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Adsorption of selected 3- and 4-ring polycyclic aromatic hydrocarbons on polyester microfibers – preliminary studies

Ewa Wiśniowska*, Maria Włodarczyk-Makuła

Department of Sanitary Networks and Installations, Water and Wastewater Treatment, Czestochowa University of Technology, 69 Dąbrowskiego Str., 42-200 Częstochowa, Poland, emails: ewisniowska@is.pcz.czest.pl (E. Wiśniowska), mwm@is.pcz.czest.pl (M. Włodarczyk-Makuła)

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ABSTRACT

This paper presents the results of investigations on the sorption capacity of selected 3- and 4-ring polycyclic aromatic hydrocarbons (PAHs) on polyester microfibers. Adsorption of organic micropollutants such as PAHs is not a well-recognized environmental problem. Some researchers indicate that concentrations of the micropollutants adsorbed on microplastic particles can reach even several mg per one kg. The detailed studies in this problem are however missing. Recent research works indicate that microfibers are commonly present in wastewater and mainly come from cloth washing. The adsorption capacity of the fibers should be analyzed to evaluate the risk connected with their environmental fates. The study aimed to evaluate the sorption capacities of selected 3- and 4-ring PAHs on polyester microfibers. Adsorption of PAHs was provided under static conditions. Adsorption capacities of PAHs on polyester microfibers varied from 24.8 μg g^{-1} (acenaphthene) to 1,424.7 μg g^{-1} (acenaphthylene). Removal efficiencies from wastewater were in the range of 70%–100%. For most PAHs, no desorption has been observed during the 24 h experiment. The total capacity of polyester microfibers towards analyzed 3- and 4-ring PAHs was 3,833.6 μg g^{-1}. The results have confirmed that polyester fibers show high adsorption capacity towards PAHs. Further detailed studies on sorption mechanisms and toxicity of the fibers are necessary.

Keywords: Polycyclic aromatic hydrocarbons; Microplastics; Adsorption; Coke plant wastewater; Polyester microfibers

1. Introduction

Microplastics are common pollutants for several decades. They are defined as small plastic particles in sizes within the wide range from 1×10^{-9}–1×10^{-8} m to 5×10^{-3} m. Plastics of sizes smaller than 1×10^{-9}–1×10^{-8} m are called nanoplastics [1]. This definition meets the recommendation of the European Commission, in which nanomaterials are considered to be that in at least one dimension is within the mentioned previously range, or in the particle size distribution, at least 50% of the particles are on the nanometric scale [2]. The smallest microplastics particles are not visible with the nonaided eye, but the largest can be observed and counted without any specialist equipment [1].

Based on literature data it can be stated that the most frequently analyzed fractions of microplastics are those in the range of 0.303×10^{-3} to 5×10^{-3} m. Based on the shape various classes of microplastics can be distinguished including fibers, fragments, foils, pellets, flakes, granules, etc. [1]. Among them, fibers are one of the most abundant in wastewater. They originate mainly from washing synthetic textiles such as polyester, acryl, polyamide (nylon), acetate, and polyparaphenylene terephtalamide [3]. Research studies have shown that under real household conditions an average emission rate of synthetic fibers from the 6 kg load of synthetic textiles could be even 18,000,000 fibers. What was interesting only 7% of them were the ones larger than 0.5×10^{-3} m, the smallest fraction

* Corresponding author.

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(0.05×10^{-3}–0.1×10^{-3} m) comprised more than 59% of the fibers load [4]. The more detailed data were given by De Falco et al. [5] who have stated that one kg of washed synthetic fabric can release up to 300 mg of microfibers to the wastewater, which is equivalent to even 1,500,000 microfibers. The authors have also stated that the data on microfiber concentration in wastewater may be affected by the presence of cellulose fibers, and a blend of polyester/cellulose fibers can be present. Loads of microfibers discharged from washing vary because of many factors affect this process, such as garment age, type of washing machine, detergent use [6].

A report given by HELCOM [7] confirmed high concentrations of microplastics in influents, with concentrations up to 467,000/m^3 of raw wastewater. These data are comparable to the ones given by other authors, who stated fibers concentration in raw wastewater in the range of more than 200,000 fibers m^{-3} (Table 1).

