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# Removal of BTX Contaminants with O<sub>3</sub> and O<sub>3</sub>/UV Processes

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Additional information is available at the end of the chapter

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## Abstract

The legal basis for the monitoring of priority and priority hazardous substances in water, sediment, and biota follows from Directive 2008/105/EC which defines the good chemical status to be achieved by all Member States together with the Water Framework Directive 2000/60/EC. The BTX compounds are considered to be the most toxic components of gasoline. Thus, organic petroleum components can induce a serious problem to public health and the aquatic environment. The effect of ozone and ozone/UV on degradation of the BTX in a model water was studied. The results indicate that the highest BTX removal rates were observed during the first 5 min of the process for all investigated pollutants. The treatment efficiencies above 90% were observed in all investigated pollutants after 40 min of ozonation. The results show a significant proportion of stripping in the removal of BTX components. Higher overall efficiency was observed by O<sub>3</sub>/UV process after abstracting share of stripping process. Application of investigated processes appears to be a promising procedure for removal of petrol aromatic hydrocarbons from aquatic environment. However, for practical application, an improvement of process removal efficiency and investigation of impact of ozonation intermediates and products on aquatic microorganisms are required.

**Keywords:** BTX, jet-loop reactor, ozone, O<sub>3</sub>/UV, wastewater treatment

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## 1. Introduction

The adoption of the Framework Directive on water [1] provides a policy tool that enables sustainable protection of water resources. The Decision No. 2455/2001/EC of the European Parliament and the Council of November 2001 [2] established the list of 33 priority substances

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or group of substances, including the priority hazardous substances, presenting a significant risk to water pollution or *via* the aquatic environment including such risks to water used for the abstraction of drinking water.

Hazardous substances are defined as substances or groups of substances that are toxic, persistent, and liable to bioaccumulation, and other substances or groups of substances which give rise to an equivalent level of concern. The EC member countries have extended this list with relevant pollutants for individual countries. Thus, in the supplement of the Water Act [3] there have been identified altogether 59 relevant substances for SR.

The BTEX contaminants consist of benzene, ethyl benzene, toluene, and three isomers of xylene. These compounds are the volatile organic compounds (VOCs) found in petroleum derivatives such as petrol (gasoline). They represent one of the main groups of soluble organic compounds that is present in wastewater from refinery. They are the most toxic components of gasoline. These substances can lead to serious health problems ranging from irritation of eyes, skin, and mucous membranes and ending with weakened nervous system, decreased bone marrow function, and cancer. Benzene in particular is highly toxic. The World Health Organization classifies the substance as carcinogenic. It is also on the list of the priority substances [4].

Many oil substances have acute toxic effect on aquatic microorganisms with possible chronic consequences [5].

Commonly used wastewater treatment processes usually apply physical and physiochemical processes. Thus, the discharge of organic pollutants may create some environmental problems, particularly at microlevel. The aromatic oil fraction consists mainly of polyaromatic hydrocarbons (PAHs) and is more toxic and persistent than the aliphatic hydrocarbons [6]. Leakages including release of petroleum products, e.g., gasoline, diesel fuel, lubricating, and heating oil, from leaking oil tanks are the most frequent sources of soil and groundwater contaminations with BTEX substances. They are polar and readily soluble, and thus they are able to penetrate into soil and groundwater and cause serious environmental problems. These compounds dispose of acute and long-term toxic effects [4].

BTEX are among the most frequently detected contaminants in US public drinking-water systems that rely on groundwater sources [7]. These organic compounds make up a significant percentage of petroleum. The most contaminated locality of hazardous BTEX substances in the Slovak Republic is the airport at Sliač, Sliač-Vlkanová territory, contaminated by the Soviet Army, and the gas station in Rajecké Teplice where BTEX contaminants were identified as dominant in the groundwater. BTEX were also found in groundwater in Bratislava due to poor technical conditions of technological equipment (old stocks of aviation fuel) and the subsequent uncontrolled release of oil into the rock mass at the Airport of M. R. Štefanik [8].

Ozone is a very powerful oxidizing agent ( $E^\circ = 2.07$  V). Ozone may react with organic compounds in two ways: by direct reaction as molecular ozone or by indirect reaction through formation of secondary oxidants like free radical species [9–11]. In practice, both mechanisms may occur depending on the type of chemical wastewater pollution.

At low pH, the predominant reaction mechanisms are the direct electrophilic attack by molecular ozone [12], i.e., ozonolysis.

Under such conditions, ozone is a selective oxidant and reacts with multiple bonds (C=C, C=N, N=N, etc.) , but only at low rates with single bonds (C-C, C-O, O-H). At high pH, indirect reaction occurs, i.e., organics are degraded by secondary oxidants/chain reaction involving powerful radicals including OH, which are produced by ozone decomposition. These radicals are very strong and nonselective oxidants. Hydroxyl radicals can be formed by increasing pH or by decomposition of O<sub>3</sub> with homogeneous and heterogeneous catalysts.

The main goal of our research was to study the feasibility of ozone and combination of O<sub>3</sub>/UV processes for removal of selected benzene, toluene, and xylenes (BTX) from water/wastewater. Investigation of process kinetics and stripping of volatile substances were also performed.

