

Electrochemically modified with osmium graphite: catalytic activity and application to the amperometric detection of hydrogen peroxide

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A new electrochemical method for deposition of Os on carbon carrier is reported. The metal phase was electrodeposited onto graphite under potentiodynamic conditions, using cyclic voltammetry over the potential range from -0.8 to 0 V (vs. Ag/AgCl, 3 M KCl). In order to obtain modified electrode with high catalytic activity in the reduction of hydrogen peroxide, the number of cycles and the scan rate of the electrodeposition process were optimized. It was found that the graphite modified with Os for 10 cycles with a scan rate of 0.01 V s⁻¹ possesses the highest catalytic activity in respect to the target reaction. The electrochemical behavior of the so produced electrode-catalyst was examined at different pHs at potentials of 0 V and -0.05 V. The modified electrode shows good analytical performance at hydrogen peroxide determination in neutral medium: a wide dynamic range (up to 5 mM) and a sensitivity of 426 $\mu\text{A mM}^{-1} \text{cm}^{-2}$ at an applied potential of -0.05 V (sensitivity of 364 $\mu\text{A mM}^{-1} \text{cm}^{-2}$ at potential of 0 V, respectively), which makes it a promising transducing material for the development of electrochemical peroxide sensors and, eventually, biosensors.

Key words: osmium, modified electrodes, electrodeposition, electrocatalyst, hydrogen peroxide reduction

INTRODUCTION

In the recent years, the electrochemical properties of osmium (Os) have been intensively studied mainly for the development of fuel cells. The interest to this transition metal is connected not only with the significant electrocatalytic activity in numerous processes, but also with the substantial advantage in the price – osmium being four times cheaper than platinum, traditionally used in developing catalysts.

It is well known that the electrocatalytic activity of metal deposits depends on their sizes, shapes, structural features and population on the surface of the carrier. Such parameters depend on both nature and pre-treatment of the carrier, and the procedure for deposition of the metal phase. Numerous different physical, chemical and electrochemical methods for obtaining osmium catalysts have been applied: vapor deposition at high temperature [1, 2], microwave synthesis [3], chemical (sol-gel) deposition [4], electrochemical deposition under either constant current [5,6] or potential [7,8], as the most commonly used precursor for the deposition of Os is hexachloroosmiate (IV) anion $[\text{OsCl}_6]^{2-}$.

Kua and Goddard suggested that pure metallic Os should be examined as Direct Methanol Fuel Cell (DMFC) catalyst because this metal is capable of catalyzing both dehydrogenation of methanol and water dehydrogenation without a co-catalyst [9]. The electrocatalytic properties of osmium

electrodeposits on Pt in acidic solutions of methanol, ethanol, propanol, formaldehyde and acetaldehyde have been studied [10]. The results showed that at low overpotentials the electrocatalytic activity of Os exceeded that of Pt during electrooxidation of methanol, ethanol and acetaldehyde. The adsorption and electrooxidation of CO, methanol, ethanol, and formic acid on glassy carbon modified with Os were examined in details by Orozco and coworkers by means of cyclic voltamperometry (CV) and infrared spectrometry with Fourier transformation (FTIRS) [11]. In order to develop a new anode material for Direct Borohydride Fuel Cell (DBFC), Os-film with thickness up to 140 nm on Pt/quartz was obtained [12]. The film was electrodeposited from a solution of $(\text{NH}_4)_2\text{OsCl}_6$ in HClO_4 by applying a differential pulse voltammetry (DPV). The analysis of the film carried out by X-ray photoelectron spectroscopy (XPS) indicated the presence of both Os and oxide (OsO_2). The authors compared the electrocatalytic activity of several electrodes modified with Os-films with varying thickness, in the reaction of electrooxidation of BH_4^- in 2 M NaOH.