In a wastewater treatment plant, the microfibers are removed from sewage and tend to accumulate in sludge. Various removal efficiencies are reported by researchers. According to some data, preliminary, secondary, and tertiary treatment can remove up to 99% of microfibers present in influent [9]. Other authors report lower removal efficiencies, in the range of 90% [10,11]. In some cases, the removal efficiency was only about 60% [12]. The efficiency of microfibers removal from wastewater depends on many factors, including, for example, dose and type of coagulating agent, polyelectrolyte, the composition of sludge, the concentration of grease, type of polymer of which the microfibers are manufactured [13]. Detailed studies determining the effect of cleaning processes on the efficiency of microfiber removal have not been conducted since now. It is however obvious that by using such tertiary treatment methods as filtration or membrane processes efficiency of microfibers removal can be increased [11].

Most research studies on microfibers focus on their concentrations, loads, and removal efficiencies. Until now, less attention has been paid to the potential role of microfibers as adsorbents cumulating other micropollutants such as, for example, polycyclic aromatic hydrocarbons (PAHs). These micropollutants tend to adsorb on solids, for example, sewage sludge. The affinity of chemical compounds to solids can be generally characterized by partition coefficient – $\log K_{ow}$. PAHs of $\log K_{ow} > 7$ are expected to strongly adsorb to solids, whereas the ones with $\log K_{ow} < 4$ are well water-soluble. PAHs of $\log K_{ow} > 7$ should theoretically tend to adsorb also on microfibers [14,15].

Table 1
Concentration of microfibers in raw wastewater according to the data given by various researchers

Concentration of microfibers in raw wastewater (fibers m^{-3})	Percent share of microfibers in raw wastewater	Reference
467,000	–	[7]
495,000	62%	[8]
200,000	54.5%	[8]

The ones with $\log K_{ow}$ lower than 7 can interact differently. It should be however emphasized that despite the fact that many authors use partition coefficient to predict PAHs behavior towards solids, $\log K_{ow}$ is not directly related to adsorption in chemistry. This coefficient is precisely used to describe the ratio of concentrations of substances in two immiscible solvents at equilibrium. One of these solvents typically is water, and the second one is hydrophobic n-octanol. Because of this $\log K_{ow}$ is really measured for the non-ionized form of the substance under known pH and it is related to substance lipophilicity. This coefficient can be only used as an indicative parameter of adsorption behavior. Adsorption of PAHs on microfibers can be additionally affected by various pollutants present in wastewater, such as surfactants, detergents [14,15].

Previous studies have shown that PAHs are well adsorbable on activated carbon (both granular and powdered), nano-silica, and other adsorbents [16,17]. The effectiveness of PAHs removal from wastewater by sorption process depended on the kind of sorbent, initial concentration of PAHs, and technical parameters. For individual PAH compounds, the effectiveness can vary in a wide range from 10 to more than 90% [18–22]. Research studies concerning the sorption of PAHs on microplastics are rarely undertaken and are mainly related to the adsorption of these compounds by microplastics present in natural waters. No research studies on adsorption of PAHs present in wastewater by defined type of microplastics were undertaken since now.

The investigation aimed to monitor 3- and 4-ring PAHs concentration during adsorption processes on the polystyrene fibers. Percent removal of PAHs from wastewater, sorption kinetic parameters, and sorption capacity of the microplastic fibers have been evaluated.

2. Materials and methods

2.1. Materials

The experiments were carried out using biologically treated coke-plant wastewater. Effluents were taken from the coke wastewater treatment plant. They were effluents after biological treatment with activated sludge (solids retention time > 10 d, conventional organic loading). The wastewater samples were taken once during the experiment.

