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THE QUANTITATIVE DETERMINATION OF HYDROGEN PEROXIDE BY VOLTAMMETRY ON THE CARBOSITALL ELECTRODE

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Key words: determination; hydrogen peroxide; voltammetry; carbositall rotation electrode; antiseptic (skin disinfectant)

The electrochemical behaviour of hydrogen peroxide (H_2O_2) has been studied using the alternating current voltammetry with square wave modulation in the potential range of +1.0…–1.0 V on the carbositall electrode as a working and auxiliary electrode (vs Ag,AgCl/KCl(sat)). The peak was obtained at E_p = +0.16 V on the background of 0.1 Mol L^{-1} Na_2SO_4 and 0.01 Mol L^{-1} $KHSO_4$ (pH≈2.4) with its height rising proportionally to the increase of H_2O_2 concentrations. The linear dependence was observed in the H_2O_2 concentration range of (1.7-10.2)×10^{-5} Mol L^{-1}, the calibration curve equation was I_p = (8.6±0.7)×10^3c (r = 0.998); LOD = 6.16×10^{-6} Mol L^{-1}, LOQ = 2.05×10^{-5} Mol L^{-1}. To determine H_2O_2 in solutions of antiseptic drugs the standard addition method was used.

Hydrogen peroxide (H_2O_2) is one of the most versatile oxidants, which oxidizing activity exceeds the known oxidizing agents – chlorine, chlorine dioxide and potassium permanganate; as a result of catalysis, H_2O_2 can be transformed into the hydroxyl radical (OH•), which is the second after Fluorine by its reactivity. In addition to the oxidizing properties ($H_2O_2+2H^++2e^-\rightarrow 2H_2O$, $E^o = 1.78$ V) it can be used as a reducing agent ($H_2O_2 + 2OH^-\rightarrow O_2 + 2\ H_2O + 2e^-$, $E^o = -0.15$ V) [22].

Thus, H_2O_2 is widely used in various industrial processes, such as the textile and paper industries for bleaching materials [20], and promotes controlled fibre swelling [22]. In the work [17] H_2O_2 was used to enhance the oxidizing potential in remediation of soil and aquifer layers, and it was also reported to be a source of oxygen for biological treatment of environmental objects [13, 24, 25]. H_2O_2 is used for decontamination (detoxification) of organic pollutants (formaldehyde, phenol, amine, penicillins, surface-active substances (surfactants), herbicides, etc.) [3, 4, 18].

For the overall assessment of the residual toxicity of the treated water it is necessary to consider the content of H_2O_2 since such an assessment is carried out by biological organisms, which are quite sensitive to it and, therefore, should continuously monitor its concentration in the aquatic environment [8, 14, 18, 28].

Probably, H_2O_2 is used most widely in medicine and pharmacy as an active ingredient of many antiseptics and disinfectants (the pharmacotherapeutic group: Antiseptics and disinfectants; ATC code D08A X01), such as 3%, 6% solutions for external use, Hydroperite, Grillen, Peramine, PEMOS-1, Perkat. Recently, the more advanced forms of these drugs have appeared. For example, there is drug "Peroxygel" (3% gel) with the bactericidal, mild cauterizing and hemostatic effect. The product contains H_2O_2 with the concentration of 3% as an active substance. In the aqueous medium under the action of catalase it breaks down to water and atomic oxygen. This reaction is accelerated in the presence of traces of hemoglobin of the blood, pus, and necrotic tissues; the foam formed in this reaction loosens the eschar, mechanically cleanses the wound, and after drying and/or removal of the necrotic tissue, pus, etc., a protective film protecting the wound from the secondary infection is formed.

"Peroxygel, 3.0%" is a colourless or pale white, odourless disperse system. It has the thermoplastic quality: at temperatures below 20°C and above 45°C it is liquid, but once applied to the skin (i.e. at a temperature of about 36.6°C) it takes the form of a gel. It is contained in a 15 g aluminium tube with a protective membrane and with a polyethylene bouchon in a cardboard box.

