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POLYANILINE COATED PT ELECTRODES : CHARACTERIZATION AND ELECTROCHEMICAL RESPONSE

M.A. Abdel Rahim, Amel A. Abdel Rahman and M.W. Khalil
Department of Chemistry, Faculty of Science, Cairo University, Giza, Egypt.

ABSTRACT

Polyaniline was electropolymerized on Pt electrodes in an aqueous medium of 0.5 M H_2SO_4. The electropolymerization was performed by the potential scan method (PSM) and the potential pulse method (PPM). It was found that polyaniline films formed by the two methods ares stable upon soaking in a sulphate mixture composed of 0.1 M Na_2SO_4 + 0.5 M H_2SO_4, pH 0.5, and have almost the same electrochemical characteristics which were studied by the cyclic voltammetric technique.

The kinetics of the redox reaction of $[Fe(CN)_6]^{3-/4-}$ was studied at the Pt-polyaniline modified electrodes in the acidic sulphate mixture. Films prepared by PPM are more efficient catalysts than those prepared by PSM. The reactions at those films were found to proceed reversibly under diffusion control. A concentration down to 1 x 10^{-4} M of $[Fe(CN)_6]^{3-}$ was detected at the polyaniline films.

1. INTRODUCTION

Conducting polymers have been extensively studied due to their potential applications in microelectronic devices (1), analytical sensors (2), electrocatalysis (3), electrooptic displays (4,5) and rechargeable batteries (6,7). Polyaniline appears to be one of the conducting polymers, that is generally homogeneous, strongly adherent to the support and chemically stable in acid medium (8,9). The electropolymerization mechanism of aniline and its film growth have been extensively investigated by several authors (10-12).

It is reported that, polyaniline films exhibit both ionic and

electronic conductivities. Unlike all other conducting polymers, the conductivity of polyaniline depends on two variables : namely, the degree of oxidation of the polyaniline and the degree of protonation of the material (8). The conductivity also depends dramatically on the pH of aqueous solution (13). With increasing solution pH, electrochemical activity of the films are destroyed, and they are finally lost if the pH is greater than 3-4 (10,14). This difference in conductivity is closely related to the polymer morphology (15). The films are conducting in the oxidized state, and are insulating in the reduced state (10,16,17), independent of the polymerization conditions. It is expected that they behave as good electronic conductors at intermediate oxidation levels (1, 18). Good stability and conductivity were observed only in a narrow potential range from -0.2 to 0.7 V (SCE) and the films readily undergo oxidative degradation at potential above 0.7 V (19). Yoshikawa *et al.*, (20) prepared highly conducting polyanilines by selecting the appropriate conditions in electrolysis.

Polyaniline films act as a barrier to ion / solvent transport, but they permit electron exchange with the electrolyte (21). They could be used as an electrode material for mediating the oxidation or reduction of a dissolved electroactive species. Some appreciable experimental work in the field of kinetics of solute redox-species reaction on electrodes covered with electronically conducting polymers have been published in the literature (22,23). It is aimed in this paper to use the Pt - modified polyaniline as a sensor electrode for ionic species in solution; $[Fe(CN)_6]^{3-}$ is used for this purpose.

2. EXPERIMENTAL

2.1. Electrochemical cell and reagents.

Aniline of reagent grade was purified by distillation in the usual

manner before use. AR H_2SO_4, sodium sulphate and potassium ferricyanide were used as received without further purification. These were prepared by using triply distilled water.

A three - electrode glass cell with a saturated calomel electrode (SCE) as a reference electrode is used. The reference electrode was placed in an external salt bridge filled with the same electrolyte and connected to the cell through a quick fit joint with a Luggin capillary whose tip was placed close to the working electrode. A platinum wire was employed as a counter electrode, it was separated from the rest of the cell by a sintered glass disk. The working electrode was a spectroscopic grade Pt disk (area is 0.07 cm^2) sealed in a Teflon tube. The electrode was polished with 0.3 μm alumina slurry, rinsed well with triply distilled water. Care was made to remove residual polishing materials from the electrode surface. All potentials were referred to the SCE unless otherwise stated.

2.2. Instrumentation.

The electrochemical measurements were performed using system AMEL 5000 (supplied by Amel instrument, ITALY) driven by an IBM PC enabled the processing the data. The PC was interfaced with the instrument through a serial RS - 232C card. Amel Easyscan software was used in connection with the PC to control the Amel 5000 system. All the reported potentials were corrected up to 90% of their values by the positive feedback technique.

2.3. Polyaniline film growth.

The films were prepared by the electropolymerization of aniline on a Pt - disk electrode, in an aqueous solution containing 0.1 M aniline and 0.5 M H_2SO_4, using two different techniques :

Potential scan method (PSM) : continuous potential cycling between -0.2 and 0.8 V vs. SCE at a scan rate of 50 mV s^{-1}.

Potential pulse method (PPM) : repeated potentiostatic pulses with a duration time of 500 ms and amplitudes between -0.05 and 0.80 V were applied to the working electrode (24).

No colored soluble products were seen near the working electrode during the electropolymerization process, which indicates a quantitative polymerization with almost 100% yield (9).

3. RESULTS AND DISCUSSION

3.1. Electrochemical characteristics of polyaniline.

The techniques used in this study, PSM and PPM, produce a high - quality smooth cohesive film on the substrate. The difference between the two techniques lies in the different growth rates of the polyaniline films. Film growth using PSM is slower than the PPM. The electrochemical characterization of polyaniline films was performed by cyclic voltammetric technique.

