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The different effects of oxygen and air DBD plasma byproducts on the degradation of methyl violet 5BN

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ABSTRACT

Through a novel design of the dielectric barrier discharge (DBD) plasma plume used in fabric-fiber surface modification, its discharge byproducts mainly including downstream gases and ultraviolet light were used to treat the dye solution. The different influence of oxygen and air DBD plasmas on the degradation of methyl violet 5BN (MV-5BN), which is widely used in textile industry, was investigated in this paper. The results showed that the cooperation between ultraviolet light and active species generated by the DBD plasma can decolorize MV-5BN effectively, and the chromophore peaks attributed to the –N═N– bonds in MV-5BN molecule disappeared entirely when the azo dye solutions were treated for 25 min by the air and oxygen DBD plasmas. The degradation reaction followed an exponential kinetics over time, and the peak of aromatic derivatives at 209 nm in UV–vis spectra increased nearly 2.7 times when the dye solution was treated for 30 min by air DBD plasma. However, the oxygen DBD plasma could deplete the aromatic derivatives entirely. It is found that the formation of O_3 and NO_x in the downstream gases of air and oxygen plasmas may be responsible for the different effects on the azo dye degradation.

1. Introduction

Organic synthetic dyes have been widely used as colorants in different industries such as textile, paper, color photography, pharmaceutical, food, and cosmetic. More than 0.7 million tonnes of organic synthetic dyes are produced annually worldwide, and nearly 12% of these synthetic dyes are lost during manufacturing and processing operation [1,2]. From the textile industries alone, the organic dyes have imposed high stress on the environment, especially for China who has the biggest textile industries in the world. Wastewater from the textile-dyeing industry has an unstable pH, high temperature and a high concentration of coloring material. Azo dyes, which hold 70% of all reactive dyes, are the most extensively utilized dyes and are normally major pollutant in dye effluents [3]. Due to the toxicity and solubility of wastewater containing azo dye, it is very difficult for the conventional effluent treatment techniques including adsorption and biological oxidation to degrade the azo dye solutions. Furthermore, these physical and biological processes produce secondary wastes.

In the past few years, several advanced oxidation processes (AOPs), such as UV–TiO_2 catalysis [4–6], nonthermal plasma technology [7–10], supercritical water oxidation [11,12], have been explored to degrade dye pollutants. These technologies have shown great potential in the decontamination of recalcitrant wastewater coming from the textile industry. DBD plasma based technology for wastewater treatments is an AOP because the high oxidation potential species (O_3, O, etc.) are generated in aqueous discharge system. Mok et al. [7] used a DBD plasma system to treat the azo dye, and the energy requirement for the degradation was only 0.654 kJ/mg which is far lower than those consumed by the ultraviolet/photocatalytic approach.

It should be noted that DBD plasma, especially the atmospheric pressure glow discharge, is a potential technique for fiber surface modification in textile industry. For example, Borcia et al. [13] used the DBD plasma to pretreat natural and synthetic textiles, and the textiles wettability was increased remarkably; Nema and coworkers [14] also modified the rabbit fibers by DBD plasma, and the shedding of the fiber decreased obviously. In recent years, our team has developed a room temperature DBD plasma plume [15–17], and it is more suitable to treat the textile than the ratio frequency plasma jet ($T > 375$ K) [18]. Normally, air and oxygen are introduced into the plasma plume to modify the fabric fibers. Thus, the byproducts (e.g. O_3, UV), which are unfriendly to the surrounding and human health, are formed in the plasma modification system. It is well known that the ozone contained in the byproducts is one of the most effective materials for the degradation of water contaminants [6,19–21]. Is it possible to design a novel DBD plasma plume system which not only modifies the textile but also treats the dye pollu-

