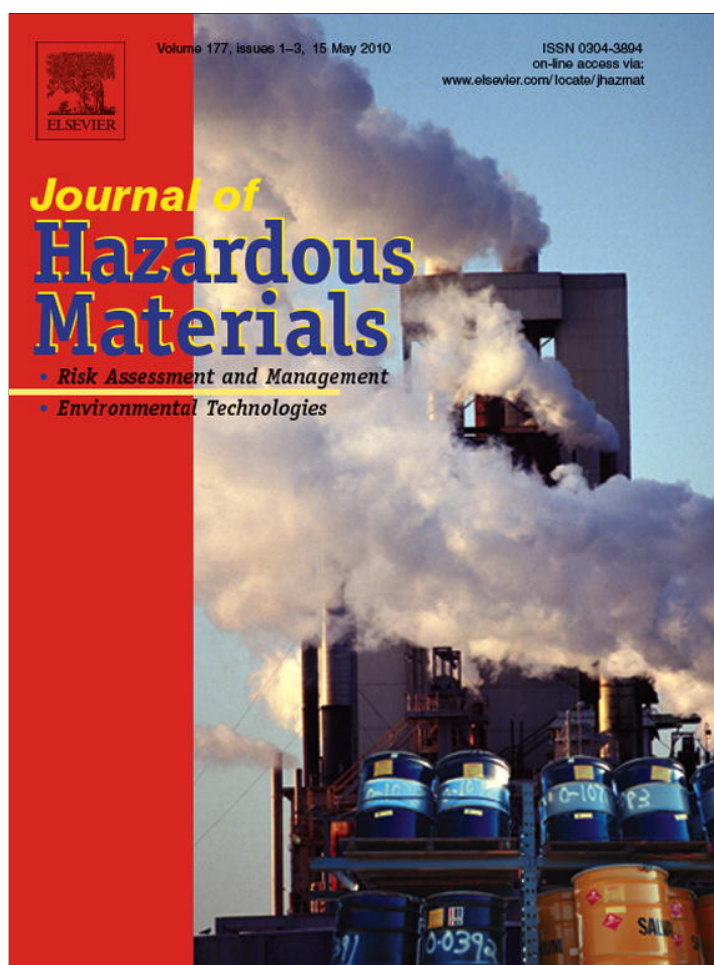




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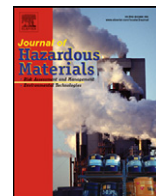
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Abatement of malodorants from pesticide factory in dielectric barrier discharges

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ABSTRACT

Traditional odor control methods are limitative technically and economically for the abatement of odor from pesticide factory due to its toxicity and complicated composition. Non-thermal plasma (NTP) methods, typically characterized by high removal efficiency, energy yields and good economy, offer possible alternative solutions. This paper provides laboratory scale experimental data on the removal of simulated odors from pesticide factory with various humidity (0–0.8 vol%) and oxygen contents (0–21%) by a dielectric barrier discharge (DBD) reactor. Peak voltage and initial dimethylamine (DML) concentration are important factors that influence the DML removal efficiency and energy yield. The conversion of DML of 761 mg/m³ reaches 100% at a peak voltage of 41.25 kV. Under the experiment conditions, the conversion of DML increases with an increase of oxygen contents. And the highest DML conversion was achieved with the gas stream containing 0.3% water. Simultaneously, the concentration of O₃ and OH radical in reactor was measured. Higher conversion, higher energy yield and fewer byproducts were found in mixed odor (DML + dimethyl sulfide (DMS)) treatment than that in single odor treatment. The energy yield is promoted from 2.13 to 5.20 mg/kJ.

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1. Introduction

Odor control from pesticide factory has become an increasing concern because of the detrimental effect on both human health and global environment. Serious human health problems attributed to such odors include damage to the liver, kidneys, respiratory system and central nervous system [1]. Due to the odors' toxicity and low detection threshold, substantial complaints from neighbors and many conflicts between surrounding residents and factories have occurred. And the exhaust streams from pesticide factory have variable composition, humidity and oxygen content [2]. Traditional odor control methods, such as wet scrubbing, active carbon adsorption, ozone oxidation and biofiltration, are limitative technically and economically [3].

