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DETERMINATION OF THE PEROXIDES BY ELECTROCHEMICAL METHODS

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In review the electrochemical behavior of peroxides, typically used as disinfectants, antiseptics and bleaching agents (e.g. hydrogen peroxide, potassium peroxomonosulphate (Oxone®), sodium perborate, organic peracids, hydroperoxides, dialkyl peroxides and acyl peroxides) on the different electrode materials was introduced. At some electrodes an excellent separation of their redox peaks can be obtained and the results are utilized in the amperometric and voltammetric electroanalysis of these peroxides over a wide range of their concentrations. A simple novel methods was given for a simultaneous potentiometric analysis of these peroxides using platinum, gold, stainless steel, carbon glass electrodes etc. Factors affecting the selectivity, sensitivity and response time were examined in detail. The current research on peroxides detection is mainly focused on electrode modifications including the application of nanomaterials in order to decrease the overpotential and increase the electron transfer kinetics was showed. The possibility of peroxides determination by electrochemical methods with applications ranging from the medical, pharmaceutical and paper industry to household washing powder was demonstrated.

Keywords: determination, peroxides, electrochemical methods, disinfectant and pharmaceuticals.

Hydrogen peroxide (H_2O_2) and other peroxides are involved in a wide variety of industrial processes. Hydrogen peroxide, potassium peroxomonosulfate (PMS) and peroxydicarboxylic acids (PCA) are used as disinfecting and bleaching agents [1–7] with applications ranging from the medical, pharmaceutical and paper industry to household washing powder, and they are also attractive reactants for decontamination of chemical warfare agent [8–10].

Therefore development of analytical methods their quantitative determination has an outstanding important value. Numerous methods for determination of peroxides are described in the literature. In this review we will consider the modern electrochemical methods of quantitative determination of the most used peroxides in medicine and industry. Most of them are based on the oxidizing properties of peroxides.

In the early work [11] a titration (potentiometrically or with visual definition titration endpoint) of Caro acid with H_2O_2 in sulfuric acid medium solution by NaAsO_2 was investigated.

The original version of iodometric method for simultaneous determination of H_2SO_5 - $\text{H}_2\text{S}_2\text{O}_8$ - H_2O_2 , based on H_2O_2 titration by permanganate in acid solution followed by the

addition of iodine solution and back titration with thiosulfate to determine of H_2SO_5 was developed by Bodin [12]. Then Cu_2I_2 and KI were added to the solution and iodine was titrated by thiosulfate for determination $\text{H}_2\text{S}_2\text{O}_8$. It is also possible used potentiometric titration: H_2O_2 by permanganate, H_2SO_5 by thiosulfate and then $\text{H}_2\text{S}_2\text{O}_8$ by iodometric titration (after adding to solution a mixture Cu_2I_2 and KI).

Improvement of this method by adjusting the determination conditions was described in the [13]. A platinum electrode was used in titration of $\text{K}_2\text{S}_2\text{O}_8$, H_2SO_5 and KMnO_4 at pH 1.8. The pH was increased to 4 with sodium acetate for the titration H_2SO_5 with $\text{Na}_2\text{S}_2\text{O}_3$, and KI– CuSO_4 was added for final titration of $\text{K}_2\text{S}_2\text{O}_8$ with $\text{Na}_2\text{S}_2\text{O}_3$.

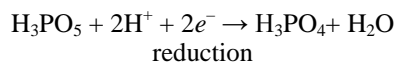
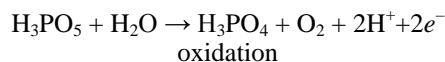
In the early Hungarian scientists work the methods based on direct titration each of peroxides by solution orthoarsenic acid with amperometric method of titration endpoint definition was described [14–15]. This method is also suitable for the determination of other peroxide compounds in the simultaneous presence, for example, H_2O_2 and PAA or peroxomonophosphoric acid (PMPA).

Spectrophotometric, titrimetric, and polarographic techniques were compared for stability testing of commercially available benzoyl peroxide (BP) pharmaceuticals. Initial testing revealed comparable results but significant discrepancies were observed on prolonged testing. The titrimetric procedure employed showed little degradation of the BP at elevated temperatures, while both the spectrophotometric and polarographic methods demonstrated radical potency losses under the conditions of testing. Agreement was excellent between the polarographic and spectrophotometric procedures above 70 per cent of the original assay with a significant divergence being noted only below this level. A rapid, reproducible procedure of sufficient accuracy for pharmaceutical product stability and control purposes was presented [16].

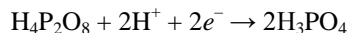
Potassium peroxydisulphate-alkali halide systems have been studied by direct current (dc-) and alternative current (ac-) polarography. When the mercury drop of the mercury electrode becomes covered by a film of adsorbed bromide or iodide ions, a peak due to peroxydisulphate (PDS) ion is found in the ac-polarogram at a potential more negative than that of the halide peak. This peak was probably due to a product of a chemical reaction between the PDS ion and halide ion close to the surface of the mercury electrode (DME). The determination of the PDS ion by ac-polarography was described [17].

In the paper [18] the possibility of selective potentiometric titration PMS by tin (II) solution in the presence of sulfuric acid and potassium bromide. Titration was carried out using spiral platinum and saturated calomel electrodes. Auxiliary electrode was connected by Agar bridge.

The voltammetric behavior of PMPA and peroxodiphosphoric acid (PDPA) on a platinum microelectrode with periodical renewal of the diffusion layer was studied. PMPA gave an oxidation and a reduction wave which corresponded to the following overall irreversible processes:

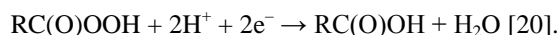


PDPA gave a wave corresponding to the irreversible reduction of $\text{H}_4\text{P}_2\text{O}_8$ according to the following scheme:



The voltammetric technique provided a method for the determination of the two peracids, even in the presence of H_2O_2 . The kinetics of the hydrolysis of PDPA to give PMPA had been studied by this method and the results supported the mechanism suggested by Crutchfield. The kinetic analysis of the reduction wave of $\text{H}_4\text{P}_2\text{O}_8$ at various acidities showed that one hydrogen ion is involved in the rate determining step of the overall electrode process and gave a lower limit for the second dissociation constant of PDPA. The mechanism of electrochemical reduction of PDPA and PDS was compared with the mechanism of their reaction with the iodide ion. Similarities between the electrode reactions and the reactions in the homogeneous phase were observed [19].

A voltammetric study of the reduction of some substituted perbenzoic acids in aqueous solution had been performed by using a platinum microelectrode with periodical renewal of the diffusion layer. The acids gave a cathodic wave corresponding to the irreversible process. Analysis of the reduction waves shows that the potential-determining step involved a molecule of undissociated peracid and one electron. The effect of substituents on the half-wave potential had also been investigated and the mechanism of electroreduction of peroxyacids was compared with the mechanism of their reduction by iodide ions. It reduced according to the overall equation:



An iodometric method for the rapid microdetermination of organic peroxides, utilizing electrochemical reduction in the iodine-iodide system, was developed. After complete reaction between peroxide and iodide in an acidic semi-aqueous medium the iodine produced is reduced at a rotating platinum electrode, at constant potential. Electronic integration is used. Organic peroxides of different reactivities had been analyzed by making use of the increased rate of reaction at elevated temperatures. Accurate and precise results were obtained for samples at the 1 mg level and the total time of analysis varied from 8 to 20 min, depending on reactivity [21].

