

Electrosynthesis of H₂O₂ from O₂ in a Gas-Diffusion Electrode Based on Mesostructured Carbon CMK-3

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Abstract—Mesostructured carbon CMK-3 (Carbon Mesostructured by KAIST) synthesized by the template method is studied as the electrocatalyst for electrosynthesis of H₂O₂ from O₂ in a gas-diffusion electrode (GDE) in alkaline and acidic solutions. The texture characteristics of the original material and its mixture with hydrophobizer (polytetrafluoroethylene) are studied by the method of low-temperature nitrogen adsorption. The rate constants for hydrogen peroxide decomposition on these materials in alkaline and acidic solutions are calculated. Kinetic parameters of oxygen reduction in alkaline and acidic solutions are determined as well as the capacitance of gas-diffusion electrodes based on mesocarbon. The selectivity of the electrocatalyst is estimated by finding the current fraction γ consumed in oxygen reduction to hydrogen peroxide. Data on the kinetics of hydrogen peroxide accumulation during electrosynthesis of H₂O₂ from O₂ are obtained. The acidic solution of hydrogen peroxide with the concentration more than 3 M is obtained with the current efficiency higher than 80%.

Keywords: electrosynthesis, gas-diffusion electrode, template synthesis, mesostructured carbon, hydrogen peroxide, electrocatalyst, double layer capacitance, current efficiency

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INTRODUCTION

Mesostructured mesoporous carbon materials are a new class of promising materials with various applications in electrochemistry. It is well known that the main advantage of mesostructured carbon materials (MCM) synthesized by the template method is their high surface area, ordered mesoporous structure, and high conductivity [1]. The authors of [1–5] studied the use of various mesostructured materials in supercapacitors based on the electric double layer. In certain studies, MCM were used as the composite electrodes in lithium-ion batteries [6]. Nanoparticles of metal oxides capable of the fast Faradaic reactions, such as copper oxide, were introduced into MCM in order to increase the capacity [7, 8]. The composite Li₂MnSiO₄ with MCM was used as the anode [9]. MCM with the surface covered with nanoparticles of platinum-group metals as the catalysts can be used as fuel cell electrodes [10]. It should be noted that the potential application field of MCM is constantly extended. It was reported [11] that mesostructured carbon was used as the template in the synthesis of mesoporous oxides of different metals, e.g., magnesium oxide. By using a rotating disk electrode, it was

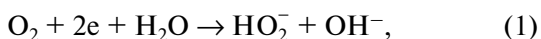
shown [12–15] that mesoporous carbon can be used in the electrosynthesis of hydrogen peroxide from oxygen in various aqueous electrolytes. It was noted that the structure of such material favors the fast removal of synthesized H₂O₂ from the reaction zone, which prevents its chemical decomposition or further electroreduction to water. It was shown [15] that mesostructured carbon can be used for mineralization of Methyl Orange in a weakly acidic aqueous solution at indirect oxidation of Methyl Orange by electrosynthesized H₂O₂.

It is known that the high electrochemical activity of carbon-based gas-diffusion electrodes (GDE) in electrosynthesis of H₂O₂ from O₂ depends on their structure characteristics which are determined to the large extent by the texture of original materials, in our case, original carbon and its composites with the hydrophobizer-binder, e.g., polytetrafluoroethylene (PTFE, fluoroplastic-4D, or F-4D) [16, 17].

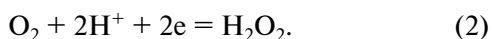
Within the frames of the Green Chemistry approach [18, 19], the special place belongs to the wide use of aqueous solutions of hydrogen peroxide as the versatile environmentally clean oxidant. As the highly active redox reagent, hydrogen peroxide allows

various organic and inorganic compounds to be oxidized both in alkaline and acidic solutions [20–23].

