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КИНЕТИКА РАЗЛОЖЕНИЯ ПАРАЦЕТАМОЛА В ВОДНОМ РАСТВОРЕ ПОД ДЕЙСТВИЕМ РАЗРЯДА ПОСТОЯННОГО ТОКА АТМОСФЕРНОГО ДАВЛЕНИЯ В ВОЗДУХЕ

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Исследована кинетика разложения парацетамола в его водном растворе под действием на него разряда постоянного тока атмосферного давления в окружающем воздухе. Диапазон исследованных концентраций парацетамола составлял 7-37 мг/л (0,046-0,25 ммоль/л). Токи разряда менялись от 20 до 50 мА. Кинетика разложения определялась в диапазоне времен 0-600 с. Концентрация парацетамола измерялась спектрофотометрически по поглощению на длине волны 242 нм (максимум полосы поглощения парацетамола). Обработки растворов проводились для случаев, когда раствор являлся жидким катодом и жидким анодом. Показано, что кинетика разложения описывается формальным уравнением первого кинетического порядка по концентрации парацетамола. Определены формальные константы скоростей разложения, которые зависели как от начальной концентрации раствора, так и тока разряда. Константы росли с ростом тока разряда и уменьшались с ростом начальной концентрации. Типичные значения констант скоростей лежали в диапазоне ~$(8\cdot10^{-2}$-$8\cdot10^{-3})$ c^{-1}, а степени разложения при некоторых параметрах достигали 100%. При фиксированном времени плазменного воздействия степень разложения была тем больше, чем меньше начальная концентрация и больше ток разряда. При прочих равных условиях константы скоростей разложения, скорости и степени разложения в растворе, который служил катодом, были выше, чем растворе, который служил анодом. На основе этих измерений рассчитаны энергетические характеристики процесса разложения. Величины энергетических выходов разложения лежат в диапазоне ~(0,05-0,019) разложившихся молекул парацетамола на 100 эВ вложенной энергии. Несмотря на более высокие скорости и степени разложения в разряде с жидким катодом, из-за различия в параметрах разрядов, энергетическая эффективность в разряде с жидким анодом оказывается выше.

Ключевые слова: разряд, парацетамол, водный раствор, разложение, кинетика

KINETICS OF DECOMPOSITION OF PARACETAMOL IN AQUEOUS SOLUTION UNDER THE ACTION OF A DC DISCHARGE AT ATMOSPHERIC PRESSURE IN AIR

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The kinetics of decomposition of paracetamol in its aqueous solution under the action of a direct current discharge at atmospheric pressure in the ambient air has been studied. The range of studied concentrations of paracetamol was 7-37 mg/l (0.046-0.25 mmol/l). The discharge currents varied from 20 to 50 mA. The decomposition kinetics was determined in the time range of 0–600 s. The concentration of paracetamol was measured spectrophotometrically by absorption at a wavelength of 242 nm (maximum of the absorption band of paracetamol). The solutions were processed for cases when the solution was a liquid cathode and a liquid anode. It is shown that the kinetics of decomposition is described by a formal first-order kinetic equation with respect to the concentration of paracetamol. Formal decomposition rate constants were determined, which depended both on the initial concentration of the solution and on the discharge current. The constants increased with an increase in the discharge current and decreased with an increase in the initial concentration. Typical values of the rate constants were in the range of ~$(8\cdot10^{-2}$-$8\cdot10^{-3})$ s^{-1}, and the degree of decomposition reached 100% for some parameters. At a fixed time of plasma exposure, the degree of decomposition was the greater, the lower the initial concentration and the greater the discharge current. Other things being equal, the rate constants of decomposition, the rate and degree of decomposition in the solution that served as the cathode, were higher than the solution that served as the anode. Based on these measurements, the energy characteristics of the decomposition process were calculated. The energy yields of decomposition lie in the range of ~(0.05–0.019) of decomposed paracetamol molecules per 100 eV of input energy. Despite the higher rates and degrees of decomposition in a discharge with a liquid cathode, due to the difference in the parameters of the discharges, the energy efficiency in a discharge with a liquid anode is higher.