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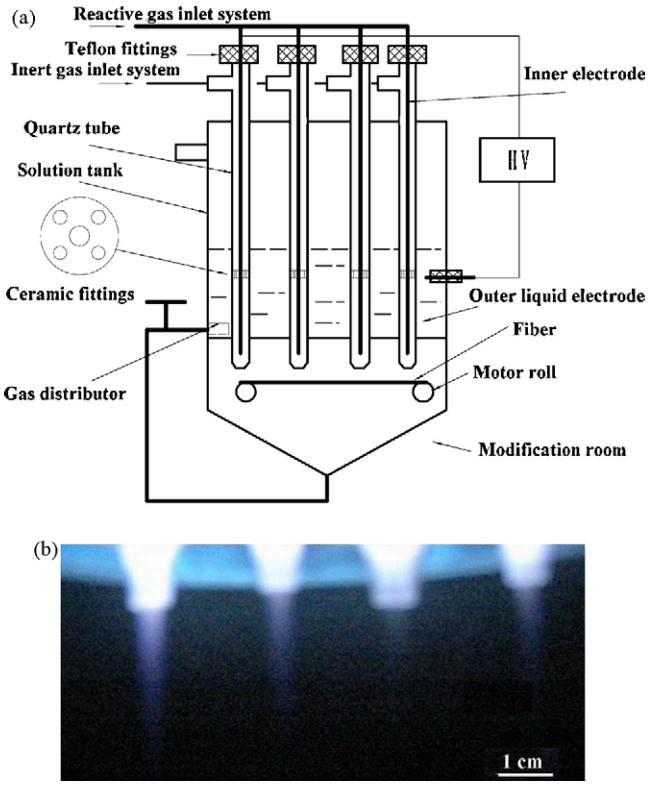

Fig. 1. (a) The schematic diagram of the plasma plume array for textile modification and dye degradation. (b) A photograph of the plasma plume array with an Ar velocity: 5 L min^{-1}; power: 50 W.

tants simultaneously by its byproducts? What will take place when the azo dye solution is treated by the byproducts of air and oxygen plasma plumes? Inspired by these questions, we chose the azo dye of MV-5NB as the target contamination to examine the effects of the byproducts of air and oxygen DBD plasma plumes.

2. Experimental

2.1. Experimental apparatus

Fig. 1 shows the schematic diagram of the experimental apparatus for the decomposition of MV-5BN. Through a special design, a DBD plasma micro-plume array was built. This system cannot only modify the textile homogeneously but the byproducts are also used to degrade the dye solution. In the reactor, four quartz tubes (inner diameter: 6 mm, outer diameter: 8 mm) were used as the dielectric layer and were fixed in an organic glass tank by the Teflon fittings. The copper tubes (inner diameter: 3 mm, outer diameter: 4 mm) fixed in the quartz tubes center by ceramic fittings acted as one electrode, and the dye solution contained in the tank acted as the outer electrode. The two electrodes were connected to an AC power with a maximum peak voltage of 30 kV and adjustable frequencies ranging from 8 kHz to 30 kHz. The dye solution was in a closer contact with the dielectric layer than the metal electrode. Therefore, the distribution of the electrical field became more uniform, which was a desirable feature for generating homogeneous plasma. On the other hand, the liquid electrode served as the coolant of the system.

In order to explore the effect of DBD plasma byproducts on the azo dye degradation, the joint caps were connected to solution tank and were closed to cover the outlet of modification room. The active gases (air and oxygen) transported to the discharge area by the inner electrode were controlled by a mass flowmeter, and the inert gas (Ar) flow was adjusted by a normal flowmeter. The byproducts from the plasma plume, as shown in Fig. 2(b), were transported by a glass tube and bubbled in the solution tank. At the same time, the ultraviolet light generated from the discharge area was absorbed by the dye solution. As a result, the joint influence of the byproducts from both emission lights and oxidization species formed in the discharge system on the dye degradation was achieved.

2.2. Experimental and analytical methods

The simulated dye solution was prepared by dissolving a given amount of an azo dye MV-5BN in distilled water. The molecule of MV-5BN was shown in Fig. 2, and the concentration of the dye solution was 22.4 mg/L. The ozone-containing gas from the DBD plasma reactor contacted the organic contaminant by the gas distributor and the samples were taken from the wastewater tank at preselected times. The solution samples were tested with a UV–vis spectrophotometer (Hitachi, U-3010) to monitor the degradation of MV-5BN, and the areas of peaks at 580 nm (–N=N–) and 209 nm in UV–vis spectrophotometer were used to calculate the degradation kinetics of the azo dye and the aromatic derivatives, respectively. The ozone acting as the indicator of the oxidation species was measured by an O_3 analyzer (USA-EPA, Model 49), and it should be noted that far lower air and oxygen velocity than the actual application

