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SIMULTANEOUS VOLTAMMETRIC DETERMINATION OF PERACETIC ACID AND THE COEXISTENT HYDROGEN PEROXIDE USING CARBOSITALL ROTATION ELECTRODE

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ABSTRACT: The aim of the present work is to determine the feasibility of peracetic acid (PAA) and the coexistent hydrogen peroxide (HP) simultaneous quantitative determination in disinfectant solution by voltammetry using carbositall rotation electrode (CE) as indicating electrode. It has been experimentally proved that the optimum pH for analysis is approximately 3.6. The linear relationship has been observed in the PAA concentration range $(3.12–12.50)\times10^{-5}$ mol L^{-1}, the calibration curve equation was $I_p = (3.78 \pm 0.46)\times10^3 c$ (r = 0.995); in the HP concentration range of $(0.94–3.76)\times10^{-4}$ mol L^{-1}, the calibration curve equation was $I_p = (3.57 \pm 0.26)\times10^3 c + (0.11 \pm 0.07)$ (r = 0.998). Determining PAA in the working solution with the concentrations of 6.24×10^{-5}, 7.80×10^{-5} and 9.36×10^{-5} mol L^{-1} the RSDs were 0.035, 0.028 and 0.022 respectively (δ = +0.30...+0.90%), determining HP in the working solution with the concentrations of 1.88×10^{-4}, 2.35×10^{-4} and 2.82×10^{-4} mol L^{-1} the RSDs were 0.028, 0.018 and 0.011 respectively (δ = –0.77…+0.92%). Determining PAA in the test solution of "Delakson" disinfectant with the concentrations of 0.1%, the RSD was 0.024 (δ = +0.9%). Determining HP in the test solution of "Delakson" disinfectant, the RSD was 0.012 (δ = +1.69%). Thus, a new voltammetric method of simultaneous determination of peracetic acid and the coexistent hydrogen peroxide in disinfectant on CE has been developed, and the possibility of its quantitative determination has been shown.

INTRODUCTION: Peracetic acid (PAA, CAS Number 79-21-0) was introduced as an antibacterial agent in 1955. It has a broad spectrum of activity, including bacteria, spores, molds, yeasts, algae, and viruses. PAA, a possibly safe oxidizing agent. It is increasingly used, especially as a high-level disinfectant in hospital establishments [1-2]. Hydrogen peroxide (HP, H_2O_2, CAS Number 7722-84-1) is a widely used antimicrobial chemical. It is used for preservative, disinfection, and sterilization applications [3-4]. It also has advantages concerning its toxicity and environmental profile [5].

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HP presence in PAA solutions is imminent according to several reasons: so as the synthesis of PAA is carried out by HP reaction and acetate acid - that is why the hydrogen peroxide is a constant technological admixture, and also, due to the course of the hydrolysis reaction, in aqueous solutions, there is a continuous unilaterally hydrolytic decomposition of PAA into HP and acetate acid.

PAA in combination with HP is used in certain disinfectants such as "Delakson", “Nukdez” (Ukraine), "Septacid", "Dezynfektor", "Steridial W" (Poland), "Sterisyl", "Sterioks" (Estonia) *etc.* This mixture shows bactericidal, tuberculocidal, virucidal, sporicidal and fungicidal properties [6-8] and intended for the final, flow-line and preventive object disinfection in health care institutions and nidus of intestinal and respiratory infections of bacterial and viral etiology, tuberculosis, dermatophytes and Sibirian plague, as well as for the sterilization of medical products (including rigid and flexible endoscopes) and suture material [9-11].

The extensive literature survey reveals various methods of PAA with HP determination [12,] such as titration [13-14], electrochemical [15-20], chromatographic [21-23,] and spectroscopic [24-25] methods have been used. It is obvious that two stepped titration method is not suitable for continuous monitoring because it is very time-consuming. Conductivity measurements are rapid and convenient, but their common disadvantage is their low selectivity. Spectroscopic methods have often been used for direct determination of a few species in aqueous solutions. Near-infrared (NIR) spectroscopy has recently been of keen interest as a practical technique for a variety of water and aqueous solution analyses. So far, a UV spectroscopic method for the direct determination of HP has not been reported probably because its UV absorption maximum is located at a very short wavelength (below 180 nm) and the extinction coefficients, obtained by ordinary UV-visible spectrometer, are very low. But all these methods are not sufficiently sensitive and furthermore require conducting of many chemicals, complicated cleaning and extraction procedures, and the use of cumbersome equipment may interfere with the whole procedure.