Key words: discharge, paracetamol, aqueous solution, decomposition, kinetics

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INTRODUCTION

Paracetamol (PC) and its toxic metabolites are present in surface and waste water and drinking water. Its aqueous solutions have a toxic effect on microorganisms [1, 2]. For example, its content in European wastewater was 11.3 mg/L [3], in the United States, up to 150 mg/L [4], and in China, up to 218 mg/L [5]. Therefore, water purification from PC is an urgent task. The use of non-equilibrium plasma of gas discharges in contact with a solution is one of the most effective methods for destroying any organic and inorganic pollution [6-12]. The purpose of this work was to determine the efficiency of using a direct current discharge for the decomposition of PC in the case when the solution served as a cathode and an anode. We are not aware of such studies for a direct current discharge. A few results obtained in other types of discharges will be considered further in the analysis of the data obtained by us.

EXPERIMENTAL PART

The object of the study was aqueous solutions of paracetamol (para-acetylaminophenol, $HO-C_6H_4-NH-CO-CH_3$) (purity according to GCMS analysis 99.5%, Shimadzu GCMS-QP 2010) with a concentration in the range of 7-37 mg/L (0.046-0.25 mmol/L). The pH of the prepared solution was ~5.5. The solutions were processed on a setup, the scheme of which is shown in Fig. 1.

An atmospheric pressure direct current discharge in air was excited by applying a constant voltage between external titanium electrodes and the surface of the treated liquid. An H-shaped reaction vessel was used to separate the processes occurring in the solution under the action of a glow discharge in the anode and cathode regions. The cells were separated by a cellophane membrane 5 μm thick. The working volume of paracetamol solution was 200 ml. The distance from the electrode to the solution surface is 5 mm. The discharge was ignited by breakdown of the discharge gaps. The treatment was carried out in ambient air at atmospheric pressure with different durations of discharge burning from 30 to 600 s. The discharge current was varied from 20 to 50 mA. To find the power deposited in the discharge, the electric field strengths in the plasma and the electrode potential drops were measured using the moving electrode method [13, 14].

The concentration of PC was determined spectrophotometrically (spectrophotometer SF-56, Russia) by absorption at a wavelength of 242 nm (maximum of the absorption band of PC [15]).

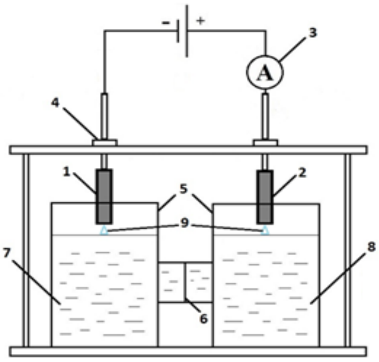

Fig. 1. The setup of DC glow discharge : 1 – cathode; 2 – anode; 3 - ammeter; 4 – electrode lowering mechanism; 5 – H-shaped cell; 6 - cellophane membrane; 7 – cell with liquid anode (LA); 8 – cell with liquid cathode (LC); 9 – discharge

Рис. 1. Схема тлеющего разряда постоянного тока: 1 – катод; 2 – анод; 3 - амперметр; 4 – механизм передвижения электрода; 5 – Н-образная ячейка; 6 - целлофановая мембрана; 7 – ячейка с жидким анодом (ЖА); 8 – ячейка с жидким катодом (ЖК); 9 – разряд

RESULTS AND ITS DISCUSSION

Qualitatively, the dependences of the change in the concentration of PC for all the studied concentrations and currents and the liquid cathode and anode are similar and have the form shown in Fig. 2, 3. Formally, the kinetic curves are very well (determination coefficient $R^2 > 0.98$) described by a relation of the form:

$$[C(t)] = N - [C_0] \times \exp(-K_D \times t) \qquad (1)$$

where N = const, $[C_0]$ – initial concentration, K_D - rate constant.

