Decomposition of benzene by non-thermal plasma processing: Photocatalyst and ozone effect

[1]*T. Zhu; [1]J. Li; [1]Y. Jin; [2]Y. Liang; [2]G. Ma

[1]*College of Environmental and Energy Engineering, Beijing University of Technology, Beijing 100022, China*

[2]*Department of Environmental Engineering, Xi'an University of Architecture and Technology, Xi'an 710055, China*

ABSTRACT: Plasma technology has some shortcomings, such as higher energy consumption and byproducts produced in the reaction process. However non-thermal plasma associated with catalyst can resolve these problems. Therefore this kind of technology was paied more and more attention to treat waste gas. A hybrid system comprising a non-thermal plasma reactor and nanometer titanium dioxide catalyst was used for benzene removal in the air. The paper described the synergistic effect of ozone and photocatalyst in the plasma reactor. Except of electric field strength, humidity and flow velocity, the synergistic behavior of ozone and photocatalyst was tested. The removal efficiency of benzene reaches nearly 99% when benzene concentration is 600 mg/m^3, and the removal efficiency of benzene also reaches above 90% when benzene concentration is 1500 mg/m^3. The plasma reactor packed with photocatalyst shows a better selectivity of carbon dioxide than that without photocatalyst. The final products is mostly carbon dioxide, water and a small quantity of carbon monoxide.
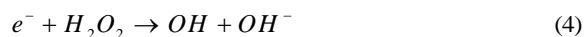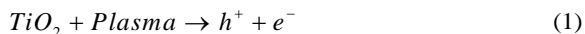
Key words: *Synergistic effect, removal efficiency, non-thermal plasma, selectivity of carbon dioxide*

INTRODUCTION

Volatile organic compounds (VOCs) are triggering serious environmental problems such as stratospheric ozone depletion and photochemical smog. Although benzene is an important chemical feedstock and additive, it should be removed from exhaust gases emitted from various moving and stationary sources due to its carcinogenicities and respiratory disorders(Noel, 2000). The traditional methods of VOCs removal such as absorption, adsorption, and incineration and so on, which were referred to the new environmental condition had many technical and economical disadvantages (Ma, 2003). In these years, some new technologies, such as biologic process, photocatalysis process, and plasma technology, were paid more and more attention (Ogata, 1999; Futamura *et al.,* 2004; Li *et al.,* 1998; Magureanu, 2007; Urashima *et al.*, 2000; Zhu *et al.,* 2007a). In particular, non-thermal plasma has attracted much attention as an method for VOCs control for two decades due to its unique properties such as quick response at ambient temperature, achievement of high electron energies within short residence times, system compactness, and easy operations (Muhamad, 2000; Li *et al.*, 2006). In

✉ *Corresponding Author Email: bamboozt@sohu.com*
Tel.: +86 10 673 920 80; Fax: +86 10 673 920 80

order to improve the energy efficiency of the VOCs decomposition process by the plasma, the cooperation with catalyst has been tested by some researchers (Einaga *et al.*, 2001; Guo *et al.,* 2006; Jim, 2008; Krawczyk, 2001; Magureanu *et al.,* 2007a and b; Ogata *et al.,* 2003; Wallis *et al.,* 2007; Li *et al.,* 2007; Zhu *et al.,* 2007b). These studies showed that the combination of discharge plasma with catalyst is a very effective method in VOCs removal. (Wallis, 2007; Durme *et al.,* 2007; Magureanu, 2005; Delagrange *et al.,* 2007; Guo *et al.*, 2007). In this paper, the combination of non-thermal plasma was tried with nanometer titanium dioxide (TiO$_2$) photocatalyst for benzene removal, in order to further reduced the energy consumption and harmful byproducts in plasma process. Discharge plasma as a driving force of photocatalyst furnished a mess of UV light. Hole-electron pairs are produced by supplying energy larger than the band-gap energy of TiO$_2$ (3.2 eV for Anatase type). High-energy particles, such as electrons, excited molecules, and radicals may transfer their energy to TiO$_2$ by bombardment when TiO$_2$ is placed in a nonthermal plasma (Thevenet *et al.,* 2007; Yoichi *et al.,* 2008). Various chemical reactions are induced on the excited TiO$_2$ surface through the following reactions:

$$TiO_2 + Plasma \rightarrow h^+ + e^- \tag{1}$$

$$h^+ + H_2O \rightarrow OH + H^+ \tag{2}$$

$$e^- + O_2 \rightarrow O_2^- \tag{3}$$

$$e^- + H_2O_2 \rightarrow OH + OH^- \tag{4}$$

$$O_2^- + H^+ \rightarrow HO_2 \tag{5}$$

$$Radicals + Reactant \longrightarrow Products \tag{6}$$

This paper illuminates the experimental results of the synergistic effect of non-thermal plasma generated by dielectric barrier discharge (DBD) and TiO$_2$ photocatalyst on benzene decomposition.