"Delakson" (“Delana”, Kyiv, Ukraine) is a complex disinfectant in the form of granulated water-soluble powder, which contains PAA (5-15%), HP (10-22%), acetate acid (22-26%) and stabilizing agents. The preparation is used in the form of water working solutions, and it’s prepared straight before the usage. It is allowed to store unused working solution for 5 days after producing in a container with a tightly closed lid at room temperature. The solution is used for disinfection during the day. The daily necessity of working solution concentration control is evident. The determination (mass fraction) of PAA in "Delakson" disinfectant is carried out by redox titration [26]. The usage of carbositall electrode for electrochemical determination of some peroxides in preparations has been studied in our previous works [27-31].

The aim of the present work is to determine the feasibility of peracetic acid and the coexistent hydrogen peroxide simultaneous quantitative determination in disinfectant solution by voltammetry using carbositall rotation electrode (CE) as indicating electrode.

MATERIALS AND METHODS: The stock solution of PAA with HP. The stock solution was prepared by accurately commercial preparation dissolving, and it is standardized by the recommended procedure [26]. 10.00 mL of obtained PAA solution was diluted in 100 mL volumetric flask with double distilled water to obtain 1.56×10^{-3} mol L^{-1} solution of PAA.

The solution of acetate buffer (pH = 3.6) was prepared by diluting of 425 mL of 1.00 mol L^{-1} solution of acetic acid and 50.0 mL 1.00 mol L^{-1} solution of NaOH in 500 mL volumetric flask with double distilled water at 20 °C.

The solution of ice acetic acid, 17.5 mol L^{-1} (AA) was prepared by diluting of 28.6 mL of concentrated (ice) solution of acetic acid in 500 mL volumetric flask with double distilled water at 20 °C.

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

The background solution consists of a mixture of the acetate buffer solution background (pH 3.6) and 0.1 mol L^{-1} Na_2SO_4.

The sample preparation which was subjected to the analytical procedures for the analysis of HP was "Delakson" disinfectant (“Delana”, Kyiv, Ukraine).

The test solution of "Delakson" disinfectant (0.1 % PAA, 0.9% HP) was prepared by dissolving 1.0 g of preparation in 1000 mL volumetric flask by double distilled water (standardized by [26]).

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

The electrochemical measurements have been carried out in 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 have been measured by the electrochemical sensor "Module EM-04" with the accuracy of ±5 mV. The CE has been used as a working and auxiliary electrode, and Ag, AgCl/KCl(sat) electrode type EVL-1M4 as a reference electrode.

The procedure of obtaining results for the calibration graph. Working solutions have been prepared by diluting different volumes of stock solution (1.00-4.00 mL) and 5 mL 1 mol L^{-1} Na_2SO_4 in 50 mL volumetric flask by the acetate buffer solution. 25 mL of the working solution was transferred to the cell. The voltammograms have been recorded by scanning the potential towards the negative direction in the potential range from +1.0 V to –1.0 V (*vs*. Ag, AgCl/KCl(sat)).

The graph was positioned in the following coordinates: the height of peaks I_p in μA at E_p = +0.15 V for PAA and E_p = –0.65 V for HP on the ordinate axis and corresponding concentration of PAA and HP respectively, *c* in mol L^{-1} on the abscissa axis. The graph equation coefficients have been calculated by the least square method.

The procedure of PAA and HP quantitative determination in "Delakson" disinfectant. Working solutions have been prepared by diluting different volumes (10.00-15.00 mL) of the stock solution and 5 mL 1 mol L^{-1} Na_2SO_4 in 50 mL volumetric flask by the acetate buffer solution. The voltammograms have been recorded by scanning the potential towards 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 has been calculated by the equation:

$$C_x = (I_p - b / a$$

Where I_p – the current peak of the working solution; *a*, *b* – graph equation coefficients.

PAA and HP mass fraction, % in the test solution has been calculated by the equation:

$$X\% = C_x \times M \times 100 \times V_0 \times 100\% / m \times 1000 \times 10 \times V$$

Where *M* – molar weight; V_0 – volumetric flask capacity; *V* – test solution volume; *m* – sample weight; 10 – stock solution volume; 100, 100 – volumetric flask capacities.

RESULTS AND DISCUSSION: Effect of nature and pH of background solution. The effect of pH on the reduction process has been investigated by recording voltammograms of stock solution (c(PAA) = 3.12×10^{-5} mol L^{-1}; c(HP) = 9.36×10^{-5} mol L^{-1}) at several pH values ranging from 2.15 to 4.78.

