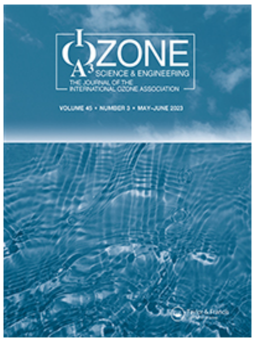

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# Physicochemical Modifications and Decolorization of Textile Wastewater by Ozonation: Performance Evaluation of a Batch System

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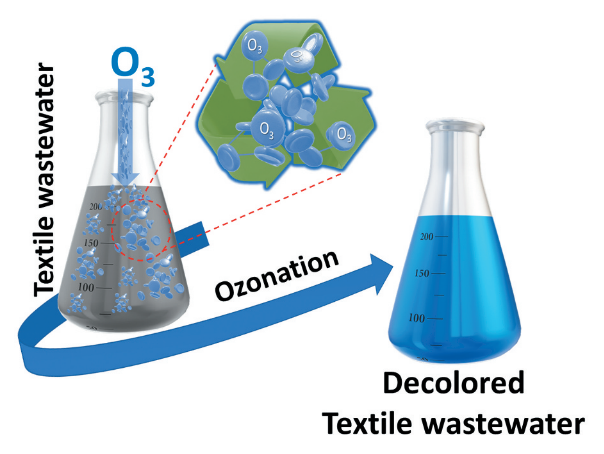
## ABSTRACT

This is an experimental study on the decolorization efficiency and the degradation of organic compounds from textile wastewater by the ozonation process in a batch system. The effects of different sample volumes of textile wastewater over time were investigated. The experiments were performed in a 1 L glass reactor with a magnetic stirrer and a bubble diffuser at the bottom to feed the ozone. The applied cumulative ozone dosage varied at 120 $gO_3\ L^{-1}$, 60 $gO_3\ L^{-1}$, and 30 $gO_3\ L^{-1}$, and the total interaction time for each test was 1 h. To investigate the physicochemical properties of the textile wastewater (solid and liquid phases) before and after the treatment, multiple analytical characterization methods were used: Thermal Gravimetric Analysis, Scanning Electron Microscopy coupled with Energy-Dispersive X-ray Spectroscopy, X-ray diffraction, Fourier Transform Infrared spectroscopy, and Spectrophotometer. The most perceptive change was observed in the color of the liquid medium, which turned from black to transparent, and a visual color number indicator known as *DurchsichtFarbZahl* (DFZ) was used for the evaluation of this process. Absorbance values decreased about 3.5 times after 5 min of treatment with a 0.15 L sample volume, and these values differed for tests with larger sample volumes. FTIR spectroscopy demonstrated that the bands' intensities associated with the C – H, C – N, and C – O decrease during treatment. On the other hand, it was possible to conclude that combining treatment methods to improve the degradation of persistent compounds after the ozonation process is necessary. Finally, the ozonation of the textile wastewater proved to be effective at removing color due to its high reaction capacity.



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**KEYWORDS**
Decolorization; ozonation; textile wastewater; treatment

## Introduction

The textile industry is one industrial sector that is constantly looking for new technological implementations on an industrial production scale. The main disadvantages of fabricating textiles are related to the consumption of water, dyes, and auxiliary chemicals and the significant production of wastewater (Mohan and Oke, 2021; Silva et al. 2018). In this sense, the average water consumption is about 100

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liters for each kilo of the textile product (depending on the variety of fabric processed) (Silva et al. 2018; Vajnhandl and Valh, 2014). Furthermore, 90% of the water input is destined for treatment to be later discharged into water bodies following the current environmental laws for each location (Silva et al. 2018; Vajnhandl and Valh, 2014). Wastewater from textile industries contains different types of dyes with high molecular weight and complex structures, resulting in low biodegradability, and it cannot be treated efficiently by the activated sludge process or any combination of biological, chemical, and physical methods (Radetski, Rosa, Rosa, De Souza Sierra, Simionatto 2002; Sevimli, Sarikaya, Yazgan 2003; Verma, Dash, Bhunia 2012). Textile wastewater (TW) problems mainly arise from three different sources (Bechtold, Burtscher, Hung 2004):

- dyestuff: colored effluents, adsorbable organic halogen (AOX), heavy metal content (Cu, Ni);
- dispersing agents in dyestuff formulation: chemical oxygen demand (COD), low biodegradability;
- auxiliaries, chemicals added: salt content (NaCl, $Na_2SO_4$), sulfide, pH value (NaOH, soda, silicates), COD (glucose, hydroxyacetone), nitrogen content (urea).

Most of these parameters are easily adjusted by conventional chemical coagulation and biological treatment methods, except for highly polymer structured colors (Selcuk 2005). Additionally, undesirable TW color changes caused by dyes reduce light penetration and inhibit photosynthetic activity, consequently reducing the amount of dissolved oxygen (Mcyotto et al., 2021).

Several recent review studies have reported the use of various techniques and systems for the treatment of TW (Alvares, Diaper, Parsons 2001; Behera, Nayak, Banerjee, Chakrabortty, Tripathy 2021; Holkar, Jadhav, Pinjari, Mahamuni, Pandit 2016; Ananthashankar, 2013; Samsami, Mohamadi, Sarrafzadeh, Rene, Firoozbahr 2020; Yaseen and Scholz, 2019). Existing methods can be categorized into three major treatment categories: biological, chemical, and physical treatments. Advanced oxidative processes (AOP) (Alinsafi et al. 2007; Boczkaj and Fernandes, 2017; Deng and Zhao, 2015; Dias and Azevedo, 2009; Lučić Škorić et al. 2016; Neamtu, Yediler, Siminiceanu, Macoveanu, Kettrup 2004; Peik-See, Pandikumar, Ngee, Ming, Hua 2014; Perkowski, Kos, Ledakowicz 2000), biological treatment (Mcyotto et al. 2021; Ghosh, Dastidar, Sreekrishnan 2018; Meerbergen et al. 2018; Wang, Li, Zhang, Yu, Zhu 2017), electrochemical treatment (Martínez-Huitle and Brillas, 2009; Silva et al. 2018; Torres et al. 2019), and membrane filtration (Erkanlı, Yilmaz, Çulfaz-Emecen, Yetis 2017; Wei et al. 2013) are some of the important methods commonly using for dye removal.

AOP is one of the most powerful methods for the decolorization of TW (Lin and Liu, 1994; Neamtu, Yediler, Siminiceanu, Macoveanu, Kettrup 2004; Sevimli and Sarikaya, 2005; Tehrani-Bagha, Mahmoodi, Menger 2010). It produces direct and selective oxidation and reacts preferably with the ionized and dissociated forms of organic compounds (Collivignarelli, Abbà, Miino, Damiani 2019; De Araujo et al. 2020). One of the most promising AOP is the process with ozone, which is a powerful oxidant with a high redox potential of 2.08 V (Collivignarelli, Abbà, Miino, Damiani 2019).

