PRIORITY SUBSTANCES IN WATER AND SEDIMENTS OF ALBANIAN PORTS AUREL NURO, BLEDAR MURTAJ, JONIDA TAHIRAJ,

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Abstract

This paper presents the levels of organochlorine pesticides, polychlorinated biphenvls (PCBs) and polycyclic aromatic hydrocarbons (PAHs) in water samples and sediments from the Albanian ports. In this study were considered seven main ports starting from Saranda to Shengjin ports. Water and sediment samples were taken at the same stations (64 stations in total) inside (38 stations) and outside (26 stations) port areas. The samples were taken in May 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-Ims column for PAH isolation. Organic pollutants were found in more than 80% of analyzed samples. The highest levels of contamination were found in sediments (except Saranda stations) compared to the water samples. The different profile of analyzed pollutants in water and sediments could be because of sources of pollution, pollutant properties, new arrivals from rivers (agricultural and industrial activities far from the port areas), momentum values, hydrogeology of ports (depth, water currents), etc. Analysis of organic pollutants in water and sediment samples of port areas should be continuous by authorities.

Key words: Albanian ports, OCP; PCB; PAH; Water analyzes; GC/ECD/FID. Përmbledhje

Ky punim paraget nivelet e pesticideve klororganike, poliklorbifenilet (PCB) dhe hidrokarburet policiklike aromatike (PAH) në mostra uji dhe sedimente nga portet kryesore të Shqipërisë. Në këtë studim janë marrë në shqyrtim shtatë nga portet kryesore duke nisur nga Saranda deri në Shëngjin. Mostrat e ujit dhe sedimenteve janë marrë në të njëjtat stacione (64 stacione në total) në brendësi (38 stacione) dhe jashtë (26 stacione) zonave të porteve. Mostrat u morrën në Maj 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 në më shumë se 80% e mostrave të analizuara. Nivelet më të larta të ndotjes ishin në sedimente (me përjashtim të Sarandës) krahasuar me ato të ujit. Profili i ndryshëm, i ndotësve të analizuar, në ujë dhe sedimente mund të jetë për shkak të burimeve të ndotjes, vetive të ndotësve, prurjet e reja që vijnë nga lumenjtë (aktivitete bujqësore dhe industriale nga zona larg porteve), vlera të momentit, hidrogjeologjia e porteve (thellësia, rrymat detare), etj.. Analiza/monitorimi i ndotësve organikë në mostrat e ujit dhe sedimentit të porteve duhet të jetë i vazhdueshëm nga autoritetet përgjegjëse.

Fjalë kyçe: Portet Shqiptare, OCP; PCB; PAH; Analizat e ujit; GC/ECD/FID.

Introduction

In this study, the pollution of priority substances in the main ports of Albania was considered. Albania faced by the Ionian Sea and the Adriatic Sea in a coastline of 316 km of which about 260 km. The entire coastline of our country has a great diversity including sandy and rocky beaches, lagoons, river estuaries, ports, etc. Since the early times, sailing has been a tradition of the Illyrian and later Arberor tribues that have lived near these seas. Their naval navals were known and used not only for trade but also as important war flotillas for the time. This is confirmed by many writings and remains found in coastal areas of Albania. In our country there are many areas used as ports (large, small) and/or anchoring places for boats/ships/ferries.

The development/systematization of the ports in Albania started after World War II and it is still continuing. The main ports of the country are located in Durres, Vlora, Shengjin and Saranda. With the growth of trade and tourism, these ports are being developed and new ports such as Petrolifera (Vlore), Porto-Romano (Durres), etc. are being built. These ports are usually built in areas of natural bays (such as the port of Vlora), in protected areas by artificial barriers (Port of Durres) or a combinations of natural bays and barriers (Saranda and Shengjin ports). Ports are considered important gates of the country, therefore nowadays there is an increased attention to them (Gjeografi e Shqiperise, 2016).

On the one hand, the geography (geographical position) and hydrogeology of the ports (depth, sea water currents) and on the other hand the intense activity in the port areas (in the marine and terrestrial areas) are the main reasons for the pollution of the port areas more than other coastline areas. This pollution is caused mainly by anthropogenic activity. The pollution sources of port areas include discharges of urban waste (sewage from cities, villages, residential areas, buisnesses and ships), industrial waste from processing (import/export) and their storage, new arrivals from agricultural lands (rainfall), atmospheric deposits, sewage pipelines, etc.