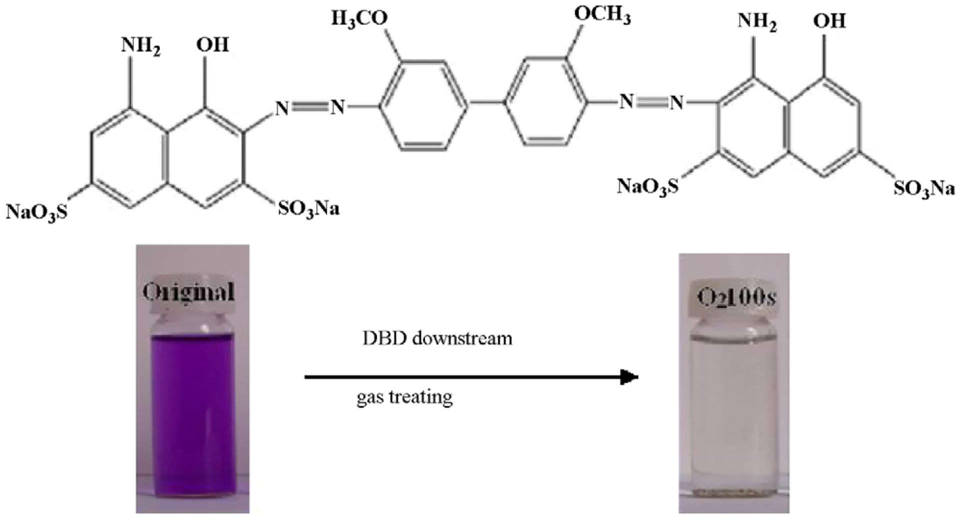

Fig. 2. The molecular structure of the MV-5BN and its color change treated by DBD downstream gas.

in the dye degradation was used to simulate the O_3 concentration because the maximum measuring range of the O_3 analyzer was only 1 ppm. The downstream gas compositions of air DBD plasma were analyzed by a gas analyzer (Testo Germany, Testo-360). A commercial O_3 generator (Hengtai China, JC-7/100W) was also used to make a comparison to our DBD system and to estimate the dye degradation efficiency by using the byproducts generated in the DBD system.

3. Results and discussion

3.1. The azo dye degradation by air and oxygen DBD plasmas

Fig. 3 shows the relationship between the absorption spectrum of MV-5BN solution and the treatment time by an air DBD plasma. In the range of UV–vis light, there are four obvious absorbance peaks for the original MV-5BN solution at wavelengths of 209, 244, 300 and 580 nm, respectively. When the azo dye solution was treated by the air and Ar DBD plasma for 1440 s, the maximum absorbance peak at 580 nm disappeared entirely. As a result, the MV-5BN solution was decolorized. This indicated that the chromophores of azo bonds (–N=N–) responsible for the violet color (see Fig. 2) were broken down when the dye molecules were exposed to the ultraviolet light and ozone generated in air DBD plasma. The inset in Fig. 3 presents the area change of peaks at 209 nm with different treatment times. Compared to the peak at 508 nm, the peak intensity at 209 nm shows a reversed process with the treating time, and the absorbance of sample treated for 1440 s is 2.7 times larger than the original dye solution. The phenomenon disclosed that the double azo bonds in MV-5BN molecule were broke down entirely when the dye solution met the byproducts of air plasma. Thus, the concentration of aromatic derivative in solution was improved three times, and the only 2.7 times improvement in peak area at 209 nm indicated that a few of aromatic derivatives were degraded in the treatment process of air DBD plasma. Fig. 4 shows the dye concentration decrease in solution over time. It can be seen that the degradation of dye solution followed an exponential kinetics. Plotting $\ln(C_t/C_0)$ versus time yielded a linear relationship (see the inset of Fig. 4), and the simulated degradation rate constant (k_a) was about $0.0019\,s^{-1}$ when the discharge power and air velocity were 40 W and $0.3\,m^3\,h^{-1}$, respectively.

Fig. 5 displays the UV–vis spectral changes of MV-5BN solution treated by the oxygen and Ar DBD plasma. When the treatment time was increased, the peaks at 580 nm exhibited a similar change as seen in the air plasma, and an exponential kinetics was also found between the dye concentration and the treatment time. However, the k_a of dye solution in the oxygen plasma was bigger than that in the air plasma under the same discharging conditions. Moreover, the peak at 209 nm (the inset figure in Fig. 5) disappeared entirely when the dye solution was treated by oxygen DBD plasma for 24 min. The results indicated that most of the aromatic derivatives were degraded by the oxidization species generated in the oxygen plasma.