2.2. Experimental procedure

In raw wastewater samples, initial PAHs concentration was determined. After that 300 mL of wastewater was poured into glass conical flasks of 500 mL volume. 0.5 g L^{-1} of polyester microfibers were added, the microfibers have been obtained from polar cloth via cutting. The experiment was carried out at a constant temperature of 20°C. pH of wastewater was on average equal to 7.2. The samples were placed on the shaker with a horizontal motion for 24 h. Concentrations of 3- and 4-ring PAHs in wastewater were determined threefold after 1, 2, 16, 18, 20, and 24 h after the polyester microfibers were added. Taking into consideration the difference between initial PAHs concentration and concentration of these micropollutants after a defined time, PAHs removal from wastewater

was calculated. The amount of PAHs removed was compared to the number of microfibers and presented in mg per one g. Simultaneously blank sample was prepared to evaluate the changes in PAHs concentration in wastewater samples by other processes than adsorption (during 24 h). The results were corrected taking into consideration blank sample results.

2.3. Qualification and quantification of PAHs

For selected 3- and 4-ring PAH analysis wastewater samples were extracted by using the liquid-liquid method. Such compounds as acenaphthene, acenaphthylene, fluorene, anthracene, phenanthrene, fluoranthene, pyrene, chrysene, benzo(a)anthracene were analyzed.

Cyclohexane/dichloromethane mixture (v/v 20:5:1) was used as extraction solvent. The samples were shaken for 1 h at a shaker with horizontal motion at constant amplitude. The extracts were then separated from wastewater and cleaned by using liquid–solid extraction using silica gel columns. After that, they were concentrated to 2 mL under a nitrogen stream. PAHs were determined quantitatively and qualitatively by gas chromatography coupled with a mass spectrometer (GC-MS) technique. The analysis was performed on a column DB-5 ms, length of 30 m and a diameter of 0.25 mm. Helium was used as a carrier gas (flow rate 1.08 mL min^{-1}). The time of analysis was equal to 50 min. The flow rate was 1.5 mL min^{-1}.

2.4. Adsorption capacity and kinetics of the adsorption process

Adsorption capacity was calculated by using the following formula:

$$q_e = \frac{(C_0 - C_i)\cdot V}{M} \quad (1)$$

where q_e is the adsorption capacity, μg g^{-1}; C_0 is the initial PAH concentration, μg L^{-1}; C_i is the equilibrium PAHs concentration, μg L^{-1}; V is the volume of solution, L; M is the mass of microplastic, g.

Two of the most popular kinetic models were used for kinetic evaluation of the adsorption process – Langmuir's model and Freundlich model [23,24]:

Langmuir's model:

$$\frac{C_e}{Q_e} = \frac{1}{K_L \cdot Q_{\max}} + \frac{C_e}{Q_{\max}} \quad (2)$$

where K_L is the Langmuir constant; $Q_{\max}$ is the maximum amount of PAHs adsorbed per unit mass of adsorbent, μg g^{-1}; Q_e is the amount of adsorbate per mass unit of adsorbent at equilibrium, μg g^{-1}; C_e is the equilibrium concentration, μg L^{-1}.

Freundlich model:

$$Q_e = K_f \cdot C_e^{\frac{1}{n}} \quad (3)$$

where K_f is the Freundlich constant, L g^{-1}; n is the model constant related to the intensity of adsorption.

3. Results and discussion

The initial concentration of total 3- and 4-ring PAHs reached 5,550 μg L^{-1}. As a result of the adsorption process, the total concentration of PAHs in wastewater decreased. After 24 h total concentration of 9 analyzed compounds decreased to 424 μg L^{-1}. It means on average 92% decrease in the concentration of 3- and 4-ring PAHs in wastewater.

In raw wastewater, the most abundant PAH compound of the analyzed was 3-ring acenaphthene, followed by benzo(a)anthracene, fluorene, phenanthrene, and fluoranthene – Fig. 1. The less abundant ones were acenaphthylene, chrysene and anthracene.

At the end of the experiment (after 24 h), the pattern of PAHs concentration was similar to the one obtained for raw wastewater in the case of 4-ring compounds, with dominant fluoranthene and benzo(a)anthracene (Fig. 2).

In the case of 3-ring compounds phenanthrene has become the most abundant, whereas concentrations of acenaphthene and fluorene decreased significantly. The highest removal efficiency for individual PAHs was obtained in the case of anthracene which was completely removed from wastewater after a 24-h retention time.