However, the wider use of hydrogen peroxide is limited by its relatively high cost if it is synthesized by traditional technologies [21]. In connection with this, the studies on the development of alternative methods for producing aqueous hydrogen peroxide solutions with different pH, aimed at simplification and cheapening of the synthesis of ready-to-use target product, gain in importance. The method of preparation of dilute hydrogen-peroxide solutions by the cathodic reduction of oxygen on carbon materials, which was extensively studied in the past 30 years [16, 17, 20–24], meets these requirements. The synthesis proceeds by the following reactions. In alkaline solutions (the Berl process [24]),



in acidic solutions,



It should be noted that this method is wasteless and makes it possible to synthesize H₂O₂ in the places of its consumption in the form of aqueous solutions with different pH to be used as the finished product without preliminarily extraction of H₂O₂, which considerably lowers its cost [25]. At present, a large number of carbon materials already studied as the electrocatalysts for 2-electron reduction of oxygen are known and the search for the new promising materials goes on [17, 26]. Taking into account the aforementioned properties of mesostructured carbon materials and the results on oxygen reduction on the rotating disk electrode made of MCM [12–15], the studies on their application as carbon electrocatalysts for electrosynthesis of H₂O₂ from O₂ in GDEs seem to be promising.

The following main requirements are imposed on carbon materials: high selectivity (γ) with respect to two-electron oxygen reduction (reactions (1) and (2)) and the low catalytic activity as regards the heterogeneous decomposition of synthesized hydrogen peroxide [17]. The rate of hydrogen peroxide accumulation in catholyte is determined by the balance between the rates of its formation and decomposition [27].

The goal of this study is to elucidate how efficient the mesostructured mesoporous carbon CMK-3 [28] synthesized by template method is as the electrocatalyst in the synthesis H₂O₂ from O₂ in gas-diffusion electrodes in alkaline and acidic solutions.

To attain this goal, the investigation program included solving the following problems: determination of the texture characteristics for the original mesocarbon and its mixtures with hydrophobizer (fluoroplastic-4D) and their effect on the electrochemical activity of GDE and the efficiency of electrosynthesis, determination of rate constants for hydrogen peroxide decomposition on these materials, determination of γ , determination of kinetic parameters of oxygen reduc-

tion in a GDE based on mesocarbon and its electric double layer capacitance, finding the data on the kinetics of hydrogen peroxide accumulation and its current efficiency (CE) during the electrosynthesis.

EXPERIMENTAL

CMK-3 Synthesis Procedure

Carbonaceous materials were synthesized by the procedure described in [28, 29]. Mesostructured silicate material SBA-15 was used as the template [30]. The replica plating procedure can be conditionally divided into 3 stages: (1) introduction of the carbon source into the silicate matrix; (2) carbonization of the carbon source; (3) removal of the silicate template. According to the original procedure, the introduction of the carbon source meant the two-fold impregnation of the silicate sample with saccharose solutions. The solution for the first impregnation was prepared from 5 mL water, 1.25 g saccharose, and 0.14 g sulfuric acid per g of silicate matrix. For the second impregnation, we mixed 5 mL water, 0.8 g saccharose, and 0.09 g sulfuric acid. Sulfuric acid was used as the catalyst of the saccharose hydrolysis to fructose and glucose and their carbonization. After each impregnation, the substance was dried at 100°C for 6 h; simultaneously with the water removal, the polycondensation and dehydration of fructose (caramelization) occurred. Then, the temperature was raised to 160°C at which the further carbonization proceeded for 6 h and the substance acquired the grayish-brown color. The further carbonization was carried out in the nitrogen flow at 850–900°C (temperature rise for 2.5 h, exposure for 2.5 h). The silicate template was removed by its dissolution in NaOH solution at 100°C for 16 h. In view of the low wettability of the matrix with water, its dissolution after carbonization was carried out in 1 M NaOH solution in ethanol-water mixture. After filtering or centrifuging, the silicate residues were dissolved in 1 M NaOH solution at 80°C for 6 h. After the second stage of dissolution, the deposit was filtered or centrifuged again, washed with distilled water, and dried in air at 120°C.

