Kinetics of Destruction of Diisopropyl Methylphosphonate in Corona Discharge

OLEG P. KOROBEINICHEV, ¹ ANATOLY A. CHERNOV, ¹ VLADIMIR V. SOKOLOV, ¹ LEV N. KRASNOPEROV²

¹Institute of Chemical Kinetics and Combustion, Siberian Branch Russian Academy of Sciences, Intitutskaya Street 3, Novosibirsk, 630090, Russia

²Department of Chemical Engineering, Chemistry and Environmental Science, New Jersey Institute of Technology, Newark, New Jersey 07102

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ABSTRACT: The kinetics of the destruction of diisopropyl methylphosphonate (DIMP) in corona discharge has been studied using a flow tubular coaxial wire dielectric barrier corona discharge reactor. The identification and quantitative determination of DIMP, its destruction intermediates, and phosphorus-containing destruction products were performed using molecular beam mass spectrometry and gas chromatography/mass spectrometry. Active discharge power was varied in the range 0.01–5 W. The destruction products such as isopropyl methylphosphonate, methylphosphonic acid, and orthophosphoric acid were found on the reactor walls. The dependence of the extent of the destruction, $D(D = 1 - X/X_0)$, where X and X_0 are DIMP mole fractions at the outlet and the inlet of the reactor), on the specific energy deposition $E_x (E_x = PF^{-1}X_0^{-1})$, where F is the carrier gas flow and P is the power dissipated in discharge reactor) was measured over the DIMP mole fraction range 60–500 ppm at the pressure of 1 bar and the temperature of 340 K. Over the range of the experimental conditions studied the destruction obeys the "pseudo-first-order" kinetic law: $\ln(1 - D) = -KE_x$. Plausible mechanisms of the destruction are discussed. It was concluded that ion mechanism is the major one responsible for the destruction process. © 2002 Wiley Periodicals, Inc. Int J Chem Kinet 34: 331–337, 2002

INTRODUCTION

Recently, corona discharge emerged as a promising novel technology for the treatment of low concentration of pollutants (volatile organic compounds (VOCs), NO_x , SO_x) from stack emissions [1–21]. During the last few years a significant progress was achieved in the application of corona discharge plasma for the conversion of VOCs [1–6], silicon-containing compounds [7], NO_x [2,3,8–11], and SO_x [11–13], and in the understanding of the kinetics of the destruction of these pollutants. Initial research on the decontamination of organophosphorus compounds (OPCs), simulants of sarin in corona discharge, was performed in the mid-80s [14–17]. Fraser, et al. [15–17] determined

Correspondence to: Oleg P. Korobeinichev; e-mail: korobein@ ns.kinetics.nsc.ru.

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hydrocarbons as DMMP (dimethyl methyl phosphonate) destruction products. But phosphorus-containing compounds (PCCs, except phosphine) have not been detected as destruction products and therefore the balance on phosphorus has not been determined. The approach based on the determination of the kinetics of pollutants destruction and the measurement of electrical power consumed by discharge has been developed in [2,3]. The obtained data have been presented using coordinates X_{out}/X_{in} and $E_x = P/(FX_{in})(X_{out}, X_{in} \text{ are}$ concentrations of pollutant at the outlet and the inlet of the reactor, P is the consumed power, and F is the flow). For many investigated compounds (VOCsmethyl chloride, methane, chlorobenzene, toluene, mehtyl ethyl ketone, 1-pentene, and cyclohexene-as well as NO and NO₂) the obtained data follow the law $X_{out}/X_{in} = \exp(-KP/(FX_{in}))$. It was concluded [2,3] that ion mechanism is the major one responsible for the destruction process. Some other researchers [4,10,13] came to the same conclusion. In contrast, other groups of researchers [18] consider that the radical mechanism is the major one responsible for the destruction of toluene and dichloromethane. We have initiated a systematic study of the destruction of simulants of sarin [19-21] in corona discharge. However, the research on the destruction of actual chemical warfare agents as well as their surrogates is still very limited. In the current study, the kinetics and the products of the destruction of a simulant of sarin-diisopropyl methylphosphonate (DIMP)-in a dielectric barrier corona discharge were studied over a range of experimental conditions. The objectives of this research were to apply the approach based on the determination of kinetics of DIMP destruction and on the measurement of power consumed by discharge to destruction of DIMP in corona discharge, to determine intermediate and final destruction products of DIMP as a function of parameter E_x , to check out the balance on phosphorus, to measure OPC destruction extent as a function of parameter E_x , to determine whether the law $(1-D) = \exp(-KP/FX_0)$ describes the destruction of OPC in corona discharge, and to use the obtained data for discussion of possible mechanism of the destruction of OPC in corona discharge.

