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# The study of optimal ozone dose for industrial ozone installation in textile wastewater reuse

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**ABSTRACT**

Textile production is one of the most water-consuming industries. Closing the water loop by treatment and recycling is highly desirable in this regard. However, textile wastewater treatment is not standard on an industrial scale, and the Polish textile factory Bilinski is one of very few with a functional wastewater recycling system. This study investigates the operational conditions of the industrial ozone system at Bilinski Co., (Konstantynow Lodzki, Poland). An ozone reaction column from Thies GmbH (Germany) with a volume of 7 $m^3$ was used to determine the optimal ozone dose in a closed water loop for textile wastewater reuse. An ozone measurement system from BMT Messtechnik GmbH (Germany), a 965OG ozone concentration metre, and a DH7 dehumidifier were used to determine the ozone in the gas phase. The applied ozone dose and transferred ozone dose (TOD) were calculated based on these data. Three values of TOD, 62.9, 37.7, and 27.0 g/$m^3$, were used for wastewater. A colour reduction of 97% was achieved after 8, 9, and 11 min of treatment. The test showed that the higher the TOD was, the shorter the treatment. Consequently, the average optimal ozone concentration was 32.4 ± 5.5 g/$m^3$. However, this value was roughly estimated because of the industrial scale of the process. It can be assumed that after transferring this ozone concentration, 97% colour removal is possible. Finally, the faster the optimal ozone concentration was transferred, the shorter the treatment time. The experiment showed how operational conditions could be investigated in a high-volume industrial system.

*Keywords:* Industrial ozone system; Textile wastewater; Wastewater recycling; Ozone measurements

## 1. Introduction

Apart from global industrialization, the problem of international environmental protection seems to be at the centre of public interest. As far as massive industrial production allows everyday goods to be delivered to everyone, postproduction waste should be limited. However, unfortunately, this is not the case. Textiles and fashion are some of the largest markets for commercial goods, and in this way, the textile branch is one of the more severe polluters. According to a recent study by Islam [1], textile production led to 20% of the overall freshwater contamination. At the same time, the greenhouse emissions estimated for textile production were as high as 10% on the global scale [1]. Moreover, 5% of the landfill area is occupied by textile waste [1]. These alarming numbers should inspire action against producing textile pollution. Primarily, wastewater emissions should be restricted because of the enormous water consumption in the textile branch. Surprisingly, even as much as 20% or more of textile wastewater is released directly into the environment without treatment [1], and this should be changed.

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Textile wastewater is a relatively resistant medium to be treated. As it is postindustrial waste, it contains several chemical impurities from the intense processing of raw textile substrates. Therefore, textile wastewater characteristics, such as colour, high salinity, and pH, are far different from neutral, organic, and nonorganic wastewater, resulting in low biological oxygen demand (BOD). The chemical oxygen demand (COD) of effluent from textile plants was found to be between 150 and 15,000 (depending on the type) with a very low BOD, such as 20 and 300 mg·$O_2$/dm$^3$ [2]. These characteristics imply that this wastewater is hardly biodegradable and demanding to treat for biological treatment plants. Research by GilPavas et al. [3] showed that the use of ozone improves the biodegradability of textile wastewater.

Thus, ozone treatment is a promising tool in textile wastewater treatment. Since molecular ozone has a high oxidative potential (2.08 V), and oxygen species, for example, hydroxyl radicals, can willingly support ozonation with a potential as high as 2.8 V [4], many recalcitrant textile pollutants can be removed. Consequently, ozone is a favourable solution used in textile wastewater purification. Numerous literature reports have shown how beneficial ozone could be when used for dye removal and the more complete purification of real textile effluents [2,5–11]. However, experiments run on an industrial scale or at least on a pilot scale are rather limited in published papers. Even though experiments have been performed on real wastewater from industrial plants, the maximal operating volumes, such as 0.012 m$^3$, in Cardoso et al. study [12] or the 0.018 m$^3$-volume pilot-scale installations proposed by Somensi et al. [13], were closer to the laboratory scale than the industrial scale. Correspondingly, Somensi et al. [13] showed interesting results after the ozonation of real textile wastewater originating from cotton and polyester dyeing. The experiments showed an enhancement in biodegradability and a reduction in toxicity towards *Vibrio fischeri*. The author recommended ozonation as a pretreatment before biodegradation in this work. One of the most interesting studies on pilot-scale ozone installation was presented by Ma et al. [14]. A high-volume, 2.93 m$^3$, reactor was used for catalytic ozone treatment accompanied by iron shavings as a source of catalyst. However, this work did not report the directly transferred ozone dose. The authors showed 70% COD removal, purportedly due to the catalytic production of reactive oxygen species and effective in situ coagulation originating from the iron [14].

