A comparison of carbon tetrachloride decomposition using spark and barrier discharges

Abstract: The decomposition of CCl$_4$ in air was investigated at atmospheric pressure in two discharges. Reactors used to generate electrical discharges were powered by the same electric power supply. In both reactors, nearly 90% conversion of CCl$_4$ was obtained. All chlorine was in the form of Cl$_2$ in the process carried out in the barrier discharge, while in the spark discharge, COCl$_2$ was formed. The conversion of CCl$_4$ to COCl$_2$ ranged from 2 to 12%. NO was formed in both discharges but the NO content in the gas leaving the reactors was 1.7–2.7% for the spark discharge and 0.045–0.06% for the barrier discharge. O$_3$ was produced only in the barrier discharge and its content ranged from 0.1 to 0.2%.

Keywords: plasma, gas cleaning, decomposition, DBD, spark discharge

2 Experimental procedure

CCl$_4$ (extra pure 99%) is used as a model compound for experiments on the destruction of chlorinated organic compounds because of the stability of its structure. Other compounds, for example CHCl$_3$, are more reactive and easier to decompose than CCl$_4$ [8,12,13]. Thus, it is anticipated that methods for efficient CCl$_4$ conversion can also be useful in the destruction of chlorine compounds with less stable structures.

CCl$_4$ decomposition processes were carried out under the followed conditions:
- gas mixture of CCl$_4$ and air (H$_2$O < 10 ppm)
- the CCl$_4$ content was 0.1% vol.,
- total gas flow rate was 10 L h$^{-1}$.

2.1 Reactors

Barrier discharge was generated in a reactor as shown in Fig. 1. The grounded electrode was made from silver paste deposited on the outside of the quartz tube. The
outer diameter of the quartz tube was 54 mm and the wall thickness was 2 mm. A high-voltage electrode was grooved. The distance between the high-voltage electrode and grounded electrode was 1.5 and 5 mm for top and bottom of the grooves respectively.

Spark discharge was generated in a reactor as shown in Fig. 2. Electrodes were made of 2 mm rods of platinum. The distance between electrodes was 12 mm. A case was made of a quartz tube of 9-mm diameter and 40-mm length.

2.2 Electrical measurements

The reactors were powered by one pulsed power supply system of 30-kHz pulse repetition frequency. The frequency, the current, and the voltage waveforms were recorded by using oscilloscope Tektronix TDS 3052 with a Tektronix P6015A voltage probe and a Tektronix TCP 312 current probe with a Tektronix TCP A300 amplifier.

The power of spark discharge was calculated according to the formula:

$$P = f \int I(t)U(t)d(t)$$  \hspace{1cm} (1)

$P$ – power, W
$f$ – frequency, Hz
$I$ – current, A
$U$ – voltage, V
$t$ – time of the pulse start and end, s

The Lissajous-figure method was used to measure the discharge power of barrier discharge:

$$P = f \cdot C \cdot S$$  \hspace{1cm} (2)

$C$ – capacitance of the measuring capacitor, 20 nF
$S$ – enveloped area of Lissajous-figure

The specific energy was calculated according to the formula:

$$SE = \frac{3.6 \cdot P}{G}$$  \hspace{1cm} (3)

$SE$ – specific energy, kJ g\(^{-1}\) of gas
$G$ – total gas (air and VOCs) flow rate at the inlet, g h\(^{-1}\)
2.3 Gas analysis

The content of CCl₄ was analyzed using a Hewlett-Packard HP 6890 with an FID detector and packed column with 5% Fluorocol on 60/80 Carbopack B. Chromatographic measurement error was less than 5%. This method allowed for determination of the content of other organic compounds containing chlorine.

Cl₂ and COCl₂ were analyzed using titration analysis. Gas was periodically passed by two bubblers with solution of KI (10 mmol) in water (100 mL). In this solution Cl₂ reacted with KI to form I₂ and KCl. COCl₂ reacted with H₂O to form HCl and CO₂. The solution was titrated with a solution of Na₂S₂O₃ (2.5 mmol) in water (50 mL) to determine the amount of I₂ and with a solution of NaOH (5 mmol) in water (50 mL) to determine the amount of HCl. Titration analysis error was less than 3%.

The content of O₃ was determined using a BMT 961TC ozone analyzer.