To the best of our knowledge, there are no results concerning the application of osmium as a catalytically active component in electrocatalysts for reduction of hydrogen peroxide (H_2O_2). The development of effective and accessible materials

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with pre-defined operational characteristics for quantitative determination of H_2O_2 at low potentials is relevant not only for the establishment of amperometric analysis of H_2O_2 , but also for developing selective first generation biosensors. Hydrogen peroxide has wide and diverse applications in many fields – food processing, textile industry, paper bleaching, disinfecting and cleaning products, pharmaceutical, clinical and environmental analyses. H_2O_2 is also a catalytic by-product from enzyme-catalysed oxidation of metabolites such as glucose, lactate, ethanol, cholesterol, xanthine, etc. Therefore, the development of highly sensitive, interference-free and simple methods for reliable, fast and low-cost quantitative analysis of H_2O_2 over the micro- and nanomolar concentration ranges applicable in the control of numerous technological processes, as well as environmental and biochemical monitoring, is practically important and widely investigated.

In this connection, the present work deals with the studies on the electrochemical behavior of graphite modified with osmium and the characterization of the produced electrode-catalyst with respect to quantitative amperometric determination of H_2O_2 at low applied potentials. A new electrochemical method for deposition of osmium on carbon carrier was reported – the metal phase was electrodeposited onto graphite under potentiodynamic conditions, using cyclic voltammetry over the working potential range from -0.8 to 0 V (vs. Ag/AgCl, 3 M KCl). In order to obtain modified electrode with high catalytic activity in the reduction of H_2O_2 , the number of cycles and the scan rate of the electrodepositing process were optimized.

MATERIALS AND METHODS

Materials

The working electrode was disc from spectroscopic graphite with diameter of the working surface $d=5.6$ mm and visible surface area *ca.* 25 mm² (RWO, Ringsdorf, Germany).

$(\text{NH}_4)_2\text{OsCl}_6$ (Fluka); NaClO_4 (Fluka); H_2O_2 (Fluka); Na_2HPO_4 and NaH_2PO_4 (Sigma-Aldrich) were of analytical grade and used as received. 0.1 M buffer solutions were prepared with monobasic and dibasic sodium phosphates dissolved in double distilled water with various pHs, adjusted with a pH meter MS 2006 (Microsyst, Bulgaria).

Apparatus and measurements

The electrochemical measurements were performed using computer controlled electrochemical workstation EmStat2 (PalmSens BV, The Nederland), equipped with PSTrace 2.5.2

software, in a conventional three-electrode cell, including a working electrode (modified with osmium graphite or a bare graphite electrode), a Ag/AgCl (3 M KCl) reference electrode, and a platinum auxiliary electrode. All the electrochemical measurements were carried out in 0.1 M phosphate buffer solutions (PBS) at temperature of 25 °C.

Preparation of the modified electrodes

Before modification the bare graphite electrode was carefully polished to mirror-like finish with emery paper with decreasing particle size (P800, P1200 and P2000), rinsed with double distilled water and sonicated in water for 3 min. The working surface of the cleaned and polished electrode was modified through direct electrodeposition of osmium by means of CV over the potential range from 0 to -0.8 V (vs. Ag/AgCl, 3 M KCl) from electrolyte containing 5 mM $(\text{NH}_4)_2\text{OsCl}_6$ and 0.1 M NaClO_4 . The scan rate and the number of cycles are varied to obtain modified electrode with optimized electrocatalytic properties in the target reaction (electroreduction of H_2O_2). To indicate the type of the modified with osmium graphite electrode we take the following notations: Os_number of cycles_scan rate/Gr (for example: graphite modified by applying 10 cycles at a rate of 5 mV s⁻¹ will be denoted in the text as Os_10c_v5/Gr).

RESULTS AND DISCUSSION

Fig. 1 displays the CVs of electrodeposition process of osmium particles onto graphite electrode which begins at potential of 0 V and ends at the negative potential of -0.8 V. It can be seen that the reduction peak of Os particles occurs at the potential of -0.45 V and remains unchanged with increasing the number of cycles. At the same time the oxidation process takes place at -0.25 V (for the first cycle) and this peak is shifted to more positive potentials by increasing the number of cycles (-0.19 V for the 20th cycle). For the continuous cycles, the reduction and the oxidation peaks of the Os particles were clearly increasing. The observed features confirm that the electrodeposition of osmium takes place at the graphite surface.