A direct reductimetric method for the determination of PDS, hydrogen peroxide, sodium perborate, nitrate and nitrite in fairly concentrated phosphoric acid medium with iron(II) was developed, with both potentiometric and visual end-point detection. Cacoetheline, Methylene Blue, thionine, Azure A, Azure B, Azure C, Toluidine Blue, new Methylene Blue, ferroin, N-phenylanthranilic acid, p-ethoxychrysoidine and barium diphenylaminesulphonate were used as indicators. The method is useful in the analysis of binary mixtures of PDS and H_2O_2 or perborate and in the estimation of the nitrate content of fertilizers [22].

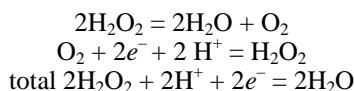
The electrochemical behavior of PMPA on platinized platinum was studied. The process of electroreduction was irreversible and involved one electron in the rate-determining step. From the dependence of the half-wave potential on the acidity it was concluded that the species H_3PO_5 , $[\text{H}_2\text{PO}_5]^-$, and $[\text{HPO}_5]^{2-}$ were simultaneously reduced. The electrode reaction was interpreted in terms of a mechanism involving nucleophilic attack by the electrode on the outer peroxidic oxygen of the depolarizer, and mechanistic analogies with the reduction of H_3PO_5 by iodide ion were discussed [23].

In [24] the electrochemical studies of H_2O_2 at platinum disc electrode under quiescent and steady state conditions, in the pH range of 2–12.3 in a universal buffer medium were carried out. At all pH values, a mixed cathodic-anodic wave was obtained. The anodic had being due to oxidation of peroxide to oxygen. At pH values below 6, one

more cathodic wave in addition to the mixed wave was obtained. The cathodic part of the mixed wave was due to the reduction of oxygen (founded by the catalytic decomposition of peroxide) to hydrogen peroxide. The additional cathodic wave was, however, due to the reduction of the undecomposed H_2O_2 to water. At pH values below six, the cathodic part of the mixed wave decreased, with the equivalent rise in the additional cathodic wave. Electrode kinetic-parameters were evaluated employing chronoamperometry, chronopotentiometry and rotating disc electrode voltammetry. Thus the possible mechanism for the reduction of peroxide can be summed up as follows:

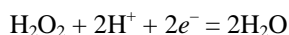
(1) At $\text{pH} < 6$ oxygen obtained from the decomposition of peroxide forms the cathodic part of the mixed wave and the second additional cathodic wave was due to the reduction of H_2O_2 to water.

(a) Cathodic part of the mixed wave.

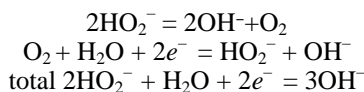


The observed reaction was the reduction of peroxide to water.

(b) Second cathodic wave



(2) At $\text{pH} > 6$, the reduction process was the same as (a) above. In alkaline solution



The voltammetric behavior of the catalytic reduction of perborate at carbon paste electrodes modified with Prussian blue was compared with that of hydrogen peroxide. Good linearity was observed between the voltammetric current and the concentration from 1 to 100 ppm H_2O_2 as perborate. The influence of the paste composition and other variables were studied [25].

The voltammetric procedures for PCA and acyl peroxides determination in mixtures based on selective PCA (peroxybutylacetic and peroxytetradecanoic acids) electrochemical reduction on a gold rotation electrode and PCA and acyl peroxides (lauryl peroxide, diacetyl dipersebacyanate) mixture electrochemical reduction on HCE on the background of dimethylformamide (C_2H_5)₄ NClO_4 0.1 mol l^{-1} solution were described. The peroxyacids reduction current on a gold disk electrode was directly proportional to the concentration at $E_{1/2} = 0.12$ V (vs. SCE) within $0.5\text{--}5 \times 10^{-3}$ mol l^{-1} [26].

The H_2O_2 concentration by a voltammetric method involving a rotating disk electrode (RDE) was determined. The oxidation limiting current at +0.4 V/SCE was proportional to the concentration of H_2O_2 founded in 1 mol l^{-1} KNO_3 containing a phosphate buffer, pH 7.5. An analytical blank was prepared «in situ» by addition of catalase enzyme to avoid interferences. The detection limit obtained was 5×10^{-9} mol l^{-1} [27].

A chemically modified electrode for the amperometric detection of H_2O_2 was described by electrodeposition of a copper heptacyanonitrosylferrate(II) (CuHNF) film on the surface of a glassy carbon (GC) electrode, the overvoltages for both oxidation and reduction of H_2O_2 can be drastically decreased. Cyclic voltammetry at the resulting modified electrode revealed the presence of two pairs of redox peaks corresponding to the $\text{Cu(I)}\text{--Cu(II)}$ and $\text{Fe(II)(CN)}_5\text{NO--Fe(II)(CN)}_5\text{NO}$ redox couples, respectively. The

modified electrode was less prone to fouling and shows enhanced sensitivity for hydrogen peroxide. Various electrochemical response characteristics were investigated. When applied as an electrochemical sensor in a flow-injection system, the modified electrode permitted detection of H_2O_2 at lower potentials than at the unmodified electrode surface. Thus greatly improved the selectivity. A linear amperometric response range with H_2O_2 was obtained between $0.4 \mu\text{mol l}^{-1}$ and 1 mmol l^{-1} . The relative standard deviation for 30 repetitive injections with a $10 \mu\text{mol l}^{-1}$ H_2O_2 solution over a 30 min period was less than 4 % [28].

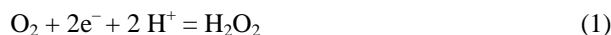
The electrocatalytic reduction of H_2O_2 was studied for carbonaceous electrodes modified with horse-radish peroxidase (HRP), microperoxidase (MP), and lactoperoxidase (LP). The carbonaceous electrodes were of three different graphites, carbon and GC. The peroxidase modified electrode was inserted as the working electrode in a flow through amperometric cell of the wall jet type and connected to a flow injection system. The effect of different pretreatments of the electrode surface prior to adsorption of the enzyme was investigated. Heating the electrodes in a muffle furnace at 700°C for 1.5 min was found to yield the highest currents. The electrocatalytic current for HRP-modified electrodes starts at about +600 mV vs. Ag/AgCl (pH 7.0) and reaches a maximum value at about -200 mV. For MP- and LP-modified electrodes the currents start at a lower potential (≈ 300 mV). For the best electrode material for HRP, straight calibration curves were obtained between 1 and $500 \mu\text{mol l}^{-1}$ H_2O_2 at 0 mV. The mechanism for the electron transfer from the electrode to the adsorbed peroxidase is discussed. Deliberate modification of the electrode surface with quinoid type electroactive species was found to mediate the reaction. It is proposed that spontaneously occurring electrochemically active surface groups mediate the electron transfer to the adsorbed enzyme. However, a contribution to the observed current from a direct electron transfer cannot be ruled out [29].

H_2O_2 in aqueous solutions can be determined directly by square wave voltammetry. The method was applicable to samples with a large range of pH in matrices ranging from distilled de-ionized water to sea-water. Its dynamic range extended from 0.5 to at least $1000 \mu\text{mol l}^{-1}$ and the precision was about $\pm 6\%$ at $2.5 \mu\text{mol l}^{-1}$ and $\pm 2\%$ at $215 \mu\text{mol l}^{-1}$. In comparison to dc- and differential pulse polarography, by using square wave voltammetry the scan time was reduced from minutes to a fraction of a second, the sensitivity was increased by several-fold and the dynamic range had been greatly expanded at both the lower and the upper end by at least an order of magnitude. The low detection limit allowed this method to be applied to the determination of H_2O_2 in some samples of rainwater [30].