Recently, NTP application in odor control has been receiving increasing attention because of high removal efficiency, energy yields and good economy. Different types of reactors and power supplies have been developed for the production of NTP in the early researches. And the early researches mainly paid attention to the improvement of the conversion efficiencies, reduction of energy cost and product analysis in single odor treatment with dry air [4–12]. Whereas, it was mentioned above that the odors from pesticide factory had complicated composition with various humidity and oxygen contents. To realize the decomposition char-

acteristics of odors in pesticide factory adequately, in this study, the decomposition of DML and DML + DMS (representative mixed odor), which are typical gaseous pollutant from pesticide factory, was investigated in air with various humidity and oxygen contents. A wire-cylinder DBD reactor energized by a high voltage source with BPFN was adopted. Special consideration was given to some important parameters that may affect odor decomposition, including peak voltage, initial concentration, the density of O₃ and OH radical in plasma system, chemical structures and chemical properties of odors. The comparison of energy yield and byproducts in single odor treatment and in mixed odor treatment was studied.

2. Experiment

2.1. Experimental setup, reactor and power supply

Fig. 1 shows a schematic diagram of the experimental setup. Air, N₂ or O₂ from appropriate gas cylinders is allowed into an odor feeding system at a controlled flow rate through Teflon tubes. A portion of the gas flow goes through a bubbler filled with DML to an appropriate level. For experiments with mixed odors treatment, another stream passes through the DMS generator and takes out DMS vapor. For experiments with humid air, a portion of the air flow goes through another bubbler filled with deionized water to an appropriate level. Then all the streams are mixed in a buffer tank, where humidity is measured by a hygrometer (Rotronic A1H). At last, the mixed stream (total flow rate of 1000 mL/min) is introduced to the DBD reactor. Samples of the treated gas for off-line

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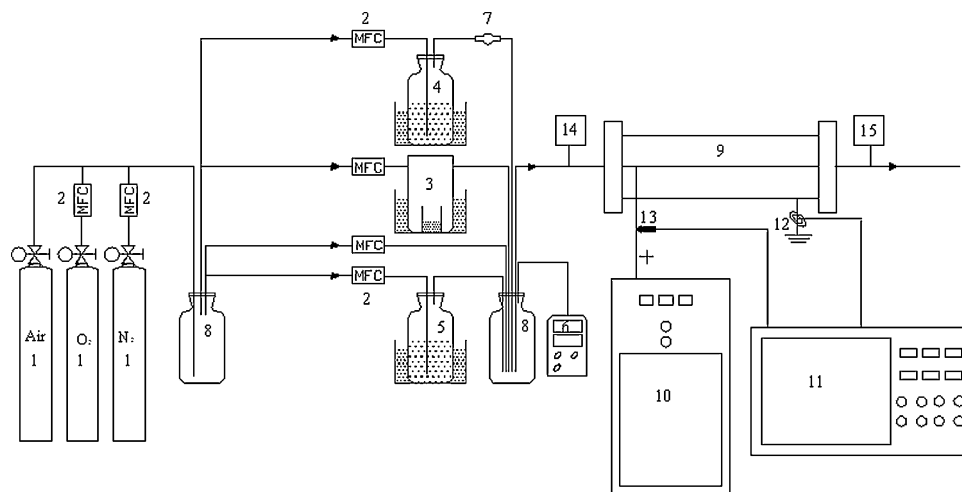


Fig. 1. Schematic diagram of the experimental setup (1. gas cylinder; 2. mass flow control; 3. DMS generator; 4. DML generator; 5. water vapor generator; 6. hygrometer; 7. drying tube; 8. buffer tank; 9. wire-cylinder DBD reactor; 10. high pulse voltage source; 11. digital storage oscilloscope; 12. current probe; 13. high voltage probe; 14. sampling of inlet gas; 15. sampling of outlet gas).

chemical analyses are collected with a gastight syringe from the sampling ports located at the inlet and the outlet of the reactor. The plasma reactor is a 30 mm (inner diameter) $\times$ 90 mm epoxy resin cylinder with a Ni–Cr alloy wire (the energized electrode) of 0.5 mm in diameter fixed along its axis. A ceramic tube made of alumina was used to form the dielectric barrier wall. The ground electrode is a film conductor out of aluminum and embedded between the epoxy resin tube and ceramic tube. A Blumlein pulse forming network (BPFN) type of narrow pulse generator is described in detail in previous work [9]. The pulse frequency adopted in this study is 100 pps (pulse per second). The pulse voltage and current waveforms were measured by using a four channel Tektronix TDS 2014B 350 MHz digital storage oscilloscope capable of sampling 1 GS/s (Giga Sample per second), a Texas HVP-3020 high voltage passive probe and a CT4 TCP202 current probe.

