



Combination of photocatalysis downstream the non-thermal plasma reactor for oxidation of gas-phase toluene

Haibao Huang^{a,b}, Daiqi Ye^{a,*}

^a College of Environmental Science and Engineering, South China University of Technology, Guangzhou 510006, China

^b Research Center of Environmental Engineering and Management, Graduate School at Shenzhen, Tsinghua University, Shenzhen 518055, China

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ABSTRACT

Ozone is an undesirable byproduct of non-thermal plasma (NTP) for volatile organic compounds (VOCs) control. Photocatalysis combined downstream the NTP reactor and ozone was utilized to oxidize toluene. The multiple synergies of $O_3/UV/TiO_2$ system and the mechanism of toluene decomposition were investigated. The influence factors such as energy density, humidity and UV sources were also intensively studied. The combination of photocatalysis in the post-plasma increased the conversion of toluene and ozone to almost 80 and 90%, respectively. Water vapor played a dual role in toluene destruction and ozone removal. In total, 0.75% humidity had the best conversion of toluene and ozone at these experimental conditions. The conversion of toluene enhanced with increasing ozone removal. Among the multiple oxidation processes in the $O_3/UV/TiO_2$ system, the O_3/TiO_2 process played a key role in the decomposition of toluene.

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1. Introduction

The use of NTP to decompose VOCs in the air exhaust from industrial processes has been extensively studied. It has unique properties including a quick response at ambient temperature, system compactness and ease of operation. On the other hand, NTP alone has disadvantages such as low energy efficiency and undesirable byproducts such as ozone [1–4], which may cause additional environmental pollution. An attempt to overcome these limitations is the combination of NTP with catalysis [5,6].

In recent years, many researchers have tried the combination of NTP with a photocatalyst, namely a plasma-activated or plasma-driven photocatalysis system, and reported that there was a synergistic effect in the system [7–9]. Some studies reported that the photocatalyst was activated by high-energy particles in the plasma instead of UV irradiation [7,10]. However, our previous study indicated that catalytic ozonation rather than photocatalysis activated by plasma played the dominant role in VOCs removal [11]. Other studies considered that photocatalysis was activated by the internal UV light emitted from the plasma [7,12,13]. However, Sano et al. [14], Ogata et al. [15] and our group [16] have reported that the UV light emitted from NTP was very weak and its contribution to toluene removal in this system could be ignored.

In order to enhance the photocatalysis process, external UV light was added into the plasma-activated photocatalysis system, and a novel system of plasma-synergized photocatalysis was formed [3,17,18]. Compared with the earlier system, the new system could increase the removal efficiency of VOCs. The present study mainly focuses on the combination of photocatalysis with the plasma *in situ*. However, this system still has the drawback of ozone pollution. Ozone itself is harmful to human health and should be removed before emission. To our knowledge, the role of ozone in the VOCs destruction was not studied in any of the previous studies. Our previous study has primarily shown that photocatalysis combined with the post-plasma was more efficient in toluene removal and O_3 control than the current plasma-synergized photocatalysis system [16].

In this study, ozone, the undesirable byproduct of NTP used in VOC control, was utilized to oxidize toluene by combining photocatalysis in the post-plasma. The multiple synergies of $O_3/UV/TiO_2$ system, the mechanism of toluene decomposition, and the factors influencing toluene removal (energy density, humidity and UV sources) were also thoroughly investigated.

2. Experimental

2.1. Experimental setup

The experimental setup is illustrated in Fig. 1. Gas samples from the outlet were analyzed on-line by a gas chromatograph

* Corresponding author. Tel.: +86 20 39380516; fax: +86 20 39380518.
E-mail address: cedqye@scut.edu.cn (D. Ye).

