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Authors' review

AUTHORS' REVIEW

Carbon paste electrodes in electroanalytical chemistry[#]

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Abstract: An overview is given dealing with the applications of carbon paste electrodes in equilibrium potentiometry as well as in electrochemical stripping analysis using both voltammetric and potentiometric modes. Various modifications of carbon pastes and carbon paste-based biosensors are also mentioned. The main emphasis in this article is directed at summarizing recent results of the authors' research group during the past few years.

Keywords: carbon paste electrodes; potentiometry; electrochemical stripping analysis; flow injection analysis; electrode modification; heavy metals determination; biosensors.

CONTENT

1. INTRODUCTION

Carbon paste electrodes (CPEs) belong to promising electrochemical or bioelectrochemical sensors of wide applicability. In 2008, it was exactly a half century since Ralph Norman Adams from the University of Kansas published a short one-page report[1] in which he introduced this kind of electrode, which was originally designed as an alternative to the dropping mercury electrode. Although the concept of a dynamic renewable electrode surface was not successful, it turned

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out that the material with paste-like consistency could be practically employed in voltammetric analysis. After the pioneering work of Kuwana,[2,3] who actually first modified a CPE by introducing an electrochemically active surface into the material and, after the first chemical modification of an electrode for electrosynthesis,[4] the modification was soon applied to carbon paste electrodes.[5,6] Finally, Baldwin described a simple method of direct mixing of a solid modifier to the paste,[7] which was the commencement of explosive research activity in this field. Quite a few reviews are exclusively devoted to CPEs.[8–14] Presently, CPEs represent one of the most frequent types of working electrodes. The overwhelming number of CPEs used worldwide belongs to pastes with insulating liquids (paraffin oil, silicon oil, bromonaphthalene, tricresyl phosphate and others). The basic requirements for a pasting liquid are its practical insolubility in the solution under measurement, a low vapor pressure to ensure both mechanical stability and long lifetime, and further, in the case of voltammetric and amperometric applications, its electrochemical inactivity in the potential window of interest. In contrast to the relatively complicated modifications of solid substrates, carbon pastes can be modified simply to obtain quantitatively new sensors with desired, often predefined, properties.[15] Very recently, a review with 333 references was published[16] in the form of a retrospective compilation presenting the field by means of various facts, notes, data, surveys, and summaries that illustrate individual achievements and milestones. A brief overview based mainly on the authors' group contributions is thus given in this paper.

2. CONSTRUCTION OF CARBON PASTE ELECTRODES

Common types of carbon pastes are soft and non-compact, and have to be kept in special bodies. A holder for carbon pastes can be realized as a well drilled into a short Teflon rod,[17] a glass tube[18] or a polyethylene syringe[19] filled with a paste, which is electrically contacted *via* a conducting wire. Such constructions are very simple; however, there is one aspect which makes them not very convenient for practical use and this is the necessity of refilling the carbon paste in experiments requiring a regular removal of the electrode surface layer. More sophisticated constructions circumventing this time-consuming procedure were designed by Monien *et al*.[20] and Lindquist,[21] who proposed piston-driven electrode holders where the desired amount of the used paste could simply be extruded from the electrode body and smoothed away or cut off. Similar types of home-made piston-driven carbon paste holders are used by our research team.[15,22]

Recently, our research group also devoted particular attention to the development of novel or innovated carbon paste electrode holders. Two typical examples of these activities are illustrated in Fig. 1. Image "1A" shows two variants of the so-called carbon paste groove electrode (CPGrE) as atypical construction of CPE, copying the planar design of some screen-printed electrodes.[23] The CPGrE assembly comprises a miniature plastic prismatic bar with a horizontal channel for carbon paste filling, a metal contact and additional plastic insert(s) defining the electrode surface *via* its mechanical coverage/exposure. The whole construction can be devised as an electrically heated electrode (left), applicable for batch measurements and allowing the sensitivity of measurements that have to be made in still solutions to be enhanced or as the working electrode for hydrodynamic amperometry, and as the detector in flowing streams (right). The usefulness of the latter was demonstrated in a very recent study concerning its intimate testing in the FIA mode.[24]