2.2. Chemicals

Air (Jingong Co., Ltd.) was a synthetic mixture (80% nitrogen and 20% oxygen) from pure liquid nitrogen and pure liquid oxygen with specified impurities of H₂O. The purity of N₂ and O₂ (Jingong Co., Ltd.) were 99.999% and 99.8%. The purity of DMS (ACROS ORGANICS, Belgium) was 99+%. DML (WULIAN Chemical Plant, China) was supplied in solution with water and salicylic acid ($\geq 99.0\%$) was also purchased from WULIAN Chemical Plant, China.

2.3. Experimental procedure

The following standard procedure was developed to achieve stable and reproducible conditions for the gas flow rate and composition. The reactor is first flushed with air at 1000 mL/min for a few minutes. Odor-containing gas is then allowed in at an appropriate flow rate for 30 min, at which point the odor concentration measured at the reactor outlet is the same as that at its entry. The desired flow rate for the specific experiment is then set and the pulse voltage is applied. After a stabilization time of about 5 residence times to obtain a steady concentration of the residual odor at the reactor outlet, two consecutive samples of the treated gas are withdrawn and subjected to GC analysis. After a group of experiments is completed, the gas flow is stopped and the pulse voltage source is switched off. The apparatus is finally cleaned by flushing air at 1000 mL/min for 15 min. In experiments with humidified gas, preconditioning of the reactor is achieved by flushing humidified air through it for 30 min. Then, the odor-containing gas, humidified as

described above, is introduced into the system. All the experiments above were achieved at ambient temperature.

2.4. Chemical analyses

The concentrations of DML and DMS were measured by the gas chromatograph GC7890II (Tianmei Corporation) which was equipped with six-port gas sample valve. The concentration of sulfur dioxide and nitrogen oxides was analyzed by a gas analyzer (KM9106, Kane International Limited) equipped with KMCLP20 standard probe. The concentrations of ozone and ammonia were measured by the gas indicator tubes (Sanhuan Corporation), and the minimum detectable limits of which were 0.2 ppm and 10 mg/m³.

2.5. Measurement of hydroxyl radicals

A film, which was impregnated with salicylic acid, was used to detect OH radical in plasma reactor [13]. Salicylic acid reacts with OH radical and produces 2,5-dihydroxybenzoic acid (2,5-DHBA). Then, a high performance liquid chromatography (HPLC) was carried out to detect the concentration of 2,5-DHBA. Therefore, OH radical in NTP reaction could be calculated.

One piece of filter paper was cut to a calculated size (300 mm $\times$ 45 mm), then a solution, which was made by 0.3 g salicylic acid dissolved in 10 mL anhydrous ethanol (99.9%), was dripped equably by a injector to this filter paper and dried. The prepared sampling film was puckered and then placed in plasma reactor. The collecting time was set as 3 h. A shorter or longer time made the results below the minimum detectable limits or inaccurate [13]. After the sampling film was taken out and was cut to fragments, all these fragments were dipped in water (15 mL). After that, an ultrasonic cleaner was used to accelerate dissolution of the products. Then the concentration of 2,5-DHBA was detected by a HPLC, equipped with an auto sampler (model G1329A) and an ultraviolet spectroscopic photometer (300 nm).

The concentration of OH radical could be calculated as the following equation:

$$C_{OH} = \frac{C_{2,5-DHBA} \times V_L \times N}{F_{2,5-DHBA} \times F_g \times t \times \eta \times (1 - \alpha)} \quad (1)$$

where, $C_{2,5-DHBA}$ is the concentration of 2,5-DHBA (mol/L); V_L is the volume of absorbing liquid (L); N is Avogadro constant (6.02×10^{23}); $F_{2,5-DHBA}$ is the producing ratio of 2,5-DHBA (In this

experiment, almost all salicylic acid that reacted with OH radical produced 2,5-DHBA, therefore, $F_{2,5\text{-DHBA}}$ was assumed to be 1; F_g is gas flow rate (mL/min); t is sampling time (min); η is pick-up efficiency (it was assumed to be 1 in this experiment because of almost all 2,5-DHBA picked up from the film); α is the product losing ratio (it was assumed to be 0).