An excellent example of industrial wastewater ozonation in wool dyeing, and finishing was provided by Baban et al. [15]. Ozonation was preceded by biotreatment in this example. Colour removal was not the only factor in this study, it was focused primarily on toxicity. The authors showed a 92% toxicity decrease after dosing $5.8 \times 10^4$ mg/m$^3$ of ozone. However, ozonation was found to only slightly affect COD removal [15]. Similar observations were made by Paździor et al. [16], who investigated general textile wastewater mixed from many textile processing operations. Paździor et al. [16] mostly investigated toxicity removal by ozonation, which was shown to be dependent on biotreatment. Alternatively, Shaikh et al. [17] proposed textile wastewater ozonation just after the dyeing process, which took place inside the dyeing machine without transporting wastewater elsewhere. However, the issues to consider include how to cope with the safety risks of ozone and the occupation of the dyeing machine for the time needed for ozonation, which is an extra cost. These issues were not discussed in the paper [17]. Furthermore, Wang et al. [18] employed ozonation supported by UV as a mature technique for textile wastewater treatment under industrial conditions. The object of the study was pretreated textile wastewater from an onsite textile treatment plant. Despite not having a high COD, which was equal to 242 mg·$O_2$/dm$^3$, the wastewater was shown to be highly contaminated with salts, which was problematic. The study examined the benefits of an ozone-based pilot-scale installation working under pressurized conditions to produce microbubbles of $O_3$, which resulted in 85% and 43.2% colour and COD removal, respectively. The authors discussed the advantage of their ozonation technique over the Fenton process in terms of membrane fouling in the subsequent RO operation. Despite the relevant results of the study, it must be noted that the capacity of the pilot was only 0.011 m$^3$, and even if the author showed that they used between 20 and 200 g of ozone gas per during their experiments, it could not be directly correlated with applied ozone dose (AOD) or transferred ozone dose (TOD). Therefore, the actual ozone dose, applied or transferred could not be estimated [18].

As indicated by this short literature overview, there is still a gap in knowledge regarding ozonation at a full industrial scale. To the best of the authors' knowledge, no experiment has been performed under industrial conditions to develop a practical method for investigating the optimal ozone dose.

The objective of the present study was to perform a comprehensive industrial-scale ozonation experiment to examine the ozone dose necessary for real textile wastewater treatment. The object of the treatment was textile wastewater supplied directly from a treatment plant on an industrial scale.

The investigation of colour and COD removal provided information on the efficiency of ozonation, which was compared to that of other stages of the treatment (biotreatment, nanofiltration, adsorption). The TOD values were examined in terms of operational conditions. *Vibrio fischeri* was used as a testing organism in toxicity assessment, allowing us to compare ozonation with other stages of the treatment.

## 2. Experimental set-up

### *2.1. Chemicals and wastewater*

#### *2.1.1. Chemicals*

The reagents for analysis were used as follows: cuvette tests for analysis LCK 514 (COD) were supplied by Hach Lange (Poland). Silver nitrate was supplied by Chempur (Piekary Slaskie, Poland) for Mohr's titration. Analytes for the toxicity of Microtox™ Modern Water (dried-bacteria inoculum, osmotic adjusting solution, dilution solution, reconstitution solution by modern water) were supplied by Tigret (Poland).

