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**THE TESTING OF CARBON PASTE ELECTRODES:  
AN EXAMPLE ON THE CHARACTERISATION  
OF A CARBON PASTE ELECTRODE PREPARED  
FROM NEWLY USED GRAPHITE POWDER**

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*In this article, the principles of physico-chemical and electrochemical characterisation of carbon paste electrodes are demonstrated on a practical example. The carbon paste electrode prepared from a commercial spectroscopic graphite powder whose properties are to be characterised has been tested in parallel with another carbon paste electrode of known composition by means of special testing procedures. These experiments comprised basic physico-chemical characterisation, polarisation studies, and voltammetric measurements with various model systems. Typical properties of the carbon paste electrode tested are discussed with*

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*respect to its applicability in electrochemistry.*

## **Introduction**

Carbon paste electrodes (CPEs) represented by carbon paste packed into a specially designed electrode body can be classified as a unique type of solid carbon electrodes [1]. Carbon paste, i.e. a mixture of graphite (carbon) powder and a suitable liquid binder, is typical heterogeneous electrode material. Carbon paste-based electrodes offering numerous advantageous properties and characteristics have found use in various fields of electrochemistry, especially in voltammetric measurements [1–9].

Commercially marketed electrodes are commonly manufactured by unified technologies and their electrochemical characteristics are comparable for a given type of electrode. In contrast to this, CPEs are usually hand-made in laboratories from various carbon paste components and using often different preparation procedures. Thus, each carbon paste represents an individual with unique physico-chemical and electrochemical properties. This implies that the success in experimental work with CPEs substantially depends upon experimental experience of user who should be able to reveal and interpret individual nuances in behaviour of various types of CPEs. It is also apparent that electrochemical properties of carbon paste-based electrodes can be predicted only approximately and their more detailed characterisation requires suitably chosen testing measurements [5,7].

The testing of CPEs can be defined as an experimental, almost empirical approach based on measurements and observations which permit to characterise all fundamental physico-chemical and electrochemical properties of carbon pastes. The proper strategy of how to choose individual testing procedures and measurements and how to arrange their overall structure naturally depends on the purpose and aims for which such a characterisation is to be performed. During four decades since CPEs were introduced in electrochemistry, a number of various procedures for the testing carbon paste-based electrodes have been proposed ([5] and Refs therein). More recently, this topic has for the first time been reviewed in a report, providing an up-to-date and comprehensive source of important information, numerous practical notes and examples mostly based on authors' own experience [10].

The article presented here brings a practical example of the testing of a CPE made from graphite powder whose properties are to be characterised. The characterisation has been performed with the aid of recommended and standardised testing procedures [10,11]. Hence, the results discussed in this paper can conveniently supplement the experimental material summarised in the above-mentioned review [10].

## Experimental

### *Apparatus*

Voltammetric experiments were carried out using a PAR 174 polarographic analyzer (EG & G Princeton Applied Research, U.S.A.) which was connected to a personal computer via an interface card for A/D conversion of the data (PC ADDA-14, Model FPC-011; Flytech Technology Co., U.S.A.). This set-up was combined with an SMDE electrode stand (Laboratorní přístroje, Prague, Czech Republic) adapted for measurements with CPEs as the working electrode. A Pt auxiliary electrode and an Ag/AgCl reference electrode (both self-made) completed the three-electrode cell. Stirring was performed with a Teflon-coated magnetic bar (20 mm, o.d. 4 mm) agitated by an electromotor.

The pH values were measured using a digital pH meter (Radelkis, Budapest, Hungary) equipped with a combined glass pH sensor (glass vs Ag/AgCl electrode, Model OP-0808P; Radelkis). The electric resistance of CPEs was measured with conventional resistance meter (Tesla, Kolín, Czech Republic).

### *Chemicals and Reagents*

All the chemicals used were of analytical reagent grade and purchased from Lachema (Brno, Czech Republic). Stock solutions and buffers for the preparation of the supporting electrolytes were made 1 M in concentration unless stated otherwise. Standard solutions of individual test substances were mostly 0.01 M; more diluted standards were prepared freshly before use. Redistilled water was used throughout. All solutions to be analysed were deaerated with argon (purity: 99.996 %, Linde Technoplyn, Czech Republic).

### *Working Electrodes*

According to a previously recommended procedure [5,8], both tested and „standard“ carbon pastes were made by thoroughly mixing 0.5 g graphite powder with 0.2 ml “LUKOOIL MV-15 500” silicone oil (Lučební závody, Kolín, Czech Republic) using a pestle and mortar.