Following the development of effective electrocatalyst of H_2O_2 reduction, we focused on the optimization of the number of cycles and the scan rate of the electrodepositing process. The effect of the number of cycles on the electrocatalytic activity of the electrodes was first investigated. Four different types of electrodes were fabricated by cycling at scan rate of 10 mV s⁻¹ for 5, 8, 10 and 20 cycles, respectively. In order to obtain a prior information about the catalytic activity in reduction of hydrogen peroxide for each of the so-produced

electrodes an amperometric response was recorded in the presence of 0.5 mM H₂O₂ at a constant potential of 0 V in 0.1 M PBS (pH 7.0). Cathode currents in presence of H₂O₂ resulting from its electrochemical reduction were observed for all four modified electrodes; the background subtracted steady-state responses of the electrodes are shown in Fig. 2. The current response increased remarkably as the number of cycles increased and reached a maximal value (52 μA) at an electrode modified with 10 cycles. As shown, the amperometric response does not change with further increase of the number of cycles and the catalytic ability of the electrodes type Os_10c_v10/Gr and type Os_20c_v10/Gr toward reduction of H₂O₂ is identical. Therefore electrode type Os_20c_v10/Gr was not used in further investigations.

In the next step, the other three types modified electrodes were tested for amperometric detection of H₂O₂ at low working potentials. The concentration dependence of the amperometric response of the electrodes was investigated by means of constant potential amperometry at potentials of -0.05 and 0 V in 0.1 M PBS at pH 7.0 and the basic operational parameters of each modified electrode were deduced (Table 1). The chronoamperometric records indicate that for all types of modified electrodes the reductive current increased stepwise upon introducing in the buffer aliquots of the H₂O₂ stock solution. The electrode sensitivity was determined as the slope of the linear portion of the calibration graph (divided by geometric electrode area), build on the basis of the chronoamperometric measurements. From the data presented in Table 1 it is evident that for all modified electrodes at pH 7.0 as the polarization potential become more negative, the sensitivity increases and the range of the strict linear dependence of the signal shortens.

In order to establish the effect of electrodeposition scan rate on the activity of the catalysts in the target reaction, graphite electrodes were modified for 10 cycles at scan rates of 5, 10, 25 and 50 mV s⁻¹, respectively. With all four types of modified electrodes, the current signal in the presence of 0.5 mM H₂O₂ at a constant potential of 0 V (pH 7.0) was examined.

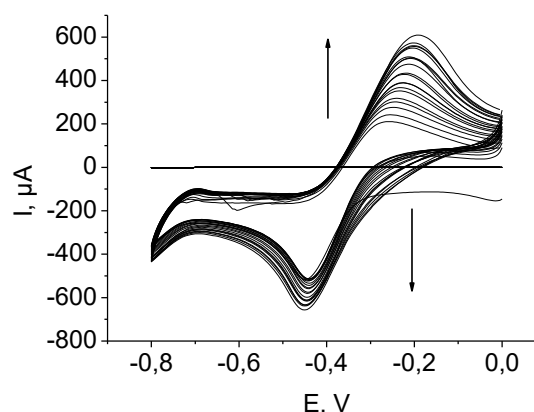


Fig. 1. Potentiodynamic electrodeposition of Os particles on graphite electrode from 5 mM (NH₄)₂OsCl₆ containing 0.1 M NaClO₄; potential scan between 0 to -0.8 V for 20 cycles; scan rate of 10 mV s⁻¹.

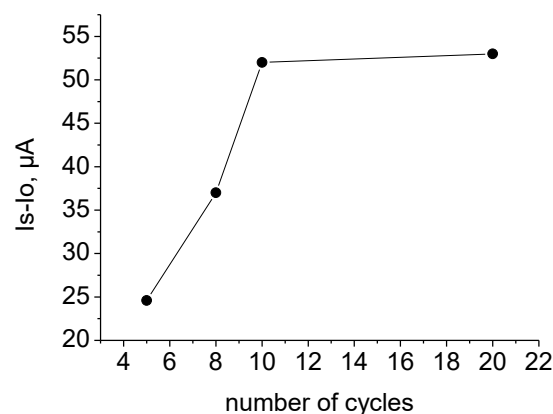


Fig. 2. Effect of the number of electrodeposition cycles (scan rate of 10 mV s⁻¹) on the amperometric responses of the modified electrodes toward the reduction of H₂O₂. Supporting electrolyte: 0.1 M PBS (pH 7.0) containing 0.5 mM H₂O₂; applied potential: 0 V.

