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Voltammetric and *in situ* FT-IRS study of the electropolymerization of *o*-aminobenzoic acid at gold and graphite carbon electrodes: Influence of pH on the electrochemical behaviour of polymer films

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ABSTRACT

The electrochemical homopolymerization of *o*-aminobenzoic acid has been performed on both gold and graphite surfaces in perchloric acid aqueous medium by cyclic scanning of the potential. Electroactive polymeric materials are formed in all cases but the amount of electropolymerization products seems to be increased by the graphite carbon electrodes. The polymers obtained have been characterized electrochemically. In addition, *in situ* FT-IR spectroscopy and scanning electron microscopy were combined to study the redox behaviour of the oxidation products and the surface morphology of the coatings. Characteristic absorption features related with benzenoid and quinoid rings and different types of C–N bonds point to the operation of redox processes similar to those undergone by the parent compound polyaniline. Owing to the presence of the –COOH group in the carbon backbone, polymer films obtained from *o*-aminobenzoic acid, contrary to polyaniline, electroactive at pH values over 3. Besides, it was observed that repeated oxidation–reduction cycles in working solutions with pH above 6 led to the dissolution of the deposited material.

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1. Introduction

Polyaniline (PANI) has been known in a variety of forms for over a century, being often considered the oldest of the conducting polymers [1,2]. It has always been at the forefront in the global search for commercially viable conducting polymers because of its unique reversible proton dopability, excellent redox cyclability, good environmental stability, low cost and easiness of preparation. In this way, PANI can be used as an electrode material [3,4] in the fabrication of secondary batteries [5–7] in microelectronics [8,9] or as an electrochromic material [10–12]. Future high-technology applications in the area of recordable optical disks [13] and chemical sensors have also been suggested [14].

A major limitation to the industrial exploitation of PANI derives from the fact that, on increasing the pH either during electrosynthesis or during operation, a significant drop of the electrical conductivity takes place. It was thought that the problem could be solved by the selection of specific counterions [15–19]. The role played by counterions is to enter the film to achieve what physicists called *doping*. From a chemical point of view, counterions pen-

etrate the polymer structure to compensate the positive charges generated along the backbone during its oxidation. The copolymerization of aniline monomers with other species has been suggested as an interesting option to achieve this purpose [20–22].

The use of substituted polyanilines has been also suggested to reduce the pH dependence of the pristine polymer. The introduction of anionic ionizable groups in the polymer structure results in a polymer with self-doping properties. Great number of the studies devoted to this topic focused on the effect played by sulphonic [23–27] and phosphonic [28] acid substituents. It is believed that the presence of anionic groups bound to the carbon rings affects the acidity constants of the amine moieties making the modified PANI structure less affected by the pH. Hence, the polymer becomes electroactive in slightly acidic, neutral or even in alkaline medium [29–31]. Despite the extensive literature dealing with the synthesis and characterization of sulfonated polyanilines, there are just a few papers paying attention to their parent carboxylated derivatives [32–41] and references cited therein. A key factor to explain that disproportion could be the ease of preparation of the sulfonic derivatives. Either post-sulfonation with fuming sulphuric acid or the polymerization of –SO₃H containing monomers are available to the scientists. On the contrary, to obtain carboxylated structures by post-modification very specific and

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laborious synthesis routes are required. Besides, the polymerization of carboxylated monomers is not often successful.

The present contribution aims to characterize the homopolymer obtained by the electrochemical oxidation of *o*-aminobenzoic acid on two different surfaces: gold and graphite carbon. The characterization will include both electrochemical and morphologic studies. It is expected that *in situ* FT-IR spectroscopy could provide valuable information about the redox processes undergone by poly(*o*-aminobenzoic acid) at the molecular level in the electrochemical environment. Finally, owing to the presence of carboxylic groups in the material synthesized, it is believed that the poly(*o*-aminobenzoic acid) will preserve part of its electroactivity at pH values where PANI is fully inactive (above pH 3).

