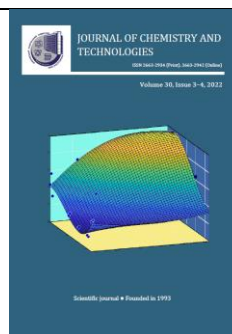




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LEAD DIOXIDE-PERFLUOROOCETANESULFONATE COMPOSITES: ELECTROSYNTHESIS AND APPLICATION

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Abstract

The regularities of electrosynthesis, properties, and electrocatalytic activity of the composites lead dioxide-perfluorooctanesulfonate have been investigated. Comprehensive data on the regularities of PbO₂ nucleation in the presence of perfluorooctane sulfonate in the deposition electrolyte are obtained. The kinetics of the deposition of composites in the presence of surfactant in the deposition electrolyte has been studied, it is shown that the effect of inhibiting of PbO₂ deposition, which is evident more with increasing of fluorine-carbon chain length, no longer evident when the chain reaches eight carbon atoms. The morphology and phase composition of composites were studied. The influence of the composition of composites on the reaction of oxygen evolution and oxidation of 4-chlorophenol was studied.

Keywords: potassium perfluorooctanesulfonate; lead dioxide; electrosynthesis; nitrate electrolyte; 4-chlorophenol.

КОМПЗИТИ ПЛЮМБУМ(IV) ОКСИД-ПЕРФТОРОКТАНСУЛЬФОНАТ: ЕЛЕКТРОСИНТЕЗ ТА ЗАСТОСУВАННЯ

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Анотація

Досліджено закономірності електросинтезу, властивості та електрокаталітичну активність композитів плюмбум(IV) оксид-перфтороктансульфонат. Отримано комплексні дані про закономірності нуклеації PbO₂ за наявності перфтороктансульфонату в електроліті осадження. Вивчено кінетику осадження композитів за наявності в електроліті осадження ПАР, показано, що ефект інгібування осадження PbO₂, що проявляється більше зі збільшенням довжини флуор-карбонового ланцюга, за досягнення ланцюга у вісім атомів карбону більше не проявляється. Досліджено морфологію та фазовий склад композитів. Вивчено вплив складу композитів на реакцію виділення кисню та окиснення 4-хлорфенолу.

Ключові слова: калію перфтороктансульфонат; плюмбум(IV) оксид; електросинтез; нітратний електроліт; 4-хлорфенол.

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Introduction

PbO₂ is traditionally used as an anode material in applied electrochemistry. Due to the fact that the potentials of the anode during electrolysis reach strongly positive values, the ozone evolution becomes possible at the anode [1; 2]. Oxygen atoms, which are part of the oxide layer, are known to play a significant role in the mechanism of the processes of anodic evolution of oxygen and ozone, [3]. It is noted [4; 5], that it is the vacancies of O²⁻ ions in the surface layers of the oxide material that affect the electrocatalytic properties and, most importantly, the stability of oxide materials in the process of real electrolysis. The composition of the oxide layers on lead dioxide anodes can, in principle, be varied. For example, the introduction of antimony in the composition of PbO₂ anodes [6], used in the electrolysis of sulfuric acid solutions, increases the amount of oxygen in the surface layer, which leads to a decrease in the overvoltage of O₂ evolution and an increase in the anode corrosion rate. The literature describes lead dioxide anodes doped with cerium atoms [7], silver [8], bismuth [9] and other elements. It is noted that in the case of joint electrodeposition of PbO₂ with cerium [7] or bismuth [9] these elements are included in the composition of the anode deposit. Bismuth compounds reduce the rate of electrocrystallization of lead dioxide due to their adsorption on the surface of the growing PbO₂ deposit. This affects the electrochemical properties of the resulting anodes. For example, the oxygen evolution overvoltage on bismuth-doped lead dioxide anodes increases. The literature also describes methods for modifying the surface of PbO₂ anodes with fluoride ions [5; 10]. This treatment reduces the number of vacancies in the lead dioxide layer, which significantly increases the stability of the developed anode material.