Table 1. Operational parameters of graphite electrodes modified with osmium by means of CV (from 0 to -0.8 V) with 5, 8 and 10 cycles at scan rate of 10 mV s⁻¹; reference electrode Ag/AgCl, 3 M KCl; background electrolyte 0.1 M PBS.

E, V	Electrode sensitivity, μA mM ⁻¹ cm ⁻²		
	Linearity, mM		
	Os_5c_v10/Gr	Os_8c_v10/Gr	Os_10c_v10/Gr
-0.05	188 7 mM (r ² =0.98 ₉)	264 5.6 mM (r ² =0.99 ₄)	426 5 mM (r ² =0.99 ₇)
0	144 7.5 mM (r ² =0.98 ₈)	232 6.3 mM (r ² =0.99 ₃)	364 5.6 mM (r ² =0.99 ₅)

Table 2. Operational parameters of modified electrode type Os_10c_v10/Gr at various pH values; reference electrode Ag/AgCl, 3 M KCl; background electrolyte 0.1 M PBS.

E, V	Electrode sensitivity, $\mu\text{A mM}^{-1} \text{cm}^{-2}$				
	Linearity, mM				
	pH 5.0	pH 6.0	pH 7.0	pH 8.0	pH 9.0
-0.05	308	430	426	288	188
	0.7 mM ($r^2=0.99_4$)	2.7 mM ($r^2=0.99_3$)	5 mM ($r^2=0.99_7$)	3.5 mM ($r^2=0.98_9$)	1.7 mM ($r^2=0.96_6$)
0	176	390	364	256	
	1.2 mM ($r^2=0.99_8$)	4.7 mM ($r^2=0.99_8$)	5.6 mM ($r^2=0.99_5$)	5.3 mM ($r^2=0.98_1$)	

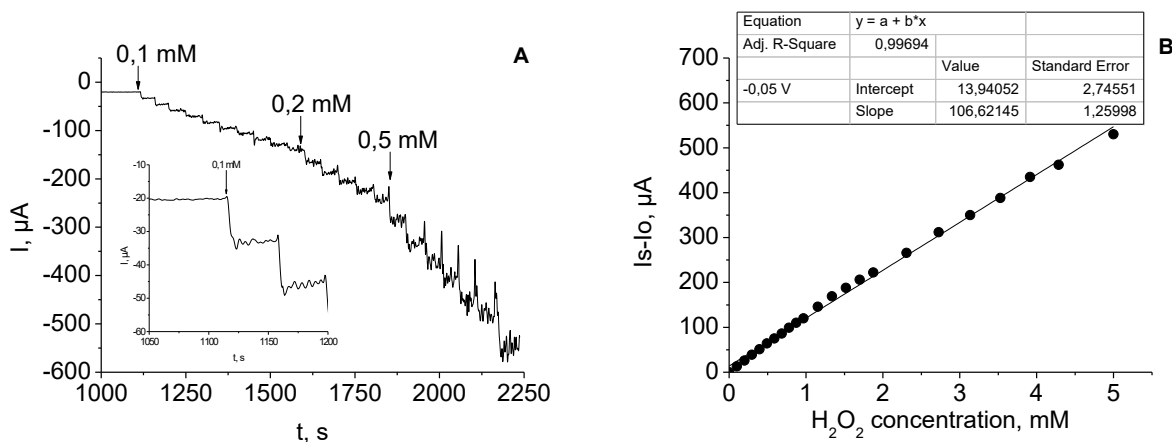


Fig. 3. A) Amperometric response of the modified electrode type Os_10c_v10/Gr to successive addition of H_2O_2 into stirred 0.1 M PBS (pH 7.0) at an applied potential of -0.05 V; inset shows the initial section of the response upon addition of the first portions of 0.1 mM H_2O_2 . B) The corresponding calibration line (the dependence of the electrode response on the concentration of H_2O_2).