2.6. Definition of energy yield

Energy yield (EY) is defined as follows:

$$EY = \frac{(C_1 - C_2)Q}{60,000P} \quad (2)$$

$$P = Ef \quad (3)$$

$$E = \int_0^T u(t)i(t)dt \quad (4)$$

where C_1 is the initial concentration of DMS (mg/m^3); C_2 is the concentration of DMS in the effluent gas (mg/m^3); Q is the gas flow rate (mL/min); P is the discharge power (W); E is the energy of the single pulse (J); f is the pulse frequency (pps); $u(t)$ is the instantaneous voltage of a single pulse (V); $i(t)$ is the instantaneous current in a single pulse (A); T is the time of a single pulse (s).

3. Results and discussion

3.1. Decomposition of DML in dry air

Conversion of DML was studied as a function of peak voltage varied in the range of 11.5–41.25 kV at a fixed pulse frequency of 100 pps. These conditions correspond to the energy density of 52–675 J/L. The initial concentrations of DML are 761, 1271 and 2652 mg/m^3 , respectively. As seen from Fig. 2(a), the DML conversion of various initial concentrations increases with the increasing peak voltage. It is commonly considered that at higher peak voltage, application of plasmas with higher electron energies is favorable for the removal process due to the enhanced production of active radicals, ions, and other excited species [14,15].

Fig. 2(a) shows that the initial DML concentration is also an important factor that influences the DML removal process. The lower concentration of DML is easier to clean. At a fixed peak voltage of 27.5 kV, the conversions of DML of 761, 1271 and 2652 mg/m^3 are 97, 70 and 27%, respectively. From Fig. 2(b), it can be seen that the energy yield increases with an increase of initial concentration of DML and decreases with the increasing peak voltage under the experimental conditions. The phenomenon can be explained by that with the same amount of active species produced in the processed stream, the portion of species used for decomposing DML is higher for higher DML concentrations. This causes the reduction of energy cost with the increase of the initial pollutant concentration [16]. When the peak voltage is too high, it causes energy waste in the chemical process. Therefore, the energy cost could be reduced when the odors from pesticide factory are concentrated within an appropriate range before DBD processing and the energy input is control within an appropriate range.

3.2. Decomposition of DML with various oxygen contents

The decomposition of DML was investigated under various oxygen concentrations. Oxygen content varies from 2 to 21 vol%. The gas flow rate is 1000 mL/min and the initial concentration of DML is 1272 mg/m^3 . Fig. 3 shows the relationship between the conversion of DML and the concentration of O_2 . The result shows that the decomposition of DML increases sharply with an increase of O_2 concentration when the O_2 concentration is below 10%. And the