## 2. Experimental methods

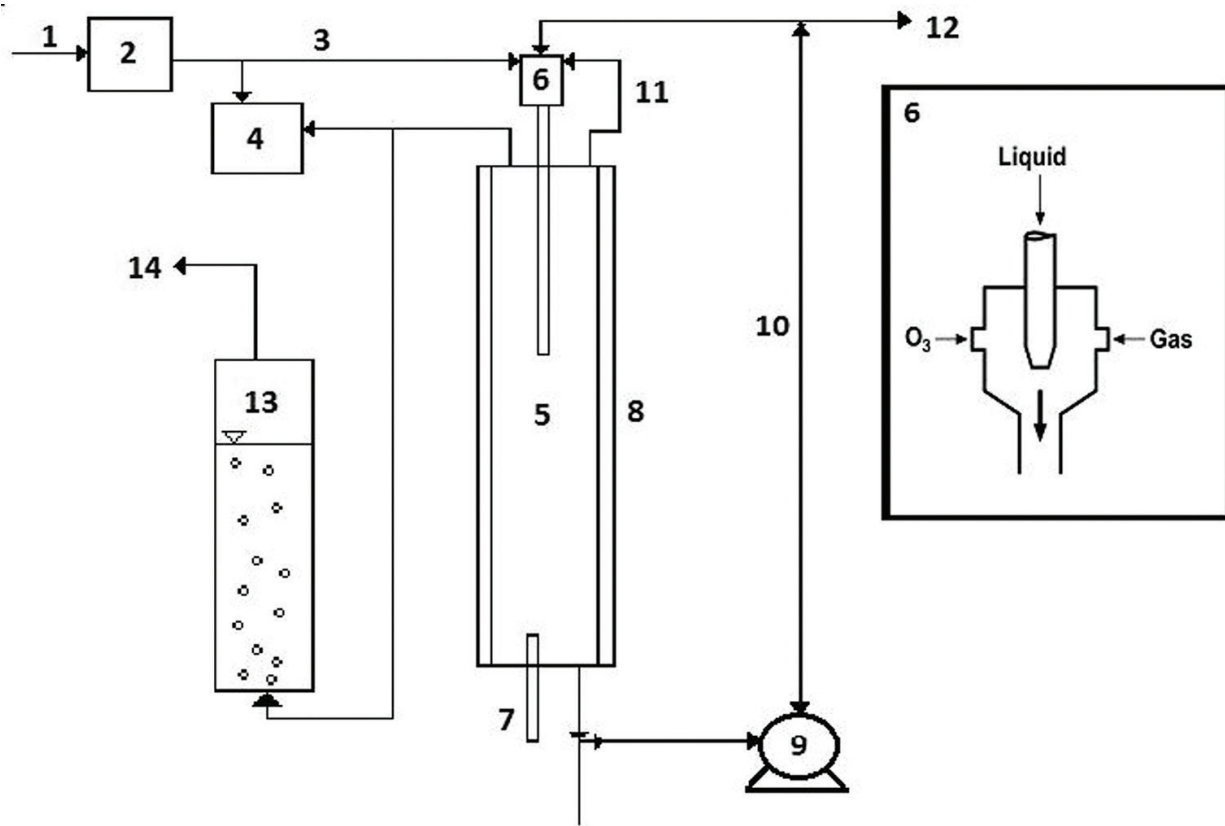
### 2.1. Experimental equipment

Ozonation trials were performed in a laboratory ozonation reactor. A schematic of the ozonation apparatus is illustrated in **Figure 1** [13]. Ozonation jet-loop reactor was operated in batch mode with regard to wastewater and in continuous mode with regard to gas. Active volume of the reactor was 3.5 L. The treated wastewater was transferred into ozonation reactor before starting operation of the reactor. A membrane pump was used to maintain external circulation of liquid reaction mixture. Pulsation of recirculated external flow was minimized with diaphragm pulsation damper (SERA 721.1, Seybert & Rahier, Immenhausen, Germany). Ozone was generated using a Lifetech generator with maximum production of 5 g h<sup>-1</sup>. Ozone was generated at 50% of the maximum ozone generator's power and maintaining continuous oxygen flow of 60 L h<sup>-1</sup>. A mixture of O<sub>3</sub> and O<sub>2</sub> was injected into a wastewater sample through a Venturi ejector. At the same time, the ejector sucked the mixture of O<sub>3</sub> and O<sub>2</sub> from the reactor headspace.

This, together with external circulation, should improve the efficiency of ozone utilization in the ozonation reactor. Pen-Ray UV lamp with wavelength 254 nm was used to generate hydroxyl radicals in the reactor. The outfall of reaction-gas mixture was transported into a destruction glass column by a fine-bubble porous distributive device. The destructive reactive column contained a potassium iodide solution. The active volume of the destructive reactive column was 1.0 dm<sup>3</sup>. An excess O<sub>3</sub> was destructed in this device [13].

### 2.2. Analytical methods

For determination BTEX compounds in model wastewater, gas chromatography was used with MS detector in connection with headspace autosampler.



**Figure 1.** Schematic scheme of the ozonation equipment. 1— $O_2$ , 2— $O_3$  generator, 3— $O_2/O_3$  gas flow, 4—UV detector of  $O_3$ , 5— $O_3$  reactor, 6—Venturi nozzle, 7—UV source, 8—tempering jacket, 9—recirculation pump, 10—external reaction mixture recirculation loop, 11—sucking of gas from head space, 12—sampling device, 13—outfall of gas, 14—destruction device for residual  $O_3$ .

Gas chromatographic method after static headspace extraction was used for quantification of the BTX in water. Headspace part was analyzed by gas chromatography with MS detector (Agilent Technologies 7890A GC Systems). All substances used for preparation of model wastewater and standard stock solutions were purchased from Dr. Ehrenstorfer.

### 2.3. Processing of experimental data

Desorption of volatile pollutants from water solution is proportional to its concentration in water solution

$$\frac{dS}{dt} = -k \cdot S \quad (1)$$

where  $k/h^{-1}$  is desorption rate.