Fig. 1 shows a typical cyclic voltammogram of the electropolymerization process of aniline by PSM. It is noted from this figure that the oxidation - reduction peak current densities of aniline increases by cyclization. This indicates that increasing the number of cycles increases the polymer thickness. The oxidation charge of electropolymerized aniline was simultaneously obtained every cycle, and on the other hand for every pulse in the PPM, and is used to calculate the polymer film thickness (25). The thickness was calculated using a density of 1.2 g cm^{-3} (26) and 0.7 electron per aniline ring are exchanged during the redox process of the polymer (16). The oxidation charge obtained at every cycle (and pulse) is plotted as a function of thickness in Fig. 2a which shows two straight lines with two different slopes. One straight line up to 70 cycles (in case of PSM) and up to 200 pulse (in case of PPM), and another of higher

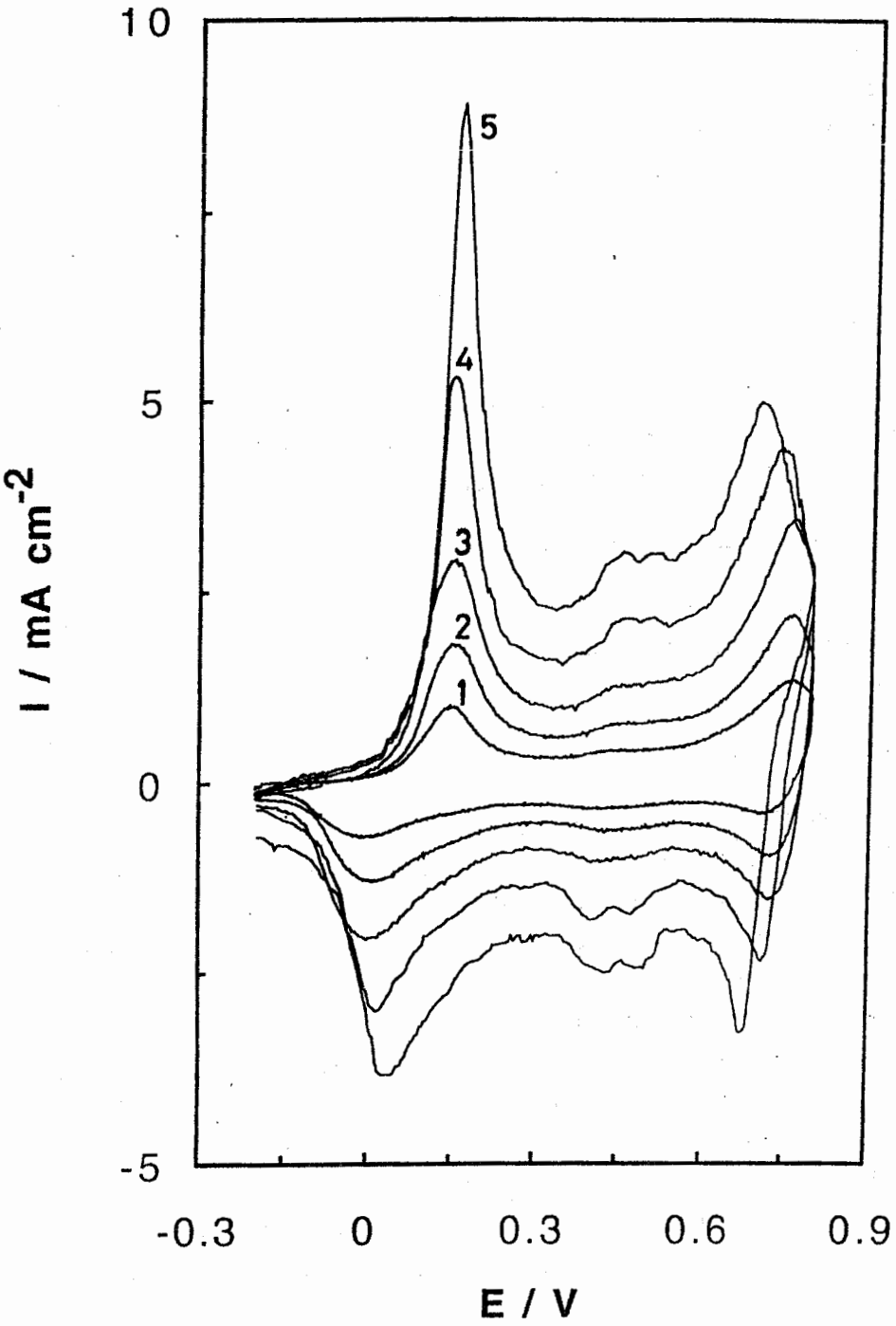

Fig. (1).

slope above these limits. This indicates that the film grows with higher rate after it reaches a certain thickness probably due to the cocatalytic phenomena. A linear relation was also obtained for polyaniline by Oyama *et al.*, (27) and for poly (naphthidine) by Arevalo *et al.*, (28). Fig 2b represents the relation between thee calculated film thickness and the oxidation charge. Values of the film thickness thus obtained are comparable with those obtained by other authors (16).

In the present study, during the film preparation in each case, the last potential cycle was stopped at a potential value of + 0.80 V to insure that the polymer film is in its conducting state due to :

- The film thus obtained is in its oxidized form.

- Insure the doping of the sulfate anion, SO_4^{2-}, from the electrolyte into the polymer film which contributes to its conductivity.