A carbon paste to be tested—denoted as “*Ald / SO*”—was made from commercial spectroscopic graphite marketed by Aldrich Co., (cat. No. 28 286-3; particle size: 1 – 2  $\mu\text{m}$ ). Standard carbon paste (“*RW-B / SO*”) used for parallel measurements as the reference CPE contained RW-B graphite powder (Ringsdorff Werke, Germany) with particles diameter of about 5  $\mu\text{m}$ . Both carbon pastes were packed into piston-like electrode holders [4,5].

## Procedures

**Voltammetry:** Anodic and cathodic curves were recorded using either direct current voltammetry (DCV) or differential pulse voltammetry (DPV). The scan rate was  $20 \text{ mV s}^{-1}$ , the pulse height  $50 \text{ mV}$ , and the sampling rate  $5 \text{ data s}^{-1}$ .

**Stripping Voltammetry:** The accumulation was performed in stirred solution at appropriate accumulation potential,  $E_{acc}$ , for a given period ( $t_{acc}$ ). After the rest period (in quiet solution),  $t_r$ , the voltammetric scan was recorded from the initial potential ( $E_{init}$ , always identical with  $E_{acc}$ ) up to the final potential,  $E_{fin}$ , in DPV mode. Some voltammetric parameters will be specified later, in connection with individual types of measurements.

## Results and Discussion

### *Choice of Carbon Paste Electrode for Testing and for Comparative Measurements*

Spectroscopic graphite powders offered in annual catalogues of renowned chemical companies are, in most cases, usable for the preparation of carbon pastes [4,5]. By choosing these graphites, one can obtain CPEs with satisfactory behaviour without having to purchase any specially produced and rather expensive carbon materials often recommended for carbon pastes. One of typical examples of commercially marketed spectroscopic graphites is a product from *Aldrich* which has been used for the preparation of a CPE tested in this work.

Carbon pastes made from RW-B graphite powder represent mixtures whose physico-chemical and electrochemical characteristics have been studied in detail [12–16]. This is the reason why the RW-B / SO was selected as a standard CPE for parallel measurements.

Based on good experience from previous experimental work (e.g. [15–18]), highly viscous silicone oil was selected as liquid binder for both Ald / SO and RW-B / SO.

### *Basic Physico-Chemical Characterisation of Ald / SO and RW-B / SO Carbon Pastes*

**Consistency.** Although mixed at identical ratio, i.e. with the same amount of the binder, the Ald / SO carbon paste exhibited a markedly lower compactness in comparison with that of the RW-B / SO. Thanks to this, the electrode holder filled with rather soft Ald / SO carbon paste could be better manipulated, and also the surface renewal of this paste (by wiping it with wet filter paper) was easier and gave a very smooth surface layer.

*Ohmic Resistance.* The Ald / SO carbon paste exhibited an ohmic resistance of 3.8  $\Omega$  which was slightly lower than 4.9  $\Omega$  found for the RW-B / SO. These values confirmed a very good conductivity of both carbon pastes and were within limits typical for this type of electrode material [5,7]. Moreover, measurements on resistance of ready-prepared CPEs (i.e., carbon pastes packed in the electrode body) allowed to check whether both electrodes had been prepared properly.

*Ageing („Life-time”).* Like the RW-B / SO, also the Ald / SO carbon paste showed a very stable regime in time and did not undergo any visible changes in consistency or in behaviour over the whole testing period (approx. three weeks). This was in accordance with previous observations, viz. that the ageing of carbon pastes (e.g. their dessication) can be noticed mainly in the cases when one compares the behaviour of CPEs made of qualitatively different liquid binders [5,19,20].

#### *Polarisation Studies with Ald / SO and RW-B / SO Carbon Paste Electrodes*

Investigations into polarisability in selected supporting electrolytes belong among the most important measurements involved in procedures for the testing of CPEs employing the current measurement-based electroanalytical techniques [10].

*Anodic Polarisability.* Oxidative scanning in the anodic direction serves as simple control measurement verifying the often quoted fact that carbon paste-based electrodes exhibit very low background currents over the whole positive potential range [1–5].

Voltammograms in Fig. 1 were obtained by anodic polarisation of both Ald / SO and RW-B / SO electrodes in two model supporting electrolytes. In strongly acidic medium such as 0.1 M HCl, the cathodic potential limits of about  $-0.6$  V vs AgCl indicate typically low hydrogen overpotential and are nearly the same for both CPEs.