The pharmacopoeian method for determining H_2O_2 is the method of permanganometric titration [2]. The extensive literature survey reveals that the most common method of the H_2O_2 analysis is spectrophotometry [20, 26, 32], fluorimetry [16], luminescence [30, 34], various types of chromatography [9, 29, 31, 33], electrochemical [6, 7, 12, 15, 27] and other methods of analysis [10, 11, 32].

The most selective, simple and rapid in performance, as well as economically viable electrochemical methods are considered. For example, to determine H_2O_2 the direct oxidation on the working electrode (e.g. platinum or carbon) is widely used. Such methods were described in more detail in our review published earlier [1]. However, there are relatively few methods, in which the H_2O_2 quantitative determination is performed by reduction on solid electrodes.

Therefore, as can be seen from the above data, development of analytical methods for the H_2O_2 quantitative determination is of a great practical importance for various applications, including pharmaceutical analysis. Practical requirements for methods of the H_2O_2 concentration determination include such criteria as selectivity,

high sensitivity and speed of analysis, simplicity, cheapness, and the possibility of their application to standardization of antiseptics and disinfectants.

The aim of the present work is to determine the feasibility of the H_2O_2 quantitative determination in a standard pharmacopoeian solution and preparations by cathodic voltammetry using the carbositall rotation electrode (CE) as an indicating electrode.

Materials and Methods

The standard solution of hydrogen peroxide (H_2O_2). 0.1700 Mol L^{-1} was freshly prepared and standardized permanganometrically. The stock solution was prepared by dissolving of 60% commercial preparation in a 100 mL volumetric flask by double distilled water. 10.00 mL of 0.1700 Mol L^{-1} solution of H_2O_2 was diluted in a 1000 mL volumetric flask with double distilled water to obtain 1.7×10^{-3} Mol L^{-1} of H_2O_2 solution.

The solution of potassium hydrogen sulphate. 1 Mol L^{-1} ($KHSO_4$) was prepared by dissolving of 68.1 g of $KHSO_4$ in a 500 mL volumetric flask by double distilled water.

The solution of sodium sulphate. 1 Mol L^{-1} ($NaSO_4$) was prepared by dissolving of 142.0 g of $NaSO_4$ in a 1000 mL volumetric flask by double distilled water.

The background solution consisted of the mixture of solutions of potassium hydrogen sulphate ($KHSO_4$) and sodium sulphate (Na_2SO_4).

The sample preparation, which was subjected to the analytical procedures for the analysis of H_2O_2, was "Hydrogen peroxide, 3%" antiseptic ("Farmatsevtychna fabryka", Stanyshivka, Zhytomyr region, Ukraine) and "Peroxygel, 3.0%" gel ("Hemi" Karczew, Poland).

The model solution of "Hydrogen peroxide, 3%" antiseptic was prepared by dissolving of 1.0 mL of the preparation in a 100 mL volumetric flask by double distilled water to obtain 8.8×10^{-3} Mol L^{-1} of H_2O_2 solution (standardized permanganometrically). 10.00 mL of this solution was diluted in a 100 mL volumetric flask with double distilled water to obtain 8.8×10^{-4} Mol L^{-1} of H_2O_2 solution.

The model solution of "Peroxygel, 3.0%" antiseptic was prepared by dissolving of 1.0 g of the preparation in a 100 mL volumetric flask by double distilled water.

The pH was measured using an ionmeter of I-160M type (Belarus) with a glass electrode of ESL-43-07 type paired with Ag, AgCl/KCl (sat) electrode.

Electrochemical measurements were carried out in an AVS-1.1 analyzer (Volta, St. Petersburg) with a three-electrode scheme by alternating the current mode with a square wave modulation in the potential range of +1.0…–1.0 V, W = 1000 rpm, the amplitude of 40 mV, ν = 65 Hz. The values of potential peaks directly at the maximum were measured by the electrochemical sensor "Module EM-04" with the accuracy of ±5 mV. CE was used as a working and an auxiliary electrode, and Ag,AgCl/KCl(sat) electrode type EVL-1M4 as a reference electrode.

