Decomposition of CCl₄ and CHCl₃ on gliding arc plasma

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Abstract: Decomposition of chlorinated hydrocarbons, CCl_4 and $CHCl_3$, in gliding plasma was examined. The effects of initial concentrations, total gas flow rates, and power consumption have been investigated. The conversion result was relatively high. It reached 80% for CCl_4 and 97% for $CHCl_3$. Using atmospheric air as the carrier gas, the plasma reaction occurred at exothermic reaction and the main products were CO_2, CO, and Cl_2. Transformation into CCl_4 was also detected for $CHCl_3$ decomposition reaction. The conversion of CCl_4 and $CHCl_3$ were increased with the increasing applied frequency and decreasing total gas flow rate.
Keywords: plasma; gliding arc; CCl_4; $CHCl_3$; decomposition reaction

Introduction

Chlorinated volatile organic compounds(CVOCs) were released to the atmosphere as flue gases from various industrial processes. Chlorinated hydrocarbons have been widely accepted as solvents and chemical intermediates in the manufacture of herbicides and plastics(Othmer, 1978). Unfortunately, emissions of these compounds were harmful to the environment, toxic (Meek et al., 2002; IARC, 1987; USAEPA, 2002), and much effort has been done to reduce it.

The current and common technology for eliminating these compounds was applying a high temperature (>800℃) incineration (Lou and Chang, 1997). The idea of this method was reacting the influent of CVOCs directly with combusted air. Although this method was applicable and simple, some problems that existed in the process have been scientifically reported. When the combustion occurred in un-perfect reaction condition (incomplete combustion), reaction tended to produce a large amount of complex chlorinated products (Taylor and Dellinger, 1988), which have possibility to be more toxic than the original influent itself.

Catalytic oxidation which was employing metal and metal oxide catalyst has been investigated (Alberici and Jardim, 1997; Khaleel and Dellinger, 2002). It has been reported that by applying this method, although the conversion and the product selectivity were very good, but the catalyst became easily deactivated by impurities and solid product. However, this method also required higher temperature to achieve the activate catalyst condition and higher reaction rate. Another limitation which became the barrier to apply this method in industrial level was

caused by the small amount of input flow rate.

In order to overcome those problems, many studies were carried out on the application of new technologies. Plasma-assisted technology was one of the emerging and effective technologies for destroying CVOCs. Plasma-assisted technology, such as electron beam (Prager et al., 1995), radio frequency (RF) plasma(Lee et al., 1996; Föglein et al., 2003), surface discharge reactors(Oda et al., 2002), dielectric barrier discharge reactors (Tonkyn et al., 1996), pulsed discharge reactors (Yamamoto et al., 1992), and capillary- tube type discharge reactors (Kohno et al., 1998), have been studied and developed. In this study, gliding arc plasma was used to decompose chloroform ($CHCl_3$) and carbon tetrachloride(CCl_4). Compared to the previous plasma devices, gliding arc plasma has a great chance to be utilized for industrial chemical applications. This method produced high flame temperature and power to destruct the toxic input material(Czernichowski, 1994). One advantage of this system was it could handle very high input flow rate.

1 Experimental setup

The schematic diagram of experimental setup is shown in Fig.1. CCl_4, $CHCl_3$ and atmospheric air have been used as source gas. Details of each part of the system are described in the next section.

1.1 Plasma reactor and applied power system

The reactor was made from a quartz-glass tube of inner diameter 45 mm and length 300 mm. The upper part and bottom of the reactor supplied with a teflon seal comprised two electrodes made of stainless steel. The length of the electrodes was 150 mm. The separation of the electrodes in the narrowest section was only 1.5 mm. The gas mixture was introduced between the electrodes through a capillary of inner
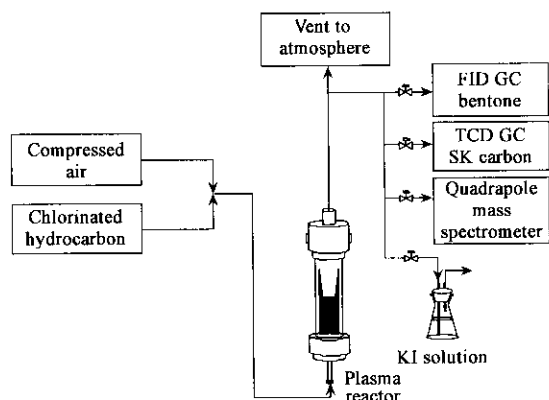
Fig.1   Schematic diagram of experimental setup