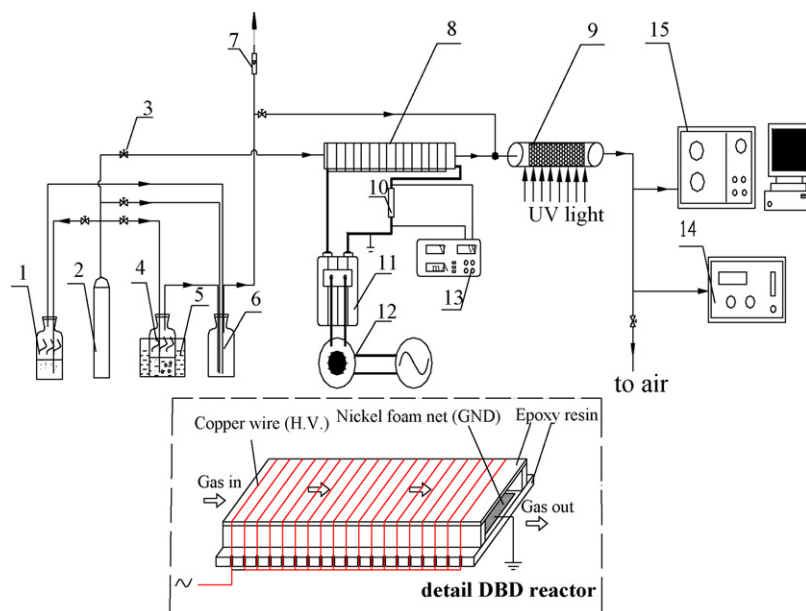


Fig. 1. Schematics of the experimental setup. (1) bubbling for water vapor; (2) dry air cylinder; (3) mass flow controller; (4) bubbling for toluene; (5) water bath; (6) buffer; (7) valve; (8) DBD reactor; (9) post-plasma reactor; (10) resistance; (11) AC transformer; (12) voltage regulator; (13) digital power meter; (14) ozone analyzer; and (15) GC.

(Kechuang, GC-900A). Ozone was monitored by an ozone analyzer (Lida, DCS-1). The experiment was carried out at room temperature and atmospheric pressure.

A wire-plate dielectric barrier discharge (DBD) reactor was used in the experiment. The post-plasma reactor was cylindrical and connected to the DBD reactor in series. This reactor was made of quartz glass, with an outer diameter of 30 mm, an effective length of 150 mm and a thickness of 1 mm. The photocatalyst was placed in the axial plane of the cylinder. In order to exclude the toluene decomposition by NTP and simplify the investigation of the post-plasma, the air stream containing toluene was directly introduced into this reactor. Post-plasma is the downstream zone of the plasma, in which long-lived ozone is usually considered as the most important oxidant. A total of 0.1 L/min air flow was fed into the DBD reactor, and then mixed with another 0.1 L/min air-flow containing toluene at the inlet of the post-plasma reactor. The total air-flow rate, the initial concentration of toluene, ozone and water vapor at the inlet of the post-plasma reactor were 0.2 L/min, 50 ppmv, 138 ppmv and 0.25 wt.%, respectively. UV radiation was provided by a 254 nm low-pressure mercury UV lamp (Philips) with approximately 16 W maximum output.

2.2. Catalyst preparation

The photocatalysts (P-25, Degussa, with the surface area of $56 \text{ m}^2/\text{g}$ and mean diameter of 18 nm) were dispersedly loaded on $\gamma\text{-Al}_2\text{O}_3/\text{nickel foam}$. The photocatalysts were prepared by impregnation method, and the preparation methods have been described in detail in our previous work [11]. Briefly, the commercial nickel foam support was impregnated in the sol of $\text{Al}_2\text{O}_3 \cdot n\text{H}_2\text{O}$, then dried at 373 K for 2 h and calcined at 723 K for 4 h to get $\gamma\text{-Al}_2\text{O}_3/\text{nickel foam}$ support. The P-25 photocatalyst was put into distilled water and dispersed fully in an ultrasonic cleaner bath to get 8 wt.% TiO_2 slurry. And the $\gamma\text{-Al}_2\text{O}_3/\text{nickel foam}$ support was dipped into the TiO_2 slurry, impregnated for 30 min and then dried in an oven at 100°C for 2 h.

2.3. Catalyst characterization

The photocatalyst was characterized with Field Emission Scanning Electron Microscopy (SEM, LEO, 1530 VP) and Energy Dispersive Spectrometry (EDS, Oxford, Inca 300).

3. Results and discussion

3.1. Characterization of catalysts

3.1.1. SEM

Fig. 2 presents SEM images of the photocatalyst at various magnification times ($50\times$, $500\times$, $5000\times$ and $50,000\times$, respectively). This figure illustrated that nickel foam had a pentagonal framework, and there were a lot of small holes on the surface of the nickel foam, which led to a large surface area. TiO_2 was loaded in a dispersed state on the surface of $\text{Al}_2\text{O}_3/\text{nickel foam}$. $\text{Al}_2\text{O}_3/\text{nickel foam}$ is a special support with a high surface area. It was thin and porous. Therefore, the pressure drop in the reactor could be reduced greatly.