*2.1.2. Wastewater*

Industrial textile wastewater taken directly from an industrial plant was investigated in this study. The wastewater was obtained from Bilinski factory of colour, one of the largest dyehouses in Europe (Poland). This textile plant produces 30 tons of goods per day, resulting in daily wastewater emissions as high as $2.5 \times 10^3$ $m^3$. The factory has an onsite wastewater treatment plant. Effluent is selected to be cleaned and recycled in this plant. Only selected wastewater streams consisting of wastewater from rinsing, acidifying baths, and washings undergo treatment in this plant. The following steps are involved in the system: filtration and heat recovery (1), biological treatment (2), membrane filtration (3), ozonation (4), and adsorption (5). The wastewater is recovered at the production site after these operations as rinsing water and dying water. The wastewater obtained after each treatment step (1–5) was investigated, but that obtained after ozonation (4) was at the centre of interest.

The characteristics of the wastewater samples (1–5) are represented by the global indicators listed in Table 1. BOD was not included in the study because biodegradability was less significant as wastewater was recycled at the production site.

*2.2. Experiment*

After biological pretreatment (3), the wastewater stream underwent ozone treatment in the industrial installation. An ozone reaction column from Thies GmbH (Germany) with a volume of 7 $m^3$ (height 3.1 m, diameter 1.7 m, with a continuous flow of 10 $Nm^3/h$ (calculated according to the DIM 1343 standard.), was used to determine the optimal ozone dose in a closed water loop for textile wastewater reuse. An ozone measurement system from BMT, a 965OG ozone concentration metre, and a DH7 dehumidifier were used to determine the ozone in the gas phase The AOD and TOD were calculated based on these data as follows:

$$\mathrm{AOD} = \int_0^t \frac{Q_{\mathrm{gas}}}{V_{\mathrm{liquid}}} C_{O3}^{\mathrm{in}} dt \tag{1}$$

$$\mathrm{TOD} = \int_0^t \frac{Q_{\mathrm{gas}}}{V_{\mathrm{liquid}}} \left(C_{O3}^{\mathrm{in}} - C_{O3}^{\mathrm{out}}\right) dt \tag{2}$$

*2.3. Analytical methods*

The colour was determined with a GENESYS 180 spectrophotometer from Thermo Fisher Scientific (USA), and the colour removal was estimated as a global relative $A/A_0$ value, calculated as the area under the integral curve between 400 and 700 nm (visible region of the spectra). The spectra of the wastewater are not characterized by any specific absorption maxima in the visible area and no specific wavelength can be selected for color characterization. Therefore, it is more reliable to calculate the integral under the curve for the visible area to express the corresponding color. The methodology adopted for calculating the area under the spectrum is commonly used in the case of wastewater analysis where the spectrum contains many colored substances. In this way, we get rid of all colors and do not focus on one, as in the case of solutions containing one dye.

The COD indicator was detected by the above-mentioned LCK cuvette tests (HACH) measured with a DR 3900 spectrophotometer (HACH) in accordance with the manufacturer's procedure. The samples weren't diluted before the COD tests. The selected method can be used with chloride content up to 1.50 g/L, the tested samples contained a maximum of 1.33 g/L.

*2.4. Toxicity tests*

The marine luminescent bacteria *Vibrio fischeri*, known to be sensitive to a wide range of toxicants, was used as the testing organism in a toxicity assessment performed according to ISO 11348-3 [19]. The methodology was based on a Microtox Model 500 Analyser (Modern Water Inc., Newark, Delaware, USA). The tests were performed according to the 81.9% basic test protocol (MicrotoxOmni 4.2, Modern Water Inc.), which consisted of nine dilutions. The freeze-dried bacteria were reconstituted with water to obtain a stock suspension of test organisms, and kept at 5°C while performing the test. The reduction in light emission indicated the correction factor in this case. The toxicity factor was measured as $EC_{50}$, which means a 50% loss in the initial luminescence caused by the specific pollutant after 300 and 900 s of exposure. Then the $EC_{50}$ was converted into toxicity units (TU) according to Eq. (3), as follows:

$$\mathrm{TU} = \frac{100}{\mathrm{EC}_{50}} \tag{3}$$

## 3. Results and discussion

The experiments were performed on a full industrial scale. Therefore, it was important to identify the optimal operational conditions for the installation. Parameters, such as volumetric ozone flow, treatment time, temperature, AOD and TOD, were crucial for the process. Correspondingly,