Experimental data of BTX degradation were fitted by the zero (Eq. (2))- , first (Eq. (3))- , and second (Eq. (4))-order reaction kinetic models. For a batch reaction system, under the assumption of a constant reaction volume, the following relationships were obtained.

$$S_t = S_0 - k_0 t \quad (2)$$

$$S_t = S_0 \exp(-k_1 t) \quad (3)$$

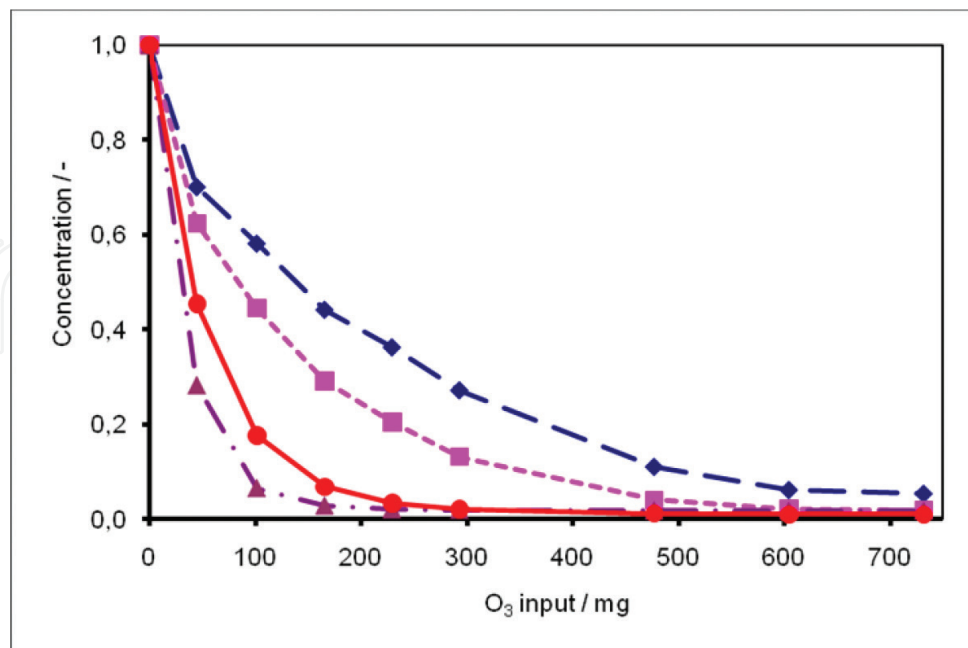
$$S_t = \frac{S_0}{(1 + S_0 k_2 t)} \quad (4)$$

where  $S_t$  (g m<sup>-3</sup>) stands for the content of BTX substances in model wastewater in time  $t$ ,  $S_0$  (g m<sup>-3</sup>) is the beginning content of BTX substances in model wastewater,  $k_0$  (g m<sup>-3</sup> h<sup>-1</sup>),  $k_1$  (h<sup>-1</sup>), and  $k_2$  (g<sup>-1</sup> m<sup>3</sup> h<sup>-1</sup>) are the rate constants for the kinetics of the zero, the first, and the second reaction order, respectively [13].

The grid search optimization method was applied to calculate values of parameters of the used mathematical models. The objective function was defined as the sum of squares between the measured and calculated values of BTX components divided with the number of measurements reduced by the number of estimated parameters [13, 14].

### 3. Results of the work

The removals of studied compounds with the ozonation time are presented in **Figure 2**. The initial concentrations of benzene, toluene, o-xylene, and p-xylene were 800, 1600, 800, and 600 µg l<sup>-1</sup>, respectively. From **Figure 2**, it is obvious that for all studied pollutants measured, the highest removal rates were observed within the first 5 min of the process. The highest affinity of ozone was measured toward p-xylene (59.6% removal efficiency). On the other hand, the lowest treatment efficiency was measured for benzene (20%) within the same ozonation time.



**Figure 2.** BTX (dimensionless values) removal during ozonation of model wastewater (◆ benzene, ■ toluene, ▲ p-xylene, ● o-xylene).

$t$ [min]	Benzene [%]	Toluene [%]	p-Xylene [%]	o-Xylene [%]
5	20.1	26.4	59.6	44.4
20	53.9	68.3	85.7	86.4
60	84.6	89.6	90.0	89.9

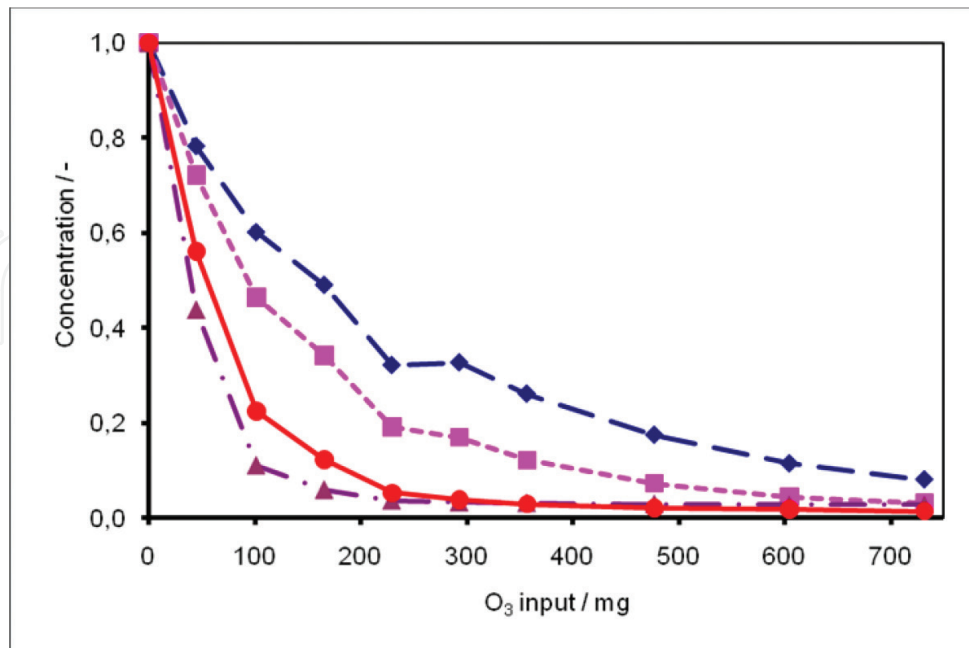
**Table 1.** Removal efficiency values of BTX compounds with ozone.