Several organic peroxides were studied by differential pulse polarography in water and water-solvent media, and compared to hydrogen peroxide. Reduction occurred usually between -0.1 V and +0.3 V vs. SCE, except for the peroxides containing a *tert*-butyl group adjacent to the -O-O- function. In this case, reduction was more difficult and was shifted to the -0.9 V to -1.1 V vs. SCE range. Diffusion coefficients were determined and are in the $4\text{--}9 \times 10^{-10} \text{ m}^2 \text{ s}^{-1}$ range. The detection limits were generally no lower than $5 \times 10^{-5} \text{ mol l}^{-1}$, due to the high irreversibility of the reduction process. Calibration plots from 5×10^{-5} to about $5 \times 10^{-5} \text{ mol l}^{-1}$ exhibited very good linearity [31].

Differential pulse and current sampled (TAST) polarography were applied for measuring H_2O_2 in actual washing systems. In addition to the possibility of the H_2O_2 determination in commercial detergent species containing sodium perborate monohydrate

as bleaching substance, the change in peroxide concentration can be followed under the conditions which usually exist in the washing process. The possibility of determining dissolved oxygen in the peroxide decomposition reaction was also demonstrated. The reduction takes place in two two-electron processes. At pH 9.2 both are irreversible and correspond to



At pH < 9.2 the half-wave potentials of both of these steps are pH-independent, indicating that electron transfers are potential determining and that proton transfers occur in processes consecutive to electron transfers. At pH 11 the reduction in the first step was reversible. Standard deviation of the analytical method described was of the order of 2 to 4 % (n = 10) [32].

The electrochemical reduction of five dialkyl peroxides in dimethyl formamide was studied by cyclic voltammetry. The electron transfer (ET) to the selected compounds is concerned with the oxygen–oxygen bond cleavage (dissociative ET) and is independent of the electrode material. Such an electrochemical behavior provided the opportunity to study dissociative ETs by using the DME and therefore to test the dissociative ET theory by using heterogeneous activation-driving force relationships. The convolution voltammetry analysis coupled to the double-layer correction led to reasonable estimates of the standard potential (E°) for the dissociative ET to dialkyl peroxides, as supported, whenever possible, by independent estimates. A thermochemical cycle based on the dissociative ET concept was employed to calculate the bond dissociation free energies (BDFEs) of the five peroxides, using the above E° s together with electrochemical or thermochemical data pertaining to the redox properties of the leaving alkoxide ion. The BDFEs were found to be in the 25–32 kcal/mol range, suggesting a small substituent effect. The dissociative ET E° s were also used together with the experimental quadratic free energy relationships to estimate the heterogeneous reorganization energies. Cyclic voltammetry is useful for quantitative estimation of these peroxides with $\pm 1\%$ accuracy, upto 10^{-4} mol l⁻¹ concentration [33].

Ultramicroelectrode arrays have been applied for monitoring the disinfectant H₂O₂ in swimming pool water. The sensors were manufactured using silicon thin-film technology. Each consisted of nine miniaturized platinum working electrodes connected in parallel. A platinum counter electrode and a silver/silver chloride reference electrode completed the measuring setup. The ultramicroelectrode arrays were characterized by cyclic voltammetry and chronoamperometry. The sensor properties could be improved and the interfering influence of the swimming pool water matrix was reduced by covering the working electrodes of the arrays with NAFION™ membranes. A comparison of the introduced chronoamperometric method using the ultramicroelectrode array with the German standard method (DIN) for the H₂O₂ determination showed that the accuracy of the NAFION™ coated sensors is suitable for monitoring and controlling the H₂O₂ concentration in swimming pool waters [34].

The possibility of evaluating the content of H₂O₂ in several authentic matrices, such as cosmetic and pharmaceutical formulations, was studied. A new catalase biosensor fabricated using an amperometric gas-diffusion oxygen sensor as electrochemical transducer and the catalase enzyme immobilized in kappa-carrageenan gel and capable of operating in both aqueous and non aqueous solvents was developed and tested for this purpose. Creams, emulsions and disinfectant solutions were analyzed. To this end, a preliminary check was needed to establish the best conditions to analyze these matrices; the choice of

solvent was one of the most important points studied. The solvents considered included dioxane, water-dioxane mixtures, water saturated chloroform and aqueous solutions. The different solubility properties of the matrices analyzed were taken into account [35].

Reversed-phase high-performance liquid chromatography with oxidative amperometric detection was optimized for the determination of several organic peroxides in drinking water under ideal conditions. The determinations were performed under isocratic conditions using acetonitrile and methanol as the organic modifiers with 0.05 mol l^{-1} potassium phosphate buffer solution. The oxidative amperometric response of the organic peroxides was dependent on the concentration of organic modifier and the electrode potential. The optimum electrode potential for the simultaneous determination of the organic peroxides was approximately $+1.15 \pm 0.05 \text{ V}$ versus the Ag/AgCl reference electrode. The maximum analytical signal for butan-2-one peroxide and *tert*-butyl hydroperoxide, when using acetonitrile, was obtained with 20% v/v organic modifier. For cumene hydroperoxide, the maximum analytical signal was achieved with approximately 35 % v/v acetonitrile. The retention time of cumene hydroperoxide, on an octyldecylsilane column (250×4 mm id), decreased sharply from > 100 to < 10 min when the organic modifier concentration was varied from 5 to 50 % v/v. The retention time of butan-2-one and *tert*-butyl hydroperoxide, under the same conditions, varied by < 10 min. The calibration curves for the aliphatic peroxides and aromatic peroxide were linear from 2 to 200 ng and from 0.2 to 200 ng injected, respectively [36].

The kinetics and mechanism for the electrochemical oxidation of H_2O_2 on platinum electrodes in phosphate buffers were studied. A mechanistic model for this reaction was developed that involves binding sites, on the surface of the electrode, that are thought to be based on some form of hydrous platinum oxide, initially identified as $\text{Pt}(\text{OH})_2$. H_2O_2 adsorbs onto the binding sites to form the complex $\text{Pt}(\text{OH})_2\cdot\text{H}_2\text{O}_2$. The complex then undergoes internal electron transfer to form a reduced platinum site, Pt, with the release of the products water and oxygen. The binding sites regenerate electrochemically to give rise to an amperometric signal together with the release of protons. Two side reactions were proposed, the first involved a competitive inhibition of the binding sites by oxygen to form the species $\text{Pt}(\text{OH})_2\cdot\text{O}_2$. The second involved a non-competitive inhibition of the complex $\text{Pt}(\text{OH})_2\cdot\text{H}_2\text{O}_2$ by protons. A maximum response was found at pH 6.8 and decreased markedly at more basic or acidic conditions. This pH-dependence suggested that H_2PO_4^- was the species involved in the formation of the binding sites. The decrease in response at $\text{pH} > 6.8$ being caused by the decrease in $[\text{H}_2\text{PO}_4^-]$, whilst an inhibition of the precursor site by protons was proposed to account for the depression in electrode response at $\text{pH} < 6.8$. The influence of chloride upon the kinetics of H_2O_2 oxidation was examined and described qualitatively in terms of the new model. It was found that the rate of oxidation was decreased markedly in the presence of chloride. Two possible inhibition modes for chloride were identified and it was established that a non-competitive inhibition of the precursor sites was likely to be the dominant cause for the chloride inhibition. The work described in this thesis has not only identified a new and comprehensive mechanism for the oxidation of H_2O_2 at platinum electrodes, but also provides information that may prove useful when designing sensors that rely upon this reaction. In particular the important role of hydrodynamic conditions, buffer composition and concentration are clearly identified [37–41].