3.2. Effect of film thickness

The reduction-oxidation cyclic voltammogram of 1 x 10^{-2} M $[Fe(CN)_6]^{3-}$ in a mixture of 0.1 M Na_2SO_4 + 0.5 M H_2SO_4, pH 0.5, was recorded by scanning the potential from 0.6 to 0 V at a scan rate of 50 m V s^{-1} on the uncovered Pt-electrode, for comparison. The result is represented in Fig. 3a as a cyclic voltammogram of Pt in the background solution and another of that containing the tested ions. The cathodic and anodic reduction and oxidation peaks appear respectively at 365 and 430 mV with a potential separation of 65 mV. After that, the effect of polyaniline film thickness (both formed by PSM and PPM) on its electrochemical responses to $[Fe(CN)_6]^{3-}$ in the background sulphate solution (pH 0.5) was studied at the same potential window and at the same scan rate (Fig 3b). A systematic

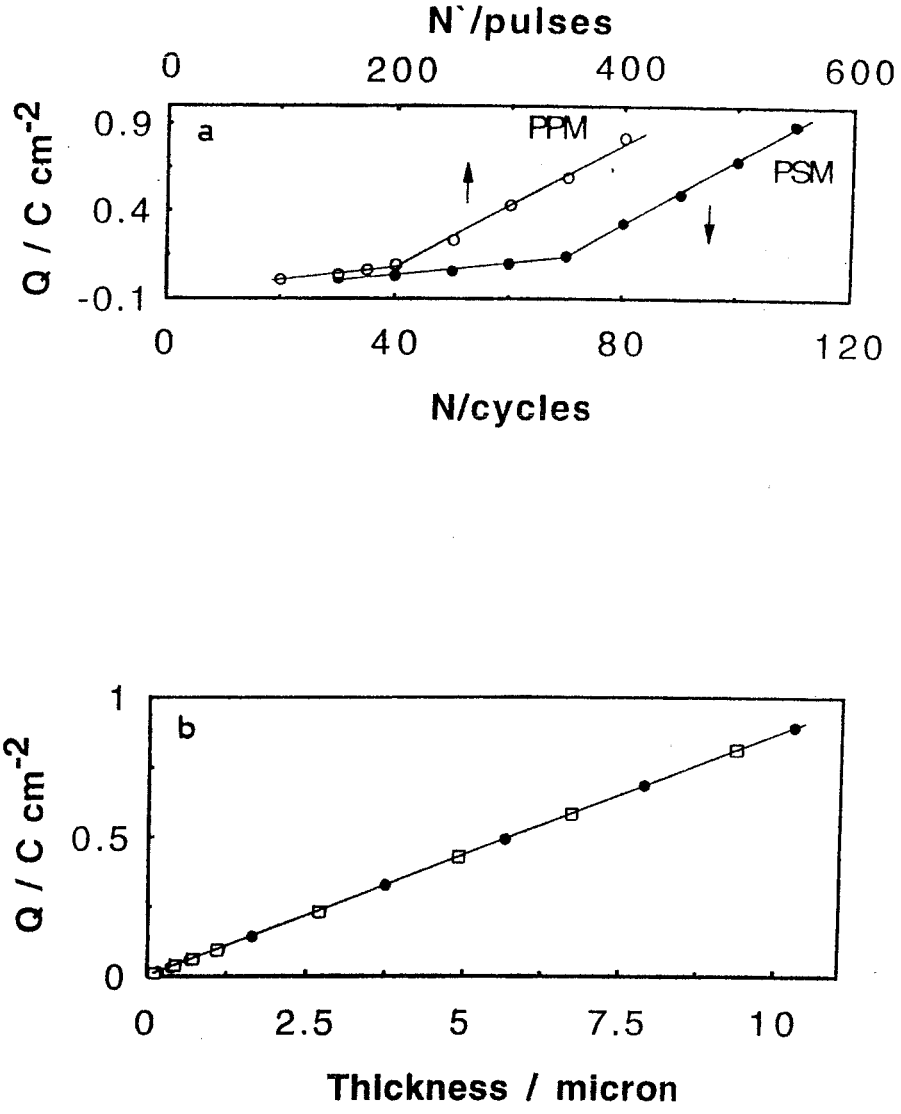
N`/pulses
0
200
400
600
Q / C cm-2
0.9
0.4
-0.1
a
PPM
PSM
0
40
80
120
N/cycles
1
0.5
0
b
0
2.5
5
7.5
10
Thickness / micron

Fig. (2).