When studying the overall character of all the anodic voltammograms given in Fig. 1, one can notice that the anodic polarisation of the Ald / SO has resulted in some undesirable signals deteriorating the shape of the base-line between  $-0.5$  V and  $+0.2$  V vs Ag/AgCl. Within this potential range, the voltammograms obtained with the RW-B / SO electrode do not suffer from such a deformation. This is more evident in Fig. 2 showing the critical parts of anodic voltammograms of both CPEs registered within limited potential range and at a higher sensitivity of the apparatus. At the Ald / SO electrode, the voltammogram (curve 1) exhibits sharp peaks superimposed upon the base-line that have practically disappeared after successive scanning (curves 2 – 4). However, as shown in these voltammograms, individual replicates give rise to a new unwanted response in an interval of  $0.0 - 0.5$  V. In order to confirm all observations, polarisation measurements with the Ald / SO electrode were carried out repeatedly and always used freshly renewed carbon paste

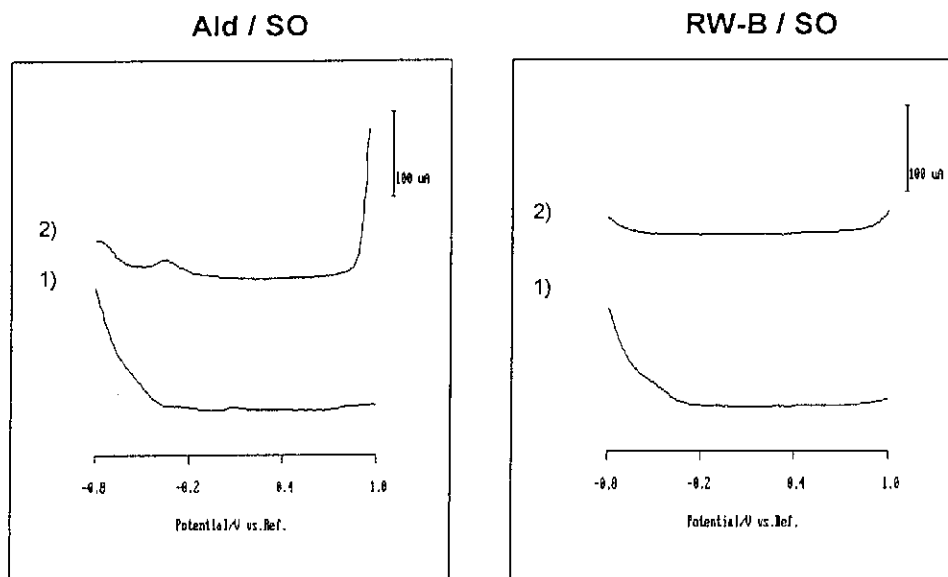


Fig. 1 Anodic polarisation of Ald / SO and RW-B carbon paste electrodes in acidic medium. 1) supporting electrolyte: 0.1 M HCl (pH 1), 2) supporting electrolyte: 0.1 M  $\text{NH}_3 + \text{NH}_4\text{Cl}$  (pH 9); DPV mode; initial potential -1.0 V vs Ag/AgCl; final potential, +1.5 V; scan rate, 20  $\text{mV s}^{-1}$ ; pulse height, 50 mV

surface.

In fact, the origin and the nature of parasitic signals at the Ald / SO carbon paste electrode during its anodic scanning are still rather unclear. (Similar behaviour of carbon paste-based electrodes with respect to their polarisation has not yet been reported.) It seems that the above-mentioned phenomena can be explained by some specific interactions at the carbon paste surface, probably by sorption effects. This could correspond to the finding that the response of the Ald / SO electrode has differed significantly from scan to scan (again see Fig. 2). Or according to another possible interpretation, signals of unknown character at the Ald / SO carbon paste electrode could be attributed to the oxidation of electroactive impurities contained in the paste [5].

*Cathodic Polarizability.* Test measurements with CPEs scanned towards negative potentials are an important tool indicating the content of oxygen entrapped inside the paste (mostly via sorption onto graphite particles). It has been shown that a plateau-like reduction signal directly proportional to the content of oxygen in the paste usually limits the applicability of a CPE to cathodic reductions [1–5].

Figure 3 shows typical voltammograms obtained by cathodic polarisation of both CPEs in two different supporting electrolytes. Compared to the RW-B / SO,

the base-line of Ald / SO is substantially deformed by a large reduction wave of oxygen. Because the only difference between both CPEs was the carbon material used, evidently higher amount of oxygen in the Ald / SO electrode was brought into the carbon paste mixture via the *Aldrich* graphite particles themselves.

