main organochlorine pesticide classes in analyzed water and sediment samples

Slika 5. Profil glavnih klasa organohlornih pesticida u analiziranim uzorcima vode i sedimenta

The concentrations of Lindane and its isomers ranged from 1.2 to 18.39 ug/l in water samples and 0.8 - 25.29 ug/kg in sediment samples (Figure 6). There was the same distribution of HCHs for all analyzed samples. HCHs profile for all samples (water and sediments) was: alpha-HCH > delta-HCH > beta-HCH > Lindan. This could be related to the same origin of HCHs in this area. HCHs concentrations could be because of previous uses of Lindane formulations and due to individual physico-chemical properties of each HCH isomer. Total of HCHs were found for all samples lower than permitted level (20 ng/L) for surface waters conform Directive 2008/105/EC.

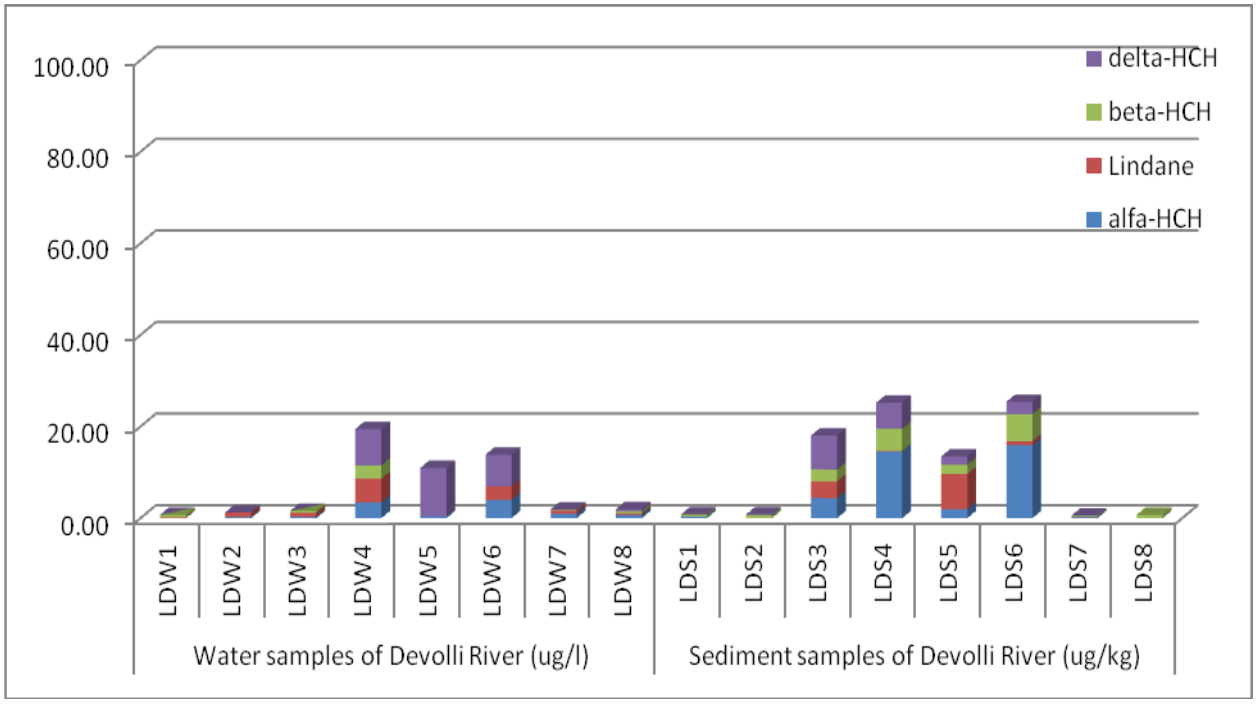


Figure 6. Lindane and its isomers in water and sediment samples of Devolli River

Slika 6. Lindan i njegovi izomeri u uzorcima vode i sedimenta reke Devolli

Total of Heptachlor and its degradation product, Heptachlor epoxide, were from N.D. (not detected or lower than limit of detection – LOD, 0.06 ug/l value for Heptachlor's) to 9.3 ug/l in water samples and 0.2 – 40.5 ug/kg in sediment samples (Figure 7). Their distribution was totally different between water and sediment samples. Heptachlor was found in higher concentrations in water samples while Heptachlor epoxide was found higher in sediment samples. Recently, Heptachlor could be in use near LD4, LD5, LD6 and LD8 stations (possibility of punctual sources). Degradation processes of Heptachlor and its adsorption in sediments are the main reasons for the higher presence of its metabolite. The presence of Heptachlor in sediments is mostly because of its previous use. The level of Heptachlor's did not exceed the permissible norms in both surface waters [11,12] and sediments.

The average level of Chlordane's (alpha and gamma-isomers) was from N.D. values (lower than 0.07 for Chlordane's) to 38.6 ug/l in water samples and between 3.7 – 91.7 ug/kg in sediment samples (Figure 8). Their distribution was the same for all analyzed samples. Alpha-Chlordane and gamma-Chlordane were found at a higher level for all sediment samples compared to their concentrations in water samples. Total of Chlordane's did not exceed the permissible norms in both surface waters [11,12] and sediments except LDW6 sample.