Table 1
Industrial wastewater characteristics

| Indicators | Raw (0) | Filtration and heat recovery (1) | Biological treatment (2) | Membrane filtration (3) | Ozonation (4) | Adsorption (5) |
|---|---|---|---|---|---|---|
| $pH_{initial}$ | 9.34 | 9.09 | 9.07 | 9.0 | 8.39 | 8.43 |
| NaCl conc., $g/dm^3$ | 1.33 | 1.33 | 1.36 | 1.38 | 1.07 | 1.29 |
| COD, $mgO_2/dm^3$ | 1,412 | 1,097 | 669 | 223 | 85.6 | 197 |

the wastewater parameters, colour, COD, and toxicity were helpful in the estimation of the ozonation efficiency.

### 3.1. Operating conditions

The operating conditions for the process of advanced after treatment process (AAP) ozone installation (Thies GmbH, Germany) were determined by measuring the ozone concentration in a gas phase at the inlet and outlet of the reactor. By using Eqs. (1) and (2), two of the most important operating parameters, AOD and TOD, were calculated. Based on integral calculations, the transferred ozone dose could be estimated. Those values were the basis for determining the ozone concentration in the liquid when experimentally measuring the ozone in the gas phase.

In Fig. 1A, the transferred ozone dose, TOD, is shown. It can be seen that there is typical exponential growth up to the point of saturation, which was typical and equal to 600 s of treatment. At a quasis steady state, the reagent dose was equal to 60 g/m$^3$ of ozone transferred from the gas into the liquid. At the same time, the nominal ozone dose in the gas phase was 83 g/Nm$^3$ (Fig. 1B), and the volumetric flow was kept at 10 Nm$^3$/h. Fig. 1B shows that the ozone concentration began to increase 600 s after the treatment outlet. This means that a quasis steady state was achieved for this specific ozonation process, and the liquid phase was saturated with ozone.

The overall TOD in the process was 360 g of ozone transferred from the gas into the liquid during the whole process (1,800 s). However, the volume of the liquid in the reaction column was 4.9 m$^3$, which yielded an ozone concentration of 5.6 × 10$^4$ mg/m$^3$ [obtained from Eq. (1)]. Therefore, a quasis steady state concentration of ozone in the liquid phase could not be identified with TOD because the TOD was a cumulative value for the whole process.

Unfortunately, thermodynamic equilibrium between the gas and liquid phases was not especially conducive to the process because of the high temperature of the liquid, which was 40°C.

### 3.2. Colour removal and optimal ozone dose

Concerning textile wastewater, colour removal is the most informative parameter of the treatment process. In Fig. 2, the colour removal is shown for the above-mentioned ozonation process wherein 360 g of TOD was used. Visible colour was present until the time between 600 s (10 min) and 900 s (15 min). Based on absorbance measurements, visible colour was removed after approximately 900 s of ozonation, which could be observed directly by visual evaluation (photos of the samples are included as supplementary materials). In contrast to Fig. 2B, which represents the visible part of the spectra and, based on it, indicates the removal of colour, Fig. 2A shows that the colourless impurities, which absorb UV light, were only partially removed. Therefore, colour might not be the best indicator of more complete purification. However, colour removal is the most prevalent factor in textile wastewater treatment [2].