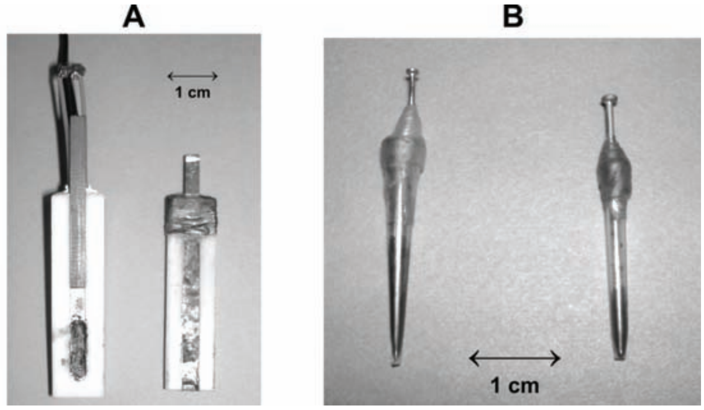

Fig. 1. Some novel construction types of electrode holders for carbon paste. Two assembled carbon paste groove electrodes; the electrically heated variant (A – left) and set-up for detection in the FIA-mode (A – right); two carbon paste mini-electrodes; with an uncut tip (B – left), cut-off variant offering a larger surface diameter (B – right). For further specification, see original reports.[23-25]

A new type of carbon paste mini-electrode (CPmE) which can be made from common plastic pipette tips, the vertical cutting of which provides the desired surface area,[25] is depicted in photograph “1B”. The advantage of this small design is the minimization of the consumption of carbon paste(s), which can be economic when using some mixtures from rather expensive carbon nanotubes, for example.

3. POTENTIOMETRY

From the viewpoint of equilibrium potentiometry, the composition of carbon pastes enables the classification of CPEs as ion-selective liquid membrane type electrodes. The pasting liquid usually exhibits good extraction ability against neutral electroactive species of non-dissociated weak acids, neutral metal chelates or ion associates. Then, the potential of an electrode containing such an organic solvent extract is predominantly governed by ionic exchange at the interface between the organic phase of the electrode and the sample solution, resulting in the so-called Donnan potential.[26] Carbon paste-based ion-selective electrodes (CPISEs) were reported and applied for the determination of several ions (see Refs.[14,26] and references therein). For example, four ion-exchangers were prepared for new perchlorate and fluoroborate CPISEs, which found their application in the direct potentiometric determinations of the two anions, as well as monitoring sensors for potentiometric ion-pair formation-based titrations.[27] Similarly, the ion-pairing principle was used in titrations of complex anions of trivalent thallium[28] or elements forming heteropolyanions.[29,30] An application of the CPGrE type construction (see above) in potentiometric pH measurements[31] is presented in Fig. 2.