Cyclic voltammetric studies on the reduction of *di-tert*-butylperoxide and *tert*-butylhydroperoxide have been carried out in three dipolar aprotic solvents, namely, dimethyl

formamide, dimethyl sulphoxide and acetonitrile, at a GC electrode. A single irreversible peak corresponding to a two-electron reduction of these peroxides to the corresponding alkoxides, was observed in each of the three solvents. The values of voltammetric peak potentials were compared in the three solvents. The reduction was observed to be diffusion limited for lower concentrations and adsorption limited at higher concentrations of the peroxide. Cyclic voltammetry was found useful for quantitative estimation of these peroxides with ± 1 % accuracy, upon 10^{-4} mol l⁻¹ concentration [42].

The combination of cathodic amperometric detection with capillary zone electrophoresis was demonstrated to be a versatile method for the quantification of organic and inorganic peroxides. A gold microelectrode, polarized at 2600 mV against an Ag/AgCl reference electrode, was placed at the end of the capillary. Since the electroosmotic flow purges the detector electrode from oxygen, no degassing of the detector cell or the sample was necessary. With an injection volume of ca. 1 nl, H₂O₂, peroxosulfate, peroxy alkanic acids and the hydroperoxides of linoleic acid can be detected down to 10 μ mol/l. Separation of the isomeric hydroperoxides of the unsaturated fatty acids was achieved by addition of β -cyclodextrin to the electrolyte [43].

The voltammetric behavior of H₂O₂ at a GC electrode in alkaline solution was studied. An anodic prewave, preceding the well-known transport controlled oxidation reaction of H₂O₂, was observed. The voltammetric current in this wave was independent of the rotation speed of the GC disc electrode, even in what looks like a limiting current plateau, denoted by a pseudo limiting current. This current was dependent strongly on pH and temperature, didn't vary linearly with H₂O₂ concentration and the reaction orders, with respect to H₂O₂ and hydroxyl ions (pH), and was dependent on the H₂O₂ concentration. A mechanism that can explain these experimental results had been proposed. It consisted of two submechanisms, occurring simultaneously at the electrode surface, which are in competition with each other. Their relative predominance was controlled by the H₂O₂ concentration, which can explain the varying reaction orders with respect to hydroxyl ions and hydrogen peroxide. The current equation resulting from the proposed mechanism showed the possibility of using the studied electrode reaction for the amperometric determination of H₂O₂ up to high concentrations [44].

The electrochemical behavior of PAA in the presence of H₂O₂ by cyclic and square wave voltammetry was studied. Using gold electrode, two well-separated peaks for the reduction of PAA and H₂O₂ were for the first time observed on their cyclic and square-wave voltammograms. The two peaks were utilized in the simultaneous analysis of both species. Good calibration curves were obtained for each species over a wide range of their concentrations [45].

A method for the polarographic determination of organic peroxyacids in aqueous solutions at DME was developed and the possibility of its determination was shown. The procedure is commonly used for mono-(C₂-C₁₂) and diperoxy carboxylic (C₄-C₁₀) aliphatic as well as *m*-chloroperbenzoic and monoperoxyphthalic acids. The possibility of simultaneous PCA and H₂O₂ determination in weakly acidic and alkaline solutions by presented method was shown. The quantity of the limiting diffusion current values (I_{lim}) was proportional to PCA and H₂O₂ concentration regardless of the PCA nature, and the quantity of values $I_{lim}/\Delta c$ in the range of 2×10^{-5} – 2×10^{-4} mol l⁻¹ to about 1.7 times higher for di-PCA than for mono-PCA. The LOD for di-PCA at pH 8–9.2 was 3×10^{-5} mol l⁻¹, for H₂O₂ – 4×10^{-5} mol l⁻¹. Relative error of 8×10^{-4} mol l⁻¹ organic peroxyacids determination was < 6% (P = 95 %, n = 7) [46].

An indirect polarographic method of organic peroxyacids determination at DME by iodate, which was obtained by the quantitative oxidation of free iodine by the determined peroxyacids, has been worked out. The peroxyacid detection limit, LOD ($P = 95\%$, $n = 7$) was 0.25–0.5 $\mu\text{g/ml}$, limit of quantitation, LOQ was 0.5–1 $\mu\text{g/ml}$, relative error of organic peroxyacids determination was 5% in the proposed analysis in ac-mode [47].

The electrochemical behavior of PAA in the presence of H_2O_2 using cyclic voltammetry and hydrodynamic techniques [RDE voltammetry and rotating ring-disk electrode (RRDE) voltammetry] was investigated. The results aiming at simultaneous electroanalysis of both species were analyzed. GC and gold electrodes were used for this investigation. It was found that the reduction of PAA, as well as H_2O_2 , is highly sensitive to the electrode material; for example, at 100 mV s^{-1} , the reduction peak potentials of PAA were 0.2 and -1.1 V at gold and GC electrodes, respectively. The well-separated steady-state limiting currents were obtained using a gold electrode for the reduction of both PAA and H_2O_2 and also a well-defined one for the oxidation of H_2O_2 . On the basis of the RDE experiments, good calibration curves were obtained for both species over a wide range of their concentrations, for PAA and H_2O_2 in the range of 0.36 to 110 and 0.11 to 34 mmol l^{-1} , respectively. The simultaneous and selective electroanalysis of PAA and H_2O_2 in their coexistence was demonstrated for the first time [48].

The simultaneous electroanalysis of PAA and H_2O_2 in the presence of a large excess of H_2O_2 , up to hundred times the concentrations of PAA, was presented for the first time. Square wave voltammetry using a gold electrode was successfully applied for this purpose. It was found that a careful electrochemical pretreatment of the gold electrode is the key for obtaining reproducible data. Good calibration curves for the reduction of both species were obtained with correlation coefficients higher than 0.996. The detection limit for PAA was found to be 45 $\mu\text{mol l}^{-1}$ [49].

A microfluidic analytical system for the separation and detection of organic peroxides, based on a microchip capillary electrophoresis device with an integrated amperometric detector, was developed. The new microsystem relies on the reductive detection of both organic acid peroxides and hydroperoxides at 2700 mV (vs. Ag wire/AgCl). Factors influencing the separation and detection processes were examined and optimized. The integrated microsystem offers rapid measurements (within 130 s) of these organic peroxide compounds, down to micromolar levels. A highly stable response for repetitive injections (RSD 0.35–3.12%; $n = 12$) reflects the negligible electrode passivation. Such a «lab-on-a-chip» device should be attractive for on-site analysis of organic peroxides, as desired for environmental screening and industrial monitoring [50].

