SONOCHEMICAL PROCESSES IN THE DECOMPOSITION OF ORGANIC MICROPOLLUTANTS FROM RAINWATER

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Abstract

A wide range of aquatic systems, drinking water, groundwater and surface water is known to include several dangerous organic micropollutants (OMPs). Additionally, a lot of the OMPs that have been found are resistant to biodegradation, allowing the possibility of persistence and accumulation in the ecosystem. Although they are present in small amounts, they have the potential to cause major health issues, such as cancer. Recent studies showed that ultrasonication is very successful in removing numerous hazardous OMPs from water. However, there is a tendency of combining this method with other advanced oxidation processes because of some of this technology's limitations. This study provided additional evidence that ultrasonication is a highly effective approach for removing micropollutants from rainwater. The efficiency of this technique can even be higher than 80%. Moreover, the addition of ozone and hydrogen peroxide during ultrasonication increased the removal ratio significantly, which was proportional to the oxidant dosage.

Keywords: ultrasound, advanced oxidation processes, sonocatalysts, organic micropollutants, OMPs

1. Introduction

One of the promising strategies of removing organic micropollutants (OMPs) from the aquatic environment is the usage of the ultrasonication process. It was found to be effective in many substances harmful to human health, such as bacteria, fungi, dyes, viruses, algae, polycyclic aromatic hydrocarbons (PAHs), pesticides, industrial chemicals, pharmaceuticals and personal care products (PPCPs) (Manariotis, Karapanagioti and Chrysikopoulos, 2011; Gągol, Przyjazny and Boczkaj, 2018; Vega, Soltan and Peñuela, 2019). Ultrasonication is considered an environmentally friendly method in water and wastewater treatment and it is an interesting alternative to other advanced oxidation processes (AOPs) (Pham

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et al., 2009). Developing new technologies in OMPs removal is crucial because as a result of the urbanization process, the number of micropollutants identified in the environment keeps constantly growing. Moreover, they also pose a threat to the entire ecosystem, despite the fact that they occur at low concentrations (mainly from ng L^{-1} to μg L^{-1}) (Arslan et al., 2017). Furthermore, their concentration in water is not regulated by guidelines, standards or legal acts. Importantly, the effectiveness of their removal in conventional treatment systems is very low, thus wastewater treatment plant facilities are considered major sources of OMPs in the aquatic environment (Gago-Ferrero, 2017; Sörengård et al., 2019).

However, although ultrasound has a large potential in water and wastewater treatment and other environmental purposes there are still considerable limitations for scale-up issues – technical and economical ones (Serna-Galvis, Porras and Torres-Palma, 2022). Hence, there is a tendency to combine sonication with other technologies which can prove to be very efficient in contaminant reduction (Gągol, Przyjazny and Boczkaj, 2018). In recent years, there has been an increasing interest in OMPs removal by using acoustic cavitation and other AOPs simultaneously such as US/UV (Rao et al., 2016; Copik, Kudlek and Dudziak, 2022), US/O_3 (Fraiese et al., 2019), US/H_2O_2 (Zhang et al., 2011; Lim, Son and Khim, 2014; Copik et al., 2021). The number of studies on sonocatalysis keeps constantly growing (Web of Science).

Therefore, the purpose of this paper is to examine the degradation of OMPs from rainwater widely spread in the environment by using ultrasound technology as a single process, combined with ozonation and hydrogen peroxide addition. Furthermore, to evaluate the effectiveness of the above-mentioned methods, gas chromatography and ecotoxicological analysis were performed.

2. Methods and materials

2.1. Sample preparation and chemicals

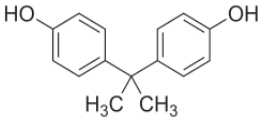
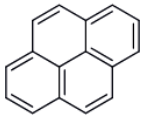
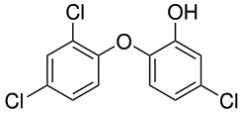
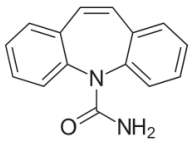
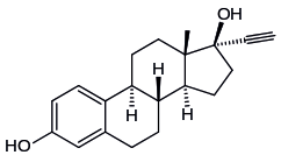
Rainwater samples were collected from the roof drainage system of the building located in an urbanized area in Southern Poland. Before measuring the basic physicochemical properties, 10 L of water were taken from a plastic rainwater storage tank and mixed. The samples were collected around 5 min after intense rain started. The pH value of the rainwater was 6.8, turbidity 5 NTU, and conductivity was 30,5 μS cm^{-1}.