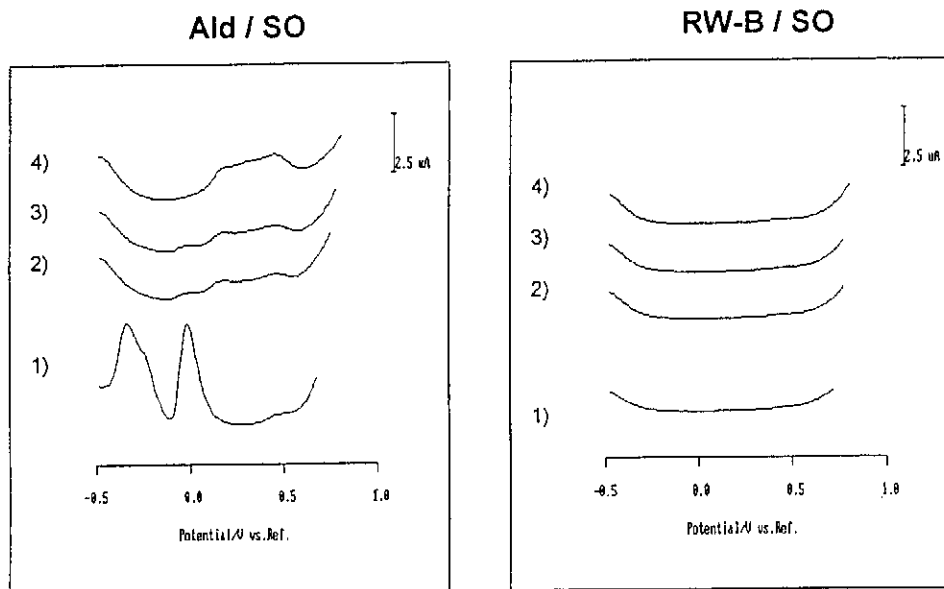


Fig. 2 Repetitive anodic polarisation of Ald / SO and RW-B carbon paste electrodes in acidic medium (within selected potential range). 1 – 4) four repetitive anodic scans. DPV mode; supporting electrolyte, 0.1 M HCl; initial potential, -0.5 V; final potential, +1.0 V; scan rate, 20  $\text{mV s}^{-1}$ ; pulse height, 50 mV.

#### *Testing of Ald / SO and RW-B / SO Carbon Paste Electrodes on Selected Model Systems and Using Some Special Studies*

*Anodic Stripping Voltammetry of Tl(I) and Pb(II) at Carbon Paste Electrodes Plated With a Mercury Film.* CPEs with deposited mercury films have been shown to be a perspective type of working electrodes applicable in electrochemical stripping analysis [20,21]. In this case, it is appreciated that the surface of carbon pastes can simply be renewed and provides sufficiently wide operational potential range for optimal formation of the film.

Figure 4 depicts voltammetric curves obtained by analysing an equimolar mixture of Tl(I) and Pb(II) at mercury film-plated Ald / SO and RW-B / SO carbon paste electrodes. In both voltammograms, the peaks of interest are well-developed

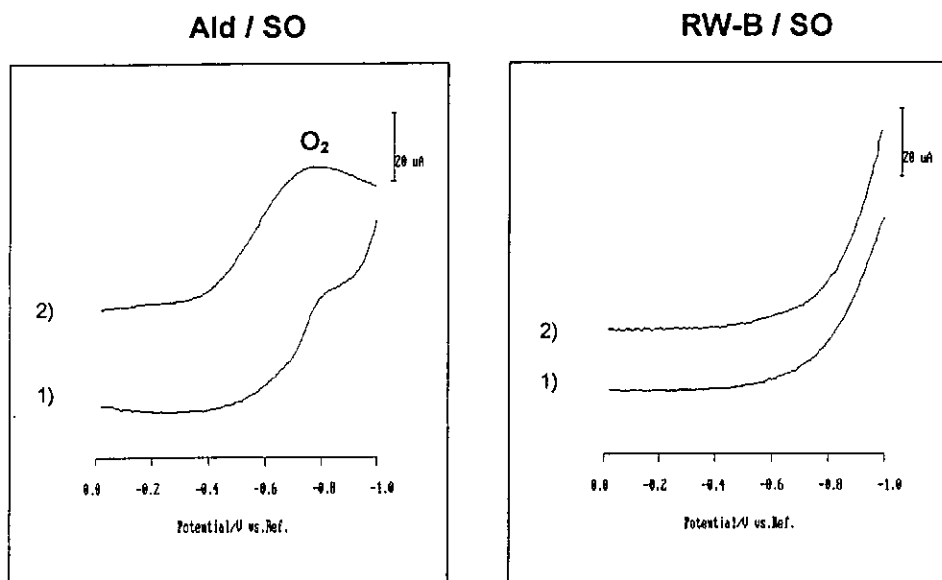


Fig. 3 Cathodic polarisation of Ald / SO and RW-B carbon paste electrodes 1) 0.1 M HCl; 2) 0.1 M  $\text{NH}_3 + \text{NH}_4\text{Cl}$ ; DPV mode; initial potential, 0.0 V; final potential, -1.0 V; scan rate, 20  $\text{mV s}^{-1}$ ; pulse height, 50 mV

and sufficiently separated. However, in this context it should be emphasised that the test was made with relatively high concentrations of both Tl(I) and Pb(II). This did not permit to reveal possible problems with the base-line of the Ald / SO which could be expected in measurements at lower concentrations of both ions. Due to this reason, the following assays with Bi(III) and Ag(I) were also included among testing procedures.