The treatment efficiencies of BTX components increased with the increase of ozonation time [15]. The highest treatment efficiency was observed for p-xylene (81.3% during the first 20 min of ozonation). The second highest treatment efficiency (72.2%) was measured for o-xylene. Final removal efficiencies of BTX constituents were observed in the range from 86.4 to 90%. The values of removal efficiency during  $O_3$  process are summarized in **Table 1**.

The best fit of experimental degradation data of all studied pollutants was obtained by the first-order kinetic model.

The removals of studied BTX by  $O_3/UV$  treatment time are presented in **Figure 3**. It is obvious that the removal rates of BTX by  $O_3/UV$  process are higher in comparison with the removal rates observed with ozone alone. This is confirmed also by the values of removal efficiency given in **Table 2**.

Comparisons of p-xylene and o-xylene removals using  $O_3$  only for oxidation and  $O_3/UV$  treatment of model wastewater are presented in **Figures 4** and **5**, respectively. Slightly higher removal rates of these two pollutants were measured when treated with ozone alone.

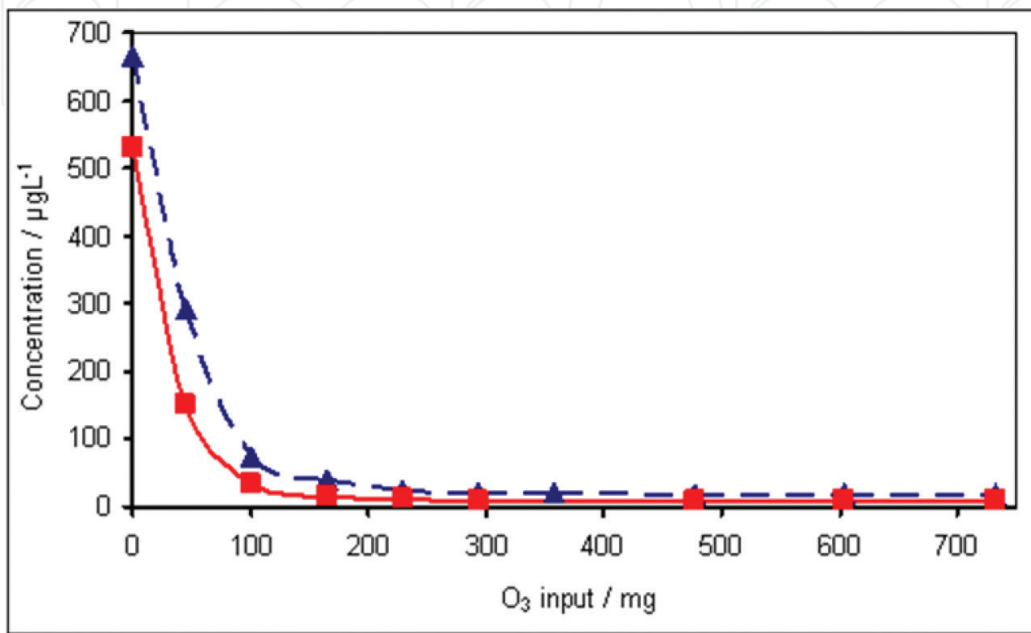


**Figure 3.** BTX (dimensionless values) removal by  $O_3/UV$  treatment of model wastewater (◆ benzene, ■ toluene, ▲ p-xylene, ● o-xylene).

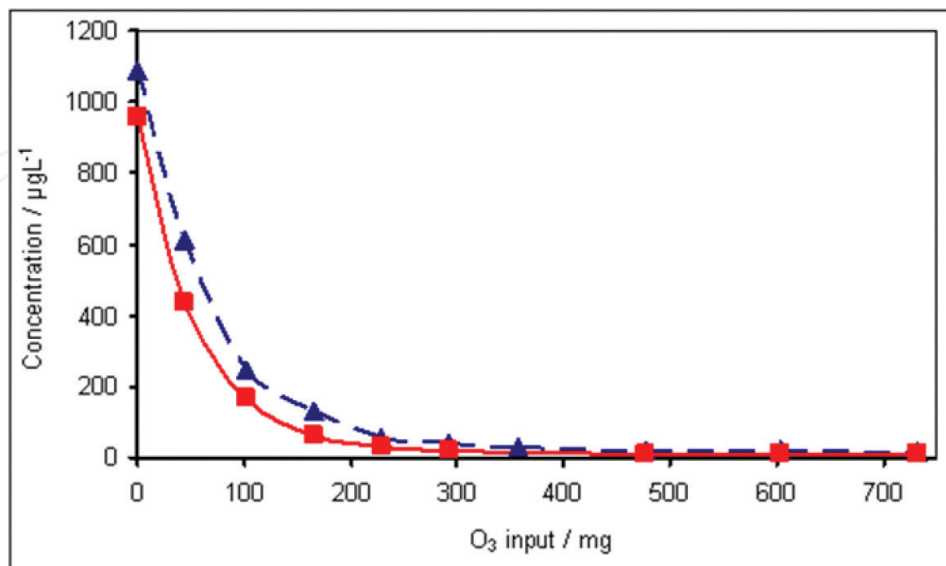


<i>t</i> [min]	Benzene [%]	Toluene [%]	p-xylene [%]	o-xylene [%]
5	21.7	27.9	56.9	43.9
20	67.8	80.8	96.3	94.7
60	91.9	96.9	97.2	98.6

**Table 2.** Removal efficiency values of BTX compounds by O<sub>3</sub>/UV process.



**Figure 4.** p-Xylene concentration profiles during O<sub>3</sub> and O<sub>3</sub>/UV treatment of model wastewater (■ p-xylene-O<sub>3</sub>, ▲ p-xylene-O<sub>3</sub>/UV).



