Improvement of efficiency of the use of pulsed corona discharge energy during the conversion of volatile organic compounds

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Abstract. The effect of electronegative additives on the conversion of volatile organic compounds in plasma of air mixtures has been studied. It was shown that during the conversion of trichloroethylene $\text{C}_2\text{HCl}_3$ and perchloroethylene $\text{C}_2\text{Cl}_4$ in air under the action of pulsed corona discharge of nanosecond duration, the addition of carbon tetrachloride $\text{CCl}_4$ to the mixture leads not only to a decrease in the discharge current, but also to an increase in energy efficiency of the conversion of toxic impurity. To explain the results obtained, the estimation of parameters of non-equilibrium non-thermal plasma and the analysis of processes in various regions of pulsed corona discharge have been carried out.

1. Introduction

The processes of electron attachment to electronegative molecules in plasma of air mixtures have a significant impact both on the composition and properties of non-thermal plasma, and on the processes occurring in it. The main electronegative component of such mixtures is oxygen. However, in some cases, toxic impurities are electronegative molecules too (SO$_2$, CS$_2$, etc.). Attachment processes reduce the concentration of plasma electrons and increase the concentration of negative ions. When plasma is formed under the action of electric discharge, this leads to a decrease in the discharge current.

If negative ions are involved in conversion of toxic impurity, then there is a significant increase in conversion efficiency. Such a case is realized in processes of sulfur dioxide conversion during flue gas treatment by pulsed electron beams and non-self-sustained discharges [1]. If negative ions do not participate in conversion of toxic impurity, their formation only leads to a decrease in the concentration of electrons, resulting in reduction of discharge current and energy input into the gas mixture. This is characteristic of carbon disulfide conversion under the action of pulsed corona discharge [2].

Numerous experiments show that the processes of dissociative attachment to some molecules (e.g., CS$_2$) can lead to the formation of various types of negative ions, depending on which bonds inside the molecule are destroyed [3]. It should also be taken into account that adding chlorine- and fluorine-containing molecules (CCl$_4$, SF$_6$, etc.) to gas mixtures, which possess the highest electron affinity energy, leads to the most noticeable changes in the properties of discharges and plasma. In these cases, even at low electron energies, mainly dissociative attachment occurs [4, 5].

Analysis of the results of experiments on the conversion of various unsaturated volatile organic compounds (VOCs) in gas discharge plasma shows that the conversion is initiated by particles capable
of activating double chemical bonds in molecules. Such particles can be electrons with energy sufficient to activate the double bond, or other active particles (atoms, radicals, ions, excited molecules, etc.). Moreover, each active particle appears as a result of interaction of components of the gas mixture with an electron that has enough energy to form the active particle. This means that the conversion degree of the VOC should be directly dependent on the number of electrons, and therefore on the current and duration of the discharge pulse.

What happens if you add a substance with high electron affinity energy to a mixture containing unsaturated VOC? It is most likely to expect that, as a result of electron attachment processes, concentration of electrons in the discharge plasma decreases, which will lead to a decrease in the discharge current and at the same time to a proportional decrease in the conversion degree of unsaturated VOC. That is, the concentration of unsaturated VOC depending on the number of discharge pulses ($C = f_1(N)$), when adding an electronegative component, should decrease more slowly, and the dependence of concentration on the value of specific energy input into the gas mixture ($C = f_2(w)$) should remain almost unchanged. In other words, electronegative additives should not lead to a change in the energy efficiency of the conversion of unsaturated VOCs in the discharge plasma.

However, experiments on the conversion of some VOCs in pulsed corona discharge plasma do not confirm the above conclusions. Thus, when converting such VOCs in discharge plasma, electronegative additives not only lead to a decrease in the discharge current and energy input into the gas mixture, but also to an increase in the energy efficiency of the conversion of VOCs [6].

To explain the existing contradiction, it is necessary to conduct additional experiments on the conversion of unsaturated VOCs under the action of pulsed corona discharge and to pay special attention to the effect of electronegative additives on the conversion processes. Of the unsaturated VOCs, the most interesting for this study are unsaturated chlorine-containing VOCs, which, on the one hand, are widely distributed and used as solvents, as anesthesia, etc., and on the other hand, are toxic and dangerous to humans and the environment. In addition, such substances have a high electron affinity energy, therefore, when analyzing processes in plasma, it is always necessary to take into account the processes of electron attachment to such molecules. We chose trichloroethylene C$_2$HCl$_3$ and perchloroethylene C$_2$Cl$_4$ as unsaturated chlorine-containing VOCs, and carbon tetrachloride CCl$_4$ as electronegative additive. The objective of this work is to study the effect of CCl$_4$ additive on the conversion of C$_2$HCl$_3$ and C$_2$Cl$_4$ in pulsed corona discharge plasma and to explain the results obtained on the basis of analysis of the processes in different areas of the discharge.