*Anodic Stripping Voltammetry of Bi(III) at Trace Concentration Level with a Mercury Film-Plated Ald / SO Carbon Paste Electrode.* Bismuth as metal with a considerable affinity to form amalgams can be effectively preconcentrated at mercury film electrodes, allowing the determination of Bi down to nanomolar level [19].

Voltammograms in Fig. 5 confirm that the Ald / SO electrode—when employed in measurements at trace concentrations—exhibits a considerably deformed base-line affecting the signal of interest.

*Anodic Stripping Voltammetry of Ag(I).* Also, anodic stripping determination of silver is characterised by extremely low detection limits (below  $1 \times 10^{-12} \text{ mol l}^{-1}$  [13]), mainly thanks to favourable deposition potential of Ag near 0.0 V vs SCE. In certain cases, the electrolytic preconcentration is performed directly onto the bare carbon paste surface.



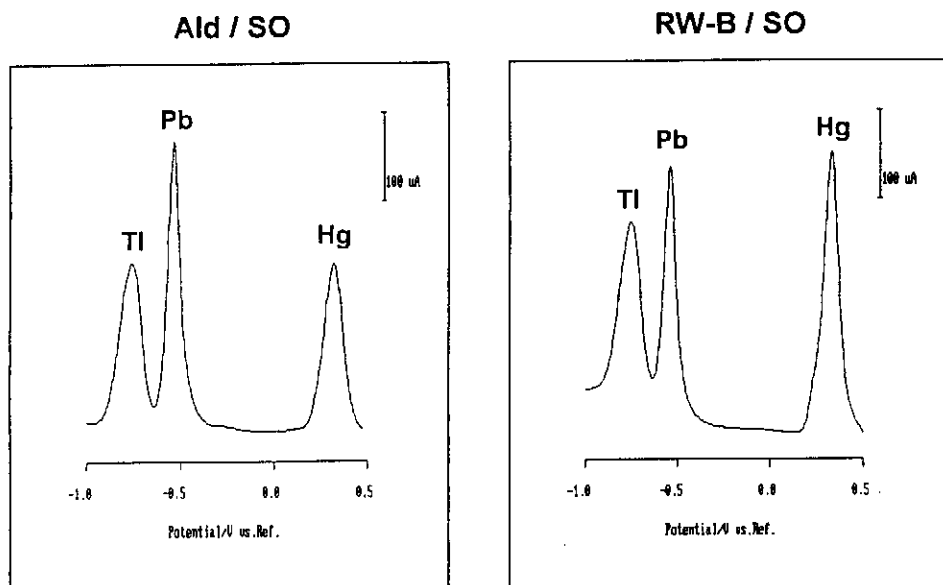


Fig. 4 Anodic stripping voltammetry of Tl(I) and Pb(II) at mercury film-plated Ald / SO and RW-B carbon paste electrodes. DPASV mode; supporting electrolyte, 0.1 M acetic buffer +  $5 \times 10^{-5}$  M  $\text{Hg}^{2+}$ ;  $c(\text{Tl}^+, \text{Pb}^{2+}) = 1 \times 10^{-6}$  mol l $^{-1}$ ; accumulation potential, -0.9 V; final potential, +0.5 V; scan rate, 20 mV s $^{-1}$ ; pulse height, 50 mV

Figure 6 shows that the determination of Ag(I) at low concentration with the Ald / SO electrode has also been negatively affected by its high background currents and the response for Ag has to be evaluated as a peak superimposed upon the baseline. However, such an approach is obviously problematic and may result in considerable errors in quantitative analysis.

*Adsorptive Stripping Voltammetry of Ni(II).* Bivalent nickel can be preconcentrated via adsorption as dimethylglyoximate complex whose reduction is then recorded voltammetrically. At CPEs, stripping voltammetry of Ni(II) appears to be useful for testing the adsorption properties of the carbon paste surface as well as for evaluating the overall character of the base-line with respect to its deformation by reduction wave of oxygen [22].

Figure 7 makes a comparison of voltammograms obtained by analysing ca  $1 \times 10^{-7}$  M Ni(II) at both Ald / SO and RW-B / SO carbon paste electrodes. It can be seen that undesirable signal of oxygen at the Ald / SO evidently deteriorates the whole base-line. In addition, a lower hydrogen overpotential of this CPE has caused marked deformations of the Ni-peak itself. This, unfortunately, did not allow to decide whether the surface of Ald / SO carbon paste could exhibit some specific adsorption capabilities resulting, e.g., in more effective preconcentration of the analyte.