3.1.2. EDS

Fig. 3 shows an EDS photograph of the photocatalyst after reaction. The EDS photograph indicates that the surface of the photocatalyst was covered with TiO_2 . Carbon was also found in EDS photograph after reaction. This also suggested that carbonate was produced and deposited on the photocatalyst after reaction. The deposition of carbonate on the catalyst surface may cause partial deactivation of the catalyst.

3.2. Multiple synergies of $\text{O}_3/\text{UV}/\text{TiO}_2$

When UV/TiO_2 was added into the post-plasma, there were various advanced oxidation processes (AOPs) including UV/TiO_2 , O_3/UV and O_3/TiO_2 . The synergistic effect of post-plasma and photocatalysis resulted in enhancing the toluene removal efficiency and lowering the O_3 concentration in the gas exhaust.

Fig. 4 shows the comparison of toluene oxidation by O_3 alone, UV alone, TiO_2 alone, O_3/UV , O_3/TiO_2 , TiO_2/UV , and $\text{O}_3/\text{TiO}_2/\text{UV}$

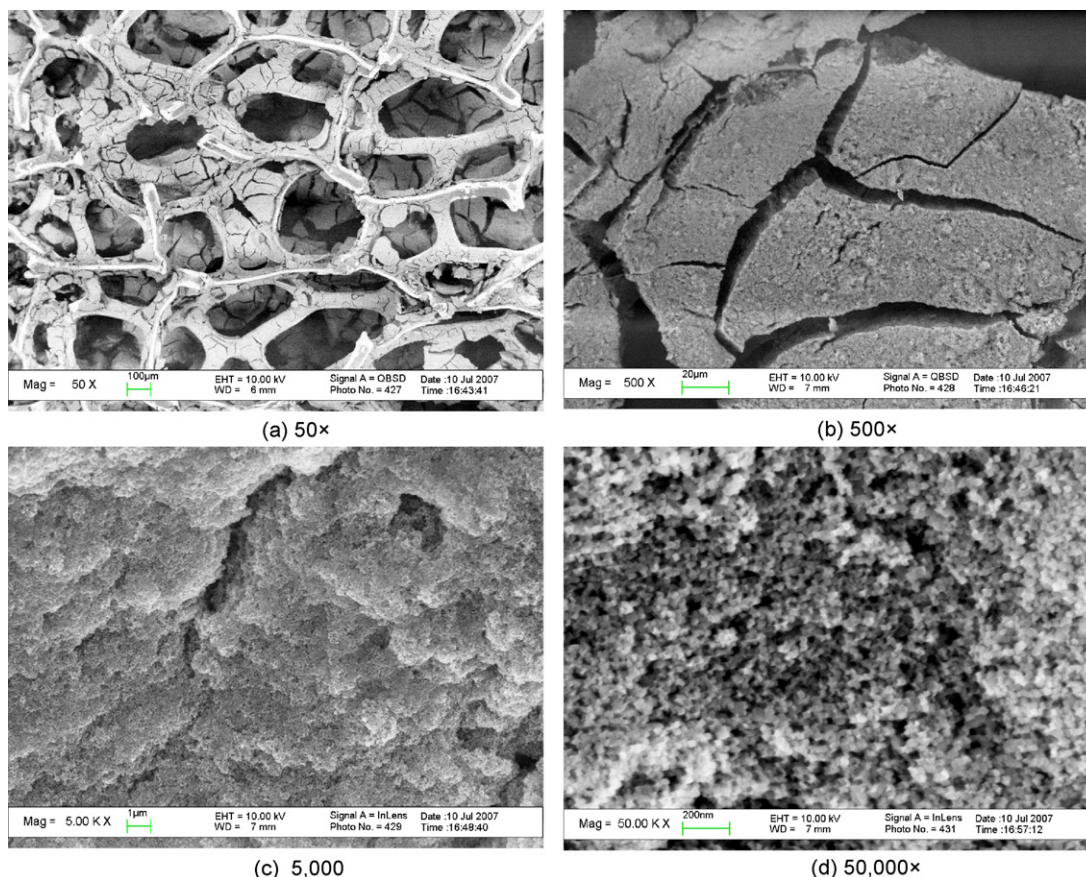


Fig. 2. SEM images of $\text{TiO}_2/\text{Al}_2\text{O}_3/\text{nickel}$ foam at various magnification times.

