

COLLOID CHEMISTRY AND ELECTROCHEMISTRY

A Model of Reactions in a Corona Discharge in the $O_2(g)$ – H_2O System

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Abstract—Reactions in the water–oxygen system during a pulse corona discharge between an electrode and the water surface (at a discharge current of 0.1 mA, voltage across the gap of 10 kV, and a negative potential on the electrode) were studied. A model was developed for describing reactions in the liquid phase initiated by active species formed in the gas phase. It was shown that H, OH, HO_2 radicals at a high concentration of ozone are not eliminated but are converted into each other via interactions with ozone: $H \longrightarrow OH \rightleftharpoons HO_2$. The yields of the reactions of the active species with substances dissolved in water may be as high as the yield of H and OH in the discharge. The experimental data obtained were used to determine the parameters of the model.

In a system consisting of a liquid and a gas, an electric discharge between the electrode and the surface of the liquid or some other source of ions, initiates the formation of chemically active radicals in the gas phase. The conditions proposed in [1, 2] provide for an effective interaction of these radicals with substances dissolved in water and for an agitation of a liquid layer up to 2 cm in thickness [2]. In the present work, we developed a model of the process on the basis of an analysis of reactions in the water–oxygen system and determined its parameters, a circumstance that makes it possible to apply this model to actual processes.

EXPERIMENTAL

We measured the steady-state concentration of ozone during a pulse corona discharge [3]. The current of the discharge was 0.1 mA, the width of the gap between electrode and surface of the liquid ~3.5 mm, and the voltage across the gap 10 kV; the potential at the electrode was negative. The scheme of the experiment was similar to that described in [1, 2]. We used glass vessels 0.5, 1.0, 2.0, and 3.0 l in volume, which were closed with polyethylene stoppers with orifices for nickel electrodes and gas inlets and outlets. In all experiments, the thickness of the water layer was 2 cm. The volumes of water were 100 ml in the 0.5- and 1.0-l vessels and 200 and 300 ml in the 2- and 3-l vessels, respectively. The oxygen flowed through the vessels at a rate of 1.0 l/h. Distilled water and chemically pure reagents were used in the experiments.

The concentration of ozone in the reactor was determined as follows. Before the discharge was switched on, oxygen was allowed to pass through the reactor for 0.5–3 h, depending on its volume. Then the voltage was applied, and the discharge occurred for 30 min. After the discharge was switched off, the electrodes were

removed so as to avoid ozone leakage, and 1% KI (up to 20 ml) and a 1 : 5 dilute sulfuric acid were added. The reactor was closed with a lid and shaken from time to time for 10–30 min, depending on the volume of the vessel, until the odor of ozone disappeared. As a result, all the ozone formed in the liquid and gas phases was consumed in the reaction with KI. The iodine formed was titrated with sodium thiosulfate to determine the total amount of ozone formed.

To determine the yield of hydrogen peroxide, we poured the treated liquid into an open vessel and kept it in air (periodically shaking) for 2 h to remove dissolved ozone. The content of hydrogen peroxide was determined by a $KMnO_4$ titration in an acidic medium.

In a special experiment, we measured the formation of iodine under the action of a 0.1-mA corona discharge in oxygen on a 0.01 mol/l solution of KI in distilled water. The reaction was studied in the 0.5-l vessel. The content of iodine after the reaction was determined by titration with sodium thiosulfate in an acidic medium.

The yield of the reaction $Fe^{2+} \longrightarrow Fe^{3+}$ was also measured. The reaction was carried out in a 36-cm³ cylindrical glass vessel. The vessel was filled with 20 ml of the solution to be studied. A platinum electrode 0.2 mm in diameter was introduced through an orifice in the bottom of the vessel, which was in contact with the liquid. The vessel was closed with a Teflon stopper with orifices for introducing the discharge electrode and two glass tubes used for filling the vessel with oxygen. The discharge electrode made from a 0.2 mm wire was mounted at a glass insulator 3.5 mm from the surface of the liquid. The parameters of the discharge were the same as in the other experiments performed in this work. The temperature of the reactor was maintained at a level of 20–21°C.