diameter 0.8 mm. A thermocouple, located 10 cm above the electrode, has been provided to measure the outlet gas temperature. A high frequency AC power supply(auto electric, A1831) with a maximum voltage 10 kV and a maximum ampere 100 mA was connected to the gliding arc electrode to generate plasma. Fig.2 shows typical waveforms of voltage and discharge current used in this experiment.
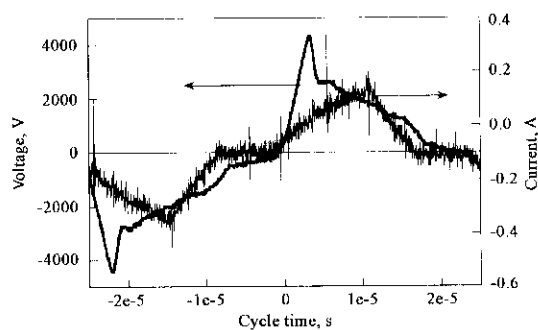

Fig.2   Voltage and current profile

1.2 Input gas

Chlorinated hydrocarbons that were used as the starting material are: (1) chloroform: $CHCl_3$, molecular weight 119.38, purity 99.0% , purchased from Junsei Chemical Co., Ltd., concentration 1, 3, 5, 8% v/v; (2) carbon tetrachloride: CCl_4, molecular weight 153.82, purity 99.5%, purchased from Kanto Chemical Co., Inc., concentration 1, 3, 5, 8% v/v.

Atmospheric air was used as the carrier gas and controlled by a mass flow controller(Tylan, FC-280S). The total mixed gas (air + chlorinated compounds) flow rates were 180, 240, and 300 L/h. The composition of the outlet mixture was analyzed before and after the plasma operation.

1.3 Measurement system

The outlet gas composition was examined by quadrupole mass spectroscopy (Balzers, QMS 200) supported by analyzer Quadstar 421 software. This

software was used for the qualitative and quantitative analysis of the reactants and products. Two gas chromatographers (GCs) have been used to obtain the quantitative amount of products. The contents of CCl_4 and $CHCl_3$ in the gas mixture before and after the reaction were determined by GC-FID (YoungLin M600D, Column: Bentone). And for measuring CO and CO_2 concentration in the outlet gas mixture, a GC-TCD (YoungLin M600D, Column: SK Carbon) was used. Chlorine gas(Cl_2) was analyzed by bubbling the reacted gas through 0.05 mol/L aqueous KI during measured-experiment time and followed by iodometric titration 0.05 mol/L Na_2SO_3.

To evaluate the performance of system, selectivity and conversion calculation were used and formulated as for decomposition of CCl_4:

$$\text{Selectivity of } Cl_2 = \frac{M_{Cl_2} \text{ formed}}{2\,M_{CCl_4}} \times 100\% \tag{1}$$

$$\text{Selectivity of } CO = \frac{M_{CO} \text{ formed}}{M_{CCl_4} \text{ converted}} \times 100\% \tag{2}$$

$$\text{Selectivity of } CO_2 = \frac{M_{CO_2} \text{ formed}}{M_{CCl_4} \text{ converted}} \times 100\% \tag{3}$$

$$\text{Conversion of } CCl_4 = \frac{M_{CCl_4} \text{ consumed}}{M_{CCl_4} \text{ introduced}} \times 100\% \tag{4}$$

for decomposition of $CHCl_3$:

$$\text{Selectivity of } Cl_2 = \frac{2\,M_{Cl_2} \text{ formed}}{3\,M_{CHCl_3} \text{ converted}} \times 100\% \tag{5}$$

$$\text{Selectivity of } CO = \frac{M_{CO} \text{ formed}}{M_{CHCl_3} \text{ converted}} \times 100\% \tag{6}$$

$$\text{Selectivity of } CO_2 = \frac{M_{CO_2} \text{ formed}}{M_{CHCl_3} \text{ converted}} \times 100\% \tag{7}$$

$$\text{Selectivity of } CCl_4 = \frac{M_{CCl_4} \text{ formed}}{M_{CHCl_3} \text{ converted}} \times 100\% \tag{8}$$

$$\text{Conversion of } CHCl_3 = \frac{M_{CHCl_3} \text{ consumed}}{M_{CHCl_3} \text{ introduced}} \times 100\% \tag{9}$$