The procedure for obtaining results of the calibration graph. The working solutions were prepared by diluting different volumes of the stock solution (0.5-3.0 mL) in a 50 mL volumetric flask with the background solution. 25 mL of the working solution was transferred to the cell. The voltammograms were recorded by scanning the potential toward the negative direction in the potential range from +1.0 V to –1.0 V (vs Ag,AgCl/KCl(sat)). The graph was plotted in the following coordinates: the height of peaks I_p in μA at E_p = +0.16 V on the ordinate axis and the corresponding concentration of H_2O_2, c in Mol L^{-1} on the abscissa axis (Fig. 3). The graph equation coefficients were calculated by the least square method.

The working solutions were prepared by diluting different volumes (1.00-2.00 mL) of the test solution (≈1×10^{-3} Mol L^{-1}) with 2.00 mL of the stock solution of H_2O_2 (1.7×10^{-3} Mol L^{-1}) in a 50 mL volumetric flask with the background solution. The voltammograms were recorded by scanning the potential toward the negative direction in the potential range from +1.0 V to –1.0 V (vs Ag,AgCl/KCl(sat)). The concentration of the test solution C_x (Mol L^{-1}) is calculated by the equation:

$$C_x = \frac{I_п - b}{a},$$

where: I_x – is the current peak of the test solution; a, b – are graph equation coefficients.

It was found that the surface active substances (SAS) being a part of the test solution of the sample preparation had the catalytic effect (current increase). Therefore, it was decided to use the addition method for analysis of the preparation.

The procedure of the quantitative determination of H_2O_2 in "Hydrogen peroxide, 3%" antiseptic. A typical procedure involves preparing several solutions containing the same amount of the unknown solution, but different amounts of the standard solution. For example, three 50 mL volumetric flasks are filled with 1.00 mL of the unknown solution each, and then the standard solution is added in different amounts, such as from 0.50 to 2.00 mL. The flasks are then diluted to the volume and mixed well. 25 mL of each solution prepared are transferred to the cell. The voltammograms are recorded by scanning the potential toward the negative direction in the potential range from +1.0 V to –1.0 V.

At first, the voltammogram of test solution is recorded, then the solution of the known aliquots of the standard solution of C_{st} (Mol L^{-1}) is added, and again the voltammogram is recorded. The concentration of the test solution C_x (Mol L^{-1}) is calculated by the equation:

$$C_x = C_{st} \cdot \frac{I_x}{I_{x+st} - I_x},$$

where: I_x – is the current peak of the test solution; I_{x+st} – is the current peak of the test solution with addition of a standard substance.

The mass fraction of H_2O_2 (w, %) in the test solution is calculated by the equation:

$$w,\% = \frac{C_x \cdot 34.01 \cdot 100 \cdot 100 \cdot V_0}{m \cdot 1000 \cdot 10 \cdot V} \cdot 100\%,$$

where: 34.01 – is the molar weight of H_2O_2, g Mol^{-1}; V_0 – is the volumetric flask capacity; V – is the volume

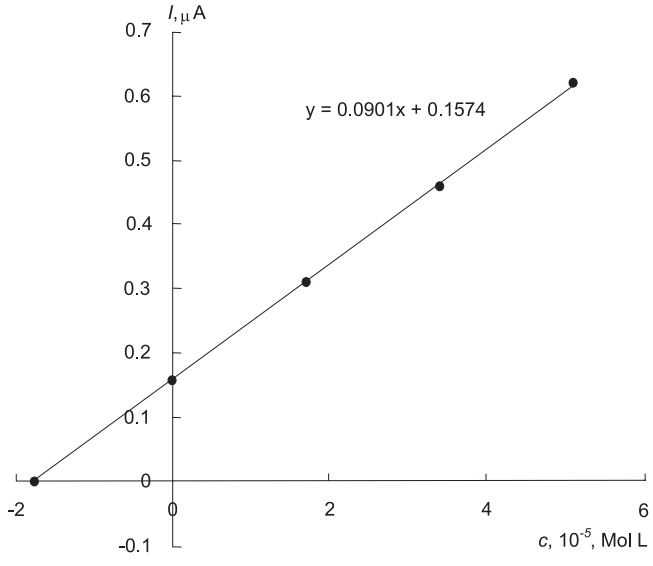

Fig. 1. The graph of the H_2O_2 reduction current peak vs. the concentration on the background of 0.1 Mol L^{-1} Na_2SO_4 and 0.01 Mol L^{-1} $KHSO_4$ (pH≈2.4) on CE (vs Ag,AgCl/KCl(sat)); E_p = +0.16 V.

of the test solution; *m* – is the sample weight, g; 10 – is the volume of the stock solution; 100, 1000 – are volumetric flask capacities.