The next stage of the experiment was to find the optimal ozone dose for colour removal from textile wastewater. To study how the ozone concentration influences the decolorization process, three volumes of the same wastewater were tested. Tank loading of 30%, 50%, and 70% were studied, which resulted in wastewater volumes of 2.1, 3.5, and 4.9 m$^3$, respectively. The parameters of the ozone supply were always kept the same for safety reasons. When the

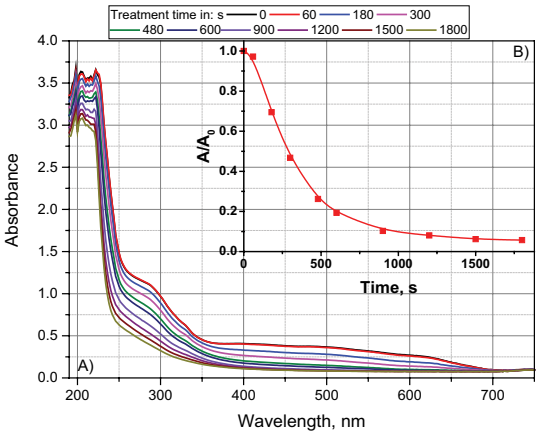


Fig. 2. Overall colour removal after application of 360 g of TOD, (A) removal of colourless impurities absorbing UV light and (B) removal of colour over time.

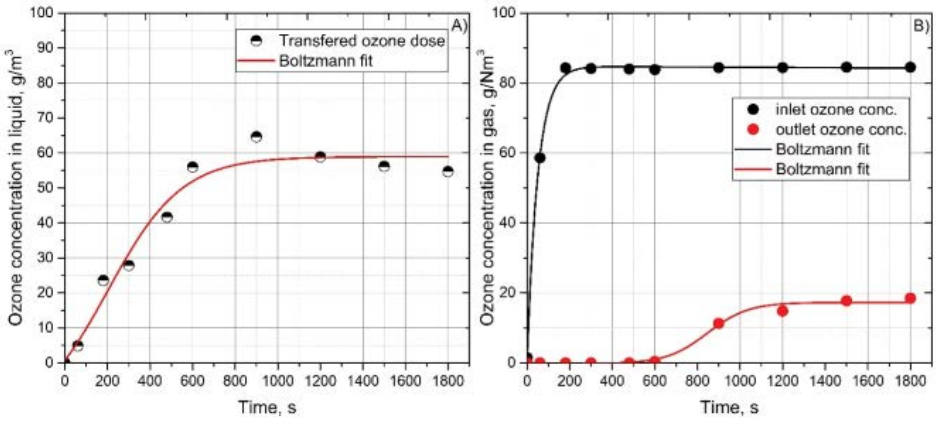


Fig. 1. Operating conditions for the ozonation process of AAP, (A) transferred ozone dose g/s and (B) ozone concentration in the gas phase at the inlet and outlet of the reactor.

filling was lower, the concentration had to be higher while the applied ozone was the same. In this way, we achieved three different ozone concentrations in the liquid for testing.

As shown in Fig. 3, colour was removed the fastest when the volume was the lowest, and at the same time, the transferred ozone concentration in g/m$^3$ was the highest. The apparent kinetic constant of decolorization was also the highest for the highest transferred ozone concentration (Fig. 3B). These results are in agreement with chemical reaction theory [4].

Table 2 shows the values of the ozone dose used to achieve 97% colour removal from treated wastewater. While more ozone, 132 g, had to be transferred into liquid when the volume of the wastewater, 4.9 m$^3$, was higher, this value was lower, 82 g, when the volume was lower (2.1 m$^3$). However, the values of the optimal ozone concentration were quite similar. After averaging, the optimal ozone concentration was 32.4 ± 5.5 g/m$^3$. After transferring this ozone concentration, 97% colour removal was possible. Consequently, the faster the optimal ozone concentration could be transferred, the shorter the treatment time needed. This experimental method for determining the optimal ozone dose might be considered inaccurate. However, it must be kept in mind that the whole experiment was run on an industrial scale. Because of safety reasons, ozone is a toxic gas, and little manipulation could be done on the ozone generator installation. While laboratory experiments can include a wide range of process parameters, industrial experiments need to be performed with a specific, sometimes much more simplified procedure.