Those parameters were used to study the effect of initial chloromethane concentrations, total gas flow rates, and power frequency. The discharge power was calculated as a product of voltage and current by oscilloscope(Agilent 54641A), defined as:

Discharge power =

$$\int (V(t) \times I(t)) dt \times \text{frequency Watt} \tag{10}$$

In this study, each data of experiment was taken after 30 min from the start of gliding plasma operation refers to the stability of bulk gas outlet temperature measured by thermocouple.

2 Results and discussion

2.1 Effect of initial concentration and total gas flow rate

Fig.3 shows the conversion trend of CCl_4 and

CHCl₃ in various values of initial concentrations and total gas flow rates. The total conversion gradually decreased when the initial concentration increased. The conversion also decreased with increasing total gas flow rate. The maximum conversion of CCl₄ reached 80% at 1% concentration and total air flow rate at 180 L/h. At similar condition, CHCl₃ conversion reached a higher value, 97%.

Although the total conversion was decreased, the removal rate efficiency has increased with increasing initial concentrations. Existing Cl^0 that was produced by plasma reaction from CCl₄ or CHCl₃ could be stated the key reason to explain the phenomena. Cl^0 could initiate the decomposition reaction and give positive effect on the decomposition process.

Different compounds, initial concentrations, and total gas flow rates also produced different values of consuming discharge power. Chemical stability of compound when it was treated by gliding plasma has an important role in destruction reaction. Table 1 shows that to destruct CCl₄, it requires more energy compared to CHCl₃. The chemical bond and structure stability of CCl₄ was stronger than CHCl₃ (Othmer, 1978). Stronger chemical bond of compounds spent more energy to initiate the arc plasma than less strong compound. Moreover, this difficulty to destruct the bond will affect the conversion values (comparison between CCl₄ and CHCl₃ conversion in Fig.3). This result has the same tendency when chlorinated hydrocarbons were treated by oxidation combustion reaction(Taylor and Dellinger, 1988).

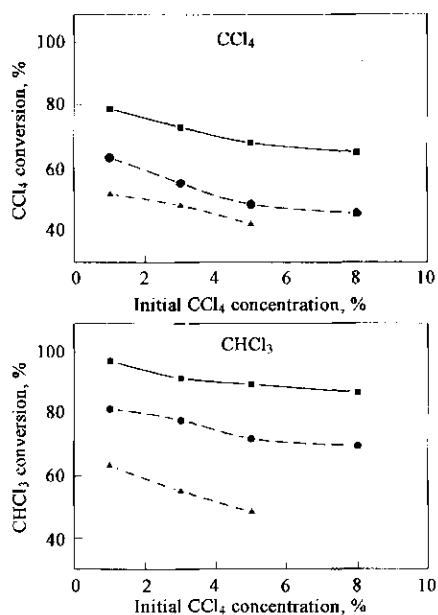Total gas flow rate also gave significant effect on

Fig.3   Influence of initial concentration of injected chlorinated hydrocarbons and total gas flow rate on its conversion
■ 180 L/h; ● 240 L/h; ▲ 300 L/h

power discharged consumption. With increasing total gas flow rate, the power consumption was decreased. Physical study related to this phenomenon has been observed and it has a correlation to the Paschen's law (Cobine, 1958).

Based on QMS spectra and GC analysis, the gaseous products were dominated by CO, CO₂, and Cl₂. The number of other C-Cl compounds was relatively small. In case of CCl₄, the selectivity of final product to Cl₂ gas was high. It could reach from 42%, up to 78.9%. Although it was not as high as CCl₄,

Table 1   Effect of initial concentration and total gas flow rate on product distribution of CCl₄ and CHCl₃ decomposition