One of the new directions in the study of lead dioxide coatings is the synthesis of metal oxide-surfactant composites [11]. There are few publications about materials of this type in the literature [12–15]. Surfactants with low surface activity, which contain fluoride ions, are also of interest [16; 17]. The hydrophobicity of the F-C tail is much greater than that of the H-C. There is a niche for research here, because such electrodes are able to influence the binding energy of oxygen-containing particles by hydrophobizing the surface. According to the literature [18], this effect will create conditions for greater reactivity in the oxidation reactions of

organic compounds. In this case, we were interested in finding out the effect of the length of the perfluorinated hydrocarbon chain on the properties of the coating. In this work, we investigated the electrodeposition and properties of the PbO₂ composite with a fluorinated surfactant heptadecafluorooctanesulfonic acid potassium salt.

Heptadecafluorooctanesulfonic acid potassium salt is a salt derivative of heptadecafluorooctanesulfonic acid that is classified under the family of poly/perfluoroalkyl alkyl substances. Their unique properties allow for numerous applications such as industrial detergents, fire-fighting foams, and water- and greaserepellent materials [19]. Perfluoroalkyl acids are more broadly referred to as perfluorosulfonic acids and have the configuration C_nF_{2n+1}SO₃H [20]. Heptadecafluorooctanesulfonic acid potassium salt contains 8 perfluoroalkyl carbon atoms [21]. The potassium salt of perfluorosulfonic acid, as a surfactant, has both hydrophobic (perfluoroalkyl group) and hydrophilic (sulfonate group) components [22].

Having in mind the above mentioned, it is of interest to study the regularities of electrosynthesis, properties, and electrocatalytic activity of the composites involved.

Experimental and Methods

All chemicals were purchased from ALSI (Ukraine). Kinetics of lead dioxide composites electrodeposition was studied on a Pt rotating disk electrode (Pt-RDE) with the surface area 0.19 cm² by steady-state voltammetry, linear potential sweep voltammetry and chronoamperometry. Voltammetry system SVA-1BM was used for the RDE experiments. The surface of electrode was treated with a freshly prepared mixture (1 : 1) of concentrated H₂SO₄ and H₂O₂ before each experiment [17]. Such pre-treatment allows one to stabilize the electrode surface and to obtain reproducible cyclic voltammograms in the background electrolyte (0.1 M HNO₃). The standard temperature-controlled three-electrode cell was used for voltammetry measurements. All potentials were recorded and reported vs. Ag / AgCl / KCl (sat.).

Electrodeposition was studied in 0.1 M HNO₃ + 0.01 M Pb(NO₃)₂. Surfactant was added into the deposition electrolyte as aqueous solutions with various concentrations.

Surface morphology of the composites involved was studied by scanning electron microscopy (SEM) with Stereoscan 440 LEO

microscope. X-ray powder diffraction data were collected on a STOE STADI P automatic diffractometer equipped with linear PSD detector (transmission mode, $2\theta/\omega$ -scan; Cu $K\alpha_1$ radiation, curved germanium (1 1 1) monochromator; 2θ -range $6.000 \leq 2\theta \leq 102.945$ ° 2θ with step 0.015 ° 2θ ; PSD step 0.480 ° 2θ , scan time 50 s/step).

Crystal structures of the phases were refined by the Rietveld method with the program FullProf.2k, applying a pseudo-Voigt profile function and isotropic approximation for the atomic displacement parameters. Microstructural parameters (i.e., size of coherently diffracting domains accepted as average apparent crystallite size D , and average maximum strain ϵ) were identified by isotropic line broadening analysis using simplified integral breadth methods for reflections with maximal intensities: (110) for α - PbO_2 and (111) for β - PbO_2 .

