

CHARACTERISTICS OF PETROLEUM COMPOUNDS AND THEIR REMOVAL FROM THE AQUATIC ENVIRONMENT

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A b s t r a c t

The aim of the study was to characterize the components of crude oil, with particular emphasis on those that have a toxic effect on organisms, and to describe the sources of pollution and the methodology for determining these compounds in aqueous solutions. In addition, the study presents the legal provisions in the field of petroleum compounds and practical methods for their removal in the event of uncontrolled emission of these compounds into the aquatic environment as well as methods used in the treatment of wastewater loaded with these compounds.

Keywords: water, wastewater, surface water, soil petroleum compounds, BTX, PAH

1. INTRODUCTION

Crude oil, both unprocessed and its products, pose a serious threat in the event of uncontrolled emissions to the aquatic and ground environment. This threat results from the possibility of migration of these compounds, relative stability and bioaccumulation in organisms inhabiting the aquatic or ground environment [1,2]. The crude oil includes paraffin (alkanes), naphthenic (cycloalkanes) and aromatic hydrocarbons, including polycyclic. The crude oil also contains elements such as sulphur, oxygen, nitrogen, iron, silicon, vanadium, sodium and

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nickel as well as organometallic and mineral compounds [2]. From the point of view of threat to the environment, the impact of components on aquatic and soil organisms as well as their presence in the air is important. It has been shown in ecotoxicological studies that some petroleum components such as volatile hydrocarbons benzene, toluene, xylenes (BTX) and polycyclic aromatic hydrocarbons have carcinogenic and mutagenic effects on test organisms [3,4]. However, in the legal regulations regarding the quality of the aquatic environment, the list of compounds which are components of crude oil is limited [5-8]. It is recommended to indicate only benzene, generally petroleum compounds as an index of mineral oil and five compounds classified as polycyclic aromatic hydrocarbons. Qualitative and quantitative identification of these compounds in the environment requires the use of a chromatographic system consisting of a gas chromatograph and a flame ionization detector or mass spectrometer [9,10]. The article presents basic information on the characteristics of petroleum compounds, legal conditions regarding the aquatic environment and analytical methods as well as methods used to remove uncontrolled emissions of these compounds into the aquatic environment, and examples of their removal from wastewater.

2. SOURCES OF PETROLEUM COMPOUNDS IN WATER ENVIRONMENT

As crude oil comes from dead organic matter (plant and animal organisms), the precursors of oil formation are proteins, lipids, lignins and carbohydrates. These components are the building material for phytoplankton, zooplankton, vertebrates and crustaceans. Thus, crude oil has various organic combinations of elements such as carbon, oxygen, hydrogen, nitrogen, sulphur, magnesium, nickel, iron, vanadium and chlorine [1,2]. Petroleum products include fuels such as gasoline or diesel fuels as well as solvents, resins, petrolatum, paraffin and ceresin and also mixtures of naphthenic, paraffinic and aromatic hydrocarbons. The chemical composition includes: saturated hydrocarbons, alkyl-naphthenic aromatic hydrocarbons, aromatic hydrocarbons and heterorganic compounds. These components constitute 97% of the crude oil composition, with the carbon content estimated at 85% [2,10]. Saturated hydrocarbons include branched, straight or cyclic alkanes, alkenes and alkynes. The content of aromatic hydrocarbons in crude oil is 15%, but in higher boiling fractions it can reach 50%. Among these compounds, benzene and toluene, xylenes (BTX) and chlorinated phenols are of great importance. These compounds are distinguished by the fact that they are volatile and have a toxic effect on organisms [11]. Therefore, reducing emissions of these pollutants into

the environment does not only apply to air but also to water and is still current and important. Toxic components of crude oil are compounds belonging to the group of polycyclic aromatic hydrocarbons, and some have carcinogenic, mutagenic and teratogenic effects on test and potential organisms on humans [12,13]. Considering the widespread use of petroleum products in the economy and in transport for combustion, it should be emphasized that both monocyclic and polycyclic hydrocarbons are not completely burned and occur in flue gas. Combustion products are therefore emitted into the atmosphere, from where they migrate to surface water and soil as a result of dry and wet deposition. In addition, the sources of these compounds in the aquatic and ground environment are:

- oil extraction and exploration work,
- transport of crude oil and petroleum products, their storage and distribution,
- processing plants such as refineries and petrochemical plants,
- combustion of crude oil and its products in industrial plants as well as in means of transport and military facilities,
- accidental breakdowns causing uncontrolled emission of petroleum derivatives into waters or into the soil
- treatment of waste containing petroleum products,
- surface runoff containing tire and asphalt abrasion products,
- insufficiently treated municipal and industrial wastewater [4,14-17].

The literature includes many works related to the presence of oil derivatives in the aquatic environment. In relation to surface waters, most of the work concerns seas and oceans, since these waters are most polluted and rainwater [16-19]. The presence of oil derivatives in sea waters is associated not only with the exploration of deposits and the exploitation of oil platforms, but mainly with transport. This is due to the current operation of tankers, transshipments in ports, automatic outflow of crude oil from the seabed as well as uncontrolled leaks in emergency situations and leaks from wrecks lying on the seabed and drainage of bilge water from vessels [18]. The content of PAHs in precipitation wastewater flowing into the separators and treated as well as in the waters of the Słupia River was studied by Siebielska and Szymański [17]. The total concentration of six PAHs (WHO list) in rainwater reached 4.7 mg/L, while the sixteen PAHs (EPA list) - 23.3 mg/L. In waters draining from separators, it did not exceed 20 mg/L. In the waters of the Słupia River, into which purified rainwater is introduced, the concentrations of six PAHs and sixteen compounds did not exceed 5 and 10 mg/L, respectively [17]. In turn, the concentration of petroleum compounds as the concentration of mineral oil index in rainwater was determined by Badowska. The values of this index ranged from 230 to 1,030 µg/L [16]. In the case of introducing oil derivatives into waters, the formation of a flood waters is characteristic, whose range is determined by a number of

factors. These include quantity, density, viscosity, temperature as well as wind time and speed. A 10-7m to 10-5m thick film forms on the water surface. For example, 40 kg of oil can form a slick of 1 km² and a thickness of 0.05µm. The film of petroleum substances hinders gas exchange, mainly oxygen between water and the atmosphere, reduces the photosynthesis process and increases the water temperature due to absorption of solar radiation [18]. Limiting photosynthesis has a destructive effect on plant development. Limiting the access of light and oxygen leads to changes in the functioning of aquatic organisms, and some fish species change their habitat. Petroleum compounds are also accumulated in aquatic organisms, which leads to metabolic disorders. The penetration of petroleum components into organisms that form a link in the food chain is a threat to humans. In addition, oil slicks are dangerous for birds and higher organisms living in the aquatic environment. In the event of coastal water pollution, petroleum compounds may migrate inland, which in turn adversely affects breeding grounds and limits or prevents the use of beaches for recreational purposes. This is important given the toxic effects of both PAHs and BTX on aquatic organisms [20, 21]. When an oil slick remains longer on water surface, petroleum compounds undergo multidirectional physical-chemical and biological changes. The intensity of these changes depends on the type of petroleum compounds, the amount and size of the surface occupied by the slick, and the time and environmental conditions. The intensity of biological changes (biodegradation, bioaccumulation) depends on the presence of an appropriate consortium of microorganisms and environmental conditions [18, 22,23].