Level of Aldrines in water samples was found between 1.3 to 20.9 ug/l while in sediment samples their total was between 7.8 to 174.22 ug/kg (Figure 9). Their distribution was the different for water and sediment samples. Aldrin was found in water and sediment samples while Endrin and Dieldrin concentrations were found at high levels only in sediment samples. Different profile in water and sediments is related to the Aldrine use in the area (time, quantity, etc.) and its degradation process. The mechanisms of passage of pollutants from water to sediments and vice versa are processes that must be considered in found profile. Aldrines concentrations did not exceed the permissible norms in both surface waters [11,12] and sediments.

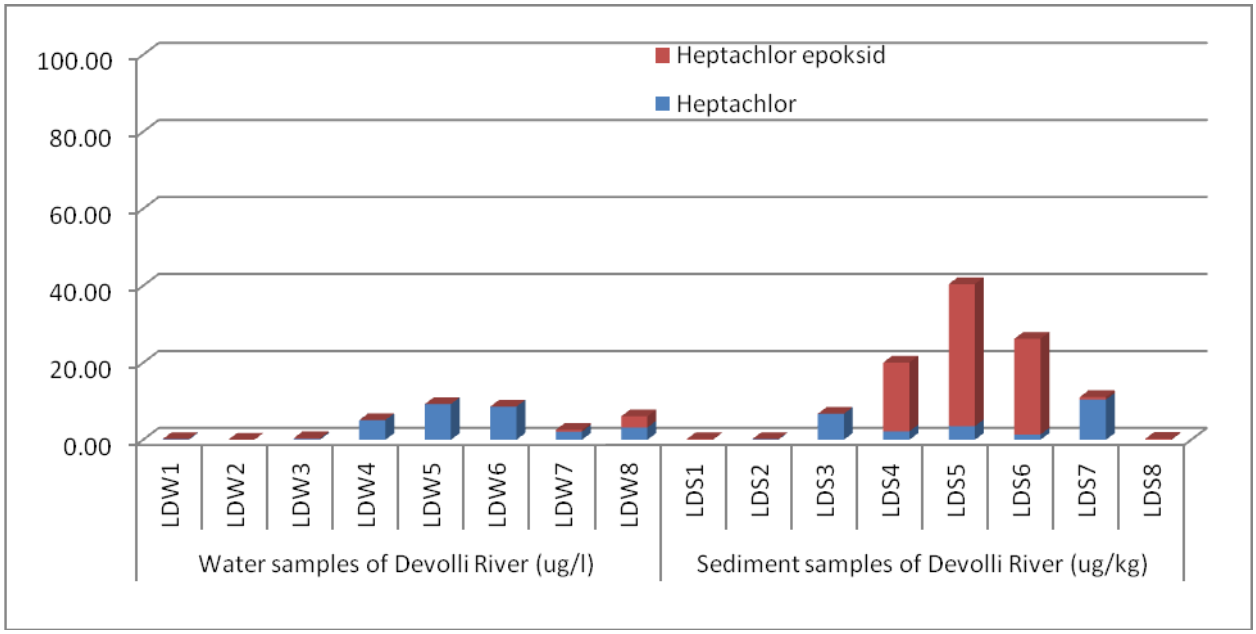


Figure 7. Heptachlor's in water and sediment samples of Devolli River

Slika 7. Heptahlor u uzorcima vode i sedimenta reke Devolli

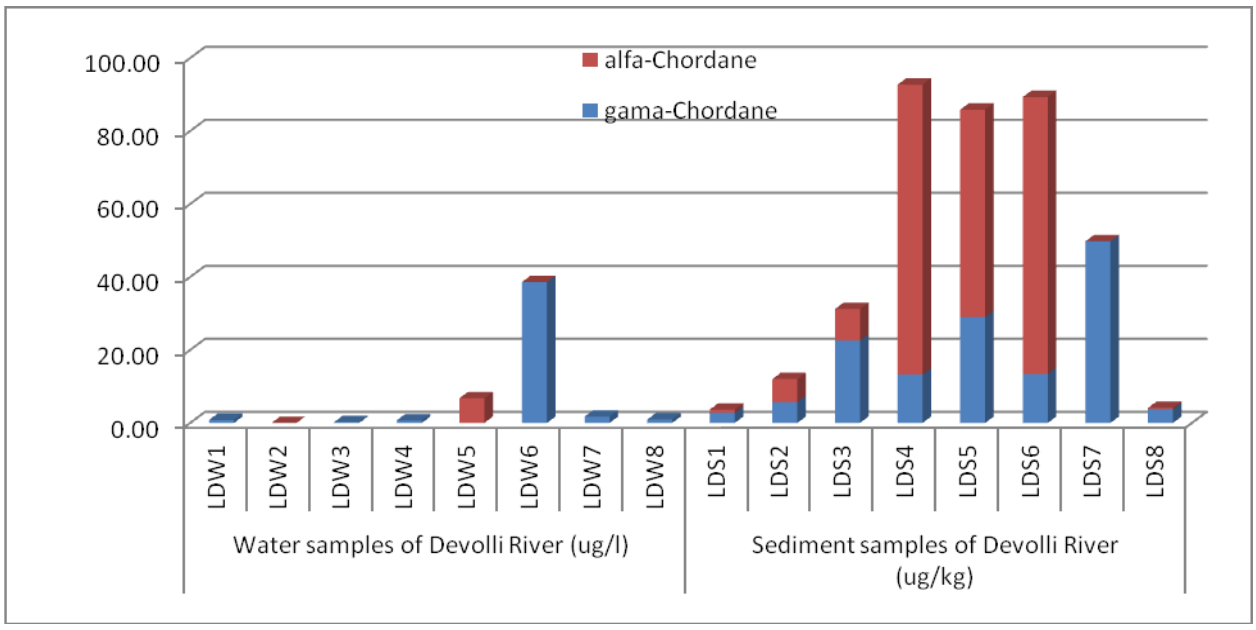


Figure 8. Chlordane's in water and sediment samples of Devolli River

Slika 8. Hlordan u uzorcima vode i sedimenta reke Devoli

Figure 10 shown total of DDTs in water and sediment samples of Devolli River. DDTs were detected for 37.5% of water samples and for 75% of sediment samples. Note that, DDT was found only in two sediments stations (LDS6 and LDS7). Its presence was related to previous uses of DDT in the Korca field and their deposition in the sediments of Devolli River. These stations represent the endpoints of Korca field. DDT was not detected in water samples, which means that it is not in use recently in this area. For water samples were detected degradation products of DDT. Their profile in water samples was: DDE > DDD > DDT while in sediments their profile was DDT > DDD > DDE. This should be related to punctual sources of DDT, its previous uses and DDT degradation processes. Note that,

degradation process of DDT in sediments is much slower than water. DDT concentration was lower than permitted level (10 ng/L) for surface waters conform Directive 2008/105/EC. Only for stations LDS6 and LDS7 the DDT levels exceeded the allowed norms for sediments.