Electrochemical Testing of GDE

The texture properties of the original mesostructured carbon and its mixtures with hydrophobizer-binder were determined by the method of nitrogen low-temperature adsorption as in [26] with the use of ASAP 2420 (Micromeritics, USA).

Insofar as our attention was focused exclusively on the GDE working layer, we prepared also the composites corresponding to the working-layer composition. Table 1 shows the results of measuring the texture properties of the original mesocarbon and the composites of GDE working layers.

Table 1. Physicochemical characteristics of mesostructured carbon and composites reproducing the GDE working layers (*)

Sample	Content, wt %		S_{BET} , m ² /g	V_{sp} , cm ³ /g	μ , cm ³ /g	V/μ	R , nm	$\Psi_{<10 \text{ nm}}$, %	S/V , m ² /cm ³
	F-4D	CMK-3							
1	0	100	963	1.26	0.096	13.12	4.05	69.7	764
2	10	90	738	0.98	0.042	23.33	3.95	54.7	753
3	20	80	565	0.79	0.021	37.62	3.95	55.0	715
4	40	60	339	0.53	—	—	3.75	59.5	640
5	60	40	156	0.26	—	—	3.78	47.2	600
6	70	30	105	0.18	—	—	3.81	62.1	583

* S_{BET} is the specific surface area determined by the BET method; V_{sp} is the total specific volume of pores; μ is the specific volume of micropores determined by the t -plot method; R is the position of the maximum in the size distribution of pores; $\Psi_{<10 \text{ nm}}$ is the volume fraction of pores with the size below 10 nm; S/V is the surface area of the working layer normalized per volume.

The constants of chemical decomposition (K_{chem}) of hydrogen peroxide in alkaline (0.5 M KOH) and acidic [0.5 M K₂SO₄ : 0.1 M H₂SO₄ (3 : 1)] solutions on original mesocarbon and its mixtures with F-4D were determined by the procedure described in [31]. The initial concentration of H₂O₂ for determination of K_{chem} was 0.23 M. Table 2 shows these results.

Gas-diffusion electrodes were prepared as bilayer planar disks with diameter of 30 mm according to the procedure described in detail in [17]. As the hydrophobizer-binder, we used water-ethanol suspension of fluoroplastic-4D with 62 wt % of dry substance and 8 wt % of stabilizer. The content of hydrophobizer in the working-layer active mass was varied from 10 to 70 wt %. The barrier-layer composite contained 50 wt % F-4D and 50 wt % acetylene black A 437-E.

Table 2. Rate constants for chemical decomposition of hydrogen peroxide on original mesocarbon and its mixtures with F-4D in alkaline and acidic solutions

Electrolyte composition: mesocarbon + F-4D, wt %	K_{chem} , h ⁻¹	
	0.5 M KOH	(0.5 M K ₂ SO ₄ : 0.1 M H ₂ SO ₄) (3 : 1)
100.0 + 0.0	1.80	0.12
90.0 + 10.0	0.80	0.10
80.0 + 20.0	0.44	0.09
60.0 + 40.0	0.24	0.08
40.0 + 60.0	0.09	0.06
30.0 + 70.0	0.06	0.05