processes. O_3 alone, UV alone and TiO_2 alone could hardly decompose the toluene because of its refractory structure [19]. Only 25% toluene was decomposed by the TiO_2/UV process and the photocatalytic activity decreased after 30 min. This indicated that the photocatalyst was gradually deactivated. When ozone was added into TiO_2/UV , the conversion of toluene increased by nearly 60% compared with the TiO_2/UV process. No significant deactivation of photocatalyst was observed even 50 min later. So the addition of ozone into the TiO_2/UV process could not only dramatically raise the removal efficiency of toluene but also greatly improve the durability of the photocatalyst compared with photocatalysis process alone. Only about 40% toluene was decomposed by the O_3/UV process. However, when TiO_2 was added into the O_3/UV process, the toluene destruction increased by nearly 40% compared with O_3/UV process. So the introduction of TiO_2 into the O_3/UV process greatly increased

the toluene conversion. Fifty minutes later, the toluene conversion by O_3/TiO_2 process reached almost 80%. Whereas, when UV was introduced into the O_3/TiO_2 process, the destruction of toluene increased by less than 5% compared with the O_3/TiO_2 process. So the combination of UV with O_3/TiO_2 process could not result in a synergistic effect for toluene destruction. It is believed that the O_3/TiO_2 process contributed most to the toluene removal among the multiple oxidation processes in the $\text{O}_3/\text{TiO}_2/\text{UV}$ system.

Fig. 4 shows that there are synergistic effects when photocatalysis was combined for toluene oxidation in the post-plasma. The efficiency of toluene destruction in different processes followed the

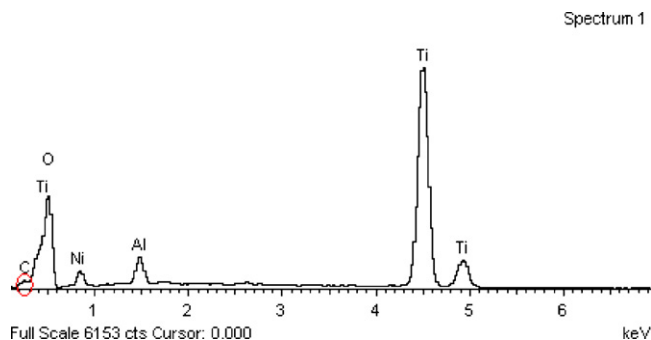


Fig. 3. EDS photograph of photocatalyst after reaction.

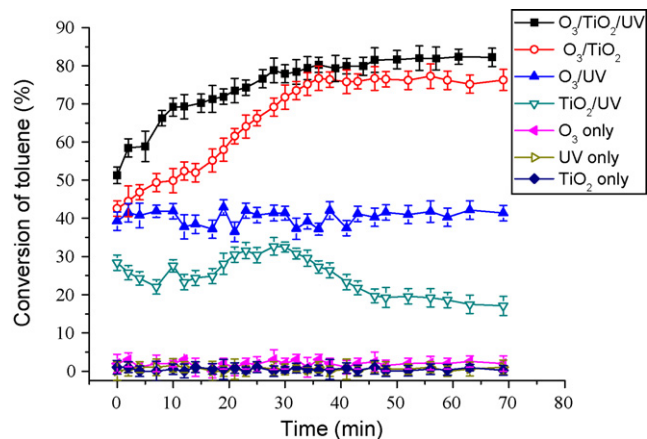


Fig. 4. Oxidation of toluene by the O_3 , UV, TiO_2 , O_3/UV , O_3/TiO_2 , TiO_2/UV and $\text{O}_3/\text{TiO}_2/\text{UV}$ processes.

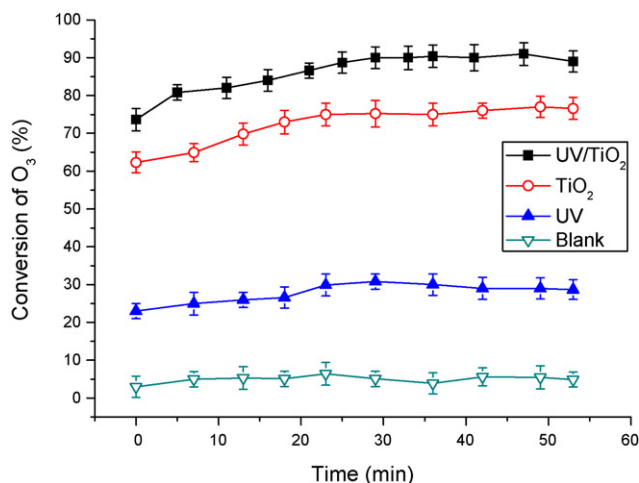


Fig. 5. Decomposition of ozone by UV, TiO₂ and UV/TiO₂ processes in the post-plasma.