MATERIALS AND METHODS

The reaction system was a tube-wire packed-bed reaction system at atmospheric pressure. The schematic diagram of the testing apparatus is shown in Fig. 1. To clarify the destruction process, dry air (78 % N$_2$, 21 % O$_2$) was used as a balance gas for benzene. Air supplied from the laboratory was divided into two air flows. Each flow rate was controlled with a flow meter. One air flow was introduced into 3 (VOCs bottle) which contained liquid VOCs. The air with almost saturated vapor of VOCs was mixed with the other air flow into 4 (blender) and diluted to the prescribed concentration of VOCs. A packed reactor with coaxial electrodes was designed as shown in Figs. 1 to 7. The reactor was made of 50 mm diameter ceramic tube, with an effective reactor length of 500 mm. One electrode (1.5 mm) was inside and the other electrode was wrapped around the outside and grounded (80 circles). For increasing discharge, the packed materials (Rasching ring) were packed between the two electrodes. The packed materials (5 mm i.d., 1 mm wall thick, 10 mm length) which were electrician ceramic rings, were divided into two groups, coated with photocatalyst or without photocatalyst. The characteristic of packed materials includes volume density is 217 g/cm^3, hole rate is 12.7 % and bibulous is 5.9 %. Nano-TiO$_2$ thin films were prepared by the Sol-Gel method (Li *et al.,* 1996; Liang *et al.,* 2006) in the experiment.

Precursor solution

1 mol Tetrabutyl Titanate (Precursor Substance) + 12 mol Ethanol (Solvent) + 1.2 mol Acetyiacetone (Chelating Reagent)

Droplet solution

6 mol Ethanol (Solvent) + 1 mol HNO$_3$ (Catalyzer) + 2.5 mol Distilled Water

Droplet solution was dropped slowly into original solution at 35 and the whole solution was mixed well together. The steady Sol would be obtained and then the Sol should be deposited at least 24 h. The packed materials must be washed by ultrasonic before they were immerged into the Sol. Later, they were pulled out from the Sol at the speed of 1.5 mm/s to get the nanometer TiO$_2$ film. The packed materials with nanometer film would be dried at 80 °C for one hour before they were put into muffle to calcine at 450 °C for 2 h. At last, the film gradually refrigerated to ambient temperature.
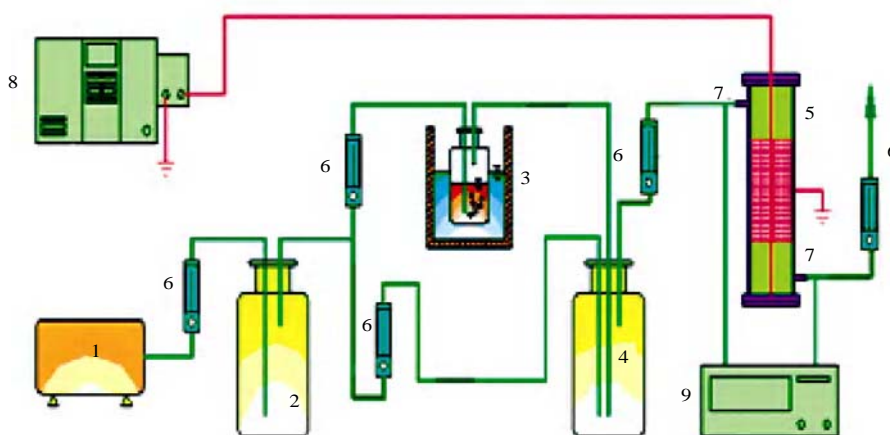

Fig. 1: The flowchart od benzene removal
1. pump 2. buffer 3.VOCs bottle 4.blender 5. reactor
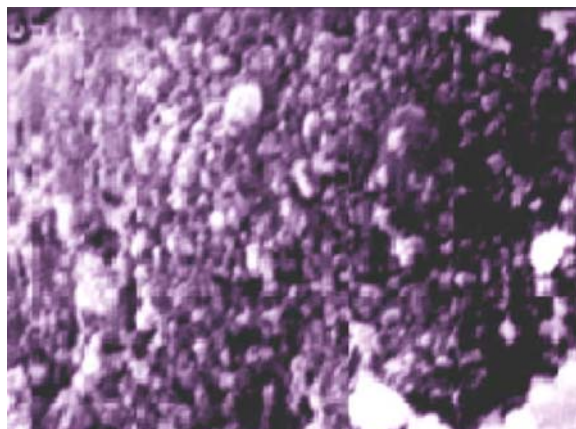6. flowmeter 7. sampling port 8. high voltage 9.GC