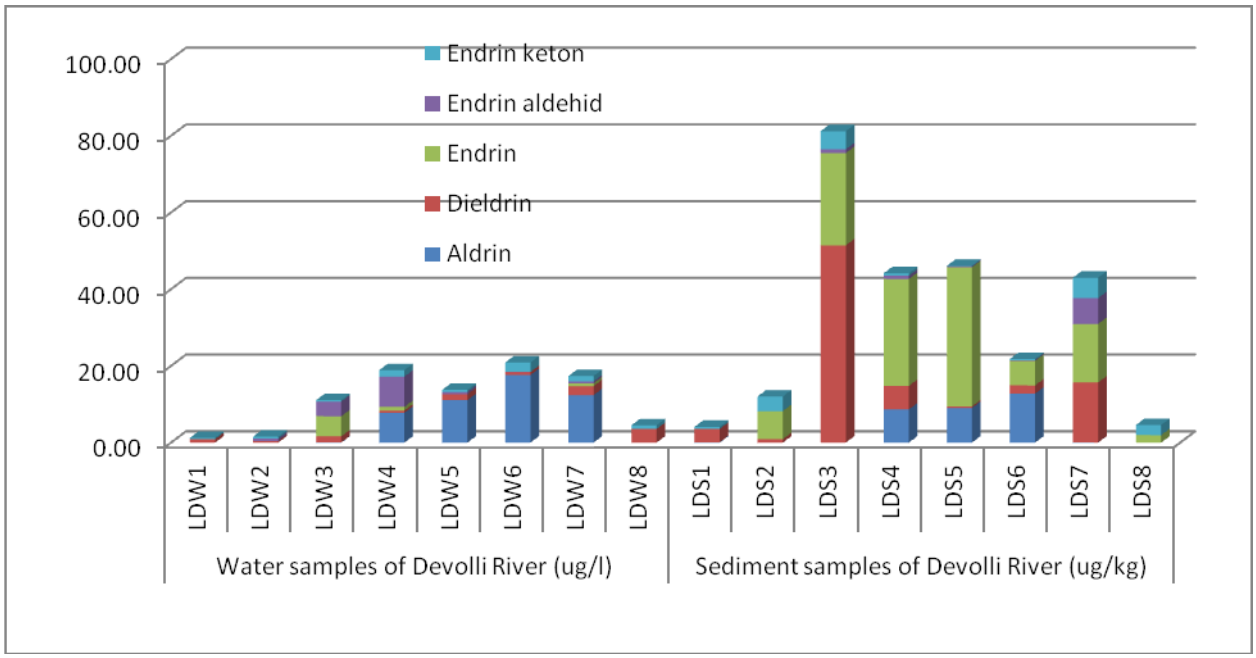


Figure 9. Aldrines in analyzed water and sediment samples of Devolli River

Slika 9. Aldrini u analiziranim uzorcima vode i sedimenta reke Devolli

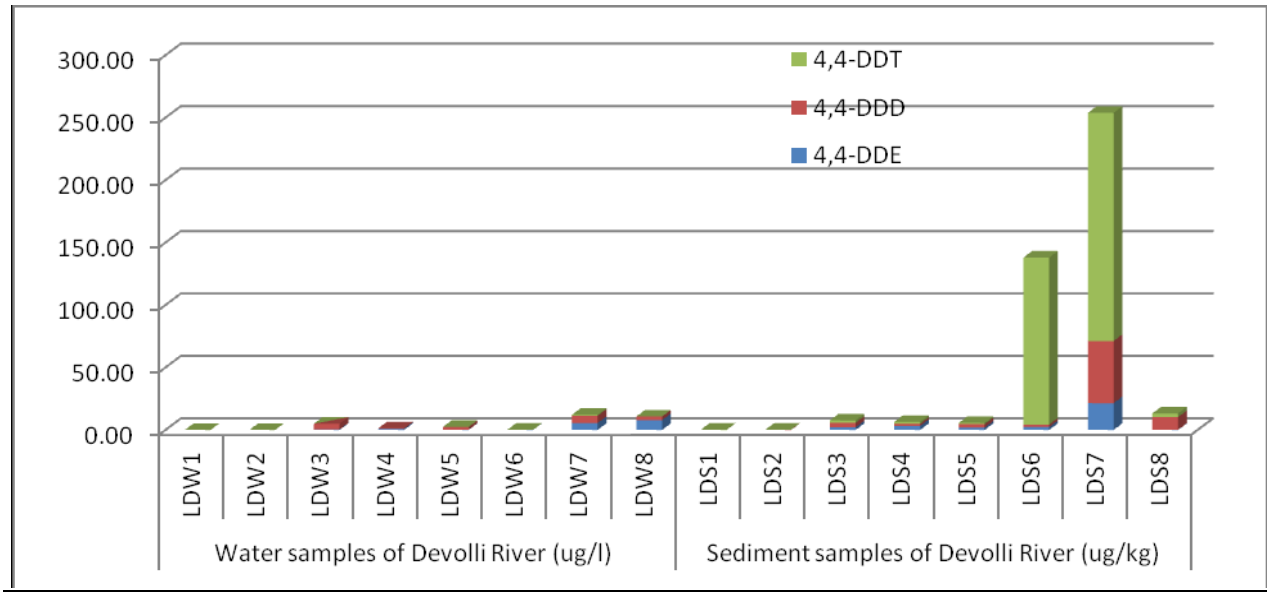


Figure 10. DDTs in water and sediment samples of Devolli River

Slika 10. DDT u uzorcima vode i sedimenta reke Devolli

Endosulfanes were detected in higher concentrations compare to other classes of analyzed pesticides. These pesticides were found in all water and sediment samples of Devolli River (Figure 11). Total of Endosulfanes in water samples ranged from 3.8 to 134.0 ug/l while in sediment samples was in interval from 5.7 to 246.7 ug/kg. Their average in water and sediment samples was respectively 72.0 ug/l and 54.5 ug/kg. were detected in higher concentrations for all analyzed samples. The higher level of Endosulfanes in water samples could be due to

recent uses of this pesticide, mainly as insecticide, in agriculture. The same distribution of these pollutants was observed in water and sediment samples as a result of recent uses and/or new arrivals from water irrigation or a punctual source for this class of pesticides. Total of Endosulfanes, in 75% of water samples, were in higher concentration than permitted level (5 ng/L) for surface waters conform Directive 2008/105/EC.

Average of Methoxychlor in water and sediment samples (Figure 12) was respectively 0.9 ug/l and 0.7 ug/kg. Its concentration was found in high level for two water stations, LDW7 (3.7 ug/l) and LDW4 (1.6 ug/l). Methoxychlor must be in use near these stations or some punctual sources can affect these levels. Its presence in sediments was because of adsorption process. Mirex was detected only in one sediment sample of Devolli River (LDS5) with 0.7 ug/kg (Figure 13). It was not used in Albania but its presence could be because of atmospheric deposition and/or as impurity in other pesticide formulation. Methoxychlor and Mirex concentration was lower than permitted level for all samples.