Table 1

Comparison of conversion of toluene and O₃ by UV/TiO₂, TiO₂ and UV in the post-plasma.

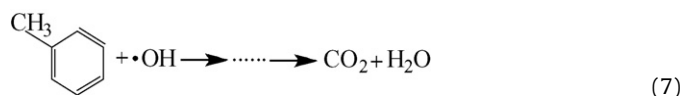
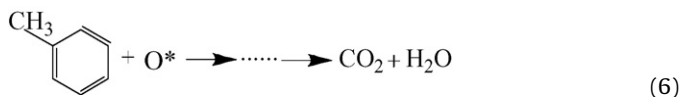
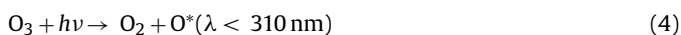
	Conversion of toluene (%)	Conversion of O ₃ (%)
UV/TiO ₂	82.2	90.6
TiO ₂	77.6	72.8
UV	40.5	27.3
Blank	1.2	2.7

order of O₃/UV/TiO₂ > O₃/TiO₂ > O₃/UV > UV/TiO₂ > O₃ alone ≈ UV alone ≈ UV alone. The O₃/UV/TiO₂ process exhibited the highest efficiency among the various processes.

Fig. 5 shows the ozone decomposition of UV, TiO₂ or UV/TiO₂ in the post-plasma. When there is no UV light or TiO₂ in the post-plasma, less than 5% ozone decomposed. When the UV light was introduced into the post-plasma, approximately 27% O₃ was removed. And when TiO₂ was added into the post-plasma, approximately 70% ozone was destroyed. However, nearly 90% O₃ was decomposed when UV/TiO₂ was introduced into the post-plasma. The conversion of ozone under different conditions was UV/TiO₂ > TiO₂ > UV > Blank. The UV/TiO₂ process had the highest O₃ removal efficiency. In the UV/TiO₂ process, ozone is mainly consumed through three reactions: decomposition by the catalyst, decomposition by UV light and acting as a hydroxyl radical (•OH) scavenger. The first reaction does not occur during the O₃/UV process and the second reaction does not occur during the O₃/TiO₂ process.

The conversion of toluene and ozone during steady-state operation by UV/TiO₂, TiO₂ and UV in the post-plasma is summarized in Table 1. The removal efficiency of toluene increased with the increase of ozone removal. This result further confirmed the importance of ozone decomposition in toluene destruction.

The plausible pathway of toluene and O₃ decomposition is [19,20]:



Where * denotes the surface site on the catalyst. The mechanism of toluene and O₃ decomposition is very complex. It includes not only heterogeneous catalysis but also O₃/UV gas-phase oxidation. Ozone can be effectively decomposed by the catalyst and UV light. O* was the intermediate highly active oxygen formed from catalytic and photolysis decomposition of ozone. The reaction of active oxygen with H₂O led to the formation of •OH whose oxidation capacity is second only to that of fluorine. There were two main kinds of active radicals in this system: •OH and active oxygen. The toluene reaction rate constant in gas-phase with hydroxyl radicals and active oxygen is 5.7×10^{-12} and $2.32 \times 10^{-13} \text{ cm}^3 \text{ molecule}^{-1} \text{ s}^{-1}$, respectively [2]. The reaction rates, combined with the high concentrations in the post-plasma, make both radicals important for toluene oxidation. However, it is still unclear how the toluene is decomposed by these active radicals and which active radical plays the key role. Further work is needed to look at the mechanism in the system.

The combination of photocatalysis with post-plasma has a synergistic effect for the removal of toluene. Ozone is abundantly formed in the NTP reactor and it is utilized as the oxidant precursor by photocatalysis in the post-plasma. The combination of photocatalysis in the post-plasma not only promoted the toluene oxidation and ozone decomposition, but also improved the durability of the photocatalyst. The O₃/TiO₂ process contributed most to toluene destruction among the multiple oxidation processes in the O₃/TiO₂/UV system.