Today, as a result of increasingly demanding regulations, especially concerning the removal of organic substances and the decolorization due to dyes, treatment processes of TW that involve ozonation are among the most studied methods (Baban, Yediler, Lienert, Kemerdere, Kettrup 2003; Hassan and Hawkyard, 2002; Hsu, Chen, Yang, Chen 2001; Konsowa 2003; Ledakowicz and Solecka, 2001; Ledakowicz, Solecka, Zylla 2001; Muthukumar, Sargunamani, Senthilkumar, Selvakumar 2005; Sevimli and Sarikaya, 2002; Soares, Órfão, Portela, Vieira, Pereira 2006; Wu and Wang, 2001). In this regard, high values of decolorization but low mineralization have been observed. The application of ozonation in the process of decolorization of wastewater is characterized by the following advantages: (1) it does not increase the volume of wastewater and sludge; (2) it removes color and reduces the organic matter in one step; (3) it needs little space, and it is easily installed in a site; (4) it is less harmful than other oxidative processes since no stock hydrogen peroxide or other chemicals are required in a site; (5) residual ozone can be easily decomposed into oxygen (Chu and Ma, 2000; Shu 2006; Tehrani-Bagha, Mahmoodi, Menger 2010).

Several studies have been carried out to confirm the effectiveness of ozone in the decolorization of TW and to optimize the experimental ozonation conditions such as pH, contaminant concentration, and applied ozone dose. However, the impact of the sample volume on the process efficiency is not evident. To evaluate the practical application of this treatment process in industrial TW, the present work aimed to study the ozonation of different volumes of TW for a fixed concentration of ozone in a batch system. Parameters, such as pH and changes in the chemical structure of TW, were studied in each working condition, and the effects of these operating parameters on decolorization were analyzed using several analytical procedures such as spectrophotometry, Fourier Transform Infrared (FTIR) Spectroscopy, and visual color number indicators known as DeutscheFarbZahl (DFZ).

## Materials and methods

### Textile wastewater

TW resulting from dyeing black was obtained from large textile industry in São Paulo, Brazil. Raw TW was initially conditioned in 20 liters barrel and stored at room temperature. Before conducting the experiments, the samples were separated into 0.15, 0.3, and 0.6 L fractions for ozone treatment. No pH adjustments were made during the treatment. The TW used in this study was collected before the filtration columns, and its characterization is presented in Table 1.

### Ozonation batch system

The system employed to conduct the experiments is depicted in Figure 1 (De Araujo et al. 2020). The components included the following: feeding ozone (A), glass reactor (B) with a capacity of 1 L, bubble diffuser (C), sampling point (D), a magnetic stirrer (E), a reservoir (E), and finally, the gas escape (G). Ozone was generated using the Ozone Solution equipment model VMUS-4 coupled with a Millenium M10 Respironics oxygen concentrator. The operating parameters of the ozone treatment were fixed at a feed flow rate of 12 L min$^{-1}$ and a gas-phase concentration of 25 mg L$^{-1}$. The ozone concentration of the gas phase at the inlet and outlet was measured using an ozone gas analyzer (model 106-H, Ozone Solutions). For calculating ozone dosage, the methodology described by (van Leeuwen 2015), was followed. First, the ozone feed rate was calculated by multiplying the gas-phase concentration and the feed flow rate, resulting in 0.3 g min$^{-1}$. In short, four experimental conditions were employed: one control (only oxygen – 0.15 L), and three sample volumes (0.15, 0.30, 0.60 L) for the ozone treatments, namely control test (CT), N1, N2, and N3, respectively. Aliquots were collected every 5, 10, 15, 20, 25, 30, 45, and 60 min for further analyses. The applied cumulative ozone dosage for each test was N1 – 120 g$O_3$ L$^{-1}$; N2 – 60 g$O_3$ L$^{-1}$; and N3 – 30 g$O_3$ L$^{-1}$, which were calculated by multiplying the total period of the ozonation process by the application rate and dividing by the total volume of the sample. Furthermore, ozone losses were measured throughout the ozonation period, and the unused ozone was subtracted from the ozone applied over the same period for calculating the ozone consumption rate.

**Table 1.** Main physicochemical characterization of the TW sample.

| Parameter | Value |
|---|---|
| Conductivity | 68.7 mS cm$^{-1}$ |
| pH | 10.3 |
| $A_{0\ \lambda=650\ nm}$ | 2.08 |
| aDFZ$_{436}$ | 216 m$^{-1}$ |
| aDFZ$_{525}$ | 205 m$^{-1}$ |
| aDFZ$_{620}$ | 190 m$^{-1}$ |

aDFZ-Visual color number (German abbreviation of DurchsichtFarbZahl), determined according to DIN ISO 7887:2012 (2012a) (*DIN EN ISO 7887 (2012–04) Water Quality – Examination and Determination of Color*).

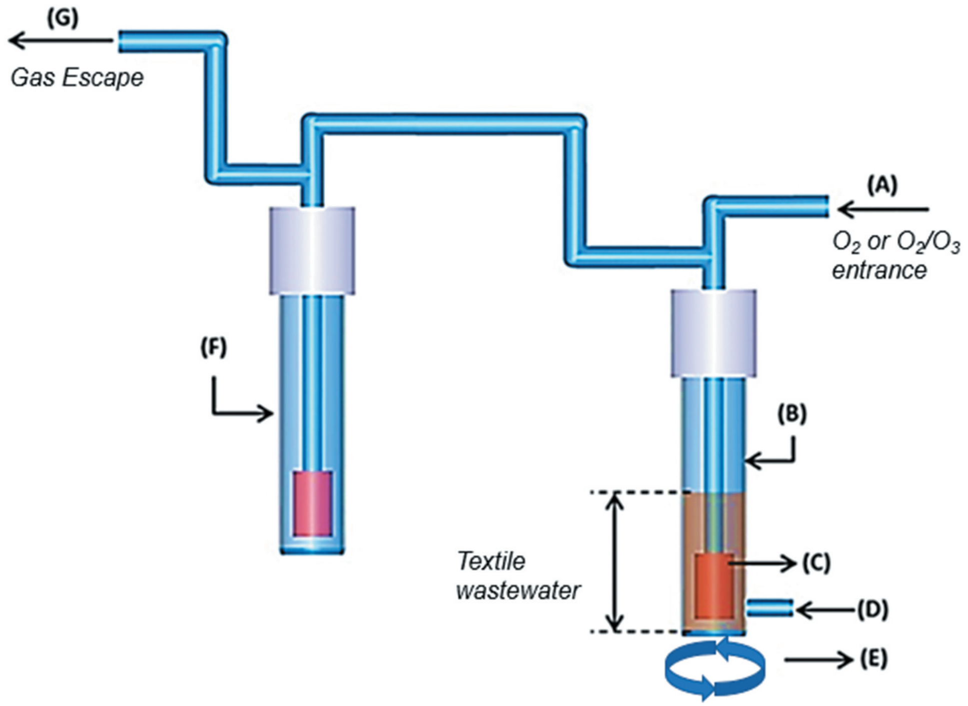

**Figure 1.** The ozonation experiments used the experimental setup (De Araujo et al. 2020).

### *Analytical methods*

Thermal Gravimetric Analysis (TGA) measurement of the TW was performed in a nitrogen atmosphere. The analysis was carried out with a simultaneous thermal analyzer (Netzsch STA 449 F3 Jupiter) in a temperature range from 30 to 300 °C for the liquid phase and from 30 to 800 °C for the solid phase. The nitrogen flow rate was set at 0.04 L min$^{-1}$ with a heating rate of 10 °C min$^{-1}$.