2. Experimental setup

In the experiments, we used the installation, the construction of which is described in [7]. The measurement methods and parameters are presented in [6]. Pulsed corona discharge was ignited between a steel cylinder (diameter 88 mm, length 56 cm) and a steel wire with a diameter of 0.24 mm located on the axis of the cylinder. The wire was a high-voltage cathode, and the cylinder was a grounded anode. The discharge was formed in atmospheric-pressure air mixtures containing toxic impurity (trichloroethylene or perchloroethylene) and an electronegative additive (carbon tetrachloride).

Waveforms of voltage, discharge current and energy input into the processed mixture containing C$_2$HCl$_3$ impurity without additives and with the addition of CCl$_4$ are shown in figure 1. The complex shape of the current pulse is explained by the presence of two components in it: the discharge current itself and the current of damped oscillations in the LC-circuit formed by structural inductance and capacity of the discharge chamber. Energy input was calculated by integrating the product of voltage and current with respect to time.

Since trichloroethylene C$_2$HCl$_3$ is an electronegative compound, the processes of electron attachment to it occur in both cases (with and without the addition of CCl$_4$). However, these processes reduce the discharge current and energy input by no more than 20 % compared to a discharge in clean air. Adding the strongest electronegative compound CCl$_4$ significantly changes the discharge parameters.
Figure 1. Waveforms of voltage (1), current in the discharge gap (2) and energy input into the gas mixture (3) per one discharge pulse in atmospheric-pressure air with an admixture of 1000 ppm C₂HCl₃ (a) and with an additional additive of 5000 ppm CCl₄ (b). Vertical scales: 75 kV/div (1), 270 A/div (2), 0.2 J/div (3); horizontal scale: 20 ns/div.

Although the amplitudes of current oscillations vary slightly, the entire waveform becomes more symmetrical about the horizontal axis, which indicates a significant decrease in the discharge current itself. As a result, a decrease in the energy input by more than 3 times is observed (from 0.5 to 0.15 J).

Similar waveforms were obtained when a discharge was ignited in air mixture containing perchloroethylene C₂Cl₄ impurity without addition and with the addition of CCl₄. It turned out that with the same content of trichloroethylene and perchloroethylene, the waveforms are almost identical. This suggests that trichloroethylene and perchloroethylene are not only close in physical and chemical properties, but also behave similarly in discharge plasma.

3. Results

The results of experiments on the conversion of trichloroethylene under the action of pulsed corona discharge are presented in figure 2.

Figure 2. Dependencies of the concentration of trichloroethylene C(TCE) on the specific energy input $w$ into the gas mixture (a) and on the number of discharge pulses $N$ (b). The initial composition of the mixture: 1000 ppm C₂HCl₃ in air at atmospheric pressure (1) with the addition of 5000 ppm CCl₄ (2).
A monotonic decrease in the concentration of C₂HCl₃ is observed, both with an increase in the specific energy input and with an increase in the number of pulses. It is seen that the addition of CCl₄ leads to a more drastic decrease in the concentration of C₂HCl₃, i.e., in addition to a decrease in the energy input, an increase in the energy efficiency of the conversion is observed. Figure 2b shows that the addition of 5000 ppm CCl₄ not only does not reduce the conversion degree of trichloroethylene, but also leads to the fact that the conversion degree of C₂HCl₃ ~ 99% in a mixture with the addition of CCl₄ is achieved in half the number of discharge pulses, compared to the mixture not containing CCl₄. Wherein the concentration of carbon tetrachloride practically does not change.

Thus, the assumption of the invariance of the energy efficiency of trichloroethylene conversion under the action of pulsed corona discharge upon the addition of electronegative compound is not confirmed.

The results of experiments on the conversion of perchloroethylene under the action of pulsed corona discharge are shown in figure 3.

As in the case of trichloroethylene, both an increase in the specific energy input and an increase in the number of pulses lead to a monotonous decrease in the concentration of toxic impurity. The addition of CCl₄ also leads to a more drastic decrease in the concentration of C₂Cl₄, i.e., an increase in the energy efficiency of the conversion occurs. It can be seen from figure 3b that the addition of 1000 ppm CCl₄ leads to the fact that the conversion degree ~ 90% of C₂Cl₄ is achieved not after 3000 discharge pulses, but only after 1800 ones, that is, much faster. The concentration of carbon tetrachloride in this case remains unchanged too. All this means that, as in the case of trichloroethylene conversion, the energy efficiency of the conversion of perchloroethylene under the action of pulsed corona discharge increases with the addition of carbon tetrachloride.