3.3. Effect of energy density

The concentration of toluene at the inlet of the post-plasma reactor was stabilized at 50 ppm. The increase of energy density through DBD improved the ozone concentration at the inlet of the post-plasma reactor and enhanced the conversion of toluene, as shown in Fig. 6. As ozone is the precursor of active oxygen [21], the increase of ozone concentration enhanced toluene removal. The increase of energy density increased the ozone concentration at the inlet of post-plasma reactor, which led to the decrease of ozone conversion. The increase of energy density is commonly used in order to obtain higher removal efficiency of pollutants. However, it will result in more ozone formation and ozone pollution is aggravated.

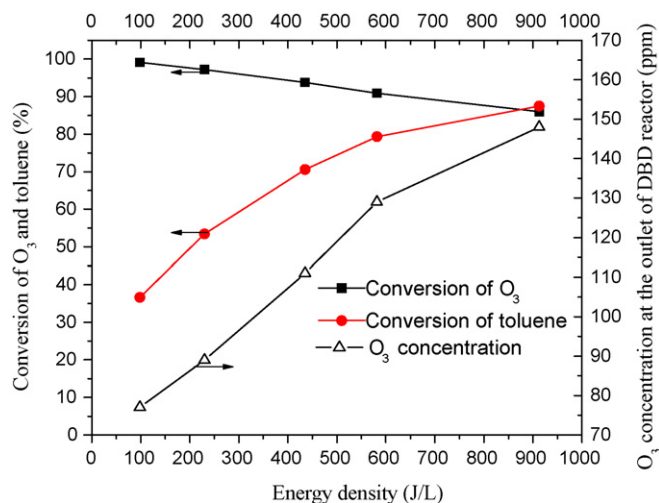


Fig. 6. Effect of energy density on ozone concentration at the outlet of DBD reactor, the conversion of toluene and O₃ in the post-plasma combined with UV/TiO₂ system.

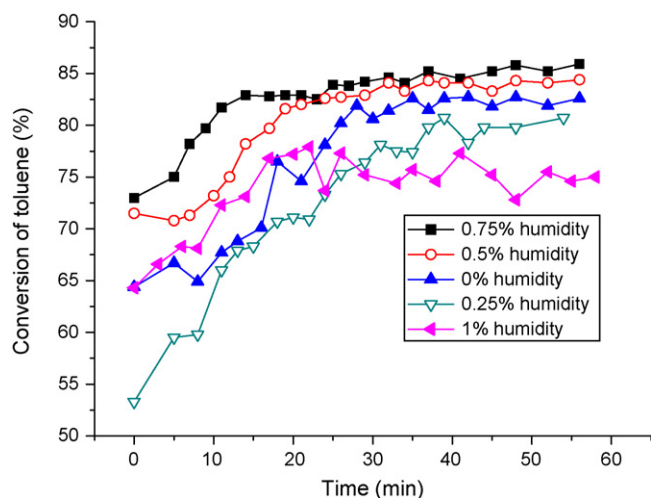


Fig. 7. Influence of humidity on the conversion of toluene in the post-plasma combined with UV/TiO₂ system.

In this system, the efficiency of toluene destruction increased exponentially with increasing energy density, whereas the conversion of ozone remained high. Although the increase of energy density promotes the conversion of toluene, it increases the energy consumption as well as the catalyst loading for ozone decomposition. Furthermore, excessive ozone did not make a significant contribution to toluene destruction due to the scavenging effect of ozone for $\cdot\text{OH}$ [22]. So the energy density is sufficient only for toluene destruction.

3.4. Effect of humidity

The influence of humidity on the conversion of toluene and ozone is illustrated in Figs. 7 and 8, respectively. As shown in Fig. 7, 0.75 and 0.5% humidity had the highest toluene conversion, followed by 0% humidity and 0.25% humidity. In total, 1% humidity performed the worst. The conversion of ozone increased with the increase of humidity from 0 to 0.75% in the air (Fig. 8). Water vapor is beneficial for the decomposition of ozone when the humidity is lower than 0.75%. However, a further increase of humidity will poi-