When we consider individual compounds as a function of time it can be stated that acenaphthene (Fig. 3) total percent removal from wastewater was equal to 97%. The highest removal rate was obtained during the first 2 h. In the case of acenaphthylene, adsorption was rapid during the first hour, when 40% of the compound was adsorbed from wastewater. During the second hour of the experiment concentration of acenaphthylene was reduced by another 39% reaching 79%. The maximum amount of adsorbed acenaphthylene after 24 h was equal to 24.8 μg g^{-1} (Table 2).

In the case of acenaphthene (Fig. 4) total percent removal from wastewater was equal to 99%. As in the case of acenaphthylene adsorption has occurred mainly during the first 2 h of the experiment with a decrease of acenaphthene concentration in the range of 60% during the first hour, and an additional 41% during the second hour of the experiment. The total amount of acenaphthene adsorbed on microfibers was equal to 1,424.7 μg g^{-1}.

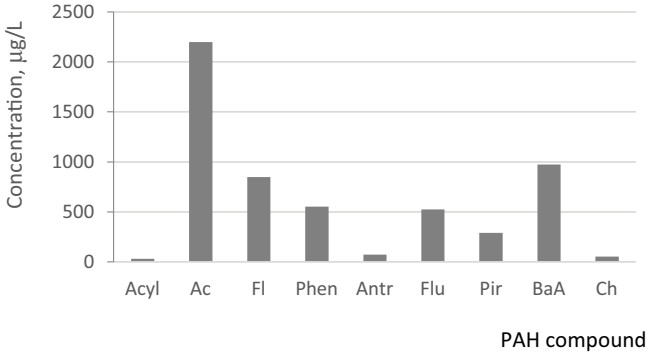

Fig. 1. Concentrations of individual 3- and 4-ring PAHs in raw industrial wastewater (Acyl – acenaphthylene, Ac – acenaphthene, Fl – fluorene, Phen – phenanthrene, Antr – anthracene, Flu – fluoranthene, Pir – pyrene, BaA – benzo(a)anthracene, Ch – chrysene).

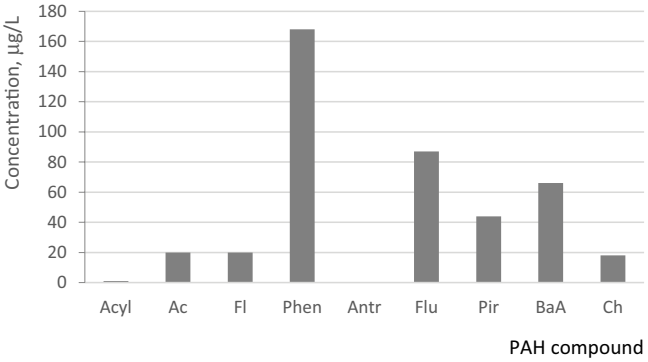

Fig. 2. Concentrations of individual 3- and 4-ring PAHs after 24 h adsorption on polyvinyl microfibers (Acyl – acenaphthylene, Ac – acenaphthene, Fl – fluorene, Phen – phenanthrene, Antr – anthracene, Flu – fluoranthene, Pir – pyrene, BaA – benzo(a)anthracene, Ch – chrysene).

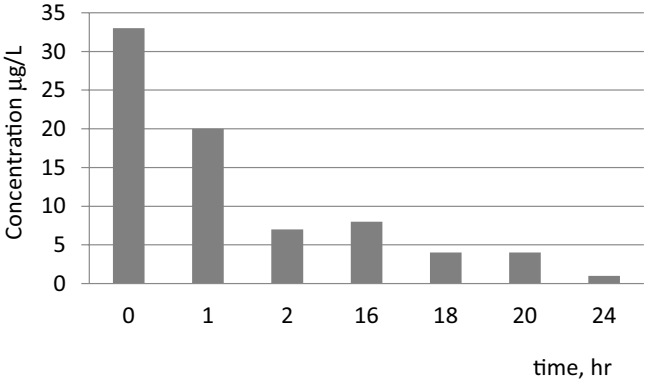

Fig. 3. Acenaphthylene concentration changes in wastewater in time.