2. Experimental

The monomer employed for the polymerization reaction was of reagent grade (Merck). The electrolytic medium for the synthesis of the polymeric materials was 0.1 M HClO₄. The aqueous media of different pH values were prepared from HClO₄/NaClO₄ solutions (Merck, p.a). All the solutions were made up with ultrapure water obtained from an Elga Labwater Purelab system (18.2 MΩ cm). The *in situ* FT-IR experiments were carried out in perchloric acid deuterated-aqueous solutions (99.9%-D) provided by Aldrich.

The working electrodes for the voltammetric study were either a spherical polycrystalline gold electrode with an area of about 3 mm² or a graphite carbon disc electrode with 0.3 cm diameter (LeCarbone Lorraine). For the *in situ* spectroscopic characterization, the polymers were grown up on a mirror-polished gold disk electrode (0.8 cm diameter). The counter electrode was a platinum wire in all cases. All the potentials were measured against a reversible hydrogen electrode (RHE) immersed in the working solutions through a Luggin capillary and are expressed in such a scale. The bare Au working electrode was thermally cleaned and subsequently protected from the laboratory atmosphere by a droplet of ultrapure water and the graphite carbon electrode was polished with a diamond suspension (Kemet International Ltd.) down to 1 μm particle size on a polishing cloth. After the cleaning step, the working electrode was transferred to the polymerization solution which was previously deaerated by bubbling N₂. Cyclic voltammograms were recorded at 50 mV s⁻¹ and at room temperature. After polymerization, the working electrode was extracted from the electrochemical cell, thoroughly rinsed with water to remove any attached monomer species and transferred to a clean background solution (free of any monomer) to be characterized by electrochemical or spectroscopic techniques.

A Nicolet Magna 850 spectrometer was employed for the *in situ* FT-IR experiments. The spectroelectrochemical cell was provided with a prismatic CaF₂ window bevelled at 60°. The interferograms were acquired with the working electrode surface pressed against this window and were collected at 8 cm⁻¹ resolution. The final spectra are presented in the usual form ΔR/R. Positive-going (upward) bands are related with vibrational modes not present at the sample potential. On the contrary, negative-going (downward) bands are displayed when the related vibration mode develops (or becomes active) at the sample potential. Scanning electron microscopy (SEM) images were acquired with a HITACHI S-3000 N microscope from films deposited on both gold and graphite electrodes.

3. Results and discussion

Figs. 1 and 2 show the cyclic voltammograms (CVs) recorded during the oxidative polymerization of 0.01 M *o*-aminobenzoic acid in 0.1 M HClO₄ aqueous solutions on gold and graphite electrodes, respectively. In both cases, the immersion of the electrode

Fig. 1. Cyclic voltammograms recorded for a gold electrode in 0.1 M HClO₄ during the electrochemical oxidation of 10 mM *o*-aminobenzoic acid. Scan rate 50 mV s⁻¹. — first, - - - second, ... 5th and —·—·— 25th sweeps.

was done at 0.1 V in order to avoid the initial oxidation of the monomer. Then, the potential was scanned in the positive direction up to 1.2 V and the anodic oxidation of the *o*-aminobenzoic acid was recorded. During the first forward scan, it can be observed that the onset of the anodic waves takes place at 1.0 V for graphite and at 0.95 V for the gold surface. However, the growth of the electroactive polymeric species is faster on the graphite electrode, as it can be inferred from the progressive development of two pair of electrochemically reversible features at around 0.5 V/0.4 V and 0.7 V/0.6 V. This two pair of peaks resemble those characteristic of polyaniline that are usually assigned to the polaron and bipolaron formation [42].

After 25 scans in the whole range of potentials, the electrodes were extracted from the electropolymerization solutions and thoroughly rinsed with water. Their respective electrochemical response were tested in background electrolytes which were free of *o*-aminobenzoic acid monomer. The cyclic voltammograms recorded in the 0.1–1.0 V potential range are depicted jointly in Fig. 3. There, it can be observed that, under the experimental conditions employed, the current densities increase with the number of cycles but these increase is more important for the graphite electrode. The voltammetric charge measured for the graphite support is approximately 200 times that of the gold support. Consequently, the amount of electroactive material deposited is larger for the carbonaceous support. There are two possible interpretations for this observation. On the one hand, it is possible that the graphite surface showed higher catalytic effect against the oxidative polymerization of *o*-aminobenzoic acid. On the other, the behaviour could be also interpreted in terms of a better adherence