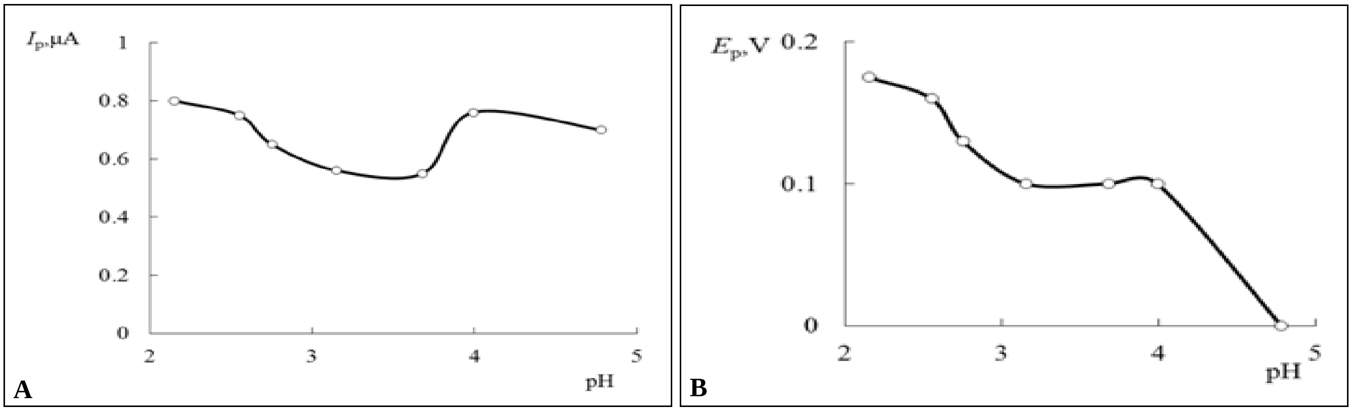

FIG. 1: THE INFLUENCE OF pH ON THE CURRENT PEAK INTENSITY (A) AND ON THE PEAK POTENTIAL (B) OF PAA REDUCTION PROCESS AT THE CE (*vs.* Ag, AgCl/KCl (sat))

A mixture of 1 mol L^{-1} Na_2SO_4 with acetate buffer solution has been used as a background solution, and the pH of the solution has been changed gradually by adding NaOH 0.2 mol L^{-1}. Two well-separated peaks (I_p) are observed at pH approximately 2.5-3.7, and at pH, about 4.78 analytical signal almost disappears. The effect of pH on the peak potential (E_p) shows the following: when pH value increases in the interval from 3 to 4, E_p remains almost constant, but E_p decreases markedly to the negative value with pH increasing over 4 **Fig. 1, 2**. That is why the optimum pH for analysis is approximately 3.6.

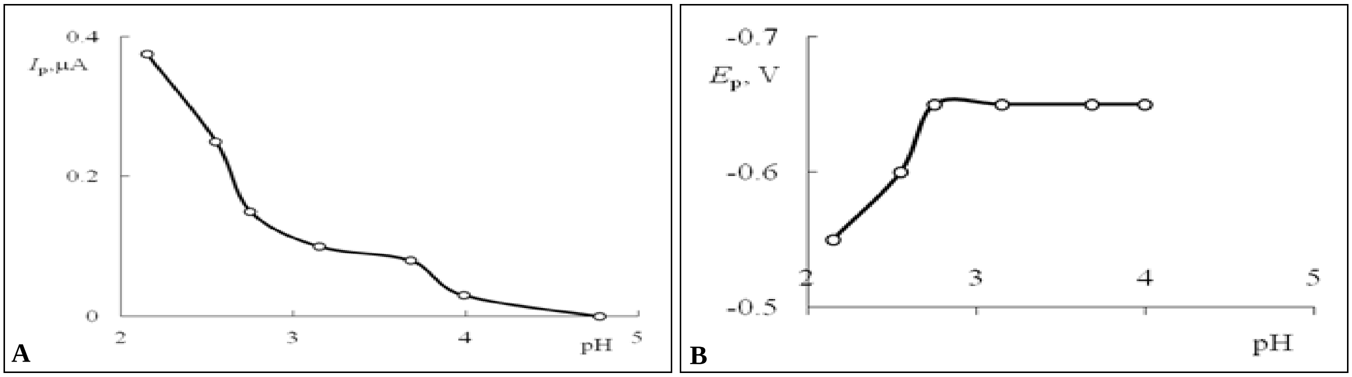

FIG. 2: THE INFLUENCE OF pH ON THE CURRENT PEAK INTENSITY (A) AND ON THE PEAK POTENTIAL (B) OF HP REDUCTION PROCESS AT THE CE (*vs.* Ag, AgCl/KCl (sat))