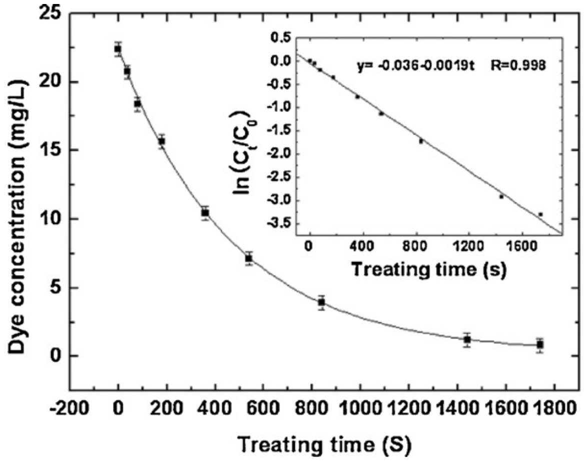

Fig. 4. The dye concentration evolution over treatment time at an air velocity of $0.3\,m^3\,h^{-1}$, an argon velocity of $0.1\,m^3\,h^{-1}$ and a discharge power of 40 W. The inset is the plot of $\ln(C_t/C_0)$ versus time.

The oxygen flow rate greatly affects the quantities of active species formed in the discharge system. In addition, this rate determines the residence time of the gas in the reactor as well as the reaction time of the active species (such as O_3) with pollutant molecules in solution, which finally determines the utilization ratio of the active species and the production costs. Fig. 6 shows the effect of the oxygen flow rate on the dye degradation. As the gas flow rate was increased, more reactive species generated in the plasma reactor diffused into the liquid phase through the gas distributor and then reacted with the azo dye. Moreover, the increased gas velocity strongly agitated the dye solution, which may cause more dye molecules to interact with the active oxidizing species. Thus, the dye degradation efficiency was improved with increased oxygen velocity.

In the dye treating process, the byproducts of the air and oxygen plasma plumes could degrade the MV-5BN effectively, and the decolorization reactions were found to follow a pseudo first-order

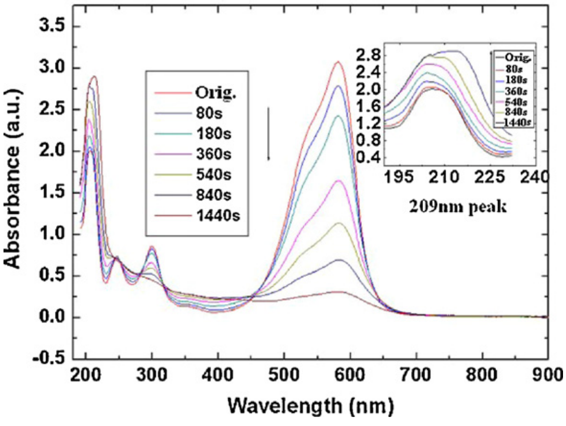

Fig. 3. The UV–vis spectra of MV-5BN solution treated by the air DBD plasma at an air velocity of $0.24\,m^3\,h^{-1}$, an argon velocity of $0.1\,m^3\,h^{-1}$ and a discharge power of 40 W.

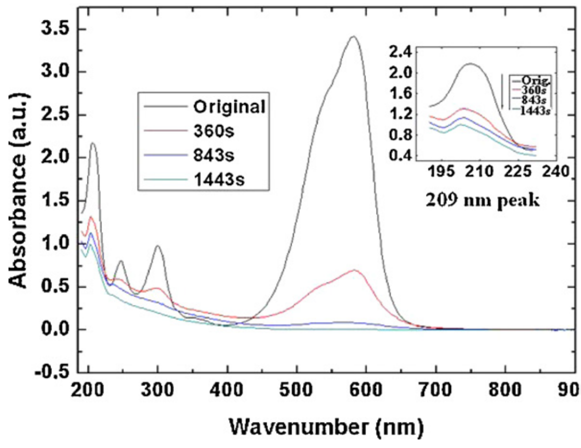

Fig. 5. The UV–vis spectra of MV-5BN solution treated by the oxygen DBD plasma at an oxygen velocity of $0.2\,m^3\,h^{-1}$, an argon velocity of $0.1\,m^3\,h^{-1}$ and a discharge power of 40 W.