An equation of this kind is obtained by integrating the following kinetic law:

$$\frac{d[C]}{dt} = -K_D \times [C] + W_{-} \qquad (2)$$

Solution (2) with the initial condition $C = C_0$ at $t = 0$ has the form

$$[C] = \frac{1}{K_D} \cdot [W - (W - [C_0] \cdot K_D) \cdot \exp(-K_D \cdot t)] \qquad (3)$$

Expressions (2) and (3) mean that, along with decomposition at a rate of $K_D \times [C]$, the reaction zone is fed with paracetamol at a constant rate W. Reversible decomposition of the PC is hardly possible. Therefore, we assume that such behavior of the dependences is due to the fact that decomposition takes place in the near-surface layer (where active particles are formed) and there is a diffusion flux of PC from the solution unaffected by the discharge. This flow provides the rate of W. At long times, this slowly decreasing flux limits the decomposition rate. A similar behavior was observed in [16] for the decomposition of phenol. The results of processing the kinetic dependences according to formula (3) for all the studied currents and concentrations are given in the Table. Degrees of decomposition were found according to the ratio:

$$\alpha = \frac{[C(t)] - [C_0]}{[C_o]} \qquad (4)$$

where $[C(t)]$ – current concentration.

The energy efficiency (the number of decomposed molecules per 100 eV of input energy) was calculated for a time equal to K_D^{-1} using relation (5) as recommended in [16]:

$$\varphi = \frac{([[C_0] - [C(K_D^{-1})]]) \times V \times e \times K_D \times N_{AV}}{P \times M \times 10} \qquad (5)$$

where V = 0.1 L – cell volume; $[C]$ – concentration, mg/L; $e = 1.6\ 10^{19}$ electron charge, C; $N_{AV} = 6.02 \cdot 10^{23}$ – Avogadro number, mol^{-1}; P – power inputted in the discharge, W; M = 151 – PC molar mass $g \cdot mol^{-1}$.

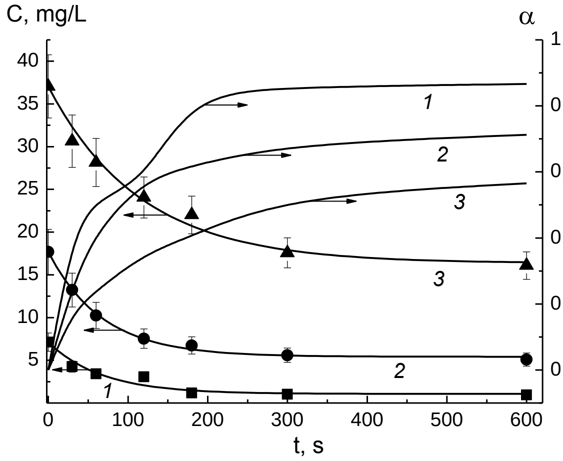

Fig. 2. Dependence of concentration (C) and degree of decomposition (α) on time at a current of 20 mA in a liquid anode. The initial concentrations of PC 1-3 - 6.3, 15.7, 37.5 mg/L, respectively. The dots are an experiment. Lines - calculation according to (1)

Рис. 2. Зависимость концентрации (С) и степени разложения (α) ПЦ от времени при токе 20 мА в жидком аноде. Начальные концентрации ПЦ 1-3- 6,3, 15,7, 37,5 мг/л, соответственно. Точки – эксперимент. Линии – расчет по (1)

Таблица

Константы скоростей (Kd), степени разложения (α) и энергетические эффективности (φ, молекул на 100 эВ)