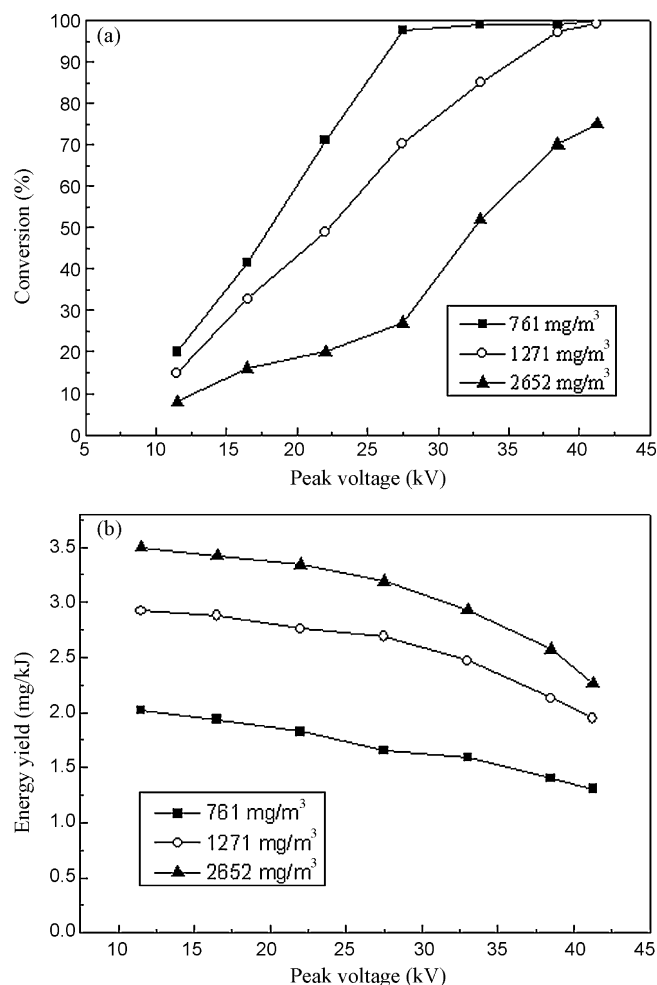


Fig. 2. (a) Decomposition of DML in dry air and (b) energy yield of DML decomposition in dry air (frequency pulse, 100 pps; gas flow rate, 1000 mL/min).

increase of the decomposition of DML becomes slight when the O_2 concentration is above 10%. When the oxygen content is 21% and the peak voltage reaches 41.25 kV, the DML conversion of 95% is achieved. Concerning the reason for the influence of O_2 in plasma system, researches reported O free radical and ozone play impor-

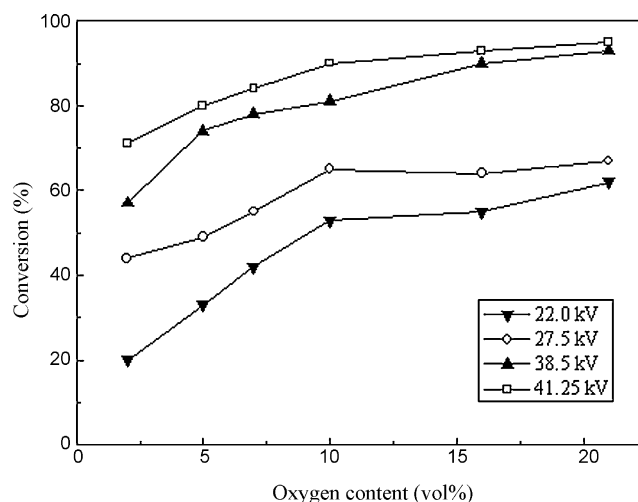


Fig. 3. Decomposition of DML in O_2/N_2 (balance gas, O_2/N_2 ; gas flow rate, 1000 mL/min; initial concentration, 1272 mg/m^3 ; humidity, 0%).

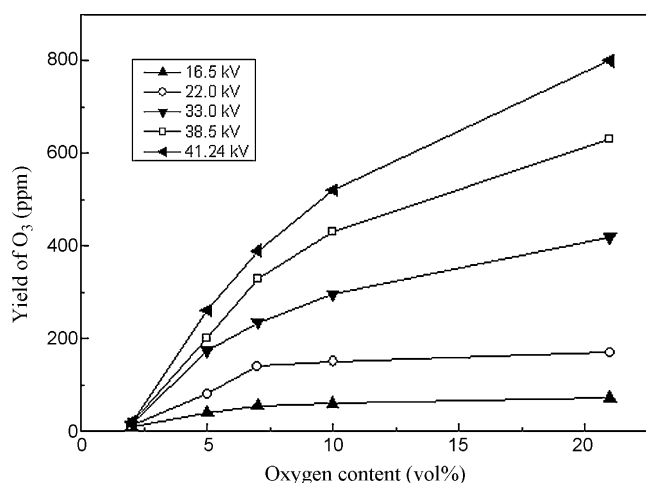


Fig. 4. The amount of ozone produced in corona plasma reactor without DML with various O₂ concentrations (balance gas, O₂ and N₂; gas flow rate, 1000 mL/min).

tant roles in plasma chemical process [17,18]. In order to investigate the role of ozone in decomposition of DML, the amount of ozone formed in plasma system without DML was investigated (Fig. 4). As seen from Fig. 4, the yield of ozone increases with the increasing O₂ content at a fixed peak voltage. When the O₂ increases from 2 to 10%, the amount of ozone rapidly increases from 15 to 295 ppm at a fixed peak voltage of 33 kV. On the other side, when the O₂ increases from 10 to 21%, only 115 ppm of ozone increase is obtained. The variation of the amount of ozone is quite consistent with that of DML conversion under the same O₂ content. Therefore, the change of O₂ content leads to the variation of ozone produced by DBD, resulting in enhancing or restraining the decomposition of DML.

