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# Photolysis of gaseous butyl acetate using built-in microwave discharge electrodeless lamps

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#### ARTICLE INFO

### ABSTRACT

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*Keywords:* Microwave discharge electrodeless lamps Photolysis Butyl acetate Gaseous pollutants This paper examined the photolysis of gaseous butyl acetate by built-in microwave discharge electrodeless lamps. Bromine and iodine lamps were used to investigate the effects of microwave input power, inlet concentration, gas residence time, and different filling materials on butyl acetate decomposition and energy yield. Water vapor and ammonia were utilized as additional chemicals to determine if they can improve removal efficiency. Built-in structure and external structure were also compared. When the gas flow velocity is 0.025 m/s, and the inlet concentration is 1.5 mg/m<sup>3</sup>, the removal efficiency of butyl acetate can reach over 78%, which is the highest in this study. Additional water vapor and ammonia can hardly improve the degradation. To some extent, built-in MDELs are better than external MDELs. The results will help promote the study of microwave discharge electrodeless lamps and their effect on the degradation of gaseous pollutants. Maybe, as the research goes on, it could realize the span from laboratory scale to practical application, and will become an advanced technology for the gaseous pollutants control.

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## 1. Introduction

Volatile organic compounds (VOCs) are major pollutants and so have been the focus of a number of studies in recent years [1,2]. Butyl acetate, an important VOC, is a kind of malodorous gas widely used in many industries in the production of paints, artificial leather, films, nitrocellulose, and drugs, among other products. However, butyl acetate (34–50 mg/L) can intensely irritate the eyes and the nose, and at high concentrations, it may even have anesthetic effects.

Butyl acetate can be treated by biofiltration [3]. However, in industrial applications, the exhaust emission is cleaned intermittently, which makes treatment by biological systems difficult. Furthermore, the biological method requires a large occupancy area and a long treatment cycle [3,4].

To complement conventional techniques, Demidiouk et al. [5] used a plasma-catalytic system to decompose butyl acetate. However, this technique is not practical because platinum is too costly as catalyst. Microwave discharge electrodeless lamps (MDELs), a unique light source, were first reported in 1968 [6]. The principle involved in MDELs is the absorption of high-frequency electromagnetic waves produced by a microwave (MW) generator by the noble gases and vaporizable elements in the lamp, which results in UV emission. Mercury, sodium, sulfur, selenium, and cadmium can be used as vaporizable elements, whereas the noble gases are generally argon, neon, and krypton, among others [7]. Compared with normal UV light sources, MDELs do not produce blackened electrodes caused by wear and tear, oxidation, and sealing due to the absence of electrodes. In addition, MDELs have many advantages such as their low price, low energy cost, high light intensity, and simple reactor. Moreover, there are many options for light-emitting materials so that MDELs can provide different wavelengths of UV radiation [8].

Electrodeless discharge lamps started to be used for the degradation of pollutants in the late 1990s. However, most studies involving microwave ovens for microwave production examined the degradation of pollutants in the water phase, with the UV wavelengths mostly over 250 nm [9–13]. Most chemical bond energies of the molecules of organic pollutants, such as C=C, C-F, C=O, and N=O, are greater than 5.0 eV [14], but the 250 nm photon energy is 4.97 eV, which can only be absorbed by a small number of organic pollutants. On the other hand, the penetration rate of the photon energy of UV in lamp tubes is poor. For instance, the penetration rate of UV light in the frequently used 185 nm mercury water vapor lamp is only 10%. Therefore, its radiation efficiency is low, and its radiant intensity is merely about 20 mW/cm<sup>2</sup> [15]. Given this, UV radiation with a wavelength of 190-210 nm is suitable for the degradation of pollutants. However, although the photodegradation of organic pollutants by MDELs in the gas phase is feasible, literature on this topic is rare.

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Fig. 1. Schematic of the built-in MDELs reaction system.

Our research group has carried out several pioneering studies on gaseous pollutants. Since 2004, we have been examining the photolysis of simulating  $H_2S$  and  $CS_2$  using self-developed MDELs [16,17]. However, the MDELs used in these studies are of an external style, i.e., only a part of the UV light tubes is in the resonant cavity. This phenomenon results in low luminous efficiency and low energy efficiency.