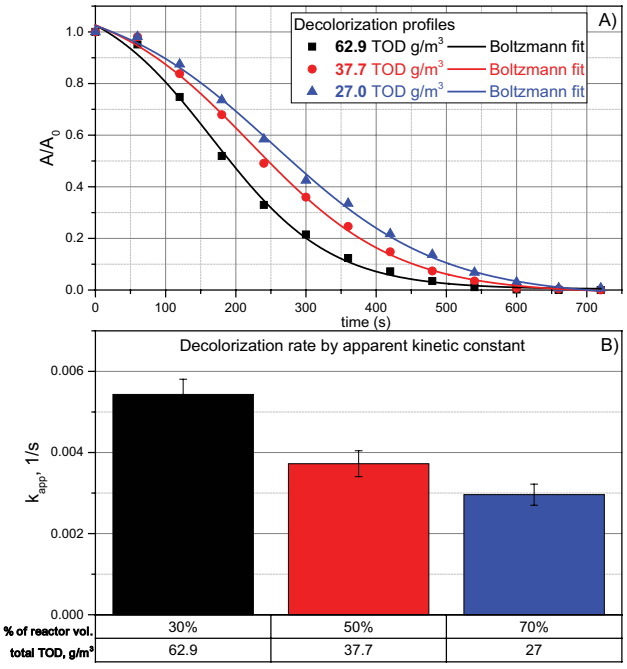


Fig. 3. (A) Colour removal profiles and (B) values of the apparent kinetic constant of decolorization (pseudo-first-order).

### 3.3. COD removal

The above-mentioned absorbance measurements indicated that visible colour was removed after approximately 900 s of ozonation (Fig. 2A). However, the UV region of these spectra was not much changed, suggesting a high residual content of colourless impurities. When this observation is in agreement with the COD removal results, it can be seen that COD was not efficiently removed during ozonation (Fig. 4A). As shown in Fig. 4A, the COD removal after transferring 360 g of ozone into 4.9 m$^3$ of wastewater was not very spectacular. Only 35% of COD was removed after 1,800 s (30 min). It must also be considered that efficient reduction was observed between 600 s (10 min) and 900 s (15 min). It is also worth mentioning that during that specific time, some small pH drop could be observed (Fig. 4B).

The results of COD decay (Fig. 3A) and those of colour removal (Fig. 2) suggested the two phases of the ozonation process. The first was colour removal, and the second was COD removal for as long as the colour was intense, which lasted for approximately 10 min, as the COD was not being removed. This was also correlated to the steady ozone concentration appearance (in the liquid). When the colour was removed, the ozone saturation phase started, which might have been caused by a fast reaction with the residual colour pollutants at the beginning of the process. After this stage, hardly degradable residuals remained and were slowly oxidized, but ozone was no longer effectively used.

### 3.4. Toxicity assessment

Fig. 5 shows the toxicity unit values, TU, measured for raw wastewater and the wastewater after each treatment step. Two exposure times, 300 and 900 s were tested. The TU was investigated based on $EC_{50}$ values measured towards the marine bacterium, *Vibrio fischeri*. Since TU is the factor classified in the quantified groups of exact limiting values, it is more straightforward to analyse than $EC_{50}$; consequently, the $EC_{50}$ was converted into TU [Eq. (3)]. Fig. 5 shows that the TU of the raw wastewater was equal to 36 and 41 after 300 and 900 s of exposure, respectively. According to the classification proposed by Persoone et al. [20], this result qualified the raw wastewater in Class IV, which means high acute toxicity in hazard classification for wastes discharged into aquatic environments. After the first treatment

Table 2
Process parameters tested in the experiment

| % Tank filling | Volume in m$^3$ | Total TOD g/m$^3$ | Colour removal | Treatment time, s | $O_3$ dose for 97% removal, g | $O_3$ conc., g/m$^3$ |
|---|---|---|---|---|---|---|
| 30 | 2.1 | 62.9 | | 480 | 81.6 | 38.8 |
| 50 | 3.5 | 37.7 | 97% | 540 | 108.9 | 31.3 |
| 70 | 4.9 | 27.0 | | 660 | 132.1 | 27.0 |