For morphology analysis, samples of the TW were dried in an oven at 80 °C and ground. The techniques employed were Scanning Electron Microscopy (SEM) and Energy-dispersive Spectroscopy (EDS). The analysis was performed in electron microscopy Vega 3 XMU (Tescan). X-ray diffraction (XRD) analyses of the solid portion were performed using a PANalytical Empyrean diffractometer (Cu-K$_\alpha$–$\lambda$ = 1.5406 Å) with 2θ varying from 15 to 90° with a step size of 0.02°, at room temperature and step time of 10s.

The functional groups' variation of the TW was observed using FTIR spectroscopy analyses on a Perkin Elmer Frontier apparatus in mode universal attenuated total reflectance (UATR) by collecting 32 scans at a 4 cm$^{-1}$. The spectrum was analyzed in the region from 400 to 4000 cm$^{-1}$.

The spectra of the samples collected at specified time intervals were measured by a spectrophotometer (Evolution 220, Thermo Scientific). The absorbance values were collected in the visible absorption region (range 420 nm – 650 nm). The DFZ indicators were used to obtain quantitative color measurements. In this regard, the estimation of DFZ allows the evaluation of the effluent coloration as a function of process time by considering different characteristic wavelengths, λ = 436 nm, λ = 525 nm, and λ = 620 nm from Equation. [1]:

$$DFZ = \frac{A}{D} \qquad [1]$$

where *A* is the absorbance and *D* is the optical path length of the cuvette expressed in meters. Based on the German TW discharge norms, to properly discharge these effluents, the maximum permitted DFZ values are 7 m$^{-1}$ for 436 nm, 5 m$^{-1}$ for 525 nm, and 3 m$^{-1}$ for 620 nm, respectively (*DIN EN ISO 7887 (2012–04) Water quality – examination and determination of color*; Dos Santos, Costa, da Silva, Garcia-Segura, Martínez-Huitle 2018). The percentage of color removal or decolorization efficiency was then calculated by Equation. [2] (Dos Santos, Costa, da Silva, Garcia-Segura, Martínez-Huitle 2018; Florenza et al. 2014; Martínez-Huitle and Brillas, 2009):

$$\%Color\ removal = \frac{A_0 - A_t}{A_0} \times 100 \qquad [2]$$

where $A_0$ and $A_t$ are the absorbance at the initial time and time t at $\lambda_{max}$ = 650 nm, respectively.

## Results and discussion

### *Characterization of the tw before the ozonation process*

The characterization of the TW before the ozone treatment was performed in the liquid and solid phases. Figure 2 shows the TGA for the liquid and solid phases as a function of temperature.

The main decomposition process took place below 100 °C, taking up at least 93% of the total conversion in the nitrogen atmosphere. Therefore, the sample of the TW used in this work has 7% total solids (organic and inorganic). No significant mass loss was observed at higher temperatures, from 100 to 300 °C (maximum temperature analyzed for the liquid phase). The TGA analysis for TW solid-phase highlighted the prominent presence of inorganic compounds once only 3% of mass loss was presented, even for temperatures reaching 800 °C. This result demonstrates that all organics compounds are presented in the liquid phase.

The morphology and the elemental composition of the TW investigated by SEM/EDS analysis are shown in Figure 3. The solid sample is heterogeneous, and the presence of the Na, Cl, O, C, S, and Al can be noted. These results are in line with those obtained by XRD, which ensures the correct identification of the main components of this sample.

According to Figure 3 (b), the chemical composition of the TW sample presents sodium (30.9 ± 0.3%), chlorine (27.4 ± 0.3%), oxygen (25.6 ± 0.2%), carbon (15.3 ± 0.7%), sulfur (0.5%), and aluminum (0.4%), which are considered common elements in the process of textile dyeing using reactive organic dyes as reported by Holkar et al. (2016); Ananthashankar and G (2013); Sevimli and Sarikaya (2002); Yaseen and Scholz (2019).

The XRD pattern of the solid part of TW is presented in Figure 4. It shows the presence of sodium chloride (NaCl) cubic phase and aluminum oxide ($Al_2O_3$) in its cubic phase. The identification of the main diffraction peaks was made with the HIGHSCORE software. It is noteworthy that no changes occurred in the XRD patterns of TW after ozonation, which confirms the finding of the study reported by Allègre et al. (2004) that it is possible to recycle mineral salt in the dyeing process.