Adsorption measurements were carried out on PbO_2 powder (Merck) without polarization in the presence of 0.1 M KCl (indifferent electrolyte). Adsorption equilibrium was achieved within 24 hours. Parameters of perfluorinated hydrocarbons adsorption were calculated using the Frumkin equation [23]:

$$Bc = \frac{\theta}{1-\theta} \exp(-2\alpha\theta),$$

where B is adsorption constant;

c – equilibrium concentration;

θ – surface coverage;

α – interaction parameter.

Oxygen evolution reaction was investigated by steady-state polarization on computer controlled EG & G Princeton Applied Research potentiostat model 273A in 1M $HClO_4$.

The electrooxidation of organic compounds was carried out in divided cell at $j_a = 50$ mA cm^{-2} . Solution, containing phosphate buffer (0.25 M Na_2HPO_4 + 0.1 M KH_2PO_4) + 10^{-4} M organic compound (pH=6.55) was used as anolyte; phosphate buffer as catholyte. Composite PbO_2 -perfluorooctanesulfonate electrodes with surface area 2.5 cm^2 were used as anodes. Stainless steel was used as cathode.

The concentration of the organic substance during the electrolysis was measured spectrophotometrically in the ultraviolet and visible region (wavelength range 200-350 nm) with Kontron Uvikon 940 spectrometer.

Reaction products were analyzed by high performance liquid chromatography (HPLC) with Shimadzu RF-10A xL instrument equipped with an Ultraviolet SPD-20AV detector and a 30 cm Discovery® C18 column.

Results and Discussion

Chronoamperograms on Pt-DE were obtained in order to investigate the initial stages of lead oxide deposition. A typical I-t PbO_2 deposition curve is shown in Fig. 1. The chronoamperogram can be conditionally divided into several characteristic sections: current jump in the initial period of electrode polarization, which corresponds to the charge of double electric layer; induction period due to the time required to start phase formation; the maximum current due to the decrease in the concentration of electroactive particles in the near-electrode zone and the achievement of the quasi-stationary current [24].

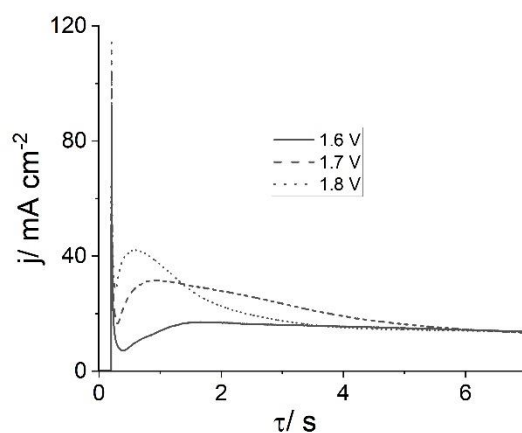


Fig. 1. J-t curves of PbO_2 -perfluorooctanesulfonate electrodeposition at different potentials. 1 - 1.6; 2 - 1.7; 3 - 1.8 V. Deposition solution is 0.1 M $Pb(NO_3)_2$ + 0.1 M HNO_3 + 3×10^{-4} M $C_8F_{17}SO_3K$

Consider the regularities of PbO_2 deposition in the presence of $C_8F_{17}SO_3K$ in the deposition

electrolyte. On typical cyclic voltammograms, the anode branch of the curve (Fig. 2) at potentials

above 1.4 V is characterized by an exponential increase in current, which corresponds to the simultaneous reactions of oxidation of lead (II) and the oxygen evolution. On the cathode branch of the curve, a current peak can be observed due to the reduction of lead oxide at potentials between 0.8 and 1.2 V. As has been pointed earlier [16], inhibition of PbO_2 formation was

observed in the presence of fluorine-containing surfactants in the deposition electrolyte. As the length of F-C chain increases, this effect was more pronounced. As one can see from Fig. 2, when a certain length of the perfluorinated hydrocarbon chain is reached, namely 8 atoms, the constants cease to change.