Concentration, % v/v	Flowrate, L/h	CCl₄			CHCl₃			
		Power, W	Selectivity		Power, W	Selectivity		
			CO+CO₂	Cl₂		CO+CO₂	CCl₃	Cl₂
1	180	242.6	48.7	78.9	234.5	31.1	6.4	33.0
3	180	240.5	32.4	73.0	232.1	40.3	9.7	34.9
5	180	239.3	28.9	68.0	226.8	42.5	10.9	33.8
8	180	229.6	25.4	65.0	212.8	48.6	11.8	
1	240	236.8	48.0	63.3	222.1	31.1	9.4	42.7
3	240	234.2	49.0	55.1	216.6	34.4	10.0	38.8
5	240	229.4	41.0	48.4	205.1	41.0	10.2	48.3
8	240	219.6	41.7	45.6	175.0	43.8	11.8	47.4
1	300	227.7	26.7	51.6	220.0	22.2	5.3	50.5
3	300	225.6	25.2	48.0	212.6	27.4	7.9	43.3
5	300	218.2	18.2	42.0	197.4	24.6	10.0	47.5

product probability of Cl_2 from $CHCl_3$ decomposition was also relatively high, in the range of 33% to 50.5%. Increasing CCl_4 initial concentration has decreased the product selectivity of $(CO+CO_2)$, and Cl_2. It means the result distribution would be broadly varied to produce other C-Cl compounds. This phenomenon did not occur on $CHCl_3$ reaction. The selectivity of Cl_2 gas was relatively constant while the selectivity of $(CO+CO_2)$ was slightly increased.

In the case of CO and CO_2 production, the selectivity of those compounds has the same trend as Cl_2, especially for CCl_4 decomposition. The idea of free-C vacancy was possibly used to explain this experiment result. When CCl_4 was destructed into Cl_2, the reaction produced free-C (C^0, radical) as an intermediate species. It created chance for free-C radical to collide with another compounds and high possibility to collide with oxygen, the concentration about 20% in the air flow, to produce CO or CO_2. In term of stability, CO and CO_2 have a stable structure and production of those CO and CO_2 were higher compared to other compounds, such as C-O-Cl compounds. Based on the calculation of QMS spectra, the concentration of oxygen(O_2) in the product stream (after plasma process) was 5—10% lower than concentration in inlet stream (before plasma process). This calculation result could directly support the previous idea that the losing oxygen was caused by reaction and consumed into CO and CO_2. Ratio of produced CO_2 to CO at the final product stream was around from 1/10 up to 1/5.

Decomposition of $CHCl_3$ also produced CCl_4 as one of the products. The selectivity was not exceeded more than 12%. During the experiment, some other trace compounds were also detected, such as $COCl_2$ and HCl. Liquid product was also produced and should be counted as the remaining C and Cl species.

2.2 Effect of frequency

As mentioned above, supplied discharge power to the plasma system has an important role on the decomposition reacfion. This factor was also important to draw the products distribution. In the plasma reaction experiment, voltage, current, and frequency could give great influence on the product distribution value. Among these variables, only power frequency could be adjusted manually in this experiment. Voltage and current were automatic fixed into a specific value after initial breakdown of gliding arc.

The effect of power frequency is prescnted in Fig. 4. The maximum power frequency was 20 kHz. As shown in Fig.4, the conversion increased with the increasing frequency. For CCl_4 in Fig.4a, the rate of conversion achieved 4% higher per 1 kHz increment of frequency, and slightly lower for $CHCl_3$ in Fig.4b. Increasing frequency leads to incrcase the supplied discharge power. Fig.4c shows the increasing discharge power as the effect of increasing power frequency. Increasing frequency gave contribution on producing different AC wave formation. Higher frequency produced more number of peaks per one cycle and supplied more energy to the system. The change of this power wave form pattern due different applied power frequency can be seen in Fig.5.

