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Elimination of volatile organic compounds in paint drying by absorption reaction in water combined with the ozone oxidation technique

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ABSTRACT

The aim of this work was to evaluate the treatment of Volatile Organic Compounds (VOCs) released during the painting and drying process of an industrial paint by absorbing them in a water curtain and then oxidizing them with ozone. For this purpose, laboratory equipment was built consisting of a paint drying cabin and a bubble column where the VOCs were absorbed and treated. Tests were carried out with simulated effluents using distilled water and service water, to which 3.5 µg/L of formaldehyde, 11.6 mg/L of butyl acetate, 5.3 mg/L of ethanol and 5.6 mg/L of methyl ethyl ketone were added, according to data provided by the paint manufacturer; these tests were then repeated using the industrial paint currently used in the processes. The water saturation with the VOCs and the simultaneous treatment by means of ozone dosage was studied, taking the Chemical Oxygen Demand (COD) value as a treatment effectiveness parameter. VOC saturation is achieved for exposure times between 5 and 15 min in the aqueous effluent. The system of absorption and ozonation is effective for the elimination of the released VOCs, achieving a 94% reduction in the COD of the wastewater. Finally, it was observed that when O_3 is injected together with the paint, the reduction of COD in the effluent is favored due to a first instance of direct ozonation of the VOCs, and a second, slower indirect stage, generated by the presence of free radicals from the reaction of O_3 with water [hydroxyl radical (HO•), superoxide anion radical (O_2^{-}•) and hydroperoxyl radical (HO_2•)] favored by the pH of the solution. The reaction kinetics of VOC oxidation is of first order.

Introduction

Volatile organic compounds (VOCs) are those organic compounds that have a Reid vapour pressure of more than 10.3 Pa under normal temperature and pressure conditions, which encompasses a group of carbon-based chemicals that evaporate easily at room temperature (Kamal et al., 2016; Ojala et al., 2011). VOCs emissions can come from a wide range of sources including chemical industries, paper production, food processing, paint drying, transportation, oil refineries, petrochemicals, etc. (Morin et al., 2019; Zhang et al., 2016; Drobek et al., 2015). Typically, aldehydes, aromatics and halo-hydrocarbons are the most common contaminants in the large family of industrial VOCs.

VOCs emission can be controlled using methods based on recovery and destruction. Recovery-based techniques include absorption, adsorption, membrane separation and condensation. According to Kujawa (2015) the high cost of adsorbents and the need for their frequent regeneration are the main limitations of the adsorption processes. In methods based on destruction through oxidative processes, VOCs are converted to carbon dioxide and water. Within the oxidative processes, there are several methods such as catalytic oxidation (Wang et al., 2017; Chen et al., 2020a, 2020b; Shu et al., 2018; Li et al., 2018a, 2018b), photocatalytic oxidation (Deng et al., 2017; Krichevskaya et al., 2017) and plasma oxidation (Zhu et al., 2015; Chang et al., 2019), which have given positive results in the treatment of VOCs.

According to Zhu et al. (2017)Zhu (2017), the catalytic ozone oxidation method is a promising alternative technique for low temperature VOC oxidation (Huang et al., 2015; Zhao et al., 2011). Zhu et al. found that relative humidity plays an important role in the process of catalytic oxidation of ozone, as the presence of water significantly improved the oxidative removal of HCHO. They concluded that the positive effects of relative humidity on the process of catalytic oxidation of ozone gives a more favorable oxidation route for HCHO in a wet gas stream.

Ozone oxidation is another potential technique that has been applied to decompose VOCs. Because of its strong oxidizing ability, ozone was commonly used as a cleaning agent in some air purifiers for the removal of VOCs. Yu-Hua Li and co-workers (2018) developed a continuous flow system that combines two reactors, one with the ozone oxidation technique and the other with the photocatalytic oxidation technique

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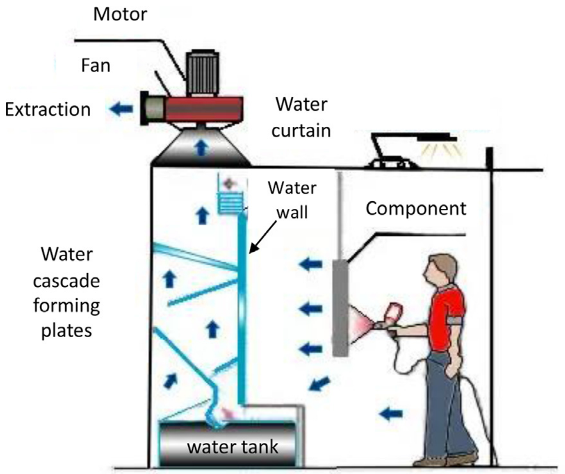

Fig. 1. Water curtain paint cabin.