3. POLISH LEGISLATION

Petroleum substances are listed in the Regulation of the Minister of Maritime Economy and Inland Navigation on the classification of ecological status, ecological potential and chemical status as well as the method of classifying the status of surface water bodies as well as environmental quality standards for priority substances [8]. Among the group of specific synthetic and non-synthetic pollutants for all river basins, the limit value for petroleum hydrocarbons was determined as the mineral oil index for the first and second class of surface water quality at the level of 0.2 mg/l. However, no limit values have been established for waters in the other classes. The same Regulation sets out environmental quality standards for priority substances set out in the Act of 2017 - Water Law and other pollutants. These quality standards are understood as the concentration of a substance or group of substances in water, fauna or flora, or bottom sediments, which should not be exceeded for reasons of

environmental protection and human health. Among the priority substances given in environmental quality standards are benzene from the group of volatile aromatic hydrocarbons and, benzo(a)pyrene BaP, benzo(b)fluoranthene BbF, benzo(k)fluoranthene BkF, benzo(ghi)perylene BghiP and indeno(123,cd)pyrene IP from the group of polycyclic aromatic hydrocarbons - Table 1 [8]. However, the average annual and maximum values were given only for benzo(a)pyrene, considering this relationship as an indicator of the occurrence of other PAHs and therefore.

Table 1. Environmental quality standards for selected priority substances, $\mu\text{g/L}$ [8]

Indicator	Average annual concentration in water		Maximum concentration allowed in water	
	Natural watercourses and lakes	Transitional and coastal waters	Natural watercourses and lakes	Transitional and coastal waters
Benzene	10	8	50	50
BaP	$1.7 \cdot 10^{-4}$	$1.7 \cdot 10^{-4}$	0.27	0.027
BbF	*	*	0.017	0.017
BkF	*	*	0.017	0.017
BghiP	*	*	$8.27 \cdot 10^{-3}$	$8.27 \cdot 10^{-3}$
IP	*	*	-	-

For benzene, the average annual concentration is 10 and 8 $\mu\text{g/L}$ for natural watercourses and lakes as well as for transitional or coastal. The maximum permissible concentration of this compound regardless of the type of water was set at 50 $\mu\text{g/L}$. Of the PAHs, the average annual and maximum concentration and content in organisms were determined only for BaP. And so the average annual BaP concentration is $1.7 \cdot 10^{-4} \mu\text{g/L}$, and the maximum allowable - 0.27 and 0.027 $\mu\text{g/L}$ for natural watercourses and lakes, as well as transitional or coastal streams, respectively. For the remaining three compounds from the PAH group, only maximum concentrations are given at the same level for natural watercourses, lakes and transitional or coastal waters (Table 1). It should be emphasized that no indene(1,2,3-cd)pyrene limits were given, which is of great importance considering the comparable toxicity of this compound with BaP [8]. Regarding groundwater, there is a regulation on the criteria and how to assess the status of groundwater bodies [7]. It lists petroleum hydrocarbons as an index of mineral oil, benzene, BTX (volatile aromatic hydrocarbons) as well as benzo(a)pyrene and 5 other PAHs (BbF, BkF, BghiP, DahA, IP). Table 2 presents the limit values of these indicators for the hydrogeochemical background and five classes of groundwater purity [7].

Table 2. Limits for petroleum derivatives in groundwater µg/L [7]

Indicator	Hydrogeochemical background	Limit values for classes				
		I	II	III	IV	V
Petroleum hydrocarbons	0	10	100	300	5,000	Over 5,000
Benzene	0	1	5	10	100	Over 100
BTX	0	5	30	100	100	Over 100
BaP	0.001 – 0.01	0.01	0.02	0.03	0.05	Over 0.05
PAHs	0.001 – 0.1	0.1	0.2	0.3	0.5	Over 0.5

In the scope of sewage quality, the ordinance of the minister of maritime economy and inland navigation regarding substances particularly harmful to the aquatic environment and the conditions to be met when introducing sewage and rainwater or snowmelt into the environment is currently in force [6]. Persistent mineral oils and petroleum hydrocarbons are listed in the list of substances particularly harmful to the aquatic environment, causing water pollution, which should be eliminated, but no limit values for these compounds are given in this document. With regard to waste water, the legal provisions only regulate the permissible values for some pollution indicators in industrial waste water discharged into sewage systems. And so in the announcement of the minister of infrastructure and construction regarding the obligations of industrial wastewater suppliers, the limit values for petroleum hydrocarbons are listed at 15 mg/L, and volatile aromatic hydrocarbons BTX at 1 mg/L. PAHs are also mentioned, but without specifying individual compounds, specifying the limit value per carbon 0.2 mgC/L [5].