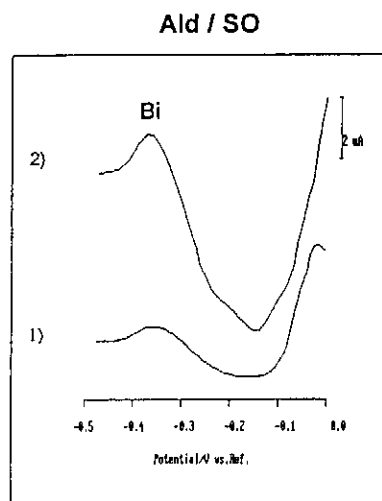


Fig. 5 Anodic stripping voltammetry of Bi(III) at mercury film-plated Ald / SO carbon paste electrode: 1) base-line, 2)  $c(\text{Bi}^{3+}) = 5 \times 10^{-8} \text{ mol l}^{-1}$ ; DPASV mode; supporting electrolyte, 0.1 M HCl +  $1 \times 10^{-5}$  M  $\text{Hg}^{2+}$ ; accumulation potential, -0.5 V; final potential, +0.5 V; scan rate, 20  $\text{mV s}^{-1}$ ; pulse height, 50 mV

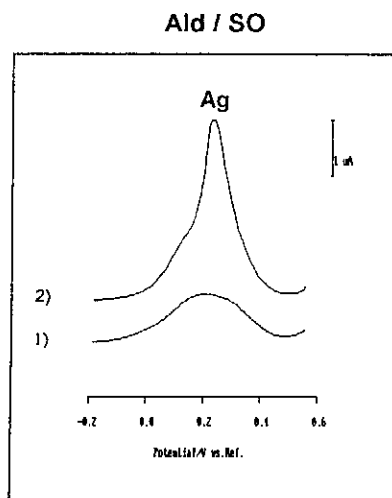


Fig. 6 Anodic stripping voltammetry of Ag(I) at Ald / SO carbon paste electrode: 1) base-line, 2)  $c(\text{Ag}^+) = 1 \times 10^{-7} \text{ mol l}^{-1}$ ; DPASV mode; supporting electrolyte, 0.05 M acetic buffer; accumulation potential, -0.2 V; final potential, +0.6 V; scan rate, 20  $\text{mV s}^{-1}$ ; pulse height, 50 mV

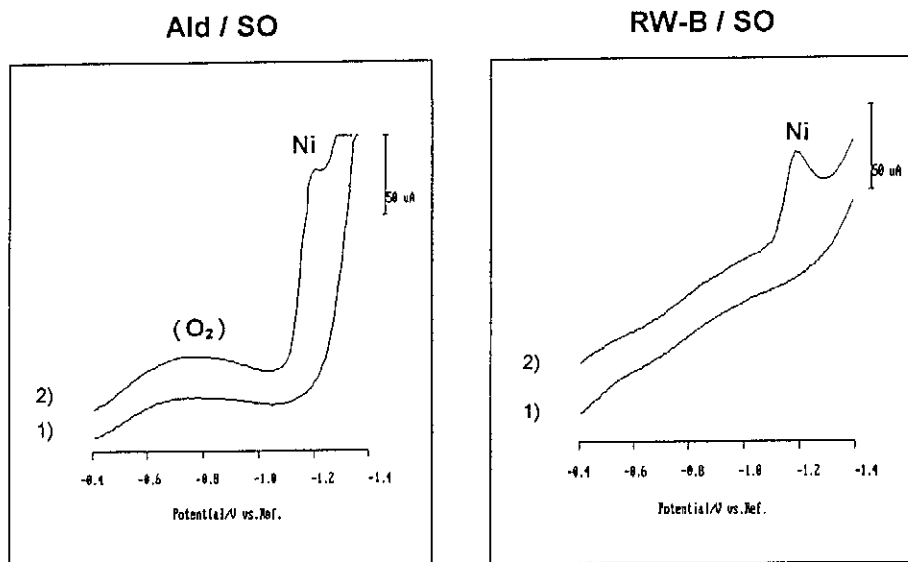


Fig. 7 Adsorptive stripping voltammetry of Ni(II) at Ald / SO and RW-B carbon paste electrodes: 1) base-line, 2)  $c(\text{Ni}^{2+}) = 1 \times 10^{-7} \text{ mol l}^{-1}$  DPCSV mode; supporting electrolyte, 0.5 M  $\text{NH}_4\text{Cl}$  + 0.05 M triethanolamine +  $1 \times 10^{-4}$  M dimethylglyoxime; scan rate,  $20 \text{ mV s}^{-1}$ ; accumulation ( $\equiv$  initial) potential,  $-0.3 \text{ V}$ ; final potential,  $-1.5 \text{ V}$

*Effect of Electrolytic Activation on Surface Characteristics of Carbon Pastes.* Activation procedures performed by potentiostatic electrolysis in quiet solution at extreme potentials usually lead to principal structural changes at the carbon paste surface, which can then be observed and interpreted with the aid of some model systems via their reaction kinetics [10].