Fig. 2. Cyclic voltammograms recorded for a graphite carbon electrode in 0.1 M HClO_4 during the electrochemical oxidation of 10 mM *o*-aminobenzoic acid. Scan rate 50 mV s^{-1} . — first, - - - second, ... 5th and — 25th sweeps.

of the poly(*o*-aminobenzoic acid) film to the graphite surface. In fact, it is believed that the interaction of the coating with gold is weaker than with graphite because noble metal are generally more electrocatalytic than carbon surfaces. Hence, the poor adherence might cause a partial and continuous release of the deposited material during the repetitive potential scans in the polymerization solution (Fig. 1), preventing the growth of a thicker layer on gold. Despite the different amount of material isolated, the voltammetric profiles reported in Fig. 3 strongly suggest that the chemical nature of the synthesized films is actually the same. Two distinct redox processes appear, the first one at 0.48/0.42 V (which results in a peak separation, ΔE_p , close to 60 mV) and the second at 0.73/0.61 V with $\Delta E_p = 120 \text{ mV}$.

Fig. 4 shows scanning electron micrographs obtained for gold (a) and graphite (b) electrodes covered with poly(*o*-aminobenzoic acid). The coatings were deposited in experiments parallel to those shown in Figs. 1 and 2, respectively. The film appears very smooth and thin on the gold electrode, supporting the results obtained in the cyclic voltammetry experiments. On the contrary, for the carbon electrode, the film morphology is more granular and the film reaches a higher thickness. The higher amount of material isolated on the carbon support is probably a consequence of the combination of the following effects: on the one hand, the higher porosity of the graphite electrode compared with the gold surface and, on the other, the better adherence of the film to the graphite substrate.

During the last years, there has been some controversy about the true electrochemical polymerization mechanism of aniline derivatives. However, it seems accepted that the first step involves the formation of a radical cation [43]. Later, this intermediate forms a dimeric species by *para*-coupling with either an unoxidized monomer or another radical cation giving rise to the propa-

Fig. 3. Voltammetric response of poly(*o*-aminobenzoic acid) film in 0.1 M perchloric acid background solutions. Films deposited on a: (a) gold electrode and (b) graphite carbon electrode, after 25 potential cycles in 0.1 M HClO_4 containing 10 mM *o*-aminobenzoic acid. Scan rate 50 mV s^{-1} .

gation of the reaction. This mechanism (involving *para*-coupling) is assumed to be valid also for the oxidative polymerization of *o*-aminobenzoic acid, as it has been illustrated in Scheme 1. It will be shown below that *in situ* FT-IR spectroscopy results are also compatible with the polymer structure derived from this kind of mechanism.

The previous voltammetric study suggests that only a little amount of polymerization products can be isolated on the surface of the gold working electrode from the oxidation of *o*-aminobenzoic acid monomer. In spite of this fact, the high sensitivity of the *in situ* FT-IR spectroscopy technique allows to obtain the vibrational spectrum of poly(*o*-aminobenzoic acid). In addition, working with D_2O solvent allows the acidic H atoms of poly(*o*-aminobenzoic acid) to be replaced by D atoms (the H/D ratio is about 0.001 in the electrolytic medium). As a result, the amino and carboxyl groups of the polymer will be deuterated whereas backbone hydrogen atoms will keep unaffected. Characteristic absorption bands in the surroundings of 1500 cm^{-1} are better resolved in deuterated medium owing to the vanishing of the O-H bending mode of water.