Fig. 2: SEM micrograph of the sample

The nanometer TiO_2 thin film was inspected and analyzed by Scan Electric Mirror (SEM, Made in Japan, S-2700). Fig. 2 shows SEM photograph for surface situation of the samples. The average particulate diameters of TiO_2 were less than 100 nm by SEM. Therefore TiO_2 samples made by self were of nanometer levels. The conjugate chemical bonds of benzene (C_6H_6) locate the same plane. Due to benzene possessing syntony stability, this kind of chemical bonds is more fastness and steady than common chemical bonds. So benzene was chose as a kind of VOCs to decompose in the experiment because of its stabilization of chemical structure. The plasma reactor employed an AC power supply of 60 Hz. The AC voltage was applied to the reactor in the radial direction, and the voltage extension changed from 10 kV to 30 kV. Chromatographic analysis and calculation were carried out by gas chromatography (manufactured by American Thermo Finnigan Co., TRACE-GC ULTRA) with a flame ionization detector (FID). Separately, another GC (SC-1001) equipped with an FID detector and an methane converter was used to analyze concentration of CO_2 and CO. Reaction gas samples were taken by a syringe from the sampling ports of the reactor. The concentration of ozone (O_3) was measured by iodine-titration and the byproducts were identified by GC-MS with a 30-m-long wide-bore capillary column (DB-1). The experimental condition was in atmospheric pressure (760 mmHg) and temperature (20 °C).

RESULTS AND DISCUSSION

Fig. 3 shows the relationship between removal efficiency of benzene and electrostatic field strength in the plasma reactor with or without packed materials.
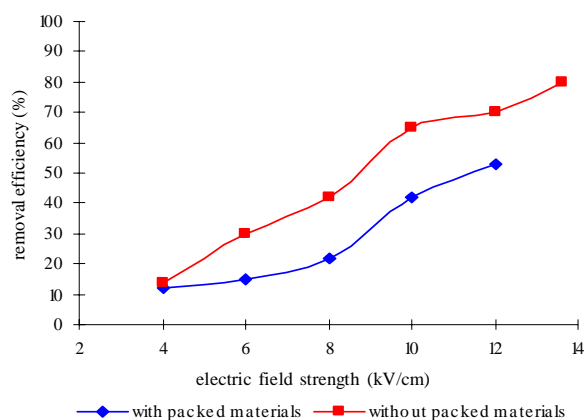

Fig. 3: The effect of removal efficiency with or without packed materials
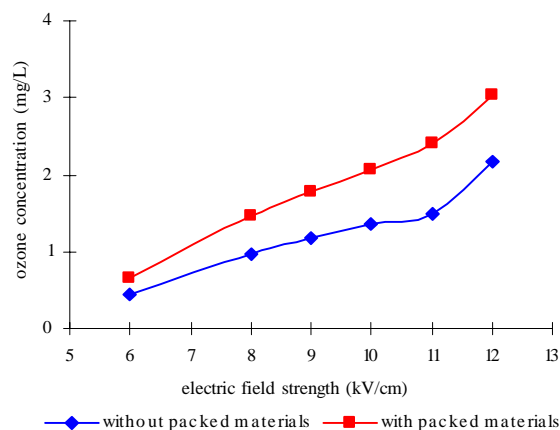

Fig. 4: The effect of ozone concentration with or without packed materials

Fig. 4 shows the relationship between ozone concentration and electrostatic field strength in the plasma reactor with or without packed materials. With increasing electrostatic field strength, the removal efficiency of benzene increases. When initial concentration of benzene is 1300 mg/m³, the average electrostatic field strength is 13.6 kV/cm and gas flux is 100 L/h., the removal efficiency of benzene arrives at 80 % in the reactor with packed materials as shown in Fig. 3. In the reactor, the space occupied by contamination air is always full of high energy electrons (Yang, 1983). When effective collisions between high energy electrons and benzene molecules take place in the reactor, electron energy will destruct molecular structure of benzene and benzene molecules will be

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converted into inorganic little molecules like carbon dioxide (CO_2), carbon monoxide (CO) and water (H_2O). Thus, removal efficiency of benzene are proportional to the electrons. In Fig. 3, when packed materials placed in the plasma reactor, with electrostatic field strength increasing, more and more high energy electrons are produced due to mediums polarization of packed materials. So packed materials in the reactor increases the removal efficiency of benzene. Fig. 4 shows when initial concentration of benzene is 1300 mg/m^3, the average electrostatic field strength is 12 kV/cm and gas flux is 100 L/h., ozone concentration is about 3.04 mg/L with packed materials and ozone concentration is about 2.16 mg/L without packed materials. Ozone concentration with packed materials heightens 1 mg/L than that without packed materials in the plasma reactor. It is obvious that packed materials in the reactor is helpful of increasing ozone concentration. The reason is high energy electrons and radicals are generated in the early discharge phase. They possess high energy compared to the dissociation energy of O_2 so that a series of reaction takes place in the plasma. The oxygen dissociation is the most important radical formation reaction.

