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Synergistic effects of non-thermal plasma-assisted catalyst and ultrasound on toluene removal

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Abstract
A wire-mesh catalyst coated by La₀.₈Sr₀.₂MnO₃ was combined with a dielectric barrier discharge (DBD) reactor for toluene removal at atmospheric pressure. It was found that toluene removal efficiency and carbon dioxide selectivity were enhanced in the catalytic packed-bed reactor. In addition, ozone and nitrogen monoxide from the gas effluent byproducts decreased. This is the first time that ultrasound combined with plasma has been used for toluene removal. A synergistic effect on toluene removal was observed in the plasma-assisted ultrasound system. At the same time, the system increased toluene conversion and reduced ozone emission.

Key words: La₀.₈Sr₀.₂MnO₃ catalyst, dielectric barrier discharge, plasma-assisted catalyst, ultrasound, toluene
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Introduction
In gaseous pollution control, volatile organic compounds (VOCs) control has received increasing attention due to their prolific emission from paint production factories, chemical plants, and printing industries. In addition, VOCs emitted into the atmosphere have long term effects on human health.

The conventional means for VOCs abatement include liquid absorption, activated carbon adsorption, and thermal and thermo-catalytic oxidation (Holzer et al., 2002). However, their drawbacks restrict their applications. For example, adsorption generates byproducts that require further treatment and oxidation is expensive and energy consumptive. Among the new treatment methods, non-thermal plasma (NTP) technology shows good performance on the decomposition of VOCs (Okubo et al., 2000; Rudolph et al., 2002; Roland et al., 2002; Inoue et al., 2011). To improve the performance of the plasma technique, it is frequently combined with catalysts (Demidiouk et al., 2003) such as MnO₂ (Zhu et al., 2009).

In this article, a new kind of wire-mesh catalyst coated by La₀.₈Sr₀.₂MnO₃ was used as a catalyst, and placed in the discharge zone. This kind of catalyst has many application advantages, such as low pressure drop, high thermal conductivity, and good mechanical properties. It has been reported that perovskite-type catalysts with a general formula ABO₃ (A represents a La ion and B a transition mental ion) can catalyze the oxidation of CO, and the decomposition of NOₓ (Niu et al., 2007) and VOCs (Blasin-Aubé et al., 2003). The synergistic effect of plasma and La₀.₈Sr₀.₂MnO₃ catalyst were investigated for the first time for toluene removal, CO₂ selectivity, and the byproducts of O₃ and NO. Additionally, to make use of the large amounts of ozone generated in the dielectric barrier discharge (DBD) reactor without catalyst and improve toluene removal efficiency, an ultrasonic system was introduced into the experiment to a form ultrasound (US)/ozone (O₃) combination system. To the best of our knowledge, this was the first time that NTP has been combined with sonolysis to make full use of the byproduct (O₃). Results showed that ultrasound improved the removal efficiency of ozone and also decreased ozone emissions at the same time.

1 Materials and experiments
The experimental system included a gas supplying and controlling system, a high AC power supply system (Vareg, Switzerland), a DBD reactor, electric and gaseous analytical systems and an ultrasonic irradiation system (Fig. 1). Dry air supplied by a compressor was divided into two air flows, one part was allowed to pass through the pure toluene liquid kept in a water bath (25 ± 1°C), the other part was mixed with the former stream in a blender. The mass flow controllers (MFC) maintained the toluene concentration at 900 mg/m³ and kept the flow rate 1 L/min. The wire-cylinder reactor was made of quartz glass with an inner diameter of 18 mm and wall thickness of 1.5 mm wrapped by sheet copper 160 mm long and 0.2 mm thick as a ground electrode. A stainless steel rod with a...
A layer of Al$_2$O$_3$/Al was first formed on the wire mesh surface as a carrier membrane by electrophoretic deposition and thermal treatment. Then coprecipitation prepared La$_{0.8}$Sr$_{0.2}$MnO$_3$ was coated onto the Al$_2$O$_3$/Al-wire mesh. A process of evaporation was followed after impregnation which was kept for 1 hr at normal atmospheric temperature after calcination at 500°C for 2 hr.

A gas chromatograph (Clarus 400, PerkinElmer, USA) equipped with a hydrogen flame ionization detector (FID) and 30 m long SE-30 capillary column was used to analyze the concentration of toluene at the inlet and outlet. The ozone concentration was measured with an ozone monitor (DCS-1, Lida Instrument, China). The CO and CO$_2$ produced in the reactor were monitored by a CO analyzer (TX2000, Oldham, France) and the CO$_2$ detector (GXH-3010E, Huayun, China), respectively. The concentration of NO was detected by an NO analyzer (PGM-1140, Rae, USA).