The experiments on electrochemical testing of GDE were carried out in a thermostated electrochemical cell similar to that used in [17]. Before the experiments, the electrodes were impregnated in 0.5 M KOH at the current of 0.1 A for 1 h and the electrolyte porosity was determined by hydrostatic weighing in water. On the electrodes under study, the current fraction γ was determined in alkaline and acidic solutions. For this purpose, the time required for absorption of a certain amount of oxygen was determined by the gasometric method [27]. Table 3 shows the results of measurements. It deserves mention that the value γ includes not only the “direct” consumption of oxygen, but also the oxygen that appears after the heterogeneous decomposition of hydrogen peroxide in the electrode pore volume (see Table 2). The current fraction consumed in one or another stage was determined not only by the electrosynthesis conditions but also by the electrode material and its structure. Hydrogen peroxide also decomposed in the catholyte volume, beyond the electrode. Thus, γ characterizes the processes that proceed in the electrode and the current efficiency shows the efficiency of the overall electrosynthesis of the target product. Naturally, γ is equal to unity if all oxygen is reduced to H₂O₂, and is equal to zero if all oxygen is reduced to water or hydroxide ions.

The electric double layer capacitance was determined as in [26] by recording cyclic voltammograms in 3 M KOH and 1 M H₂SO₄. Inert gas was passed through the gas chamber. The charging current was measured at the potential scan rate of 0.5–5 mV s⁻¹ in the potential intervals of –0.14–0.0 V in alkali and 0.2–0.6 V in acid. The current of double layer charging was calculated at –0.075 V in alkali and 0.4 V in acid. Then, the electrode capacitance was determined from the tangent of the dependence of the charging current on the potential scan rate (Fig. 1). The polarization curves of oxygen electroreduction were recorded at the potential scan rate of 1 mV/s and then curves were

Table 3. Characteristics of electrodes of mesostructured carbon with different content of F-4D

Content of F-4D, wt %	Electrolyte porosity, vol %	Capacitance per electrode geometrical surface, F/cm ²	Capacitance per g of carbon, F/g	Coefficient in equation $E^* = -(a + b \log i)$, mV		γ^{**} , fractions of unity at the current density (mA/cm ²) and the potential ($-E^{***}$, V)			
				<i>a</i>	<i>b</i>	50	100	150	190
0.5 M KOH									
60	17	0.190	15.1	190	73	0.88 (0.36)	0.86 (0.47)	0.88 (0.58)	0.87 (0.66)
70	12	0.129	13.6	180	64	0.92 (0.33)	0.90 (0.40)	0.91 (0.46)	0.94 (0.50)
0.5 M K ₂ SO ₄ : 0.1 M H ₂ SO ₄ (3 : 1)									
60	17	0.437	34.7	140	110	0.87 (0.36)	0.94 (0.49)	0.93 (0.59)	0.95 (0.70)
70	12	0.216	22.8	120	110	0.91 (0.31)	0.92 (0.38)	0.92 (0.43)	0.90 (0.47)

**E* is the electrode potential, *i* is the current density, mA/cm².

**Current fractions consumed in the reduction to HO₂⁻ and H₂O₂.

***Electrode potentials at which γ is determined.

plotted in Tafel coordinates in order to determine the parameters of linear segments. Table 3 shows the results.

Based on the recorded potentiodynamic curves of oxygen reduction on GDEs with different contents of F-4D in alkaline and acidic electrolytes (are not shown in this paper), we selected the electrodes with the optimal content of hydrophobizer, namely, 60 and 70 wt %. Figure 2 shows the polarization curves of oxygen electroreduction on these electrodes in alkaline and acidic solutions.

The kinetics of hydrogen peroxide accumulation was studied by carrying out preparative electrosynthesis in a glass cell with anodic and cathodic compartments separated by a cation-exchange membrane MF-4SK-100 by the procedure described in [17]. A platinum plate with the surface of 3 cm² served as the anode. The GDE was placed between the catholyte and gas chambers. Oxygen was uninterruptedly supplied into the electrode from its rear side through the gas chamber at atmospheric pressure. The preparative electrosynthesis was carried out in the galvanostatic