The procedure of the quantitative determination of H_2O_2 in "Peroxygel, 3.0%" gel. The working solutions are prepared by diluting different volumes (0.5-1.5 mL) of the stock solution with the same amount of the test solution of H_2O_2 (1.7×10^{-3} Mol L^{-1}) in a 50 mL volumetric flask with the background solution. The voltammograms are recorded by scanning the potential toward the negative direction in the potential range from +1.0 V to –1.0 V (vs Ag,AgCl/KCl(sat)). The graph is plotted in the following coordinates: the height of peaks I_p in μA at E_p = +0.16 V on the ordinate axis and the corresponding concentration of H_2O_2 *c* in Mol L^{-1} on the abscissa axis (Fig. 1).

The mass fraction of H_2O_2 (%) in the test solution is calculated by the equation:

$$X,\% = \frac{C_x \cdot 34.01 \cdot 100 \cdot V_0}{m \cdot 1000 \cdot V} \cdot 100\% ,$$

where: 34.01 – is the molar weight of H_2O_2, g Mol^{-1}; V_0 – is the volumetric flask capacity; V – is the volume of the test solution; m – is the sample weight, g; C_x – is the graph of the H_2O_2 (regression line equation) found concentration, Mol L^{-1}: $y = ax + b$ where *a, b* – are the graph equation coefficients; $y - I_p$ (μA).

When $y = 0$; $C_x = x = -b/a$.

Results and Discussion

The effect of the nature and pH of the background solution. The effect of the pH on the reduction process was studied by recording voltammograms of H_2O_2 in the concentration of 6.8×10^{-5} Mol L^{-1} at several pH values ranging from 1.4 to 4.5 (Fig. 2). The mixture of 0.1 Mol L^{-1} Na_2SO_4 + 0.01 Mol L^{-1} $KHSO_4$ was used as a background solution, and the pH of the solution was changed when gradually adding NaOH 0.2 Mol L^{-1}.

As can be seen from the graph (Fig. 2), the height of the H_2O_2 reduction peak decreases, and the potential of the reduction peak is shifted toward more electronegative values with increasing the background electrolyte pH from 1.4 to 4.5. The maximum peak (I_p) is at the pH of approximately 2.2 and at a the pH around 4 the analytical signal almost disappears. The effect of the pH on the peak potential (E_p) shows the following: when the pH value increases in the interval from 2 to 3, E_p remains almost constant, but E_p decreases sharply to a negative value with the pH increasing over 3.5. Therefore, the optimal peak for the analysis (E_p = +0.16 V) was obtained at pH≈2.2-2.4 on the background of Na_2SO_4 and Mol L^{-1} $KHSO_4$.

For the quantitative determination of H_2O_2 in a standard pharmacopoeian solution the calibration curve method was used. The calibration curve equation was $I_p = (8.6\pm0.7)\times10^3\times c$ (r = 0.998) (Fig. 3). The results obtained are summarized in Tab. 1.

The high sensitivity of this method is accompanied by very good reproducibility. The reproducibility was evaluated from 5 repeated electrochemical signal measurements of model solutions with H_2O_2 concentrations of 5.10×10^{-5}, 6.80×10^{-5} and 8.50×10^{-5} Mol L^{-1}. Precision of the method developed with reference to the relative standard deviation (RSD) was 4.24%, 3.27% and 2.30%, respectively (n = 5, P = 0.95). The results obtained are summarized in Tab. 2.