***Table*. Rate constants (Kd), decomposition degree (α) and energy efficiencies (φ, molecules per 100 eV)**

Discharge current 20 mA						
Concentration, mg/L	Liquid anode			Liquid cathode		
	K_D, s^{-1}	α,	φ	K_D, s^{-1}	α	φ
7.1	$(1.5\pm0.3)\cdot10^{-2}$	0.88	$2.2\cdot10^{-2}$	$(3.9\pm0.5)\cdot10^{-2}$	0.98	$3.2\cdot10^{-2}$
17.7	$(1.5\pm0.1)\cdot10^{-2}$	0.7	$4.4\cdot10^{-2}$	$(1.9\pm0.3)\cdot10^{-2}$	0.91	$3.5\cdot10^{-2}$
37.5	$(8.7\pm0.1)\cdot10^{-3}$	0.57	$4.4\cdot10^{-2}$	$(9.8\pm0.1)\cdot10^{-3}$	0.82	$7.1\cdot10^{-2}$
Discharge current 40 mA						
8.9	$(6.4\pm1)\cdot10^{-2}$	0.82	$6.0\cdot10^{-2}$	$(3.7\pm1)\cdot10^{-2}$	~1	$1.8\cdot10^{-2}$
19	$(1.9\pm0.2)\cdot10^{-2}$	0.76	$3.7\cdot10^{-2}$	$(2.8\pm0.6)\cdot10^{-2}$	~1	$2.5\cdot10^{-2}$
33.8	$(3\pm0.6)\cdot10^{-2}$	0.6	$7.4\cdot10^{-2}$	$(1.6\pm0.3)\cdot10^{-2}$	0.89	$2.4\cdot10^{-2}$
Discharge current 50 mA						
7.8	$(8.4\pm1)\cdot10^{-2}$	0.93	$6.5\cdot10^{-2}$	$(6.8\pm2)\cdot10^{-2}$	~1	$2.3\cdot10^{-2}$
18.2	$(4.7\pm1)\cdot10^{-2}$	0.84	$7.3\cdot10^{-2}$	$(3.4\pm1)\cdot10^{-2}$	~1	$2.5\cdot10^{-2}$
35.6	$(2.5\pm0.4)\cdot10^{-2}$	0.71	$3.2\cdot10^{-2}$	$(1.2\pm0.3)\cdot10^{-2}$	0.95	$1.8\cdot10^{-2}$

Примечание: энергетическая эффективность φ рассчитывалась для времени равного KD-1. Степень разложения α приведена для времени 600 с

Note: energy efficiency φ was calculated for a time equal to K_D^{-1}. The degree of decomposition α is given for a time of 600 s

It is difficult to compare the obtained results with the data of other works in a correct way, due to the use of different types of discharges and reactor designs. In [17], the decomposition of PC under the action of DBD in air (frequency 0-20 kHz, power 500 W, flow rate of the solution circulating in a closed system with a volume of 1500 ml was 0.84 ml/s) was studied. It was found that the decomposition kinetics also obeys the 1st order equation, and the rate constants fall from $7.4\cdot10^{-3}$ to $4.7\cdot10^{-4}$ s^{-1} with increasing concentration from 10 to 100 mg/L.

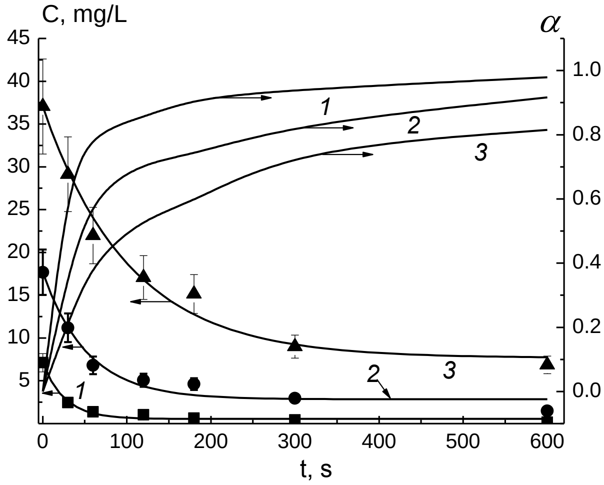

Fig. 3. Dependence of concentration (C) and degree of decomposition (α) on time at a current of 20 mA in a liquid cathode. The initial concentrations of PC 1-3 - 6.3, 15.7, 31.5 mg/L, respectively. The dots are an experiment. Lines - calculation according to (1)

Рис. 3. Зависимость концентрации (C) и степени разложения (α) ПЦ от времени при токе 20 мА в жидком катоде. Начальные концентрации ПЦ 1-3- 6,3, 15,7, 31,5 мг/л, соответственно

Complete decomposition at a concentration of 10 mg/L was achieved in 10 min, and at a concentration of 100 mg/l in 1 h. In [18], the decomposition of PC was studied in DBD in air (the flow rate of the circulating solution was 1.4 ml/s; the frequency and power were not indicated, although the power was measured). The concentration of the solution was 20 mg/L. The maximum degree of decomposition reached 0.9 at a treatment time of 18 min. and voltage 24 kV. The energy efficiency was ~$1\cdot10^{-3}$ molecules per 100 eV. This is about one order of magnitude less than what we have achieved. An energy efficiency close in order of magnitude to our results (~$8\cdot10^{-2}$ molecules per 100 eV) was found in [13] for DBD in a mixture of gases O_2:Ar = = 1:9 (frequency 500-900 Hz, closed in solution system with a volume of 40 ml, PC concentration 25 mg/L, power 0.06 W). PC decomposed completely within ~45 min. Thus, the glow discharge in terms of its indicators (degree of decomposition, decomposition rate, energy efficiency) generally exceeds dielectric barrier discharges.