$$e + O_2 \rightarrow e + O + O \qquad (7)$$
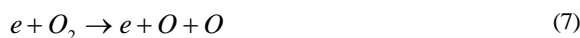
Electronically excited atomic oxygen (O(^{1}D)) are very short-lived radicals, whereas ground state atomic oxygen (O) and hydroxyl (OH) have a longer lifetime. O(^{1}D) reacts with H_2O resulting in formation of OH radicals. O$^-$ and OH$^-$ radicals are removed by formation of O_3. In Figs. 3 and 4, the test results also indicate ozone is helpful for benzene removal, at least, ozone acts as the oxidant precursor. With ozone concentration increasing, the removal efficiency of benzene increases. Because ozone as a kind of oxidative species produced by the initial oxidation just like OH radical, has an effect on further reaction of benzene. The humidities of contaminated air in the reactor have influence on ozone concentration as shown in Fig. 5a. Ozone concentration without vapor is higher 35 % than that with relative humidity 67 %, and ozone concentration decreases with humidity increasing. Because H_2O molecule have electronegative, it will consume the electrons in the plasma. At the same time, H_2O will react with O (^{1}D) which is the origin of formation of O_3.

$$H_2O + O(^1D) \rightarrow 2OH \qquad (8)$$
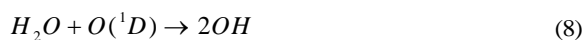
So humidity counteracts the formation of ozone.

In Fig. 5b, the findings show the removal efficiency reduces with humidity increasing. Probably, the active sites for benzene removal are reduced by water vapor occurring competitive adsorption on the surface of nano-TiO_2. Thus, humidity affects the benzene removal in three ways: deactivation of high energy electrons, inhibition of ozone formation, and suppression of the catalyst activity of nano-TiO_2 for benzene oxidation with ozone in the plasma reactor. Fig. 6 shows the relationship between ozone concentration and gas flux with catalyst or without catalyst in the plasma reactor under three initial concentration of benzene. When gas passes the reactor, and electrostatic field strength is
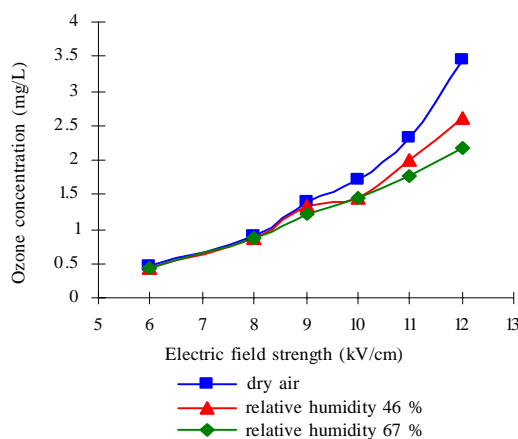
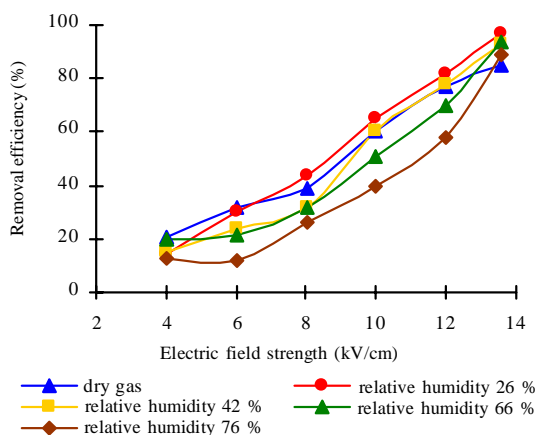Fig. 5a: The effect of humidity on ozone concentration without photocatalyst

Fig. 5b: The effect of humidity on removal efficiency with photocatalyst
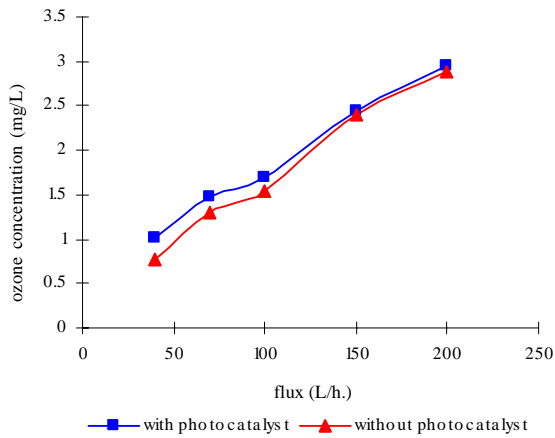
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Fig. 6a: Relationship between ozone concentration and flux with or without catalyst when benzene concentration is 0 mg/m³
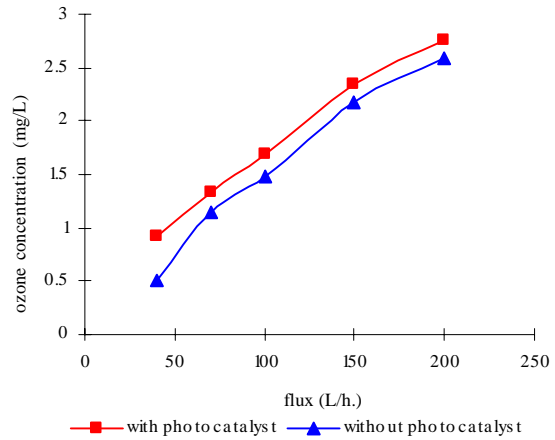