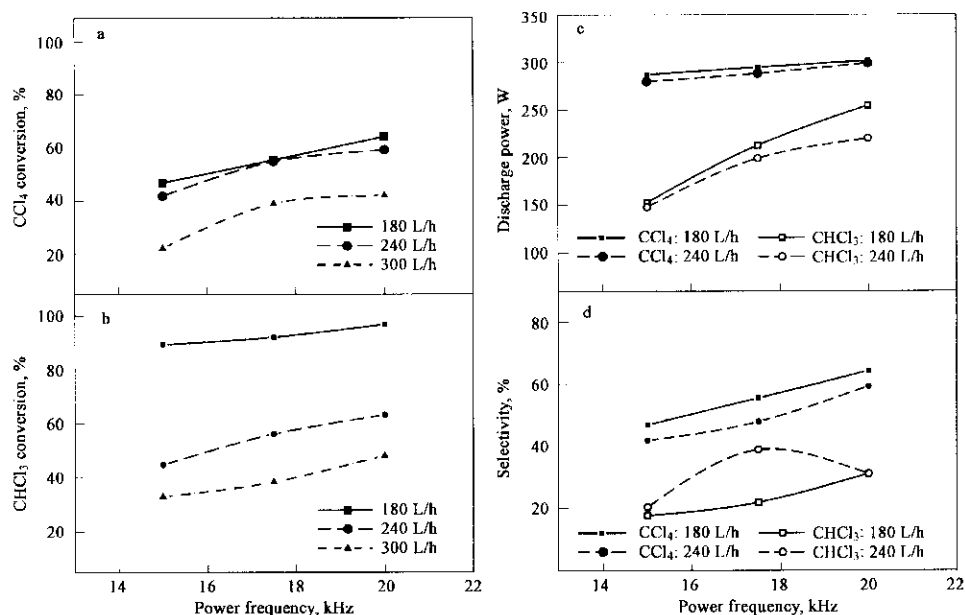Fig.4d shows the different value of frequency or

Fig.4   Effect of applied input power frequency

a. CCl_4 conversion; b. $CHCl_3$ conversion; c. discharge power; d. Cl_2 product selectivity; all data was takcn at 1% of initial both CCl_4 and $CHCl_3$
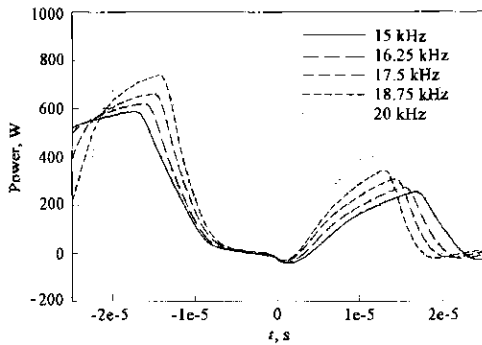
Fig.5   Effect of applied different power frequency on powerprofile wave form

Data was taken at 5% of CHCl₃ and total gas flow rate 180 L/h

power discharge also gives different value of Cl_2 gas production although the initial concentration and flow rate were same. It means that power frequency or power discharge has a significant role on governing the way of reaction. With the increasing power frequency, the selectivity of Cl_2 was getting increased. It could be proposed that in the higher energy field, the plasma reaction tended to produce Cl_2 gas as the final product.

2.3 QMS spectroscopy

A QMS spectroscopy of the conversion of CCl_4 is shown in Fig.6. Fig.6a describes the condition of components in the 180 L/h feed stream containing atmospheric air with 1% of CCl_4. The baseline spectra of CCl_4 were 83/85 (CCl_2^-), and 117 (CCl_3^+). CO_2 has spectra at 44 (CO_2^+) and CO spectra was collide with minor N_2 spectra at 29(CO^+/N_2^+). Major N_2 spectra was located at 28 (N_2^+). The concentration of CO in the air was quite small, CO compound could be negligible in this spectroscopy. When the plasma was applied in
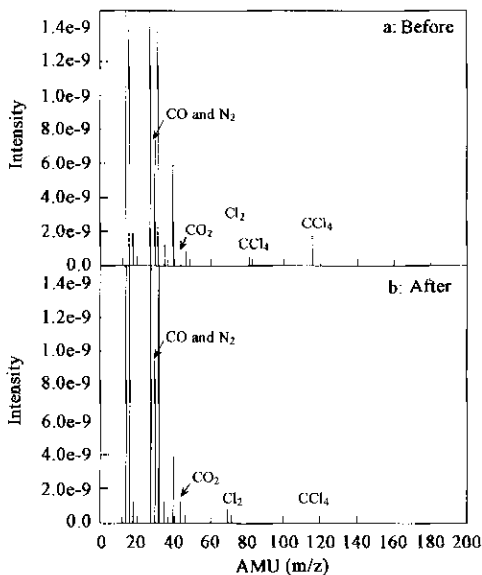

Fig.6   QMS spectrogram of CCl₄ decomposition

Before (a) and after (b) gliding plasma treatment; applied frequency 20 kHz; 1% of CCl₄; total gas flow rate 180 L/h

Fig.6b, the peak intensity of 83/85 disappeared together with decreasing intensity of spectra 117 and appared a new peak, 71, which is known as $Cl_2(Cl_2^+)$. Intensity of CO_2 was increased also but the difference was very small. High increment occurred with spectra 28. Because N_2 was relatively stable gas and no source of N compound to produce N_2, it could be said that the increasing intensity of spectra 28 was due to production of CO by plasma.