EXPERIMENTAL

The setup represents a molecular beam mass spectrometry (MBMS) based experimental facility [19] modified for the kinetic studies on the destruction of DIMP in a corona discharge flow reactor. A flow tubular dielectric barrier corona discharge reactor coupled to an online molecular beam inlet system of quadrupole mass spectrometer was used. The reactor consisted of a quartz tubing 115 cm long, 1.2 cm OD, 0.9 cm ID, with a coaxial nichrome wire 0.5 mm in diameter which served as one of the electrodes. The tube was wrapped with nickel copper braid which was the second, grounded, electrode. The active length of the reactor determined by the wrapped length was 105 cm. The reactor tube was located inside a water jacket. The reactor temperature was kept at 340 K by circulating water from a bath circulator. Carrier gases (N₂, He) were supplied from cylinders ("SibTechGas," purity 99.7%). A mass-flow controller (MKS Instruments) controlled the carrier gas flow. The OPC (DIMP in this study) was introduced to the reactor by passing the carrier gas above the surface of the liquid reactant in an evaporator. The mass flow of the reactant was established by measuring the mass loss of the evaporator to the accuracy of 0.1 mg. Typical mass loss at the gas carrier flow rate of 2.5 sccm was 20 mg/h. The mole fraction of DIMP was varied in the range 60–500 ppm. Organophosphorus compounds were obtained from Aldrich: diethylmethylphosphonate (DEMP), 97% (683-08-9); orthophosphoric acid (OPA), 85% (7664-38-2); methylphosphonic acid (MPA), 98% (13590-71-1). The DIMP, 98%, was purchased from Alfa AESAR (Johnson Matthey). N,Obis(trimethylsilyl)trifluoroacetamide (BSTFA), 99.5% (25561-30-2), was obtained from Supelco. Inc.

The output voltage from a 1 kW high voltage AC power supply was controlled by a variac. The active power measurements were based on the electrical measurements [2,3,19]. The maximum amplitude of the output voltage was 20 kV (frequency 50 Hz). The voltage and the current were recorded using a digital oscilloscope (LeCroy, Model 9310AL). The measurements were performed by summing up five hundred 100 ms (five periods) sweeps of the voltage and current. The voltage and averaged. The active power (P) dissipated in the plasma reactor was determined by subtracting the measured power without load from that measured with load.

The samples were analyzed using a molecular beam inlet system with a quadrupole mass spectrometer MC7302 produced by the Experimental Plant of the Russian Academy of Sciences. The detection system allowed to determine the extent of DIMP destruction at mole fractions as low as 1 ppm with the accuracy of 10%. The mole fractions of DIMP and of the destruction products were determined via measuring peak intensities of their parent ions and the calibration coefficients. In addition, mole fractions of DIMP and other PCCs at the inlet (X_0) and the outlet (X) of the reactor were measured using gas chromatography/mass

spectrometry (GC/MS). In these measurements, the gas was passed through a system of three liquid nitrogen traps. A glass wool filter was placed in the last trap to trap possible aerosols. To control the initial mole fraction of DIMP it was measured twice, before and after the experiment. To increase the accuracy of measurements of the initial mole fraction of DIMP the gas was passed through the trapping system for 1 h and more. After that the traps were removed and rinsed with acetone. The resulting solution was evaporated using a rotor evaporator, producing a residue. The volume of the residue (0.3-0.4 ml) was determined in additional test experiments. The efficiency of the trapping and recovery of DIMP under the conditions of trapping and the solution evaporation used was better than 95%. The typical scatter of the experimental data was ca. 5-20% for molecular beam inlet system of mass spectrometer and 7-30% for GC/MS analysis, depending on their mole fractions.

