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T. Luk'yanenko^a, *O. Shmychkova*^a, *L. Dmitrikova*^b, *L. Borschevich*^c, *A. Velichenko*^a**THE COMPOSITION AND ELECTROCATALYTIC ACTIVITY OF COMPOSITE PbO₂-SURFACTANT ELECTRODES**^a Ukrainian State University of Chemical Technology, Dnipro, Ukraine^b State Institution «Dnipropetrovsk Medical Academy of the Ministry of Health of Ukraine», Dnipro, Ukraine^c Oles Honchar Dnipro National University, Dnipro, Ukraine

It is shown that anionic surfactants, sodium laureth sulphate in particular, are included in electrodeposited lead dioxide. The content of organic substance in the composite coating can vary from 3.4 to 12.5 wt.%. The change in deposition conditions (temperature, pH and anodic current density) allows controlling the content of surfactant in the resulting oxide. The electrocatalytic activity of the materials involved was investigated in respect to the oxygen evolution reaction and the electrochemical oxidation of 4-chlorophenol. The calculated value of Tafel slope is 229 mV dec⁻¹ for non-modified PbO₂, while it is 178 mV dec⁻¹ for composite of PbO₂ with 10.2 wt.% of sodium laureth sulfate. According to absorption spectra, the initial solution of 4-chlorophenol is characterized by two peaks at 220 and 280 nm. At first, the electrolysis leads to a decrease in the peak at 220 nm, a slight increase of the peak at 280 nm and the appearance of the plateau at 240–270 nm. This is caused by a decrease in the concentration of 4-chlorophenol and accumulation of benzoquinone in the solution. A further increase in the electrolysis duration results in the disappearance of the peaks at 220 and 280 nm and the reduction of the plateau at 240–270 nm due to a decrease in the concentrations of both 4-chlorophenol and benzoquinone. Already after 4 hours of electrolysis, the aromatic compounds are completely destroyed with the formation of only aliphatic electrolysis products (mainly maleic acid), that was evidenced by high performance liquid chromatography. The processes of electrooxidation of 4-chlorophenol on lead dioxide and PbO₂-based composite materials proceed qualitatively in the same way and differ only in the rate.

Keywords: nitrate electrolyte, lead dioxide, 4-chlorophenol, sodium laureth sulfate, electrooxidation.

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Introduction

The development of methods for directed synthesis of new materials with specified properties is known to be the one of the priorities of modern science [1,2]. At the same time, the great attention is paid to various electrochemical methods in connection with the simplicity of their implementation, low cost equipment and the ability to control the composition and properties of the materials obtained by changing the electrolysis regimes and the composition of electrolytes. Methods for the electrochemical synthesis of oxide composites are of considerable interest among others [3–5].

Lead dioxide is considered to be an

exceptionally successful object in this regard because of its following specific features: (i) it is formed at high anodic polarization by the electrodeposition from aqueous Pb(II) solutions; (ii) its physicochemical properties and electrocatalytic activity can be altered by the modification with various additives during the electrodeposition [6,7]; and (iii) PbO₂ has a high electrical conductivity, is chemically stable in both acidic and alkaline media and is characterized by a rather high basic electrocatalytic activity towards most anodic reactions [8,9].

Composite materials based on lead dioxide and containing surfactants are of considerable interest

for research, since while maintaining the basic properties of PbO₂, the composition, physicochemical properties, and electrocatalytic activity of such materials can vary widely [10,11]. At the same time, there is a lack of information regarding the effect of surfactants on the characteristics of electrodeposition of materials based on lead dioxide, especially the physicochemical properties of the oxides obtained and their electrocatalytic activity. In this connection, the effect of sodium lauryl sulfate (SLES) on the PbO₂ chemical composition and electrocatalytic activity was investigated in this work.

Material and methods

All chemicals were reagent grade. Lead dioxide was electrodeposited from nitrate electrolytes that contained 0.1 M HNO₃ and 0.1 M Pb(NO₃)₂.

Platinized titanium was used as a sheet. It was treated as described in ref. [6] before platinum layer depositing. The content of SLES (CH₃(CH₂)₁₁(OCH₂CH₂)_nOSO₃Na), depending on purposes of the experiment, varied within the range of 1·10⁻⁶ to 0.01 M. Electrolyte compositions and conditions of the deposition of composite coatings were selected hereby that in all cases the current efficiency of lead dioxide deposition was about 100%.