3.3. Decomposition of DML with various humidity

The effect of humidity on DML decomposition is examined by passing dry and humidified air (0–0.8 vol%) through the DBD reactor. Fig. 5 shows that humidity strongly affects DML decomposition in plasma. For a peak voltage of 33 kV, the conversion of DML is 85% in dry air. The conversion of DML is promoted to 93% as the humidity increases to 0.3%. What is interesting is that when the humidity increases to 0.5 and 0.8%, the conversion of DML sharply drops to 71 and 69%. This indicates the promoting effect of low humidity

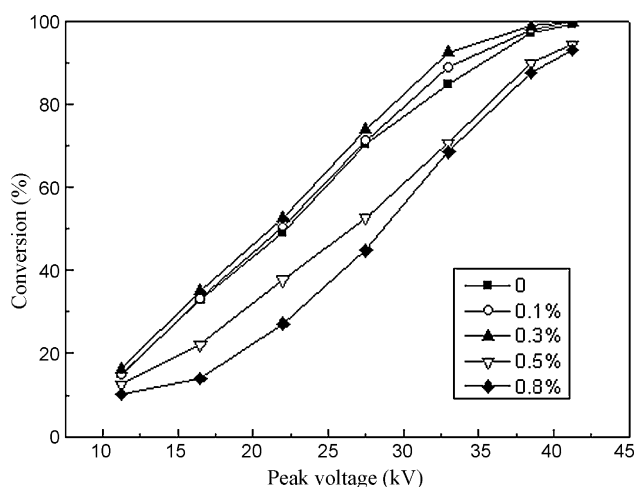


Fig. 5. Effect of humidity on DML decomposition (balance gas, air; gas flow rate, 1000 mL/min; initial concentration, 1272 mg/m³).

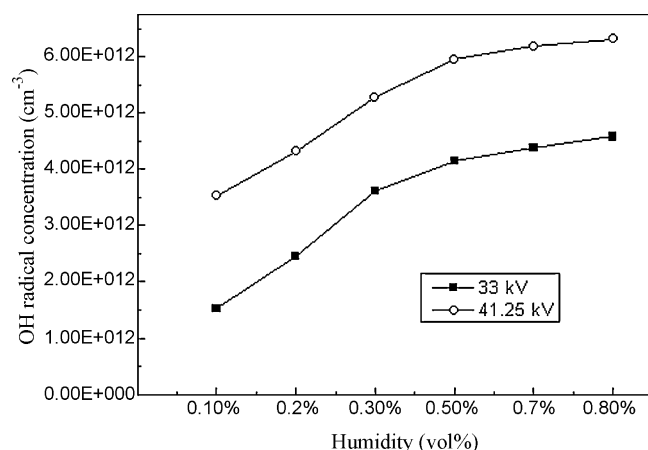


Fig. 6. OH radical density as a function of humidity in the plasma system (balance gas, air; gas flow rate, 1000 mL/min).

(≤0.3%) and the adverse effect of high humidity (≥0.5%) on DML decomposition in plasma system. Similar results are obtained for other peak voltages.

In agreement with these findings, Guo et al. [19] reported water plays a very important role in the reaction by forming OH radicals inside the plasma system. Fig. 6 shows the OH density as a function of humidity in the plasma system. At lower humidity (≤0.3%), OH density increases with an increase of humidity. OH is extraordinarily active and takes part in many chemical processes [19]. It explains the promoting effect of low humidity on DML decomposition. At a higher humidity (≥0.5%), OH production is saturated as humidity increases. A possible reason of low OH formation at high humidity is decrease in electron mean energy by H₂O molecules. However, the conversion of DML decreases dramatically when the humidity is higher than 0.5%. It implies that next to OH formation, also other processes play an important role towards DML decomposition.