**Figure 5.** o-Ylene concentration profiles during O<sub>3</sub> and O<sub>3</sub>/UV treatment of model wastewater (■ o-xylene-O<sub>3</sub>, ▲ o-xylene-O<sub>3</sub>/UV).



However, insignificant differences in the removal rates and treatment efficiencies follow from the treatment of the investigated BTX compounds using  $O_3$  and  $O_3/UV$ . The data presented above represent overall removal of pollutants during  $O_3$  or  $O_3/UV$  treatment, i.e., an effect of stripping of pollutants content is also included in the data.

The effect of gas stripping of the investigated BTX compounds at the conditions of ozonation and  $O_3/UV$  trials was also studied. Volatility of substances depends on the size of molecules as well as on the vapor pressure [16]. With the increase of the molecular weight, the solubility of substance in water decreases. Important factor influencing solubility in water is hydrophobicity of substances. Solubility in water decreases with increase hydrophobicity of substance [17]. The information on hydrophobicity gives octanol-water partitioning coefficient value. Evaporation of substances correlates with vapor pressure [17] and is strongly influenced by temperature and the pressure of the system [18].

Volatility of substances can be quantified by the values Henry low. With the increase of the Henry's constant value, solubility of substance in water decreases. The values of basic physico-chemical properties of BTX components are given in **Table 3** [19].

Comparison of o-xylene, p-xylene, benzene, and toluene concentration profiles measured during the stripping only and ozonation treatment of model water are presented in **Figures 6–9**, respectively. As it was already mentioned, 10 min of ozonation corresponds to input of 45 mg  $O_3$  per liter of active volume of the jet-loop ozonation reactor.

It is obvious from the presented results that stripping can significantly contribute to the removal of investigated compounds during the ozonation and  $O_3/UV$  treatments. The higher contribution of stripping to the overall removal of the component during the ozonation is for benzene. This observation correlates very well with the physical properties of the different components (**Table 3**). Similar results were obtained for toluene ( $36.8 \mu\text{g L}^{-1} \text{min}^{-1}$ ) in comparison to benzene ( $32.0 \mu\text{g L}^{-1} \text{min}^{-1}$ ).

	Benzene	Toluene	o-Xylene	p-Xylene
Molecular weight[ $\text{g mol}^{-1}$ ]	78.11	92.13	106.16	106.16
Water solubility[ $\text{mg L}^{-1}$ ]	1700	515	175	198
Vapor pressure(at $20^\circ\text{C}$ ) [kPa]	12.6923	3.7863	0.8799	0,8799
Boling point[ $^\circ\text{C}$ ]	80.1	110.6	144.4	138.4
Octanol-water partition coefficient (at $20^\circ\text{C}$ )[log Kow]	2.13	2.69	2.77	3.15
Henry's law constant(at $25^\circ\text{C}$ ) [ $\text{kPa m}^3 \text{mol}^{-1}$ ]	0.55	0.67	0.50	0.71
AA-EQS [ $\mu\text{g L}^{-1}$ ]	10 <sup>a</sup>	100 <sup>a</sup>	10 <sup>b</sup>	10 <sup>b</sup>

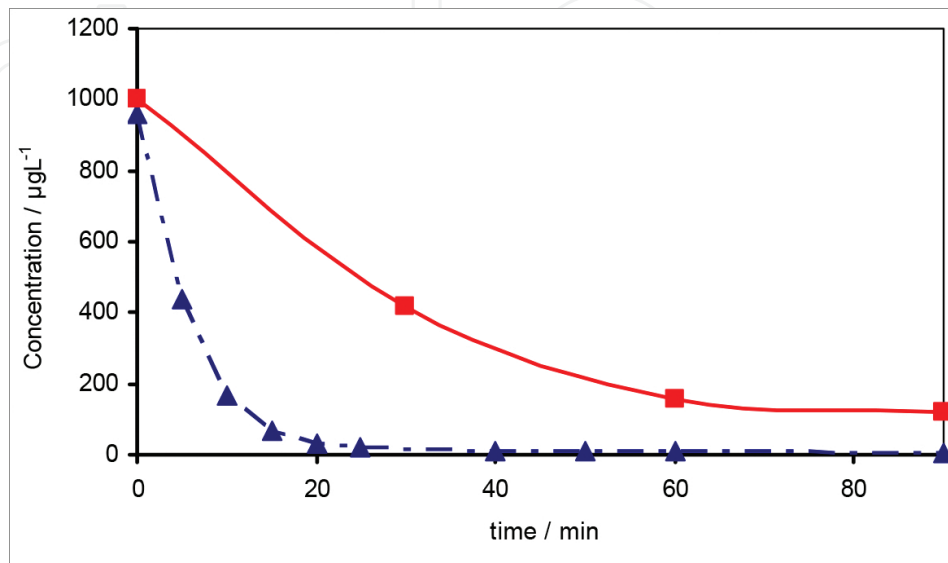
Notes: AA-EQS, Annual Average Value of Environmental Quality Standard (EQS).

<sup>a</sup> European Commission (2008) List of EQS [20].