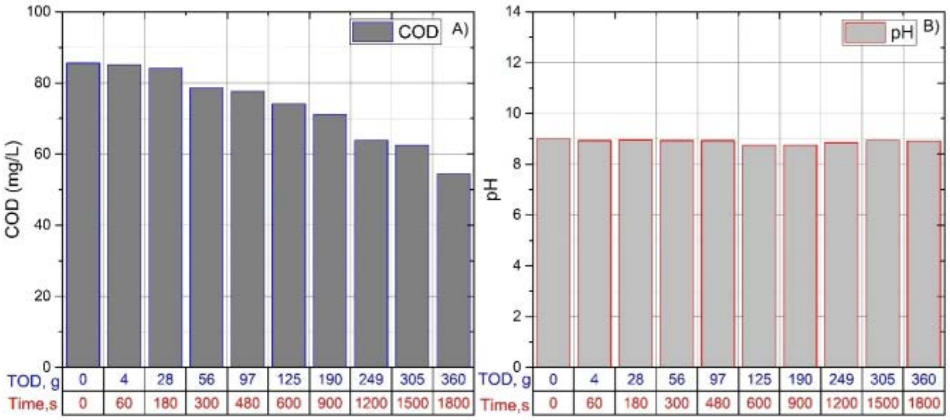


Fig. 4. (A) Chemical oxygen demand removal and B) pH change during ozonation.

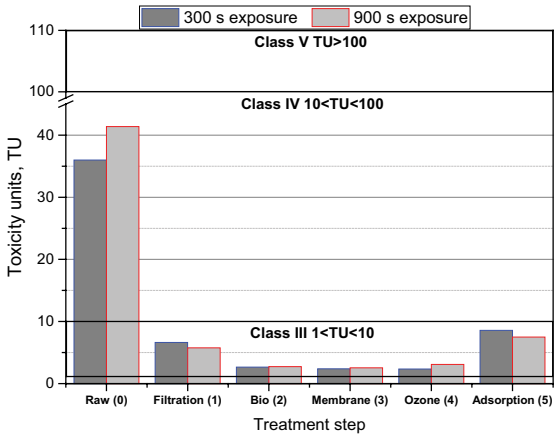


Fig. 5. Values of toxicity units before and after subsequent treatment steps with Persoone's toxic hazard scale [19].

step, filtration (1), the TU was decreased to the value ca. 6 units (300 and 900 s of exposure time), which qualified our wastewater as Class III with an acute level of toxicity [20]. The step of biotreatment (2) decreased the TU to the value ca. 2.5 unit, and it did not change much after the membrane filtration (3) and ozonation step (4). Fig. 5 shows a positive result in toxicity removal for treatment steps 1–4, including ozonation. However, the TU of treated wastewater was not lower than 1, and the threshold of Class II could not be reached. Even though the class of slight acute toxicity was not reached, the toxicity was reduced more than 15 times after the ozonation step. Surprisingly, the adsorption step, after ozonation, increased the TU from 2.3 to 8.6. This poor result was possibly caused by salt redistribution from the adsorbent recovery system into the treated water, resulting in higher toxicity. Conclusively, using Persoone's toxic hazard scale [20], the biotreatment combined with ozonation was determined as beneficial for toxicity removal.

## 4. Summary

After analysing the experimental data, the complex mechanism of ozone treatment was determined. At least two phases of the process were distinguished. The first was a very fast colour removal phase, which was evidenced in interphase gas–liquid areas with a high mass transfer speed. After all, the Hatta number, which describes the relationship between mass transfer and pure chemical reaction in gas–liquid systems, cannot be directly determined for real industrial wastewater; however, the kinetic regime of decolouration must be fast. After that, when the colour was removed, the second phase started. This phase was much slower, taking place (probably) in the bulk of the liquid phase (then the regime must slow down). This means that it took place in the tank. Therefore, the quasis stationary state appeared quickly during this phase, and the liquid became saturated with ozone. Most of the COD was removed in this phase.