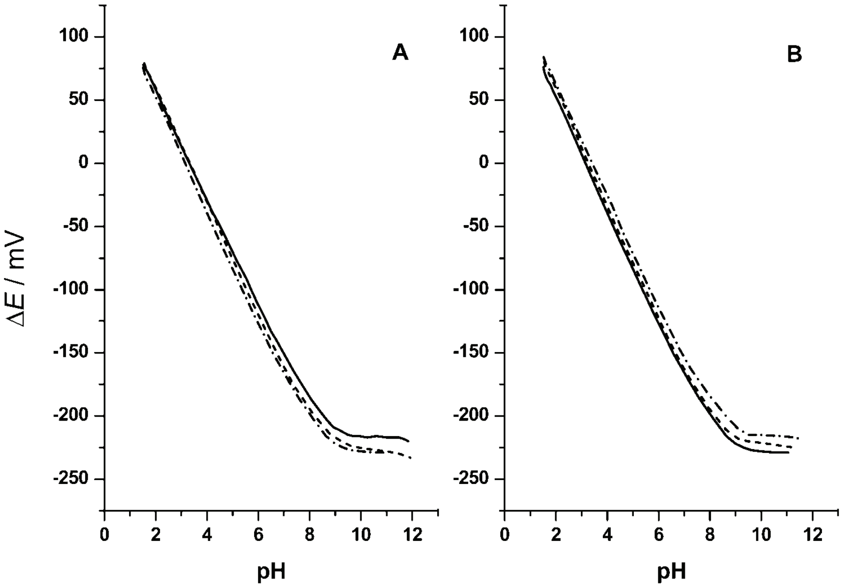

Fig. 2. Potentiometric responses of carbon paste groove electrodes with dispersed bismuth powder (17 % w/w) in Britton–Robinson buffer solution (three repeated measurements). The electrode surface was renewed after each set of measurements (A); all sets measured at the same surface (B).

A distinct advantage of carbon paste-based electrodes is their very low ohmic resistance (less than 10 Ω instead of up to MΩ values for electrodes equipped with polymeric membranes). Thus, experimental work with CPISEs is more convenient and simpler potentiometers allowing voltage measurements of potentiometric cells with lower inner resistance may be applied. This fact accompanied with a very quick response time is especially appreciated in automatic titration procedures.[32] Thus, all procedures elaborated for potentiometric indication of ion-pair formation-based titrations with polymeric membrane electrodes[33] can simply be monitored using CPISEs; the determination of surfactants can serve as an excellent example.[34]

3.1. Electrochemical stripping analysis with unmodified paste electrodes

In voltammetric stripping analysis, the choice of the main constituents for the preparation of the carbon paste was rather conservative and, in fact, there were no attempts to seek new alternative materials. The properties of conventional paste mixtures from spectroscopic graphite and paraffin oils were found satisfactory for the majority of applications. An example can be given dealing with application of such an electrode for trace iron(III) determination in compounds of pharmaceutical significance.[35] In 1993, some new types of CPEs containing more polar organic liquids, often used as plasticizers of polymeric membrane-based ion-selective electrodes, were introduced.[36] Consequently, it was shown that these pasting liquids act as anion exchangers in acidic media due to the presence of some protonizable functional groups. For example, a CPE containing tricresyl phosphate was found suitable for the accumulation of some lipophilic anions, forming ion-pairs with the protonized pasting liquid. At that time, some interesting applications of these pastes in both stripping voltammetry[37,38] and stripping chronopotentiometry[39–42] were introduced.

3.2. Modified carbon pastes in stripping analysis

Carbon pastes undoubtedly represent one of the most convenient materials for the preparation of modified electrodes. A modifier can be dissolved in a binder or admixed mechanically to the paste during its homogenization. For example, a CPE modified with cobalt(II) phthalocyanine was used for voltammetric determination of ascorbic acid in foodstuffs.[43] *In situ* modification of the electrode paste surface is often used as well; examples may be given dealing with applications of an anionic surfactant for voltammetric determination of silver(I) at ultratrace levels.[44] Recently, some procedures utilizing cationic surfactants for *in situ* modifications of CPEs were reported for determinations of chromium(VI),[45] osmium(IV)[46] and other platinum metals,[47] whereby negatively charged complex anions were formed.

4. CARBON PASTE AS A SUPPORT FOR METALLIC FILM ELECTRODES

In order to prepare electrodes plated with metallic films, numerous conductive materials have been proposed for use; glassy carbon is probably the most frequently employed material. It was confirmed that carbon paste as a support for plating with metallic films is able to offer results similar to those of well established solid electrodes and, in addition, their easy and inexpensive preparation and no risk of mechanical damage of the electrode surface are very advantageous.