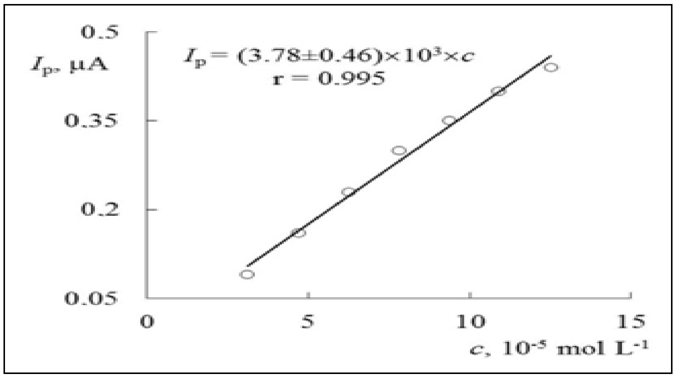

FIG. 3: THE CALIBRATION GRAPH OF THE PAA REDUCTION CURRENT PEAK *vs.* CONCENTRATION; E_p = +0.15 V

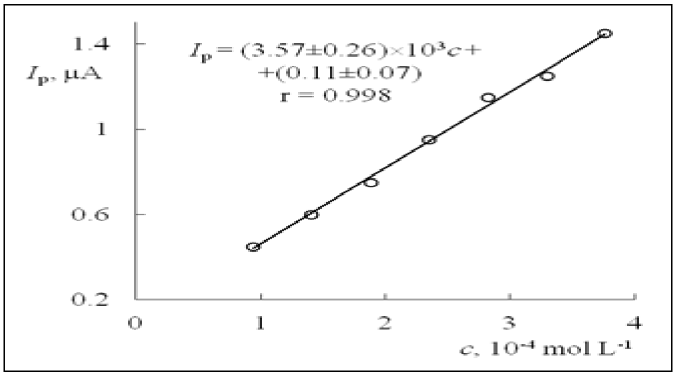

FIG. 4: THE CALIBRATION GRAPH OF THE HP REDUCTION CURRENT PEAK *vs.* CONCENTRATION; E_p = –0.65 V

For quantitative determination of PAA and HP in working solutions the calibration curve method was used. The calibration curve equation in the PAA concentration range $(3.12\text{-}12.50) \times 10^{-5}$ mol L^{-1} was $I_p = (3.78 \pm 0.46) \times 10^3 \times c$ (r = 0.995) **Fig. 3**; in the HP concentration range $(0.94\text{–}3.76) \times 10^{-4}$ mol L^{-1} was $I_p = (3.57 \pm 0.26) \times 10^3 c + (0.11 \pm 0.07)$ (r = 0.998) **Fig. 4**. Analytical characteristics of the calibration graph of the PAA and HP voltammetric determination procedure are given in **Table 1**.

TABLE 1: ANALYTICAL CHARACTERISTICS OF THE CALIBRATION GRAPH OF PAA AND HP VOLTAMMETRIC DETERMINATION PROCEDURE (y = *ax*+*b*)

Parameters	Data	
	PAA	HP
Concentration ranges (mol L^{-1})	$(3.12\text{-}12.50) \times 10^{-5}$	$(0.94\text{-}3.76) \times 10^{-4}$
Regression equation	$I_p = (3.78 \pm 0.46) \times 10^3 c$	$I_p = (3.57 \pm 0.26) \times 10^3 c + (0.11 \pm 0.07)$
a	3.78×10^3	3.57×10^3
b	0.013	0.11
S_a	0.46×10^3	0.26×10^3
S_b	0.038	0.07
Δa	0.18×10^3	0.10×10^3
Δb	0.015	0.025
Correlation coefficient (*r*)	0.995	0.998
LOD (mol L^{-1})	1.19×10^{-6}	2.15×10^{-5}
LOQ (mol L^{-1})	3.97×10^{-5}	7.18×10^{-5}

The high sensitivity of this method is accompanied by very good reproducibility. The reproducibility was evaluated from 5 repeated electrochemical signal measurements of working solutions with PAA concentrations of 6.24×10^{-5}, 7.80×10^{-5} and 9.36×10^{-5} mol L^{-1} and with HP concentrations of 1.88×10^{-4}, 2.35×10^{-4} and 2.82×10^{-4} mol L^{-1}. Precision of the method developed with reference to the relative standard deviations (RSDs) were 0.035, 0.028 and 0.022 respectively (δ = +0.30...+0.90%) for PAA; and 0.028, 0.018 and 0.011 (δ = –0.77…+0.92%) for HP. The results obtained are summarized in **Table 2**.