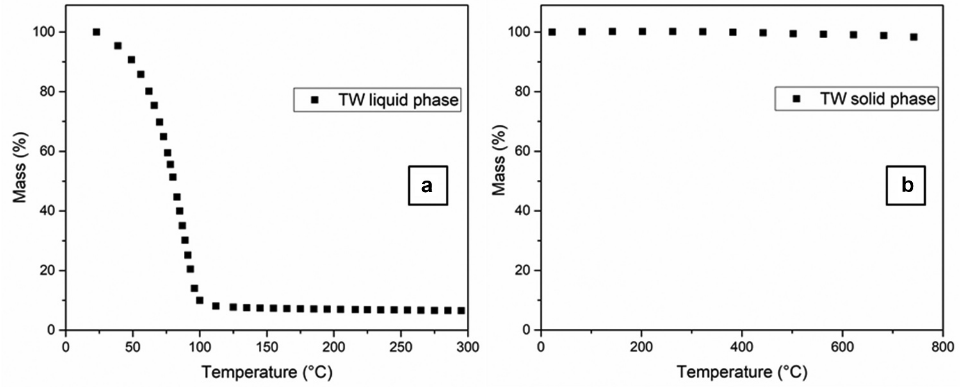

**Figure 2.** TGA pyrolysis curves of TW samples. Liquid phase (a) and solid-phase (b).

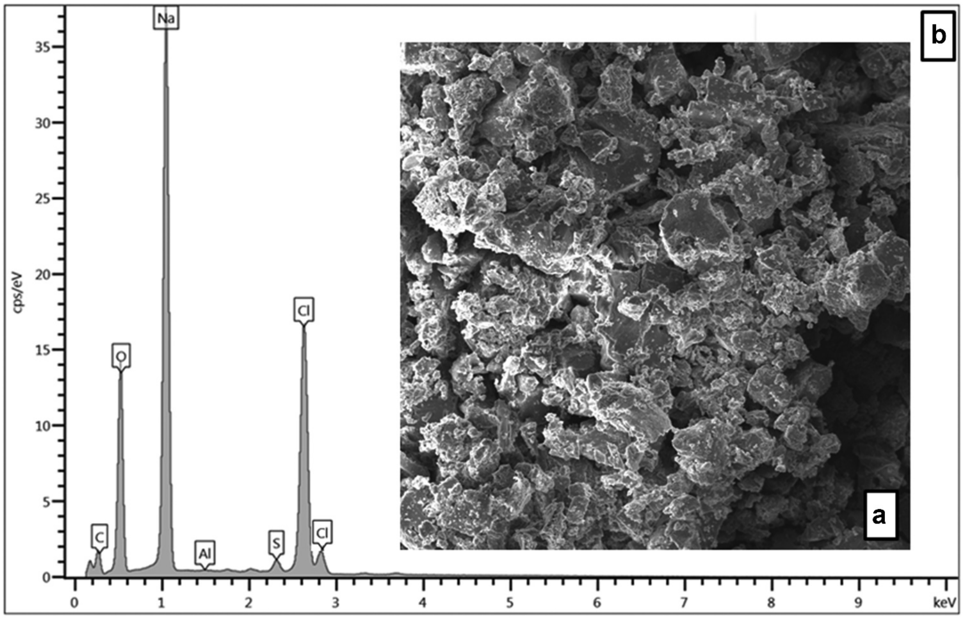

**Figure 3.** Micrograph (a) and energy-dispersive X-ray spectra (b) of the solid part of the TW sample.

In the case of TW after dyeing operations, the most important characteristic is the presence of NaCl and $Na_2SO_4$. They can act as exhausting as well as retarding agents. For example, they act as exhausting agents in the application of direct dyes on cellulosic fibers, acid milling dyes on wool, and reactive dyes on cotton, and as retarding agents in the application of leveling acid dyes on wool and basic dyes on acrylic (Bilińska, Gmurek, Ledakowicz 2017; Muthukumar and Selvakumar, 2004).

Several authors have investigated the influence of salts or alkalines on the ozone treatment (Bamperng, Suwannachart, Atchariyawut, Jiraratananon 2010; Colindres, Yee-Madeira, Reguera 2010; Muthukumar and Selvakumar, 2004) all reported that the decolorization performance of dye solution with the presence of NaCl has no significant efficiency changes. Muthukumar and Selvakumar (2004) were the only authors who have made a detailed study about the effect of salt during ozone decolorization of TW. At higher pH levels, the presence of salt lowers the decomposition of reactive dyes; in addition, complete decolorization time is the lowest when the effluents contain no salt content in them, irrespective of their pH levels.

## Characterization of textile effluent after ozonation

### Effect of ozone treatment on pH

The initial pH measurement of TW is 10.23 and therefore of an alkaline nature. Figure 5 shows that while no pH variation was observed in CT, there was a significant pH decrease after 5 min of ozone treatment. The final

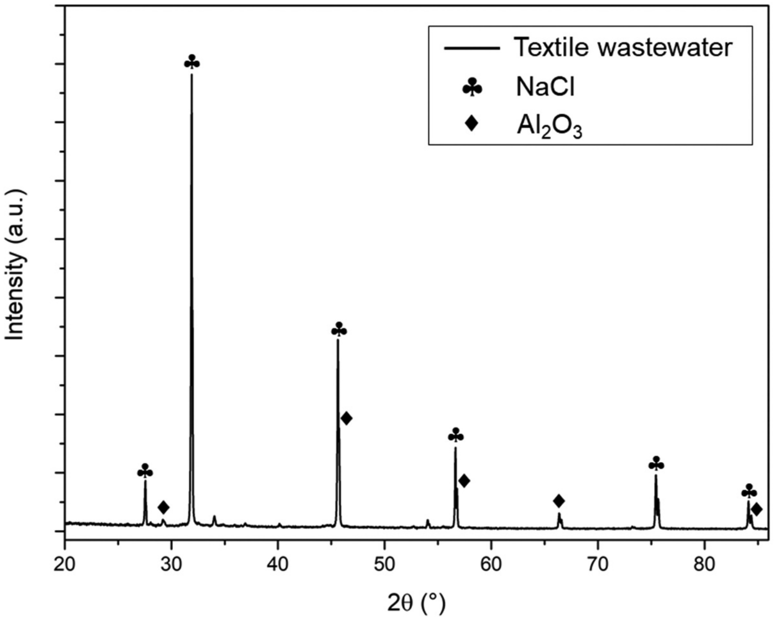

Figure 4. X-ray diffractogram of the solid part of the TW sample.

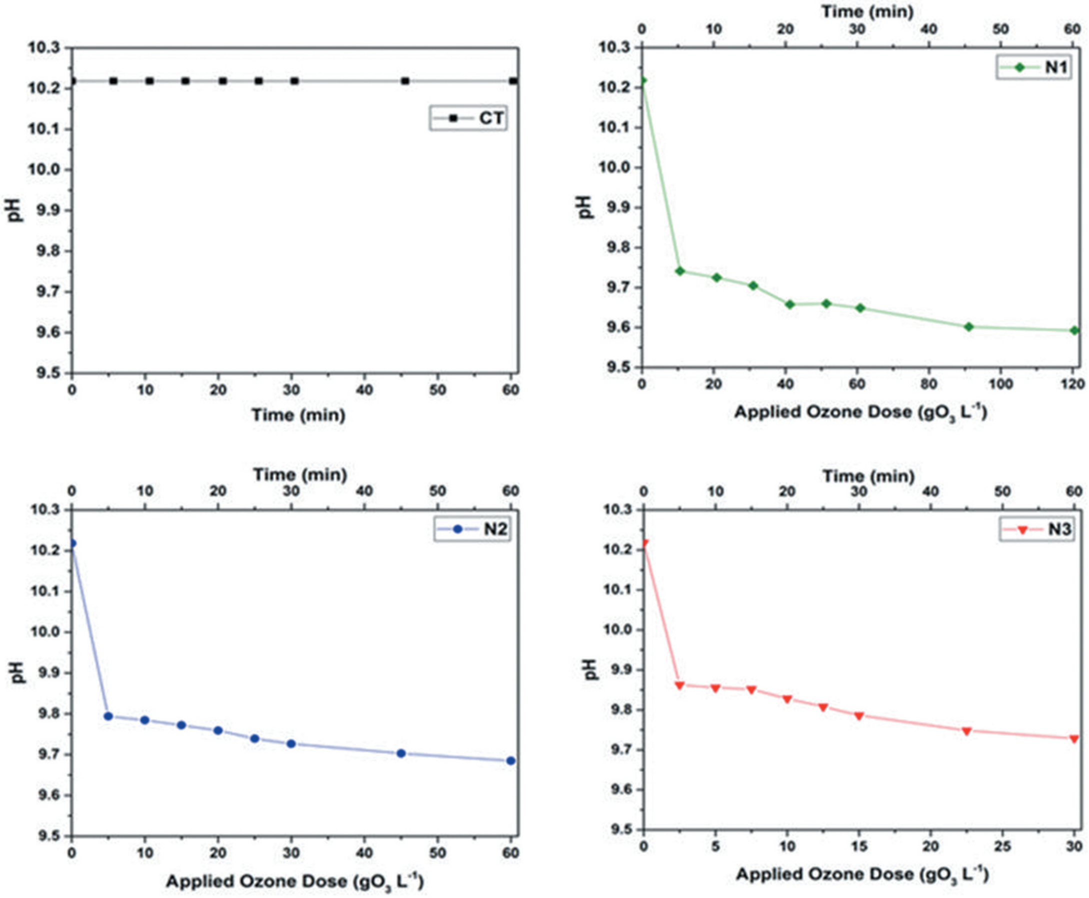

Figure 5. Temporal evolution of pH during the ozonation process.

pHs were 9.59 (N1), 9.70 (N2), and 9.74 (N3), indicating no significant variation regardless of the initial wastewater volume treated.