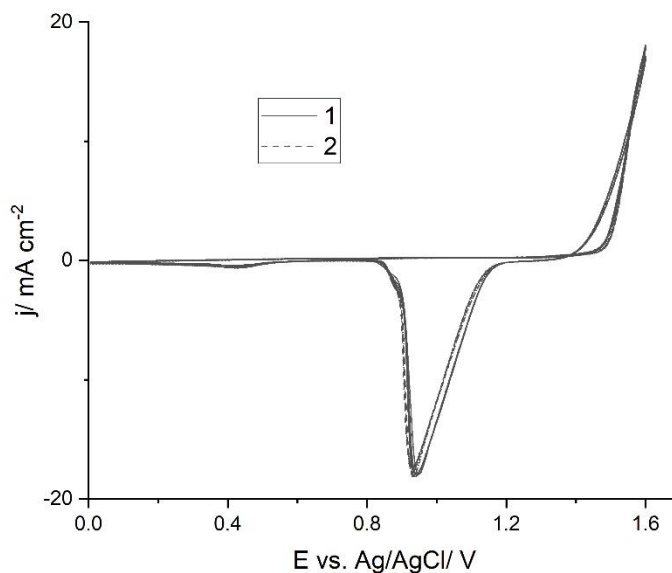


Fig. 2. CV curves (scan range 0.0-1.6 V) for PbO_2 (1) and $\text{PbO}_2\text{-C}_8\text{F}_{17}\text{SO}_3\text{K}$ (2) in 0.1 M $\text{Pb}(\text{NO}_3)_2$ + 0.01 M HNO_3 + 3×10^{-4} M $\text{C}_8\text{F}_{17}\text{SO}_3\text{K}$. Scan rate is 50 mV s^{-1}

The adsorption of surfactants on the PbO_2 powder was investigated in the background solution in the absence of polarization [16]. The adsorption of perfluorooctanesulfonate is satisfactorily described by the Frumkin isotherm (Fig. 3, correlation factor 0.999). Adsorption parameters were calculated according to this

isotherm, which are unity for interaction parameter and energy of adsorption interaction ($-\Delta G$) $25.12 \pm 0.01 \text{ kJ mole}^{-1}$ that indicates the slight interaction between adsorbed molecules and specific character of adsorption, respectively.

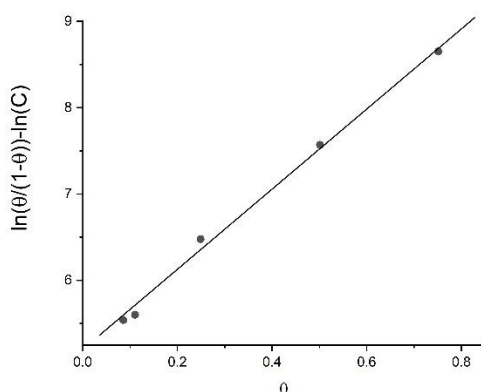


Fig. 3. Adsorption isotherm of $\text{C}_8\text{F}_{17}\text{SO}_3\text{K}$ on PbO_2 powder in the absence of polarization

Potentiometric titration revealed a shift of the zero charge pH of the oxide to alkali region, when $\text{C}_8\text{F}_{17}\text{SO}_3\text{K}$ is present in the solution, which suggests adsorption occurring not only as a result

of electrostatic attraction of anionic surfactant to a positively charged PbO_2 surface, but also as a result of some specific interaction; which consistent with the simulated data on material

balance of adsorption [17] under conditions of chemical interaction of $C_8F_{17}SO_3K$ with the electrode surface.

The morphology of the deposits was investigated by scanning electron microscopy (Fig. 4).