The experiments were carried out with solutions of FeSO₄ in 0.4 mol/l H₂SO₄. The solutions were prepared as described in [4], but without adding NaCl. The concentration of FeSO₄ was varied from 5×10^{-5} to 0.1 M. Depending on the yield of the reaction, its duration was varied from 1 to 10 min so as to provide for a sufficient accuracy of measurements. We examined whether the yield depends on the duration of the reaction. In all cases, this dependence was linear. The concentration of Fe³⁺ in solution during the reaction was measured spectrophotometrically at 300 nm; high concentrations were determined by titration.

MODEL

It was assumed that the action of the electric discharge on a mixture of water vapor and oxygen (oxygen was saturated with water vapor to a partial pressure of ~2.4 kPa at 20°C) yield the OH, H, and O radicals and ozone. Then these active species interact with each other in the gas phase. We composed a scheme containing all possible reactions, both between the initial radicals and the products of their transformations. The scheme is presented in Table 1 (the rate constants were borrowed from the handbook [5] and the original papers [6, 7]). The secondary species formed by these reactions are the HO₂ radical, hydrogen peroxide, and molecular hydrogen.

In the system under study, interactions between molecules occur in the gas and liquid phases and at the interface. Interactions of species dissolved in water with active species formed in the gas phase can occur only after these species are absorbed by the liquid phase.

The process under consideration involves species that slowly and rapidly react with substances dissolved in water. The concentration of the species of the first type (ozone and hydrogen peroxide) is almost the same throughout the entire volume of the liquid; the species of the second type (radicals, such as OH) only have time to react in an upper layer of the liquid. In [8], the equation of diffusion is solved for various cases of the diffusion of gases in liquids at different rates of reactions in the liquid phase. It was assumed that the diffusion of a gas into a liquid insignificantly affects the physical properties of the liquid; therefore, the temperature and diffusion coefficient in the subsurface layer should not differ markedly from those in the bulk of the liquid. For noninteracting gases, the absorption of each should occur independently, according to its properties and partial pressure. For the rate of diffusion in an agitated liquid, it is possible to obtain the following formula

$$W \sim s(A^* - A^0/H)(D_A/\pi\theta)^{1/2}, \quad (1)$$

where A^* is the concentration of the gas near the gas-liquid interface, A^0 is the average concentration of the dissolved gas in the liquid phase, H is the Henry's law

Table 1. Rate constants k_{ij} [l/(mol s)] for reactions between active species in the gas phase at 293 K [5]