The electrode response was found to decrease linearly with increasing the scan rate from 10 to 50 mV s^{-1} ($y = -0.3776x + 56.031$, $r^2 = 0.99_8$). These experimental data suggest that the increase of the scan rate during electrodeposition results in smaller amount of catalytically active metal phase formed onto graphite surface. At the same time the catalytic current (48 μA) of the modified electrode type Os_10c_v5/Gr is lower compared with that of the electrode type Os_10c_v10/Gr, i.e. the application of scan rate lower than 10 mV s^{-1} does not result in obtaining more active metal deposition. Based on these results, 10 cycles at scan rate of 10 mV s^{-1} were selected as optimal conditions for electrodeposition.

The effect of pH of the PBS on the catalytic activity towards the reduction of H_2O_2 was also investigated. The basic operational characteristics of the electrode type Os_10c_v10/Gr, determined in 0.1 M PBS over pH range 5.0 – 9.0 at potentials of -0.05 and 0 V, are provided in Table 2. The modified electrode showed higher electrocatalytic activity at an applied potential of -0.05 V in PBS with pHs 6.0 and 7.0. The linear dynamic range of the electrode response also differs depending on the applied potential and the pH value of the buffer solution. The linearity range of the signal shortens gradually with

increasing the acidity of the background electrolyte. The same effect was observed when increasing pH value over 7.0. Thus the pH value 7.0 was selected as optimal for the determination of H_2O_2 with the here presented modified electrode.

Fig. 3A shows the typical current-time (I-t) plot upon the successive injection of H_2O_2 at an applied potential of -0.05 V. A well-defined response was observed during the successive additions of 0.1 mM, 0.2 mM and 0.5 mM H_2O_2 , respectively. These results evidence a stable and efficient catalytic property of osmium. It can also be observed that the modified electrode type Os_10c_v10/Gr responds rapidly to the changes of H_2O_2 concentration, producing steady-state signal within 10 s. The corresponding calibration curve is presented in Fig. 3B. The linear response was proportional to the H_2O_2 concentration up to 5.0 mM (correlation coefficient of 0.997) with a sensitivity of 106.62 $\mu\text{A mM}^{-1}$ (or 426 $\mu\text{A mM}^{-1} \text{cm}^{-2}$) and a detection limit of 10 μM (at a signal-to-noise ratio of 3). The reproducibility of the current signal for the same electrode to 1 mM H_2O_2 was examined. The relative standard deviation (RSD) was calculated to be 3 % for 4 successive measurements.

Table 3. Comparison of the analytical characteristics of amperometric sensors for H₂O₂ detection reported in the literature with the achieved in the present work.

Electrode modifier/(Electrode)	E, V (vs. Ag/AgCl)	Electrolyte	Sensitivity, $\mu\text{A mM}^{-1} \text{cm}^{-2}$	Detection limit, μM	Ref.
Fe ₃ O ₄ /chitosan/(GC rotating electrode)	-0.2	pH 7.0	9.6 $\mu\text{A mM}^{-1}$	7.4	[13]
PtNW-CNT-CHIT/(GC)	-0.1 (vs. SCE)	1/15M PB pH 6.98	260	-	[14]
Co ₃ O ₄ nanowalls/(GC)	-0.2	0.01 M PBS pH 7.4	80.74 * (4.84 $\mu\text{A mM}^{-1}$)	10	[15]
PdNPs-PAA/(Au)	-0.1 (vs. SCE)	PBS pH 7.0	232	5	[16]
Ag/(GC)	-0.2	0.1 M PBS pH 7.6	104.53 * (7.39 $\mu\text{A mM}^{-1}$)	0.5	[17]
PVA-MWCNTs-PtNPs/(GC)	0	0.1 M PBS pH 7.0	122.63	0.7	[18]
Ag microparticles/(SPC)	-0.4	0.01 M PBS pH 7.4	138.4	50	[19]
Pd/PEDOT nanospheres/(GC)	-0.2 (vs. SCE)	0.1 M PBS pH 7.0	215.3	2.84	[20]
Graphene-AgNPLs/(GC)	-0.5	0.2 M PBS pH 7.0	183.5	3	[21]
MWCNT/Pt nanohybrids/(Pt)	0	PBS pH 7.0	205.8	0.3	[22]
Os/(Gr)	-0.05 0	0.1 M PBS pH 7.0	426 364	10	This work