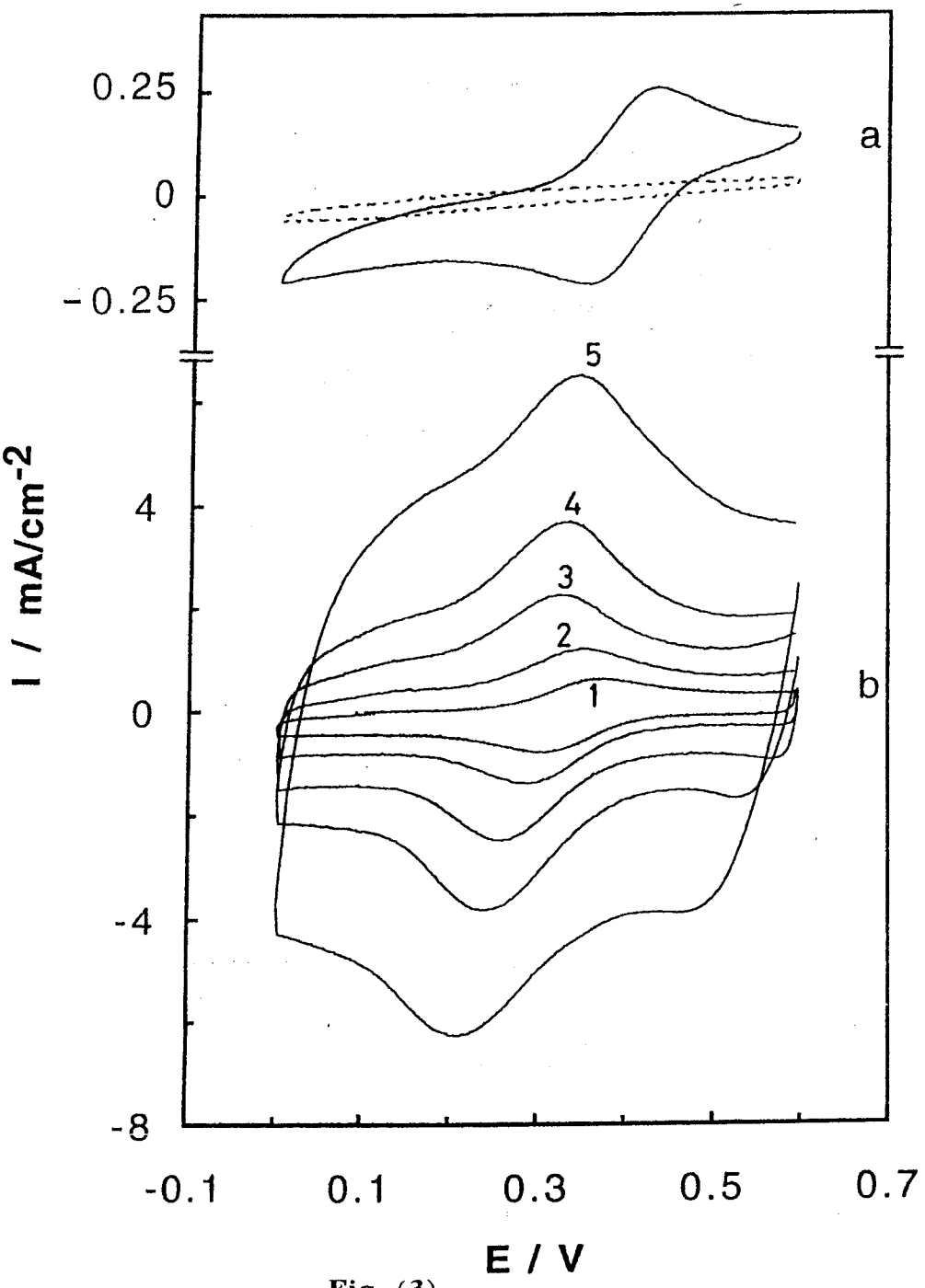

Fig. (3).

procedure was carried out with respect to these experiments : after electropolymerization, at the desired number of cycles (or number of pulses) the polyaniline coated electrodes were well washed with the sulfate mixture, pH 0.5, to free them from traces of aniline and to remove any soluble compounds left over from the growth medium. The Pt-polyaniline electrode was then transferred into a cell containing only the sulfate mixture and continuous cycling of potential, between 0.6 and 0 V range at a scan rate of 50 mV s^{-1}, was carried out for about 20 cycles in order to :

- stabilize the film in that solution.
- get a stationary state cyclic voltammogram in the background solution for point-point subtraction with that containing the tested redox system.

It was noted in the above experiments that, when the higher limit of the potential scan is expanded to more positive values (more than 0.6 V), the film loses its characteristic response, although it remains adherent to the Pt electrode surface. In addition, this potential window, 0 - 0.6 V, is far from the switching reaction of the polymer film (14) and is suitable for studying the reduction - oxidation of $[Fe(CN)_6]^{3-}$ ions because their standard electrode potential lies in this range. On the other hand, the metallic behaviour of polyanilines had to match high exchange current density values at this range of potential (29). Furthermore, in each experiment the potential was stopped at 0.6 V. With this upper potential, degradation reactions are supposed to be limited (19).

Fig. 3b shows the electrochemical response of polyaniline films to 1 x 10^{-2} M $[Fe(CN)_6]^{3-}$ for different film thickness. It should be remembered that, as denoted above, the film was stabilized by repeatedly cycled from 0.6 to 0 V in the background solution after each electropolymerization and before carrying out the experiment in

the redox - containing solution. It is noted, in each stabilization experiment, that the voltammogram responses show higher capacitive current produced with films electropolymerized to higher number of cycles, and on the other hand to higher number of pulses. We assume, in agreement with other authors (11), that high capacitive currents cause the insertion of the electrolyte anions into the polymer film during its growth, and consequently the increase of film conductivity is expected. After every experiment in the redox - containing solution, the electrode was introduced again into the sulphate mixture in which cyclic voltammetry tests run again at 50 mV/s^{-1} in the range 0.6 - 0 V. In such cases and in the first stabilization cycles, the voltammogram contains the peaks corresponding to the reduction and oxidation of $[Fe(CN)_6]^{3-}$ and $[Fe(CN)_6]^{4-}$ ions, respectively, which readily disappear by cycling the potential. This confirms that these ions are incorporated into the polymer film during the electron transfer process (30). Certainly, the release process of the incorporated ions proceeds much slower if the polymer film was grown to a higher number of cycles (or pulses), i.e., thick films. On the other hand, when comparing the two types of films, it was found that the release process of the incorporated ions proceeds much faster in case of polymer films prepared using the PPM.

Inspection of the cyclic voltammograms of Fig. 3b leads to the conclusion that increasing the number of cycles (or pulses) in the film preparation process, i.e., increasing the polymer film thickness, results in increasing the current peaks corresponding to the redox species, $[Fe(CN)_6]^{3-}$. This is expected, where, the increase of film thickness is accompanied by .

- Increase of its effective area and hence more sites would be available for the electron exchange process.

- Insertion of more SO_4^{2-} ions from the forming electrolyte into the polymer film and thus improving the film conductivity.

Values of peak potential of reduction and oxidation (E_{pc} and E_{pa}, respectively) of $[Fe(CN)_6]^{3-/4-}$ on Pt and Pt-polyaniline are very close to the values obtained by Rourke *et al.*, (30). When comparing the voltammograms of Fig. 3b (especially those of lower number of cycles) with the voltammogram of Fig. 3a; the peak potential of the redox species $[Fe(CN)_6]^{3-}$ is by about 55 mV more negative than the corresponding values at the uncovered Pt. A similar shift was observed by some authors (30,31).

For films formed at a number of cycles ≥ 60 cycle (with respect to PSM) and ≥ 200 pulse (with respect to PPM) the cathodic-anodic peak separation (ΔEp) increases with increasing the film thickness. The relatively large separation (> 100 mV) at high film thickness is considered to the due to increase of film resistance. The film thickness that shows satisfactory electrochemical response is that corresponding to an electropolymerization at about 50 cycles in PSM and 175 pulses in PPM. These responses are; namely, reversible cathodic and anodic peaks separation (not more than 60 mV) and relatively high peak current densities for the electrochemical response to $[Fe(CN)_6]^{3-}$ and high rate of releasing of these ions by cyclization in the background solution (see above). Accordingly, films formed at 50 cycles in the PSM and 175 pulses in the PPM were then used in the next experiments.