Table 2
Maximum adsorption capacity of polyvinyl microfibers towards selected 3- and 4-ring PAH compounds

Compound	Amount of PAH adsorbed on microfibers (µg g^{-1})
Acenaphthylene	24.8
Acenaphthene	1,424.7
Fluorene	530.2
Phenanthrene	289.2
Anthracene	34.2
Fluoranthene	334.3
Pyrene	198.1
Benzo(a)anthracene	47.8
Chrysene	950.3
Total	3,833.6

Fluorene and anthracene removal by adsorption was also in a similar way – Figs. 5 and 6. In the case of fluorene, about 46% of the initial concentration has been removed during the first hour of the experiment, followed by a further 34% during the second one. The total percent removal

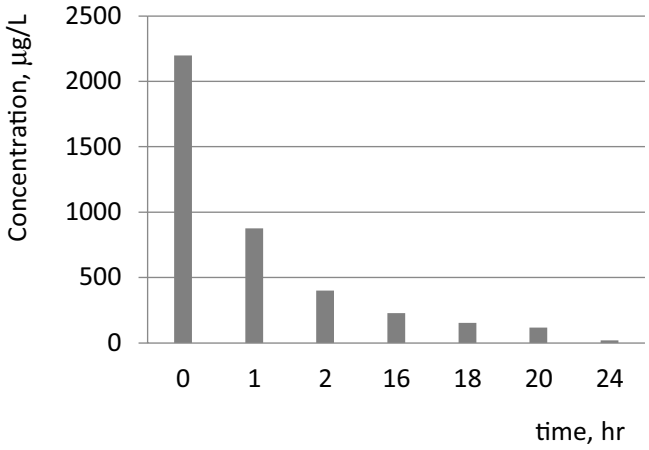

Fig. 4. Acenaphthene concentration changes in wastewater in time.

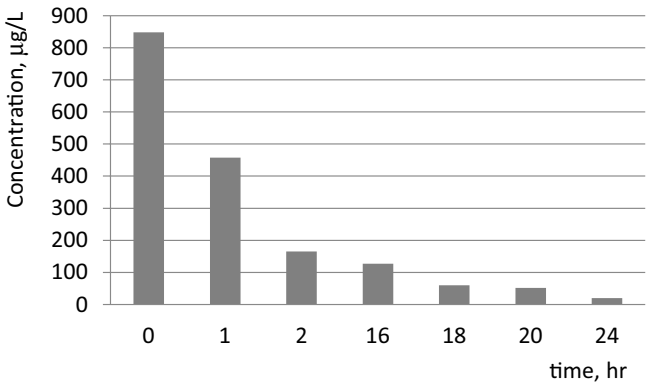

Fig. 5. Fluorene concentration changes in wastewater in time.

of fluorene after the 24 h experiment was equal to 97.6%. At the end of the experiment, 530.2 µg g^{-1} of fluorene was adsorbed on microfibers.

Removal patterns for anthracene and phenanthrene were untypical. During the first hour of the experiment, only about 16% of anthracene has been removed. During the second hour the removal was more effective and after that time 62% of this compound was removed. Finally, no anthracene was found in wastewater, which means that the whole compound has been adsorbed. Contrary to other 3-ring compounds part of anthracene was desorbed to wastewater after 18 and 20 h. The total amount of anthracene adsorbed on microfibers was equal to 34.2 µg g^{-1}.

Phenanthrene was not removed during the first hour of the experiment (Fig. 7). After 2 h about 40% of this compound was removed. Finally, after 24 h about 70% of this compound have been removed. Removal rates varied during the time and no linear correlation has been stated between the removal rate and time. The adsorption capacity of microfibers towards phenanthrene was 289.2 µg g^{-1}.