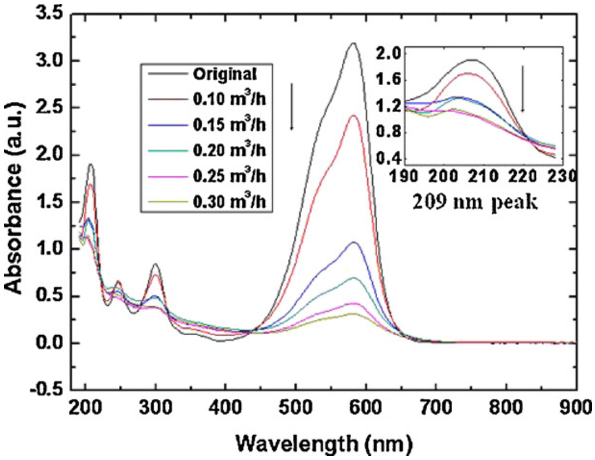

Fig. 6. The effect of oxygen gas velocity on the dye degradation at an argon velocity of 0.1 $m^3 h^{-1}$, a discharge power of 40 W and a treatment time of 360 s.

kinetics which was consistent with other results [3,10]. The oxidizing species in dye solution, such as O_3, $OH^\bullet$, played an important role to decolorize the pollutants in the plasma system. In our previous work [17], we found that no obvious reaction occurred when the dye solution with low concentration was treated by the ultraviolet light alone. However, the ultraviolet light increased O_3 decomposition and hydrogen peroxide (H_2O_2) formation, and therefore, more hydroxyl radicals were generated as follows [22,23]:

$$O_3 + h\nu \rightarrow O_2 + O \quad (1)$$

$$O + H_2O \rightarrow OH^\bullet + OH^\bullet \quad (2)$$

$$O + H_2O \rightarrow H_2O_2 \quad (3)$$

$$H_2O_2 + h\nu \rightarrow OH^\bullet + OH^\bullet \quad (4)$$

Furthermore, the aromatic derivatives could not be removed even if the air plasma containing the same amounts of oxygen has the same discharge conditions as the oxygen plasma.

3.2. The byproducts composition measurement in the air and oxygen plasma plume

In order to explore the reasons that caused the different effects on the MV-5BN degradation between the oxygen and air plasma, it is very necessary to analyze the compositions of the downstream gases generated in the air and the oxygen DBD plasmas, respectively. Fig. 7(a) shows the effect of discharge power on the ozone concentration in air and Ar plasma plume. When the applied discharge power was increased, more electron avalanches were generated in the plasma plume, and the ozone concentration at the plasma reactor outlet was also improved. On the other hand, the dose of the ultraviolet light was also promoted with the stronger discharge intensity. Therefore, the dye degradation ratio was improved when a higher discharge power was used in the air plasma plume.

Compared to the air and Ar plasma plume, the ozone concentration at oxygen plasma reactor outlet was enhanced greatly when the same discharge power was applied in the plasma plume system. A linear relationship was found as shown in Fig. 7(b). From the figure, the ozone concentration was only 110 ppb when the discharge power was 12 W, but 400 ppb ozone was produced in the oxygen plasma. The main ozone generation reactions are described below [24]:

$$e^* + O_2 \rightarrow O + O + e \quad (5)$$

$$O + O_2 + M \rightarrow O_3 + M \quad (6)$$

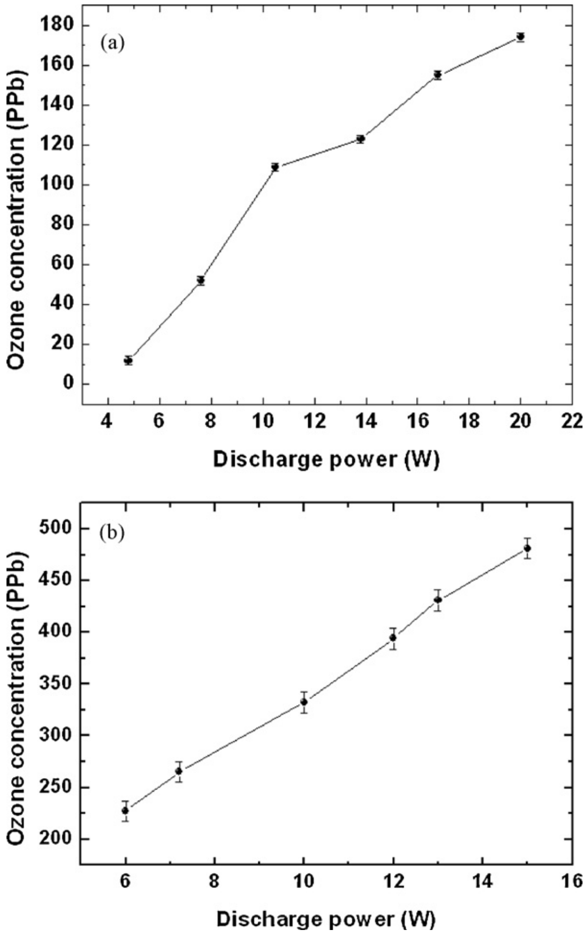

Fig. 7. The relationship between the discharge power and ozone concentration in the air plasma at an argon velocity of 0.1 $m^3 h^{-1}$ and a discharge time of 10 s. (a) The air mass is 5 sccm and (b) the oxygen mass is 1 sccm.

In the air plasma, nitrogen atoms may consume some electrons because the ionization energy of nitrogen atom (14.5 eV) is similar with oxygen ionization energy (13.6 eV), and some NO_x compounds were formed in the following possible reactions [10,24,25]:

$$e^* + N_2 \rightarrow N + N + e \quad (7)$$

$$e^* + N_2 \rightarrow N_2(a'1\Sigma^-{}_u) + e \quad (8)$$

$$N + O \rightarrow NO \quad (9)$$

$$O + NO \rightarrow NO_2 \quad (10)$$

From reactions (5)–(10), it can be seen that the nitrogen molecules in air not only quench some electrons but also combine with some oxygen atoms (O). Therefore, the ozone concentration in the air plasma is far lower than in the oxygen plasma under the same discharge conditions. Fig. 8 presents the concentration evolution of NO and NO_2 in air plasma as a function of discharge time. The NO and NO_2 concentrations exhibited a similar reaction as described by Eqs. (9) and (10). In the initial discharge stage, the NO concentration was about 25 ppm, and the NO_2 was only 2 ppm. When the discharge time was increased to 20 s, the NO concentration decreased to a stable value (3 ppm). Meanwhile, an inflexion occurred on the line of NO_2 reactive rate, after which the NO_2 concentration increased slowly. It is well known that some acids, such as HNO_3, HNO_2, are formed from the dissolution of nitrogen oxides, as described in Eqs. (11)–(13) [10].

$$2NO_2 + H_2O \rightarrow 2H^+ + 2NO_3{}^- + NO \quad (11)$$

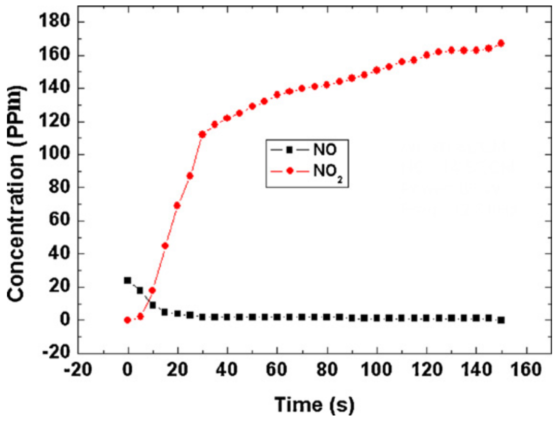

Fig. 8. The influence of discharge time on the NO and NO_2 concentrations with an air mass of 80 sccm, an argon velocity of 0.1 $m^3 h^{-1}$ and a discharge power of 48 W.

$$2NO_2(g) \rightarrow 2N_2O_4 + H_2O(l) \rightarrow HNO_3 + HNO_2 \quad (12)$$

$$NO_2(g) + NO(g) \rightarrow 2N_2O_3(g) + H_2O \rightarrow 2HNO_2 \quad (13)$$

Therefore, the pH of dye solution decreased when the MV-5BN dye was treated by the air plasma. Certainly, the pH of dye solution treated by the oxygen plasma also decreased due to the formation of carboxylic acids generated from the MV-5BN oxidization. It was reported that the direct attack of hydroxyl radicals ($HO^{\bullet}$) on many organic compounds was strongly affected by pH [26]. Under acidic conditions, more $HO^{\bullet}$ radicals were generated, and this led to the increased degradation efficiency of MV-5BN. According to Du et al. [10], the pH of azo dye solution treated by the air plasma was a litter lower than that by the oxygen plasma. Compared to the theory analysis and the experimental results, the pH value was not the main factors that caused the different effects on the MV-5BN degradation under the air and oxygen plasma plume.