This paper aims to evaluate the removal efficiency, operating modes, and decomposition mechanism of butyl acetate using built-in MDELs in order to overcome the disadvantages of external MDELs, namely, UV light tubes that are absolute in the resonant cavity. The use of built-in MDELs processes for butyl acetate degradation may be present good efficiency. On the other hand, the main advantage of this kind of treatment is the need of a short time to obtain an efficient treatment. Compared to external MDEL methods, this style can attain high luminous efficiency and high energy efficiency.

A number of studies have found treatment style effects on degradation of waste gas efficiency [16,17]. However, each study used different methods and studied difference of the target compounds ecosystems, so it is impossible to simply compare these studies to ours. In our study, the use of built-in MDELs to degrade gaseous butyl acetate, using this new type treatments methods, will be shown to yield useful industrial application information.

#### 2. Materials and methods

#### 2.1. Reagents

The sources and purity of the reagents used are as follows. Butyl acetate (purity 99.9%) was obtained from the Shanghai Baorui Chemical Corporation, nitrogen (99.99%) from Shanghai Pujiang Special Gas Corporation, compressed air from Shanghai Bixiou Gas Industry Corporation, and concentrated NH<sub>3</sub>·H<sub>2</sub>O (25%) from Jiangsu Tongcheng Chemical Corporation.

These reagents were not further processed before use.

#### 2.2. Built-in MDELs and reaction procedures

As shown in Fig. 1, a high-quality electric cable was used to transmit the high voltage produced by a high-voltage power (developed with the assistance of Shanghai Yalian Microwave Tec. Co.) to the magnetron in order to establish microwave (2.45 GHz). Microwave was reflected and converged in the resonant cavity.

When it reached a certain intensity, the electrodeless lamps were illumined in 1–2 s and issued UV radiation.

The electrodeless lamps, made of quartz tubes with a 20 mm external diameter, were sealed up at both ends. Two MDELs were examined in the experiments: MDEL-Br<sub>2</sub> filled with Br<sub>2</sub> (0.5 Torr) and Kr (1 Torr), and MDEL-I<sub>2</sub> filled with I<sub>2</sub> (0.5 mg) and Kr (2 Torr). Both MDELs were 20 cm long and were fixed in the middle of the resonant cavity.

Butyl acetate, a simulating pollutant, was in 1–6 bottles placed in a thermostat tank to keep the temperature constant. Due to the high volatility of butyl acetate, the gas from the compressed air cylinder flowed through the bottles, carrying gaseous butyl acetate to the liquid surface. The gas was mixed and diluted with the surrounding air in a cyclone device made of pot scourers to form gaseous butyl acetate with a certain concentration, which was fed axially into the MW/UV reactor under a tunable air blower. On account of the 1000 Pa pressure loss of the blower, the pressure in the discharge zone was lower than but near atmospheric pressure.

#### 2.3. Analysis methods

The gaseous butyl acetate samples were analyzed by a gas chromatograph (GC-930, China) with a 2 m long Porapak Q column maintained at 473 K and a hydrogen flame ionization detector. The removal efficiency of butyl acetate ( $\eta = (C_{in} - C_{out})/C_{in}$ ,  $C_{in}$ , and  $C_{out}$ are the inlet and outlet concentrations of butyl acetate, mg/m<sup>3</sup>) was calculated from the GC peak areas before and after discharge. The concentration of butyl acetate is measured by gas chromatography on line (T-column: 160 °C, T-vaporizer: 180 °C, T-detector: 200 °C).

The UV radiation emitted by MDELs was detected by an Acton VM-505 VUV Monochrometer (Acton, America). Products and intermediate products were collected by liquid nitrogen freezing or deionized water absorption using an air sampler. The products were then analyzed by gas chromatography–mass spectrometry (GC–MS, HP 5973-6890, America) or ion chromatography (IC, Dionex ICS3000, America). Butyl acetate was examined by the S-3100 Photodiode Array Detector (PDA, Sinco CO.) on the spectral range of 190–1100 nm at room temperature. The software version used was LabPro plus Build 342.4. Gas flow velocity (*v*) was measured by a hot bulb anemoscope (QDF-3, Beijing Detection Instrument Co.).

To measure energy efficiency, energy yield  $[Ey = QC_{in}\eta/P (mg/kWh)$ , where *Q* is the gas flow rate  $(m^3/h)$ , and *P* is the input power (kW)] was used.