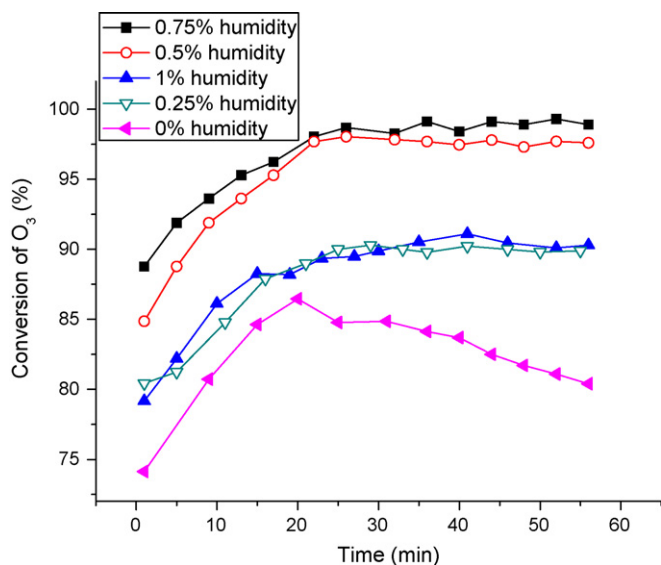


Fig. 8. Effect of humidity on the decomposition of ozone by the UV/TiO₂ process in the post-plasma.

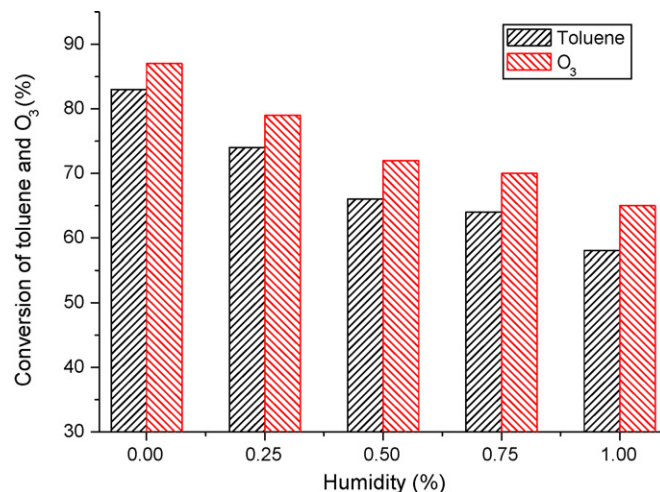


Fig. 9. Influence of humidity on the decomposition of toluene and ozone by TiO₂ in the post-plasma.

son the photocatalyst. As demonstrated in reactions (3)–(5), water promoted the consumption of ozone. The photocatalyst activity decreased after 25 min with 0% humidity. Meanwhile, no obvious deactivation of photocatalyst was observed even after 45 min in the humid air-flow. Therefore, nearly 0.5 and 0.75% humidity can effectively inhibit the deactivation of photocatalyst in a plasma-synergized photocatalysis system because water enhances the generation of $\cdot\text{OH}$, which is similar to the effect of water vapor in the photocatalysis system.

H₂O enhances the decomposition of toluene and ozone through the reactions (5) and (7), which was mentioned above. Water promoted the formation of $\cdot\text{OH}$, which was a very strong oxidant and assisted in the oxidation of toluene.

However, water vapor had a negative effect on toluene and ozone decomposition. As shown in Fig. 9, the conversion of toluene and ozone both decreased with increasing humidity in the O₃/TiO₂ process. Water vapor prevents the adsorption of toluene and ozone on the catalyst because of competitive adsorption of H₂O [23]. It also poisons the photocatalyst by occupying catalytic active sites [24]. The negative effect of water in O₃/UV/TiO₂ mainly resulted from the harm of water to the toluene removal by the O₃/TiO₂ process. As demonstrated above, the O₃/TiO₂ process is very important in synergistic oxidation of toluene in the multiple-synergy system. Too much water vapor may also hinder the transmission of UV light in the reactor, resulting in a decrease of toluene and ozone decomposition.

Water vapor therefore played dual roles in toluene and ozone decomposition. In this experiment, 0.75% humidity had the best conversion of toluene and ozone. However, there was not much difference in the conversion of toluene and ozone between 0.75% humidity and 0.5% humidity in steady-state operation. Thereby, the humidity should be kept at nearly 0.5% to maintain the activity and stability of catalysts.