To detect and to quantify the low volatile PCCs by GC/MS, the procedure of silvlation was used [22,23]. This procedure consists of the substitution of the hydrogen atom in hydroxyl group of PCC by trimethylsilyl radical [7,8]. Organophosphorus compounds silylated in this way are volatile and can be detected using GC/MS. The silvlating solution used was a mixture of acetonitrile and BSTFA (9:1 v/v). To account for the effect of the column aging a reference substance was introduced in the silvlating solution of BSTFA and acetonitrile. Diethyl methylphosphonate (DEMP: $PO(CH_3)(OC_2H_5)_2$) was used as a reference compound. About 10 mg of DEMP was added to 10 ml of silvlating solution. It gives a known concentration of DEMP as reference compound in each sample. These portions of the silylating solution were added to the evaporated samples, which were obtained by rinsing the traps or the walls of the reactor. After mixing and moderate heating to accelerate the reaction these mixtures were analyzed by GC/MS. For the determination of the absolute amounts of PCC in the samples, calibration was used. A small known amount of PCC was mixed with the silvlating solution and analyzed by GC/MS. As a result of this procedure we obtained PCC calibrated peak areas which corresponded to the known PCC concentrations. The washings obtained from the reactor and the traps in the experiments were evaporated, silvlated, and analyzed using GC/MS. The calibrated PCC peak areas obtained were used to determine the PCC concentrations in the samples. For example, for DIMP, [Concentration DIMP = [Area DIMP] × [Calibrated Concentration DIMP]/([Calibrated Area DIMP] × [Area DEMP]/[Calibrated Area DEMP]). This formula allowed to quantify PCC concentrations. The efficiency of the OPC trapping depended on the gas flow rate. At low gas flow rates (<20 sccs, the residence time of the gas in the traps >10 s) the calibrated efficiency of DIMP trapping was better than 95%. The analysis of the solutions obtained by rinsing three traps in series showed 70% of PCC trapped in the first trap, 28% in the second trap, and \approx 1% in the third trap at the gas flow rate of 1.66 sccs. About 1% of the PCC was precipitated on the glass wool filter. When calibrating DIMP initial mole fraction, the data on the DIMP weight loss during the experiments were used.

RESULTS AND DISCUSSION

Destruction Products: The Kinetics of DIMP Destruction

The measurements of the extent of DIMP destruction were performed after establishing steady state of DIMP concentration at the outlet of the reactor. It was observed that the establishing of a steady state of DIMP concentration requires a prolonged period of time (about 10 min) when studying low volatile OPCs. The results below were obtained by measuring the PCC mole fractions at the inlet and the outlet of the reactor after this time. The destruction products such as isopropyl methylphosphonate (IMP), methylphosphonic acid, and orthophosphoric acid were found on the wall of the reactor. No other phosphorus-containing destruction products (PCDPs) were detected. A small amount (<10% of all PCDPs) of organophosphorus products of DIMP destruction was found in the liquid nitrogen traps. No aerosols were collected from the glass wool when the gas flow was less than 2 sccs. The results of the DIMP destruction in nitrogen carrier gas (99.7% N_2 , 0.3% O_2) are shown in Fig. 1. In this figure mole



Figure 1 E_x dependence of mole fraction of DIMP and PCDP: 1–DIMP, 2–IMP, 3–MPA, 4–OPA, 5–phosphorus element balance.

fractions of DIMP and its destruction products at the reactor outlet normalized on the inlet mole fraction of DIMP are plotted against the specific deposited energy E_x . The latter was defined as energy per unit gas volume per unit mole fraction of DIMP, $E_x = PF^{-1}X_0^{-1}$, where P is the active power dissipated in the reactor and F is the gas volumetric flow rate. The previous research on VOCs and NO_x destruction demonstrated that the specific energy defined in this way is the parameter characterizing the discharge efficiency of the corona reactor. The mass balance on phosphorus was calculated by summing the mole fractions X_i of the species containing phosphorus and comparing with the initial DIMP mole fraction. Data on phosphorus element balance are in Fig. 1. The experimental data indicated no dependence of the DIMP destruction products on the nature of the carrier gas. The dependencies of the normalized mole fraction X/X_0 on the specific energy E_x for different initial mole fractions of DIMP in the range 60-500 ppm are shown in Fig. 2. This dependency can be expressed by the following equation: $\ln(X/X_0) =$ $-KE_x = -KPF^{-1}X_0^{-1} = -160E_x$. The coefficient K was determined experimentally with the accuracy of $\approx 10\%$. The coefficient *K* has dimension as J⁻¹ sccs ppm. Similar dependence was observed previously in the study of the destruction of VOCs in corona discharge [2,3].

The Effect of the Carrier Gas on the Efficiency of DIMP Destruction

The corona onset voltage as well as the active power dissipated in the reactor at the same applied voltage strongly depend on the nature of the carrier gas. In the current study, helium, dry air, and nitrogen (with ca. 0.3% oxygen impurity) were used as the carrier gases.