The content of NO was determined using a URAS 10P industrial photometer.

The CCl₄ conversions were calculated according to formulas:

\[ X = \frac{W_{\text{in}}[\text{CCl}_4] - W_{\text{out}}[\text{CCl}_4]}{W_{\text{in}}[\text{CCl}_4]} \times 100 \]  

\[ X_{\text{COCl}_2} = \frac{W[\text{COCl}_2]}{2 \times W[\text{CCl}_4]} \times 100 \]  

\[ X_{\text{Cl}_2} = \frac{W[\text{Cl}_2]}{2 \times W[\text{CCl}_4]} \times 100 \]

3 Results and Discussion

3.1 Electrical characteristics

The waveforms of the discharge current and voltage for each power supply system are shown in Figs. 3 and 4. These waveforms are different, despite the fact that they are powered by the same power supply. Changing the current-voltage characteristics was due to the differences in properties of the reactors.

Differences in voltage and current waveforms for the two types of reactors are strongly related to the construction of the reactors. Barrier discharge reactor in the general case can be considered a cylindrical capacitor, whose capacity can be calculated by the equation:

\[ C = \frac{2\pi\varepsilon\varepsilon_0 d}{\ln\frac{R}{r}} \]  

\[ C - \text{capacity, F} \]
\[ \varepsilon - \text{relative dielectric permittivity (dielectric constant)} \]
\[ \varepsilon_0 - \text{permittivity of vacuum, } 8.854187 \times 10^{-12} \text{ F m}^{-1} \]
\[ d - \text{length of the electrodes, m} \]
\[ R - \text{radius of the outer electrode, m} \]
\[ r - \text{radius of the inner electrode, m} \]

Voltage and current waveforms for the circuit of the barrier discharge reactor are similar to the charging and discharging of the capacitor in a capacitive circuit.

Capacity of the spark reactor can be calculated by the equation:

\[ C = \frac{\varepsilon\varepsilon_0 S}{D} \]  

\[ S - \text{surface of the electrode top, m}^2 \]
\[ D - \text{distance between electrodes, m} \]

The spark reactor system has a low capacitance because the S is very small (~3.14 \times 10^{-6} m²). The spark reactor operated at several times lower voltage than the barrier reactor, because there was no additional dielectric barrier impeding electrical breakdown.

3.2 Mechanism of CCl₄ decomposition

The main product of CCl₄ decomposition was Cl₂ for both discharges (Figs. 5 and 6). This result corresponded with the results obtained in other discharges and other models of reactors [13-16]. A small amount of COCl₂ was formed only in spark discharge (Fig. 5). New organic compounds, like C₂Cl₆ or C₃Cl₄, were not formed. Based on these results and on the literature data, the mechanism of decomposition of CCl₄ in these conditions can be proposed.

CCl₄ decomposition can be initiated by reactions with electrons or radicals of oxygen, chlorine, nitrogen or hydroxide [10,17-20]:

\[ \text{CCl}_4 + e \rightarrow \text{CCl}_3 + \text{Cl} + e \]
\[ \text{CCl}_4 + \text{O} \rightarrow \text{CCl}_3 + \text{ClO} \]
\[ \text{CCl}_4 + \text{Cl} \rightarrow \text{CCl}_3 + \text{Cl}_2 \]
The CCl₃ radical takes part in many reactions [13,21-26], where M is any molecule that exchanges energy:

\[
\text{CCl}_3 + \text{Cl} + (\text{M}) \rightarrow \text{CCl}_4 + (\text{M})
\]

\[
\text{CCl}_3 + \text{Cl}_2 \rightarrow \text{CCl}_4 + \text{Cl}
\]

\[
\text{CCl}_3 + \text{O} \rightarrow \text{COCl}_2 + \text{Cl}
\]

NCl takes part in the following reactions [26]:

\[
\text{NCl} + \text{N} \rightarrow \text{N}_2 + \text{Cl}
\]

\[
\text{NCl} + \text{O} \rightarrow \text{NO} + \text{Cl}
\]