Toluene removal efficiency ($\eta$) was calculated as follows:

$$\eta = \frac{C_{\text{toluene, in}} - C_{\text{toluene, out}}}{C_{\text{toluene, in}}} \times 100\%$$

where, $C_{\text{toluene, in}}$ and $C_{\text{toluene, out}}$ are the initial and final toluene concentrations, respectively.

Because volume changes due to chemical reactions are negligible, the selectivity of CO and CO$_2$ may be defined as follows:

$$S_{\text{CO}} = \frac{C_{\text{CO}}}{7(C_{\text{toluene, in}} - C_{\text{toluene, out}})}$$

$$S_{\text{CO}_2} = \frac{C_{\text{CO}_2}}{7(C_{\text{toluene, in}} - C_{\text{toluene, out}})}$$

Selectivity of CO$_2$:

$$S_{\text{CO}_2} = S_{\text{CO}} + S_{\text{CO}_2}$$

where, $C_{\text{CO}}$ and $C_{\text{CO}_2}$ are the outlet concentration of CO and CO$_2$, respectively.

2 Results and discussion

2.1 Influence of La$_{0.8}$Sr$_{0.2}$MnO$_3$ catalyst on reaction

The catalyst was a critical design feature within the DBD reactor due to the high discharge temperature in the discharge zone. In this work, because of the good performance at high temperature, La$_{0.8}$Sr$_{0.2}$MnO$_3$ was chosen as the catalyst to study toluene removal efficiency, the selectivity to CO, and CO$_2$ and byproducts.

2.2 Toluene removal efficiency

Figure 2 presents the toluene removal efficiency as a function of applied voltage with or without La$_{0.8}$Sr$_{0.2}$MnO$_3$. Results showed that whether the catalyst was applied or not, toluene removal efficiency increased when the applied voltage increased from 11.5 to 21.5 kV. However, when the catalyst was introduced into the reactor, removal efficiency was significantly enhanced in the whole range of applied voltage. For example, toluene conversion was 89.4% at 21.5 kV with catalyst, which was greater than the 70.9% without catalyst. Similar results were observed earlier when the MnO$_x$ catalyst was introduced into the reactor (Guo et al., 2010).

The interaction of adsorbed toluene with adsorbed oxygen and lattice oxygen determines the oxidation of toluene.
Fig. 2 Effect of applied voltage on the toluene removal efficiency. Toluene concentration 900 mg/m³; gas flow rate 1 L/min; AC frequency 250 Hz.

(Mergler et al., 1997) and adsorbed oxygen species (such as O\(^{2-}\), O\(^{2-}\), and O\(^{2-}\)) play the dominant role in the total oxidation of toluene. It was found that catalytic activity of La\(_{0.8}\)Sr\(_{0.2}\)MnO\(_3\) was attributed to the extent of the redox processes: Mn\(^{4+} \rightleftharpoons \) Mn\(^{3+}\). The Sr-substitution for La on the catalyst can enhance the redox ability and desorption of the adsorbed and lattice oxygen, which, in turn, improves toluene oxidation.

### 2.3 Selectivity of CO\(_2\) and CO\(_2\)

Figure 3 shows the selectivity of CO\(_2\) and CO\(_2\) as a function of applied voltage with or without La\(_{0.8}\)Sr\(_{0.2}\)MnO\(_3\) catalyst. In the whole range of applied voltage, La\(_{0.8}\)Sr\(_{0.2}\)MnO\(_3\) assisted the formation of CO\(_2\) and CO\(_2\), and increased selectivity. Under the same applied voltage of 19.0 kV, the selectivity of CO\(_2\) was 28% without the catalyst but 59% with the La\(_{0.8}\)Sr\(_{0.2}\)MnO\(_3\) catalyst. At the same time, the selectivity of CO\(_2\) increased from 62% without the catalyst to 80% with the catalyst. In addition, when the catalyst was introduced into the reactor, the proportion of CO\(_2\) in the CO\(_2\) increased significantly, which meant that total oxidation of toluene was enhanced and more CO was oxidized to CO\(_2\). Lin et al. (1981) concluded that CO reacted with surface-adsorbed oxygen at relatively low temperature, which followed Eley-Rideal mechanism, and was oxidized by lattice oxygen at relatively high temperature.