*Calculated from the data in paper

A large number of H₂O₂ sensors have been reported in literature. The performance of the sensors is greatly dependent on the supporting electrolyte, applied potential, electrode material and surface area, therefore it is difficult to compare one sensor to others. Various H₂O₂ sensors, based on transition and noble metal particles-modified electrodes, have been listed in Table 3 with respect to the operational conditions, sensitivity and detection limit. It can be seen that the proposed Os-modified graphite electrode possess the sufficient capability for amperometric detection of H₂O₂. Compared with other modified electrodes employed as H₂O₂ sensors in neutral medium, it showed much higher sensitivity. In contrast to other H₂O₂ amperometric sensors of previous literature reports [13-17, 19, 20], the applied potential for the modified electrode Os_{10c}/v10/Gr is much lower. At such a low potentials (0 V and -0.05 V), the background current decreases and the response of other electroactive species can be minimized or totally eliminated, which is crucial for the selectivity of the analytical system.

CONCLUSION

The current work provides a new facile strategy to construct hydrogen peroxide sensor based on

osmium, electrodeposited on graphite electrode. The number of cycles and the scan rate of the electrodeposition process (cyclic voltammetry) were purposefully altered and the catalytic activity in the reduction of H₂O₂ for each of the produced electrodes was examined. The electrode modified with 10 cycles at scan rate of 0.01 V s⁻¹ exhibits the best analytical performance for the amperometric H₂O₂ detection. The optimal working pH value (7.0), the high sensitivity (426 $\mu\text{A mM}^{-1} \text{cm}^{-2}$) and the extended dynamic range of the response (up to 5 mM) at an applied potential as low as -0.05 V, makes it an attractive novel material for electrochemical H₂O₂ sensing and promising candidate for designing biosensing systems.

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ЕЛЕКТРОХИМИЧНО МОДИФИЦИРАН С ОСМИЙ ГРАФИТ: КАТАЛИТИЧНА АКТИВНОСТ И ПРИЛОЖЕНИЕ ЗА АМПЕРОМЕТРИЧНА ДЕТЕКЦИЯ НА ВОДОРОДЕН ПЕРОКСИД

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(Резюме)

Представен е нов електрохимичен метод за отлагане на осмий върху въглероден носител. Металната фаза е отложена върху графит в потенциодинамични условия, използвайки циклична волтамперометрия в диапазон от потенциали от -0.8 до 0 V (vs. Ag/AgCl, 3 M KCl). С цел получаване на модифициран електрод с висока каталитична активност при редукция на водороден пероксид, са оптимизирани броят цикли и скоростта на изменение на потенциала при процеса на електроотлагане. Установено е, че графитът, модифициран с осмий чрез 10 цикъла при скорост 0.01 V s⁻¹, проявява най-висока каталитична активност в целевата реакция. Електрохимичното поведение на така разработения електрокатализатор е изучено при различни стойности на рН при работни потенциали 0 V и -0.05 V. Модифицираният електрод има много добри аналитични характеристики при определяне на водороден пероксид в неутрална среда: широка област на линейна концентрационна зависимост на сигнала (до 5 mM) и чувствителност 426 μA mM⁻¹ cm⁻² при потенциал -0.05 V (чувствителност 364 μA mM⁻¹ cm⁻² при потенциал 0 V, съответно), които го характеризират като подходящ базов трансдюсер (преобразувател на сигнала) за разработването на електрохимични сензори за пероксида и, евентуално - биосензори.

Ключови думи: осмий, модифицирани електроди, електроотлагане, електрокатализатор, редукция на водороден пероксид