Mercury film electrodes were very popular and mostly employed in electrochemical stripping determinations of heavy metal ions instead of stationary mercury electrodes. For example, a CPE containing tricresyl phosphate was found to be especially suitable for this purpose; both voltammetric and chronopotentiometric modes were applied.[48,49] It was also confirmed that a mercury film with properties similar to those of a mercury film generated from solutions of mercury(II) salts may be obtained by reduction of mercury(II) oxide dispersed in a paste.[50] Since 1996, investigations on gold film-plated CPEs represents a new subject of our research because, hitherto, no report on such electrodes had been published. They were found useful for the determination of mercury[51] and arsenic.[52]

Bismuth film electrodes (BiFEs) were first prepared by Schwabe[53] and used in potentiometric pH measurements. Into electrochemical stripping analysis, they were introduced by Wang *et al*. in 2000.[54] This whole topic has already achieved a relevant position within research activities devoted to the development and application of mercury-free electrodes. Their most significant advantage is that they are environmentally friendly, since the toxicity of bismuth and its salts is significantly lower than that of salts of other heavy metals. Furthermore, the advantageous analytical properties of BiFEs in electrochemical stripping analysis, roughly comparable to those of mercury film electrodes, are attributed to the property of bismuth to form alloys with various metals (which is analogous to the amalgams formed by mercury). Many papers appearing since introduction of BiFEs are referred to in recent reviews.[55–59] Unfortunately, numerous authors simply followed the original procedure;[54] this means that BiFEs were prepared using either different substrates or different varieties in their constructions but they were only applied in the determinations of the same metal ions - lead(II) and cadmium(II) – in the same medium – a pH 4.5 acetate buffer. However, it has been shown that bismuth film-plated CPEs may also be applied in alkaline media, such as ammonia buffers[60] or even 0.10 M KOH solution.[61]

The role of the plating regime in the deposition of bismuth films onto a carbon paste surface was also investigated. It should be noted that the term “film” does not correspond with the real situation as it is not compact: analogously to mercury “films” deposited in form of isolated droplets, bismuth is deposited in form of isolated crystals. The new observations performed by scanning electron microscopy were focused mainly on morphological transformations of the microstructure of the bismuth film in dependence on the deposition process and its intensity during potentiostatic electrolysis.[62] To overcome such electrolytic depositions, bismuth powder-dispersed CPEs were introduced.[63] The electrode paste was prepared as a mixture of finely powdered metallic bismuth together with graphite powder and silicon oil and was characterized in solutions containing Cd(II) and Pb(II) at the microgram/liter level in conjunction with square-wave anodic stripping voltammetry. The electrode exhibited well-defined and separated stripping signals for both metals accompanied with a low background contribution. Moreover, it exhibited superior performance in comparison to the bare CPE and, surprisingly, yielded a response higher than that of an *in situ* prepared bismuth film CPE.

The introduction of bismuth(III) salts and bismuth films into electroanalysis also instigated the novel approach of potentiometric stripping analysis based on total substitution of mercury by bismuth. First, Bi(III) salts could be applied to form bismuth films. Secondly, bismuth(III) salts could substitute mercury(II) in its role as an oxidant. These facts offer quite new possibilities of an attractive use of "mercury-free", environmentally friendly PSA procedures for the determinations of some heavy metals. The highest signals were observed when working in solutions containing anions forming complexes with bismuth(III), such as chlorides or bromides.[64]

Similarly to bismuth, antimony bulk electrodes are also well known from equilibrium potentiometry. Concerning voltammetry, attempts with modifications of screen-printed carbon electrodes using either Bi_2O_3 or Sb_2O_3 were also realized,[65] but the first antimony film electrode prepared *in situ* on a glassy carbon electrode substrate and employed in combination with either stripping voltammetry or stripping potentiometry was reported very recently.[66] Simultaneously, initial studies with antimony-modified CPEs, focusing on their performance in anodic stripping voltammetry, were performed; the investigations comprised the first experiments with Sb film-plated and Sb powder-dispersed CPEs.[67,68] Again, antimony films generated *in situ* on CPEs with excess antimony(III) salts as chemical oxidants were introduced as new procedures for potentiometric stripping analysis following previous electrolytic preconcentration of the to be determined metals.[69] Moreover in acidic solutions containing halide ions, the oxidation ability of Sb(III) is adequately limited because of the formation of its corresponding halide complexes. Compared with similar total substitution of traditionally used mercury(II) by bismuth(III), the use of antimony(III) offers a higher sensitivity in the detection of heavy metals.