In Fig. 5 *in situ* FT-IR spectra obtained in $\text{D}_2\text{O} + 0.1 \text{ M HClO}_4$ solution for a gold electrode covered with poly(*o*-aminobenzoic acid) are presented. The gold electrode coated with the polymeric material was transferred to the spectroelectrochemical cell and immersed in the test solution at 0.2 V. After equilibration, the reference spectrum was collected at the same potential. In this way, the reference contains the vibrational information on the reduced polymer state. The potential was then stepped to higher values

Fig. 4. Scanning electron micrographs obtained for the films deposited on gold (a) and graphite (b) electrodes in 0.1 M HClO₄ solution during the electrochemical polymerization of *o*-aminobenzoic acid.

to collect vibrational information associated with the oxidized form of poly(*o*-aminobenzoic acid). Sample potentials of 0.6 V and 0.9 V are depicted in Fig 5, which display several absorption bands. From the higher intensity of the bands at 0.9 V it is derived that the oxidation level of the polymer progress with the applied potential. Characteristic positive-going bands appear at around 1656 cm⁻¹, 1487 cm⁻¹, 1375 cm⁻¹ and 1276 cm⁻¹ which are re-

Fig. 5. *In situ* FT-IR spectra collected during the oxidation on a gold electrode of a poly(*o*-aminobenzoic acid) film in 0.1 M HClO₄ solution. The sample potentials are indicated and the reference was acquired at 0.2 V. One thousand interferograms were collected at each potential. 8 cm⁻¹ resolution.

lated with vibrational modes of the polymer in its reduced state. On the other hand, negative-going bands appearing at 1708 cm⁻¹, 1598 cm⁻¹, 1530 cm⁻¹ and 1326 cm⁻¹ are related to vibrations coming from the oxidized state of the polymer. The negative-going band at around 1126 cm⁻¹ is related with the penetration of perchlorate anions during the oxidation of the polymeric material. With regard to the upward bands, the feature at 1276 cm⁻¹ can be easily assigned to the single bond C–N stretching in secondary aromatic amines. Its negative character clearly points to the transformation of these amines into imines (negative-going band at 1598 cm⁻¹) and C–N moieties of intermediate order (negative-going band at 1326 cm⁻¹) when the polymer is oxidized. The feature at 1487 cm⁻¹ is related with the disappearance of the aromatic C–C stretching vibration which is present at 0.2 V. This band together with the existence of a negative-going peak at 1530 cm⁻¹

Scheme 1.

(quinoid $\text{C}=\text{C}$ stretching) reveals the oxidation of benzenoid to quinoid rings at the sample potentials employed. For clarity, all the peak frequencies obtained for both the reduced and oxidized states of the polymer are summarized in Table 1.

From the *in situ* FT-IR spectra presented here, it is also deduced that the carboxylic group plays a role in the oxidation mechanism of poly(*o*-aminobenzoic acid). This is evidenced by the presence of a bipolar feature at $1656\text{ cm}^{-1}/1708\text{ cm}^{-1}$, which appears in the spectral region typical for the $\text{C}=\text{O}$ stretching vibration. If the carboxylic group were not involved in any way in the redox transformation, the band would not be present in the spectra of Fig. 5 because only those modes suffering changes induced by the applied potential appear in the *in situ* FT-IR technique. Besides, the existence of chemical interaction between the carboxylic and the neighbouring amino group in the reduced state of poly(*o*-aminobenzoic acid) is at the origin of the appearance of $\text{C}=\text{O}$ stretching in the spectra. It is well known that this kind of interaction lowers considerably the $\text{C}=\text{O}$ stretching frequency of those aryl carboxylic acids having amino groups in the *ortho* position [47]. The interaction can only be present in the reduced state (thus decreasing the carboxylic $\text{C}=\text{O}_{\text{str}}$ frequency down to 1656 cm^{-1}). When the polymer is oxidized, amine nitrogens transform into protonated imines and the interaction is not possible. The carboxylic groups are then released and the $\text{C}=\text{O}$ stretching frequency returns to its characteristic value, which is higher than 1700 cm^{-1} . The transformation has been depicted in Scheme 2.

One of the key points working with self-doping polyaniline derivatives is that of the effect of pH on the electrochemical response of the polymer. Thanks to the incorporation of the $-\text{COOH}$ group it was expected that the electroactivity of poly(*o*-aminobenzoic acid) could show better performance than polyaniline in aqueous media of higher pH. In this way, polymeric materials were synthesized on a gold surface in perchloric acid medium and then rinsed and transferred to clean background solutions of pH ranging from 1 to 6 to test their electrochemical response. The ionic strength of all the solution was kept constant by adding the required amount of NaClO_4 to the HClO_4 medium. The cyclic voltammograms recorded are presented jointly in Fig. 6. Most of the voltammetric profiles show two redox processes centred at around 0.4 V and 0.7 V , similarly to those obtained in strongly acidic med-