The cathodic peak current densities (in case of films prepared by PSM and PPM) were plotted as a function of film thickness in Fig. 4. This relation is linear in both cases up to a film thickness of about 1.5 μm above which it deviates from linearity. This value of thickness was found to correspond to the break point in Fig. 2a.

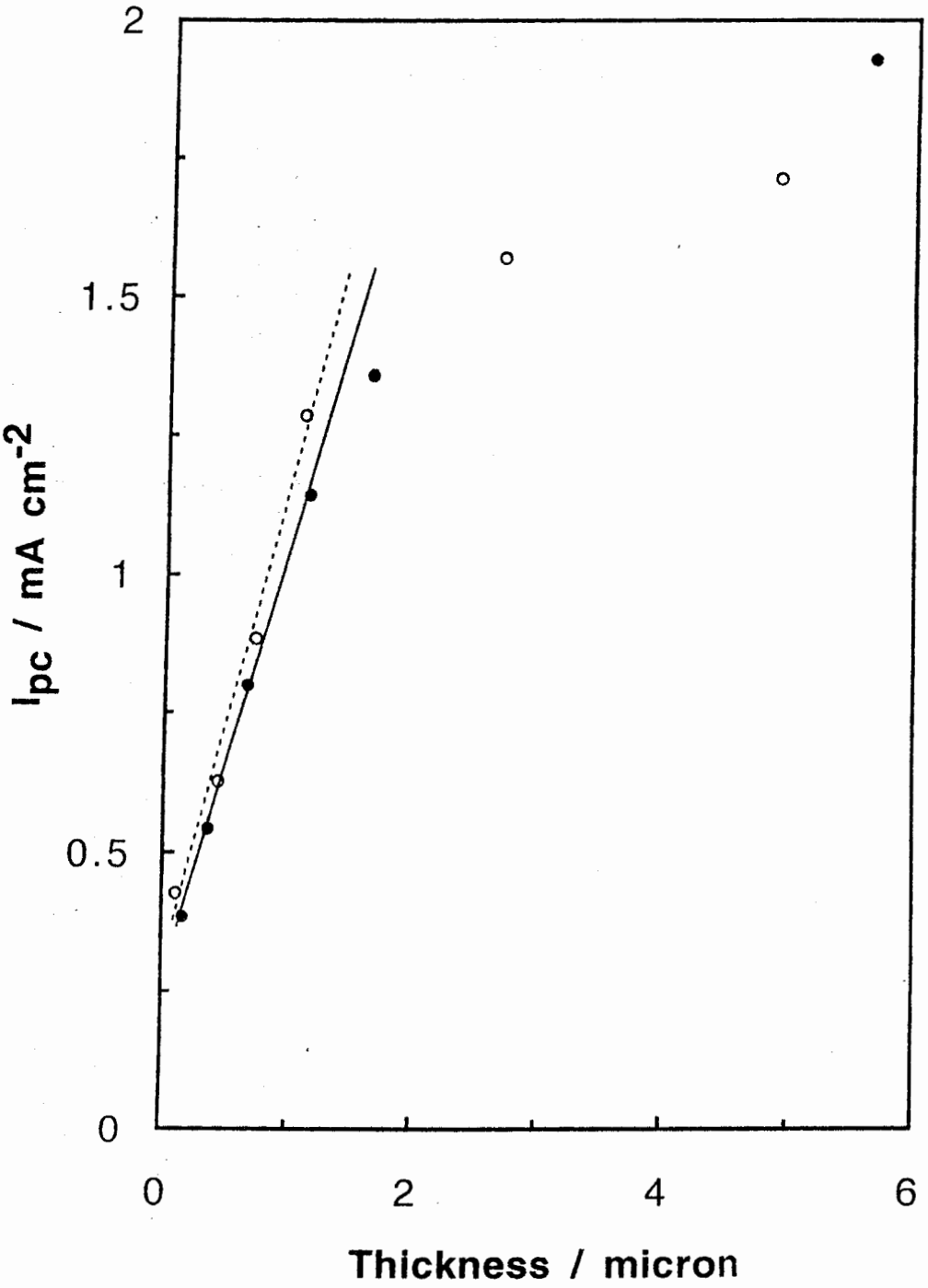

Fig. (4).

Furthermore, high $[Fe(CN)_6]^{3-}$ reduction-oxidation peak separation was observed in case of films prepared to a thickness above this limit., see Fig. 3b. Again, this is considered to be due to the increase of film resistance after 1.5 μm thickness.

3.3. Scan rate effect

The electrochemical response of polyaniline films, prepared by PSM and PPM, to 1 x 10^{-2} M $[Fe(CN)_6]^{3-}$ was studied at various scan rates. The study was carried out by two methods : varying the scan rate from high to low values in one set of experiments and from low to high values in the other set. Each set of experiments was carried out on a new layer of polyaniline film.

Plotting the peak current densities (cathodic and anodic) against the square root of the scan rate show straight line relations, as observed in Fig. 5. This confirms that the electron transfer process at the film is diffusion controlled (21). The potential peaks separation (ΔE_p of the cathodic and anodic reactions) is in the range 57 - 60 mV characteristic for the one electron reversible process. The nearly constant values of E_{pc} and Epa is another criterion for the reversibility of the redox reaction. This is observed in both cases, in case of polymer films prepared using PSM and also PPM.

The experimental results show that the electrochemical response of polyaniline to $[Fe(CN)_6]^{3-}$ is the same whether the scan rate varried from high to low values or vice versa.