The importance of ozone in plasma processing is discussed in the previous part of the research (Section 3.2). On the other hand, humidity also strongly affects forming ozone in plasma system (Fig. 7). Water molecules might compete with oxygen for high-energy electrons [20]. Indeed, for a peak voltage of 38.5 kV in dry air, the ozone concentration in the plasma outlet gas stream is 630 ppm. The ozone production in the plasma decreases at higher humidity. An ozone concentration in plasma system of 530 ppm is monitored at 0.5% humidity, and 500 ppm at 0.8% humidity. Since more ozone

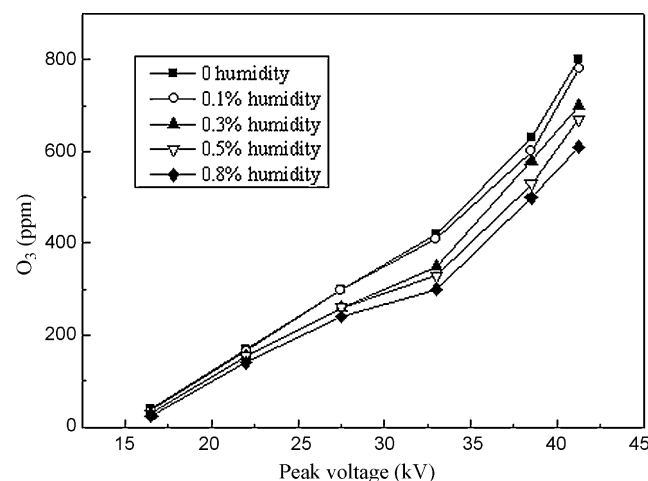


Fig. 7. The amount of ozone produced in corona plasma reactor in air with various humidity (balance gas, air; gas flow rate, 1000 mL/min).

Table 1

Comparison of energy yield between single odor treatment and mixed odors treatment (balance gas, dry air; gas flow rate, 1000 mL/min; initial concentration, DML 1271 mg/m³, DMS 1324 mg/m³).

	Single DML	Single DMS	Mixed DML	Mixed DMS
Initial concentration (mg/m ³)	1271	1324	1250	1338
Maximum conversion (%)	99	99	99	99
Energy yield (mg/kJ)	2.13	2.24	2.69 + 2.51 = 5.20	

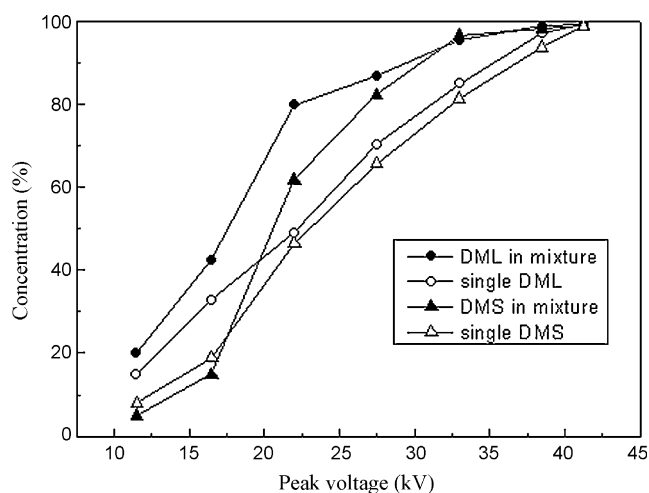


Fig. 8. Conversion of DML and DMS in the plasma system (balance gas, dry air; gas flow rate, 1000 mL/min; initial concentration, DML 1271 mg/m³, DMS 1324 mg/m³).

in reactor could promote the decomposition of DML. High humidity, resulting in inhibiting ozone production, leads to an adverse effect on DML decomposition.

In a word, from Figs. 6 and 7, it can be seen that different factors codetermine the decomposition of DML under humid conditions. Generally, the humidity in pesticide factory is high. Therefore, the odors should be controlled within an appropriate humidity level before DBD process.

3.4. Decomposition of mixed odors

Fig. 8 shows that the conversion of DML and DMS differs according to whether they were treated together. The conversion of DML is higher than the one treated separately under a fixed peak voltage when it was mixed with DMS. At a peak voltage of 22 kV, the removal efficiency of DML was improved from 62 to 80% when mixing with DMS. DML and some of its intermediate products are alkali. They can react with the byproducts such as SO₂, CO₂, and NO_x from DMS decomposition process to generate ammonium nitrates ((NH₄)₂SO₄, (NH₄)₂CO₃, NH₄NO₃, etc.), which promotes the decomposition of DML [10]. Also, the conversion of DMS is higher than that treated separately under large range of peak voltages when it was mixed with DML. The reason for this phenomenon might be similar with that for the effect on DML. However, at low peak voltages of 11.5 and 16.5 kV, the conversion of DMS reduces slightly when it is in mixed odors. It has been previously reported that high-energy electrons, active species such as radicals and ions play an important role in the

decompositions of polluting gases and VOCs [21]. The above finding suggests that although the reaction between DML and DMS improves the decomposition process at large range of peak voltages, the rivalry of high-energy electron and activated particle still exists. Thus, the removal efficiency of DMS decreased compared with the single treating when the energy put into the reactor is not high enough.