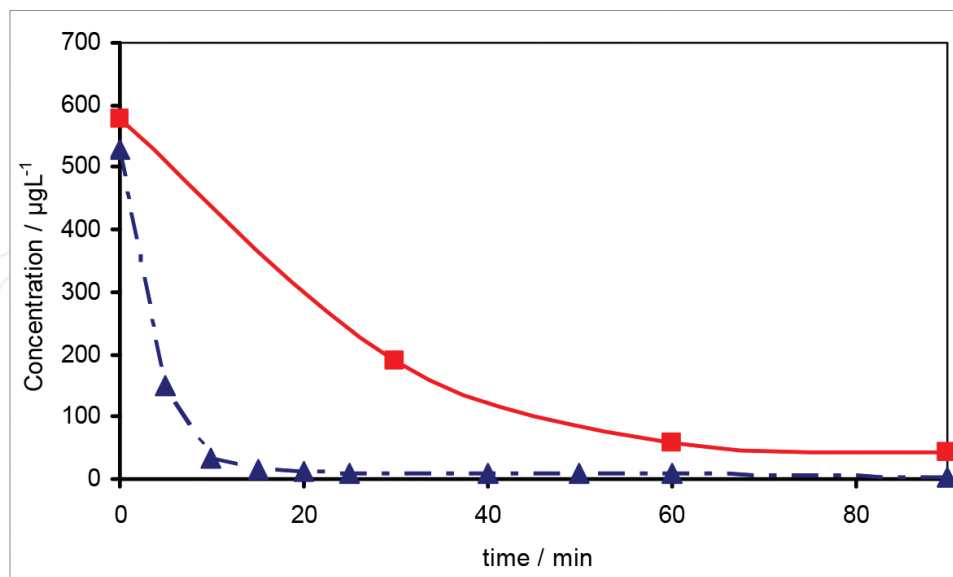
<sup>b</sup> Regulation of the Slovak Government [21] List a EQS of total xylenes of  $10 \mu\text{g L}^{-1}$ .

**Table 3.** Basic characteristics for studied organic components of BTX.

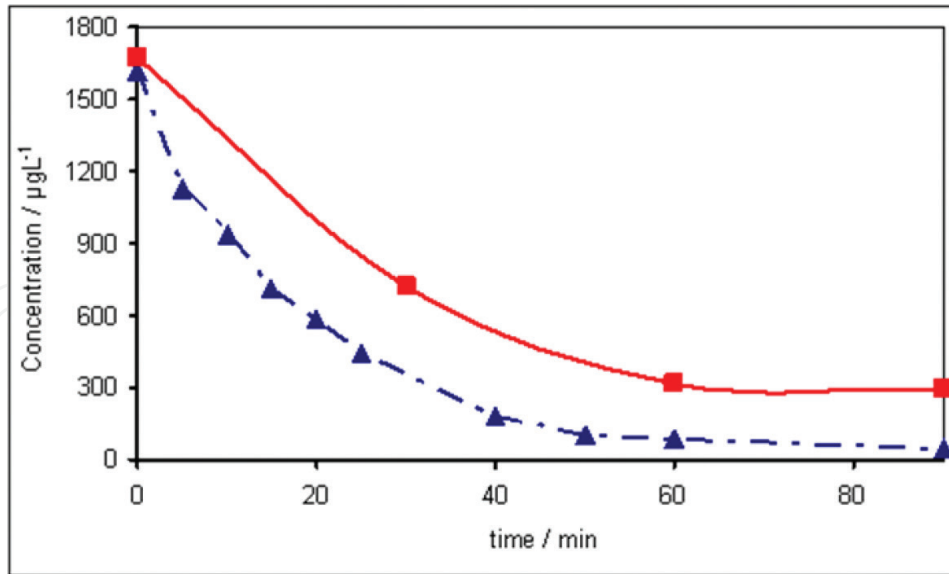
Concentration profiles of the studied BTX substances during ozonation, i.e., after excluding removal of the compounds by stripping process during ozonation of the model wastewater, are presented in **Figure 10**. In other words, the values plotted in **Figure 10** were obtained by subtraction of the concentrations of individual components due to stripping by oxygen flow from the total concentrations obtained during ozonation of the model wastewater.



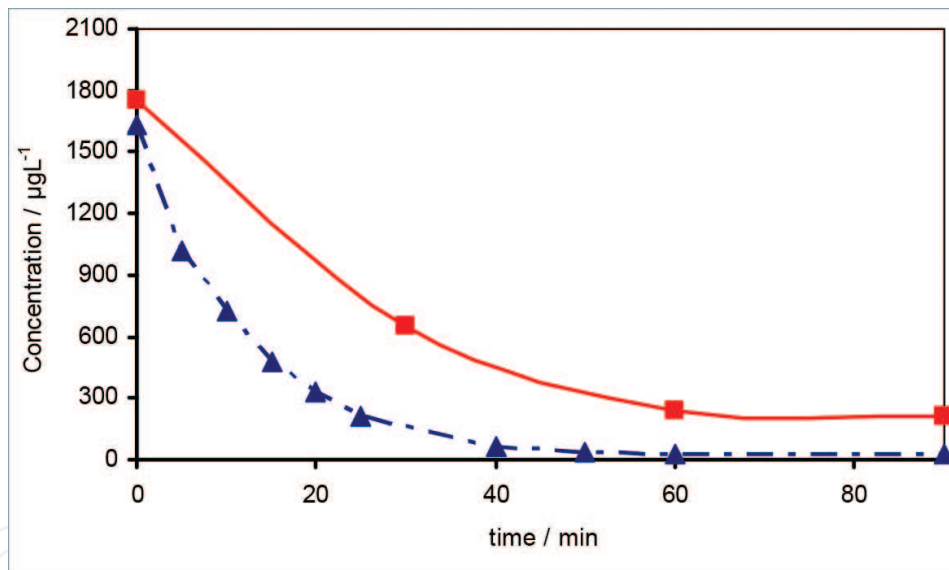
**Figure 6.** o-Xylene concentration profiles during stripping and ozonation of model wastewater (■ o-xylene-stripping, ▲ o-xylene-ozonation).



**Figure 7.** p-Xylene concentration profiles during stripping and ozonation of model wastewater (■ p-xylene-stripping, ▲ p-xylene-ozonation).