Based on the obtained results it can be stated that 3-ring PAHs were mainly adsorbed on microfibers during the first 2 h, however, the process was continued also after that time. In the process of further contact with microfibers no

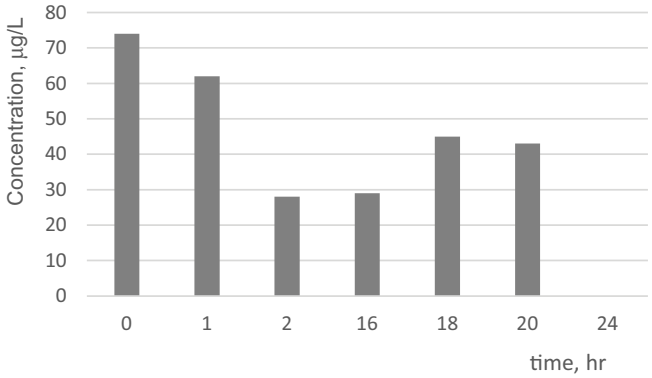

Fig. 6. Anthracene concentration changes in wastewater in time.

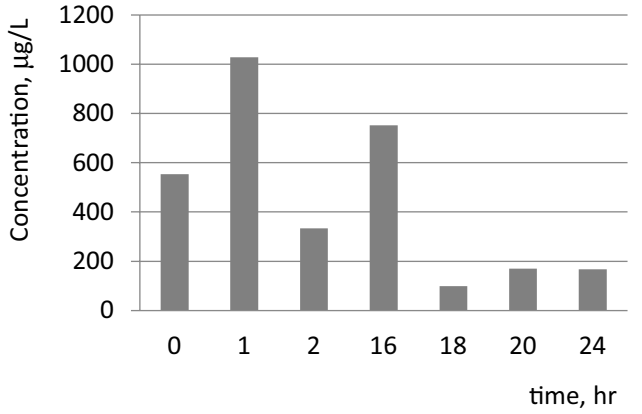

Fig. 7. Phenanthrene concentration changes in wastewater in time.

desorption of most 3-ring PAHs, except for phenanthrene and anthracene, has occurred under the conditions of the experiment. Percent of PAHs removal from wastewater by adsorption on microfibers was no lower than 70%.

In the case of 4-ring PAHs: fluoranthene, pyrene, chrysene and benzo(a)anthracene the results are presented in Figs. 8–11.

Fluoranthene and pyrene were not so rapidly adsorbed on microfibers as most 3-ring PAHs analyzed in our study. After 1 h about 10% of fluoranthene has been removed from wastewater. At the end of the experiment, 83% of fluoranthene has been removed. Also, the pyrene maximum removal rate has not exceeded 85%. The maximum rate of pyrene removal was observed after the 1st hour. During the first hour of the experiment, only 7.5% of this compound was removed. The total amount of pyrene adsorbed on microfibers was equal to 198.1 µg g^{-1}, whereas fluoranthene was 334.3 µg g^{-1}.

Benzo(a)anthracene was rapidly adsorbed during the first hour of the experiment, but after 2 h of experiments, some fluctuations have been observed. At the end of the 24-h experiment, 93% removal efficiency of benzo(a)anthracene has been achieved, with final adsorption capacity under experimental conditions equal to 47.8 µg g^{-1}.

In the case of chrysene, the pattern of removal was similar to 3-ring phenanthrene. No removal of the compound

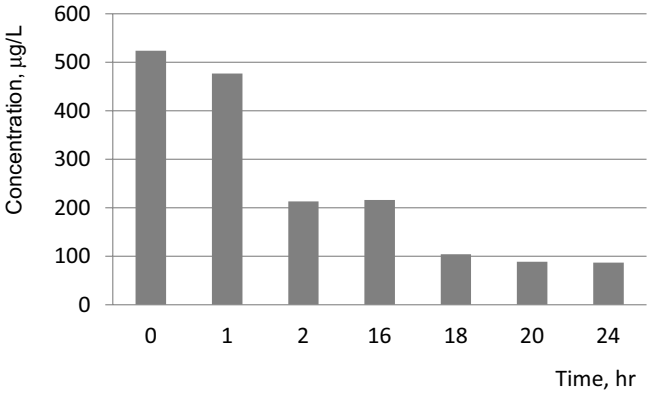

Fig. 8. Fluoranthene concentration changes in wastewater in time.