Subsequently, five OMPs, namely Bisphenol A (BPA), Carbamazepine (CBZ), 17 – α ethinyloestradiol (EE2), Pyrene (PYR), and Triclosan (TCS) were added to the collected samples to obtain a concentration of 1 mg L^{-1}. Higher concentrations than those that occur in the natural environment were used in the study to increase the accuracy of the analysis. Importantly, rainwater may not naturally contain all these substances, however, the effect of this water matrix on the efficiency of

removing harmful compounds should be determined, because rainwater enters the whole aquatic system.

These substances were of analytical grade and supplied by Sigma – Aldrich (Poznan, Poland). The pH value and conductivity of solutions were examined by using CPC – 505 device provided by Elmetron (Zabrze, Poland) while turbidity was monitored by the HI-93414-02 Turbidity meter provided by HANNA Instruments Inc. Table 1 contains the basic parameters of the analysed micropollutants.

Table 1. Physicochemical properties of analysed OMPs (Kim et al., 2016)

Compound	Chemical formula	Molar mass (g mol^{-1})	Structural formula	CAS no.
BPA	$C_{15}H_{16}O_2$	228.29		80-05-7
PYR	C_6H_{10}	202.25		129-00-0
EE2	$C_{20}H_{24}O_2$	296.40		57-63-6
CBZ	$C_{15}H_{12}N_2O$	236.27		298-46-4
TCS	$C_{12}H_7Cl_3O_2$	289.5		3380-34-5

2.2. Advanced oxidation processes

The sonication process was carried out in a horn-type, 20 kHz frequency transducer supplied by Vibracell Sonics, Sonics and Materials Inc., (Newtown Connecticut, USA). The device was equipped with a 13 mm diameter probe, which can generate high-intensity ultrasound at the maximum power of 500 W. The adoption of a cooling system prevented overheating of the treated samples. The temperature of

the solution was kept at approximately 25°C. The sonication process was operated in a 100 mL volume vessel placed on the dedicated magnetic stirrer. The sonication was carried out at a constant ultrasound amplitude level equal to 114 μm which is 80% of maximum device amplitude. The time of the treatment varied from 1 to 30 min.

The ozonation process was conducted in ozoner FM500 (WRC Multiozone, Gdansk, Poland). The O_3 produced from fresh air was introduced to the 100 mL vessel containing rainwater solution by the ceramic diffuser placed around 3 cm below the water level. The process was carried out in the following O_3 dosages: 1, 2, 5 and 10 mg L^{-1}, and the sample contact time during ozonation was 30 min. The addition of hydrogen peroxide (H_2O_2) was executed in the same volume of the beaker (100 mL), and the dosage of oxidant was equal to 3, 6, 9 and 12 mg L^{-1}. In the study, 30% of H_2O_2 provided by Sigma-Aldrich (Poznan, Poland) was used. The dosage of oxidants was selected based on preliminary research.

Finally, the ultrasonication process was combined with above – mentioned AOPs (US/O_3 and US/H_2O_2 processes). The treatment was conducted in a 100 mL vessel, at the 30 min sonication time and maximum intensity. The process was introduced at different dosages of O_3 and H_2O_2. The experimental setup is shown in Fig. 1.