3.4. Effect of $[Fe(CN)_6]^{3-}$ concentration

Using films prepared by PSM and PPM, the electrochemical response of polyaniline to different concentrations of $[Fe(CN)_6]^{3-}$ was studied. A set of experiments was carried out by varying the concentration from high to low values. In this case the same

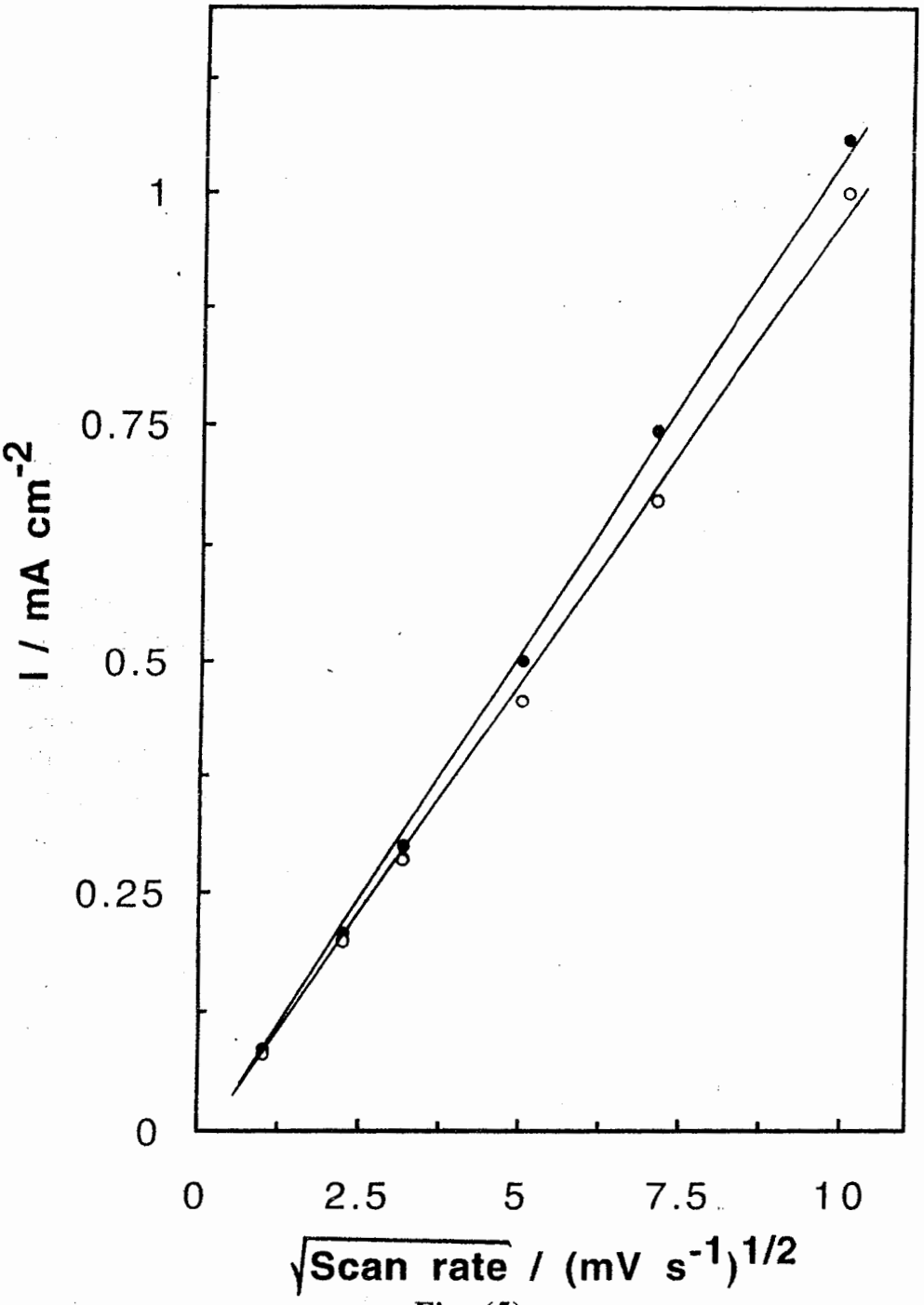

Fig. (5).

procedure as in the scan rate effect was carried out. It was observed that polyaniline responds to a lower concentration (1 x 10^{-4} M [Fe $(CN)_6]^{3-}$) than the uncovered Pt electrode (1 x 10^{-3} M).

Another category of experiments was carried out also on a polyaniline films grown at the same conditions as above, but in this case the concentration of [Fe $(CN)_6]^{3-}$ is now varied from low to high concentration . It was noted in these experiments that starting with the lowest concentration, 1 x 10^{-4} M, polyaniline does not respond to this concentration unless the cycling of potential was repeated up to 10 cycles, or soaking the electrode in the [Fe $(CN)_6]^{3-}$ - containing solution for about 5 minutes. According to these experiments, it is possible to conclude that [Fe $(CN)_6]^{3-}$ must be incorporated in the polyaniline films in order to enhance its response. The incorporated ions may play a role in the electron transfer of [Fe $(CN)_6]^{3-}$ ions in solution. It was reported that the incorporation process may proceed by one of the following or both :

- Electrostatic trapping upon soaking (32, 33).
- Ion exchange by potential cycling of the pre-formed film in the ion containing solution (30).

It is then necessary to remove the incorporated [Fe $(CN)_6]^{3-}$ ions before using the polymer film in another test with another ion concentration. Cycling the electrode in an [Fe $(CN)_6]^{3-}$ - free solution (the background sulfate mixture) for several cycles leads to the expulsion of the incorporated ions. The cycling was repeated until stable voltammogram free from peaks was obtained.