The influence of catalyst on the selectivity of CO\(_2\) was further illustrated by a blank test, in which the inlet gas components were CO (volume percentage 0.5%) and air. The experimental results are presented in Fig. 4, which show that the oxidation reaction of CO was catalyzed by perovskite catalysts. At the same time, more CO\(_2\) was detected by the CO\(_2\) detector. The selectivity of CO\(_2\) was therefore increased. These results are in accordance with some previous studies (Peña and Fierro, 2001; Lin et al., 1981; Teng et al., 2007). Thus, the selectivity of CO\(_2\) and CO\(_2\) can be strengthened.

The mechanism for the oxidation of CO to CO\(_2\) may be as follows on the catalyst (Peña and Fierro, 2001):

\[
\begin{align*}
O_2(g) & \rightarrow O_{2(ad)}^2 \rightarrow 2O_{2(ad)}^{2-} \\
CO(g) & \rightarrow CO_{(ad)}^\cdot \\
CO_{(ad)}^\cdot + 2O_{2(ad)}^{2-} & \rightarrow CO_2(ad) \\
CO_2(ad) & \rightarrow CO_{2(ad)} \rightarrow CO_2(g) + O_{2(ad)}^{2-}
\end{align*}
\]

where, subscript (ad) denotes adsorbed species and subscript (g) denotes gaseous species. It has been reported that Mn ions with different oxidation states and lattice oxygen play an important role in the oxidation reaction of CO on perovskite-type catalysts (Labhsetwar et al., 2006).

#### 2.3.1 Byproducts

The formation of byproducts, including O\(_3\) and NO\(_x\), is one of the disadvantages for the application of non-thermal plasma. When plasma was combined with a catalyst, the generation of byproducts decreases significantly (Mista and Kacprzyk, 2008). In this research, the typical byproducts O\(_3\) and NO were studied.

As shown in Fig. 5, ozone formation was significantly influenced by the La\(_{0.8}\)Sr\(_{0.2}\)MnO\(_3\) catalyst. The ozone in the gas exhaust with catalyst was much lower than that without catalyst. For example, under the same applied voltage of 21.5 kV, the concentration of ozone was about 3630 mg/m\(^3\) without the catalyst but was 630 mg/m\(^3\).
with the La$_{0.8}$Sr$_{0.2}$MnO$_3$ catalyst. It was obvious that the La$_{0.8}$Sr$_{0.2}$MnO$_3$ catalyst diminished ozone formation through the decomposition of ozone to oxygen and active oxygen atoms, which play an important role in the oxidation of toluene (Delagrange et al., 2006).

It has been reported that ozone decomposition proceeds via the following three-step process (Futamura et al., 2004):

$$\text{O}_3^+ \rightarrow \text{O}_2 + \text{O}^+ \quad (9)$$

$$\text{O}^+ + \text{O}_3 \rightarrow \text{O}_2 + \text{O}_2^+ \quad (10)$$

$$\text{O}_2^+ \rightarrow \text{O}_2 + \text{e}^- \quad (11)$$

where superscript * denotes the active site on the catalyst surface.

Atomic oxygen formed in ozone decomposition plays an important role in toluene oxidation. In addition, it can react with water to form hydroxyl radicals, which also enhance toluene oxidation.

$$\text{O} + \text{H}_2\text{O} \rightarrow 2\text{OH}^- \quad (12)$$

Although NO is one of the byproducts generated by non-thermal plasma when air is used as the balance gas, it is often neglected by researchers. Figure 6 shows the NO concentration as a function of applied voltage with or without the La$_{0.8}$Sr$_{0.2}$MnO$_3$ catalyst. More NO was generated at higher applied voltage. The results also showed that the amount of NO in the effluent decreased greatly when the La$_{0.8}$Sr$_{0.2}$MnO$_3$ catalyst was introduced into the reactor. For example, under the same applied voltage of 19.0 kV, the concentration of NO was 12.3 mg/m$^3$ without catalyst but was 3.1 mg/m$^3$ with catalyst.

The decomposition of NO on La$_{0.8}$Sr$_{0.2}$MnO$_3$ catalyst could be described as follows (Peña and Fierro, 2001):

$$\text{M-O-M} + \text{NO} \rightarrow \text{M-O} + \frac{1}{2}\text{N}_2 (\Delta = \text{oxygen vacancy})$$

$$\Delta + \text{NO} + 2\text{e}^- \rightarrow \text{O}_2^+ + \text{N}_{(ads)} (\text{O}_2 = \text{lattice oxygen}) \quad (14)$$

$$\text{N}_{(ads)} + \text{NO}_{(ads)} \rightarrow \text{N}_2\text{O} \quad (15)$$

$$2\text{N}_{(ads)} \rightarrow \text{N}_2 \quad (16)$$

where, subscript ads denotes the adsorbed species. The electron comes from the concomitant oxidation of the B-site cation (Mn$^{3+}$ → Mn$^{4+}$) neighboring the vacancy.