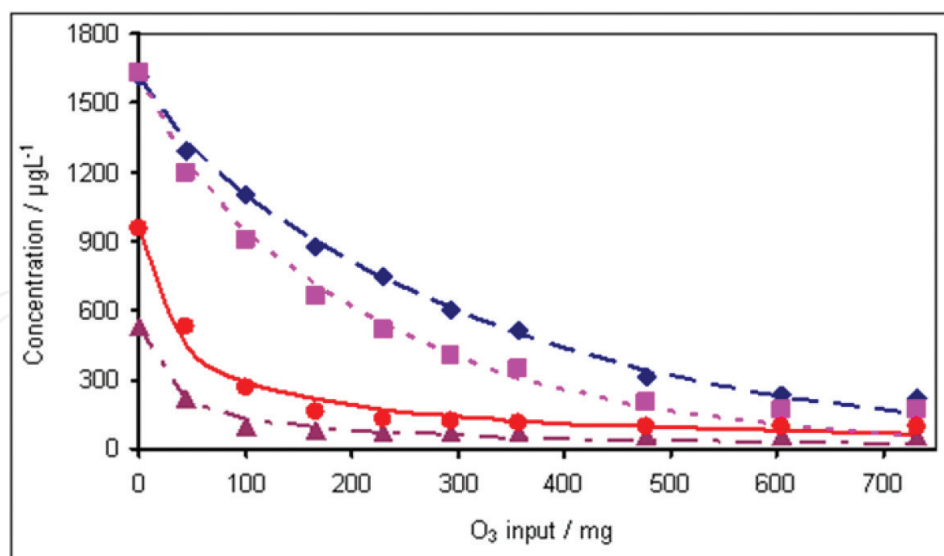
**Figure 8.** Benzene concentration profiles during stripping and ozonation of model wastewater (■ benzene-stripping, ▲ benzene-ozonation).



**Figure 9.** Toluene concentration profiles during stripping and ozonation of model wastewater (■ toluene-stripping, ▲ toluene-ozonation).

Ozonation and stripping experimental trials were performed at the same operational conditions except presence of ozone in the system for stripping tests. The removal efficiencies for BTX components due to ozone oxidation of the model wastewater sample are given in **Table 4**.

Experimental data were processed using kinetic models to evaluate an order of the reaction. The calculated concentration profiles obtained by kinetic models corresponded to the best fit of experimental data (**Table 4**) for ozone oxidation, i.e., after excluding contribution of stripping to overall BTX concentrations during ozonation.



**Figure 10.** Experimental (◆ benzene, ■ toluene, ▲ p-xylene, ● o-xylene) and calculated (lines) BTX concentration profiles during ozonation of model wastewater.

<i>t</i> [min]	Benzene [%]	Toluene [%]	p-Xylene [%]	o-Xylene [%]
5	20.1	26.4	59.6	44.4
20	53.9	68.3	85.7	86.4
60	84.6	89.6	90.0	89.9

**Table 4.** Removal efficiency values of BTX compounds by O<sub>3</sub> (stripping excluded).

<i>t</i> [min]	Benzene [%]	Toluene [%]	p-Xylene [%]	o-Xylene [%]
5	21.7	27.9	56.9	43.9
20	67.8	80.8	96.3	94.7
60	91.9	96.9	97.2	98.6

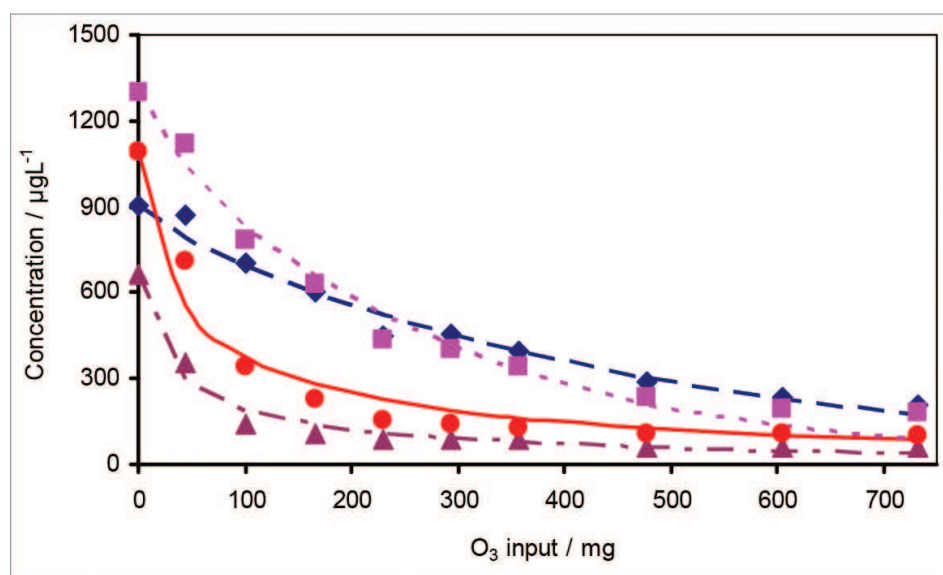
**Table 5.** Removal efficiency values of BTX compounds by O<sub>3</sub>/UV.

The highest removal rates were observed during the first 5 minutes of ozonation for all investigated pollutants. However, there is approximately 10% difference in removal efficiency (Table 1) and it is caused by ozonation reaction only (Table 4).

The best fit of experimental degradation data of benzene and toluene was obtained by the first-order kinetic model. On the other hand, the second-order kinetic model was more appropriate for description degradation of xylenes. The rate constant values and the values of the correlation coefficient  $r_{xy}$  corresponding to treated kinetic data after subtracting volatilized portions due to oxygen stripping are summarized both for O<sub>3</sub> and O<sub>3</sub>/UV treatments in Table 6.