4. Discussion
To explain the results obtained, it is necessary to analyze the processes in various areas of the pulsed corona discharge. Figure 4a shows the areas of pulsed corona discharge in “wire-cylinder” configuration.

An inhomogeneous electric field is formed in the gap. Electric field strength is

\[ E(r) = \frac{U}{\ln(R_2/R_1)} \frac{1}{r}, \]

where \( U \) is the voltage across the gap, \( R_1 \) is the radius of the wire, \( R_2 \) is the radius of the cylinder, \( r \) is the distance from the axis of the cylinder.
Estimation of the value of $E(r)$ allows to distinguish three regions in the discharge gap:

I – strong field region ($r < 5$ mm, $E > 50$ kV·cm$^{-1}$);
II – moderate field region (5 mm $< r < 20$ mm, 12.5 kV/cm $< E < 50$ kV·cm$^{-1}$);
III – weak field region (20 mm $< r < 44$ mm, 5.8 kV·cm$^{-1} < E < 12.5$ kV·cm$^{-1}$).

If ionization by collision is taken into account, then the value of $E$ in the strong field region will decrease by 10–30%.

Regions I–III differ significantly in the processes occurring in them.

Figure 4. (a) Regions of pulsed corona discharge formed between the wire cathode (1) and the cylindrical anode (2); (b) dependencies of reduced electric field strength $E/n$ (3) and electron temperature $T_e$ (4) at the distance $r$ from the axis of the cylinder.

Dependency of the reduced electric field strength $E/n$ ($n$ is the gas concentration) on the distance $r$ is presented in figure 4b (curve 3). The parameter $E/n$ determines the electron temperature of the discharge plasma. The type of the dependency of the temperature on this parameter is rather complicated. It is known [8] that in the approximation of constancy of mean free path length of electrons this dependency is linear, whereas in the approximation of constancy of mean collision frequency of electrons, the dependency is quadratic. There are various ways to estimate the electron temperature. It is possible to determine it experimentally by optical measurements [9]. Electron temperature may be estimated using simple empirical formulas, for example [10]:

$$T_e / T = 29.96 \ln(|E| / p) + 24.64,$$

where $T_e$ is the electron temperature; $T$ is the gas temperature; $|E|$ is the electric field strength module, V·cm$^{-1}$; $p$ is the gas pressure, Torr. The relative excess of the electron temperature over the gas temperature can be calculated by the formulas that take into account the collisional processes in the discharge, for example [11]:

$$\frac{T_e - T}{T} = \frac{2e^2E^2}{9k_B T^2 n^2 \Sigma \delta},$$

where $e$ is the elementary charge, $k_B$ is the Boltzmann constant, $\Sigma$ is the cross section of elastic electron-atom (electron-molecular) collisions, $\delta = 2m_e/m_i$, $m_e$ is the electron mass, $m_i$ is the mass of an ion or atom. It should be noted that for the same discharge conditions, different assessment methods can give significantly different results.

We used the estimates made by the authors of [12] for the negative corona in air with the electrode configuration closest to that used in our experiments. Based on these estimates, the electron temperature $T_e$ dependency on the distance $r$ was obtained (figure 4b, curve 4). It can be seen that $T_e \geq 6$ eV in the strong field region, $1$ eV $\leq T_e \leq 6$ eV in the moderate field region, and $T_e \leq 1$ eV in the weak field region.
First, we consider processes in a gas without electronegative components, for example, in pure nitrogen. Since the ionization potential of nitrogen is $U_i = 14.5$ eV, and the cross section for ionization by electrons is maximum at an electron energy of $\sim 100$ eV [13], it is obvious that processes of ionization by collision are possible only in the strong field region. That is, the region I is the main source of electrons and positive ions. In addition to ionization by collision, secondary electrons are generated as a result of photoemission from the cathode surface, since the electron work function (4–5 eV) is significantly less than the ionization potential [12]. In addition, electrons in the region I are capable of producing the dissociation of molecules and the electronic excitation of molecules and atoms. In the region II, the temperature of electrons allows them to participate only in two main processes – electronic and vibrational excitation of molecules, and this region can be divided into two subregions: II1, in which both processes are possible, and II2, in which only vibrational excitation of molecules occurs. In the region III, the electron temperature is not sufficient for most inelastic processes; therefore, elastic collisions with molecules and drift of electrons to the anode prevail.