($UV / TiO_2 + O_3$) to treat the cooking fumes. They found that VOCs concentrations decreased abruptly when ozone was injected into the ozone oxidation reactor, showing that ozone can react effectively with VOCs in oil fumes.

Ozone, as mentioned above, is a powerful oxidant with a redox potential of 2.07 V in an alkaline solution, so it can oxidize many inorganic and organic substances (Yang et al., 2020; De Araujo et al., 2020; Tabla-Hernandez et al., 2020; Swetha et al., 2017) in ‘ media. In general, the ozonation of pollutants can be done in two different ways: a) direct reaction by the ozone molecule and b) indirect oxidation by the OH• produced by the decomposition of ozone (Sonntag, 2012). If the ozone molecule reacts directly with the pollutants, it can happen within four possible reaction mechanisms (Wang and Chen, 2020) among which oxidation-reduction reactions, cycloadition reactions, electrophilic substitution reactions, and nucleophilic reactions may be involved; if the reaction is indirect, the pH value of the solution where the reaction occurs has a significant influence on both the efficiency of direct ozonation and on the generation of OH•. Filho et al. (2019) found that an increase in the relative humidity improved the degradation of the contaminants, which was mainly associated to the production of OH• from the reaction between O• radicals and water molecules.

Water curtain cabins are used for painting processes (Fig. 1). The cabins consist of several elements that allow collecting the paint sprays in the water tank, as well as capturing the solvents incorporated into the paints.

A water film traps the VOCs and micro-drops of paint remaining from the application process. This method avoids the use of filters, which reduces their maintenance. The disadvantage of this closed system is the accumulation of VOCs in the water tank. Consequently, this study aims to develop an innovative technique for the decomposition of VOCs by ozone oxidation, simulating the operation process of the water curtain cabin. This strategy of VOC absorption in aqueous solution prior to treatment has already been reported by Xie et al. (2019) through the use of a wet scrubber.

Experimental method

Based on the information provided by the company, the operating plant has an effluent that presents the following contaminants: 0.0035 mg/L of formaldehyde, 0.0116 g/L of butyl acetate, 0.0053 g/L of ethanol, 0.0056 g/L of methyl ethyl ketone with a COD value of 7910 mg/L. The solubility of the contaminants in water at 20 °C is 7 g/L for butyl acetate, 2.9 g/L for methyl ethyl ketone and 40% v/v for formaldehyde.

In a first stage, the wastewater was simulated in laboratory using the chemical reagents mentioned above, in analytical grade. An effluent of similar concentration to that which would be generated in the water tank of the paint cabin was prepared with distilled water. 250 mL of this effluent were treated by bubbling O_3, and chemical oxygen demand (COD) measurements before and after treatment were then compared. The ozone was generated by an Ozonizer equipment that generates an ozone flow of 650 mg/h.

In a second stage, the effluent was simulated using the service water that the company has in its process plant. As in the previous stage, 250 mL of this wastewater was treated by bubbling O_3. To determine the effectiveness of the technique, different physicochemical parameters were evaluated for both the service water, the simulated wastewater, and the supernatant of the treated effluent. At this stage, a pollutant concentration tendency was made with respect to the COD values, in order to obtain the concentration ratio that should be reached in the plant up to the maximum effluent discharge value established at 250 mg/L DOC.

A third stage was designed with a closed-circuit device, to simulate water curtain cabins used for painting processes. This device is presented in Fig. 2.

The paint sample was injected through the lower injection nozzles and distributed on the base of the drying booth. An impeller was used to circulate the flow and release the paint solvents (VOC), and the air circuit was forced to close through a bubbling column in water. This water is incorporated into the system through the fill/drain tap. The washed air is returned to the drying cabin through the return duct, generating a circulation circuit so that all the VOCs emitted by the paint are absorbed into the water column. To achieve the necessary pressure difference and generate the bubbling column, a secondary impeller was included on the return pipe. In this system, 600 mL of plant service water were loaded, and 60 mL of paint were injected through the 9 lower injection points, to then generate the evaporation of the solvents and bubble them into the water reservoir. Successive samples were taken every 5 min for 30 min, and solvent contamination was checked by measuring the COD. Subsequently, 200 mL of that sample were taken and O_3 was bubbled up to a maximum time of 8 h and the effectiveness of the method was verified by also evaluating by the final COD measurement.