4. ANALYTICAL METHODS

Petroleum compounds determined as the index of mineral oil in aqueous solutions are carried out using gas chromatography after previous solvent extraction [9]. The recommended detector for quantitative determination is the flame ionization detector (FID). This applies to hydrocarbons that can be extracted with an organic solvent with a boiling point between 36 and 69°C and do not adsorb on Florisil (prepared diatomaceous earth). The retention time of these compounds assumes values between the retention time of C₁₀H₂₂ n-decane and the retention time of C₄₀H₈₂ n-tetracontane. Therefore, this method can be used to determine the content of aliphatic (long or branched chain), alicyclic and

aromatic hydrocarbons (including those with alkyl substituents). Low polarity compounds such as chlorinated hydrocarbons and polar compounds (in high concentrations) as well as surfactants interfere with the determination. This methodology is recommended for determining the mineral oil index in surface waters and wastewater. The mineral oil concentration is calculated relative to the external standard containing the specified two mineral oils [9]. For example, the use of the above method was described by Poluszyńska and colleagues [24]. The concentration of mineral and lubricating oils was determined. N-decane and n-tetracontane solutions were used to determine the retention times of the compounds tested. Hexane and methanol were used for extraction and the process was carried out by hand shaking and in a water bath. The extract was dried with anhydrous sodium sulphate, concentrated under a stream of nitrogen, and then chromatographed on a GC-FID system. In order to verify the adopted procedure, recovery values were determined, which depended on the initial concentration and extraction temperature. Recovery values ranged from 21 to 84% [24]. On the other hand, Badowska and Grylik checked the impact of the acidification of the sample on the mineral oil index value at the stage of its preparation for analysis and its purification on Florisil [25]. The results of these tests showed that the acidification of the sample has an impact on the result of the analysis, despite the fact that the standard has freedom in its use. The results of these tests showed that the acidification of the sample has an impact on the result of the analysis, despite the fact that the standard has freedom in its use. Despite this, the recovery value was at the level of 83%, i.e. it was within the recommended range (80 – 100%) [25]. Benzene and BTX hydrocarbons are determined using gas chromatography with a GC-MS mass spectrometer or gas chromatography using a leaching and trapping technique as well as thermal desorption GC-MS P&T in accordance with PN-EN ISO 15680 [26]. The procedure for determining PAHs such as naphthalene, fluoranthene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(ghi)perylene and indene(123cd)pyrene is given in PN-EN ISO 17993. It concerns high performance liquid chromatography HPLC. In literature, the methodology most commonly described for the qualitative and quantitative identification of PAHs is the GC-MS or UPLC system.

5. REMOVAL OF PETROLEUM COMPOUNDS FROM AQUATIC ENVIRONMENT

In the event of an uncontrolled spill of petroleum substances, the following methods are used to remove petroleum compounds from the water surface: collection with adsorption, dispersion, combustion and dumping. The safest method of neutralizing spills is collection. For this purpose, rigid or flexible oil

booms and oil collectors are used. Collectors can be sorbent, adhesive or an overflow-pump. The final stage is the removal of petroleum residues using sorption barriers or organic, mineral and mixed-type sorption materials. Natural or synthetic sorbents are used. Natural sorbents include peat, straw, wood shavings, sawdust, bark, charcoal, brown shell, nut shells or diatomaceous earth or clay minerals, and sorbents (e.g. composts) from waste. Synthetic, on the other hand, are materials made of polyvinyl chloride or polystyrene [27-30]. Sorbents should have the following characteristics:

- good sorption capacity for petroleum derivatives
- adequate porosity ensuring the penetration of petroleum derivatives and their retention
- possibility of regeneration by recovery of collected substances or utilization of used material
- hydrophobicity and specific gravity preventing wind drift.