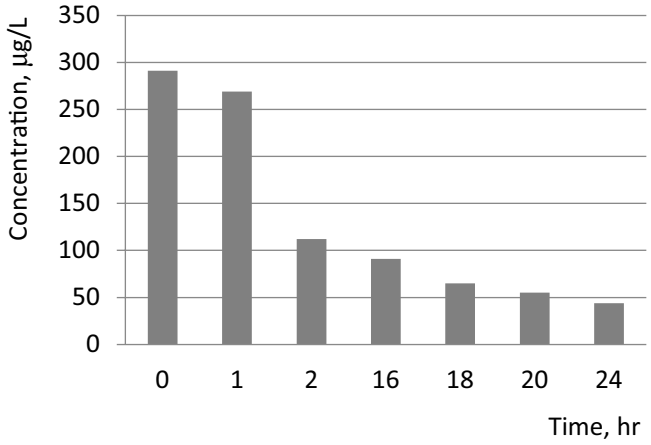

Fig. 9. Pyrene concentration changes in wastewater in time.

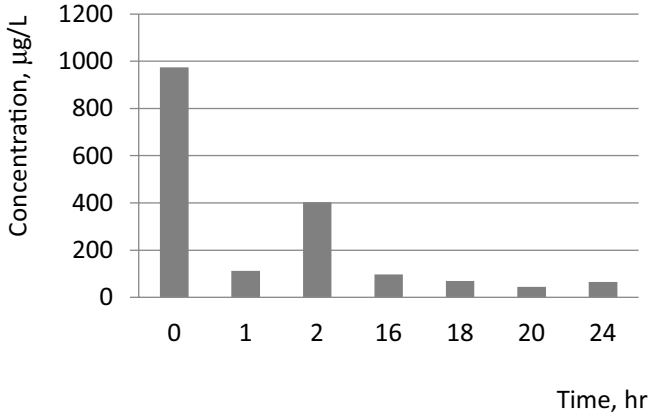

Fig. 10. Benzo(a)anthracene concentration changes in wastewater in time.

was observed during the first hour of the adsorption process. After that time 68% of this compound was removed. Finally of chrysene was removed. The final adsorption capacity for this compound was equal to 950.3 µg g^{-1}.

In the case of 4-ring PAHs no pattern of adsorption could be identified. The sorption efficiency was similar

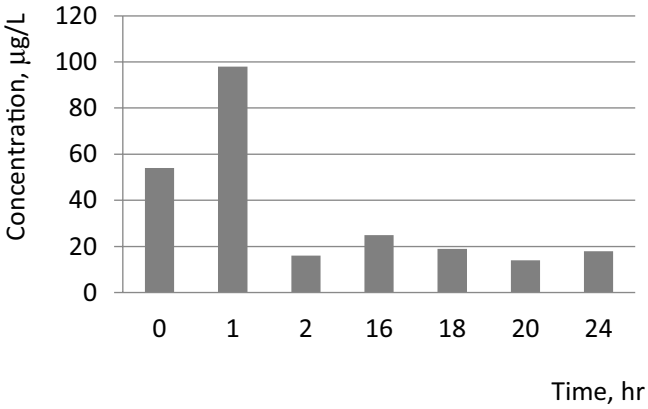

Fig. 11. Chrysene concentration changes in wastewater in time.

and for 3-ring ones it was in the range of 62%–75% and for 4-ring ones, it was in the range of 63%–88%. The experiment, however, proved and confirmed that all 3- and 4-ring compounds were adsorbed on polyester microfibers. The total amount of PAHs was equal to 3,833.6 μg g^{-1}. As for sorption patterns, the 4-rings are made of benzene rings, while the 3-rings have a cyclopentane ring. These compounds show different properties in relation to the sorbent active groups, they are also susceptible to degradation to a different extent. The exception was anthracene, which was not quantified after 24 h.