In a fourth stage, the ozonation equipment was incorporated in the bubble column (Fig. 2b), so that the unconsumed O_3 is absorbed by the return pipe and generates a secondary gas phase oxidation. Two tests were carried out in this fourth stage; the first with the ozonation equipment off, similar to stage 3, but with gradual injection of paint, 10 mL, so that a saturation curve could be built every 5 min. In the second test, the ozone generator was turned on from the beginning, and the delay in the saturation was confirmed with VOCs in the absorption water. Once the saturation was reached, the paint injection was suspended, and the O_3 oxidation test continued in the closed circuit. During the treatment phase, once the maximum of absorbed VOCs in the service water had been reached, in addition to taking samples for the determination of COD, UV spectrophotometry curves were made. The equipment used for these tests was a Hach DR 5000 spectrophotometer.

Results and discussion

VOC decomposition efficiency using the ozone oxidation technique in the simulated laboratory effluent

Using distilled water

The initial COD value of the effluent simulated with distilled water was 9950 mg/L. 270 mg of O_3 were added by bubbling to the simulated effluent, and a slight turbidity was observed in the sample. The final COD was filtered and measured, and its value was 1050 mg/L. The VOCs concentrations decreased sharply when ozone was injected into the ozone oxidation reactor, which shows that ozone can react effectively with the VOCs from the simulated paint drying in aqueous media.

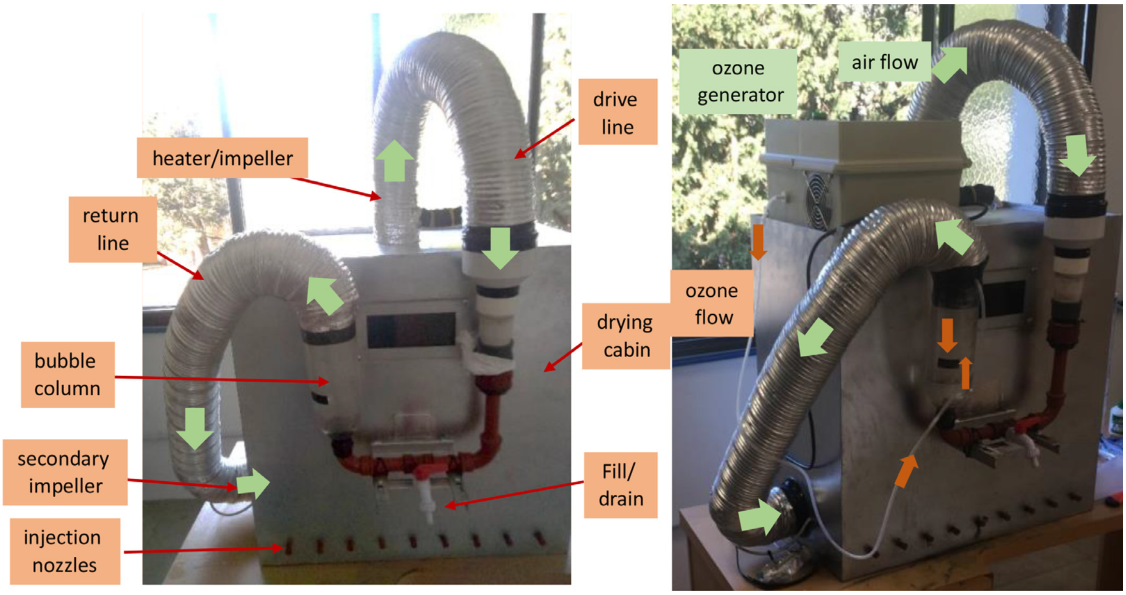

Fig. 2. a) Water curtain cabin simulator equipment. b) Cabin with attached ozone generator.