Oxygen evolution reaction was investigated by steady-state polarization using computer controlled EG & G Princeton Applied Research potentiostat (model 273A) in 1 M H₂SO₄. All potentials were recorded and reported vs. Ag/AgCl/KCl(sat.) reference electrode.

The electrooxidation of organic compounds was carried out in a divided cell at $j_a = 50 \text{ mA cm}^{-2}$. The volume of anolyte was 130 cm³. Solution containing phosphate buffer (0.25 M Na₂HPO₄+0.1 M KH₂PO₄)+10⁻⁴ M organic compound (pH 6.55) was used as an anolyte; phosphate buffer served as a catholyte. Stainless steel was used as a cathode. Composite PbO₂-SLES electrodes were used as anodes. Electrode surface area was 2.5 cm².

The change of the concentration of the organic substance during the electrolysis was measured by sampling (volume of 5 cm³) at regular intervals and measuring the absorbance of the solution in the ultraviolet and visible region (wavelength range of 200–350 nm) using a Kontron Uvikon 940 spectrometer.

Analyses of the reaction products were conducted by high performance liquid chromatography (HPLC) using a Shimadzu RF-10A xL instrument equipped with a Ultraviolet SPD-20AV detector and a 30 cm Discovery® C18 column.

Results and discussion

In some cases, small amounts of ionic additives

are incorporated into the growing oxide, forming micromodified materials based on lead dioxide [5–8,10]. On this basis, it should be assumed that the addition of organic substances would also affect the electrodeposition, composition and properties of oxide materials. It should be noted that at present, the addition of organic substances (most often surfactants and polyelectrolytes) are widely used in electroplating processes for the production of metals and alloys [12,13]. Additives can be used for various purposes, affecting both the technological parameters of the electroplating of metals and the properties of the resulting coatings. An extensive amount of publications is devoted to these investigations, in which various aspects of the use of additives of organic substances in the processes of electrodeposition of metals are considered in detail [12,13].

In contrast to the above processes, there is almost no information in the literature about the effect of polyelectrolytes and surfactants on regularities of the electrodeposition and physicochemical properties of the resulting oxide materials.

It was established that anionic surfactants, SLES in particular, are included in the growing lead dioxide. The content of organic substance in the coating can vary from 3.4 to 12.5 wt.%, a surfactant-oxide composite being formed (Table 1).

Table 1

Dependence of SLES content in PbO₂ on its concentration in the deposition electrolyte 0.1 M HNO₃+0.1 M Pb(NO₃)₂+X M C₁₆H₂₉O₆ SNa*

X, M	Content, wt.%
0.0005	3.4
0.0007	5.9
0.001	7.0
0.005	7.8
0.007	10.2
0.01	12.5

Note: * – coatings are deposited at 4 mA cm⁻².

The content of the additive in the composite can be controlled by changing the composition of the solution and electrolysis conditions. Surface effects are caused by the adsorption of additives both on the lead dioxide surface and on the colloidal PbO₂ particles formed in the near-electrode zone [10]. It should be noted that the obtained experimental data on the influence of the deposition conditions and the composition of the solution on the content of the additive in the composite coating are in good qualitative agreement with the influence of these factors on the adsorption of substances on lead

Table 2

The influence of pH and the deposition temperature on SLES content in PbO₂ ($j_a=4 \text{ mA cm}^{-2}$)

Electrolysis conditions	Content, wt. %
0.1 M HNO ₃ +0.1 M Pb(NO ₃) ₂ +0.007 M SLES, T=298 K	10.2
0.3 M HNO ₃ +0.1 M Pb(NO ₃) ₂ +0.007 M SLES, T=298 K	3.2
1 M HNO ₃ +0.1 M Pb(NO ₃) ₂ +0.007 M SLES, T=298 K	3.0
0.1 M HNO ₃ +0.1 M Pb(NO ₃) ₂ +0.007 M SLES, T=323 K	3.0
0.1 M HNO ₃ +0.1 M Pb(NO ₃) ₂ +0.007 M SLES, T=343 K	2.8

dioxide, i.e. an increase in adsorption leads to an increase in the content of surfactants in PbO₂ (an increase in the concentration of the additive or a positive charge of oxide due to a decrease in the pH of the solution or an increase in the anodic current density).

An increase in the deposition temperature and acid concentration in deposition electrolyte also leads to an increase in the content of surfactant in lead dioxide (Table 2).

The electrocatalytic activity of the obtained materials was investigated in respect to the oxygen evolution reaction and the electrooxidation of 4-chlorophenol.