Organic pollutants mainly come from urban waste, industrial/mechanical processes and agricultural activity. 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. Generally, their presence in sediment is much higher because of concentration process. This polluted sediments can cause organic/inorganic polltants presence time to time in the water column. The organic pollutants considered in this study were organochlorinated pesticides (OCPs), polychlorinated biphenyls (PCBs) and polycyclic aromatic hydrocarbons (PAHs) which are classified as priority substances because of their persistence and toxicity (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 64 stations in the seven main ports of Albania starting from Saranda Port in the South to Shengjin port in the North Albania. The sampling stations was as follow: 9 stations at Saranda's port (WSP); 12 stations at Vlore's port (WVP); 8 stations at Petrolifera (WPP); 6 stations at fishing port, Zvernec (WZP); 12 stations at Durres's port (WSP); 9 stations at Porto-Romano (WPR) and 8 stations at Shengjin's port (WSH). The samples were taken in May 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 $+4^{\circ}$ C before their analysis in the laboratory. Sediment samples were taken at the same stations using a Van Veen grape. 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 consider. The sampling stations for Albanian ports was shown in Figure 1.



Figure 1. Sampling stations in Albania's port

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 2×40 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, 15 - 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°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 0.25 μ m) 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 (Guan *et al*, 2018; Li *et al*, 2009; Nuro *et al*, 2014; Borshi *et al*, 2016; Murtaj *et al*, 2014; Mohamed *et al*, 2011)

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, 15-20 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°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 priority substances (organochlorine pesticides, PCBs, and PAHs) in water and sediment samples of Albanian ports starting from Saranda's port to the Shengjin's port. Water and sediment samples were taken in May 2023, in a total of 64 stations. OCPs and PCB markers were analyzed using the GC/ECD technique while PAH and Benzene were quantified by using GC/FID technique. Figure 2 shows the total of organochlorine pesticides in water samples (ppb - ug/l) and sediment samples (ppb - ug/kg) for the Albanian ports. The minimum level of OCPs was for the water in the port of Saranda with 3.0 ppb and Shengjin ports with 4.0 ppb. The maximum level of pesticides was in Porto-Romano with 18.3 ppb and the port of Durrës with 16.3 ppb.

The presence of pesticides in sediment samples was 2-3 times higher than in water samples. This is mainly influenced by the process of sediment formation from the deposition of particles in suspension which make a strong adsorption for these pollutants by the water. The exception was for the sediments of the port of Saranda which, due to their rocky nature, have low level, comparable to that of the water samples. Pesticide levels in water are mainly influenced by momentum values and new arrivals from water currents (in/out port areas) and rivers. Pesticide levels in sediment samples ranged from 3.8 ppb (Saranda port) to 55.6 ppb (Porto-Romano).

The presence of pesticides in port sediments could be related to water currents, new flows from rivers that bring new quantities from agricultural lands even far from them. For some ports, OCP presence may be due to pesticide deposits near the port areas, possible accidents that occurred in these areas or their discharges directly to the sea (Porto-Romano and the port of Durres). Cluster analyzes for organochlorine pesticides in water (Figure 3) and sediment samples (Figure 4) of Albanian ports have a notable similarity between each other.

Saranda and Shengjin port were grouped together for both dendograms (similarity level more than 90%) because of the lower levels of OCPs in water and sediment samples. In the second group the similarity levels for OCPs in water of studied ports was as follows: Petrolifera, fishing port of Zverneci (95.3%), Durres (92.1%), Porto-Romano (78.6%) and Vlore's port (67.8%) while in sediment stations in the second group were Vlora and Zverneci (89%), Petrolifera nad Durres (95%) connected together (79.5%) and after that with Porto-Romano with a similarity level of 77.5%.

The relatively high similarities between the different ports can be related to the level of pollution in water and sediment samples and the same pollution origin. Figure 5 and Figure 6 show respectively, distribution of OCPs in water and sediment samples.

Five groups (clusters) of pesticides and their residues were found in water as follows: the first group was built by a-HCH, b-HCH, d-HCH, Heptachlor epoxide, Aldrine, gamma-Chlordane and Endosulfan (with a similarity level more than 93%); in the second group were Lindane, Methoxychlor, Endrin aldehyde, DDE and DDT (with a similarity level more than 85%); Endrin and DDD were in third group (with around 72% similarity); in fourth group were part Heptachlor, Dieldrin, Endosulfan sulfate and alpha-Chlordane (with more than 70% similarity level) and in the fifth group was Endosulfan II and Mirex (with a similarity level of 63.6%).