Referring to the cyclic voltammetric curves, it was found that the current density of the cathodic peaks, Ipc, for [Fe $(CN)_6]^{3-}$ ions reduction within the polymer is larger than the anodic peaks, Ipa, especially for high bulk concentration. This suggests that polyaniline

stabilizes the $[Fe(CN)_6]^{3-}$ at a half of the redox couple, in agreement with Rourke *et al* (30), and the electrode reaction in polyaniline film is electrochemically less reversible. Another possible explanation for this is that, in the anodic half cycle, part of $[Fe(CN)_6]^{4-}$ produced by the reduction of $[Fe(CN)_6]^{3-}$ in the cathodic half cycle, can be loaded into the polymer film. Accordingly, the amount of $[Fe(CN)_6]^{4-}$ oxidized in the anodic half cycle become less than $[Fe(CN)_6]^{3-}$ reduced in the cathodic half cycle. This is only in the case of polymer prepared by PSM. But with respect to polymer films prepared by PPM the ratio I_{pc}/I_{pa} is close to one which reflects the reversibility of the redox reaction at this types of films. Fig. 6. represents the relation between the ratio I_{pc}/I_{pa} and the redox concentration in case of polymer films prepared by both methods. This shows that the redox reaction at polymer films prepared by PSM approaches the reversibility at low bulk concentration (1×10^{-4} M). On the other hand, such reactions at films prepared by PPM are almost reversible. As reported above, part of $[Fe(CN)_6]^{4-}$ produced by the reduction of $[Fe(CN)_6]^{3-}$ in the cathodic half cycle may incorporate inside the polymer film. The amount of incorporated $[Fe(CN)_6]^{4-}$ ions depends on the bulk concentration. This could explain the reversibility at low bulk concentration in case of films prepared by PSM. While in case of films prepared by PPM, this type of films were grown at faster rates than those prepared by PSM. Rourke *et al.*, (30) reported that a fast growing film has a granular morphology. Accordingly, the loading-deloading process of $[Fe(CN)_6]^{4-}$ ions roceed much faster in case of these films due to their high porosity, which on the other hand, could explain the reversibility of the redox reactions at polymer films prepared by PPM. Fig. 7 represents the relation between the cathodic peak current and the redox concentration. For reactions at films prepared by PPM, the cathodic peak current is linearly proportional to the concentration in range 10^{-4} - 10^{-2} M, while it is not the case with respect to the results obtained at films prepared by PSM.

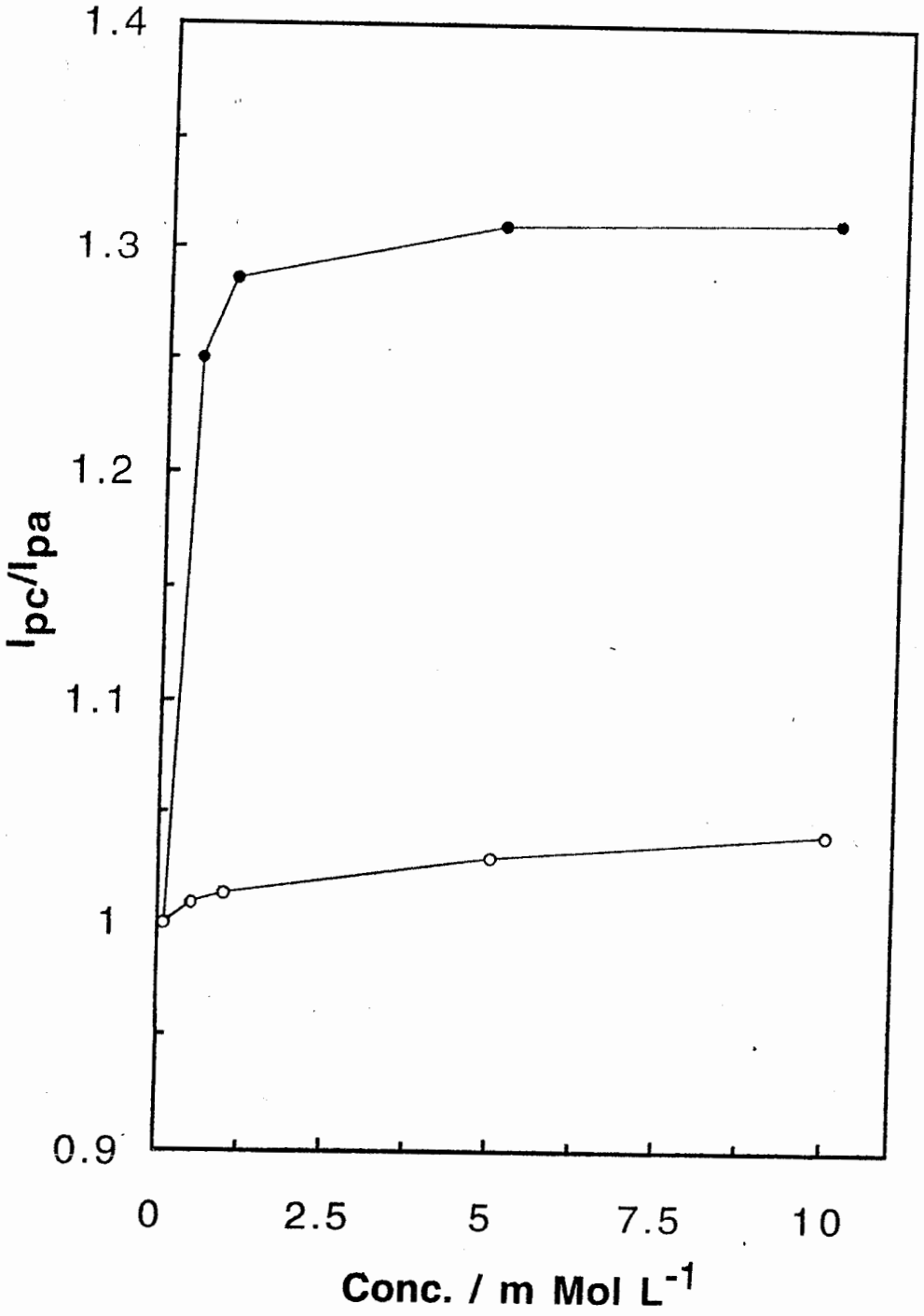

Fig. (6).