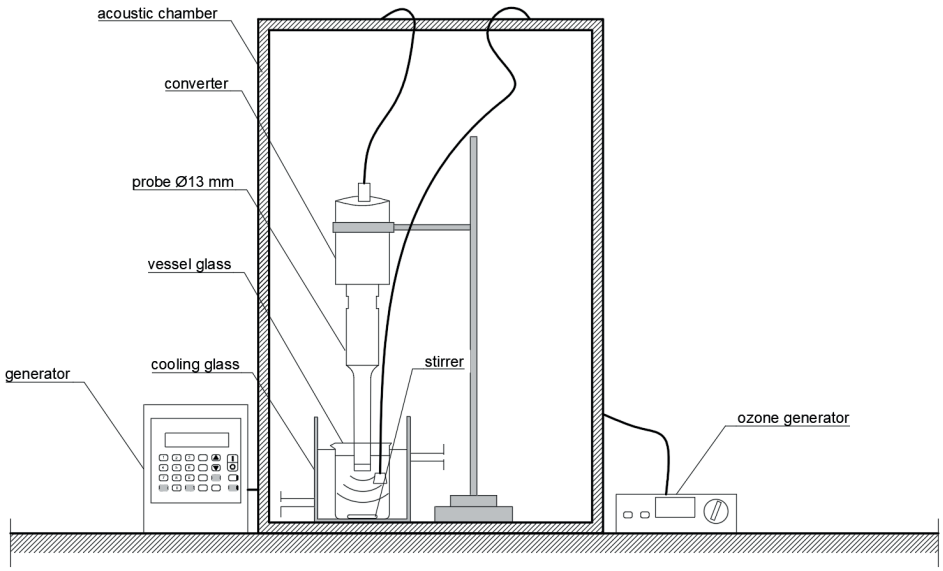


Figure 1. Experimental setup (Copik, Kudlek and Dudziak, 2022)

2.3. Solid phase extraction and gas chromatography method

To determine the efficiency of rainwater treatment by advanced oxidation processes gas chromatography analysis was performed using gas chromatograph 7890B supplied by Perlan Technologies (Warsaw, Poland). Utilizing Superclean™ ENVI™

– 18 tubes with an Octadecylsilane (C18) cartridge bed, the Solid Phase Extraction (SPE) technique was used to transform the sample matrices into a suitable form and enhance the accuracy of the chromatographic analysis. 5 mL of methanol (MeOH) and clean water were used in the conditioning process, and 5 mL of dichloromethane (DCM) were applied during the elution process. The drying time in the vacuum was set at 5 minutes, and the sample flow was set at 1 mL min^{-1}. Negative pressure ranging from 5 to 10 kPa was used during the SPE procedure.

Gas chromatography used helium as the carrier gas at a flow rate of 1 mL min^{-1}. Ion source and Ion trap temperatures were set at 150°C and 230°C, accordingly. The oven temperature ranged from 80°C to 300°C, while the injector temperature was 250°C. The capillary columns (30 m x 0.25 mm x 0.25 µm dimensions) were purchased from Sigma-Aldrich (Poznan, Poland) and were 0.25 mm thick. The degree of OMPs removal after treatment was assessed based on the peak areas as compared to the calibration process data. The recovery rate of the tested compounds ranged from 97 to 99%, and the limit of detection ranged from 0.03 ng mm^{-3} for PYR to 0.23 ng mm^{-3} for TCS. The limit of quantification ranged from 6.2 to 27 μL^{-1}.

2.4. Ecotoxicological assessment

To assess the toxicity of the post–processed samples, the Microtox toxicity test was employed. It is based on the measurement of the inhibition of luminescent marine bacterium *V. fischeri* exposed to the tested solutions. The toxicity effect of the samples was assessed based on the intensity of light emitted by the bacteria after 5 and 15 min of contact time with solutions. In the study, the Microtox Omni system procedure was employed in the Microtox 500 analyser (Warsaw, Poland). Moreover, as a reference sample 2% solution of NaCl was used. The effect was classified into four toxicity classes listed in Table 2.

Table 2. Toxicity classification system of post-treated samples (Santana et al., 2009; Bohdziewicz et al., 2016)

Toxicity (%)	Toxicity class
< 25	non-toxic
25–50	low toxicity
50–75	toxic
> 75	high toxic

3. Results

3.1. Ultrasonication as a single process

It can be seen from the data in Fig. 2 that there was a proportional correlation between sonication time and degradation ratio. The highest rate of OMPs elimination equal to 83% was achieved in the case of CBZ and the lowest for BPA (39%). The average obtained removal ratio after 30 min of ultrasonication used as a single process was 56%. Moreover, research revealed that even at the short time of sonication treatment efficiency of OMPs removal was very high.

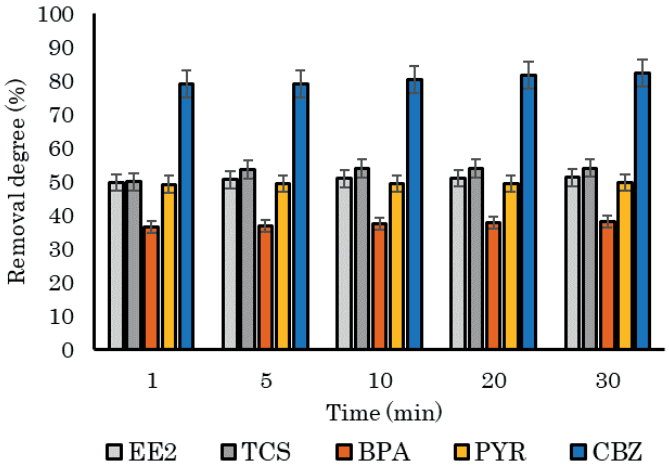


Figure 2. Removal degree of OMPs in ultrasonication treatment

It is worth noting that the sonication process was conducted at a high amplitude level, corresponding to 57 W of acoustic power and 43 W cm^{-2} acoustic intensity. These parameters affect the high production of oxidants due to bubbles implosion including hydroxyl radicals OH and hydrogen peroxide H_2O_2 following equations 1–4 (Doosti, Kargar and Sayadi, 2012):