Typical stripping voltammetric responses of metallic film, metal powder, and metal oxide-modified CPEs obtained by analyzing an equimolar mixture of Cd(II) and Pb(II) ions in model water samples are depicted in Fig. 3. The figure shows a comparison of the individual electrode configurations in two sets of voltammograms (A and B), taken from previous studies,[59,67,70] in which the respective experimental conditions and, especially, the supporting electrolyte were optimized for the antimony-modified variants.

5. AMPEROMETRIC (BIO)SENSORS

In collaboration with Austrian colleagues, the first modifications of carbon pastes with manganese dioxide films in order to obtain sensors for the determination of hydrogen peroxide commenced at the end of the nineties.[71–73] However, for application in procedures utilizing flow injection analysis, it seemed more effective to transfer all experience obtained with such CPE-based sensors to their analogous screen-printed configurations.[74] Such sensors enabled the detection of hydrogen peroxide by either mediated oxidation or mediated reduction (Fig. 4).

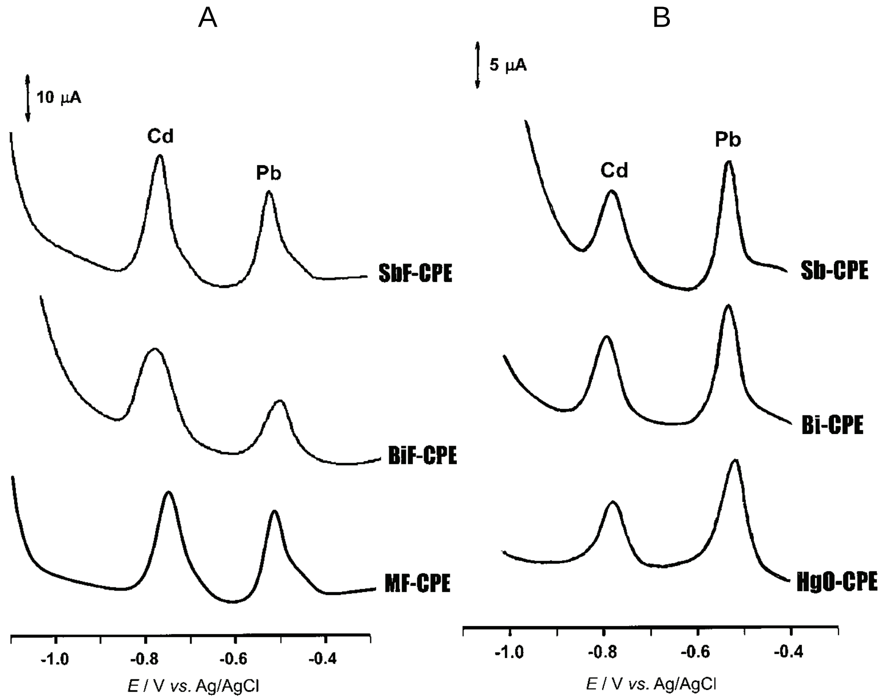

Fig. 3. Anodic stripping voltammograms of Cd(II) + Pb(II) at different types of metal film-plated and metal or metal oxide powder-dispersed carbon paste electrodes. Mercury, bismuth or antimony film-plated carbon paste electrode (MF-CPE, BiF-CPE, or SbF-CPE) (A); HgO (5 %), Bi (20 %) or Sb (20 % w/w) powder-dispersed CPE (B); supporting electrolyte, 0.01 M HCl; *c*(Cd,Pb) = 50 ppb; *c*(Hg,Bi,Sb) = = 1×10^{-5} mol L^{-1} (for A); accumulation potential, –1.2 V *vs*. Ag/AgCl/3M KCl; accumulation time, 120 s; equilibrium time, 15 s; potential range from –1.1 to –0.30 V.