Fig. 6b: Relationship between ozone concentration and flux with or without catalyst when benzene concentration is 700 mg/m³
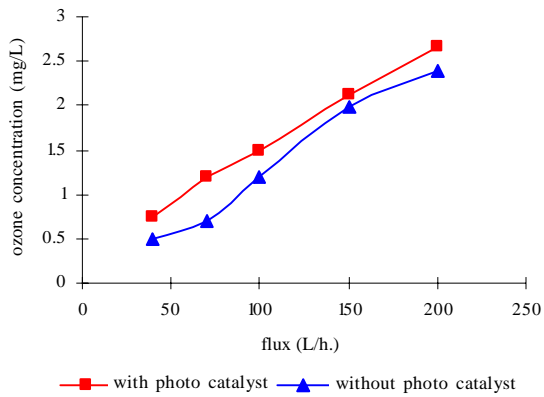

Fig. 6c: Relationship between ozone concentration and flux with or without catalyst when benzene concentration is 2000 mg/m³
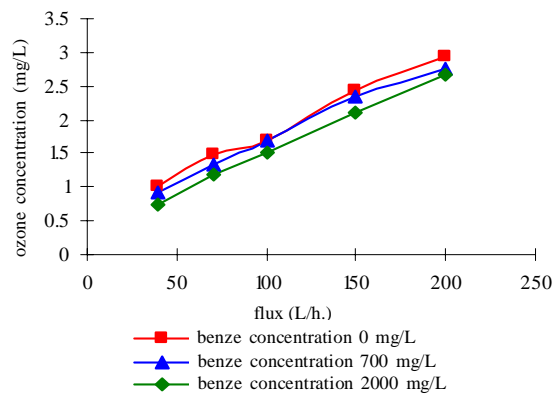

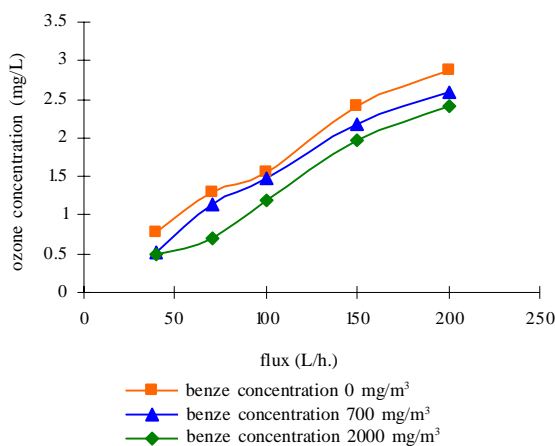Fig. 6d: Relationship between ozone concentration and flux with catalyst

Fig. 6e: Relationship between ozone concentration and flux without catalyst

10 kV/cm, ozone concentration increases with gas flux increasing as shown in Figs. 6a, b and c, regardless of with or without photocatalyst. Fig. 6d and 6e show benzene concentration reduces with initial concentration of benzene increasing, regardless of with or without photocatalyst.

The relationship between benzene degradation and electrostatic field strength with or without photocatalyst is shown in Fig. 7 where benzene initial concentrations changes from 600 mg/m³ to 1500 mg/m³. When initial concentration is 600 mg/m³, the average electrostatic field strength is 10 kV/cm, and gas flow rate is 14 mm/s, the removal efficiency of benzene attains 98 % in the reactor with photocatalyst, but the removal efficiency of benzene attains 78 % in the reactor without photocatalyst as shown in Fig. 7a. When initial

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concentration is 1500 mg/m³, the average electrostatic field strength is 12 kV/cm and gas flow rate is 14 mm/s, the removal efficiency is higher 19 % with photocatalyst than without photocatalyst in the plasma reactor as shown in Fig. 7b. The results indicate photocatalyst enhanced the benzene removal efficiency obviously with ozone. When both photocatalyst and ozone coexist, there will be an improved removal efficiency of benzene in the plasma reactor. TiO_2 is a photocatalyst material of 3.2 eV band gap. Which if it absorbs bigger energy than band gap, it makes photo-excited electron-hole pairs that could oxidize benzene. At same time, the surface hydroxyl groups are oxidized to form composition of benzene in the photocatalytic reactions. Therefore it was advantageous to use photocatalyst in plasma system to control of oxidation step of benzene.