Table IDIMP Destruction Extent in Flux of DifferentGas Carriers at $X_0 = 250$ ppm

Gas	$V (J \text{ sccm}^{-1})$			
Composition	$P\left(\mathbf{W}\right)$	ppm ^{−1})	$D_i(\%)$	K_i
He ^a	0.5	0.0008	52	900 ± 200
N_2^a	3.7	0.0059	61	160 ± 30
N ₂ (+0.3% O ₂)	3.8	0.006	66	180 ± 30
Air	4.4	0.007	71	175 ± 30

^{*a*}Estimated amount of $O_2 \le 10$ ppm.

The power consumed by the reactor as a function of the voltage applied for different carrier gases is listed in Table I. The data are also plotted in Fig. 3. The measurements of DIMP destruction in different carrier gases were performed using mass spectrometric detection and the following set of experimental conditions: $F = 5 \operatorname{sccs}, X_0 = 250 \operatorname{ppm}, U = 12 \operatorname{kV}$. It is apparent from Table I that at these experimental conditions the extent of the destruction was about 60% for all carrier gases studied. However, the power P dissipated in the reactor (and, subsequently, E_x) was approximately five to six times lower in helium than that in nitrogen. Therefore, the efficiency of DIMP destruction in helium was approximately five to six times higher than that in nitrogen and air.

Discussion

The experimental observations (kinetic laws, the effect of the concentration, power and flow rate on the removal efficiency) found for the destruction of DIMP in corona discharge are very similar to those obtained earlier for the destruction of some VOCs as well as NO_x in [2,3]. A successful mechanism of DIMP destruction



Figure 3 The discharge power against voltage for different gas carriers.

in corona discharge is to explain the following experimental observations:

- 1. The absolute energy efficiency of DIMP destruction.
- 2. The same energy calculated per one DIMP molecule is required to achieve a certain extent of destruction at different initial concentrations.
- 3. The difference in DIMP destruction efficiency in helium and nitrogen.
- 4. Primary products of DIMP destruction.

Obtained data showed that primary intermediate of DIMP destruction is IMP. This can be explained on the basis of both ion and radical mechanisms. As it was shown in [24] the formation of IMP takes place during DIMP destruction in flame as a result of the following reaction of displacement:

$$(C_3H_7O)_2(CH_3)PO + OH \rightarrow (C_3H_7O)(CH_3)(OH)PO$$

+ C_3H_7O

Similar processes occur during the destruction of other organophosphorus compounds, for example, trimethyl phosphate (TMP) in flame [25]:

$$(CH_3O)_3PO + OH \rightarrow (CH_3O)_2(OH)PO + CH_3O$$

On the other hand the formation of IMP can take place as a result of fast reaction of DIMP ionization by charge exchange and the following reaction of DIMP ion destruction:

$$DIMP + O_2^+ \rightarrow DIMP^+ + O_2$$
$$DIMP^+ \rightarrow IMP + C_3H_6^+$$

Endothermic effect of the second reaction is high, but is less than the exothermic one of the first reaction. Possibly, both stages take place nearly simultaneously. Another pathway may be suggested by analogy with radiolysis of tributyl phosphate (TBP) [26–28] which is similar to that of DIMP. The main process of the TBP destruction is dealkylation [26]. It leads to the formation of di- and mono-*n*-butylphosphate. The process of breakage of C–O bond with the formation of di-*n*-butylphosphate was observed during the decay of excited molecules of TBP and in the reactions involving ions [27]. Similarly one can expect IMP to be formed by the reactions during the radiolysis of DIMP:

$$(C_3H_7O)_2(CH_3)PO + e^-$$

$$\rightarrow 2e^- + (C_3H_7O)_2(CH_3)PO^+$$

$$(C_3H_7O)_2(CH_3)PO^+ \rightarrow (C_3H_7O)(CH_3)(OH)PO$$
$$+ C_3H_6^+$$

In the case of the decay of excited molecules of TBP radicals, C_4H_9 and $(C_4H_9O)_2OPO$ are formed. The latter is a precursor of di-*n*-butilphosphate:

$$(C_4H_9O)_2OPO + (C_4H_9O)_3PO$$

 $\rightarrow (C_4H_9O)_2(OH)PO^+ + (C_4H_8O)(C_4H_9O)_2PO$

In Ref. [27] it was postulated that the initial ion $(C_4H_9O)_3PO^+$ can be decomposed by the following reaction:

$$(C_4H_9O)_3PO^+ \rightarrow (C_4H_9O)_2P(OH)_2^+ + C_4H_7$$

At subsequent abstraction of H^+ from $(C_4H_9O)_2$ -P(OH)₂⁺ ion, di-*n*-butilphosphate is formed. Similar processes with the formation of IMP can be expected during the radiolysis of DIMP, too:

$$(C_{3}H_{7}O)_{2}(CH_{3})PO^{+} \rightarrow (C_{3}H_{7}O)(CH_{3})P(OH)_{2}^{+}$$
$$+ C_{3}H_{5}$$
$$(C_{3}H_{7}O)_{2}(CH_{3})P(OH)^{+}_{2} \rightarrow (C_{3}H_{7}O)(CH_{3})(OH)PO$$
$$+ H^{+}$$