The ClO radical takes part in the following reactions [27-29]:

\[
\text{ClO} + \text{O} \rightarrow \text{O}_2 + \text{Cl}
\]

\[
\text{ClO} + \text{Cl} \rightarrow \text{Cl}_2 + \text{O}
\]

\[
2 \text{ClO} \rightarrow \text{O}_2 + \text{Cl}_2
\]

\[
2 \text{ClO} \rightarrow \text{CO}_2 + \text{Cl}
\]

HClO takes part in the following reactions [30,31]:

\[
\text{HClO} + \text{Cl} \rightarrow \text{Cl}_2 + \text{OH}
\]
HClO + O\(_2\) → ClO\(_2\) + OH

CCl\(_4\) may be formed under high temperature according following reaction [32]:

CCl\(_4\) + CO\(_2\) → 2 COCl\(_2\)

COCl\(_2\) and ClO\(_2\) may be decomposed by reactions with the Cl radical [13,21,33]:

ClO\(_2\)+Cl → Cl\(_2\) + O\(_2\)

COCl\(_2\) + Cl → COCl + Cl\(_2\)

COCl is not a stable compound and decomposes in the following reaction [13,21-23]:

COCl + Cl → CO + Cl\(_2\)

ClCN and NCO may be decomposed in the following reactions [34,35]:

ClCN + O → NCO + Cl

NCO + O → CO + NO

The mechanism described above is shown in Scheme 1.

3.3 Nitrogen monoxide and ozone

The process was carried out in air. The components of air in the conditions of the plasma were reactive reagents which can form nitrogen monoxide and ozone. NO was formed in both discharges (Fig. 7). The content of NO in the gas leaving the reactor was 1.7–2.7% for the spark discharge and 0.045–0.06% for the barrier discharge. O\(_3\) was produced only in the barrier discharge (Fig. 8). Its content ranged from 0.1 to 0.2%. Figs. 7 and 8 show that the NO and O\(_3\) contents increase with the increasing specific energy.

The presence of NO and O\(_3\) in the post-reaction gases is undesirable, and these compounds should be removed from the gas.

Ozone decomposed relatively easy to O\(_2\) at elevated temperatures. To decompose the ozone, an oven operating at 200°C was placed downstream of the reactor. The residence time of the reactants in the oven was 0.5 s. Ozone removal was necessary because its reaction with KI interfered with the titration. NO did not disturb the analysis of gas composition, and therefore it was not removed from the gas. The removal of NO from gases is complicated. Catalytic reaction with ammonia is the most common method for removing NO.

3.4 Spark discharge

High CCl\(_4\) conversion (almost 90%) was reached in the spark discharge (Fig. 5). In the spark reactor, the main
product was Cl₂, but COCl₂ was also produced (Fig. 5). COCl₂ formation is undesirable because it is toxic. Additionally, NO was produced in the spark reactor. NO content after the reaction ranged from 1.7 to 2.7% (Fig. 7). O₃ was not produced in the spark reactor.

The spark discharge consists of individual streamers. Streamer channels have a diameter of ~0.1 mm, and their duration is 300–500 ns [36,37]. The average density of electrons decreases from 10¹⁵ to 10¹¹ cm⁻³ with the duration of streamer [36]. The energy of electrons ranged from 0.1 to 4 eV in the spark discharge [36,38]. The maximum temperature of the gas reaches 5000–6000 K in the streamer channel [36]. Electrons have too low energy to cause dissociation of O₂, for which electrons with energies above 6 eV are needed. However, binding energy of C-Cl is 3.6 eV. This means that the electrons have sufficient energy to cause dissociation of this bond.

High gas temperature in the streamer channel results in the formation of NO and COCl₂ with no O₃. The high gas temperature in the streamer channel causes ionization and thermal dissociation of the molecules. Thermal dissociation and ionization concerns mainly O₂ and N₂, which are found in the largest quantities in the reactor. The equilibrium concentration of oxygen and nitrogen atoms is ~20 and ~15% at 6000 K respectively. These atoms can initiate the decomposition of CCl₄. However, the dominant reaction should be the formation of NO. NO is formed during cooling to ~3000 K. During further cooling to ~1000 K, NO decomposes into O₂ and N₂. At a temperature less than 1000 K, the NO decomposition reaction is inhibited due to the high activation energy of the reaction. As a result of all these processes, NO concentration in the exit gas ignition is high. At high temperatures, ozone does not occur, because its decomposition reactions are faster than the reactions of its formation.