The stoichiometry of the formation of H_2O_2 from ascorbic acid in a model wine system by square-wave voltammetry (SWV) on a hanging mercury drop electrode was examined. Both ascorbic acid and H_2O_2 could be determined in the same test sample by first employing an anodic scan for ascorbic acid and then a cathodic scan for hydrogen peroxide. Linear calibration plots were obtained for ascorbic acid (up to 235 mg/L ; 1.335 mmol l^{-1}) and H_2O_2 (up to 0.12 $\mu\text{mol l}^{-1}$), both ranges being appropriate given the concentrations likely to occur in white wine. The reaction stoichiometry for the oxidation of ascorbic acid to the production of H_2O_2 showed significant deviation from the expected value of 1:1. Evidence is presented to suggest that this deviation from the expected stoichiometry is due in part to the generated H_2O_2 reacting with residual ascorbic acid. The generation of ascorbic acid in model wine base solutions containing dehydroascorbic acid was observed but H_2O_2 was not a critical component of this interconversion reaction. The implication of these observations for the browning of white wines is discussed. The advantages of

square-wave voltammetry for the real time measurement of the reaction sequences involving ascorbic acid are highlighted [51].

The determination of potassium monoperoxosulfate persisting in some disinfectants in the form of triple salt ($2\text{KHSO}_5 \cdot \text{KHSO}_4 \cdot \text{K}_2\text{SO}_4$) based on titration by tin(II) chloride solution in the presence of potassium bromide at 70°C with potentiometric registration by means of point platinum and glass pH electrodes was presented. The analysis of «Virkon» solution was performed [52]. The disadvantages of this technique are the necessity of heating the solution of the test sample to a temperature of 70°C , and the instability of the titrant solution.

The development and characterization of a biosensor for hydroperoxides was described, which was obtained by combining an oxygen gas diffusion amperometric electrode and two immobilized enzymes (peroxidase and tyrosinase) working in parallel and competing for the same substrate (catechol). The response of the biosensor to several hydroperoxides was investigated ($\text{LOD} = 5 \mu\text{mol l}^{-1}$ for hydrogen peroxide). It was experimentally founded that the biosensor is able to respond also to aqueous solutions of ionic peroxides ($\text{LOD} = 2 \mu\text{mol l}^{-1}$ for PDS). The biosensor was applied to the determination of the H_2O_2 content of pharmaceutical products, i.e. aqueous disinfectant solutions ($\text{RSD}\% \leq 0.5$; recoveries by standard addition method between 96.0 and 98.5%) [53].

A rapid and highly selective potentiometric determination method for the simultaneous analysis of PAA and H_2O_2 was proposed, for the first time, using GC as an indicator electrode and I_2/Γ potential buffer. On the basis of the large difference in the reaction rates of PAA and H_2O_2 with Γ^- a transient potential response corresponding to the reactions of the two species with Γ^- was observed. The response times were typically a few seconds and several minutes of PAA and H_2O_2 , respectively. The potential response obtained using the GC indicator electrode was found to be Nernstian over a wide range of their concentrations (from micromolar to millimolar) with slopes of 30.5 and 29.5 mV for PAA and H_2O_2 , respectively [54].

A rapid and highly selective potentiometric method for the simultaneous analysis of PAA and H_2O_2 was proposed, for the first time, using GC as an indicator electrode and I_2/Γ potential buffer. On the basis of the large difference in the reaction rates of PAA and H_2O_2 with Γ^- , which was confirmed using stopped-flow spectrophotometry, a transient potential response corresponding to the reactions of the two species with Γ^- was observed. The response times were typically a few seconds and several minutes for PAA and H_2O_2 , respectively. The effects of the concentrations of molybdate catalyst, H^+ , I_2 , and Γ^- in the potential buffer on the selectivity as well as the sensitivity were examined. The potential response obtained using the GC indicator electrode was found to be Nernstian over a wide range of their concentrations (typically from micromolar to millimolar) with slopes of 30.5 and 29.5 mV for PAA and H_2O_2 , respectively (in close agreement with the theoretical value, that is, 29.6 mV). O_2 was found to have no substantial effect on the potential change at the GC electrode in the present potential buffer [55].

The potentiometric analysis of PAA in the presence of a large excess of H_2O_2 , up to 500 times the concentrations of PAA, was presented for the first time. This method is based on the detection of the potential change of the GC indicator electrode in the Γ^-/I_2 potential buffer which is caused by the change in the concentrations of Γ^- and I_2 as a result of the redox reaction of the PAA and/or H_2O_2 with Γ^- . Based on the fact that the reaction rate of PAA and Γ^- is much faster than that of H_2O_2 and Γ^- , a high selective response for PAA was obtained. The different factors, such as the concentrations of Γ^- and I_2 in the potential buffer, affecting the selectivity and sensitivity was studied. A good

calibration curve for PAA, the slope of which is in close agreement with that expected from the Nernst equation, i.e., 29.6 mV per decade, was obtained with a correlation coefficient higher than 0.993. The detection limit for PAA was found to be in the micromolar range depending on the concentration of the coexistent H_2O_2 [56].

Ozone (O_3) and H_2O_2 (the mixture is commonly called peroxone) have been analyzed potentiometrically using Pt indicator electrode and I/I_3^- redox couple as a probing potential buffer. The analysis is based on the measurement of the change of the open circuit potential of the indicator electrode as a result of the reaction of the oxidant mixture and I^- . The large difference in the reaction rates of the oxidation of I^- by the two oxidants enabled a selective and simultaneous analysis of both oxidants: the oxidation of I^- by O_3 is instantaneous and results in a transient potential change, while that by H_2O_2 is comparatively slow and it takes a few minutes for a steady state potential change to be attained. The experimental factors affecting the oxidation of I^- by the two oxidants were examined for optimizing the selectivity. Further, the equation describing the time dependency of the potential change was derived by considering the rate constants of the reactions of I^- and the oxidants and it was found to fit the experimental results. The detection limits for O_3 and H_2O_2 were estimated to be $0.3 \mu\text{mol l}^{-1}$ with correlation coefficients higher than 0.995, over their concentration range of micro and submicromolar level [57].

The kinetics of electroreduction of PAA at a gold electrode in acetate buffer solutions of pH in the range 3–6 was studied. The electroreduction of PAA was irreversible. The relevant kinetic parameters including the Tafel slope, the standard rate constant (k^0), the electrochemical reaction order (m), the product (an_a) of the transfer coefficient and the number of electrons involved in the rate-determining step, and the number (n) of electrons involved in the overall electrode reaction were determined using cyclic voltammetry, hydrodynamic voltammetry, and hydrodynamic chronocoulometry. The Tafel slope = 0.215 V/decade, $m = 1$, $k^0 = 9.4 \times 10^{-11} \text{ cm s}^{-1}$, $an_a = 0.32$, and $n = 2$. The formal potential of the PAA/acetic acid redox couple was also theoretically calculated to be 1.762 V vs. Ag/AgCl based on the equilibrium constant for the reaction of PAA and H_2O_2 in the presence of acid catalyst and the standard redox potential of H_2O_2 . The diffusion coefficient of PAA was also estimated. Based on the obtained results, the overall electroreduction of PAA is considered to be composed of two one-electron reduction steps with the formation of acetate or hydroxyl radical as probable reaction intermediates [58].