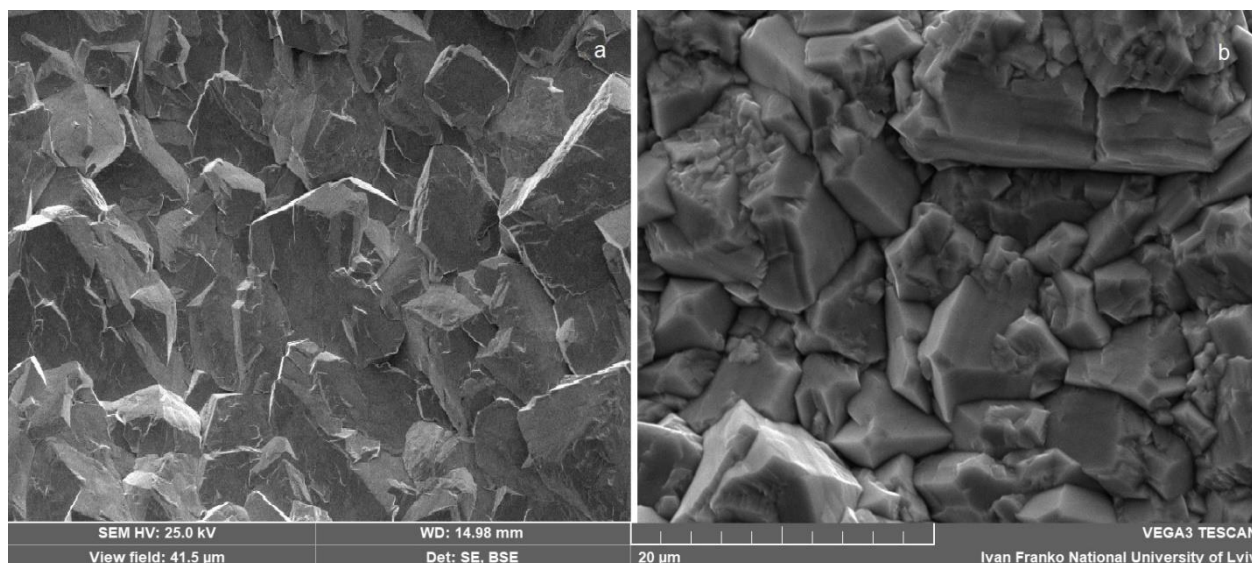


Fig. 4. SEM images of PbO_2 anodes. Deposition solution is 0.1 M $Pb(NO_3)_2$ + 0.1 M HNO_3 (a) + 3×10^{-4} M $C_8F_{17}SO_3K$ (b)

Unlike lead dioxide, which is characterized by polycrystalline blocks with a pronounced crystallographic orientation, the metal oxide-surfactant composites involved are characterized by a decrease in the crystal size. Indirect evidence of the formation of smaller crystals is also a decrease in the full width at half maximum (Figs. 5, 6). However, the most pronounced crystal

orientations consist of crystals of the same size (Table 1).

The crystalline phase in the samples is represented by a mixture of alpha and beta phases of PbO_2 , and the presence of a fluorine-containing surfactant in the coating helps to reduce the content of the dioxide α -phase by almost half (see Table 1).

Table 1

Phase composition, crystallographic data and microstructural parameters of the investigated samples								
Sample description	Phases	Content (mass %) α - PbO_2 / β - PbO_2	Lattice parameters, Å			Unit cell volume, Å ³ V	Reliability factors $R_1 / R_p, R_{WP}, R_{EXP}, \chi^2$	$D, \text{Å} / \epsilon$
			a	b	c			
0.1M $Pb(NO_3)_2$ + 0.1M HNO_3	α - PbO_2	30(2) /	5.005(3)	5.945(4)	5.453(3)	162.24(19)	0.0558	157 /
	β - PbO_2	70(2)	4.9538(7)		3.3829(5)	83.02(2)	0.0336 / 0.0739, 0.0985,	0.0099
0.1M $Pb(NO_3)_2$ + 0.1M HNO_3 + 3×10^{-4} $CF_3(CF_2)_7SO_3K$	α - PbO_2	18(1) /	5.031(6)	5.969(9)	5.428(5)	163.0(3)	0.0526	260 /
	β - PbO_2	82(1)	4.9528(5)		3.3815(3)	82.949(13)	0.0305 / 0.0773, 0.102,	0.0153
							0.0949, 1.14	225 / 0.0078

The electrocatalytic activity of the samples was studied in relation to the reaction of oxygen evolution and oxidation of 4-chlorophenol.