The presence of electronegative components, such as oxygen in the air, changes the situation everywhere except in the region I. In this region, the electron attachment cross section is minimal and there is practically no attachment to oxygen molecules. The processes of ionization by collision, dissociation, and electronic excitation of nitrogen and oxygen, as well as photoemission from the cathode occur. Dissociative attachment of electrons to oxygen in the region II1 and three-body attachment of electrons to oxygen in the regions II2 and III are added to the above processes.

Since practically all electrons are born in the region I, and no attachment processes occur here, the average electron concentration in this region does not depend on the presence of electronegative components and can be estimated from current waveforms in pure nitrogen as $n_e \approx 10^{12}$ cm$^{-3}$. Accounting for the drift of electrons from the cathode in strong field over the duration of the discharge pulse leads to a decrease in $n_e$ by about an order of magnitude. Electron-ion recombination does not have a significant effect on $n_e$ during the discharge pulse.

The proposed division of the discharge gap into three regions is quite reasonable. Thus, the authors of [12], investigating the negative DC corona in “wire-cylinder” configuration, also distinguish three regions in the discharge, but call them differently: the ionization region ($E/n \geq 120$ Td), the plasma region ($E/n \geq 80$ Td) and the region of negative ions ($E/n < 80$ Td). In [12], the electron temperature $T_e = 1.85$ eV corresponds to the value of $E/n = 80$ Td, and the authors note that at this $T_e$, about 5% of plasma electrons have an energy higher than the dissociation energy of the oxygen molecule (5.1 eV).

When a pulsed corona discharge is ignited in air mixture containing trichloroethylene or perchloroethylene impurities, processes of dissociative attachment of electrons to the impurity molecules are added in the moderate and weak field regions. Thus, according to [14], during the conversion of trichloroethylene in plasma, the primary process is the reaction

$$\text{C}_2\text{HCl}_3 + e \rightarrow \text{CHCICCl}_3 + \text{Cl}. \quad (4)$$

Under the conditions of our experiments, when discharge is ignited in air mixture with 1000 ppm $\text{C}_2\text{HCl}_3$ or $\text{C}_2\text{Cl}_4$, $\sim 27\%$ of plasma electrons attach to impurity molecules, and the attachment processes occur only in the moderate and weak field regions. Average concentration of negative ions is $n_e \approx 3.7 \cdot 10^6$ cm$^{-3}$. It is important that for all the mixtures used, the concentration of positive ions in the strong field region is equal to the concentration of electrons in this region when the discharge is ignited in nitrogen: $n_e \approx 10^{12}$ cm$^{-3}$. During the discharge pulse, the positive ions drift towards the cathode, but they do not have time to change their position significantly.

The processes of electron attachment to carbon tetrachloride have a number of features. First, the attachment of electron to $\text{CCl}_4$ leads, as a rule, to the dissociation of the molecule. Thus, at $T_e < 0.6$ eV, only $\text{Cl}^-$ ions are formed, at $T_e > 0.6$ eV, other ions are also formed ($\text{Cl}_2^-$, $\text{CCl}_2^-$, $\text{CCl}_3^-$), and the maximum formation efficiency of $\text{Cl}_2^-$ and $\text{CCl}_4^-$ is observed at $T_e \approx 1.3$ eV [15, 16]. However, with further increase in $T_e$, the attachment efficiency decreases sharply [5]. Second, the characteristic times of attachment of electrons to $\text{CCl}_4$ are few nanoseconds, i.e. during the action of the discharge
pulse, attachment has time to occur in full. (For comparison: the characteristic times of attachment of electrons to \( \text{O}_2 \) are few microseconds; therefore, during the action of a nanosecond pulse, attachment to oxygen can be ignored.) Third, the \( \text{CCl}_4 \) molecule has the highest value of the electron attachment rate constant: \( k_a(\text{CCl}_4) = 2.4 \times 10^{-7} \text{ cm}^3 \cdot \text{s}^{-1} \) [17].

Taking into account the listed features and waveform analysis, the following estimates can be made. When a discharge is ignited in air mixture with 1000 ppm \( \text{C}_2\text{HCl}_3 \) or \( \text{C}_2\text{Cl}_4 \), the addition of 5000 ppm \( \text{CCl}_4 \) to the mixture results in attachment of \( \approx 79\% \) of electrons to electronegative molecules (27% to \( \text{C}_2\text{HCl}_3 \) or \( \text{C}_2\text{Cl}_4 \), 52% to \( \text{CCl}_4 \)). In this case, the attachment processes also occur only in regions II and III, and the average concentration of all negative ions reaches \( n_\text{e} \approx 1.1 \times 10^{10} \text{ cm}^{-3} \). Negative ions drift towards the anode, but during the discharge pulse they do not have time to change their position significantly.