Dispersion involves the use of surfactants and agitation of water. Dispersants, reducing the surface tension, create microdroplets facilitating the access of light and oxygen to water. This enables the development of microorganisms capable of decomposing petroleum compounds, which indirectly accelerates biodegradation [27, 31]. Therefore, the scattering method involving the addition of SPCs that cause a change in interfacial tension may stimulate the biodegradation processes of petroleum compounds. It has been shown that the rate of biochemical changes can increase by 10 to 100 times under these conditions [28, 31]. However, the use of dispersants may increase the toxicity to aquatic organisms, as surfactants have a beneficial effect on the dissolution of various sparingly soluble and toxic compounds such as aromatic hydrocarbons and their halogenated derivatives. In this form, these compounds become more easily available to organisms. Therefore, the use of this method is limited to areas that are difficult to access, away from water intakes or places where it is not possible to use sorbents [31,32].

Removal of petroleum compounds from wastewater is carried out using physical, chemical and biological methods. Physical include flotation, stripping, coalescence, sorption and membrane methods, while chemical methods include coagulation and electrocoagulation as well as advanced oxidation [33-37]. These processes are usually used in hybrid systems that are a combination of unit processes. Biodegradation of petroleum compounds by microorganisms capable of metabolic or cometabolic transformations is also possible. Examples of studies on the removal of petroleum compounds from aqueous solutions using integrated coagulation with reverse osmosis were conducted by Piekutin and co-authors [31, 33]. The removal efficiency of BTX compounds such as toluene, ethylbenzene, xylenes (m+p xylene, o-xylene) was determined. A decrease in

the mineral oil index in this process was also determined. The use of aluminium sulphate as a coagulant and RO allowed the removal of test compounds in the range of 75-81%. The combination of reverse smear with stripping allowed to remove petroleum compounds in 80% [33,35]. In other studies where aeration and reverse osmosis were combined, the removal of C7-C35 hydrocarbons designated as mineral oil index reached 99% [34]. Sample research on changes in the content of aromatic hydrocarbons (BTX) in municipal wastewater during purification was conducted by Mrowiec [4]. Adsorption on activated sludge flocs has been shown to be significant in removing BTX in a treatment plant. At the same time, the inhibitory effect of these compounds on the nitrification process was confirmed. In the anaerobic process, the removal efficiency of BTX compounds ranged from 38 to 86%. Variable anaerobic conditions have contributed to increasing the removal efficiency of these compounds by 99% [4]. Biodegradation studies of petroleum compounds in the marine environment have shown that the percentage degradation of toluene ranged from 23 to 100% of initial values after 71 days of incubation. The degree of PAH degradation varied from 66 to 100% depending on the properties and decreased with the increase in the number of aromatic rings [23, 32, 38-40].

6. CONCLUSION

Petroleum substances are a mixture of many hydrocarbons of various structures, some of which are toxic to organisms. In the case of pollution of the aquatic environment, these compounds can penetrate into aquatic organisms and adversely affect gas exchange and inhibit photosynthesis. Therefore, it is important to take measures to reduce emissions of these compounds into the aquatic environment (wastewater treatment) and to quickly remove any spills in the event of uncontrolled pollution of the aquatic environment. In the second case, the basic action is to limit the spread of oil derivatives on the water surface through sorption and dispersion. Sorbents play a significant role here, as their sorption capacity and regeneration (oil recovery) as well as availability are considered the most important application criteria. In the case of wastewater loaded with petroleum compounds, only deepened oxidation methods allow their relatively quick degradation. Biodegradation requires appropriate consortia of microorganisms and stimulating conditions and is long-lasting, while the physico-chemical methods only allow the separation of these impurities, which creates further problems with their disposal.

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