One can conveniently assess the nature of the effects changing the rate of oxygen evolution reaction. The regularities of the reaction of

oxygen evolution on PbO_2 can be satisfactorily explained within the framework of the mechanism proposed by Pavlov and Monahov [8]. According to this mechanism, the evolution of oxygen takes place at active sites located in the hydrated layer of PbO_2 .

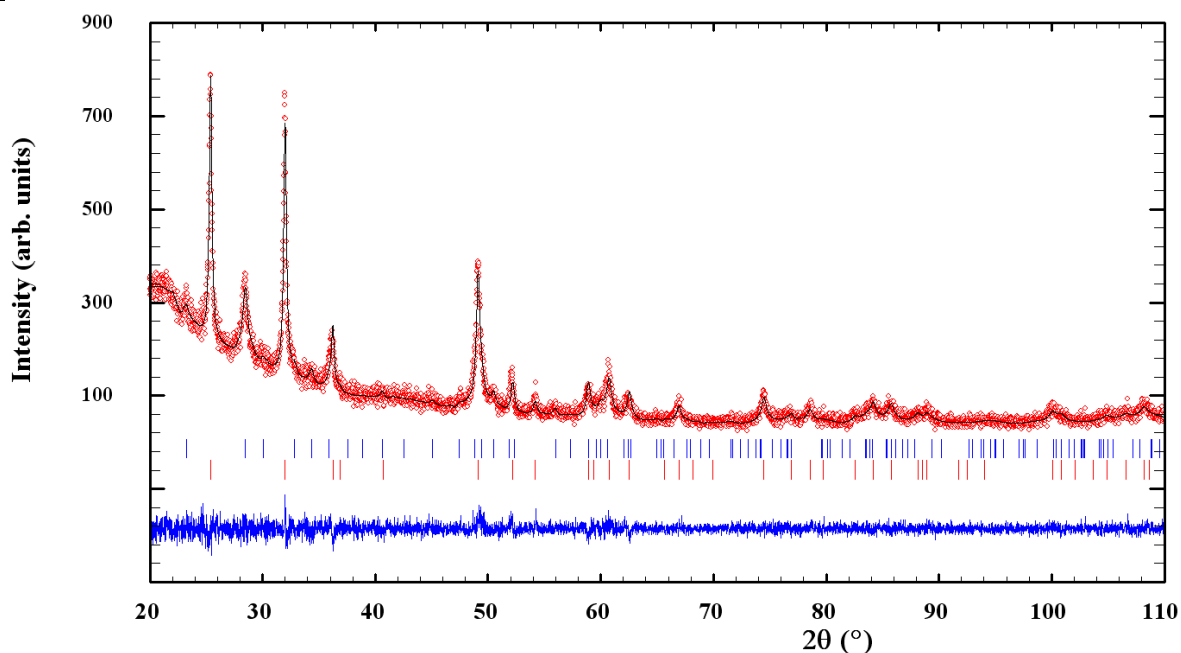


Fig. 5. Observed and calculated X-ray powder profiles for undoped PbO_2 . Experimental data (circles) and calculated profile (solid line through the circles) are presented together with the calculated Bragg positions (vertical ticks) and difference curve (bottom solid line). Upper ticks: α - PbO_2 , bottom ticks: β - PbO_2 .