$$H_2O \rightarrow H^{\cdot} + OH^{\cdot} \quad (1)$$

$$H^{\cdot} + OH^{\cdot} \rightarrow H_2O \quad (2)$$

$$H^{\cdot} + H^{\cdot} \rightarrow H_2 \quad (3)$$

$$OH^{\cdot} + OH^{\cdot} \rightarrow H_2O_2 \quad (4)$$

It is a well-known fact that increased time is connected with more cavitation events. Nevertheless, the increment of treatment efficiency is limited.

As is well known, increasing the time of sonication leads to an increase in the number of cavitation events, however, an excessive amount of generated

bubbles can inhibit the process efficiency due to the inhibition of ultrasonic wave propagation (Chen, 2011).

3.2. O_3 and US/O_3 process

To evaluate the effectiveness of OMPs degradation from rainwater ozonation as a single process, ozonation combined with ultrasonication was used. In this stage, the same parameters of ultrasonication were applied and the sonication time was 30 min. The degradation of selected micropollutants by using the above mentioned advanced oxidation processes is shown in Fig. 3.

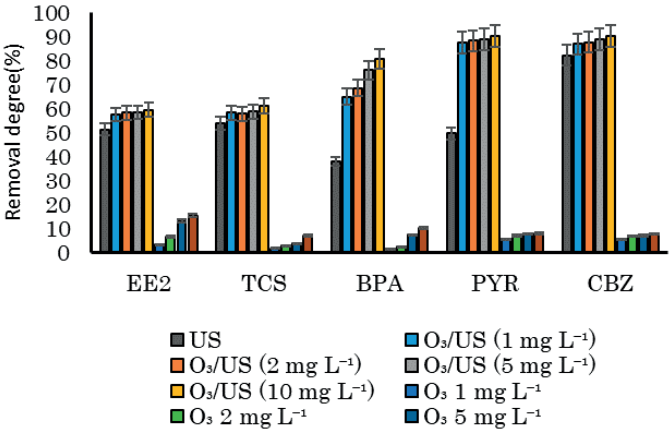


Figure 3. Removal degree of OMPs in ultrasonication and ozonation treatment

Data shows that the removal ratio of OMPs increases as the ozone dosage is increased.

However, it can be observed that the ultrasonication technique was more effective than ozonation used as a single process. The average removal degree of OMPs was equal to respectively 56 and 10% (at maximum ozone concentration). As is well known, ozone has a very high oxidation-reducing potential equalling to 2.07 V, therefore it has a strong ability of removing harmful substances from aquatic environment. Furthermore, the O_3 presence in water during sonification can increase the hydroxyl radicals' generation, which is accredited to the thermolytic ozone decomposition during acoustic cavitation according to the equations 5-7. As a result of these phenomena, a synergic effect can be obtained (Rekhate and Srivastava, 2020).

$$O_3 \rightarrow O_2 + O^{\cdot} \tag{5}$$

$$O^{\cdot} + H_2O \rightarrow 2HO^{\cdot} \tag{6}$$

$$2HO^{\cdot} \rightarrow H_2O_2 \tag{7}$$

It is worth noting that in this study the ozonation method significantly increased the efficiency of the ultrasonication technique. After the addition of 10 mg L^{-1}, the average removal of analysed substances in US/O_3 was 76%, and so the average enhancement of efficiency was equal to 20%. In this process, the maximum degradation degree was obtained in the case of CBZ, and PYR (respectively 90 and 91%).

3.3. H_2O_2 and US/H_2O_2 process

Fig. 4 shows the degradation of selected micropollutants during hydrogen peroxide treatment as a single process and combined with ultrasonication technique.