Precision and accuracy of the voltammetric determination of H_2O_2 in the model solution of preparations were studied by analyzing five replicates of the sample

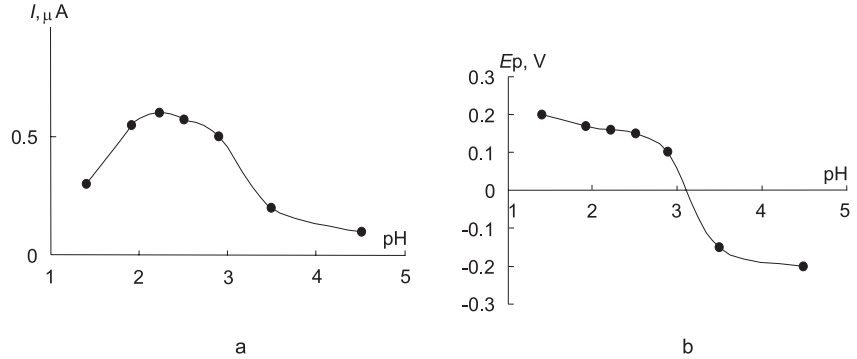

Fig. 2. The effect of the pH on the current peak intensity (*a*) and the peak potential (*b*) of the reduction process of H_2O_2 on CE (vs Ag,AgCl/KCl(sat)).

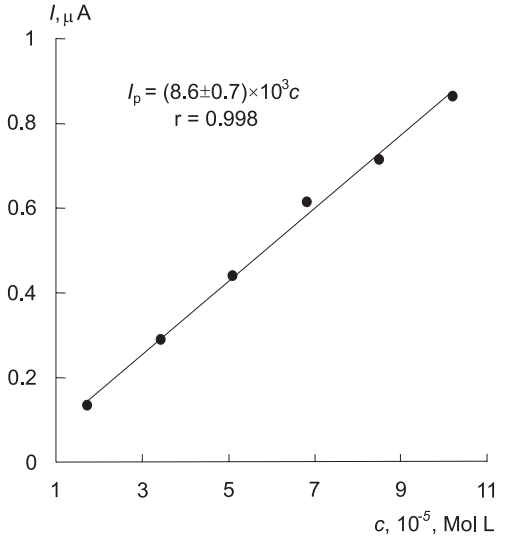

Fig. 3. The calibration graph of the H_2O_2 reduction current peak vs. the concentration on the background of 0.1 Mol L^{-1} Na_2SO_4 and 0.01 Mol L^{-1} $KHSO_4$ (pH≈2.4) on CE (vs Ag,AgCl/KCl(sat)); E_p = +0.16 V.

Table 1

Analytical characteristics of the calibration graph of the H_2O_2 voltammetric determination procedure in a standard pharmacopoeian solution (y = *a*x + *b*)

Parameters	Data
Concentration ranges (Mol L^{-1})	$(1.7\text{-}10.2)\times10^{-5}$
Regression equation	$I_п = (8.6\pm0.7)\times10^3 c$
a	8.6×10^3
b	0.011
Δ*a*	0.7×10^3
Δ*b*	0.05
S_a	0.3×10^3
S_b	0.02
Correlation coefficient (*r*)	0.998
LOD (Mol L^{-1})	6.16×10^{-6}
LOQ (Mol L^{-1})	2.05×10^{-5}

Table 2

The assessment of accuracy and precision of the H_2O_2 voltammetric determination procedure in the model solution of the standard pharmacopoeian solution (*n* = 5; P = 0.95%)

Taken (Mol L^{-1})	Found (Mol L^{-1})	Reproducibility (%±SD)	RSD (%)	ε (%)	δ* (%)
5.10×10^{-5}	$(5.13\pm0.27)\times10^{-5}$	100.64±5.31	4.24	5.27	+0.60
6.80×10^{-5}	$(6.86\pm0.28)\times10^{-5}$	100.88±4.10	3.27	4.06	+0.90
8.50×10^{-5}	$(8.51\pm0.24)\times10^{-5}$	100.06±2.86	2.30	2.85	+0.05

Note: * – In relation to the average reference method of permanganatometric titration [2].