No.	Rate	k_{ij}
1	$\text{OH} + \text{OH} \rightarrow \text{H}_2\text{O}_2$	1.3×10^{10}
2	$\text{OH} + \text{OH} \rightarrow \text{H}_2\text{O} + \text{O}_2$	1.1×10^9
3	$\text{OH} + \text{H} \rightarrow \text{H}_2\text{O}$	1.1×10^{10}
4	$\text{OH} + \text{HO}_2 \rightarrow \text{O}_2 + \text{H}_2\text{O}$	6.6×10^{10}
5	$\text{OH} + \text{O} \rightarrow \text{O}_2 + \text{H}$	1.8×10^{10}
6	$\text{OH} + \text{O} \rightarrow \text{HO}_2$	2.2×10^9
7	$\text{OH} + \text{O}_3 \rightarrow \text{HO}_2 + \text{O}_2$	4.0×10^7
8	$\text{OH} + \text{H}_2 \rightarrow \text{H} + \text{H}_2\text{O}$	4×10^6
9	$\text{OH} + \text{H}_2\text{O}_2 \rightarrow \text{HO}_2 + \text{H}_2\text{O}$	1.0×10^9
10	$\text{H} + \text{H} \rightarrow \text{H}_2$	1.0×10^9
11	$\text{H} + \text{HO}_2 \rightarrow 2\text{OH}$	4.3×10^{10}
12	$\text{H} + \text{HO}_2 \rightarrow \text{H}_2 + \text{O}_2$	3.4×10^9
13	$\text{H} + \text{HO}_2 \rightarrow \text{H}_2\text{O} + \text{O}$	1.4×10^9
14	$\text{H} + \text{H}_2\text{O}_2 \rightarrow \text{OH} + \text{H}_2\text{O}$	2.2×10^7
15	$\text{H} + \text{H}_2\text{O}_2 \rightarrow \text{H}_2 + \text{HO}_2$	2.6×10^6
16	$\text{O} + \text{H}_2\text{O}_2 \rightarrow \text{OH} + \text{HO}_2$	1.0×10^6
17	$\text{HO}_2 + \text{HO}_2 \rightarrow \text{H}_2\text{O}_2 + \text{O}_2$	1.3×10^8
18	$\text{O}_3 + \text{H} \rightarrow \text{OH} + \text{O}_2$	5.4×10^8
19	$\text{O}_3 + \text{HO}_2 \rightarrow \text{OH} + 2\text{O}_2$	1.2×10^6
20	$\text{O} + \text{HO}_2 \rightarrow \text{OH} + \text{O}_2$	3.2×10^9
21	$\text{H}_2\text{O}_2 \rightarrow 2\text{OH}$	1.33×10^{-7}
22	$\text{O}_3 \rightarrow \text{O}_2 + \text{O}$	1.73×10^{-4}
23	$\text{O} + \text{O}_3 \rightarrow 2\text{O}_2$	4.8×10^6
24	$\text{O} + \text{H}_2 \rightarrow \text{OH} + \text{H}$	4.0×10^3
25	$\text{HO}_2 + \text{H}_2 \rightarrow \text{H}_2\text{O} + \text{OH}$	1.0×10^3 [6]
26	$\text{H}_2 \rightarrow \text{H} + \text{H}$	1.0×10^{-3}

Note: According to [7], for reaction (9) in the liquid phase, $k_{ij} = 4.07 \times 10^7$ l/(mol s).

constant, s is the surface area of the interface, D_A is the diffusion coefficient for species A, and θ is a coefficient characterizing the agitation.

When diffusing species are consumed in a fast reaction of the second order with substance N, solution of the equation of diffusion at a long time yields

$$w \sim k_{A,N} A^* N S D_A^{1/2}, \quad (2)$$

where $k_{A,N}$ is the reaction rate constant and N is the concentration of the impurity in water. Thus, under all possible conditions, the rates of absorption of species from the gas phase by water are proportional to their concentrations over the interface and are dependent on the coefficient of their diffusion in the liquid phase, i.e., on their physicochemical properties.

The equation for the rate of the consumption of radicals and impurity molecules (denoted by R) by the reaction in given volumes of gas V_g and liquid V_l yields according to (2)

$$\frac{d[R]}{dt} = \frac{dN}{dt} - k_{R,N} S \frac{[R]}{V_g} N D_R^{1/2}.$$

The rate of consumption for the interaction of radicals with each other and with ozone are $\frac{k_{R,R}}{V_g} [R]^2$ and $\frac{k_{R,O_3}}{V_g} [R][O_3]$, respectively. The impurity interacts with ozone dissolved in water at a rate of $\frac{k_{O_3,N}}{V_1} [O_3]wN$.

The consumption of ozone by water proceeds at a rate of

$$D_{O_3}^{1/2} s \left(\frac{[O_3]}{V_g} - \frac{[O_3]w}{V_1 H} \right),$$

where $H = 1.7$ [9].

Finally, the system of equations takes the form

$$\begin{aligned} \frac{d[R_i]}{dt} = & [R_i]_0 - \frac{k_{i,j}}{V_g} [R_i][R_j] - k_{i,N} a_1 D_{R_i}^{1/2} s \frac{[R_i]}{V_g} N \\ & - \frac{k_{O_3,i}}{V_g} [R_i][O_3] - V_{R_i}, \end{aligned} \quad (3)$$

$$\frac{dN}{dt} = -k_{R,N} a_1 D_{R_i}^{1/2} s \frac{[R_i]}{V_g} N - \frac{k_{O_3,N}}{V_1} [O_3]wN, \quad (4)$$

$$\begin{aligned} \frac{d[O_3]}{dt} = & [O_3]_0 - s D_{O_3}^{1/2} a_2 \left(\frac{[O_3]}{V_g} - \frac{[O_3]w}{V_1 H} \right) \\ & - \frac{k_{O_3,i}}{V_g} [R][O_3] - V_{O_3}, \end{aligned} \quad (5)$$

$$\begin{aligned} & \frac{d[O_3]w}{dt} \\ & = s D_{O_3}^{1/2} a_2 \left(\frac{[O_3]}{V_g} - \frac{[O_3]w}{V_1 H} \right) - \frac{k_{O_3,N}}{V_1} [O_3]wN. \end{aligned} \quad (6)$$