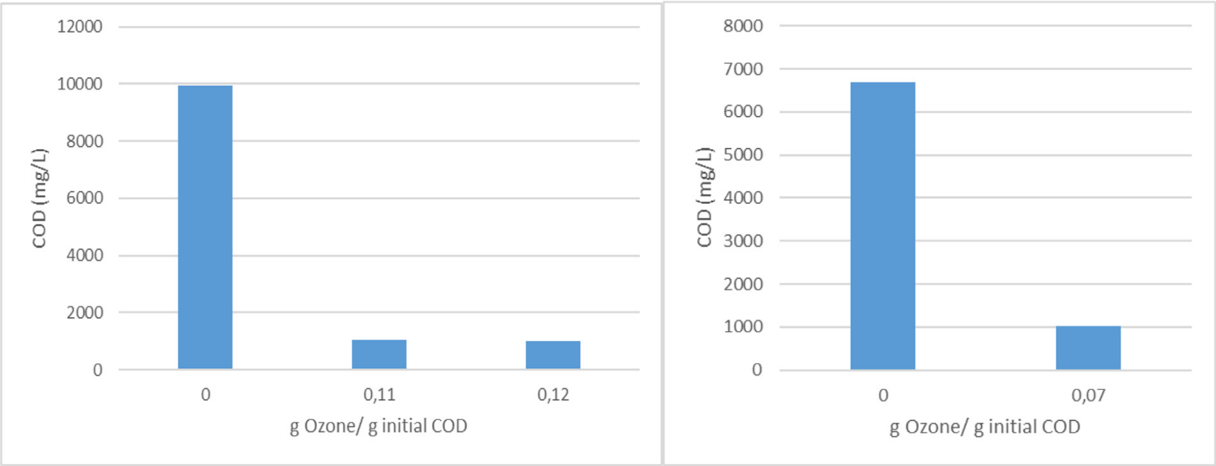

Fig. 3. Ozone oxidation test. Simulated wastewater with a) distilled water and b) service water.

Table 1
Physicochemical parameters of the samples.

Parameters	Service water	Simulated wastewater	Treated wastewater
COD (mg/L)	10	6880	1022
pH	8,14	8,19	8,94
Conductivity (µS/cm)	1428	1435	1460
Hardness (mg/L)	360	378	351
Alkalinity (mg $CaCO_3$/L)	550	500	450
Silica (mg/L)	30,2	28,5	27,4
Aluminium (mg/L)	≤ 0,05	≤ 0,05	≤ 0,05

A subsequent bubbling addition of 30 mg of O_3 to the sample reduced the COD value to 1000 mg/L (Fig. 3a).

Using service water

The service water has a negligible initial COD value of 10 mg/L, which increases considerably to 6880 mg/L when the analytical reagents are added. Subsequently, 250 mL of the simulated effluent was taken and 120 mg of O_3 was bubbled into it, reducing the COD value to 1022 mg/L (Fig. 3b). The physicochemical parameters evaluated in the service water and in the simulated wastewater before and after the O_3 oxidation test are presented in Table 1.

In both cases, using distilled and service water, the VOC concentration decreased rapidly when ozone was injected into the oxidation reactor. This result corroborates the evidence for the beneficial role of water in the ozone oxidation process as documented in previous reports (Yu and Lee, 2007; Zhao et al., 2011; Zhu et al., 2017; Huang et al., 2015). On the other hand, the pH value of the reaction solution has a significant influence on both the efficiency of direct ozonation and the generation of OH (indirect ozonation). At high pH (especially above 8),

Table 2
Service water absorption of VOCs and ozonation treatment.

E_1 - Service water contamination		E_2 - Ozone treatment			
		Contaminated service water		Simulated wastewater	
Saturation time	COD (mg/L)	Dosed ozone (g)/ g initial COD	COD (mg/L)	Dosed ozone (g)/ g initial COD	COD (mg/L)
0	10				
5	11650	0	11870	0	11610
10	11710	0,27	10220	0,56	11200
15	11770	0,68	6240	1,12	4788
20	11820	0,96	3890	1,68	2000
25	11820	1,23	2460	2,24	665
30	11870	1,78	500		

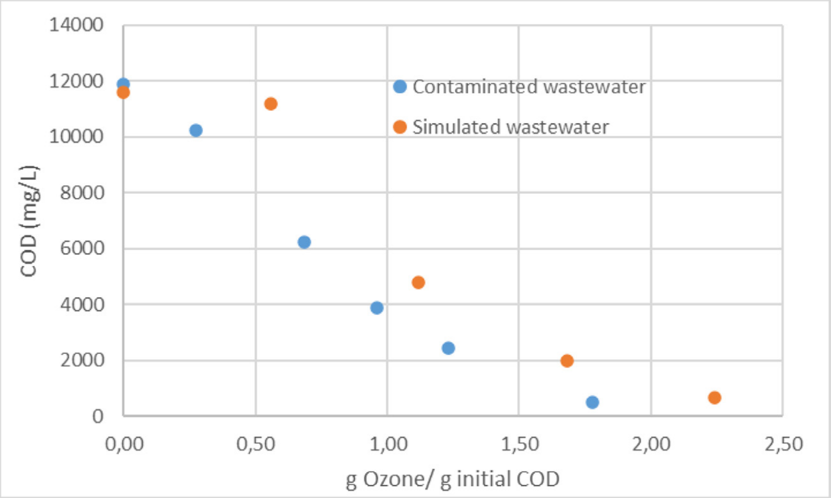

Fig. 4. E_2 – Ozone treatment. Ozonation of VOCs as a function of dosed ozone.

the abundance of OH- can improve the generation of OH, being this a usually optimal pH for ozonation treatments (Wang and Chen, 2020; Chen et al., 2020a, 2020b; De Witte et al., 2010).