It should also be assumed that oxide-surfactant composite materials will differ significantly in electrocatalytic activity in respect to lead dioxide. Since at high anodic potentials the vast majority of electrochemical processes occur with the participation of oxygen-containing particles adsorbed on the electrode (so-called oxygen transfer reactions), it is convenient to evaluate the nature of the effects by the determination of oxygen evolution overpotential.

The observed change in the electrochemical properties of lead dioxide-based composite materials with respect to the oxygen evolution reaction is mainly because of the change in the chemical properties of the oxide surface, leading to a change in the bond strength of oxygen-containing particles chemisorbed on the electrode [5–8]. As it is seen from the obtained data (Fig. 1), modification of lead dioxide by SLES leads to an increase in oxygen evolution overvoltage.

In this way, the Tafel slope is 229 mV dec^{-1} on non-modified PbO₂, while it is 178 mV dec^{-1} at 10.2 wt.% SLES content (Fig. 2). As one can conclude from the calculated results, the Tafel slope significantly exceeds the theoretical value, which indicates a decrease in the degree of filling of oxygen-containing particles on the surface of the electrode, probably due to blocking by sulfate ions. This effect was described in detail in previous publication [14]. A decrease in the slope in the case of PbO₂-SLES

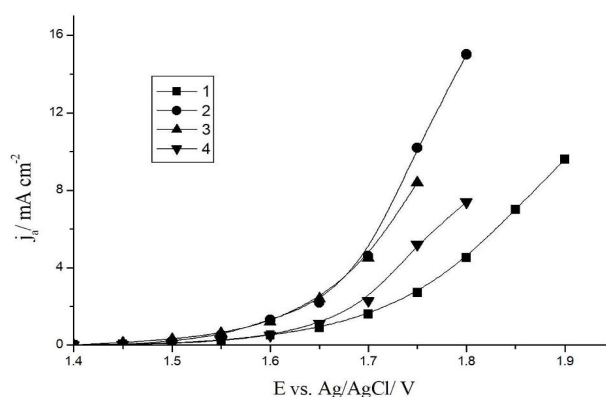


Fig. 1. Steady-state polarization curves of oxygen evolution in 1 M H₂SO₄ on the following electrodes: PbO₂ (1); PbO₂-3.2 wt.% SLES (2); PbO₂-7 wt.% SLES (3); PbO₂-10.2 wt.% SLES (4)

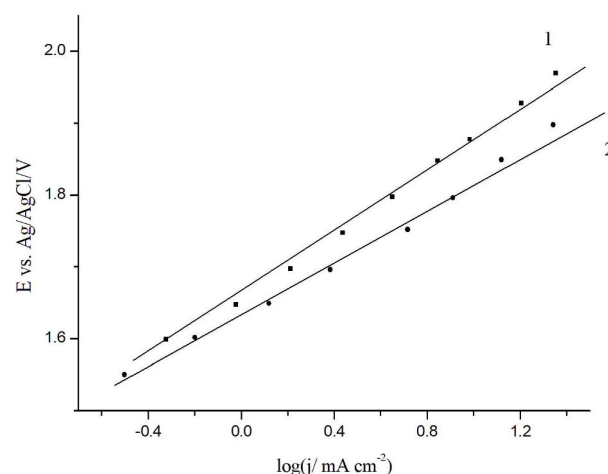


Fig. 2. Tafel plots (1 M H₂SO₄) for lead dioxide anodes electrodeposited from the following solutions: (1) – 0.1 M Pb(NO₃)₂+0.1 M HNO₃; (2) – 0.1 M Pb(NO₃)₂+0.1 M HNO₃+0.0007 M SLES

denotes an increase in the degree of filling of lead dioxide by oxygen-containing radicals, which are directly involved in the electrochemical stages of oxygen evolution. The most likely, surfactants on the electrode surface are mediators in the process of oxygen evolution and inhibit the surface blocking by sulfate ions on such materials.

Phenolic compounds, 4-chlorophenol in particular, and anode materials, based on lead dioxide, were selected to evaluate the electrocatalytic activity of the materials involved in the oxidation of organic substances. Since processes of electrooxidation of phenolic compounds on various electrodes are rather well studied, the focus can only be aimed on clarifying the role of anode material.

According to the literature [15], a highly large number of intermediate products are formed during the anodic oxidation of 4-chlorophenol. The main intermediate products include benzoquinone and maleic acid.

A simple and convenient way to estimate the electrocatalytic activity of an electrode material in the investigation of phenol oxidation is the disappearance time of aromatic intermediates which can be determined from the UV spectra of solutions at different electrolysis time.