The mass spectroscopy of CHCl₃ before and after plasma reaction is presented in Fig.7. Fig.7a shows the components in the 240 L/h feed stream containing compressed air mixed with 1% of $CHCl_3$. The baseline spectra of $CHCl_3$ were 82/84/86 (CCl_2^+). CO_2 has spectra at 44(CO_2^+) but it was collided with major N_2O spectra(N_2O^+), which has possibility to exist in the gas product. CO was at 29 (CO^+) but it was also collided with minor N_2 spectra 29 (N_2^-). It made difficult to distinguish them using QMS. In this experiment, CO and CO_2 were analyzed by gas chromatography. When the plasma was applied, Fig.7b, the peak intensity of 83/84/86 got decreased and formed some new peaks: 63/65, which is known as $COCl_2(COCl^+)$, 69/71 as Cl_2 (Cl_2^+), 117 as $CCl_4(CCl_3^-)$.
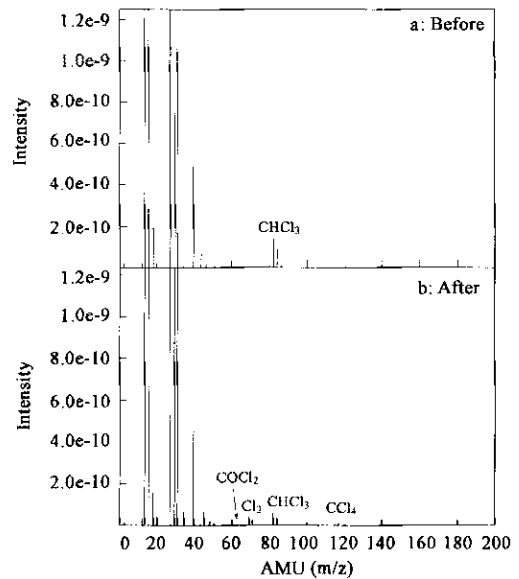

Fig.7   QMS spectrogram of CHCl₃ decomposition

Before (a) and after (b) gliding plasma treatment; applied frequency 20 kHz; 1% of CHCl₃; total gas flow rate 180 L/h
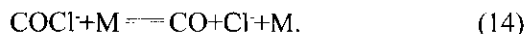
2.4   Reaction pathway

The kinetic reaction of CO, CO_2, and Cl_2 formation which were produced from chlorinated hydrocarbons has been studied (Prager et al., 1995; Lou and Chang, 1997; Koch et al., 1995; Nichipor et al., 2000; Chang and Senkan, 1998; Feiyan et al.,
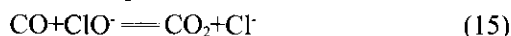
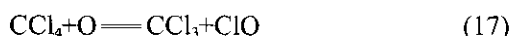2002; Wu and Won, 2000). The major pathways responsible for the formation of Cl_2 were:

$$COCl_2 + Cl^- = COCl^- + Cl_2 \quad (11)$$
$$CHCl_3 + Cl^- = CHCl_2 + Cl_2 \quad (12)$$
$$COCl^- + Cl^- = CO + Cl_2 \quad (13)$$
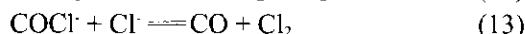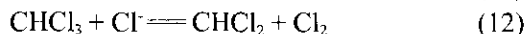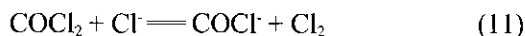
However, the third reaction (Eq.13) was also one of the primary reaction paths for the formation of CO. Other one was:

$$COCl^- + M = CO + Cl^- + M. \quad (14)$$

It was seen that CO formation was initially created through $COCl_2$. The major reaction path for the formation of CO_2 was come from CO:

$$CO + ClO^- = CO_2 + Cl^- \quad (15)$$
$$CO + O + M = CO_2 + M \quad (16)$$

in which ClO forms via:

$$CCl_4 + O = CCl_3 + ClO \quad (17)$$
$$CCl_3 + O_2 = COCl_2 + ClO \quad (18)$$

It means oxygen and oxygen radical have significantly contribution to held the reaction. The production of O radical came from dissociation reaction(Penetrante *et al.*, 1995):