Here, $[R_i]_0$ and $[O_3]_0$ are the rates of production of radicals and ozone; a_1 and a_2 are proportionality coefficients, which are dependent on agitation conditions and the reaction order; $k_{i,j}$ is the rate constant for the reaction between radicals (molecules) i and j ; and V_{R_i} and V_{O_3} are the rate of the removal of radical i and ozone by the flow of oxygen through the vessel. The time evolution of the concentrations of each species was calculated by solving the system of equations (3)–(6).

RESULTS AND DISCUSSION

Role of ozone. The active species H, OH, and HO_2 interact with ozone [reactions (7), (18), and (19) in Table 1]. These reactions play the main role, because the concentration of ozone is much higher than those of the other active species. In these reactions, the active

species are not eliminated but are transformed into each other:



The steady-state ratio between the concentrations of OH and HO_2 is given by

$$[OH]/[HO_2] = k_{19}/k_7 = 0.03,$$

i.e., the steady-state concentration of OH is much lower than that of HO_2 .

Consumption of impurities dissolved in water. The main routes for the consumption of OH are reactions (1)–(5) in Table 1. At small concentrations of the active species and a high concentration of water in the gas phase, OH radicals predominantly react with the substance dissolved in water, a situation that is characterized by the inequality [2]

$$[OH] \ll A_0 N k_{OH,N} / k_{OH,OH}. \quad (7)$$

Here, $A_0 = a_1 D_{OH}^{1/2}$ [see equation (3)], $k_{OH,N}$ is the rate constant for the reaction of OH with substance N dissolved in water. OH radicals are formed in the discharge at rate k_{OH} and by the reaction of atomic hydrogen with ozone [reaction (18)] and are consumed by the reaction with the impurity if inequality (7) is fulfilled; thus, under steady-state conditions, we obtain

$$dN/dt = k_{OH,N} [OH] N s A_0 = k_{OH} + [H][O_3] k_{18}. \quad (8)$$

The main route for the consumption of hydrogen atoms is reaction (18), because the concentration of ozone is much higher than the concentrations of the other species; hydrogen atoms are formed in the electric discharge at rate k_H . Thus, under steady-state conditions, $[H][O_3] k_{18} = k_H$, and equation (8) takes the form

$$dN/dt = k_{OH} + k_H,$$

which means that if condition (7) is met, the rate of the consumption of the impurity by reactions with radicals is equal to the rate of the formation of the radicals and that practically all radical are involved in the process of decomposition of substances dissolved in water.

Yield of OH and H and the coefficient of diffusion of OH. We measured the yield of the reaction $Fe^{2+} \longrightarrow Fe^{3+}$. This process may involve reactions with radicals, hydrogen peroxide, and ozone. Under the experimental condition used in the present work, the yield of hydrogen peroxide is small [1]. The rate constant for the reaction of Fe^{2+} with ozone is also small, only ~ 250 l/(mol s) [10]. In addition, the duration of the experiment (below 10 min) is insufficient for the steady-state concentration of ozone to be established. As a result, the yield of the reaction between Fe^{2+} and ozone is negligibly low. In acidic media, ozone is stable and does not initiate the formation of OH radicals [11].