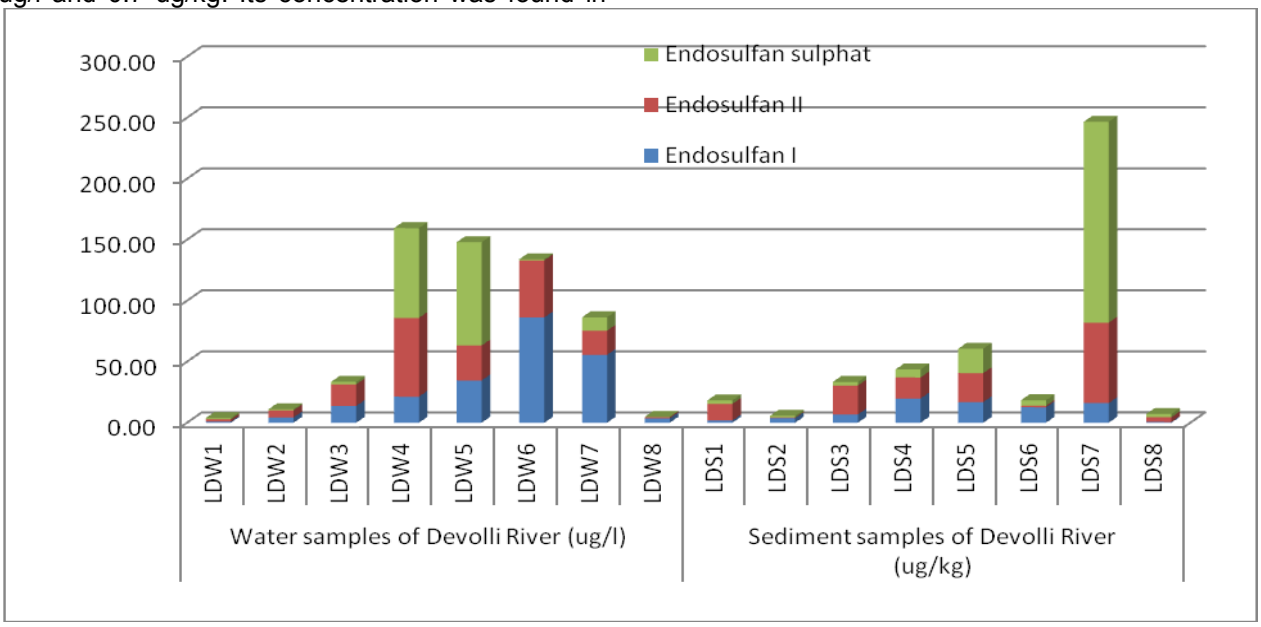


Figure 11. Endosulfanes in water and sediment samples of Devolli River

Slika 11. Endosulfani u uzorcima vode i sedimenta reke Devolli

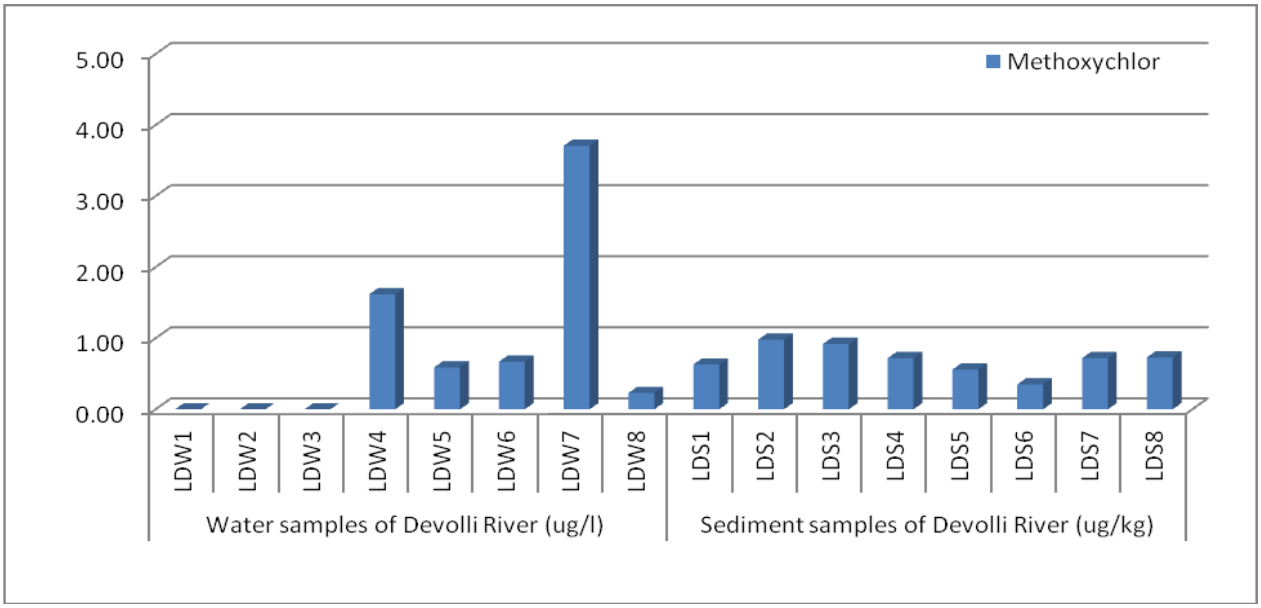


Figure 12. Methoxcychlor in water and sediment samples of Devolli River

Slika 12. Methoxcychlor u uzorcima vode i sedimenta reke Devolli

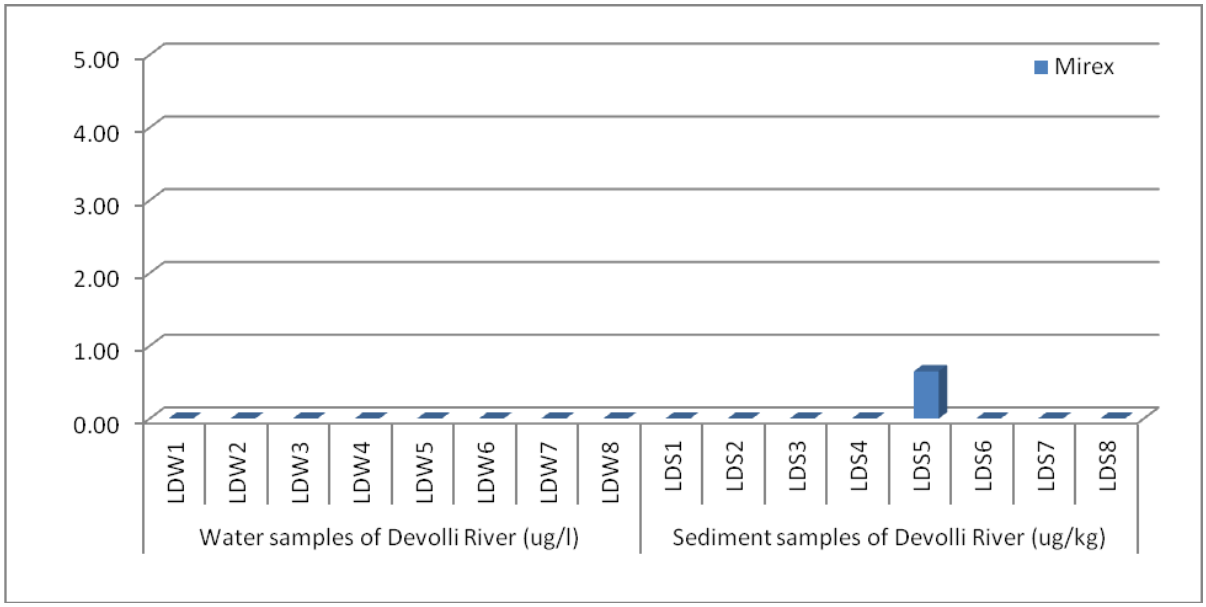


Figure 13. Mirex in water and sediment samples of Devolli River

Slika 13. Mirex u uzorcima vode i sedimenta reke Devolli

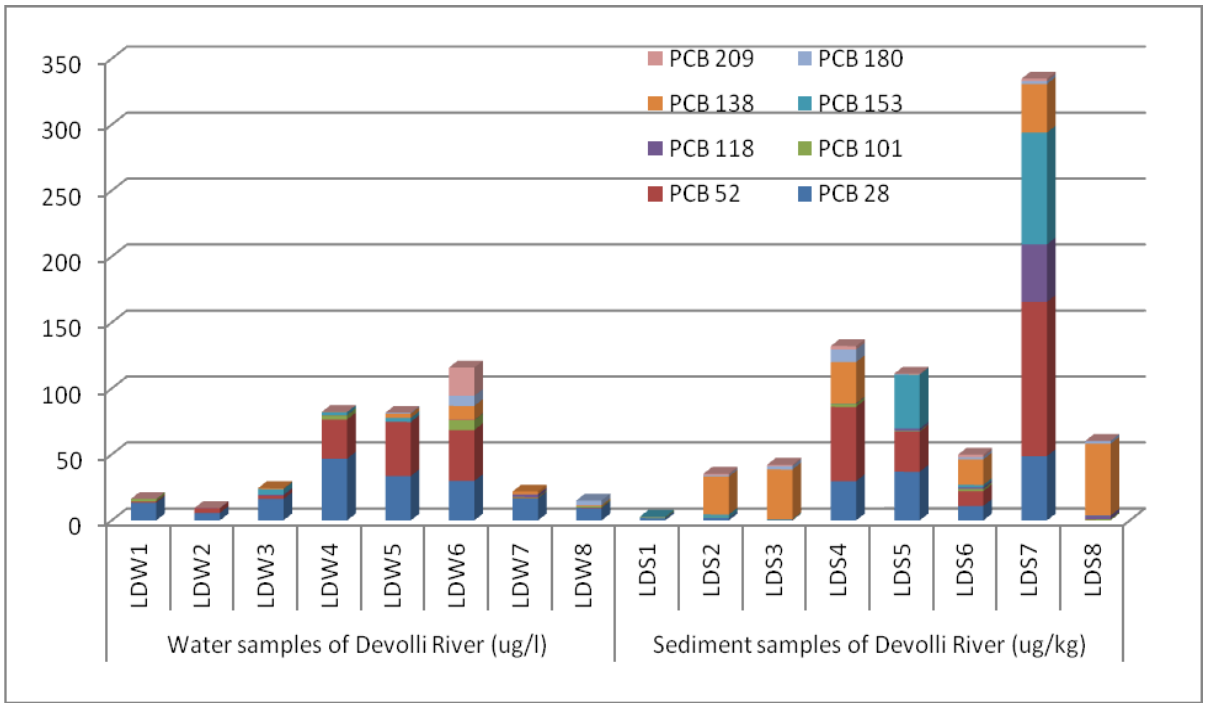


Figure 14. Total of PCBs in water and sediment samples of Devolli River

Slika 14. Ukupan broj PCB-a u uzorcima vode i sedimenta reke Devolli

Figure 14 shown total of PCB markers in water and sediment samples. Also, PCB markers were detected in all analyzed samples of Devolli River. Their average value in the water samples was 41.5 ug/l and in sediment samples was 95.7 ug/kg. The highest PCB levels were found at LD4, LD5, LD6 and LD7 station for both water and sediment samples. This is related to the elevated mechanical, industrial and agricultural activities near these stations and because concentration process in the second part of the river. The highest PCB levels in sediment samples could be connected with deposition time, molecular weight, affinity to absorb with the particles found in water,