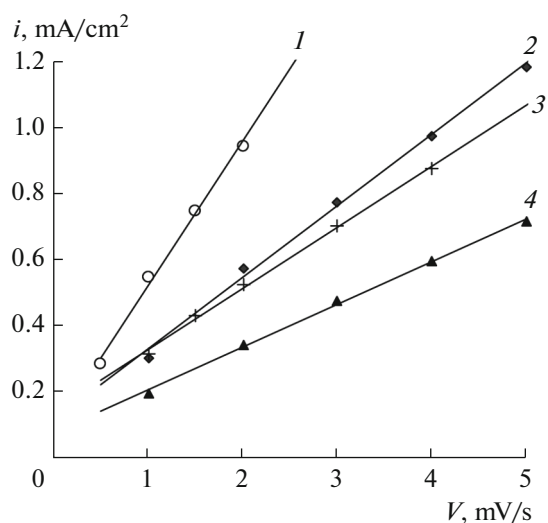


Fig. 1. Dependences of the current density of electric double layer charging on the potential scan rate on electrodes with the fluoroplastic-4D content, wt %: (1, 3) 60 and (2, 4) 70 in (1, 2) 1 M H₂SO₄ and (3, 4) 3 M KOH.

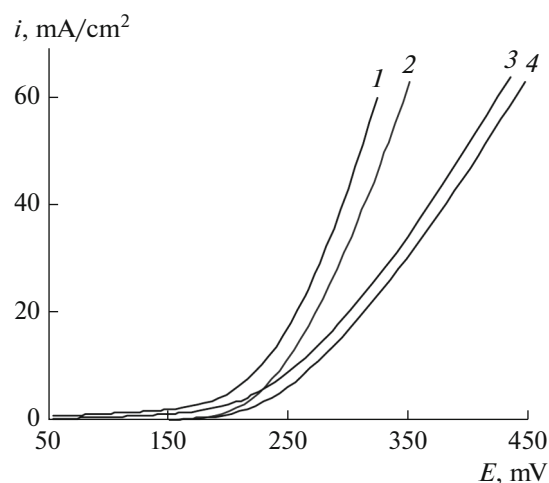


Fig. 2. Polarization curves of O₂ reduction in GDE with (1, 3) 70 and (2, 4) 60 wt % F-4D in (1, 2) acidic and (3, 4) alkaline solutions.

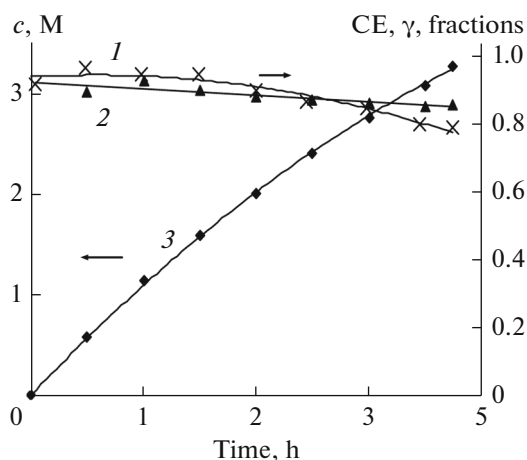


Fig. 3. Results of preparative electrosynthesis of H_2O_2 from O_2 in GDE with 70 wt % F-4D in acidic solution at the overall current density of 150 mA/cm^2 : (1) γ , (2) current efficiency in H_2O_2 , (3) H_2O_2 concentration.

mode in the GDE with 70 wt % F-4D at the overall current density of 150 mA/cm^2 in $0.5 \text{ M K}_2\text{SO}_4 : 0.1 \text{ M H}_2\text{SO}_4$ (3 : 1) at $20\text{--}22^\circ\text{C}$. The anolyte represented $2 \text{ M H}_2\text{SO}_4$ solution. The catholyte was continuously stirred with air at a rate of 45 mL/min . The potential was measured at the frontal side of GDE with respect to the silver/silver-chloride electrode (SCE). For electrolysis and polarization measurements, we used a potentiostat IPC-Pro MF (Russia). The current efficiency with respect to hydrogen peroxide was determined every hour during 7.5 h electrosynthesis. The quantitative content of hydrogen peroxide in catholyte was determined by permanganatometry [32]. Figure 3 shows the results of preparative electrosynthesis of H_2O_2 .