**Fig. 2.** Comparison of the removal efficiency by MDEL-Br<sub>2</sub> and MDEL-I<sub>2</sub> ( $C_{in} = 0.89 \text{ mg/m}^3$ ,  $\nu = 0.08 \text{ m/s}$ ).

#### 3. Results and discussion

#### 3.1. Characteristics of the MDELs

The UV radiation emitted by MDELs was detected by an Acton VM-505 VUV monochrometer. The main UV wavelengths of MDEL-Br<sub>2</sub> were 207 nm (KrBr<sup>\*</sup>  $\rightarrow$  Kr+Br+ $h\nu$ ) and 291 nm (Br<sub>2</sub><sup>\*</sup>  $\rightarrow$  Br+Br+ $h\nu$ ) [18]. MDEL-I<sub>2</sub> emitted six atomic I emission lines at 178.3, 180.1, 183 (5p<sup>4</sup>6s(<sup>4</sup>P<sub>5/2</sub>)-5p<sup>5</sup>(<sup>2</sup>P<sub>3/2</sub>)), 184.4 (5p<sup>4</sup>6s(<sup>4</sup>P<sub>3/2</sub>)-5p<sup>5</sup>(<sup>2</sup>P<sub>1/2</sub>)), 187.6 (5p<sup>4</sup>6s(<sup>4</sup>P<sub>1/2</sub>)-5p<sup>5</sup>(<sup>2</sup>P<sub>1/2</sub>)), 206.2 nm (5p<sup>4</sup>6s(<sup>2</sup>P<sub>3/2</sub>)-5p<sup>5</sup>(<sup>2</sup>P<sub>1/2</sub>)), and one I<sub>2</sub><sup>\*</sup> emission line at 342 nm [19,20]. We aimed to obtain 207 nm from MDEL-Br<sub>2</sub> and 206.2 nm from MDEL-I<sub>2</sub> in the range of 190–210 nm. In the preliminary experiment, light intensity of MDEL-Br<sub>2</sub> is found higher than MDEL-I<sub>2</sub>.

Prior to examining the parameters of photodecomposition, a preliminary experiment was conducted to determine which between MDEL-Br<sub>2</sub> and MDEL-I<sub>2</sub> is better for the degradation of butyl acetate. Fig. 2 shows the removal efficiency using MDEL-Br<sub>2</sub> and MDEL-I<sub>2</sub> separately under the same experimental conditions:  $C_{in} = 0.89 \text{ mg/m}^3$ , v = 0.08 m/s. In Fig. 2, under each equivalent MW input power, the removal efficiency of butyl acetate by MDEL-Br<sub>2</sub> is constantly higher than that by MDEL-I<sub>2</sub>. The results meet with the spectrogram that light intensity of MDEL-Br<sub>2</sub> is higher than MDEL-I<sub>2</sub>. Therefore, MDEL-Br<sub>2</sub> was used in the subsequent experiments.

#### 3.2. Factors affecting butyl acetate removal efficiency

#### 3.2.1. Effects of MW input power

The maximum MW input power is 200 W. MDELs are not lit until the input power is high enough. MDEL-Br<sub>2</sub> needs 152 W, whereas MDEL-I<sub>2</sub> needs 124 W. As shown in Fig. 3, both removal efficiency and energy yield increase quickly with an increase in input power. The higher the power is, the higher is the UV light intensity, and the better is the decomposition of butyl acetate. When the input power is between 152 and 200 W, a higher removal efficiency and energy efficiency result. Therefore, 200 W as the best input power was used in the subsequent experiments.

#### 3.2.2. Effects of butyl acetate inlet concentration

The impact of inlet concentration on removal efficiency and energy yield was detected under two gas flow velocities: 0.1 and 0.025 m/s. In theory, with an increase in inlet concentration, the removal efficiency decreases because the photon energy produced



**Fig. 3.** Effect of input power on  $\eta$  and Ey ( $C_{in} = 1.50 \text{ mg/m}^3$ ,  $\nu = 0.05 \text{ m/s}$ ).

by MDELs is limited, and the photon energy obtained by unit butyl acetate is reduced. However, as seen in Fig. 4, the inlet concentration has little effect on the removal concentration. All of the inlet concentration data are no more than 20 mg/m<sup>3</sup>, indicating that low-concentration gaseous butyl acetate was the main focus. The results imply that under a low inlet concentration, MDELs are influenced slightly by inlet concentration. Due to the limitation of the gas generation method, the simulation of high-concentration under a high inlet concentration was not tested. On the other hand, an increase in inlet concentration obviously results in a sharp increase in the energy efficiency, as illustrated in Fig. 4.