punctual sources, etc. The distribution of PCB markers was the same for water and sediment samples of Devolli River (Figure 15). Some individuals were noted to be at higher levels for some stations (PCB 52 and PCB 180). This could be related mostly to the punctual sources of these pollutants. Volatile congeners, PCB 28 and PCB 52, were found abundantly in water samples due to their atmospheric deposition. These congeners have been reported in earlier study in other areas of Albania. Their presence is a consequence of atmospheric deposits, common in our country. Heavy PCBs (PCB 138, PCB 153 and PCB 118) were found frequently in sediment samples. Also, PCB 52 and PCB 28 were detected in the sediment samples. Their presence can be because of any accidental spillage of transformer oils or other equipment that uses PCBs as oils near these stations. Industrial wastewaters near Korca and Maliqi cities can influence the found levels. The level of PCB markers did not exceed the permissible norms in both surface waters [11,12] and sediments based on Albanian and EU norms.

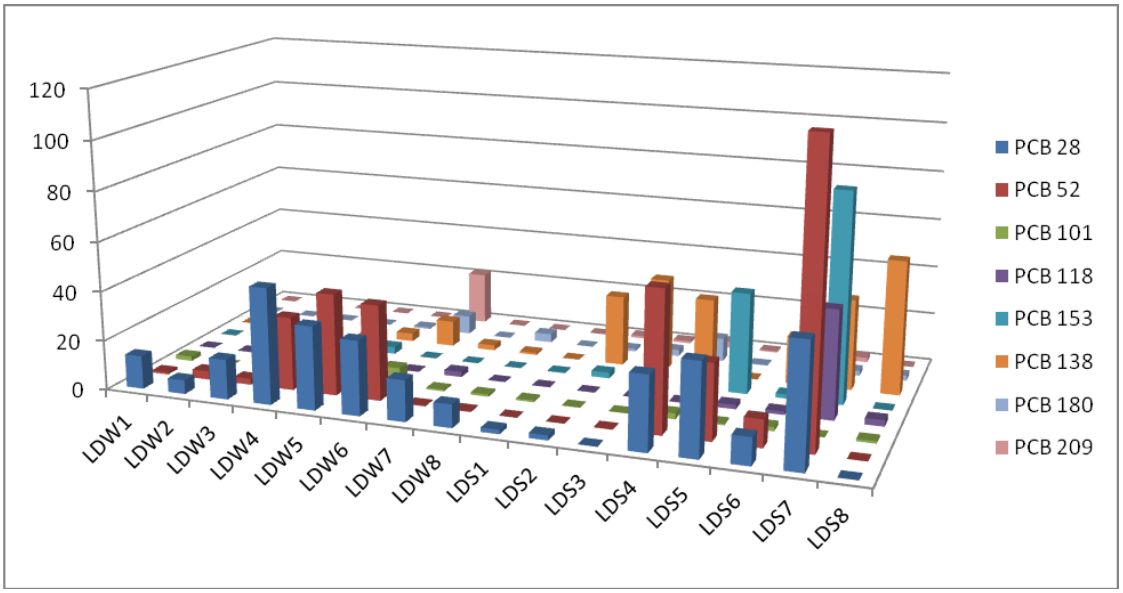


Figure 15. Distribution of PCBs in water and sediment samples of Devolli River

Slika 15. Distribucija PCB-a u uzorcima vode i sedimenta reke Devolli

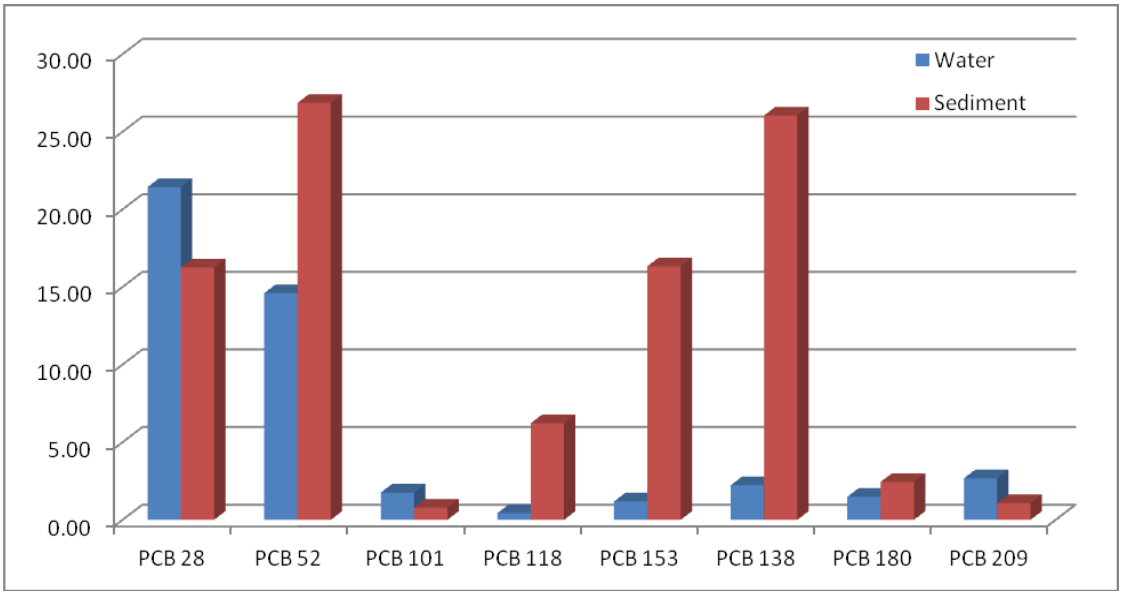


Figure 16. Profile of PCBs in analyzed water and sediment samples of Devolli River

Slika 16. Profil PCB-a u analiziranim uzorcima vode i sedimenta reke Devolli

4. CONCLUSIONS

This study presents first data on concentrations of organochlorine pesticides and polychlorinated biphenyls in water and sediment samples of Devolli River. Pesticides and PCBs were detected in all analyzed samples. Presence of pesticides and their degradation products could be related to previous uses of organochlorine pesticides in the Korca field for agricultural purposes. The most polluted stations were found on the second part of the river because agricultural processes near these stations. The most polluted sediment stations was LDS7 which is located at the end of the Korca field due to the concentration of these organic pollutants from the drainage canals of this field. It was observed difference on the distribution of pesticides and PCBs between water and sediments stations. Presence of pesticides and/or PCBs in water samples shown mainly recent use or new arrivals from soil leaching.