The influence of the catalyst on ozone formation is presented in Figs. 6a, b and c. It shows the catalyst could reduce the ozone formation to a certain extent. This is because ozone as the main long-living radical can capture free electrons which are produced by photocatalysis and produce OH radical. It avoids not only hole-electron pairs compounding but also increases photons efficiency. Further more, OH radical is a kind of good oxidant and it can transform organism into mineral. (Zhang *et al.,* 2003; Maria *et al.,* 2002).

$$O_3 + e^- \rightarrow O_3^- \bullet \qquad (9)$$

$$H^+ + O_3^- \bullet \rightarrow HO_3 \bullet \qquad (10)$$

$$HO_3 \bullet \rightarrow O_2 + \bullet OH \qquad (11)$$

$$O_3 + h\nu \rightarrow \bullet O + O_2 \qquad (12)$$

$$\bullet O + H_2O \rightarrow 2 \bullet OH \qquad (13)$$

From 9 to 13, it could be arrived at conclusions. Ozone increases photons efficiency of photocatalusis that is helpful of benzene removal. On the other hand, photocatalyst promotes ozone to separate into OH radical and reduces ozone output.Complete oxidation of benzene to CO_2 is the final goal of the experiment, and the catalytic effect on the product distribution had been investigated. Photo-oxidation activity should be expressed as selectivity to CO_2 because other byproducts except CO_2 were emitted in plasma process. Here, the selectivity of CO_2 was calculated as follows:

$$CO_2 \ selectivity(\%) = \frac{[CO]_2}{6([Benzene]_0 - [Benzene])} \times 100 \qquad (14)$$

where the $[Benzene]_0$ and $[Benzene]$ indicate inlet and outlet concentrations of benzene, respectively.

Though GC-MS, the main products in the plasma reactor were CO_2, H_2O, and a small quantity of CO. Ozone was the only byproduct, and no other byproducts could be detected in the tail gas. In addition, certain brown-yellow products that were observed in the plasma reactor regardless of with or without catalyst appeared. The composition of the brown-yellow products was indistinct, and maybe it was aromatic polymer detected by GC-MS.

Figs. 7a and 7b show, the removal efficiency of benzene reaches nearly 99 % when benzene concentration is 600
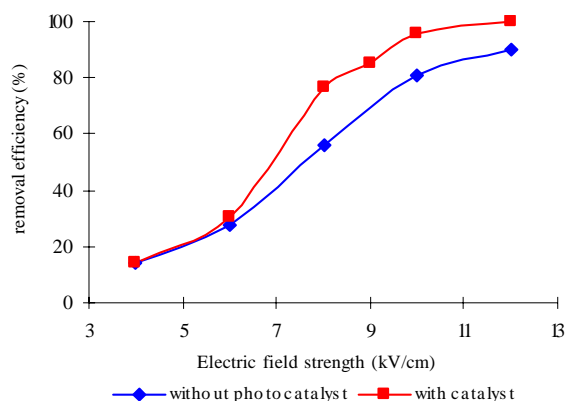

Fig. 7a: Relationship between benzene removal and electric field strength with or without catalyst when benzene concentration is 600 mg/m³
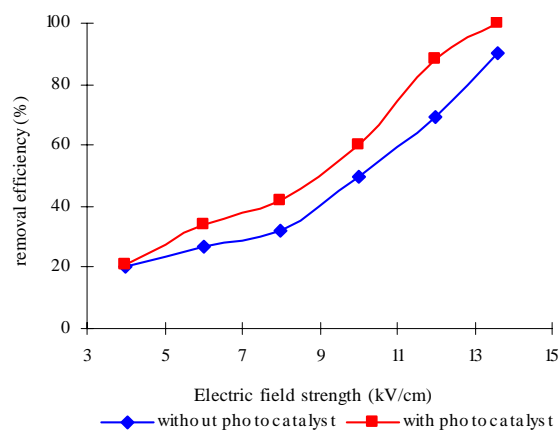

Fig. 7b: Relationship between benzene removal and electric field strength with or without catalyst when benzene concentration is 1500 mg/m³

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mg/m^3, and the removal efficiency of benzene also reaches above 90 % when benzene concentration is 1500 mg/m^3. TiO_2 can generate higher concentrations of different types of active oxygen species in non-thermal plasma, and the hybrid system has an effective utilization of active oxygen species in benzene removal based on the data presented in Fig. 7.

The minimum of CO/CO_2 is 0.286 and CO/CO_2 decreases with electrostatic field strength increasing as shown in Figs. 8a and 8b. There are no products except CO_2 and H_2O at 11 kV/cm and 12 kV/cm. CO/CO_2 of byproducts is lower 8.2 % with catalyst than that without catalyst. These findings show the plasma reactor packed with materials with catalyst has a better selectivity of CO_2 than that without catalyst.

From Fig. 8c, it is found the selectivity of CO_2 ranges from 65 % to 69 % in the plasma reactor without catalyst, while the selectivity of CO_2 ranges from 68 % to 73 % in the reactor with catalyst. The selectivity of CO_2 is independent of electrostatic field strength. The selectivity of CO_2 is enhanced due to the benzene oxidation near or on the photocatalyst surface. For that, it could be thought that intermediates and secondary products are more oxidized to CO_2 on

photocatalyst surface. With benzene concentration increasing, the total output of CO_2 increases.