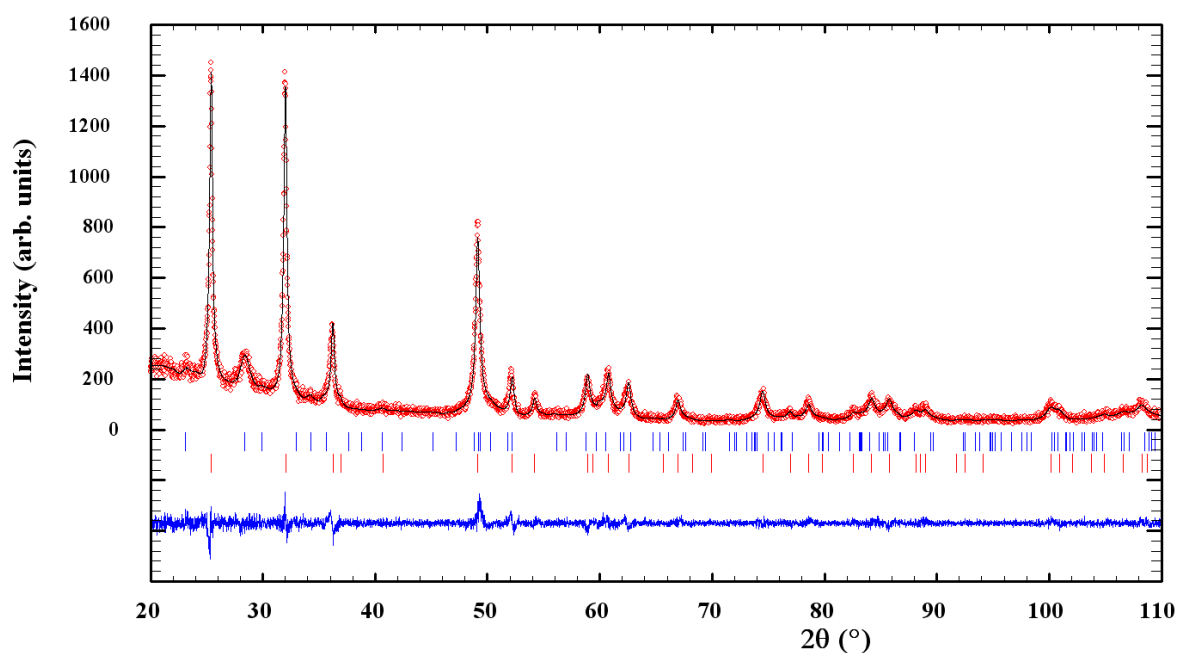


Fig. 6. Observed and calculated X-ray powder profiles for PbO_2 - $\text{C}_8\text{F}_{17}\text{SO}_3\text{K}$ composite. Experimental data (circles) and calculated profile (solid line through the circles) are presented together with the calculated Bragg positions (vertical ticks) and difference curve (bottom solid line). Upper ticks: α - PbO_2 , bottom ticks: β - PbO_2 .

It is known that the oxygen evolution on PbO_2 is limited by second electron transfer, i.e. electrochemical desorption [24]. Having this in mind, as the binding energy of oxygen-containing radicals to the electrode surface increases, the overvoltage of oxygen evolution will increase.

Fluorine-containing surfactants due to hydrophobization increase the bond strength [18], which leads to an increase in the overvoltage of oxygen. Such data are confirmed

by XPS data, where one can see an increase in the proportion of inert oxygen-containing radicals, which was also observed in the case of the use of fluorine as an additive to the electrolyte of PbO_2 deposition [10].

It can be seen that in both cases of lead dioxide-surfactant composites, the Tafel plot changes its slope above 1.65 V (Fig. 7). In low polarizations region the contribution of the ohmic drop to the measured potential leads to

distortion of the anodic polarization curve resulting in a deviation of the Tafel plot from linearity. At low polarization area Tafel slope is 390 and 474 mV dec⁻¹ for PbO₂-6 wt.% C₈F₁₇SO₃K

and PbO₂-8 wt.% C₈F₁₇SO₃K, respectively. At high polarization area it is 158 and 200 mV dec⁻¹ accordingly. For undoped sample the slope is linear at all polarizations and it is 156 mV dec⁻¹.