Perkowski et al. (2000) and Sevimli and Sarikaya (2002) reported that under alkaline conditions, the decomposition of pollutants by ozonation occurs from their reactions with highly reactive intermediates, such as $\bullet HO_2$ and $\bullet OH$ radicals. In the ozonation process, the pH parameter influences the oxidation efficiency. The pH of the solutions decreases due to the production of organic and inorganic acids during the oxidation of the TW solutions (Tehrani-Bagha, Mahmoodi, Menger 2010). As soon as these acids are formed, they may also be degraded and fully mineralized to $CO_2$ and $H_2O$, resulting in a pH increase (Goulart de Araujo, Santos, Teixeira 2017).

## Effect of sample volume variation on consumed ozone dose

Ozone inputs and losses over the reactor were measured, and the ozone consumed in the reactor was related to the ozone applied dose as a function of ozonation time, as shown in Figure 6.

As the applied ozone dose increases, in this case, due to the variation in sample volume, consequently color removal efficiency increases (Sevimli, Sarikaya, Yazgan 2003). On the other hand, when the applied ozone dose was increasing, the dose of ozone consumed reached a threshold and decrease; besides, in the experiments, N1, and N2, the saturation of the ozonation process occurred in 30 min and 50 min, respectively, this indicates a depletion of substances that can react with ozone. Experimental results showed that the major part amount of applied ozone is lost, in view of this, it is possible to conclude that high doses of ozone are not necessary, but what determines the effectiveness of the treatment is the residence time of the ozone in the TW in function the volume sample. At the same time, the consumed ozone dose in experiments N1 and N2 reached 13.9 and 17.2 $gO_3\ L^{-1}$, respectively, and then decreased, whereas the N3 experiment with a sample volume of 0.6 L obtained an ozone consumption rate in relation to the applied ozone dose higher and constant during the 60 min of treatment. In the experimental configuration implemented in this work, the most effective ozone utilization was obtained in larger sample volume applications. These results might be similarly explained by the works of (Sevimli, Sarikaya, Yazgan 2003) and (Alvares, Diaper, Parsons 2001) the mass transfer of ozone from the gaseous to aqueous phase can be inefficient due to gas/liquid ratios, causing a proportion significant of applied ozone to be lost in the off-gas. Decreasing the feed flow of the ozone gas phase should enhance the mass transfer causing an increased contact time with the TW sample. However, such an overestimation of ozone dosage may be insignificant compared to the amount of ozone used during the experiments performed in this work for decolorization.

Therefore, the focus of this work is to demonstrate in detail the physicochemical modifications in the solid and liquid phases during the ozonation process.

## Characterization of tw after ozonation process

The ozonation process fostered significant changes in the color of the treated TW samples, visually highlighting the decomposition of compounds via oxidation, as shown in Figure 7. Color changed with increasing oxidation times and, as expected, larger sample volumes resulted in longer times (60 min; 0.6 L) to achieve about the same visual appearance obtained with the lowest sample volume (15 min; 0.15 L). The higher the organic content, the longer the process will take to reach a similar outcome. Furthermore, as the sample volume increases, there is a decrease in gas interacting with the TW, hampering the process efficiency.

The changes related to the color of the TW samples are consequences of composition changes during AOP treatment. Figure 8 shows the absorbance spectra in the visible range (425–650 nm) for variations of sample volumes. The raw TW absorbance is also presented for comparison.

The temporal evolution of the absorbance curve for the three experimental runs confirms the visual observation (Figure 7). The absorbance value decreased significantly in the treated samples compared to the raw TW in the visible range studied for all experiments. A comparative analysis of the absorbance profile of each experiment spotlights that for the lowest sample volume (0.15 L), 5 min was enough to 3-fold decrease the absorbance of the chromophoric species. On the other hand, it took longer for the intermediate and largest sample volumes to reach that same level, with 15 min for the former and about 20 min for the latter level. The spectrum of the initial solution exhibited a visible bandwidth of a strong peak centered at $\lambda = 506$ nm. According to Florenza et al. (2014) and Pinheiro et al. (2004), this band may be associated with the azo dye and other possible organic aromatic products that absorb in the region of similar wavelength.