Connections between groups were from 50% to 4%. Clusters of OCP distributions in the sediment samples were as follows: in the first cluster were a-HCH, g-Chlordane, Lindane, b-HCH and Heptachlorepoxide with a similarity level more than 95% (OCP individuals and similarity level was almost the same with water); Endrin aldehyde and Endosulfan sulfate were in the second cluster (93.6%); a-Chlordane and Methoxychlor were in third cluster (91.2%); DDE and Mirex were part of fourth cluster (86.6%); the fourth cluster was built by Heptachlor, DDT, DDD, Aldrine, Endosulfan I and Endrin keton with a similarity level more than 80% and in the last group were Dieldrin, Endosulfan II and Endrin (87.4%).

Note that, clusters of pesticides were built based on their respective concentrations in water and sediment samples from Albanian ports. Generally, there are no levels of individual pesticides that exceed the norms allowed in Albanian or international legislation (Directive 2008/105/EC). Also, OCP concentrations were comparable to previous reports for the Adriatic Sea (Nuro at al, 2017; Myrtaj et al 2013).

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Figure 2. Total of organcohlorine pesticides in water and sediments samples from Albanian ports



Figure 3. OCPs in water samples of Albanian ports



Figure 4. OCPs in sediment samples of Albanian ports





Figure 5. Distribution of OCPs in water of Albanian ports

Figure 6. Distribution of OCPs in sediments of Albanian ports

Figure 7 presents the total of PCB markers in water (ppb - ug/l) and sediment samples (ppb - ug/kg) in Albanian ports, May 2023. Their minimum for water samples was in the port of Saranda with 2.0 ppb. The waters of this port are less polluted by organic pollutants due to the stronger water currents of the Ionian Sea. The maximum of PCBs was in the port of Durrës with 15.1 ppb. The presence of PCBs in the sediment samples was in the same level to those of water samples. This is related to recent pollution for these pollutants. PCB levels in sediment samples were from 3.6 ppb (Saranda port) to 16.7 ppb (Durres port).

Their presence may be due to atmospheric deposits, mechanical and industrial activities near port areas, water currents and river flows. Figure 8 shows PCB markers in water samples of Albanian ports. These clusters were almost the same as those found for OCPs in water. Saranda and Shengjin port were part of first group (96.3%) while Vlora port, Petrolifera, fishing port of Zverneci, Porto-Romano and Durres (with a similarity level from 99% to 85%).

Connection between groups were around 65%. The similarity level was relatively high for PCBs in water samples because of the same pollution origin. There were three groups (clusters) of PCBs in analyzed sediments. Saranda and Shengjin ports (65%) in first cluster; fishing port of Zverneci and Porto-Romano (62%) were part of second cluster and in third cluster were Vlora port, Petrolifera and Durres port (96 - 73%).

The connections between groups were with a similarity level from 54 to 8%. The low similarity levels between groups show differences in the source of pollution with PCBs in port sediments. Figure 10 shows the distribution of PCB markers in water samples from Albanian ports while Figure 11 their distribution in sediment samples. The clusters for the distribution of PCBs in water samples were: PCB 52, PCB 118 and PCB 52 (first group of volatile PCBs with similarity level more than 87%); PCB 153 and PCB 153 and PCB 180 (second group of heavy PCBs with similarity level of 94%) and third group built by PCB 101 and PCB 138 (with similarity level of 76%). Connection between groups were from 55% to 11%. Volatile PCBs were the most frequently detected PCBs in water samples due to atmospheric deposition. Heavy PCBs were found more abundantly in sediment samples due to their physico-chemical properties.

Three were clusters of distribution for PCBs in sediment samples as follows: PCB 28 and PCB 53 (first group of PCB volatile was a similarity level of more than 98%), PCB 101 and PCB 153 (second group with a similarity level of around 75%) and in third group were PCB 138, PCB 180 and PCB 118 (heavy congeners with a similarity level from 87% to 61%). Connections between groups were from 52% to 19%. PCB marker levels do not exceed the norms allowed in Albanian or international legislation for all water samples. These concentrations are also comparable to previous reports made earlier for the Adriatic Sea (Nuro *et al*, 2018; Myrtaj *et al*, 2017; Menarini et al 2006).

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Figure 7. Total of PCB markers in water and sediment samples of Albanian ports





Figure 8. PCBs in water of Albanian ports

Figure 9. PCBs in sediment of Albanian ports



Figure 10. Distribution of PCB markers in water samples of Albanian ports



Figure 11. Distribution of PCB markers in sediment samples of Albanian ports

Figure 12 shows the total of PAH in water (ppm - mg/l) and sediment samples (ppm - mg/kg) of Albanian ports. Minimum concentration of PAHs in water samples was for Saranda port with 0.6 ppm. Again, the strong water currents of the Ionian Sea affect the lowest levels of pollutants in the waters of this port.