Fig. 6. Cyclic voltammograms recorded for a poly(*o*-aminobenzoic acid) film deposited on a gold electrode in test solutions of increasing pH: (—) pH 1; (---) pH 3; (- - -) pH 5; (· · ·) pH 6.

ium (Fig. 3). The electric charge associated with these processes drops slightly (less than about 10%) in the pH range from 1 to 5, as can be deduced from the plot in Fig. 7. This result strongly suggests that the poly(*o*-aminobenzoic acid) retains most of its electroactivity even at pH values where the conventional polyanilines are inactive. The role played by the ionizable $-\text{COOH}$ group is therefore of extreme importance to guarantee an adequate doping level of the polymer backbone in solutions having moderately acidic pH. In spite of this, it can also be observed in Fig. 6 that increasing the pH of the test solution over 6 results in the disappearance of the characteristic voltammetric features for poly(*o*-aminobenzoic acid) and to an irreversible loss of its associated electric charge (more than 70%). In a further experiment, after recording the cyclic voltammogram in the pH 6 medium, the electrode was transferred again to a clean 0.1 M HClO_4 solution and the potential cycled in the $0.1\text{--}1.0\text{ V}$ range. It was expected that the poly(*o*-aminobenzoic acid) could re-activate in strongly acidic medium, however the cyc-

Table 1

Assignments of the vibrational bands for the reduced and oxidized forms of poly(*o*-aminobenzoic acid) in acidic D_2O [35,36,44–47]

Oxidation state	Frequency/ cm^{-1}	Assignment
Reduced	1656	Carboxylic acid ($\text{C}=\text{O}$) str.
	1487	Aromatic ($\text{C}=\text{C}$) str.
	1375	($\text{C}-\text{H}$) bending
	1276	Secondary amine ($\text{C}-\text{N}$) str.
Oxidized	1708	Carboxylic acid ($\text{C}=\text{O}$) str.
	1598	Imine ($\text{C}=\text{N}$) str.
	1530	Quinoid ring ($\text{C}=\text{C}$) str.
	1326	Intermediate order ($\text{C}\cdots\text{N}$) str.
	1126	ClO_4^-

Scheme 2.

Fig. 7. Fraction of the voltammetric charge lost by the poly(*o*-aminobenzoic acid) films after potential cycling in aqueous media of increasing pH.

lic voltammogram of the polymer was not completely recovered. That result strongly suggests the lack of voltammetric features at pH 6 (Fig. 6) is due, in fact, to the partial dissolution of the deposited material. The presence of the unprotonated -COO^- group (pK_a roughly 2) increases the electrostatic interactions between the polymer and the solvent as the pH becomes more alkaline. As a result, at pH values around 6, the polymer is forced to dissolve.

4. Conclusions

Cyclic voltammetry has been employed to synthesize poly(*o*-aminobenzoic acid) films on both gold and graphite electrodes. The graphite surface seems more appropriate to obtain thicker films, probably as a consequence of the higher chemical interaction with the polymer, which results in better adherence to the support.

From an electrochemical point of view, the redox response of the deposited films resembles that of polyaniline: Both polymeric materials show two separate voltammetric processes which seem related with the formation of polaronic and bipolaronic species. The *in situ* FT-IR spectroscopy showed that, in parallel to polyaniline, the transformation of benzenoid into quinoid rings and amine into imine structures also operates during the oxidation of poly(*o*-aminobenzoic acid).

However, the presence of the -COOH group attached to the poly(*o*-aminobenzoic acid) backbone confers better electrochemical performance on this material. As a result, poly(*o*-aminobenzoic acid) is able to retain most of its oxidation-reduction characteristics in aqueous solution of pH as high as 5.

Finally, it has been observed that poly(*o*-aminobenzoic acid) dissolves in test solutions of pH over 6. The stronger interaction between the unprotonated R-COO^- and the solvent seems at the origin of this observation. This result opens the possibility of obtaining a water-soluble polyaniline derivative to be applied as a paint material on non-conducting surfaces.

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