Thus, the main contribution to the oxidation of Fe^{2+} comes from OH radicals. The dependence of the yield of Fe^{3+} on the concentration of Fe^{2+} is displayed

in Fig. 1. As can be seen, the experimental dependence levels off, with the yield at the plateau being 34 ± 2 mol/mol electrons. A preliminary estimate of the yield of active species was made under the assumption that the ionizing effects of an electron with an initial energy of 10 keV and of an electron passed through the potential difference of 10 kV are the same. As a result, we obtained $Y(OH) = 14.4$, $Y(H) = 18.0$, and $Y(O) = 2.5$ mol/mol electrons [12]. The total yield of active species is 32.4. This value coincides within the error limits with the experimentally measured maximum yield of the reaction, which is realized when condition (7) is fulfilled for all radicals reacting with the impurity.

Results of this experiment were used to determine the product of the diffusion coefficient and the kinematic factor. For this purpose, we calculated the dependence of the yield of Fe^{3+} on the concentration of Fe^{2+} at various values of A_0 [see relation (7)] by solving the system of equations (3)–(6) for the formation and consumption of active species (OH , H , O , HO_2 , H_2O_2 , and O_3) in reactions with each other and with Fe^{2+} . We used the rate constant of $k_{OH, Fe^{2+}} = 2 \times 10^9$ l/(mol s) [13]. The calculation results are displayed in Fig. 1. As can be seen, most of the experimental points lie between the curves plotted at $A_0 = 1 \times 10^{-4}$ and 3×10^{-4} cm 2 , somewhat closer to the curve calculated at $A_0 = 3 \times 10^{-4}$ cm 2 . We set, therefore, $A_0 = (2-3) \times 10^{-4}$ cm 2 .

Yield of ozone and the diffusion coefficient for ozone in water. The dynamics of the establishment of the equilibrium content of ozone in the vessels was calculated and measured. Figure 2 shows the results for the 0.5-l vessel. As can be seen, the characteristic time of establishment of the stationary state is ~ 15 min. The contents of ozone in the vessels established within 30 min are given in Table 2. The stationary content of ozone was calculated by solving the system of equations (3)–(6). The rate of the formation of ozone in the electric discharge $[O_3]_0$ was a free parameter. In the steady-state regime, the distribution of the concentration of ozone between the gas and liquid phases is determined by the Henry's law constant and geometric factors; therefore, a comparison of the calculated and measured values makes it possible to determine the yield of ozone in the electric discharge. The obtained ozone yield is $k_{O_3} = 180$ mol/mol electrons, which corresponds to an energy expenditure of 31 W per gram of ozone. The calculated contents of ozone in the vessels are listed in Table 2; the calculated dependence of the ozone content in the 0.5-l vessel on the duration of the reaction at $k_{O_3} = 180$ mol/mol electrons is shown in Fig. 2. As can be seen for Table 2 and Fig. 2, the model closely describes the experimentally measured contents of ozone in the different vessels and the evolution of the ozone concentration.

To calculate the characteristics of reactions involving ozone, it is necessary to know the diffusion coeffi-

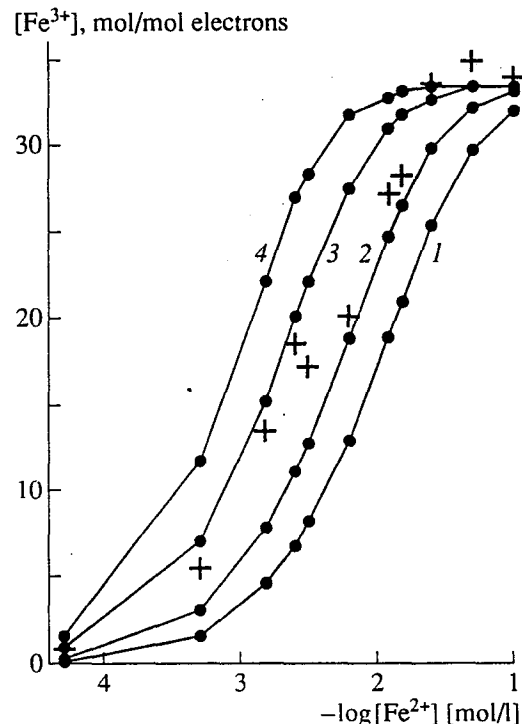


Fig. 1. Experimental (crosses) and calculated (closed circles) dependences of the yield of Fe^{3+} (mol/mol electrons) on the concentration of Fe^{2+} in solution. The values of the yield at the same A_0 are connected by straight lines: $A_0 = (1) 5 \times 10^{-5}$, (2) 1×10^{-4} , (3) 3×10^{-4} , and (4) 7×10^{-4} cm 2 .