$$e + O_2 = e + O(^3P) + O(^3P) \quad (19)$$
$$e + O_2 = e + O(^3P) + O(^1D) \quad (20)$$
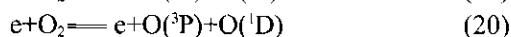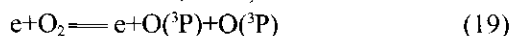
Cl radical in this formula can be formed from:

$$CCl_4 = CCl_3 + Cl \quad (21)$$
$$CCl_3 + O = COCl_2 + Cl \quad (22)$$
$$CCl_3 + Cl_2 = CCl_4 + Cl \quad (23)$$

Reverse reaction could be happened on formula 21 as the main path way to produce CCl_4 in decomposition of $CHCl_3$,

$$CCl_3 + Cl = CCl_4 \quad (24)$$

3 Conclusions

The performance of CCl_4 and $CHCl_3$ decomposition on gliding arc plasma at atmospheric pressure, related with initial concentration, total gas flow rate, and input power frequency, was studied. Gliding plasma could generate effective electrons and ions enough to produce the fragment of CCl_4 and $CHCl_3$ molecules. The maximum conversion of CCl_4 was 80% and $CHCl_3$ was 97% for feed gas stream containing 1% of target material and total air flow rate 180 L/h. The major final products were CO, CO_2, and Cl_2. Cl_2 selectivity was relatively high and reached up to 78.9% for CCl_4 and 50.5% for $CHCl_3$. Conversion into CCl_4 was found in case of $CHCl_3$ decomposition, but not exceed than 12%. $COCl_2$ has an important role as intermediate species to produce Cl_2 and CO. Mostly, CO_2 was produced from CO reaction. From these results, it could be concluded that the gliding arc plasma system was very useful for the decomposition of CCl_4 and $CHCl_3$. Development was needed to

remove some traces unwanted compounds, such as: $COCl_2$ in the final product.

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Introduction to the State Key Laboratory of Environmental Aquatic Chemistry

State Key Laboratory of Environmental Aquatic Chemistry (SKLEAC) is a member of the State Key Joint Laboratory of Environmental Simulation and Pollution Control, responsibly constructed by Chinese Academy of Sciences (CAS) with a loan from the World Bank and financial input from CAS. SKLEAC was founded based on several laboratories in the Research Center for Eco-Environmental Sciences (RCEES) in the field of water quality sciences and technologies. SKLEAC is a multi disciplinary basic research center including environmental aquatic chemistry as core, geography, biology, ecology, hydrology, as well as environmental engineering and technology.

The goal of SKLEAC is to promote the basic research on water quality transformation processes in natural environment and new water purification technologies and processes. Specific objectives include: develop modern sciences and high-effective technologies for dealing with water quality problems in natural waters and water treatment processes; to establish applicable environmental water quality simulation system and computer software package; to develop industrialized water and high-effective waste-water treatment technology as well as the unit facilities; to evaluate the safety of water and water environment ecology; to optimize the scientific strategy for water quality management.

SKLEAC has a strong research group, including one academician of CAS, 10 research professors, more than 20 research follows, and about one hundred doctoral and master students. In order to support more research work, SKLEAC established an advanced chemical instrumental analysis platform, which includes more than 40 analytical instruments such as AFM, HPLC-MS, GC-MS, TOC, etc. And five professional technicians are employed to maintain and manage these instruments.

Research topics

1. Water quality identification: qualitative, quantitative and speciation analysis of trace contaminants and particulate in different types of water.
2. Water quality processes: physical, chemical, interfacial, geobiochemical processes of water.
3. Aquatic eco-toxicology: structure and function of aquatic ecosystems and ecotone, ecological and toxicological effects of contaminants, as well as their criterion and pollution control.
4. Water quality simulation: models for distribution and transfer of pollutants, models for environmental behaviors of pollutants, models for water treatment systems.
5. Water purification technology: reactors, facilities, unit operation and industrial processes for water and wastewater treatment, specifically on flocculation, adsorption, chemical and biological oxidation techniques.
6. Water quality management: water quality assessment, criterion, controlling strategy, optimal utilization scheme, as well as restoration, rehabilitation or reclamation technologies.

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