The initial solution of 4-chlorophenol is characterized by two peaks at 220 and 280 nm (Fig. 3). At first, the electrolysis shows a decrease in the peak at 220 nm, as well as a slight increase in the peak at 280 nm and the appearance of the plateau at 240–270 nm which is caused by a decrease in the concentration of 4-chlorophenol and accumulation of benzoquinone in the solution. A further increase in the time of electrolysis leads to the disappearance of the peaks at 220 and 280 nm, as well as the reduction of the plateau at 240–270 nm due to a decrease in the concentrations of both 4-chlorophenol and benzoquinone. Already after 4 hours of electrolysis, the aromatic compounds are completely destroyed with the formation of only aliphatic electrolysis products (mainly maleic acid), that was evidenced by HPLC [15].

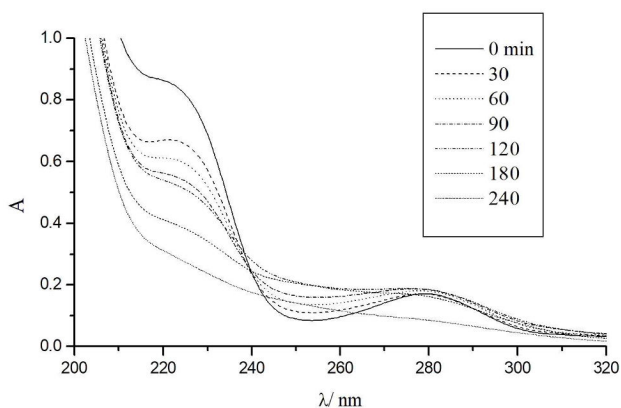


Fig. 3. The electronic absorption spectra of 0.5 M Na_2SO_4 +0.1 mM 4-chlorophenol+phosphate buffer solution at different electrolysis duration on the PbO_2 -10.2 wt.% SLES anode

As was shown earlier [5–8,15], the modification of the lead dioxide results in a significant increase in its electrocatalytic activity with respect to reactions involving oxygen-adsorbed particles on the electrode. In this regard, we investigated the electrooxidation of 4-chlorophenol on composite anodes based on lead dioxide containing different amount of surfactant (Fig. 4). It should be noted that the processes of electrooxidation of 4-chlorophenol on lead dioxide and PbO_2 -based composite materials proceed qualitatively in the same way and differ only in the rate. This suggests the invariance of the mechanism of 4-chlorophenol oxidation on different materials which makes it possible to correctly compare their electrocatalytic activity with respect to the reaction involved.

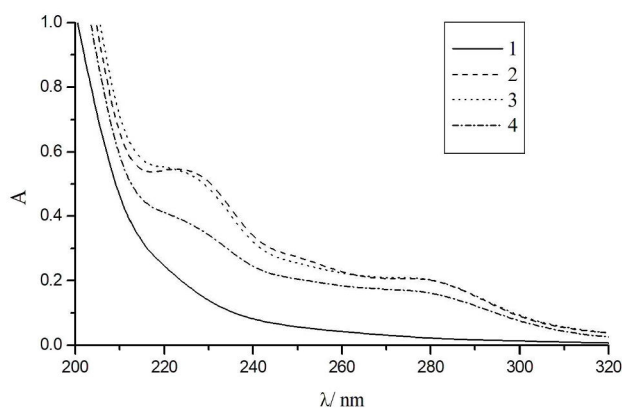


Fig. 4. The electronic absorption spectra of 0.5 M Na_2SO_4 +0.1 mM 4-chlorophenol+phosphate buffer solution after 180 min of electrolysis on the PbO_2 (1); PbO_2 -3.2 wt.% SLES (2); PbO_2 -7 wt.% SLES (3); PbO_2 -10.2 wt.% SLES (4) anodes

Conclusions

Anionic surfactants, SLES in particular, are included in the electrodeposited lead dioxide. The content of organic substance in the surfactant-oxide composite can vary from 3.4 to 12.5 wt.%. An increase in the temperature and acid concentration in deposition electrolyte leads to an increase in the content of surfactant in lead dioxide-based composite.

The electrocatalytic activity of the materials involved was investigated in respect to oxygen evolution reaction and the oxidation of 4-chlorophenol. The calculated value of Tafel slope is 229 mV dec^{-1} on non-modified PbO_2 , while it is 178 mV dec^{-1} on 10.2 wt.% SLES- PbO_2 . A decrease of the slope in the case of PbO_2 -SLES denotes an increase in the degree of filling of lead dioxide by oxygen-containing radicals, which are directly involved in the electrochemical stages of oxygen

evolution. The most likely, surfactants on the electrode surface play the role of mediators in the process of oxygen evolution and inhibit the surface blocking by sulfate ions on such materials. The processes of electrooxidation of 4-chlorophenol on lead dioxide and PbO₂-based composite materials proceed qualitatively in the same way and differ only in the rate.