Colour removal, as an operating parameter of the treatment, allowed the opportunity to determine an effective ozone dose equal to 32.4 ± 5.5 g/m$^3$. After transferring this ozone concentration into the liquid colour of the wastewater, 97% was removed, which was invisible to the eye. After estimating the effective ozone dose, the only matter was the time needed for effectively transferring this ozone dose from the gas into the liquid. COD was found to be unsuitable for ozone treatment parametrization. For effective COD removal, ozonation should be combined with other treatments, such as biotreatment. Ozonation was not regarded as a self-standing treatment method but rather post- or pretreatment in the more complex system. The results of $EC_{50\%}$ towards *Vibrio fischeri* converted into TU showed that in contrast to other methods, such as adsorption, ozonation was not harmful in terms of toxicity.

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**Supporting information**

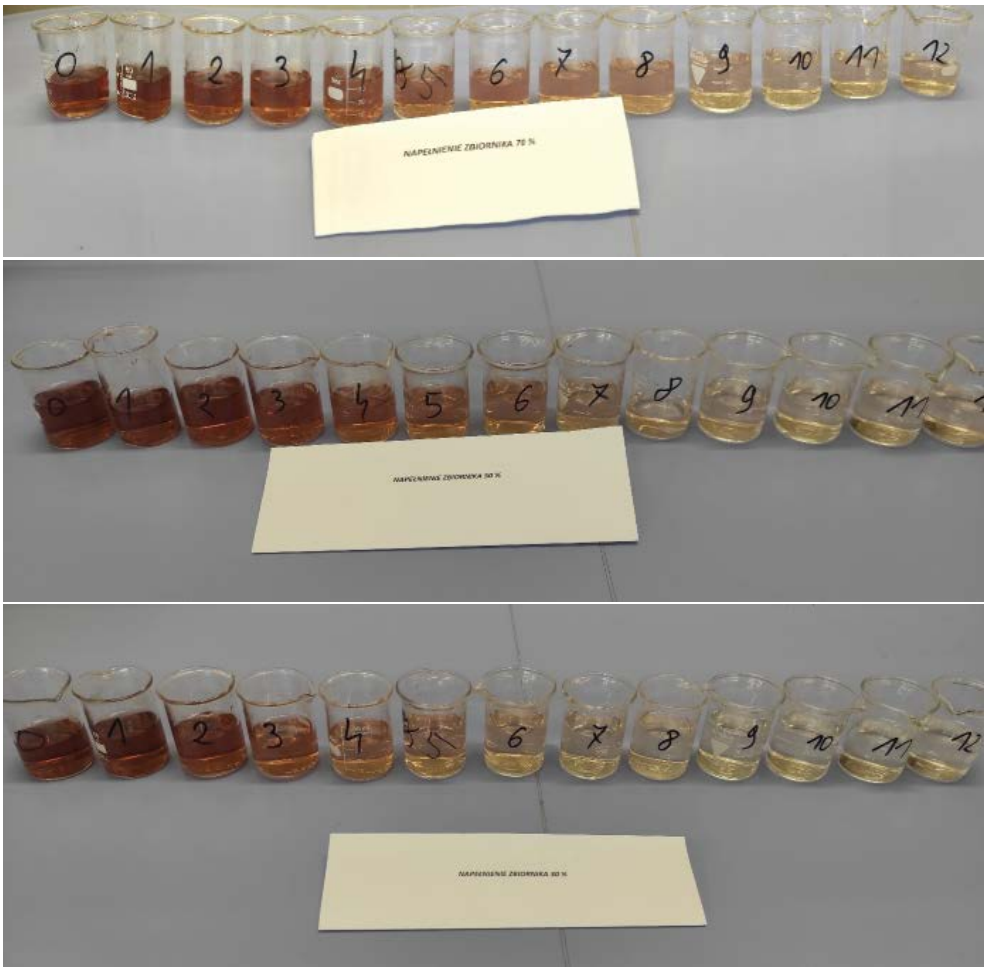


Fig. S1. Photos of wastewater samples when the reactor was loaded in 70%, 50%, and 30%, respectively.