Electrochemical behavior of PAA and H_2O_2 was examined at various metal and carbon electrodes (i.e., Au, Ag, Cu, Pt, Pd, Rh, Ti, W, Hg, Ni, Fe, GC, and basal-plane pyrolytic graphite (BPG)) in 0.1 mol l^{-1} acetate buffer solution (pH 5.5) using potentiostatic (i.e., cyclic voltammetry and RDE voltammetry) and galvanostatic techniques. It was found that the electro-reductions of PAA and H_2O_2 are highly sensitive to electrode material. Both species were found to be electrochemically and separately reduced at Au, Ag, Cu, Pt, Pd, GC, and BPG electrodes. On the other hand, at Fe, Ni, Hg, Rh, Ti, and W electrodes, voltammetric response for the PAA reduction was not obviously observed. The kinetics of electro-reduction of PAA in 0.1 mol l^{-1} acetate buffer solution was studied at Au, Ag, and GC electrodes in details, and the relevant kinetic parameters (i.e., the exchange current density, j_0 , the standard rate constant, k_0 , and cathodic transfer coefficient, α_c) were estimated from the Tafel plots. The cyclic voltammetric reduction peak potentials obtained for the PAA reduction at Au, Ag, and GC electrodes were

compared with those calculated using the kinetic and thermodynamic parameters obtained under the same experimental conditions. The measured and calculated reduction peak potentials at each electrode were found to be in agreement with each other, indicating that the evaluated values of kinetic parameters for the reduction of PAA at Au, Ag, and GC electrodes are reasonable [59].

BP was determined by differential pulse voltammetry using a GC electrode in dichloromethane-acetic acid (1.5×10^{-2} mol l⁻¹) solution and tetrabutyl ammonium perchlorate (0.01 mol l⁻¹) as the supporting electrolyte. The peak potential was -0.045 V (vs. Ag/AgCl). There was a good linear relationship between the peak current and BP concentration in the range of 2.5×10^{-6} – 1.0×10^{-4} mol l⁻¹. The detection limit of the method was 2.5×10^{-7} mol l⁻¹. The samples of the local wheat flour were directly determined with the desirable results [60].

This paper [61] presented an on-line monitoring device for PDS concentration that uses a coulometric measuring method. The method was based on the redox potential control in a miniaturised electrochemical reactor. This paper presented the design of the measuring cell and described the integration of this cell in an on-line measuring device which can be applied in a process environment. Experimental results illustrated its dynamic characteristics and accuracy.

In review [62] the electrochemical behavior of oxidants, typically used as disinfectants (e.g. hydrogen peroxide, ozone, hypochlorite and peracids) on the different electrode materials was introduced. At some electrodes an excellent separation of their redox peaks can be obtained and the results are utilized in the amperometric electroanalysis of these oxidants over a wide range of their concentrations. Further a simple novel method was given for a simultaneous potentiometric analysis of these oxidants using the iodide/iodine redox couple as a so-called potential buffer. Factors affecting the selectivity, sensitivity and response time were examined in detail.

Based on preliminary voltammetric investigations at both Pt and Au electrodes in aqueous solutions buffered at different pH values in the range 0–10, two possible profitable triple-pulse amperometric approaches were developed for determining simultaneously PAA and H₂O₂ present in the same samples. At both surfaces a pulsed waveform applied at rotating-disc electrodes was adopted to take advantage on one hand of the optimized signal reproducibility achieved by this potential multistep antifouling approach and on the other hand of the constant thickness of the diffusion layer, which is necessary when the recording of time-independent currents is desired. At a rotating disc Pt electrode an anodic selective signal was indeed recorded for H₂O₂ alone, while PAA contents could be inferred only from the difference of convenient signals, since at all pHs explored its sole cathodic reaction could be observed at potentials coincident with those proper for the reduction of H₂O₂ too. The same pulse approach at Au electrodes instead provided totally independent signals for the two analytes considered, thus proving to be suitable for their independent detection. In fact, H₂O₂ alone undergoes anodic oxidation also at this surface, while the reduction of PAA occurs at potentials less cathodic than those required for H₂O₂. At both electrodes, the best results turned out to be achieved at pH 0 in terms of both precision (± 2 –4%) and detection limits (0.2–0.3 mmol l⁻¹), as well as of linear range which extended for about three orders of magnitude. The kinetics of the equilibrium involving the generation of H₂O₂ from the reaction of PAA with water was also evaluated, since it was suspected of making unreliable the proposed amperometric approaches [63].

Oxidative chemisorption and cathodic stripping reductive desorption of iodide have been studied at a smooth polycrystalline gold (Au(poly)) electrode. Potential-dependent surface coverage of iodide has been controlled on the basis of its reductive desorption in 0.1 mol l^{-1} KOH alkaline media and its quantitative oxidation to aqueous iodates in acidic media. The Au (poly) electrode surface catalyzes the decomposition of H_2O_2 to O_2 . Specific adsorption of iodide on the Au electrode inhibits fully the catalytic decomposition and electrochemical oxidation of H_2O_2 as well as the adsorption of unknown impurities and the oxidative degradation of the electrode surface by H_2O_2 . A quantitative characterization/detection of H_2O_2 at the iodide-modified Au (poly) electrode in the alkaline media has, thus, been achieved. Performance of the electrode toward the detection of H_2O_2 with respect to response time and sensitivity as well as operational stability has been evaluated. It has a sensitivity of $0.272 \text{ mA cm}^{-2} \text{ m mol}^{-1}$ in amperometric measurements with a detection limit of $1.0 \text{ } \mu\text{mol l}^{-1} \text{ H}_2\text{O}_2$, and the response time to achieve 95 % of the steady-state current is $<20 \text{ s}$. The effect of O_2 in the air-saturated solution can be minimized by subtracting the additional current for the O_2 reduction. Experimental measurements were based upon cyclic voltametric and amperometric techniques [64].

Peroxydic acid (PCA) was successfully synthesized for the first time as an equilibrium mixture with citric acid and H_2O_2 using sulfuric acid (homogeneous) and Nafion (heterogeneous) catalysts. The formation of PCA in the equilibrium mixture was detected by the potentiometric measurement based on the triiodide/iodide redox couple potential buffer and the concentration of PCA was determined from the obtained potential change. The equilibrium % yield of PCA was found to largely depend on the concentration and the nature of catalysts. The peculiar roles of the heterogeneous and homogeneous catalysts in the formation of PCA were discussed briefly [65].

The electrochemical reduction of peroxydic acid (PCA) coexisting with citric acid and H_2O_2 in the equilibrium mixture was extensively studied at a gold electrode in acetate buffer solutions containing $0.1 \text{ mol l}^{-1} \text{ Na}_2\text{SO}_4$ (pH 2.0–6.0) using cyclic and hydrodynamic voltammetric, and hydrodynamic chronocoulometric measurements. The reduction of PCA was characterized to be an irreversible, diffusion-controlled process, and the cyclic voltammetric reduction peak potential (E_p) was found to be more positive by ca. 1.0 V than that of the coexisting H_2O_2 , e.g., the E_p values obtained at 0.1 V s^{-1} for PCA and H_2O_2 were 0.35 and -0.35 V , respectively, vs. $\text{Ag|AgCl|KCl (sat.)}$ at pH 3.3. The E_p of PCA was found to depend on pH, i.e., at $\text{pH} > 4.5$, the plot of E_p vs. pH gave the slope ($-64 \text{ mV decade}^{-1}$) which is close to the theoretical value ($-59 \text{ mV decade}^{-1}$) for an electrode process involving the equal number of electron and proton in the rate-determining step, while at $\text{pH} < 4.5$, the E_p was almost independent of pH. The relevant electrochemical parameters, Tafel slope, number of electrons, formal potential (E^0), cathodic transfer coefficient and standard heterogeneous rate constant (k^0) for the reduction of PCA and the diffusion coefficient of PCA were determined to be ca. $100 \text{ mV decade}^{-1}$, 2, 1.53 V (at pH 2.6), 0.29 , $1.2 \times 10^{-12} \text{ cm s}^{-1}$ and $0.29 \times 10^{-5} \text{ cm}^2 \text{ s}^{-1}$, respectively, and except for E^0 , the obtained values were almost independent of the solution pH. The overall mechanism of the reduction of PCA was discussed [66].