Two types of tests, referred to as E_1 and E_2, were carried out with the service water and effluent simulated in the laboratory, using the closed-circuit device in Fig. 2. Table 2 refers in its first two pairs of columns to E_1, and the remaining pair to E_2. 600 mL of service water were saturated after 5 minutes and then this value remained constant until it reached 30 min. Afterwards, 200 mL of the sample saturated with VOC were taken and O_3 was injected during 6.5 h in the ozonation reactor; in the same way the simulated sample was ozonated.

Fig. 4 illustrates the variation in VOC decomposition efficiencies with the injected ozone concentration. Direct chemical reactions between ozone and VOCs would be limited when ozone concentrations were low, resulting in lower VOC decomposition efficiencies.

Finally, the equipment shown in Fig. 2 was used to carry out three tests with 600 ml of service water and the paint VOCs. The first test (E'_1) was performed to construct the saturation curve of the VOCs in the water, without adding ozone; the second test (E'_2) was performed to evaluate the effect of ozonation; the third test (E'_3) of long duration was performed to record the evolution of the reaction kinetics. The results obtained are shown in Table 3. It can be seen in the second column that the 600 ml sample is saturated with VOC after 20 min, with 40 mL of paint injected; then, in the last two columns, the contamination and simultaneous treatment tests are shown. In the first stage, ozone is injected continuously, and 10 mL of paint are added every 5 min until the first 20 min (E'_2). Once the 20 min of the test have been reached, the paint is no longer injected into the system, but the ozone bubbling continues until the end of the test at 90 min (E'_3).

Fig. 5 shows the UV absorbance curve from the saturation of the service water to the end of the treatment. There is an absorbance maximum at about 268 nm, which decreases as ozone is injected. Fig. 6 clearly shows the difference between E'_1 (only paint) and the first section of E'_2 (ozone + paint), as the VOC concentrations decreased when ozone was injected. When stopping the injection of paint and continuing with the dosage of ozone in line, E'_3, the change of slope in the reaction kinetics of the ozonation mechanism is clearly observed. The first stage is a direct ozonation of the VOCs in the presence of ozone and the second is a slower reaction, generated by the presence of •OH, favored by the pH of the solution. At the end of the test, after dosing 2.38 g of ozone, a COD removal of 94% is achieved.

Table 3
Contamination and simultaneous treatment of service water.

	Contamination E'_1	Contamination and treatment E'_2 and E'_3	
Time (min)	COD (mg/L)	Ozone (g)	COD (mg/L)
0	0	0,00	0
5	7179	0,05	3761
10	10,383	0,11	6632
15	11,407	0,16	8115
20	11,452	0,22	9393
25	11,470	0,27	9257
35		0,38	7947
45		0,43	7408
50		0,65	5384
55		0,87	3778
60		1,08	3335
65		1,30	2310
70		1,52	1624
75		1,73	1381
80		1,95	1295
85		2,17	803
90		2,38	744