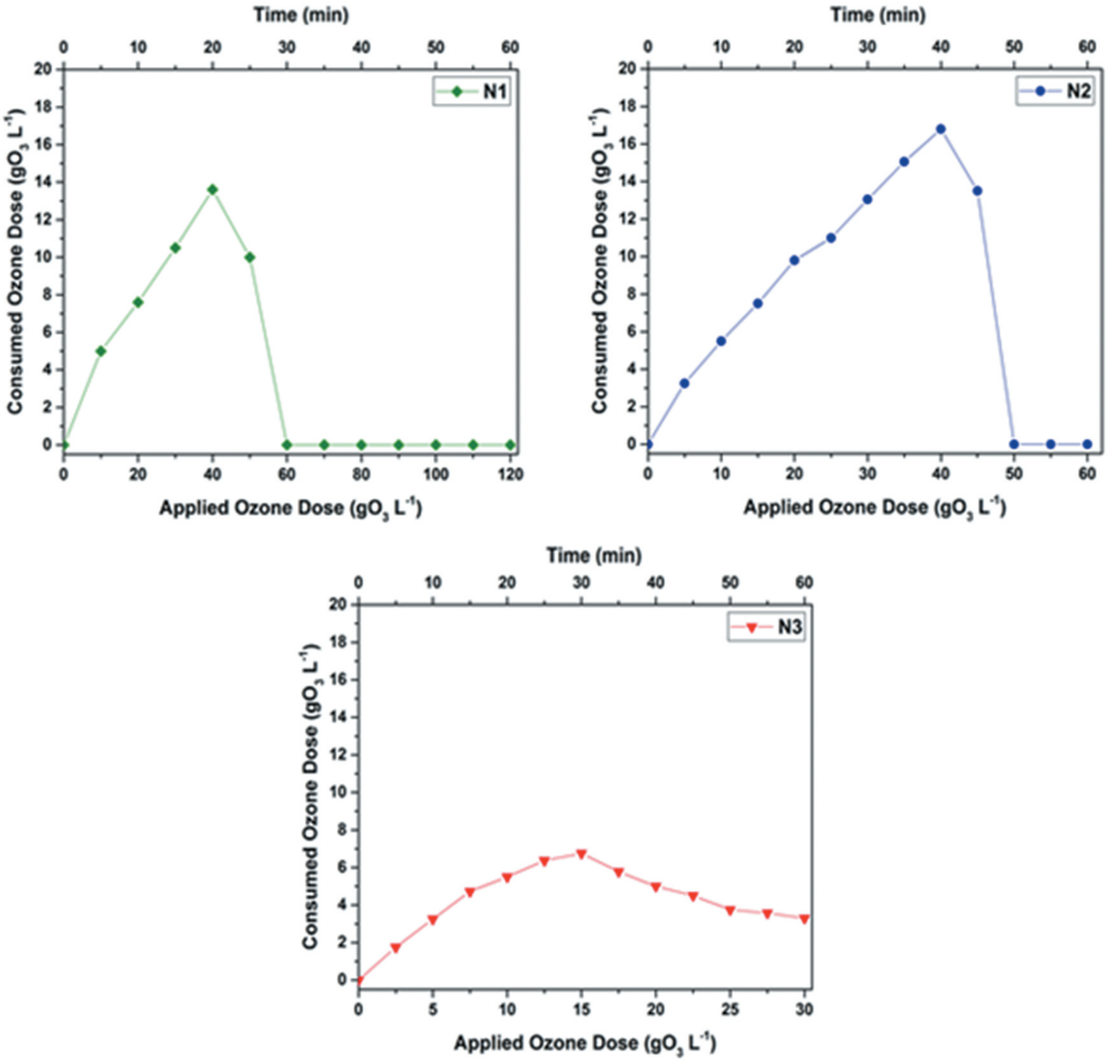

**Figure 6.** Relationship between consumed ozone dose and the applied ozone dose.

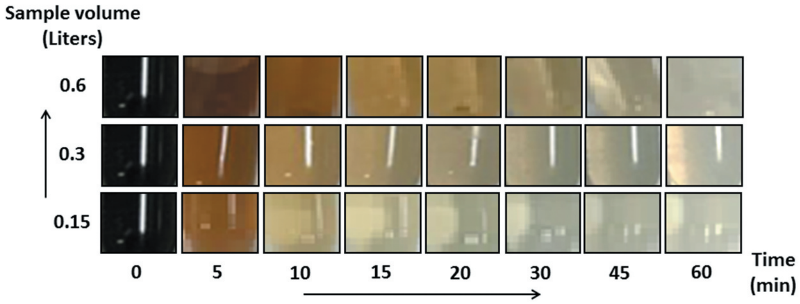

**Figure 7.** The color change of the TW samples during the ozonation process.

The presented results indicate that the azo dye is the principal species present in the TW sample during the decolorization process. The azo group may be bonded to benzene rings, naphthalenes, aromatic heterocycles, or enolizable aliphatic groups. These are essential to give the color of the dye, with their shades of different intensities. In general, the chemical structure of an azo dye is represented by a backbone, the auxochrome groups, the chromophoric groups, and the solubilizing groups; according to Figure 9, the color of the azo dyes is determined by the azo bonds and their associated chromophores and auxochromes (Benkhaya, M'rabet, El Harfi 2020).

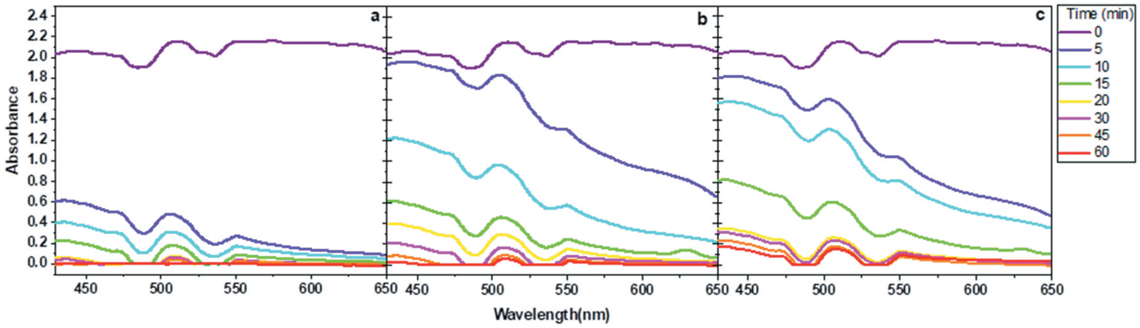

**Figure 8.** Absorbance analyses for samples of the raw TW and after ozonation process for times up to 60 min and sample volumes of (a) 0.15, (b) 0.30, and (c) 0.60 L.

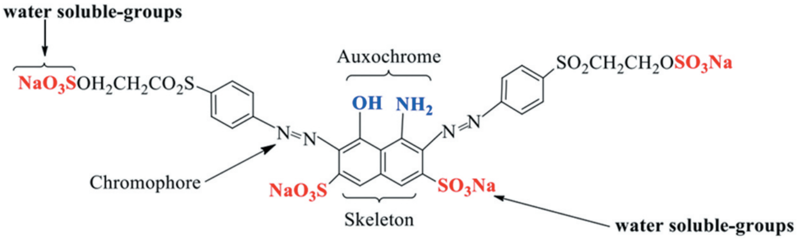

**Figure 9.** Structure of the azo dye (Benkhaya, M'rabet, El Harfi 2020).

Reported by van Leeuwen et al. (2009), the decolorization analysis does not indicate all changes in the ozonation process, as the first reactions of the azo dye with ozone produce by-products that still obtain color; because of this, the chemical structure and composition of the untreated and ozone-treated TW samples were determined by using FTIR. The results may be explained by the production of small organic molecular fragments and the partial destruction of the azo dye, not being utterly mineralized under the prevailing oxidation conditions.

Figure 10 depicts few changes in the bands' intensities even under an oxygen-only – CT run. These small changes are indications of the interaction between the ozone and the degradation of the organic content of the samples; therefore, it is possible to confirm that would still require another biological stage to degrade the intermediates (formed upon ozonation). In this present work, was performed only the evaluation ozonation process. The identification in the spectrum, assigned vibrations, and wavelength are listed in Table 2.