RESULTS AND DISCUSSION

The results shown in Table 1 suggest that in the original MCM CMK-3, the specific volume of micropores was only 7.6% of the total pore volume. After the addition of 10 wt % fluoroplastic-4D, the micropore volume was more than halved and after the addition of 40 wt % F-4D, the specific volume of micropores could not be determined by the method we used (t -plot), i.e., micropores were the first to be blocked by the hydrophobizer-binder. As the content of F-4D increased from 0 to 70 wt %, the specific surface S_{BET} decreased more than 9-fold and the total specific volume of pores decreases 7-fold. The quantities R and $\Psi_{<10\text{nm}}$ passed through extremum as the F-4D content increased, whereas the surface per unit volume decreased linearly ($R_2 = 0.98$).

The data in Table 2 show that the rate constant for heterogeneous decomposition of H_2O_2 k_{chem} in alkaline solutions is high, more than one-order-of-magni-

tude higher than its value in acidic solution. As the F-4D content in the mixture increased from 0 to 70 wt %, the value of k_{chem} decreased 30-fold in alkaline medium and 2.4-fold in acidic medium. The so high k_{chem} in alkaline solution is associated first of all with the thermodynamic instability of hydrogen peroxide in alkaline solutions and, as a consequence, with the high rate of decomposition on the well-developed heterogeneous surface of the original mesocarbon and its mixtures, i.e., with the well-developed surface of the H_2O_2 /mesocarbon contact.

Figure 1 shows that as the F-4D concentration increases, the current of double-layer charging decreases, which is associated with the decrease in the electrolyte-wetted surface with the decrease in the volume of electrolyte pores (Table 3). In the acidic electrolyte, the current is higher than in alkaline electrolyte. This is explained by the difference in the potential ranges and types of electrolyte ions.

The analysis of results in Table 3 shows that the coefficient b is higher in the acidic solution as compared with the alkaline solution. Polarization is higher in alkaline solution than in acidic one, which agrees with the higher coefficient a in alkali.

As the F-4D concentration decreased from 70 to 60 wt %, the volume of electrolyte pores (electrolyte porosity) increased, in our case, by a factor of 1.4. The capacitance of electrodes also increased by a factor of 1.5 both in alkali and acid, the BET surface of the electrode mass increased by the same factor (Table 1). Probably, this is the reason for a certain decrease in γ in alkaline solution, because as the carbon surface increases, the rate of H_2O_2 decomposition increases. In acidic solution, γ remained unchanged because the rate constant for H_2O_2 decomposition is several times lower in acid as compared with alkali (Table 2). The slope of polarization curves remains unchanged, and coefficient a increases. The polarization of the electrode becomes higher, which may be associated with the decrease in the surface of three-phase boundary carbon/electrolyte/oxygen with the increase in electrolyte porosity.

Earlier [33], we performed experiments with furnace black SN600 ($705 \text{ m}^2/\text{g}$) with the surface of the same order of magnitude as for MCM studied here, the volume fraction of micropores in the total volume of pores in the original material was 6.3%. It was of interest to compare these materials, for instance, the samples containing 70 wt % F-4D. The polarization and the γ value were virtually the same as for electrodes of furnace black CN600 with the same content of hydrophobizer, but the electrolyte porosity was higher by 20% and the capacitance was higher by a factor of 3.3 in alkali and 3.9 in acid. The specific volume of all pores (cm^3/g) decreased as compared with the original carbon material by a factor of 10.2 in CN600 and 7 in MCM. The BET surface for electrodes with mesostructured carbon was higher as compared with

CN600 by a factor of ~1.7 and decreased with the addition of 70 wt % F-4D as compared with the initial value: by a factor of 11.75 for CN600 and a factor of 9.2 for mesostructured carbon. Hence, the surface of furnace black is better blocked by fluoroplastic as compared with the MCM surface.