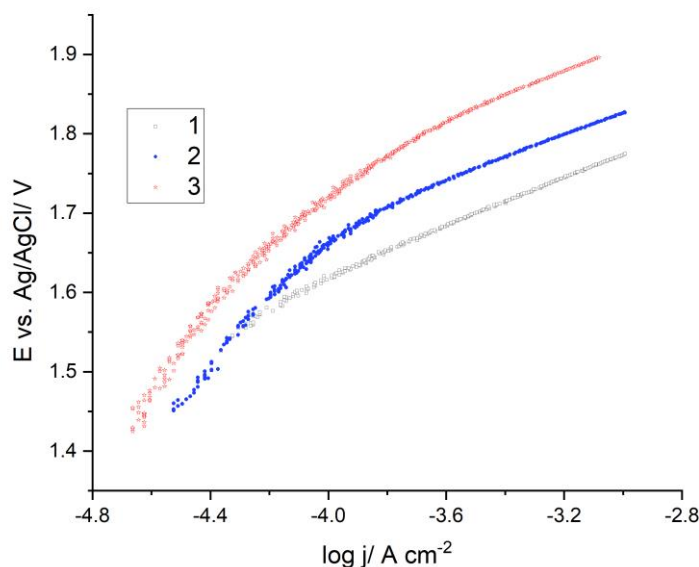


Fig. 7. Tafel plots built from current-potential curves recorded in 1 M HClO₄ at $\nu = 1 \text{ mV s}^{-1}$ on next electrodes; 1 - PbO₂; 2 - PbO₂-6 wt.% C₈F₁₇SO₃K; 3 - PbO₂-8 wt.% C₈F₁₇SO₃K

One might suggest that such a kink is due to the blocking of oxygen evolution by functional groups, as observed during oxygen evolution on BDD [25]. In the second area, the Tafel slopes are also much higher than the theoretical ones, which is typical for lead dioxide in sulfate solutions [26]. In our case, it can be assumed that the semiconductor component of the composite manifests itself in this region as a result of the interaction of divalent lead ions in the places of cationic vacancies not only with oxygen ions, but also with sulfo-groups of surfactant.

The partial oxidation rate constant of 4-chlorophenol increases from 2.0×10^{-2} for undoped sample to $2.2 \times 10^{-2} \text{ min}^{-1}$ for PbO₂-8 wt.% C₈F₁₇SO₃K.

One can assume that such an effect, as in the process of oxygen evolution, is associated with the formation of particles with higher bond strength, which, based on experimental data, are involved in the oxidation of organic compounds.

Conclusions

Regularities of electrosynthesis of lead dioxide-perfluorocyanesulfonate composites are satisfactorily described by the well-known four-

stage scheme of formation of lead dioxide. When the length of the perfluorinated hydrocarbon chain is eight carbon atoms, the heterogeneous oxidation rate constant does not differ from that in the electrolyte without additives. Adsorption parameters were calculated according to Frumkin isotherm, which are unity for interaction parameter and energy of adsorption interaction ($-\Delta G$) $25.12 \pm 0.01 \text{ kJ mole}^{-1}$ that indicates the slight interaction between adsorbed molecules and specific character of adsorption, respectively.

The metal oxide-surfactant composites involved are characterized by a decrease in the crystal size, which is confirmed by a decrease in the full width at the half maximum of peaks on the diffractogram.

Fluorine-containing surfactants due to hydrophobization increase the bond strength, which leads to an increase in the overvoltage of oxygen. Composites characterized by abnormally high Tafel slopes, which is due to the blocking of oxygen evolution by functional groups, as well as the interaction of divalent lead ions in the places of cationic vacancies not only with oxygen ions, but also with sulfo-groups of surfactant.

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