It is well known that the profit of textile-dyeing factories becomes lower and lower with the intensified competition, and the production cost will decrease in textile-dyeing factories if the plasma byproducts can be used effectively. Fig. 9 illustrates the comparison of degradation rate between the byproducts and the commercial O_3 generator. Under the same reactive conditions, the dye peak intensity at 580 nm treated by the DBD byproducts was about two times stronger than the commercial O_3 generator. Although the dye degradation rate for the DBD system was only half of the O_3 generator, the byproducts application was still a potential choice to reduce the production cost for textile-dyeing industry.

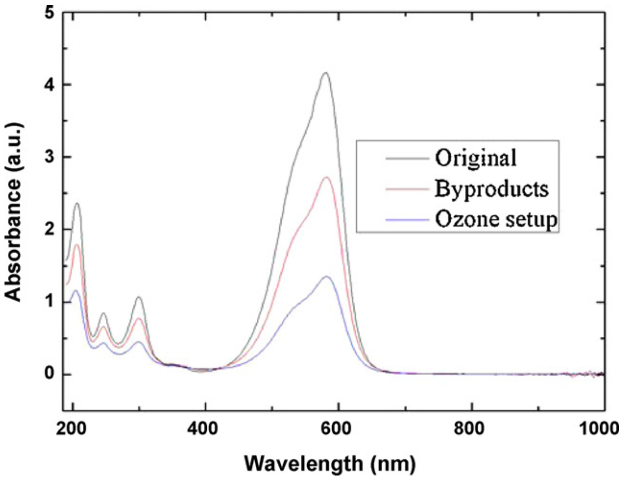

Fig. 9. The comparison of MV-5BN degradation ratio between the DBD byproducts and the commercial ozone generator with a oxygen velocity of 0.1 $m^3 h^{-1}$, a discharge power of 50 W and a treating time of 90 s.

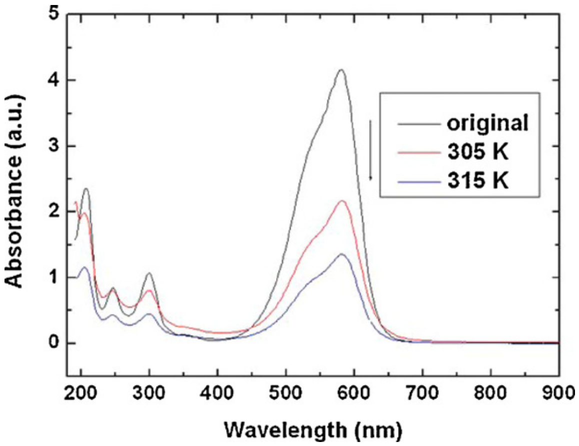

Fig. 10. The influence of solution temperature on the MV-5BN degradation with an oxygen and argon velocity of 0.2 $m^3 h^{-1}$, a discharge power of 40 W and a treatment time of 80 s.

3.3. *The effect of temperature on the dye degradation*

The influence of temperature on the dye degradation is shown in Fig. 10. For the oxygen plasma, the peaks intensity at both 580 and 209 nm decreased when the MV-5BN solution temperature was increased from 305 to 315 K. Normally, the increased temperature can enhance the reaction rate and improve the activity of the species, thus increasing the dye decomposition rate in the solution. On the other hand, increasing temperature accelerates the reactive radicals to collide and leads to quenching. Thus, the dye degradation rate may decrease. In our study, high temperature favored the degradation more, and its negative influence can be neglected.

4. Conclusion

In this paper, the unfriendly byproducts of air and oxygen plasma plumes were used to treat the azo dye MV-5BN, and the different effects on the dye degradation were explored. The experimental results show that the downstream gases of air and oxygen plasmas can decolorize the MV-5BN solution effectively. The aromatic derivatives were nearly degraded entirely by the byproducts of oxygen plasma. The nitrogen gas in air plasma and the NO in downstream gas consumed some portion of ozone. Moreover, the acidic gases generated in the air plasma lowered the solution pH and inhibited the degradation rate of intermediate products to form carboxylic acids. Therefore, the lower ozone concentration and acidic gases generation might be the reasons that the air plasma byproducts could not degrade the MV-5BN entirely. However, the toxicity of intermediate products is far lower than the parent compound, and can be further decayed by the biodegradation method. Based on the above results, it proved that the DBD plasma plume array is a potential technique at low cost for textile surface modification and dye degradation.

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