The polarization curves of O₂ electroreduction in Fig. 2 depend on the quantitative content of F-4D and the electrolyte solution composition: the electrochemical activity of the GDE with 70 wt % F-4D is noticeably higher than that of GDE with 60 wt % and, in acidic electrolyte, the electroreduction of O₂ proceeds with the lower overpotential. Based on these data, for preparative electrosynthesis of H₂O₂ we have chosen the GDE with 70 wt % F-4D and the acidic solution containing 0.5 M K₂SO₄ : 0.1 M H₂SO₄ (3 : 1).

The results of preparative electrosynthesis shown in Fig. 3 point to the high efficiency of electrode operation: the kinetics of H₂O₂ accumulation obeys the parabolic law, the current efficiency is 80%, the selectivity of the process γ is higher than 0.8. The fact that the curve of the target product accumulation obeys the parabolic law is associated with the insufficient decomposition of this product on the heterogeneous surface in the electrode porous volume. In 7.5 h, the hydrogen peroxide concentration exceeding 3 M was obtained with the current efficiency higher than 80%. It deserves mention that in the GDE containing 70 wt % F-4D, in its porous volume, the well-developed three-phase boundary is created, as well as the system of well-connected pores for the highly efficient delivery of reagent (oxygen) and electrolyte to this boundary and the removal of synthesized product from the porous volume. This unambiguously follows from the stable operation of the electrode in the potential interval of 0.48–0.55 V during the studied time interval; in the process, the solution volume changed from 20.0 to 27.4 mL. The obtained results undeniably point to the importance of mesostructured carbon CMK-3 as the electrocatalyst for electrosynthesis of H₂O₂ from O₂ in gas-diffusion electrodes.

CONCLUSIONS

As the amount of hydrophobizer-binder fluoroplastic-4D increases in the mixture from 0 to 70 wt %, the specific surface of MCM (S_{BET}) decreases more than 9-fold, the total specific volume of pores (V_{sp}) decreases more than 7-fold, the specific volume of micropores (μ) disappears at the F-4D content of about 40 wt % and cannot be determined by the t -plot method, i.e., the micropores are the first to be blocked.

The rate constant for heterogeneous decomposition of H₂O₂ k_{chem} in alkaline solution is high: more than one order of magnitude higher than in acidic solution. As the content of F-4D in the mixture

decreases from 0 to 70 wt %, k_{chem} decreases 30-fold in alkaline and 2.4-fold in acidic medium.

As the fluoroplastic concentration increases, the current of charging of the electric double layer decreases, which is associated with the decrease in the volume of electrolyte pores. In the acidic electrolyte the current is higher than in the alkaline electrolyte. This is explained by the difference in potential intervals and the types of electrolyte ions. The selectivity of two-electron reduction of oxygen γ in GDEs with 60 and 70 wt % F-4D varies from 86 to 95% for the overall current densities from 50 to 190 mA/cm² and the relatively low electrode polarization.

The results of preparative electrosynthesis of the target product in the GDE with 70 wt % F-4D point to the high efficiency of electrode operation: the kinetics of H₂O₂ accumulation obeys the parabolic law, the current efficiency exceeds 80%, the selectivity of the process γ is higher than 0.8. In 7.5 h electrosynthesis, the hydrogen peroxide concentration above 3 M was obtained in 0.5 M K₂SO₄ : 0.1 M H₂SO₄ (3 : 1) with the current efficiency exceeding 80%.

Based on the totality of results obtained, it can be said that mesostructured carbon material, as compared with nonstructured material (CN600), exhibits the more ordered mesoporous structure which provides the higher accessibility of the GDE surface at the formation of the electric double layer and the high efficiency of the inner-pore transfer. This favors the high electrochemical activity and stable operation of GDE.

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