**Fig. 4.** Effect of  $C_{in}$  on  $\eta$  and Ey at (a) input power=200 W, v=0.1 m/s; (b) input power=200 W, v=0.025 m/s.



**Fig. 5.** Effect of gas residence time on  $\eta$  and Ey (input power=200W,  $C_{in} = 1.50 \text{ mg/m}^3$ ).

#### 3.2.3. Effects of gas residence time (gas flow rate)

Gas residence time, which is determined by the gas flow rate, is the duration of the entry and exit of exhaust gases to the reactor. The results in Fig. 5 were achieved at an input power of 200 W, C<sub>in</sub> of 1.50 mg/m<sup>3</sup>. As presented, with the residence time increased from 0.61 s to 13.32 s, the removal efficiency of butyl acetate increased dramatically from 0% to 78.55%, which in turn led to an increase in both collision time and collision possibility. On the other hand, Ey increased from 0 to 10.23 mg/kWh when the residence time increased from 0.61 s to 3.33 s, but it started to decrease as the residence time further increased. Therefore, gas residence time is a key point of photolysis in the experiments. Generally, a longer residence time results in a higher removal efficiency. However, if the residence time is too long, the flow rate of exhaust gases dealt with in unit time decreases, which results in a low energy efficiency and high energy cost. Therefore, when MDELs are used, the residence time comprehensively takes into account the inlet concentration, volume of degradation, capacity of devices, and admitted costs.

#### 3.3. Impact of additional chemicals on removal efficiency

Butyl acetate is a kind of ester, so we hypothesize that the reaction mechanism of butyl acetate under MDELs is similar to the general hydrolysis reaction, and alkali and H<sub>2</sub>O can accelerate the reaction. Therefore, water vapor and ammonia were selected for separate addition to gaseous butyl acetate in order to ascertain if they can enhance removal efficiency. Water vapor was produced from compressed air that passed through deionized water and was



**Fig. 6.** Effect of additional chemicals on  $\eta$  (input power=200 W,  $C_{in}$  = 3.66 mg/m<sup>3</sup>, v = 0.15 m/s).

introduced into the reactor, whereas ammonia was produced from compressed air that passed through concentrated NH<sub>3</sub>·H<sub>2</sub>O.

After the addition of either water vapor or ammonia, however, the removal efficiency of butyl acetate was not enhanced. Instead, it decreased intermittently, as shown in Fig. 6. For water vapor, it can absorb some microwave, which affects the luminous efficiency and removal efficiency. For ammonia, due to the complexation between Cu (the material of resonant cavity) and NH<sub>3</sub>, the effect of ammonia is weakened. To some extent, the wear and tear of the resonant cavity caused by the complexation disturbs the convergence of microwave. In addition, both water vapor and ammonia possibly have little effect on the reaction, so the removal efficiency was not enhanced.

#### 3.4. Comparison between built-in MDELs and external MDELs

The devices of external MDELs are shown in Fig. 7 [16]. Most of the quartz light tubes are outside the resonant cavity, so luminous efficiency is affected. In our previous study, MDEL-Br<sub>2</sub> was used. However, MDEL-Br<sub>2</sub> was the most difficult to stimulate among the four MDELs. The length of the electrical discharge arc stretching to the outside of the resonant cavity was merely about 3 cm [21], which was the main reason we did not use MDEL-Br<sub>2</sub> to degrade H<sub>2</sub>S and C<sub>2</sub>S [16,17]. When built-in MDELs are adopted, however, not only can MDEL-Br<sub>2</sub> be successfully illumined, but its capability to decompose pollutants also surpasses that of MDEL-I<sub>2</sub>. However,



Fig. 7. Schematic of the external MDELs reaction system.