As seen from Table 1, the energy yields of single DML and single DMS treatment are 2.13 and 2.24 mg/kJ. When the two odors were treated together, 2.69 mg DML and 2.51 mg DMS can be removed simultaneously by 1 kJ of energy. And the energy yield of mixed odors treatment is 5.20 mg/kJ. Clearly, the decomposition of actual odors (mixed odors) can save energy cost because of higher energy yield than single malodorant decomposition.

It can also be found from Fig. 8 that the conversion of DML is higher than that of DMS in a large range of peak voltage both in single treatment and in mixture treatment. The weakest bond in DML (CH₃NHCH₃), C–N (bond dissociation energy: 342.7 kJ/mol) is lower than C–S (307.9 kJ/mol) in DMS (CH₃SCH₃). However, except for discharge chemistry processes, reactivity with their own decomposition products CO₂, SO₂ and NO_x also exists in their decomposition processes. It is mentioned above that DML and some of its intermediate products are alkali, while CO₂, SO₂ and NO_x are acidic. Therefore, the reactivity between odors and their own decomposition products is more significant for DML decomposition.

It can be concluded from above that the removal efficiencies of malodorants are mostly related to their chemical structures [22], chemical properties, discharge power, reactivity with electrons, active species and decomposition products generated in a DBD discharge, gas composition [10].

3.5. Byproducts analysis

Identifying the byproducts during decomposition of odor is one of the most important issues for the plasma chemical processes. In the cases of DML and DMS removal, the main byproducts are NO_x, NH₃, SO₂ and O₃. As seen from Table 2, at a fixed peak voltage of 41.25 kV, the amount of NO_x, NH₃, SO₂ and O₃ in mixed odor treatment, which is 15, 20, 30 and 43 mg/m³, is much smaller than that in single odor treatment. It is discussed in Section 3.4 that reactivity with their own decomposition products CO₂, SO₂ and NO_x exists in DML + DMS decomposition processes. Therefore, mixed odors treatment leads to less byproducts formation.

Moreover, some sulfur and white crystal ammonium nitrates were discovered adhering to the electrode wire and reactor wall. The byproducts, such as NH₃ and NO_x, react with each other to generate ammonium nitrates like NH₄NO₃. These chemicals, being solid and soluble in water, can be easily removal. And they can also be recycled and utilized as fertilizers. Quantifying the sulfur and

Table 2

Comparison of byproducts in single treatment and in mixed odor treatment (balance gas, dry air; gas flow rate, 1000 mL/min; peak voltage, 41.25 kV; initial concentration, DML 1271 mg/m³, DMS 1324 mg/m³).

	DML			DMS			DMS + DML			
	NO _x	NH ₃	O ₃	NO _x	SO ₂	O ₃	NO _x	NH ₃	SO ₂	O ₃
Concentration in outlet gas (mg/m ³)	30	40	386	200	200	235	15	20	30	43

ammonium nitrates will be studied to set up sulfur balance and nitrogen balance in the further research.

4. Conclusion

In this study the decomposition of simulated odor (DML and DML + DMS) in pesticide factory was investigated in air with various humidity and oxygen contents. Peak voltage and initial concentration are important factors that influence the DML removal efficiency and energy yield. The conversion of DML of 761 mg/m³ reaches 100% at a peak voltage of 41.25 kV. Higher oxygen content (0–21%) promotes production of active species such as ozone, leading to higher DML conversion. Humidity enhances the amount of OH radicals and inhibits ozone production in reactor, which codetermines the optimum humidity of 0.3% under the experiment conditions (0–0.8%). When DML and DMS were decomposed together, synergistic actions exist in the processes, leading to higher conversion, higher energy yield and less byproducts formation. The energy yield is promoted from 2.13 to 5.20 mg/kJ.

Acknowledgments

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