The effect of electrolytic activation on the surface layer structure of both carbon pastes could be seen with the naked eye: the carbon paste surface becoming darker and loosing its smoothness. This observation corresponds to previous experience [10,11] reporting that activation processes may often result in total removal of the outer layer of liquid binder. Thereby, carbon particles at the electrode surface are practically naked with characteristic „velvet“ consistency. In this study, it was found that the activation done under conditions recommended in literature ( $+1.75 \text{ V}$  for 5 min, and  $-1.5 \text{ V}$  for 15 s [10]) had led to a destruction and disintegration of the surface layer of both Ald / SO and RW-B / SO. Therefore, the whole activation procedure had to be appropriately adapted; the optimum conditions found are given in the caption of Fig. 8. This figure illustrates that the activation, as expected, has resulted in a substantial lowering of the overpotential required for the anodic oxidation of model compound (ascorbic acid), which can be recognised as a distinct shift of peaks towards less positive potentials. It is also interesting to

notice a substantial peak increase at the Ald / SO electrode after activation. (In accordance with the above-described polarisation studies, such an increase can perhaps be attributed to a catalytic effect of oxygen fixed in the Ald / SO carbon paste.) Compared to measurements with the RW-B / SO, it is evident that even the activation studies have revealed significant differences in behaviour of both CPEs to be tested.

*Anodic Voltammetry of Phenothiazine in Highly Methanolic Solutions*. A study with phenothiazine derivatives can be recommended as a test on the electrode response for relatively complicated electrode reaction of organic substance with several electroactive sites [17]. Regarding CPEs, this test is especially convenient, allowing one to observe the stability of carbon pastes in mixed media since the experiment requires the use of organic solvent in the supporting electrolyte [17].

In Figure 9, the base-lines of both Ald / SO and RW-B / SO electrodes and their responses for phenothiazine are compared. For measurements in blank supporting electrolyte, the corresponding voltammograms (curves 1) document that both carbon pastes are sufficiently stable even in 50% methanolic solution. Negligible differences in the base-line responses indicate a minimal effect of gra-

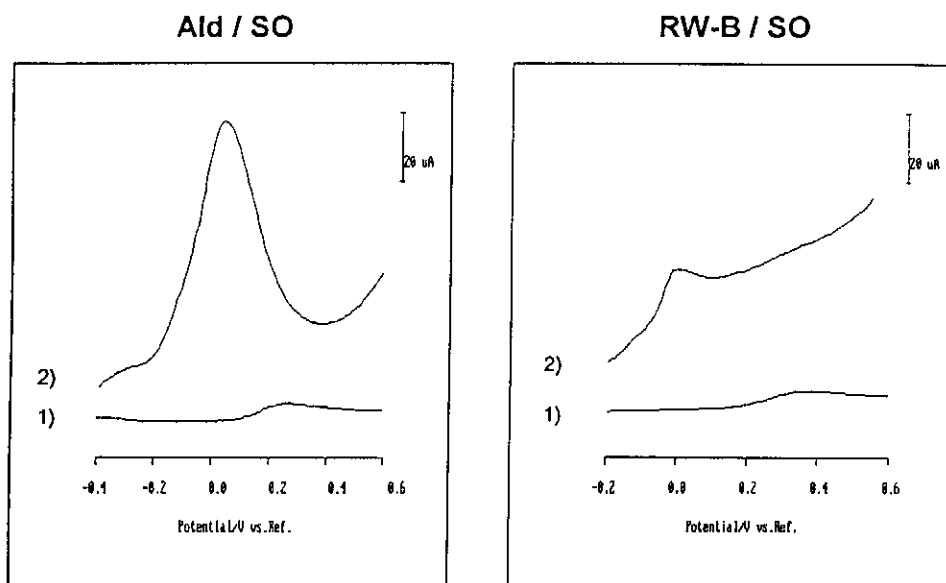


Fig. 8 Effect of electrolytic activation on the surface characteristics of Ald / SO and RW-B carbon paste electrodes. (Studies on anodic oxidation of ascorbic acid): 1) unactivated, 2) activated carbon paste surface; DC mode; supporting electrolyte, 1 M acetic buffer (pH 4.7); initial potential,  $-0.5$  V; final potential,  $+1.0$  V; scan rate,  $20$   $\text{mV s}^{-1}$ . Activation parameters:  $+1.5$  V (for 60 s),  $-1.0$  V (15 s)