Furthermore, in combination with a proper biocatalyst (glucose oxidase was used in the initial experiments), they served as basic units in the construction of the corresponding biosensors;[75,76] a similar biosensor based on sarcosine oxidase was also reported.[77] Modification with other metal oxides were also studied.[78–80] Recently, biosensors possessing very good properties were obtained when electrocatalysts based on the oxides of platinum group metals were used as modifiers/mediators.[81–85] Biosensors based on dehydrogenases were also studied.[86]

Finally, CPEs were applied in studies involving DNA sensing,[87–89] as well as for the electrochemical detection of bacterial cells.[90,91]

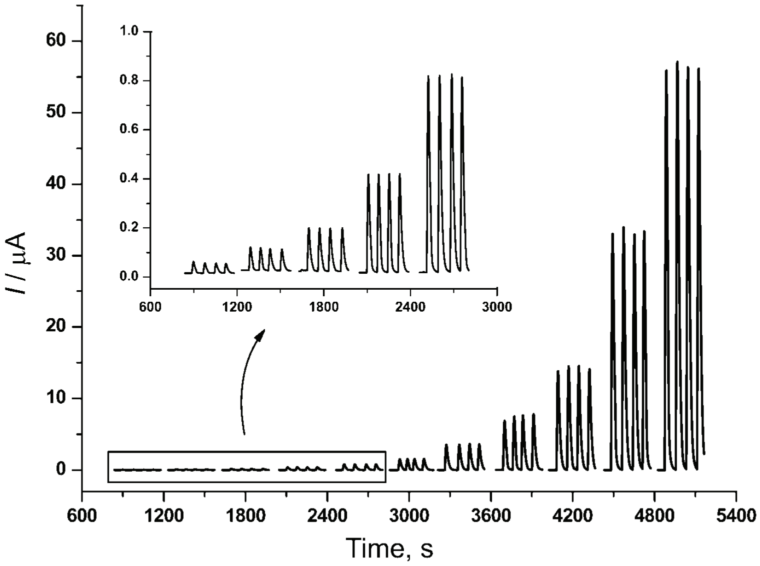

Fig. 4. Flow injection analysis of hydrogen peroxide with amperometric detection. Sensing element, CPGrE with dispersed RuO_2 powder (5 % w/w); $c(H_2O_2)$: 0.5–1000 mg L^{-1}; measured in a stream of 0.10 M phosphate buffer (pH 7.0); flow rate, 0.50 mL min^{-1}; detection potential, 0.5 V *vs*. Ag/AgCl/3M KCl.

In some case, however, carbon ink used for the fabrication of screen-printed layers, although similar to carbon pastes, can cause some different properties of sensors when compared with those based on original carbon pastes. To avoid this sometimes unexpected behavior, groove electrodes (GrEs) were recently introduced as a new alternative to using carbon pastes in electroanalysis (see above).[31] Compared to conventional CPEs, the construction specifics of GrEs offer simpler preparation and quicker exchange of the carbon paste mixture, whereas the small size and planar configuration compete with screen-printed electrodes. First experiments dealing with their application in flow injection analysis were recently presented.[23,24,31]

6. CONCLUSION

Over the past decades, as indicated in a very recent review,[16] carbon paste has become one of the most popular electrode materials used for the laboratory preparation of various electrodes, sensors, and detectors. Such a position is undoubtedly the result of the optimum constellation of physicochemical and electrochemical properties of this carbonaceous substrate. The authors also believe that a contribution coming from our laboratories, outlined in this paper, may offer an interesting insight into the field.

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ИЗВОД

ЕЛЕКТРОДЕ ОД УГЉЕНИЧНЕ ПАСТЕ У ЕЛЕКТРОАНАЛИТИЧКОЈ ХЕМИЈИ

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Дат је преглед примена електрода од угљеничне пасте у равнотежној потенциометрији као и у електрохемијској линеарној промени потенцијала и волтаметрији. Поменуте су такође и различите модификације угљеничних паста и биосензора заснованих на угљеничним пастама. Основни циљ овог рада је да сумира новије резултате истраживачке групе аутора током неколико протеклих година.

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