### 2.3.2 Influence of ultrasound on toluene removal efficiency

Figure 7 shows the influence of ultrasound on the conversion of toluene. When the DBD reactor without catalyst was combined with ultrasonic decomposition system, the ozone was utilized to generate more active free radicals and the conversion of toluene increased with the applied voltage, which meant that ultrasonic irradiation assisted the degradation of toluene. Results showed that toluene removal efficiency was 78.4% without ultrasound at 21.5 kV but was 93% with the combination of ultrasound and ozone. The significant increase in active free radicals formed in the hot cavitation bubbles and at the gas-liquid interface of the bubbles was likely due to the interaction of ultrasound and ozone. Toluene removal proceeds not only via active free radicals, but also via direct pyrolysis in the...
2.4 Mechanism of toluene destruction in DBD

Many energetic electrons are generated in the discharge zone. The distribution of electron energy in DBD is in accordance with Maxwell function. The percentage of electrons whose energy value is equal to $\varepsilon$ can be expressed as follows (Zhao, 1993):

$$f(\varepsilon) = 2.07 \left(\frac{\varepsilon}{\bar{\varepsilon}}\right)^{-3/2} e^{-1/2\varepsilon}$$  \hspace{1cm} (24)$$

where, $f(\varepsilon)$ denotes the percentage, and $\bar{\varepsilon}$ denotes the average energy of electrons in DBD system.

Using a series of mathematical operations and equation counter changes on Eq. (24) previous research determined that the percentage of electrons higher than 5.4 eV, the bond energy of C=O in an aromatic ring, was about 45.8% (Ye et al., 2008). Therefore, C=C can only be destroyed by high-energy electrons above 5.4 eV, thereby less than half the toluene can possess ring-cleavage dissociation. However, more than 50% toluene was removed in the previous experiment. Therefore, other active species and reactions must exist which led to toluene removal except for direct electron-impacts.

Former research has reported that toluene destruction via DBD can be achieved through three pathways, i.e., electron-impacts, attacks by other active radicals (such as O and OH) and ion collisions (Huang et al., 2011). However, the effects of ion collisions are negligible (Lee and Chang, 2003). The formation of O atoms and OH can be illustrated by Eqs. (25) and (12):

$$O_2 \xrightarrow{\varepsilon} O + O$$  \hspace{1cm} (25)$$

The oxygen is attacked by the energetic electrons to generate O atoms due to its relatively higher concentration as background gas and low bond energy. The O atoms react with H$_2$O and H to form OH and other oxidation products (Ye et al., 2008). The production of OH has been previously analyzed through HPLC (Guo et al., 2010). The production of OH has been counter changes on Eq. (24) previous research determined that the percentage of electrons higher than 5.4 eV, the bond energy of C=O in an aromatic ring, was about 45.8% (Ye et al., 2008). Therefore, C=C can only be destroyed by high-energy electrons above 5.4 eV, thereby less than half the toluene can possess ring-cleavage dissociation. However, more than 50% toluene was removed in the previous experiment. Therefore, other active species and reactions must exist which led to toluene removal except for direct electron-impacts.

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The H-abstraction from the methyl group by electrons should be the first step of toluene oxidation because of the lowest dissociation energy of C–H bond in methyl. The aromatic intermediates will then be further attacked by electrons and OH, causing direct cleavage of the aromatic ring. All of the intermediates proceed by a series of complex oxidation steps by O/H attack, finally leading to the production of CO$_2$ and H$_2$O.

3 Conclusions

The synergistic effect of NTP and La$_{0.8}$Sr$_{0.2}$MnO$_3$ catalyst was observed in this study. The La$_{0.8}$Sr$_{0.2}$MnO$_3$ catalyst increased toluene removal efficiency and the selectivity of CO$_2$ and CO$_2$, as well as suppressed byproducts (such as O$_3$ and NO) formation at the discharge temperature. In addition, the combination of DBD reactor with ultrasonic
system revealed certain effects on toluene removal efficiency. Ultrasonic energy not only enhanced the mass transfer of gaseous ozone to the liquid phase, it also increased the production of active radicals, which played an important role in the degradation of toluene and the reduction in ozone emissions.

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