The maximum of PAHs was in Petrolifera at 2.9 ppm. The PAH values in the sediment were 2-5 times higher than those of the water. PAH levels in sediment samples ranged from 2.0 ppm (Saranda port) to 4.5 ppm (Porto-Romano). The presence of PAH must be mainly due to marine and automobile transport in port areas, accidental/intentional spills of fossil fuels, water currents, river flows, etc. In Porto-Romano and Petrolifera there is an activity of hydrocarbons (deposits of oil by-products from export/import).

This is also supported by the fact of the presence of higher amounts of PAH with a small molecular mass (non-pyrogenic) in both ports. Figure 13 shows PAHs in water samples of Albanian ports. Saranda, Porto-Romano (85%) and Shengjin port were part of first group (60.3%) while Vlora and Durres ports (99%) and after that Petrolifera (94%) and fishing port of Zverneci, (85%) built the second group (cluster).

Connection between groups were around 53%. Could see that similarity level was relatively high for PAHs in water samples. There were three clusters of PAHs in port sediments (Figure 14). Saranda and Shengjin ports (85%) built first cluster; Vlora port, Durres port (96%) and Petrolifera (85%) were part of second cluster and in third cluster were fishing port of Zverneci and Porto-Romano (85%).

The connections between groups were with a similarity level of around 60%. Figure 15 shows the distribution of PAHs in water samples from Albanian ports while Figure 16 their distribution in sediment samples. The clusters of PAHs in water samples were: Acenaphthylene, Fluorene, Pyrene and Benzo[ghi]perylene (first group with a similarity level more than 70%); Benzo[b]fluoranthrene and Benzo[k]fluoranthrene (second group with similarity level of more than 98%); Anthracene and Benzo[a]anthracene (third group with a similarity level of 76%); Chrysene and Perylene (fourth group with a similarity level of more than 93%) and the last group was built by

Indeo[123cd]pyrene, Dibenzo[ab]anthracene (91%) and Phenanthrene (63%). Connection between groups were from 35% to 2%.

Four were clusters of distribution for PAHs in sediment samples as follows: Acenaphthylene, Fluorene, Pyrene and Benzo[k]fluoranthrene (first group with a similarity level of more than 98%), Phenanthrene, Chrysene and Benzo[a]anthracene (second group with a similarity level of around 95%); in third group were Benzo[b]fluoranthrene, Dibenzo[ab]anthracene and Perylene with a similarity level from 93% to 79% and the fourth was built by Anthracene, Benzo[ghi]perylene and Indeo[123cd]pyrene with a similarity level of more than 70%. Connections between groups were from 45% to 27%.

Although there are differences in the distribution of PAHs in water and sediment samples, similarities can also be found between the groups due to the same origin of pollution with these hydrocarbons. In no case are there PAH levels that exceed the norms allowed in Albanian or international legislation (Directive 2008/105/EC). These concentrations are also comparable to reports made earlier for the Albanian part of the Adriatic Sea (Frohner *et al*, 2018; Magi *et al*, 2002; Mandic and vrancic 2017; Marini and Frapiccini 2013).



Figure 12. PAHs in water and sediment samples of Albanian ports



Figure 13. PAH in water samples of Albanian ports



Figure 14. PAH in sediment samples of Albanian ports



Figure 15. Distribution of PAH in water of Albanian ports



Figure 16. Distribution of PAH in sediments of ports

Conclusions

In this study, were evaluated the levels of organochlorine pesticides, PCBs and PAHs in water and sediment samples from Albanian ports. These compounds are part of priority substances list due to their persistence and toxicity. Organochlorine pesticides were detected almost in all analyzed samples. The level of OCP was 2-5 times higher in the sediment samples. This must be related mainly to the previous uses of these compounds, their degradation processes, sedimentation process, and new arrivals from rivers and water currents. PCBs were detected in more than 80% of water and sediment samples.

Also, PCBs were found at higher levels for sediment samples. Their presence must be consequence of atmospheric deposition (volatile PCBs), mechanical activity and water currents (heavy PCBs) could be the main pollution factors. The presence of PAHs must be mainly a consequence of ship transport and punctual sources of these pollutants near the study stations. Cluster analyses shown the similarities between pollutants, their respective levels and the pollution origin for each considered port. The pollution level for pesticides, PCBs and PAHs in Albanian ports were higher/comparable with previous reports for the Adriatic Sea (Borshi *et al*, 2016; Borshi *et al*, 2018; Murtaj *et al*, 2014). The presence of priority substances in the water and sediment samples of ports should be continuous by authorities because of their tendence to be polluted by anthropogenic pollution.

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