The following observed regularities of decomposition kinetics can be noted. At a given discharge current, an increase in the PC concentration leads to a decrease in the effective decomposition rate constants and decomposition degree. At a given PC concentration, an increase in the discharge current is accompanied by an increase in the effective constants of the decomposition rates and degrees of decomposition. The energy efficiency of decomposition weakly depends on both the discharge current and the initial concentration. The rate constants found are effective, i.e., they include the concentrations of active species involved in the decomposition reactions of PC. As shown in [19], an increase in the discharge current leads to an increase in the fluxes of almost all active particles in the plasma. This ensures the growth of effective rate constants. The decrease in the constants with increasing concentration indicates that the active species involved in the decomposition of PC are also consumed in other reactions. Indeed, the rate of PC decomposition can be written as:

$$\frac{d[C]}{dt} = -\left[\frac{K_D \cdot V_F}{\sum \beta_i + K_D \cdot [C]}\right] \cdot [C] \qquad (6)$$

where V_F is the rate of entry of active particles from the gas phase, $\Sigma\beta_i$ is the rate of the death of active particles in all processes, except for the reaction with PC. The value in square brackets is the effective rate constant. As the PC concentration increases, the constant drops from $(K_D \cdot V_F)/\Sigma\beta_i$ to $V_F/[C]$. That is, at high concentrations of PC ($K_D \cdot [C] >> \Sigma\beta_i$), all active particles react only with PC. The formation of active species becomes the rate-limiting step, and the reaction must proceed according to the zero order.

According to the achieved degrees of decomposition and rate constants, the action of the discharge on the liquid anode and liquid cathode leads to similar results. But the energy efficiency for a liquid anode is about 2-3 times higher. This is due to the specific features of the electrical characteristics of a DC glow discharge. In a glow discharge, the total voltage drop across the discharge gap is the sum of the voltage drop on the positive column of the discharge and the voltage drops near the electrodes (cathode and anode potential drops). The cathodic potential drop for different metal electrodes is 100-300 V. At the same time, the cathodic potential drop for distilled water lies in the range of 600-800 V [20]. For this reason, at a given discharge current, the total input power for a discharge with a liquid cathode should be 2-3 times greater than for a discharge with a liquid anode. Measurements (Fig. 4) show that, in fact, the power inputted in a discharge with a liquid anode at the same current is approximately 2 times less than for a discharge with a liquid cathode.

CONCLUSION

Thus, exposure to a direct current glow discharge in air is an effective means of purifying water from paracetamol. Almost complete decomposition of PC is achieved in a short time (less than 10 min.) with satisfactory energy consumption. In this case, it is energetically more favorable to use a solution that serves as the discharge anode.

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The authors declare the absence a conflict of interest warranting disclosure in this article.

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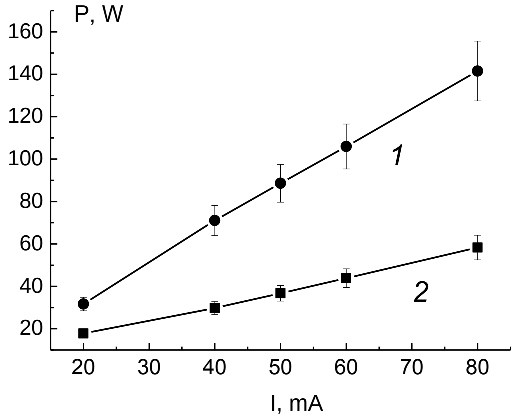

Fig. 4. Dependence of the power inputted in the discharge on the discharge current. 1 - liquid cathode. 2 - liquid anode

Рис. 4. Зависимость мощности, вкладываемой в разряд, от тока разряда. 1 – жидкий катод. 2 - жидкий анод

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