Thus, lead dioxide based surfactant-oxide composite coatings are of great interest because of high electrocatalytic activity in respect to oxygen transfer reactions.

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СКЛАД ТА ЕЛЕКТРОКАТАЛІТИЧНА АКТИВНІСТЬ КОМПОЗИЦІЙНИХ ЕЛЕКТРОДІВ PbO₂-ПОВЕРХНЕВО-АКТИВНА РЕЧОВИНА

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Показано, що аніонні поверхнево-активні речовини, зокрема натрію лауреатсульфат, включаються до електроосажденного плумбум(IV) оксиду. Вміст органічної речовини в композиційному покритті «поверхнево-активна речовина-оксид» може варіюватися від 3,4 до 12,5 мас.%. Зміна умов осадження (температура, рН і анодна густина струму) дозволяє контролювати вміст поверхнево-активної речовини в одержуваному композиті. Встановлено електрокаталітичну активність досліджених матеріалів у реакції виділення кисню та окиснення 4-хлорфенолу. Розраховане значення тафелівського нахилу становить 229 мВ/дек для немодифікованого PbO₂ та 178 мВ/дек для PbO₂, що містить 10,2 мас.% натрію лауреатсульфату. Як впливає зі спектрів поглинання, вихідний розчин хлорфенолу характеризується двома піками при 220 і 280 нм. Протягом електролізу спостерігається зменшення піка при 220 нм, а також невелике збільшення піка при 280 нм і поява плато при 240–270 нм, що викликано зменшенням концентрації 4-хлорфенолу і накопиченням бензохінону в розчині. Подальше збільшення тривалості електролізу приводить до зникнення піків при 220 і 280 нм, а також до зменшення плато при 240–270 нм в результаті зниження концентрацій як 4-хлорфенолу, так і

бензохінону. Вже після 4 годин електролізу ароматичні сполуки повністю руйнуються з утворенням виключно аліфатичних продуктів (в основному, малеїнової кислоти), про що свідчать дані високоефективної рідинної хроматографії. Процеси електроокиснення 4-хлорфенолу на плюмбум(IV) оксиді та композиційних матеріалах на основі PbO₂ відбуваються якісно однаково та відрізняються лише швидкістю.

Ключові слова: нітратний електроліт, плюмбум(IV) оксид, 4-хлорфенол, натрію лауретсульфат.

THE COMPOSITION AND ELECTROCATALYTIC ACTIVITY OF COMPOSITE PbO₂-SURFACTANT ELECTRODES

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It is shown that anionic surfactants, sodium laureth sulphate in particular, are included in electrodeposited lead dioxide. The content of organic substance in the composite coating can vary from 3.4 to 12.5 wt.%. The change in deposition conditions (temperature, pH and anodic current density) allows controlling the content of surfactant in the resulting oxide. The electrocatalytic activity of the materials involved was investigated in respect to the oxygen evolution reaction and the electrochemical oxidation of 4-chlorophenol. The calculated value of Tafel slope is 229 mV dec⁻¹ for non-modified PbO₂, while it is 178 mV dec⁻¹ for composite of PbO₂ with 10.2 wt.% of sodium laureth sulfate. According to absorption spectra, the initial solution of 4-chlorophenol is characterized by two peaks at 220 and 280 nm. At first, the electrolysis leads to a decrease in the peak at 220 nm, a slight increase of the peak at 280 nm and the appearance of the plateau at 240–270 nm. This is caused by a decrease in the concentration of 4-chlorophenol and accumulation of benzoquinone in the solution. A further increase in the electrolysis duration results in the disappearance of the peaks at 220 and 280 nm and the reduction of the plateau at 240–270 nm due to a decrease in the concentrations of both 4-chlorophenol and benzoquinone. Already after 4 hours of electrolysis, the aromatic compounds are completely destroyed with the formation of only aliphatic electrolysis products (mainly maleic acid), that was evidenced by high performance liquid chromatography. The processes of electrooxidation of 4-chlorophenol on lead dioxide and PbO₂-based composite materials proceed qualitatively in the same way and differ only in the rate.

Keywords: nitrate electrolyte; lead dioxide; 4-chlorophenol; sodium laureth sulfate; electrooxidation.

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