CONCLUSION

The laboratory scale plasma reactor was used for benzene removal in an air stream and the following conclusions are obtained. With ozone concentration increasing, the removal efficiency of benzene increases. Ozone concentration with packed materials is heigher than that without packed materials in the plasma reactor. Water vapor reduces ozone concentration, and occurring competitive adsorption on the surface of TiO_2 Ozone concentration increases with gas flux increasing, and the removal efficiency of benzene reduces with initial concentration of benzene increasing. When both photocatalyst and ozone coexist, there will be an improved removal efficiency of benzene in the plasma reactor. Effective utilization of active oxygen species is essential in VOCs removal, and TiO_2 can generate higher concentrations of different types of active oxygen species in non-thermal plasma. It is facile and promising to simultaneously hybridize plasma with TiO_2 based on the data presented in this paper. Detected by GC-MS, the main
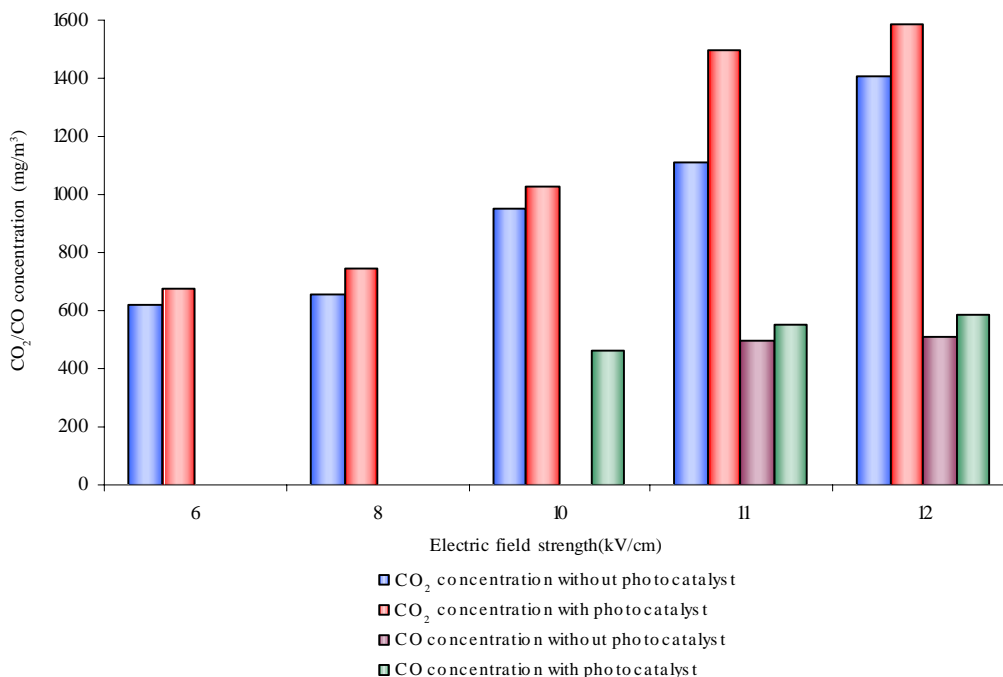

Fig. 8a: Results of byproducts detection when benzene concentration is 750 mg/m^3 with or without catalyst
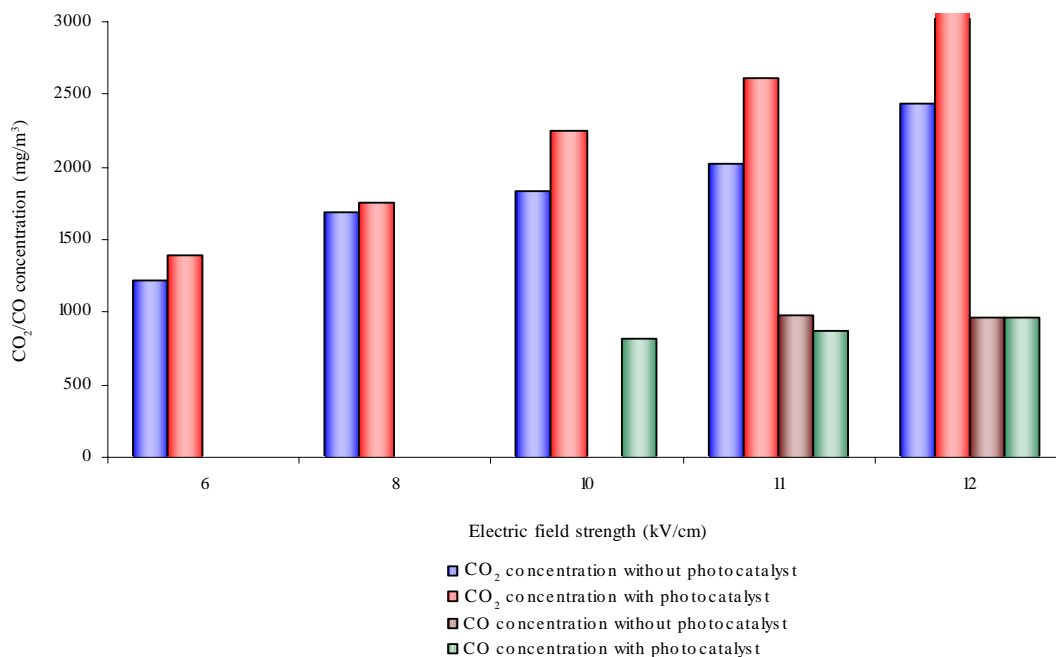
Decomposition of benzene