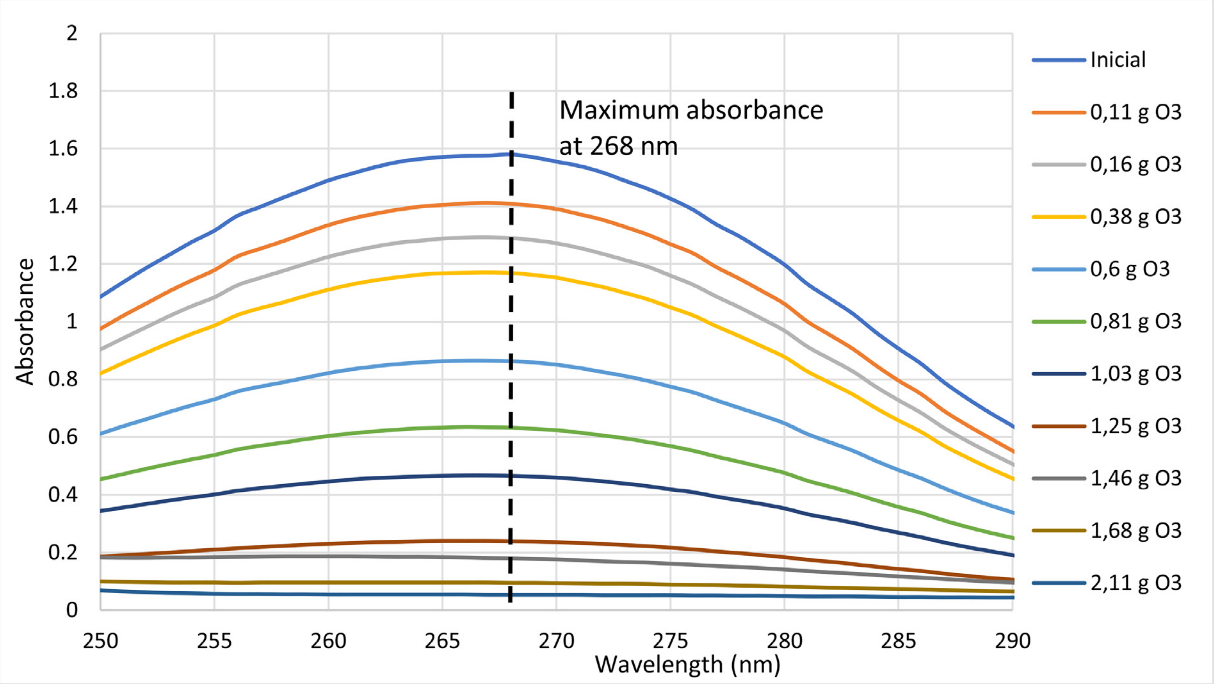

Fig. 5. UV spectra during ozonation. The decrease in absorbance evidences the conversion of the contaminants into CO_2, O_2 and OH^- (pH raised from 8.1 to 9)

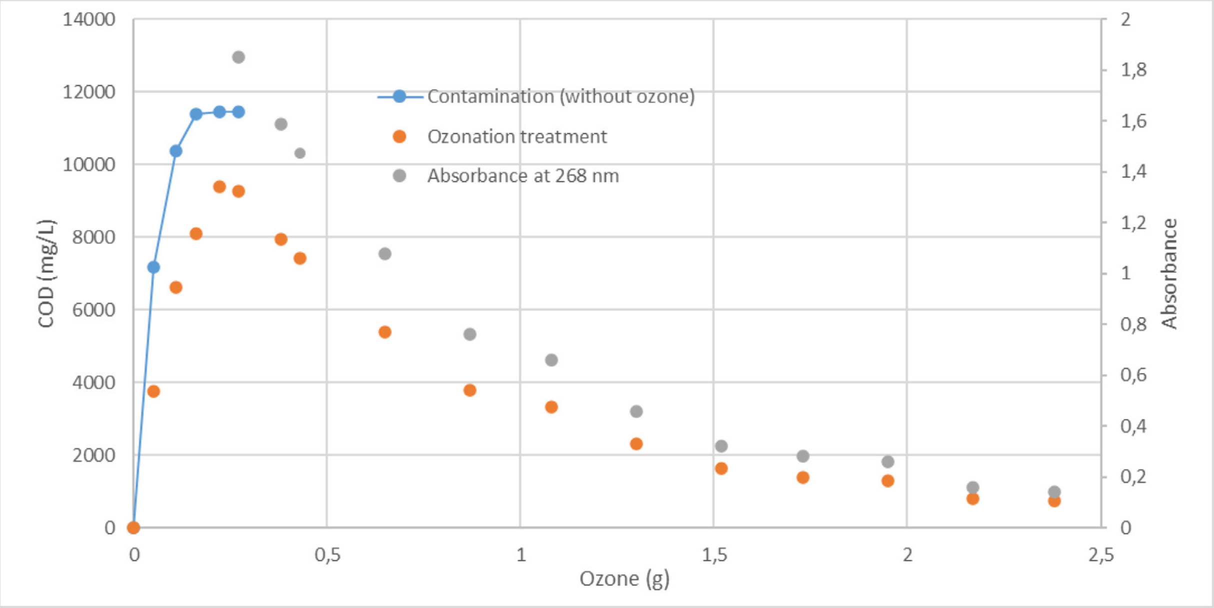

Fig. 6. Contamination and simultaneous ozonation of absorbed VOCs.

According to Merenyi (2009), in the direct reaction of O_3 with water the following reactions occur:

$$O_3 + OH^- \rightarrow HO_4^-$$

$$OH^- \leftrightarrow HO_2 \cdot + O_2^-$$

In the presence of ozone, OH• would be generated through the following equations:

$$O_2^- \bullet + O_3 \rightarrow O_2 + O_3^- \bullet$$

$$O_3^- \bullet \ \rightarrow O_2 + O^- \bullet$$

$$O^- \bullet + H_2O \rightarrow \ \bullet OH + OH^-$$

Usually the concentrations of radicals are low (less than 10^{-12} M), but their reaction rates with the organic compounds are very high, which makes the contribution in the total oxidation of organic compounds significant (Kasprzyk-Hordern et al., 2003; Boncz, 2002). The radicals formed as secondary oxidants, the hydroxyl radical (HO•) and also the superoxide anion radical (O_2^-•) (Tian et al., 2020a, 2020b) are very reactive, but not very specific (Schmitt et al., 2020). As mentioned, the free radicals formed in the decomposition of ozone almost always intervene in the oxidation processes. The most important radicals found in a solution containing ozone are the hydroxyl radical (HO•), the superoxide radical (O_2^-•) and the hydroperoxyl radical (HO_2•) (Tian et al., 2020a, 2020b). Ozone is the most selective oxidant (three orders of magnitude between the reaction rates for the slowest and the fastest