Table 3

The results of voltammetric determination of H_2O_2 in the model solution of preparations (*n* = 5; *P* = 0.95%)

Object	Taken*(%)	Found (%)	Reproducibility (%±SD)	RSD (%)	ε (%)	δ* (%)
"Hydrogen peroxide, 3.0%"	3.08±0.19	3.03±0.08	98.44±2.59	2.11	2.63	–1.56
"Peroxygel, 3.0%"	3.00±0.30	2.98±0.09	99.33±3.03	2.45	2.63	–0.67

Note: * – The calculation was made according to the average content determined by the pharmacopeian procedure.

solutions at three concentration levels. The results obtained are summarized in Tab. 3.

CONCLUSIONS

Thus, a new voltammetric method of the H_2O_2 determination in a standard pharmacopoeian solution and the model solution of preparations, such as antiseptics "Hydrogen peroxide, 3.0%" and "Peroxygel, 3.0%" using CE as an indicating electrode has been developed, and the possibility of its quantitative determination has been shown.

The linear dependence is observed in the concentration ranges of the pure substance from 1.70×10^{-5} to 10.20×10^{-5} Mol L^{-1}. The calibration curve equation is $I_p = (8.6\pm0.7)\times10^3\times c$ (r = 0.998); LOD = 6.16×10^{-6} Mol L^{-1}, LOQ = 2.05×10^{-5} Mol L^{-1}. To determine H_2O_2 in preparations the standard addition method was used. The RSD was 2.11% (δ = –1.56%) for "Hydrogen peroxide, 3.0%" and 2.45% (δ = –0.67%) for "Peroxygel, 3.0%", respectively.

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КІЛЬКІСНЕ ВИЗНАЧЕННЯ ГІДРОГЕНУ ПЕРОКСИДУ МЕТОДОМ ВОЛЬТАМПЕРОМЕТРІЇ НА ВУГЛЕСИТАЛОВОМУ ЕЛЕКТРОДІ

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***Ключові слова**: визначення; гідрогену пероксид; вольтамперометрія; карбоситаловий електрод; антисептичний засіб*

Методом катодної вольтамперометрії з використанням як індикаторного вуглеситалового електроду вивчена електрохімічна поведінка гідрогену пероксиду в інтервалі потенціалів E = +1,0… –1,0 В (відн. нас. Ag,AgCl/KCl). Пік ($I_п$) був отриманий при $E_п$ = +0,16 В на фоні 0,1 Моль/л Na_2SO_4 та 0,01 Моль/л $KHSO_4$ (pH ≈ 2,4), висота якого зростає зі збільшенням концентрації H_2O_2. Лінійна залежність спостерігалася в інтервалі концентрацій (1.70-10.20)·10^{-5} Моль/л, рівняння градуювального графіка має вигляд: $I_п$ = (8,6±0,7)·10^3c (r = 0,998); LOD = 6,16·10^{-6} Моль/л, LOQ = 2,05·10^{-5} Моль/л. Для визначення H_2O_2 у розчинах антисептичних препаратів використовували метод добавок.

КОЛИЧЕСТВЕННОЕ ОПРЕДЕЛЕНИЕ ПЕРОКСИДА ВОДОРОДА МЕТОДОМ ВОЛЬТАМПЕРОМЕРИИ НА УГЛЕСИТАЛЛОВОМ ЭЛЕКТРОДЕ

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***Ключевые слова**: определение; пероксид водорода; вольтамперометрия; углеситалловый электрод; антисептическое средство*

Методом катодной вольтамперометрии с использованием в качестве индикаторного углеситаллового электрода изучено электрохимическое поведение пероксида водорода в интервале потенциалов E = +1,0… –1,0 В (отн. нас. Ag,AgCl/KCl). Пик ($I_п$) был получен при $E_п$ = +0,16 В на фоне 0,1 Моль/л Na_2SO_4 и 0,01 Моль/л $KHSO_4$ (pH ≈ 2,4), высота которого увеличивается с ростом концентрации H_2O_2. Линейная зависимость наблюдалась в диапазоне концентраций (1.70-10.20)·10^{-5} Моль/л, уравнение калибровочного графика: $I_п$ = (8,6±0,7)·10^3c (r = 0,998); LOD = 6,16·10^{-6} Моль/л, LOQ = 2,05·10^{-5} Моль/л. Для определения H_2O_2 в растворах антисептических препаратов использовали метод добавок.