The results described above were supported by the ones concerning calculations from kinetic models (Table 3). The results are consistent with the literature data, which provide the values of the adsorption capacity and coefficients in kinetic equations and coefficients according to the Freundlich or Langmuir isotherm models in a wide range. Compared to the results obtained by other authors differences can be observed. The differences result from different types, doses and properties of sorbents, the initial concentration of the analyzed compounds, the qualitative composition of the solution, temperature and various process parameters. Gupta et al. [23] have stated that values for the adsorption capacity of naphthalene are in the range from 333–416 mg g^{-1}, fluorene 285–476 mg g^{-1}, phenanthrene 217–277 mg g^{-1}, anthracene from 0.14 to 8.35 mg g^{-1}. The values of the Freundlich equation coefficients were in the range naphthalene 21–86, fluorene 18–138 and phenanthrene 30–106. The literature also provides information on the fit of adsorption models, indicating that PAHs for individual compounds are different in different literature sources. For example, for naphthalene and pyrene, the Freundlich model was shown to be the best in the research by Yakout et al. [24].

Compared the results with the ones obtained by other authors it can be observed that equilibrium conditions in the case of PAH adsorption can be achieved even after 5–10 d [25], so the adsorption process is rapid at the beginning, however, it lasts for several days even toward individual microplastic particles. It depends on many factors, among them temperature, presence of other pollutants, and their water solubility was identified [25]. Linear adsorption observed for most PAHs during the experiment

Table 3
Kinetic models of selected PAHs adsorption on polyester microfibers

Compound	Freundlich model			Langmuir's model
	K_f	$1/n$	R^2	K_L
Acenaphthylene	4.141	0.4113	0.9095	9.7×10^{-4}
Acenaphthene	2.9153	0.1581	0.9450	8.8×10^{-2}
Fluorene	2.1855	0.3251	0.8198	6.7×10^{-5}
Phenanthrene	1.5119	0.5291	0.7664	1.7×10^{-4}
Anthracene	0.8859	0.2416	0.6420	–
Fluoranthene	0.8800	0.4841	0.4373	5.6×10^{-5}
Pyrene	0.1172	1.1070	0.6428	2.1×10^{-4}
Chrysene	2.3425	0.1456	0.9254	4.4×10^{-5}
Benzo(a)anthracene	2.7845	0.2014	0.8974	2.3×10^{-4}

was identified as typical for larger microplastics particles, especially at higher temperatures such as 20°C [25].

The amounts of PAHs adsorbed onto microplastics particles were significantly higher than the ones obtained by other authors who analyzed the adsorption of PAHs onto microplastic present in a natural environment. Nabetani et al. [26] have stated that on microplastics in Lake Biwa and Osaka Bay 1.7–27 μg g^{-1} of a total of 16 PAHs have been adsorbed. These authors have also calculated that this phenomenon was connected with $\log K_{ow}$ values, and chemicals content on microplastics increased as their octanol/water partition coefficient increased. The differences between the capacity of microplastics towards PAHs in natural water and wastewater (in our study) could be due to the lower concentration of these compounds in the water body than in industrial wastewater. Contrary to Sørensen et al. [25], Nabetani et al. [26] have stated that smaller microplastic particles have adsorbed more PAHs than microplastics with larger diameters. Contrary to this Liu et al. [27] have no observed the correlation between microplastic sizes and sorption efficiency. This aspect without a doubt needs further detailed research work. The differences between the adsorption behavior of 4- and 3-ring PAH compounds can be connected with the fact that 4-ring ones contain only benzene rings, whereas the 3-ring PAHs contain also cyclopentane rings.

4. Conclusions

It can be concluded that:

- PAHs were effectively adsorbed onto the surface of microfibers present in wastewater. Removal efficiencies of the individual compound were from 67% in the case of chrysene to 100% (for anthracene). Rapid adsorption during the first 2 h has occurred which confirms that adsorption of PAHs occurs in the wastewater probably before they inflow into the wastewater treatment plant.
- PAHs sorption capacities on polyester fibers obtained in our studies (in the range 24.8 for fluorene to 1,424.7 μg g^{-1}) were rather high compared to the ones obtained for microplastics present in freshwater systems. The total

amount of analyzed PAHs adsorbed on microfibers was equal to 3,833.6 μg g^{-1}. No data for comparison of PAHs sorption capability in wastewater are available.
- Based on the literature data it can be recommended further detailed studies on the sorption behavior of PAHs on fibers and other types of microplastics as crucial for the environmental fates of these pollutants.

Acknowledgments

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