3.5. Effect of UV sources

The effect of UV sources on toluene and ozone destruction in the O₃/UV/TiO₂ system was also investigated in this study (Figs. 10 and 11). Fig. 10 shows that the conversion of toluene under different UV sources followed the order: 254 nm/8 W > 254 nm/16 W > 365 nm/8 W > 254 nm/4 W. As shown in Fig. 11, the conversion of ozone follows the same order as that of toluene. The conversion of toluene also increased with the increase of ozone conversion in this experiment. The result indicates again

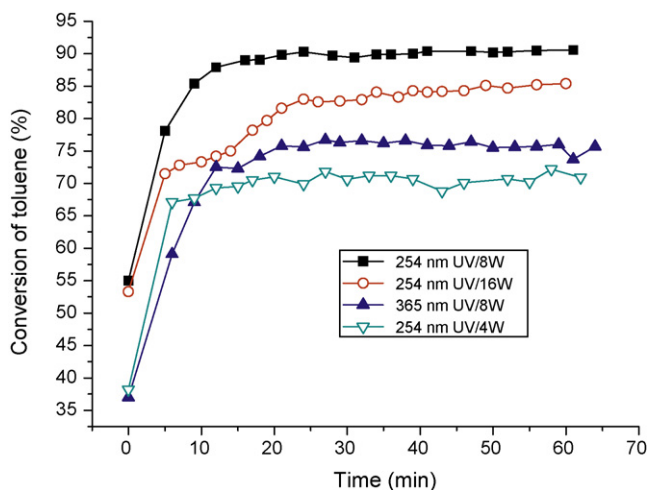


Fig. 10. Effect of UV sources on the toluene decomposition in the post-plasma system combined with UV/TiO₂.

that ozone decomposition plays a dominant role in toluene removal. The removal of toluene and O₃ is dependent on not only the intensity but also the wavelength of UV light. The UV light affects not only the TiO₂/UV process but also the O₃/UV process. The 254 nm/8 W UV lamp showed the best performance for toluene and ozone conversion among the four experimental UV sources. The conversion of toluene and ozone with the 254 nm/8 W UV light in steady-state operation reached nearly 92 and 97%, respectively. The efficiency of photocatalytic decomposition decreases on the contrary after the UV intensity increases to a certain extent. The increase of UV intensity can produce more photo-induced electron and hole; whereas, it is unfavorable to the transfer of photo-induced electron and hole. This possibly promotes the recombination rate of electron-hole pair and leads to the decrease of photocatalysis removal efficiency [25]. In addition, high UV intensity might inhibit the adsorption of toluene and ozone on the photocatalyst and result in the conversion decrease of ozone and toluene. It can be seen that the 254 nm UV light is more effective in the destruction of toluene (Fig. 10) and ozone (Fig. 11) than the 365 nm UV light. It is well known thought that ozone can be decomposed only by UV light with a wavelength between 200 and 308 nm, as described in reaction (4). Therefore, 254 nm rather than 365 nm UV light should be applied and there is an optimal intensity of UV light in this system.

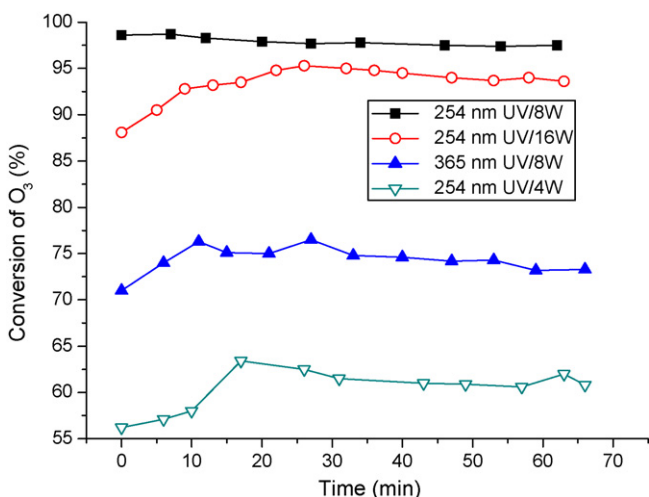


Fig. 11. Effect of UV sources on the ozone decomposition at outlet in the post-plasma system combined with UV/TiO₂.

4. Conclusion

The results of the present work clearly demonstrate that the combination of photocatalysis in the post-plasma not only dramatically enhanced the conversion of toluene, but also effectively inhibited O₃ emission in the gas exhaust. Almost 80% toluene and 90% ozone were destructed in the system. Water vapor played a dual role in toluene destruction and ozone removal. In total, 0.75% humidity and 254 nm/8 W UV light had the best conversion of toluene and ozone at the experimental conditions. The conversion of toluene increased with increasing ozone removal. O₃/TiO₂ process played a key role in the toluene decomposition in the O₃/UV/TiO₂ system.

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