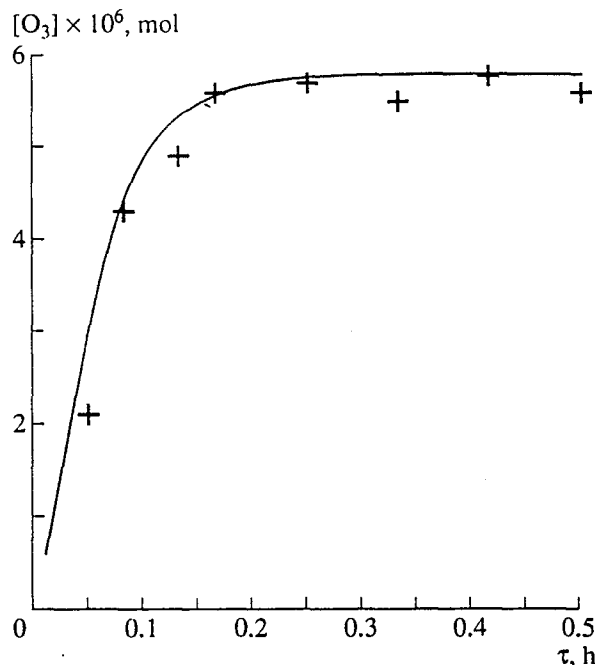


Fig. 2. Time evolution of the steady-state concentration of ozone in the 0.5-l vessel (containing 0.1 l of water); the symbols and the line designate experimental values and calculation results at an ozone yield of 180 mol/mol electrons.

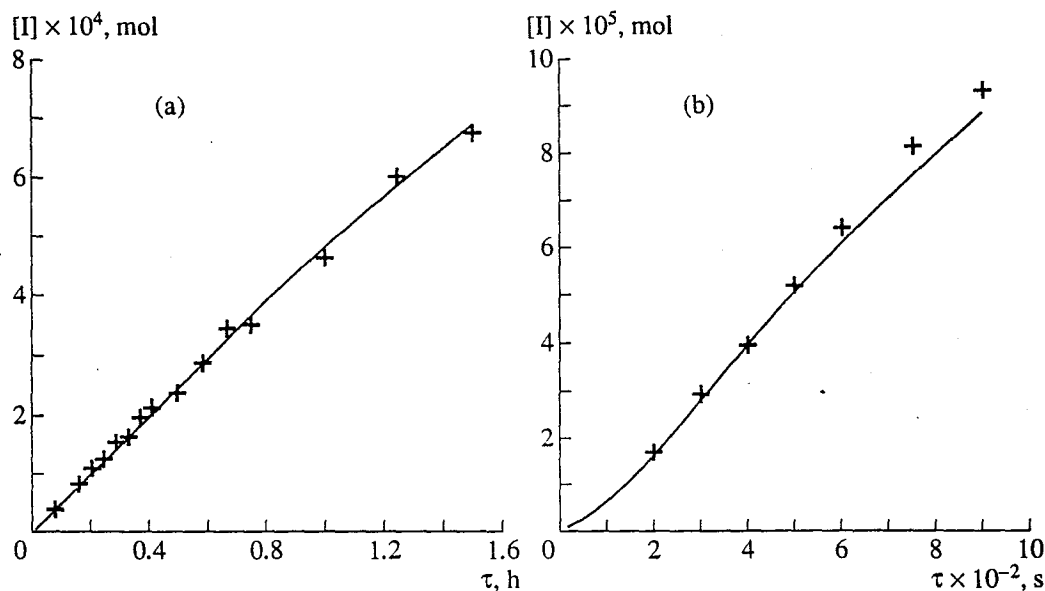


Fig. 3. Yield of I_2 as a function of time at KI concentrations in solution of (a) 10^{-2} and (b) 10^{-3} mol/l; the symbols and line refer to experimental and calculated values, respectively.