TABLE 2: EVALUATION OF ACCURACY AND PRECISION OF PAA AND HP VOLTAMMETRIC DETERMINATION PROCEDURE IN WORKING SOLUTION (n = 5; P = 0.95%)

	Taken (mol L^{-1})	Found (mol L^{-1})	Reproducibility (%±SD)	RSD	ε (%)	δ* (%)
PAA	6.24×10^{-5}	$(6.30 \pm 0.28)\times10^{-5}$	100.90 ± 4.41	0.035	4.37	+0.90
	7.80×10^{-5}	$(7.86 \pm 0.28)\times10^{-5}$	100.73 ± 3.53	0.028	3.50	+0.73
	9.36×10^{-5}	$(9.39 \pm 0.26)\times10^{-5}$	100.34 ± 2.78	0.022	2.75	+0.30
HP	1.88×10^{-4}	$(1.87 \pm 0.06)\times10^{-4}$	99.23 ± 3.42	0.028	3.45	-0.77
	2.35×10^{-4}	$(2.34 \pm 0.05)\times10^{-4}$	99.53 ± 2.34	0.018	2.35	-0.47
	2.82×10^{-4}	$(2.85 \pm 0.04)\times10^{-4}$	100.92 ± 1.41	0.011	1.40	+0.92

* In relation to the average reference method [26]

The high sensitivity of this method is accompanied by a very good reproducibility. The reproducibility has been evaluated from 5 repeated electrochemical signal measurements of test solutions of "Delakson" disinfectant. The precision of the developed method in terms of the relative standard deviation (RSD) was 0.024 (δ = +0.90%) for PAA and 0.019 (δ = +1.69%) for HP respectively. The obtained results have been summarized in **Table 3**.

TABLE 3: THE RESULTS OF HP VOLTAMMETRIC DETERMINATION IN "DELAKSON" DISINFECTANT (*n* = 5; *P* = 0.95%)

	Taken (mol L^{-1})	Found (mol L^{-1})	Reproducibility (%±SD)	RSD	ε (%)	δ* (%)
PAA	0.100	0.101 ± 0.003	100.90 ± 2.99	0.024	2.97	+0.90
HP	0.900	0.915 ± 0.013	101.69 ± 1.50	0.019	1.47	+1.69

* The calculation has been made according to the average content established using standard procedure [26]

CONCLUSION: Thus, a new voltammetric method of simultaneous determination of peracetic acid and the coexistent hydrogen peroxide in disinfectant on CE has been developed, and the possibility of its quantitative determination has been shown. The linear relationship has been observed in the PAA concentration range $(3.12–12.50)\times10^{-5}$ mol L^{-1}, the calibration curve equation was $I_p = (3.78 \pm 0.46)\times10^{3}c$ (r = 0.995); in the HP concentration range of $(0.94–3.76)\times10^{-4}$ mol L^{-1}, the calibration curve equation was $I_p = (3.57 \pm 0.26)\times10^{3}c + (0.11 \pm 0.07)$ (r = 0.998). Determining PAA in the working solution with the concentrations of 6.24×10^{-5}, 7.80×10^{-5} and 9.36×10^{-5} mol L^{-1} the RSDs were 0.035, 0.028 and 0.022 respectively (δ = +0.30...+0.90 %); LOD = 1.19×10^{-6} mol L^{-1}, LOQ = 3.97×10^{-5} mol L^{-1}. Determining HP in the working solution with the concentrations of 1.88×10^{-4}, 2.35×10^{-4} and 2.82×10^{-4} mol L^{-1} the RSDs were 0.028, 0.018 and 0.011 respectively (δ = –0.77…+0.92%); LOD = 2.15×10^{-5} mol L^{-1}, LOQ = 7.18×10^{-5} mol L^{-1}. Determining PAA in the test solution of "Delakson" disinfectant with the concentrations of 0.1%, the RSD was 0.024 (δ = +0.9%. Determining HP in the test solution of "Delakson" disinfectant, the RSD was 0.012 (δ = +1.69%).

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CONFLICTS OF INTEREST: Nil

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