In [67] a potentiometric method to determine peroxide hydrogen and glucose was studied. This method had been applied on the potentiometric determination of peroxide hydrogen and glucose in the total ionic strength adjustment buffer (TISAB) (pH 7.5) solution with the glassy electrode modified by the calix[4]arene. The GC electrode covered

with the calix[4]arene depended on the H_2O_2 concentration in the range of $\log[\text{H}_2\text{O}_2]$ from -3.3 to -1.2 in the solution of TISAB (pH 7.5) with nearly Nernstian slope of about $65.6 \pm 3 \text{ mV}$ and the detection limit of peroxide hydrogen was $4.0 \cdot 10^{-5} \text{ mol l}^{-1}$. The GC electrode covered with the calix[4]arene depended on the glucose concentration in the range of $\log[\text{glucose}]$ from -3.6 to -2.8 in the solution of TISAB (pH 7.5) with nearly Nernstian slope of about $50.2 \pm 2 \text{ mV}$ and the detection limit of glucose was $2.0 \cdot 10^{-5} \text{ mol l}^{-1}$. The electrode had the good selectivity, sensitivity, stability and repeatability.

Electrochemical reduction of H_2O_2 on a sand-blasted stainless steel electrode in an aqueous solution of NaClO_4 was studied. The cyclic voltammetric reduction of H_2O_2 at low concentrations was characterized by a cathodic peak at -0.40 V vs. SCE. Cyclic voltammetry was studied by varying the concentration of H_2O_2 in the range from $0.2 \text{ } \mu\text{mol l}^{-1}$ to 20 mmol l^{-1} and the sweep rate in the range from 2 to 100 mV s^{-1} . Voltammograms at concentrations of H_2O_2 higher than 2 mmol l^{-1} or at high sweep rates consisted of an additional current peak, which may be due to the reduction of adsorbed species formed during the reduction of H_2O_2 . Amperometric determination of H_2O_2 at -0.50 V vs SCE provided the detection limit of $5 \text{ } \mu\text{mol l}^{-1} \text{ H}_2\text{O}_2$. A plot of current density versus concentration had two segments suggesting a change in the mechanism of H_2O_2 reduction at concentrations of $\text{H}_2\text{O}_2 \geq 2 \text{ mmol l}^{-1}$. From the rotating disc electrode study, diffusion coefficient of H_2O_2 and rate constant for reduction of H_2O_2 were evaluated [68].

A series of dopant-type polyaniline-polyacrylic acid composite (PAN-PAA) films with porous structures were prepared and developed for an enzyme-free H_2O_2 sensor. The composite films were highly electroactive in a neutral environment as compared to polyaniline (PAN). In addition, the carboxyl group of the PAA was found to react with H_2O_2 to form peroxy acid groups, and the peroxy acid could further oxidize the imine structure of PAN to form *N*-oxides. The *N*-oxides reverted to their original form via electrochemical reduction and increased the reduction current. Based on this result, PAN-PAA was used to modify a gold electrode (PAN-PAA/Au) as a working electrode for the non-enzymatic detection of H_2O_2 . The characteristics of the proposed sensors could be tuned by the PAA/PAN molar ratio. Blending PAA with PAN enhanced the surface area, electrocatalytic activity, and conductivity of these sensors. Under optimal conditions, the linear concentration range of the H_2O_2 sensor was 0.04 to 12 mmol l^{-1} with a sensitivity of $417.5 \text{ } \mu\text{A/mmol l}^{-1} \text{ cm}^2$. This enzyme-free H_2O_2 sensor also exhibited a rapid response time, excellent stability, and high selectivity [69].

Behavior of H_2O_2 in alkaline medium by dc-polarography with DME aiming to apply it in antioxidant activity determination was studied. Development of a peroxide anodic current having form of a peak, instead of common polarographic wave, was investigated. As a base for this investigation the interaction of H_2O_2 with anodically dissolved mercury was followed. Formation of mercury complex $[\text{Hg}(\text{O}_2\text{H})(\text{OH})]$ was confirmed. The relevant experimental conditions, such as temperature, concentration and pH dependence, as well as time stability of H_2O_2 anodic current, were assessed. Development of an antioxidant assay based on decrease of anodic current of H_2O_2 in the presence of antioxidants was described. Under optimized working conditions, a series of benzoic acids along with corresponding cinnamate analogues for H_2O_2 scavenging activity was tested. In addition, the assay versatility was confirmed on various complex samples was confirmed [70].

In work [71] the electrocatalytic behavior of phosphotungstate-doped glutaraldehyde-cross-linked poly-l-lysine (PLL-GA-PW) film electrode towards reduction of H_2O_2 in acidic medium was described. The modified electrode was prepared by means of electrostatically trapping the phosphotungstate anion into the cationic PLL-GA coating

on GC electrode. The PLL-GA-PW film electrode showed excellent electrocatalytic activity towards H_2O_2 reduction in $0.1 \text{ mol l}^{-1} \text{ H}_2\text{SO}_4$. Under the optimized conditions, the electrochemical sensor exhibited a linear response for H_2O_2 concentration over the range 2.5×10^{-6} to $6.85 \times 10^{-3} \text{ mol l}^{-1}$ with a sensitivity of $1.69 \mu\text{A m mol}^{-1}$. The curvature in the calibration curve at high concentration was explained in terms of Michaelis–Menten (MM) saturation kinetics, and the kinetics parameters calculated by three different methods were compared. The PLL-GA-PW film electrode did not respond to potential such interferences as dopamine, ascorbic acid and uric acid. This unique feature of PLL-GA-PW film electrode allowed selective determination of H_2O_2 . Finally, the proposed electrochemical sensor was successfully applied to determine H_2O_2 in commercially available antiseptic solution and soft-contact lenses cleaning solution and the method using independent estimation by classical potassium permanganate titration method was validated. Major advantages of the method were simple electrode fabrication, stability and high selectivity towards hydrogen peroxide.

A built-in three-in-one screen-printed electrode assembly containing nano bismuth species deposited silver as working, pre-oxidized silver as reference and unmodified silver as counter electrodes (designated as SPAgE-Binano), for simple electrochemical sensing of H_2O_2 in pH 7 phosphate buffer solution was developed. The working electrode showed $\sim 250 \text{ mV}$ reduction in over-potential and a two-fold increased peak current values for electrocatalytic reduction of H_2O_2 over the respective unmodified silver working electrode (i.e. SPAgE) following a diffusion-controlled electron-transfer mechanism. 50 nm sized Bi particles on SPAgE surface was seen in SEM picture of the working electrode. Linear range of H_2O_2 detection from $100 \mu\text{mol l}^{-1}$ to 5 mmol l^{-1} , a sensitivity of $0.627 \mu\text{A m mol}^{-1}$ and detection limit ($S/N = 3$) of $56.59 \mu\text{mol l}^{-1}$ were obtained. Finally, SPAgE-Binano sensor assembly was utilized for cosmetic H_2O_2 determination, where obtained values and labeled values were in good agreement with recovery in the range of $94.75\text{--}101.03 \%$ [72].