It should be noted that the three regions of the discharge can be considered as three different resistances connecting in series, through which one current flows. Therefore, the attachment processes in regions II and III cause a change in the resistance of these regions and the redistribution of voltage drops across all three regions. Estimates show that in air mixture with 1000 ppm \( \text{C}_2\text{HCl}_3 \) or \( \text{C}_2\text{Cl}_4 \), the voltage drop across regions I, II and III is 95, 35 and 20 kV, and when added 5000 ppm \( \text{CCl}_4 \) – 80, 30 and 40 kV, respectively. Such a redistribution does not lead to a change in the main processes occurring in regions I, II and III.

Based on this, we can draw the following conclusions. The conversion of unsaturated chlorine-containing VOCs is initiated only in the region of strong field in which no electron attachment processes occur. The attachment of electrons in the regions of moderate and weak field reduces the discharge current and the energy input into the gas mixture by reducing the concentration of electrons not participating in the conversion of VOCs, whereas the conversion processes in the region of strong field are not inhibited. As a result, the energy efficiency of the conversion increases.

The above analysis of processes in the discharge plasma explains the increase in the energy efficiency of conversion of unsaturated chlorine-containing VOCs (figures 2a and 3a), but cannot explain the dependencies of VOCs concentration on the number of discharge pulses (figures 2b and 3b, from which it follows that the addition of \( \text{CCl}_4 \) leads to the conversion of more number of VOCs molecules). To explain the increase in the conversion degree of VOCs in the coordinates “concentration – number of discharge pulses” when adding \( \text{CCl}_4 \) to the mixture, it is necessary to analyze the processes of VOCs conversion taking into account the appearance of negative ions and radicals formed from \( \text{CCl}_4 \).

Authors of [14] state that dissociative attachment reaction (4) is the primary initiation step of trichloroethylene conversion in plasma. Chlorine radicals formed in (4) add to carbon atom of the double bond of \( \text{C}_2\text{HCl}_3 \) resulting in the start of a chlorine radical chain reaction. Other researchers consider chain reaction of \( \text{C}_2\text{HCl}_1 \) conversion with participation of both chlorine radicals and other active particles (for example, O and ClO radicals in silent discharge plasma [18], ClO and OH radicals in pulsed streamer corona plasma [19], excited nitrogen molecules \( \text{N}_2^* \) in barrier discharge plasma [20]). In some cases VOCs conversion includes a catalytic cycle with participation of OH radicals [21].

As noted, the processes of dissociative attachment of electrons to carbon tetrachloride lead to the formation of a significant number of chlorine radicals and other active chlorine-containing particles. Both additional chlorine radicals formed as a result of the presence of \( \text{CCl}_4 \) and some of the additional active particles can participate in the chain trichloroethylene conversion process described in [14, 18–20], and thereby increase the efficiency of this chain process significantly. As a result, with the same number of electrons initiating the conversion of trichloroethylene in the region of strong field, additional active particles capable of participating in the chain \( \text{C}_2\text{HCl}_1 \) conversion process appear in the regions of moderate and weak field. This can lead to the conversion of more number of trichloroethylene molecules, as observed in figure 2b. The proximity of the physical and chemical properties of \( \text{C}_2\text{HCl}_3 \) and \( \text{C}_2\text{Cl}_4 \), as well as the similarity of the behavior of these substances in the
discharge plasma, allow to suggest that in the case of perchloroethylene conversion, chain mechanisms involving chlorine radicals and other active particles are realized too. The efficiency of these chain processes can also be significantly improved with the addition of CCl₄ and additional production of chlorine-containing radicals. The consequence of this may be an increase in the number of converted perchloroethylene molecules.

Comparison of the concentration of plasma electrons and changes in the concentration of the gas mixture components in our experiments confirms the possibility of implementing a chain mechanism for the conversion of VOCs. It turned out that there are approximately 19 and 23 converted molecules of perchloroethylene and trichloroethylene per one plasma electron, respectively. On the one hand, this suggests that an electron in the region of strong field, successively accelerating and losing energy in collisions, can initiate the conversion of several molecules of toxic impurity. On the other hand, each active chlorine-containing particle formed as a result of the processes of dissociative attachment of electron can take part in the chain process of the conversion of toxic component and thereby lead to the removal of several VOC molecules.

5. Conclusion

Thus, the introduction of electronegative chlorine-containing additives into the mixture can be considered as a way to improve the efficiency of the use of pulsed corona discharge energy during the conversion of some unsaturated chlorine-containing VOCs. This improvement is achieved due to the spatial separation of the processes of the conversion of toxic impurity and the attachment of electrons to electronegative additives.

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