#### Table 1

Comparison of	f the energy	vields of built-in	MDFIs and	external MDFLs
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Built-in MDELs			External MDELs		
Gas residence time (s)	Inlet [butyl acetate] (mg/m <sup>3</sup> )	Ey (mg/kWh)	Gas residence time (s)	Inlet [H <sub>2</sub> S] (mg/m <sup>3</sup> )	Ey (mg/kW h)
3.33	1.5	10.3	3.6	1.2	5.1
	2.5	17.8		2.7	6.3
	5.8	39.0		5.2	8.1
	13.8	92.6		14.0	8.3

the composition of filling gases is not changed  $[Br_2 (0.5 \text{ Torr}) \text{ and } Kr (1 \text{ Torr})].$ 

The energy yields of built-in MDELs and external MDELs are compared in Table 1. Under a similar gas residence time, all energy yields under the similar inlet concentration of built-in MDELs are larger than those of external MDELs. Although the structure of MDELs and the pollutants (butyl acetate and H<sub>2</sub>S) used are different, built-in MDELs have larger energy yields when degrading butyl acetate which is more difficult to manage. Nonetheless, further studies using the same pollutants are needed to examine the difference in energy yields.

#### 3.5. Mechanism of the photolysis of butyl acetate

PDA UV-vis detector was employed to examine butyl acetate, and the spectrum is presented in Fig. 8. Butyl acetate does not absorb UV light around 254 nm, so the general UV 254 nm cannot decompose butyl acetate theoretically. On the other hand, the absorbance of butyl acetate around 206.2–207 nm is considerable, which is the premise of photolysis.

Products and byproducts are analyzed by GC-MS together with IC. Acetic acid (CH<sub>3</sub>COOH) was detected by GC–MS, whereas CH<sub>3</sub>COO<sup>-</sup> was detected by IC. Therefore, CH<sub>3</sub>COOH was confirmed as one of the products or byproducts (Figs. 9 and 10). The wavelength of MW is too long to have enough energy, so MW cannot break the chemical bonds of butyl acetate and produce radicals. Some studies have reported that pure MW, in a blank experiment, cannot degrade pollutants [9,22]. Therefore, butyl acetate is mostly photo-degraded by UV 206.2-207 nm. MW radiation may also promote the effect of the photolysis of pollutants, which is called the synergy effect [23,24]. If the synergy effect exists, builtin MDELs are superior to external MDELs whose synergy effects can then be ignored. However, due to the particularity of UV 206.2-207 nm, the production of UV lights without MW is difficult, so UV 206.2-207 nm without MW was not simulated in our experiments. The pathways of degradation are presumed to be as follows:



Fig. 8. UV-vis spectrum of butyl acetate by PDA.





Butyl acetate transits to the excitation state after irradiation by UV light:

 $CH_3COOC_4H_9 + hv \rightarrow CH_3COOC_4H_9^*$ 

The energy of UV 206.2–207 nm is about 5.77 eV, whereas the chemical bond of O–O is 5.17 eV, and that of H<sub>2</sub>O's first O–H is 5.21 eV, which are less than 5.77 eV. There is little H<sub>2</sub>O in the air, so •OH is produced through the following processes:

$$O_2 + hv \rightarrow 20$$

$$H_2O + hv \rightarrow \bullet OH + H$$

$$H + O_2 \rightarrow HO_2^{\bullet}$$

 $H_2O + O \rightarrow 2^{\bullet}OH$ 

Butyl acetate in the excitation state breaks down because of its unsteadiness, and it further reacts with •OH:

$$CH_3COOC_4H_9^* + OH \rightarrow CH_3COO^- + C_2H_5OH$$



Fig. 10. The chromatogram chart of IC analysis of degradation products.

 $CH_3COO^-$  and  $C_2H_5OH$  can react with •OH, which will create micro molecules such as  $CO_2$  and  $H_2O$ . Meanwhile, the related mechanism of photodegradation needs further research.

#### 4. Conclusions

This study presents a detailed description of butyl acetate removal with MDELs, including the operational parameters involved, pathways discussion, additional chemicals used, and a rough comparison of built-in MDELs and external MDELs. Microwave input power, inlet concentration, gas residence time, and filling materials are found to have substantial effects on the removal efficiency of butyl acetate and energy yield. The results obtained in this study showed that when the gas flow velocity is 0.025 m/s, and the inlet concentration is  $1.5 \text{ mg/m}^3$ , the removal efficiency of butyl acetate of built-in microwave discharge electrodeless lamps decomposition method is sufficient. The removal efficiency of the butyl acetate was over 78%. The advantage of the built-in-type MDELs method is connected with incorporation of microwave and UV for the decomposition of butyl acetate, this style can attain high luminous efficiency and high energy efficiency. With this new system function by built-in MDELs, the removal efficiency of the butyl acetate may obtain the maximum enhancement.

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