Fig. 8b: Results of byproducts detection when benzene concentration is 1500 mg/m^3 with or without catalyst
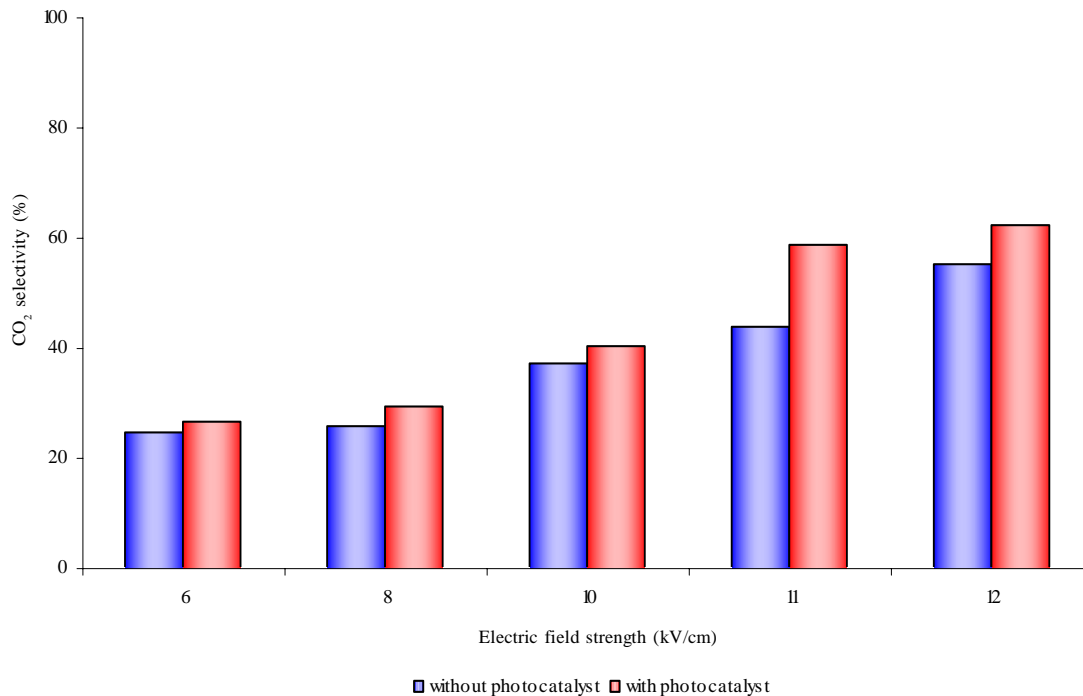

Fig. 8c: CO_2 selectivity when benzene concentration is 750 mg/m^3 with or without catalyst

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products in the plasma reactor are CO_2, H_2O and a small quantity of CO. There are no other products detected in the tail gas. The plasma reactor packed with catalyst shows a better selectivity of CO_2 than that without catalyst. The selectivity of CO_2 is independent of electrostatic field strength. The selectivity of CO_2 is enhanced due to the benzene oxidation near or on the photocatalyst surface. With benzene concentration increasing, the total output of CO_2 increases. The hybrid system comprising a non-thermal plasma reactor and nanometer TiO_2 catalyst, not only in the gas phase but on the catalyst surface, resulted in the higher energy efficiency and enhanced performance for the oxidative removal of benzene with lower medium reactivities and higher CO_2 selectivity in non-thermal plasma.

ACKNOWLEDGMENTS

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AUTHOR (S) BIOSKETCHES

Zhu, T., M. Sc, is a Ph.D. research student in air pollution control in College of Environmental and Energy Engineering, Beijing University of Technology, Beijing, China. Email: *bamboozt@sohu.com*

Li, J., Ph.D., Professor in environmental engineering in College of Environmental and Energy Engineering, Beijing University of Technology, Beijing, China. Email: *ljian@bjut.edu.cn*

Jin, Y. Q., Professor in environmental engineering in College of Environmental and Energy Engineering, Beijing University of Technology, Beijing, China. Email: *jinyq@bjut.edu.cn*

Liang, Y. H., Ph.D., Professor, Department of Environmental Engineering, Xi'an University of Architecture and Technology, Xi'an, China. Email: *lyah@xauat.edu.cn*

Ma, G. D., M. Sc, Professor, in Environmental Engineering, Department of Environmental Engineering, Xi'an University of Architecture and Technology, Xi'an, China. Email: *magd@xauat.edu.cn*