Haase et al. [29] also assumed the possibility of dissociative addition of an electron to a molecule of TBP. Breakage of the bond in molecule of DIMP was observed at electron impact ionization in ion source of mass spectrometer [30,31]. Negative and positive flame ions from a premixed low-pressure ethylene/oxygen flame doped with TMP were analyzed by mass spectrometer [32].

However radical-based mechanism is incapable to explain the above-mentioned experimental observations 1, 2, and 3. It was reported [33] that facts similar to the above cited 1, 2, and 3 have been observed on ethane destruction in corona discharge. The modeling of ethane destruction based on the mechanism involving radicals and atoms [33] failed to explain the destruction efficiency (discrepancy up to a factor of 10^4) and the product composition.

Plausible destruction mechanisms in corona discharge were discussed in [2,3]. In [2], only one mechanism was pointed out as potentially capable to explain all the experimental observations. This mechanism is based on the physical and chemical processes involving ions. Substantially higher energies are required for ion production (\approx 9.6 eV molecule⁻¹) as compared to those required for the production of free radicals (\approx 3–4 eV molecule⁻¹). However the fast charge exchange reactions provide more effective pathway for an impurity molecule destruction. This can explain high efficiency and concentration independence down to very low concentrations. Such a mechanism infers high efficiency of the destruction for those molecules which have ionization potential lower than that of oxygen molecules (the main source of ionization in air). For molecules with ionization potential higher than that of oxygen (such as CH₄, SO₂) low destruction efficiency (calculated per one molecule of the contaminant) at low concentrations is anticipated.

Such correlation was indeed observed in the study of the VOCs and SO_x destruction [2]. The results of this study are in line with such mechanism. The ionization energies of OPC under investigation ($E_i =$ 9.65 eV for DIMP) are lower than that of oxygen ($E_i =$ 12.07 eV). The measured destruction efficiency as well as the concentration dependence are similar to those previously observed for VOCs with low ionization energy.

The higher efficiency of destruction in helium can be rationalized as follows. Helium atom has a very high ionization energy ($E_i = 24.6 \text{ eV}$). Moreover, the first excited state of this atom lies at relatively high energies ($E_1 \sim 20$ eV). Therefore, the elastic collisions of electrons with helium atoms prevail at lower energies. Electrons can gain substantially high energies before they start to loose energy on excitation and ionization in collisions with helium atoms. This is in line with substantially lower corona breakdown voltage in helium compared with nitrogen and air. In such a case, an impurity with low ionization/excitation energy can serve as the major sink of the energy gained by electrons even at very low concentration. Because of the much higher electron temperature a higher energy yield of ionization and electronic excitations is expected. This is consistent with higher destruction efficiency in helium observed in this work. The mechanisms of the destruction of a number of compounds in corona discharge are currently discussed and developed. They were based on the consideration of electron-beam processing, ion-molecular reactions, and neutral reactions chemistry [2,3,7,18,34-36]. The following mechanism for the destruction of DIMP is suggested:

$$\begin{split} O_2 + e^- &\rightarrow O_2^+ + 2e^- \\ O_2^+ + DIMP &\rightarrow O_2 + DIMP^+ \\ O_2^+ + DIMP &\rightarrow O_2 + IMP^+ + C_3H_6 \\ DIMP^+ + e^- &\rightarrow IMP + C_3H_6 \\ DIMP^+ &\rightarrow IMP + C_3H_6^+ \end{split}$$

Processing of experimental data shows that *G*-value (a number of destroyed molecules per 100 eV) for DIMP is about 0.07. *G*-value for the ion production in the reduced electric field of 130 Td (the reduced critical field) is 0.17 ion per 100 eV, which is close to *G*-value measured for DIMP. So ion mechanism enables one to explain high DIMP destruction efficiency. Not all facts can be explained now. The task of future investigation is to develop a model which can explain quantitatively the dependence of X_{out}/X_{in} on $E = P/FX_{in}$.

CONCLUSIONS

The highest efficiency of OPC destruction was achieved using helium as a gas carrier. The law $\ln(X/X_0) = -KPF^{-1}X_0^{-1}$ proposed for destruction of VOCs was confirmed for OPC destruction.

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