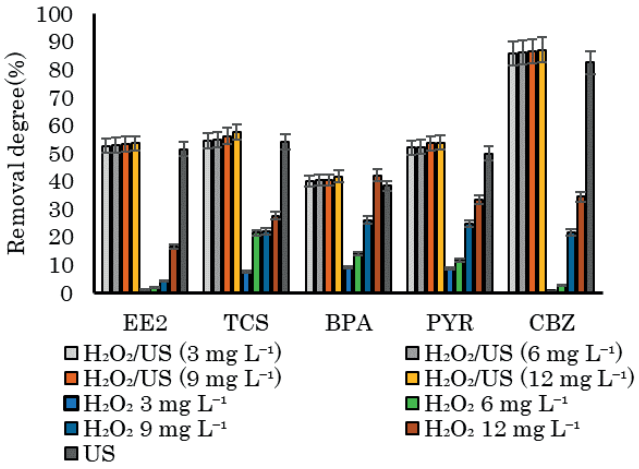


Figure 4. Removal degree of OMPs in ultrasonication and hydrogen peroxide treatment

This stage of study provided further evidence that with the increased dosage of oxidation agent, OMPs concentration decreases. It was noted that the average degradation rate after the addition of 12 mg L^{-1} without ultrasonic assistance was equal to 31%, thus the H_2O_2 process was more effective than ozonation used alone. Furthermore, the research indicated that H_2O_2 dosage combined with sonification was more effective than both processes used singularly. The highest removal ratio was obtained in the case of CBZ during US/H_2O_2 (87%). The enhancement of effectiveness in these processes is also attributed to hydroxyl radicals' generation as shown in equations 1-4. Due to hydrogen peroxide addition, more H_2O_2 is dissociated into two hydroxyl radicals and consequently can be treated as its secondary source (Nie, Wang and Qiu, 2008; Nikfar *et al.*, 2016). Despite obtaining a high degree of removal of harmful compounds, it should be emphasized that sonication is associated with high energy consumption (around 130 kJ during 30 minutes of sonication). Therefore, the economic analysis of the ultrasonication process also plays an important role. However, preliminary studies indicated (data

not shown) that the synergistic effect in the US/O_3 and US/H_2O_2 processes during 1 min sonification was significantly lower.

3.4. Toxicity

The Microtox ® assessment results are summarized in Fig. 5.