Their presence in sediments is connected mainly to their previous use. Levels of some individual pesticides in surface water samples of Devolli River were found to be higher than permitted levels according to Albania and EU norms. The found levels for organochlorinated pesticides and PCBs in surface waters of Devolli River were comparable/higher than reported levels for other ecosystems in Albania. Responsible authorities should continue to monitor water samples of this river and all area of Korca field.

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IZVOD

NEKE OD KARAKTERISTIČNIH SUPSTANCA U RECI DEVOLI, ALBANIJA

Ova preliminarna studija daje prve podatke o koncentracijama organohlornih pesticida (OCP) i polihlorisanih bifenila (PCB) u uzorcima vode i sedimenata reke Devolli. Nalazi se u jugoistočnoj Albaniji, u polju Korča, koje je druga poljoprivredna oblast u Albaniji. Stanice za uzorkovanje vode reke Devoli odabrane su u osam različitih stanica, počevši od njenog toka (planina Gramozi) do kraja polja Korča, u blizini hidroelektrane Banja (region Gramši). Uzorkovanje vode i nanosa urađeno je na istim stanicama, u decembru 2018. Pesticidi su se u prošlosti na ovom području godinama koristili uglavnom u poljoprivredne svrhe. Takođe, sliv reke Devoli je, takođe, pogođen industrijskim aktivnostima i urbanim zagađenjem. Navodnjavanje vodom i padavine doprinose njihovom transportu iz tla u podzemne i površinske vode. Organohlorni pesticidi i PCB su veoma stabilna jedinjenja koja se mogu naći u životnoj sredini mnogo godina nakon njihove primene.

Ekstrakcija tečnost-tečnost je korišćena za izolovanje OCP i PCB u uzorcima vode, dok je ultrazvučna ekstrakcija korišćena za njihovu ekstrakciju u uzorcima sedimenta. Istovremena analiza organohlornih zagađivača u uzorcima vode obavljena je tehnikom gasne hromatografije korišćenjem detektora za hvatanje elektrona (GC/ECD). Rtk-5 kapilarna kolona (30 m k 0,33 mm k 0,25 um) korišćena je za odvajanje 17 organohlornih pesticida prema EPA 8081A i 7 PCB markera.

Relativno visoke koncentracije organohlornih pesticida su otkrivene u uzorcima vode i sedimenta reke Devoli u poređenju sa drugim rekama Albanije. Njihovo prisustvo može biti posledica njihove prethodne upotrebe u polju Korča. Isparljivi PCB su pronađeni u većoj koncentraciji u uzorcima vode zbog atmosferskog taloženja. Utvrđeno je da su nivoi pojedinih pesticida u uzorcima površinske vode reke Devoli veći od dozvoljenih prema normama Albanije i EU. Nadležni organi treba da vrše kontinuirani monitoring u uzorcima vode ove reke.

Ključne reči: *reka Devolli; Organohlorni pesticidi; PCB markeri; Analiza vode; Analiza sedimenta; GC/ECD*

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