TW has many functional groups. The bands at 3424 $cm^{-1}$ in the spectrum of the samples could be assigned to the axial stretching vibrations of OH groups superimposed on the N – H stretching bands (Lučić Škorić et al. 2016). The two peaks (2919 and 2850 $cm^{-1}$) are aliphatic C – H peaks (Kaçan and Kütahyali, 2012). It is noteworthy that, for N1, there was a complete disappearance of these bands, probably due to a more significant degradation of organic compounds with a lower sample volume. The bands at 1638 and 1431 $cm^{-1}$ reflect the carbonyl group stretching (amide) and N – H bending, respectively. For these bands, no difference was observed for any of the runs performed. Bands at 1248 and 1054 $cm^{-1}$ correspond to C – N and C – O stretching vibrations, respectively (Mahmoodi, Arami, Bahrami, Khorramfar 2011). Figure 10(b) indicates the disappearance of these bands in N1. In the case of N3 and CT, there is no clear distinction between these bands compared to TW. The small peak at 893 $cm^{-1}$ is attributed to the C – H bending owing to aromatic groups (Vasantharaj et al. 2019). For N1, most of the identified peaks in the pre-treated wastewater (TW) disappeared. On the other hand, peak C ($\lambda$ = 1638 $cm^{-1}$), assigned as C – O, was kept unaltered. The presence of NaCl (Figure 4) in the solutions

**Table 2.** List of assigned vibrations and their respective wavelength.

| Identification | Assignment | Wavelength ($cm^{-1}$) |
|---|---|---|
| A | C – H | 2919 |
| B | C – H | 2850 |
| C | C – O | 1638 |
| D | N – H | 1431 |
| E | C – N | 1248 |
| F | C – O | 1054 |
| G | C – H | 893 |
| H | MAS* | 605 |

*Monosubstituted Aromatic Structures

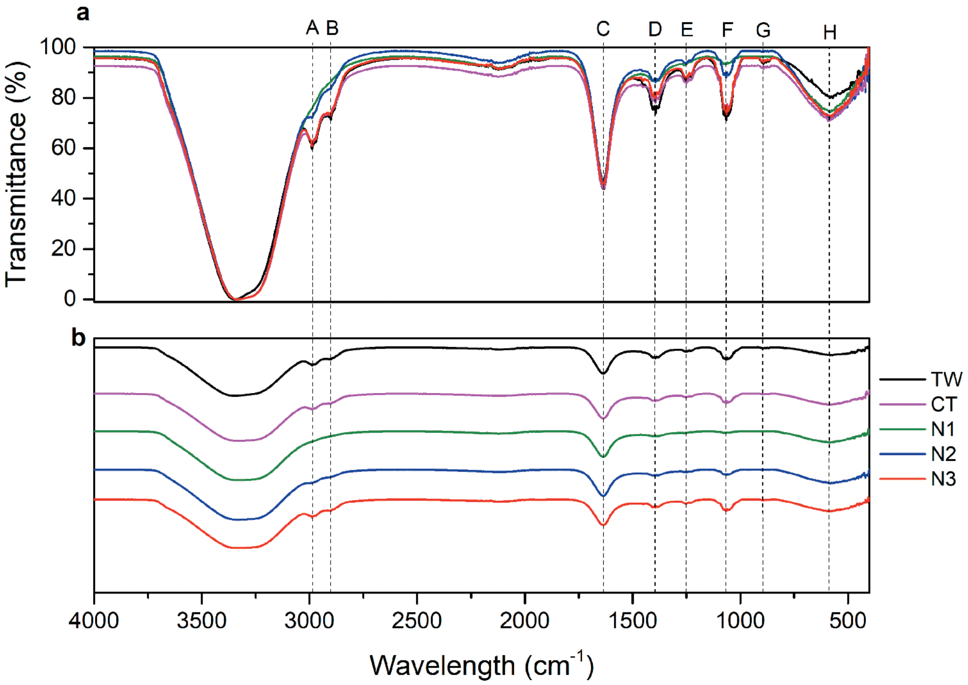

**Figure 10.** FTIR spectra of the TW before and after the ozonation process. TW: textile wastewater; CT: control test; N1 to N3: the experimental runs (a) transmittance of each solution. (b) another view of the FTIR spectra.

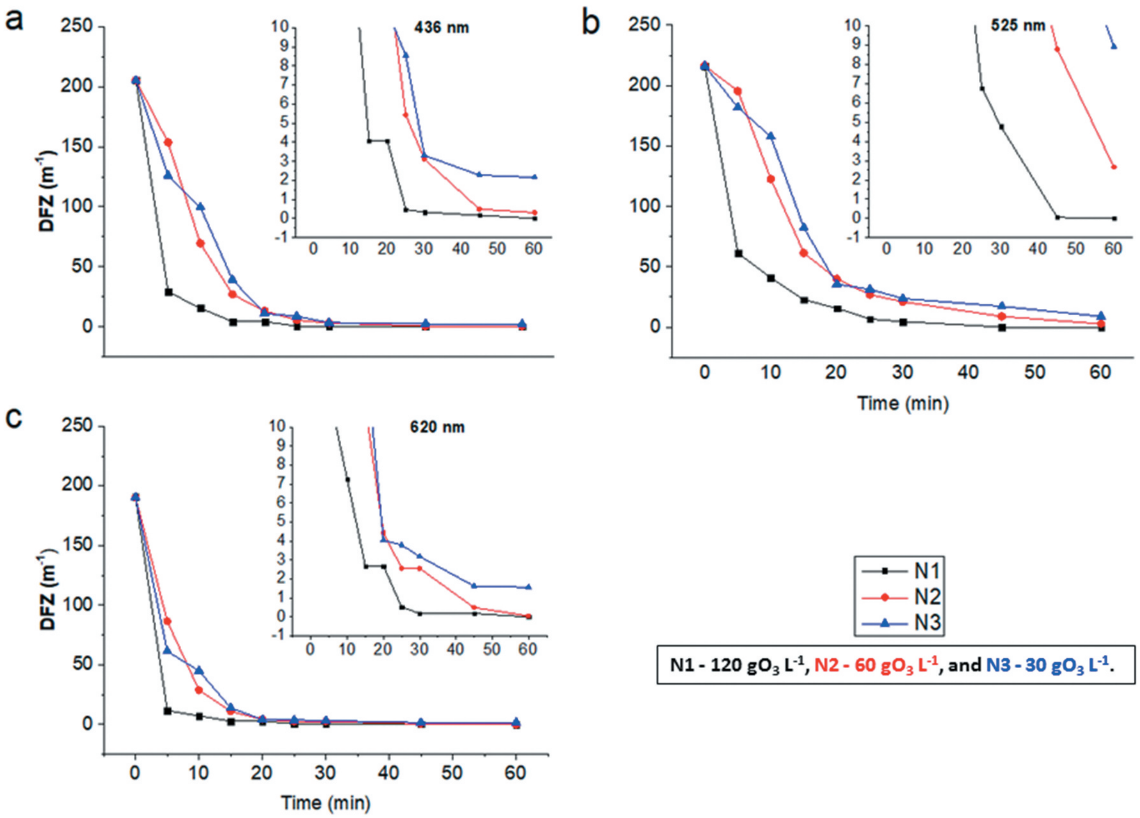

**Figure 11.** Evolution with ozonation of textile wastewater, with DFZ determined at (a) 436 nm, (b) 525 nm, and (c) 620 nm. (black square) N1, (red circle) N2, and (blue triangle) N3.