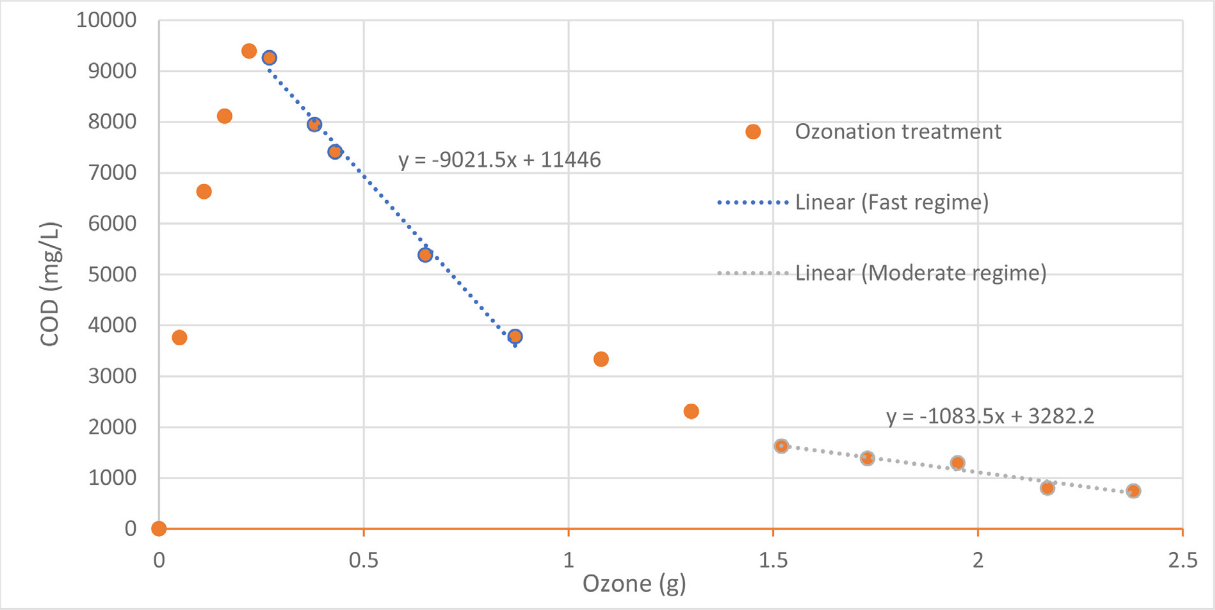

Fig. 7. . Regime transition in ozonation treatment. In the rapid stage the ratio of g/L of COD removed per gram of ozone is 9.02; in the moderate stage it decreases to 1.08.