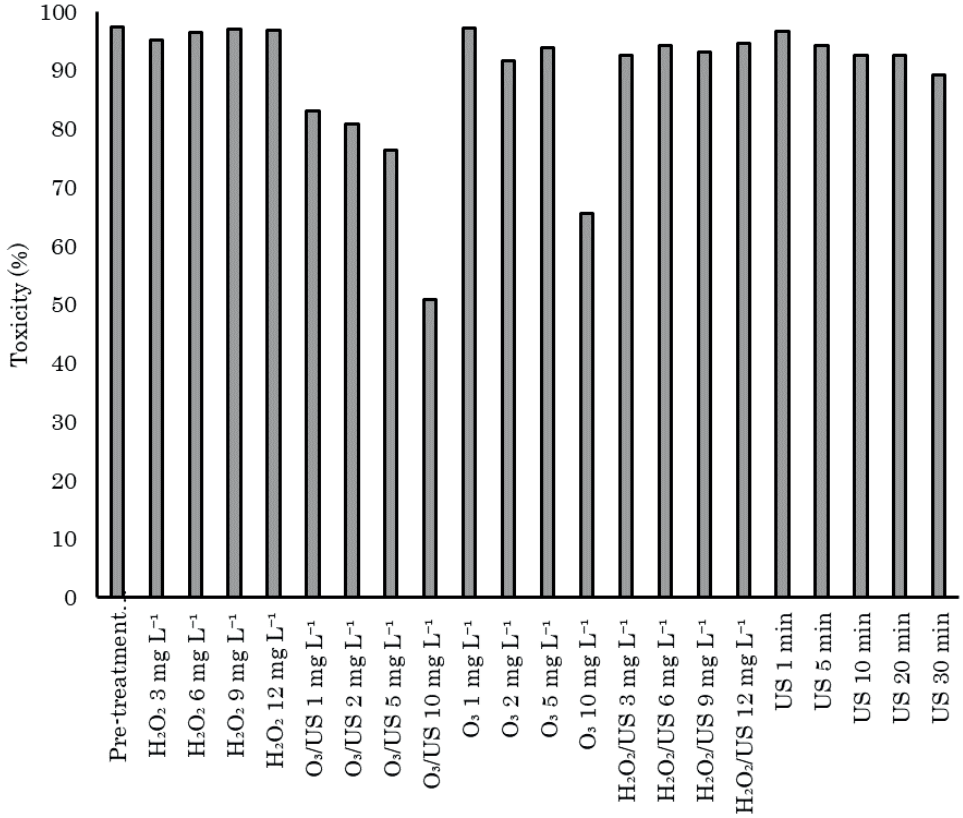


Figure 5. Toxicity of samples after ultrasonication treatment

The conducted analysis revealed that the rainwater sample was not toxic (average toxicity after 5 min and 15 min of exposition was equal to 2%). However, after the addition of 1mg L-1 of five selected OMPs the average toxicity of the sample increased to 97.3%. Research indicated that during the ultrasonication process average toxic effect decreased with sonication time. However, the sample toxicity after 30 min of sonication remained highly toxic (89% of toxicity). Similarly, after the H_2O_2 and H_2O_2/US process no significant reduction in post-treated sample toxicity was observed. At the maximum dosage of oxidant H_2O_2 and H_2O_2/US processes resulted in 1 and 3% reduction in toxic effect, respectively. Such relation might be caused by the generation of so-called degradation by-products.

Importantly, the intermediates formed during advanced oxidation processes can be even more toxic and harmful to human health than the initial compound, and so many scientists are interested in AOPs treatment, which emphasizes the necessity and significance of extensive ecotoxicological analysis (Dudziak, Kudlek and Burdzik-Niemiec, 2018; Kudlek, 2019; Copik, Kudlek and Dudziak, 2022).

Nevertheless, during ozonation used as a single process, the toxicity decreased to 65% at the maximum dosage of ozone. Furthermore, when the process was performed with ultrasonication assistance, the toxic effect decreased to 50% (i.e. low toxic).

4. Conclusions

This study set out to evaluate the effectiveness of the advanced oxidation process in BPA, CBZ, EE2, PYR and TCS removal from water, which can be commonly found in the environment as a result of anthropogenic activity. Given their toxicity and destructive effect on humans, novel techniques should be developed to remove them from water. The result of this investigation showed that considering the highest dosage of oxidant and maximum time of ultrasonication the efficiency of the treatment during analysed AOPs decreased in order: O_3/US, US/H_2O_2, US, H_2O_2, O_3. It is noteworthy that the O_3-based processes had the highest efficiency in toxicity decrement. After the addition 10 mg L^{-1} of O_3 combined with ultrasonication, the toxicity of the samples decreased from highly toxic to low toxic. It can be concluded that the ultrasonication technique can be a highly effective method in OMPs removal, especially when it is combined with other AOPs. Furthermore, the degradation rate of OMPs was very high even at the 1 min time of sonication. Nevertheless, attention should be paid to ecotoxicological analysis, the presence of formatted intermediates and their potentially harmful effect on public health. In addition, another important aspect is the high energy consumption during sonication, therefore, an attempt should be made to optimize the process in economic terms.

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SONOCHEMICZNE PROCESY W USUWANIU ZANIECZYSZCZEŃ MIKROORGANICZNYCH Z WODY DESZCZOWEJ

Abstrakt

Obecnie w wodzie wodociągowej w wodach gruntowych oraz w wodach powierzchniowych powszechnie identyfikuje się niebezpieczne małocząsteczkowe mikrozanieczyszczenia organiczne (OMPs). Ponadto wiele z nich jest odpornych na biodegradację, co stwarza możliwość ich akumulacji w środowisku. Pomimo że ich stężenie jest niewielkie, mogą powodować poważne problemy zdrowotne. Część mikrozanieczyszczeń jest również kancerogenna. Badania wykazują, że ultradźwięki mogą być bardzo skuteczne w usuwaniu wielu niebezpiecznych OMPs z wody. Istnieje jednak tendencja do łączenia tego procesu z innymi zaawansowanymi procesami utleniania (AOPs) ze względu na niektóre ograniczenia technologii nadźwiękawiania. Przeprowadzone badania dowiodły, że ultradźwięki są skuteczną metodą usuwania mikrozanieczyszczeń z wody deszczowej. Skuteczność tej techniki wyniosła nawet około 80%. Co więcej, dodatek ozonu i nadtlenku wodoru podczas procesu nadźwiękawiania zwiększył efektywność usuwania szkodliwych związków proporcjonalnie do dawki zastosowanego utleniacza.

Słowa kluczowe: nadźwiękawianie, zaawansowane procesy utleniania, mikrozanieczyszczenia organiczne, AOPs