In [73] a PAA in the presence of a large excess of H_2O_2 , about 500 fold that of PAA, using Au(111)-like gold electrode in acetate buffer solutions of pH 5.4 was selectively electroanalyzed. Au(111)-like gold electrode was prepared by a controlled reductive desorption of a previously assembled thiol, typically cysteine, monolayer onto the polycrystalline gold (poly-Au) electrode. Cysteine molecules were selectively removed from the Au(111) facets of the poly-Au electrode, keeping the other two facets (i.e., Au(110) and Au(100)) under the protection of the adsorbed cysteine. It has been found that Au(111)-like gold electrode positively shifts the reduction peak of PAA, while, fortunately, shifts the reduction peak of H_2O_2 negatively, achieving a large potential separation (around 750 mV) between the two reduction peaks as compared with that (around 450 mV) obtained at the poly-Au electrode. This large potential separation between the two reduction peaks enabled the analysis of PAA in the presence of a large excess of H_2O_2 . In addition, the positive shift of the reduction peak of PAA gives the present method a high immunity against the interference of the dissolved oxygen.

Such compounds as urea peroxide, $\text{Na}_2\text{CO}_3 \times 1.5\text{H}_2\text{O}_2$, peroxidon (N-polyvinylpyrrolidone complex with H_2O_2 , $M = 35000\text{--}1500000$) are used as the active substances. In the paper [74] a comparative study of the possibility potentiometric titration of aforementioned peroxides using redox platinum indicator electrode by various methods using as oxidation (KMnO_4 , $\text{Ce}(\text{SO}_4)_2$, NH_4VO_3), and reduction (KI, Na_2SO_3 , ascorbic acid) reagent was conducted. It was shown that it was preferred to use cerium sulfate among oxidation reactants. High speed reaction allows to direct potentiometric titration

with clear potential jumps. The indicating agreement between the results obtained by different analytical methods shows the correctness of the founded peroxides content. Peroxides reduction reaction occurred in time and analysis requires the use of back-titration. As the reducing agent, we proposed metol-sulfite mixture of sodium sulfite and metol (*p*-methylaminophenolsulfate) in the presence of oxalic acid in sulfuric acid media. Reagent excess was titrated with iodine solution. The necessity of the control experiment during reverse titration allowed avoiding errors due to insufficient impact resistance and reagent mixture components. The choice of analysis depends on the solubility of the polymer films (acetic acid, ethyl alcohol, acetone, etc.) The artificial mixtures of starting peroxides do not contain peroxides films analysis showed that the presence of dissolved polymer film does not affect the results of the analysis. A number of bleaching agents samples were analyzed. The various methods of determination were proposed.

The behavior of potassium hydrogenperoxomonosulfate (KHSO₅) has been studied using alternating current voltammetry with square wave modulation in potential range +1.0...-1.2 V at carbosital electrode as indicating and auxiliary electrode (reference electrode Ag, AgCl/KCl(sat)) ($E_p = +0.25$ V) [75]. A linear relationship between peak current and concentration was obtained in the range 0.9×10^{-5} mol L⁻¹ to 5.4×10^{-5} mol L⁻¹ of the KHSO₅ concentrations at pH ~0.8 for pure substance. It is $I_p = (8.3 \pm 0.6) \times 10^3 \times c$ ($r = 0.999$). RSD were 2.68 %, 2.55 % and 2.39 % for the 3.6×10^{-5} , 4.5×10^{-5} and 5.4×10^{-5} mol L⁻¹ concentrations of KHSO₅ model solutions, respectively ($\delta = -0.28... +0.44$ %). The addition method was used for KHSO₅ determination in disinfectant «Ecocid S», RSD were 2.01 % and 1.88 % for $c(\text{KHSO}_5) = 4.65 \times 10^{-5}$ and 7.75×10^{-5} mol L⁻¹, respectively. The obtained results have good agreement with those obtained by using the reference method of iodometric titration, with an accuracy lower than -0.42 %.

In the paper [76] the electrochemical behavior of KHSO₅ in the presence of sodium dodecylbenzenesulfonate (SDBS) has been studied using cathodic voltammetry at the carbosital electrode as indicating in the potential range of +1.0...-1.2 V (the reference electrode Ag, AgCl/KCl(sat)) ($E_p = +0.3$ V). It has been experimentally proven that the height of KHSO₅ reduction peak decreases and the potential of the reduction peak is shifted toward more electronegative values with increasing of the background electrolyte pH from 0.80 to 7.17. The maximum peak (I_p) occurs at a pH of approximately 0.8 and at a pH around 5 the analytical signal almost disappears. The effect of pH on the peak potential (E_p) shows the following: when the pH value increases in the interval from 0.8 to 2, E_p remains almost constant, but E_p decreases sharply to the negative value with pH increasing over 2. It has been experimentally proven that SDBS leads to increase of the current peak and the peak potential shifts to the more electropositive side (+0.25 → +0.3V). The influence of the present SDBS has been examined. The current peak increases with the concentration of the surfactant up to 1.2×10^{-3} mol L⁻¹ and then stays almost constant with the increase in the concentration of SDBS above 3.0×10^{-3} mol L⁻¹. The linear relationship was observed in the KHSO₅ concentration range of $(1.8-9.0) \times 10^{-5}$ mol L⁻¹, the calibration curve equation was $I_p = (4.3 \pm 1.1) \times 10^4 c$ ($r = 0.998$). When determining KHSO₅ in the test solution of «Ecocid S» disinfectant with the concentrations of 4.65×10^{-5} , 6.20×10^{-5} and 7.75×10^{-5} mol L⁻¹ the RSDs were 0.025, 0.023 and 0.021, respectively ($\delta = -0.64 ... +0.16\%$); LOD = 6.50×10^{-6} mol L⁻¹, LOQ = 2.17×10^{-5} mol L⁻¹.

Thus, the current research on peroxides detection is mainly focused on electrode modifications including the application of nanomaterials in order to decrease the

overpotential and increase the electron transfer kinetics was showed, and the possibility of peroxides determination by electrochemical methods with applications ranging from the medical, pharmaceutical and paper industry to household washing powder was demonstrated.

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РЕЗЮМЕ

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ВИЗНАЧЕННЯ ПЕРОКСИДІВ ЕЛЕКТРОХІМІЧНИМИ МЕТОДАМИ

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Оглянута електрохімічна поведінка пероксидів, які, зазвичай, використовуються як дезінфікуючі засоби, антисептики і відбілюючі засоби (наприклад, гідроген пероксид, калій пероксомоносульфат (Охоне®), натрій перборат, органічні пероксикислоти, гідропероксиди, діалкілпероксиди та ацилпероксиди) на розмаїтих електродних матеріалах. На певних електродах спостерігаються добре диференційовані окисно-відновні піки, а відтак ці результати були використані для амперометричного та вольтамперометричного визначення цих пероксидів у широкому діапазоні їх концентрацій. Представлені нові прості методики сумісного потенціометричного визначення пероксидів з використанням електродів із платини, золота, нержавіючої сталі, скловуглецю і т.п. Детально розглянуті фактори, що впливають на вибірковість, чутливість і час відгуку. Сучасні дослідження, спрямовані на розроблення нових сенсорів для визначення пероксидів, в основному зосереджені на опрацюванні нових модифікованих електродів, в тому числі з використанням наноматеріалів, яким характерні зниження перенапруги та прискорене перенесення електронів. Продемонстрована можливість здійснення визначення пероксидів електрохімічними методами у різноманітних об'єктах, починаючи з медичної, фармацевтичної та паперової галузі промисловості до побутового прального засобу.

Ключові слова: визначення, пероксиди, електрохімічні методи, дезінфекційні та лікарські препарати.

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