cient for ozone in water. For this purpose, we measured the yield of iodine in KI solutions as a function of the duration of the reaction. The reaction was performed in the 0.5-l vessel. In this case, the choice of the ozone concentration is important. The calculated time of establishment of the stationary concentration of ozone is in agreement with the experimental value of ~ 15 min; therefore, the duration of measurement of the iodine yield should be longer. The mechanism of the process

in solution is fairly complex: products of I^- oxidation may interact with ozone and OH. At KI concentrations from 10^{-4} to 10^{-3} mol/l, almost all I^- ions are consumed within 0.5–1.0 h, a period after which the interaction of ozone with products plays the main role. This makes calculations more complicated and introduces uncertainties associated with the choice of the reaction mechanism and with the values of individual rate constants. At $[KI] \sim 0.1$ mol/l, almost all ozone interacts with iodine at the gas–water interface, without penetrating into the bulk. Under these conditions, the diffusion coefficient cannot be determined; therefore, we used the KI concentration of 10^{-2} mol/l.

Table 2. Content of ozone ($[O_3] \times 10^5$ mol) in the steady-state regime during the pulse corona discharge between the electrode and the surface of the liquid (V_l is the volume of the liquid, V_v is the volume of vessel)

$V_v, \text{ l}$	$V_l, \text{ l}$	Expt.	Calc.
0.5	0.1	2.0	1.9
1.0	0.1	2.4	2.43
2.0	0.2	3.9	3.4
3.0	0.3	4.2	4.1

Table 3. Content of hydrogen peroxide in solution after treatment with the pulse corona discharge for 30 min (ϵ is the coefficient of absorption of H_2O_2)

$V_v, \text{ l}$	ϵ	$[H_2O_2] \times 10^6, \text{ mol}$	
		Expt.	Calc.
0.5	0.166	2.0 ± 1	1.6
1.0	0.091	1.5 ± 0.8	0.5
2.0	0.12	1.5 ± 0.8	0.7
3.0	0.139	1.5 ± 0.8	0.8

The dependence of the I_2 yield on the time at $[KI] = 10^{-2}$ mol/l is shown in Fig. 3a. As can be seen, there is close agreement between the calculated and measured values. The value of $a_2 D_{O_3}^{1/2}$, the parameter that characterizes diffusion and agitation, was found to be $(3.5 \pm 0.2) \times 10^{-5} \text{ cm}^{-2}$. Figure 3b shows similar dependences at $[KI] = 10^{-3}$ mol/l. As can be seen, up to a $\sim 60\%$ conversion (a $\sim 6 \times 10^{-5}$ -mol yield), the calculated and measured values are in good agreement. At higher conversions, the calculated yield approaches the level of 10^{-4} mol (KI content in water), whereas the iodine yield measured by titration with thiosulfate in an acidic medium is controlled by the formation of compounds with a higher degree of I^- oxidation (for example, IO_3^-), which are absent in the scheme.

Hydrogen peroxide yield. The yield of hydrogen peroxide 30 min after the beginning of the reaction are presented in Table 3. The average yield is 0.5–1 mol/mol electrons, which is somewhat higher than the value obtained in [1] at the same conditions of elec-

tric discharge but with a 36-cm³ vessel and a volume of the liquid of 20 ml. This is due to an increase in the probability of the consumption of radicals with increasing concentration in [1], i.e., due to a small size of the vessel.

To calculate the kinetics of the accumulation of hydrogen peroxide in water, it is necessary to introduce the coefficient of absorption by water of hydrogen peroxide formed in the gas phase. At small concentrations of H_2O_2 in water, its evaporation can be safely neglected; therefore, we assumed that all H_2O_2 molecules striking the water surface are absorbed, i.e., the absorption coefficient is equal to the ratio of the surface area of the gas-water interface to the entire surface area of the gas-filled cavity. The calculation results are listed in Table 3; within the error of measurements, they coincide with the experimental values of the H_2O_2 concentration.

Thus, the proposed model correctly describes all the processes occurring in the oxygen-water system during a pulse corona discharge between an electrode and the surface of the liquid.

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