Pollutants		$k_1$ [h <sup>-1</sup> ]	$k_2$ [g m <sup>-3</sup> h <sup>-1</sup> ]	
Benzene	O <sub>3</sub>	$3.92 \times 10^{-2}$	–	0.9964
	O <sub>3</sub> /UV	$5.42 \times 10^{-2}$	–	0.9770
Toluene	O <sub>3</sub>	$5.55 \times 10^{-2}$	–	0.9885
	O <sub>3</sub> /UV	$4.56 \times 10^{-2}$	–	0.9977
o-Xylene	O <sub>3</sub>	–	$2.38 \times 10^{-4}$	0.9793
	O <sub>3</sub> /UV	–	$1.78 \times 10^{-4}$	0.9636
p-Xylene	O <sub>3</sub>	–	$5.78 \times 10^{-4}$	0.9839
	O <sub>3</sub> /UV	–	$3.82 \times 10^{-4}$	0.9980

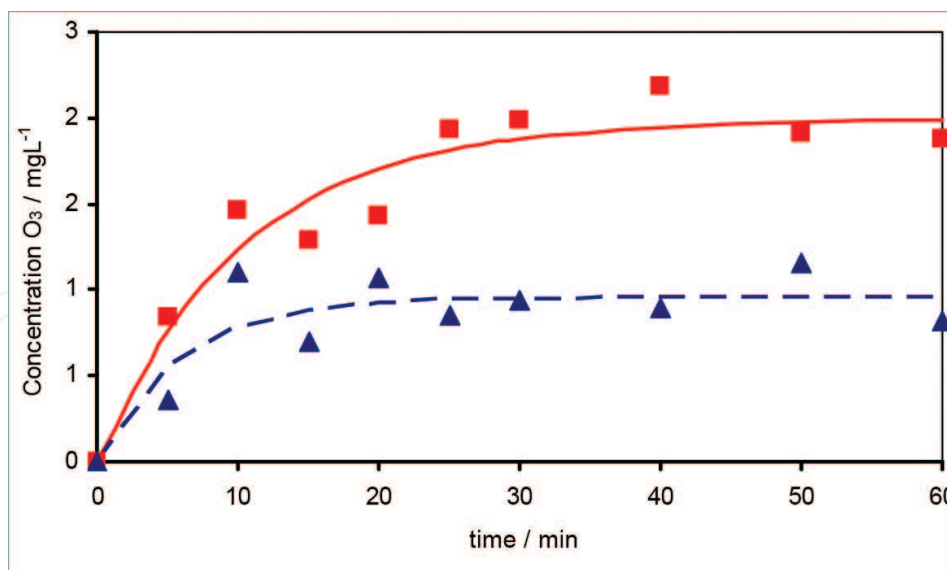
**Table 6.** Kinetic parameters and statistical characteristics—stripping excluded.



**Figure 11.** Experimental (◆ benzene, ■ toluene, ▲ p-xylene, ● o-xylene) and calculated (lines) BTX concentration profiles during O<sub>3</sub>/UV treatment of model wastewater.

Similar concentration profiles for the studied BTX compounds during the O<sub>3</sub>/UV treatment of the model wastewater are presented in **Figure 11**. The values presented in **Figure 11** were also obtained by subtraction of concentrations of individual components caused by gas stripping. The calculated concentration profiles were also obtained by kinetic models corresponding to the best fit of experimental values (**Table 6**) for O<sub>3</sub>/UV treatment, i.e., after excluding contributions of stripping of individual compounds. The removal efficiencies values are given in **Table 5**.

Removal efficiencies are very close to those given in **Table 2**. In the case of o-xylene and p-xylene, the best fit was obtained by the second-order kinetic model (**Table 6**). The first-order kinetic models for benzene and toluene may indicate significant influence of gas stripping on total removal of these compounds from solution and the process is probably determined by physical phenomena rather than chemical.



**Figure 12.** Ozone concentration profiles in the jet-loop reactor during treatment of model wastewater with O<sub>3</sub> (■) and O<sub>3</sub>/UV (▲).

**Figure 12** illustrates ozone concentration profiles in the jet-loop reactor during treatment of model wastewater with O<sub>3</sub> and O<sub>3</sub>/UV.

Lower ozone concentration in water or higher ozone consumption in the system is obvious for O<sub>3</sub>/UV treatment. Lower experimental ozone concentration for O<sub>3</sub>/UV reaction system can be explained by decomposition and hydroxyl radicals formation. However, insignificant increase of removal rates was observed as a result of radical reaction mechanism. On the other hand, the values of kinetic constant for all compounds (**Table 6**) are slightly higher when ozone alone was applied in comparison to O<sub>3</sub>/UV treatment. Thus, the higher removal rates for ozone treatment of BTX were observed in comparison to the O<sub>3</sub>/UV treatment process.

#### 4. Conclusion

Effect of ozone and O<sub>3</sub>/UV treatments on BTX components were investigated in this study. Investigation of stripping of volatile substances was also performed.

The highest removal rates for all investigated BTX components were observed during the first 5 min of processing for both ozonation and O<sub>3</sub>/UV treatment processes.

Ozone showed the highest affinity to p-xylene. The lowest removal efficiency was measured for benzene. Treatment efficiencies above 90% were observed for all investigated pollutants after 40 min of ozonation. Longer ozonation time resulted in very low enhancements of removal efficiencies of both ozonation and O<sub>3</sub>/UV treatment processes.

Application of O<sub>3</sub>/UV treatment had no significant effect in comparison with ozonation only, particularly for benzene. In case of o-xylene and p-xylene removal efficiencies, over 90% were

observed after 20 min of the process. Forty minutes of the process were needed for more than 90% removal efficiency of ethylbenzene.

Due to high volatility of BTX components their removal from liquid phase can be significantly influenced by stripping. According to physical characteristics, the highest stripping can be expected for benzene and toluene [15].

From the processing of experimental data by simple kinetic models [13, 14], removal of o-xylene and p-xylene was best achieved by the second-order kinetic model. On the other hand, best fit of experimental data for benzene and toluene was obtained using the first-order kinetic model.

From the result of the study one can conclude that ozonation is a prospective process and a promising procedure for the removal of BTX components from aquatic environment. However, further research should be performed to enhance process efficiency and to study the impact of reaction intermediates and products on aquatic ecosystem.

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