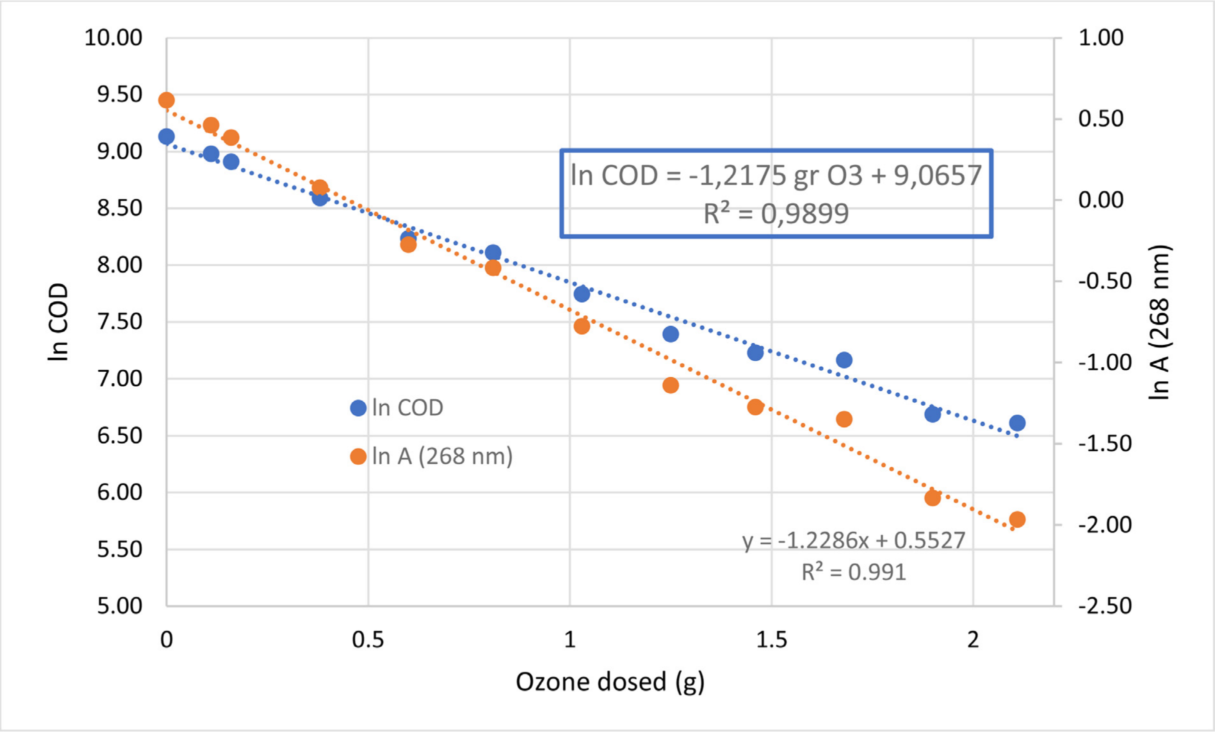

Fig. 8. Representation of ln COD and ln of absorbance at 268 nm versus ozone dosed. The oxidation reaction follows a first order kinetic.

reaction) and $O^{-}\bullet$ and $HO\bullet$ are the least selective, as can be seen from the results tests in Fig. 7, where a rapid reaction is observed up to the addition of 1 g of ozone (approximately 60 min of reaction) and then transitions to a moderate reaction rate (Deng, 2020). This rapid reduction of effluent COD followed by a slower regime was also reported by Suryawan et al. (2020) in the treatment of textile effluents and by Yang et al. (2019) in the ozonation of effluents from the chlor-alkali industry.

If the data from the E’3 test is considered, and the natural logarithm of COD versus dosed ozone is plotted, the points fit on a straight line (Fig. 8). This observation is repeated when analyzing the variation of the logarithm of the absorbance at 268 nm, also versus the grams of ozone injected, which indicates that the ozonation of the absorbed pollutants is due to a first order kinetic:

$$\frac{dC}{dt} = -k.C$$

$$\int_{C_o}^{C_f} \ln C = -k.t$$

Since the added ozone is a function of the treatment time, it can be expressed according to the regression of Fig. 8 where:

$$\frac{d\ COD}{d\ gr\ O_3} = -1,2175\ .\ COD$$

$$\ln COD = -1,2175\ .\ gr\ O_3 + 9,0657$$

According to this model, an effluent with less than 200 mg/L will be achieved by adding 3.1 g of ozone, that is, in about 4 h 45 min of treatment. As for the products obtained by the treatment, Kharel et al. (2020) found that, in the ozonation of pharmaceutical effluents, a proportion of 0.7 to 1.0 mg O_3 for each mg of dissolved organic carbon (DOC) reduces the formation of harmful by-products to insignificant levels. According to their recorded values, this corresponds to about 0.28 to 0.40 mg O_3 each mg of COD; the effluent treated in this work yields a proportion of 0.35 mg O_3 each mg of initial COD, remaining within the range mentioned by Kharel. In turn, Gunten records that the reaction of ozone with formaldehyde, through the formation of the formate ion, is (Gunten, 2003):

$$CHO_2^- + O_3 \rightarrow CO_2 + O_2 + OH^-$$

This reaction is consistent with the fact that the pH of the treated effluent increased from 8.1 to about 9 in the treatments performed. In our case study, in addition to the oxidation of formaldehyde, the ozonation of the rest of the contaminants (butyl acetate, ethanol, methyl ethyl ketone) gives the same reaction products, based on the UV curves where a strong reduction in effluent absorbency can be seen.

Conclusions

This study successfully developed a continuous flow reaction system to effectively remove VOCs from paint drying. The continuous flow reaction system combines two techniques: the water VOC absorption technique, and the ozone oxidation technique.

Ozone in water undergoes a decomposition process and is converted into hydroxyl (HO•) radicals, the superoxide radical (O_2^{-}•) and the hydroperoxyl (HO_2•) radical, for subsequent VOC reactions, and the oxidation reaction follows a first order kinetic.

It was shown that the decomposition efficiency of VOCs in water increased with the concentration of ozone, reaching a COD removal of 94%. The closed circuit allowed the treatment efficiency to be increased by recapturing any ozone that might escape from the water in the bubble column.

Declaration of Competing Interest

None.

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