phite in the paste upon the behaviour of both CPEs in organic solvent-containing electrolyte. This confirms previous experience [11,17] that the stability of a CPE in mixed media depends mainly upon the type of liquid binder used. When comparing the responses for anodic oxidation of phenothiazine (2), both voltammograms include two well-shaped peaks, but the ratio of their heights is evidently different. This phenomenon is probably associated with the process proper of the electrode

oxidation of phenothiazine at both CPEs. This heterocyclic compound is oxidised—in dependence of pH—in two successive one-electron steps via unstable cation radicals which exhibit a strong adsorbability onto the electrode surface [17]. It seems that this could explain an enlargement of the second peak measured with the Ald / SO carbon paste electrode if one considers the behaviour of this CPE described in previous paragraphs.

### Conclusion

This report has presented a practical example of the characterisation of physico-chemical and electrochemical properties of a carbon paste electrode made from a

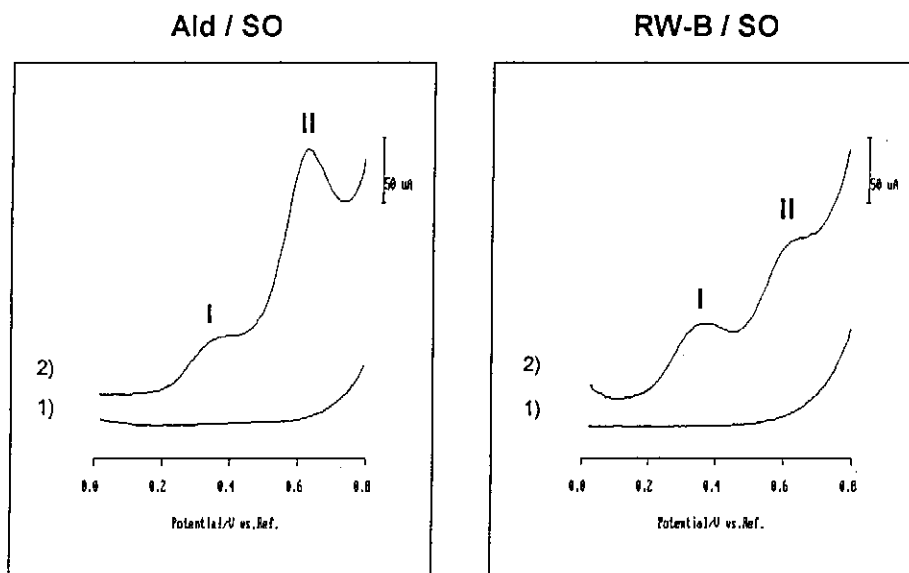


Fig. 9 Anodic oxidation of phenothiazine at Ald / SO and RW-B carbon paste electrodes in methanol-containing medium: 1) base-line, 2)  $c(\text{phenothiazine}) = 1 \times 10^{-4} \text{ mol l}^{-1}$ ; DPV mode; supporting electrolyte, 2 M  $\text{H}_2\text{SO}_4$  in 50 % methanol; initial potential, 0.0 V; final potential, +1.0 V; scan rate,  $20 \text{ mV s}^{-1}$ ; pulse height, 50 mV

new type of graphite powder.

Most observations and measurements have revealed that the Ald / SO carbon paste electrode, i.e. the electrode under test, exhibits a behaviour different from that observed for the RW-B / SO serving as standard CPE. The results of testing measurements suggest that individual nuances in the behaviour of both CPEs can be attributed to enhanced sorption capabilities of graphite used for the preparation of the electrode tested. Sorption properties of *Aldrich* graphite powder seem to be closely connected with its structure. In comparison with RW-B graphite, the particles of *Aldrich* powder have a smaller diameter (see *Experimental*) and hence, this spectroscopic graphite apparently provides a larger active surface per weight unit. Especially this can explain the behaviour of *Aldrich* graphite powder since the active surface area of any carbon material is usually in direct relationship with its sorption capabilities [5,7].

It can be concluded that the applicability of the Ald / SO carbon paste electrode—due to its sorption capabilities—appears to be relatively limited. For instance, experiments have shown that the Ald / SO electrode is not very convenient for stripping voltammetric determinations at very low concentrations because of high background currents. On the contrary, some studies (e.g., measurements of the electrode resistance and stability) have suggested that this CPE would offer a satisfying performance in other fields of electrochemistry such as potentiometry or special voltammetric investigations of qualitative character.

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