The reaction of COCl₂ formation from CCl₄ and CO₂ is an endothermic reaction; ΔH is 70 kJ mol⁻¹ [32]. Therefore, this reaction will occur at high temperature. Consequently, the conversion of CCl₄ to COCl₂ was high in spark discharge, up to 12% (Fig. 5).

3.5 Barrier discharge

High CCl₄ conversion (almost 90%) was reached in the barrier discharge (Fig. 6). The most important advantage of the barrier reactor was the possibility of running CCl₄ decomposition process in such a way that all the chlorine was present in the Cl₂. This is a significant advantage because Cl₂ can be easily neutralized; for example, it reacts with NaOH to form NaClO. NaClO is often used for water disinfection. O₃ was formed (Fig. 8) from the oxygen (in air) in the barrier discharge, and is easily decomposed. Small amounts of NO were also formed from the oxygen (Fig. 7). These are different from the results obtained in the spark discharge. This is due to the different properties of the plasma produced in the different discharges.

Barrier discharge consists of a number of microdischarges. Duration of a single microdischarge is 0.1–10 ns, the diameter of the microdischarge channel is ~0.1 mm, the density of electrons in the microdischarge channel ranges from 10¹⁴ to 10¹⁵ cm⁻³, the electron energy is 1–10 eV [39], and the temperature of gas in the microdischarge channel reaches 400 K [40]. The electron energy is sufficient to initiate dissociation of all of the reactants fed into the reactor (CCl₄, O₂, N₂, H₂O). In contrast, low temperature prevents thermal ionization and dissociation from occurring. Consequently, all of the
reactions should be initiated by the electrons. The low temperature also prevents COCl$_2$ synthesis, from CCl$_4$ and CO$_2$ from occurring. In contrast, low temperature causes the O$_3$ decomposition reactions to be slower than its synthesis reactions, and O$_3$ was in the gases at the outlet of the barrier reactor. NO was formed in the reaction between oxygen and nitrogen radicals and in the sequence of reactions initiated by the collision of CCl$_4$ and N radical.

3.6 Influence of specific energy

As seen in Figs. 5 and 6, the CCl$_4$ conversion increased with the increasing specific energy for both reactors. This result corresponded with the results obtained in other discharges [3,8-10]. The specific energy was changed by changing the ratio of the pulse duration to the interval between discharge pulses. Discharge pulse consisted of a number of microdischarges for barrier discharge and streamers for barrier and spark discharge. Extending the duration of the pulse discharge resulted in increased numbers of microdischarges and streamers. As a result, the effect of the most important factors influencing chemical processes increased in each discharge.

The most important factor was the temperature in the spark discharge. Therefore the importance of the temperature-dependent processes increases with increasing specific energy. The importance of undesirable processes increased the most, to wit:
- an increase in the content of NO in post-reaction gases from 1.7 to 2.7% (Fig. 7),
- an increase in the conversion of CCl$_4$ to COCl$_2$ from 2 to 12% (Fig. 5).

The desired decomposition of CCl$_4$ to Cl$_2$ does not depend on the specific energy. The CCl$_4$ conversion to Cl$_2$ was 72-73.5% for tested specific energies in spark discharge (Fig. 7).

The number of electrons was important in barrier discharge. Rates of the reactions initiated by electrons increased with the number of electrons. The increase of the specific energy was related to an increase in the number of discharges. Increase in the number of electrons was the result of increasing the number of discharges. Therefore, the increase of the specific energy caused:
- a slight increase of the content of NO in post-reaction gases (Fig. 7),
- a two-fold increase, from 0.097 to 0.201%, the content of O$_3$ in post-reaction gases (Fig. 8),
- an increase in the conversion of CCl$_4$ to Cl$_2$ (Fig. 6).

4 Conclusions

The results obtained for the two different reactors powered by the same power supply system may be summarized as follows:
- Both reactors effectively decompose CCl$_4$.
- In the barrier reactor, phosgene does not occur.
- In the barrier reactor, nitrogen monoxide was produced in a very small amount.
- The temperature-dependent processes, such as synthesis of NO and COCl$_2$, were important in the spark discharge.
- The processes initiated by high-energy electrons were the most important in the barrier discharge. The processes requiring high temperatures were not important.

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