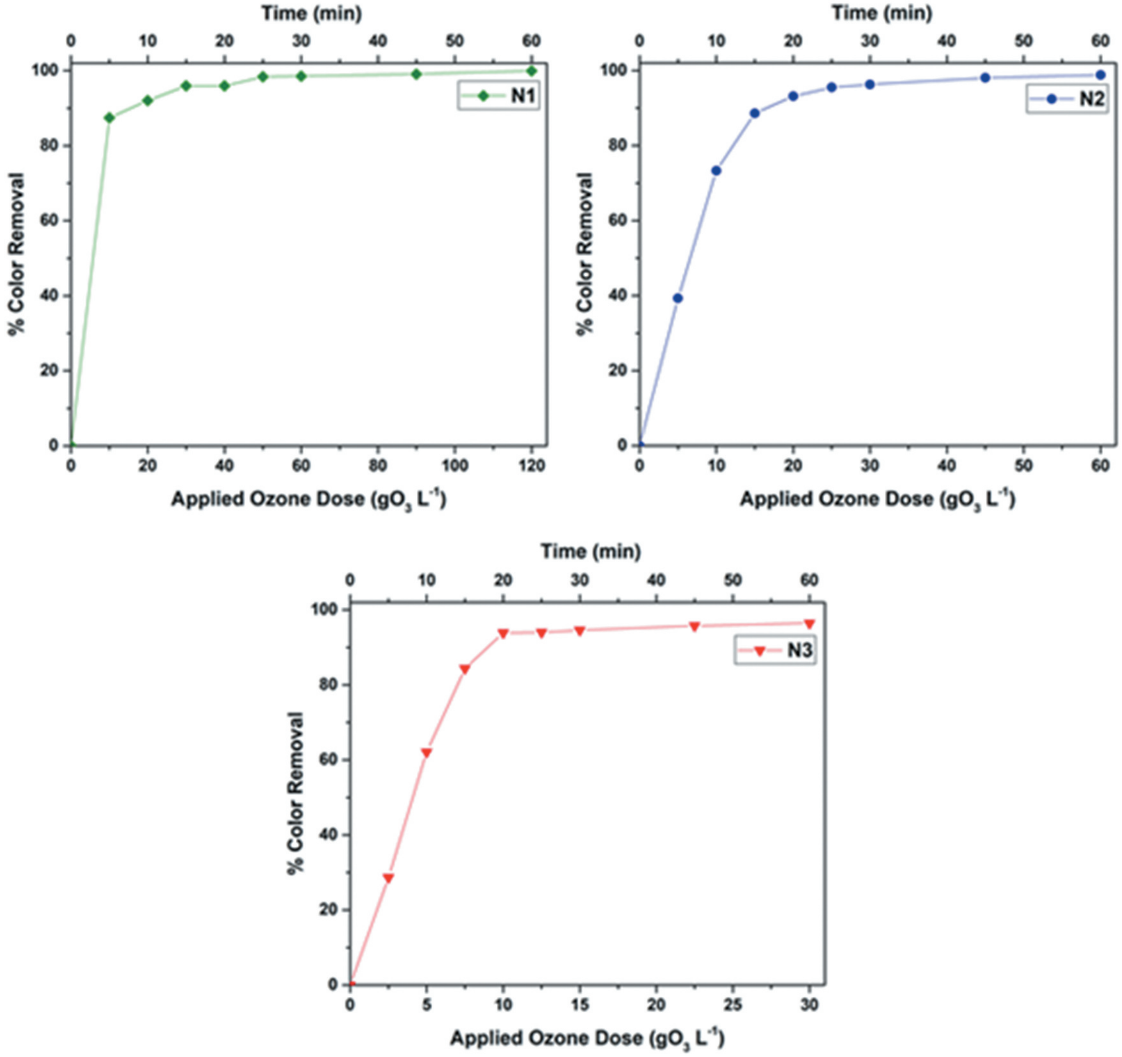

Figure 12. Color removal by employing ozonation in three different experimental conditions.

may have contributed to the formation of products with oxygen. One possible reason is that, in solutions containing only NaCl, the decomposition of ozone is only followed by the formation of products containing oxygen (Razumovskii, Konstantinova, Grinevich, Korovina, Zaitsev 2010), which may have kept high the intensity of the C – O even under oxidation. The fingerprint region of the peaks at 605 and 440 cm$^{-1}$ indicates monosubstituted aromatic structures (MAS) (Kaçan and Kütahyali, 2012).

Figure 11 shows the changes in color over time considering the parameter DFZ. Most of the experimental conditions resulted in suitable values for effluent release after 60 min of treatment. For N1 (0.15 L), the values after 60 min were: <limit of detection (LOD) for 436, 525 and 620 nm. For N2 (0.30 L), the values were: 2.66 for 436 nm, 0.30 m$^{-1}$ for 525 nm, and 0.05 m$^{-1}$ for 620 nm. For N3 (0.60 L), the values were: 8.91 m$^{-1}$ for 436 nm, 2.15 m$^{-1}$ for 525 nm, and 1.56 m$^{-1}$ for 620 nm. The only not acceptable value was observed for N3 at 436 nm, with 8.91 m$^{-1}$, higher than the established value of 7 m$^{-1}$. According to the literature, N1 and N2 possess the appropriate experimental conditions (*DIN EN ISO 7887 (2012–04) Water quality – examination and determination of color*; Dos Santos, Costa, da Silva, Garcia-Segura, Martínez-Huitle 2018).

The dark-colored TW was treated with different volumes of TW and consequently the different applied ozone doses. Figure 12 shows the color removal (%) through this variation regarding the volume in function of the time and applied ozone dose. N1 demonstrated the fastest color removal rate, reaching more than 80% removal in the first 5 min. N2 and N3 took between 15–20 min to reach a similar value, presenting similar behaviors. These results confirm the visual observations shown in Figure 7 that, although longer times were necessary for N2 and N3 to reduce color, all experimental conditions reach similar values after 60 min. However, care must be taken since the DFZ value obtained by N3 at 436 nm was superior (~8.91 $m^{-1}$) than it should have been (7 $m^{-1}$).

## Conclusion

In this work, the application of ozone with different volumes (0.15, 0.30, 0.60 L) in a batch system performance was evaluated concerning decolorization efficiency and proved to be an effective decolorization method for textile wastewater was performance evaluated for the liquid product during its process.

The TGA analysis indicated that the liquid sample contained about 7% of non-degradable compounds. The presence of Cl, O, Na, C, S, and Al in the solid samples was confirmed by XRD and SEM/EDS analysis.

FTIR spectrometry indicates that ozone promoted alterations in the bonds and structure of the samples. A slight degradation of organic compounds with a lower sample volume was observed after ozone treatment indicating that it is necessary to combine treatment methods to improve the degradation of persistent compounds after the ozonation process. The appreciable reduction of the ozonation performance can also be related to the formation of complexes with the degradation by-products, such as carboxylic acids, enhancing the parasitic reactions that consume the •OH competitively and consequently reducing the overall process efficiency.

Slight modifications were observed by the volume variation of liquid samples in the final tests; all experimental conditions resulted in a decrease in absorbance with the decrease of the sample volume, as confirmed by spectrophotometric analyses. Considering the application of the process on an industrial scale, these modifications must be considered mainly due to the significant increase in the volume of wastewater to be treated.

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## Data availability statement

The authors confirm that the data supporting the findings of this study are available within the article.

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