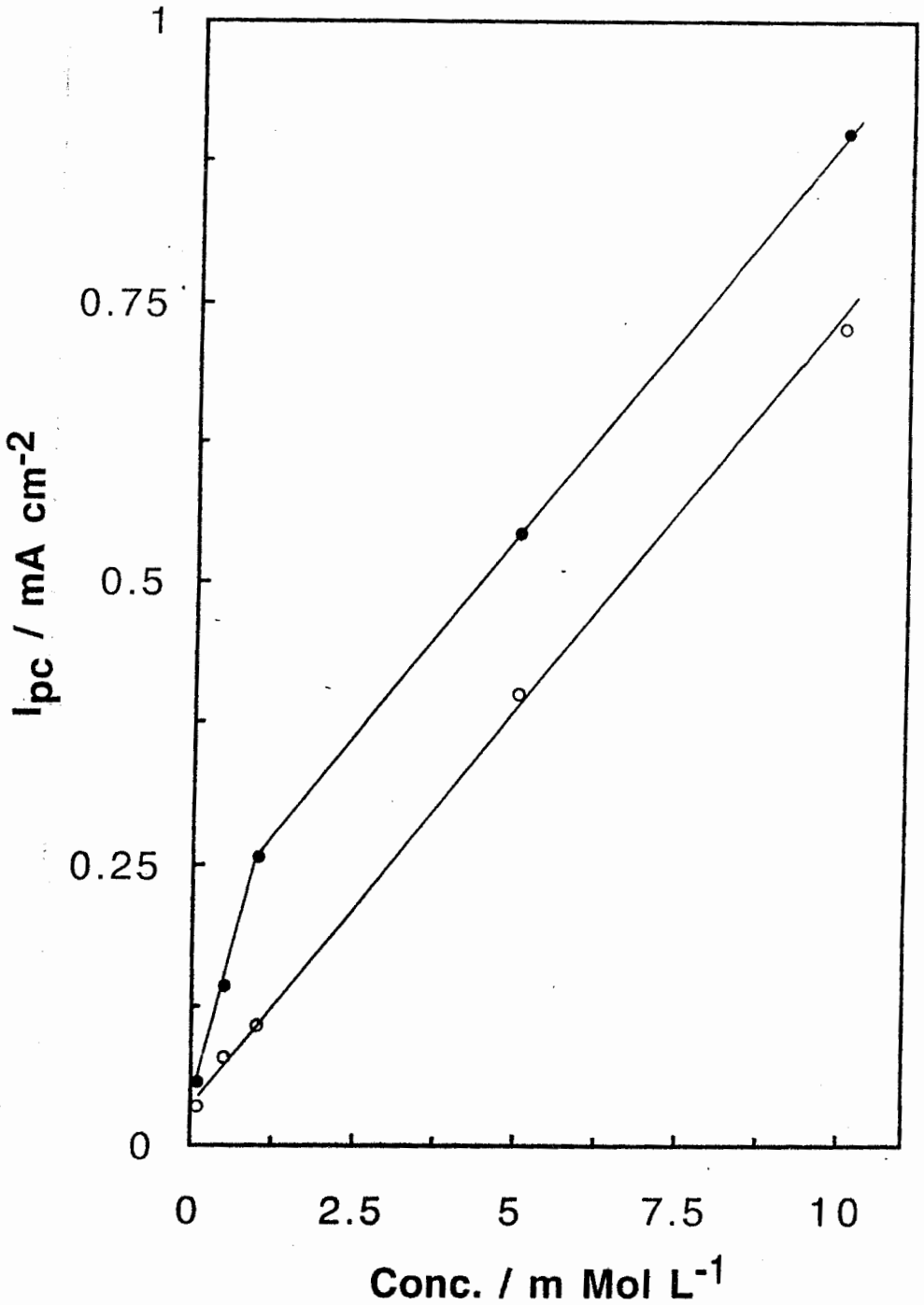

Fig. (7).

The experimental results of the concentration effect of $[Fe(CN)_6]^{3-}$ reveal that, for a particular concentration of the $[Fe(CN)_6]^{3-}$, the same peak currents and potentials were always obtained whether the concentration was increased or decreased. This effect was found to be similar for both techniques of polymer preparation. The experimental results also reveal that the redox reactions of $[Fe(CN)_6]^{3-}$ at Pt -polyaniline electrodes proceed at the polymer/solution interface and at the bulk polymer.

3.5. Polymer film stability

Some experiments were carried out to study the stability of the polymer films formed by both PSM and PPM. These experiments showed that this type of polymer (especially the one prepared by the PPM) shows good stability in the dry state at room temperature as well as in the preparing medium for several days without appreciable changes in its electrochemical response, even when used in several experiments. On the other hand, loss of the electrochemical response takes place upon storing the polymer-coated electrode in water which can be explained as due to some film degradation as a result of the deprotonation effect (13, 34).

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Figures caption

Fig. 1 : Polyaniline film growth using the PSM in the potential range between - 0.2 and + 0.8 V (SCE) at a scan rate of 50 mV s^{-1}. (1) 30, (2) 40, (3) 50, (4) 60 and (5) 70 cycles.

Fig. 2 : a) Variation of the oxidation charge, Q, with the number of cycles, N, and the number of pulses, N`.

b) The oxidation charge as a function of polymer film thickness. Films prepared using the PSM and films prepared using thee PPM.

Fig. 3 : a) Cyclic voltammograms of 1 x 10^{-2} M $[Fe(CN)_6]^{3-}$ in sulphate mixture, pH 0.5, on pt-coated polyaniline films grown using the PSM upto different number of cycles : (1) 300, (2) 50, (3) 70, (4) 90 and (5) 110 cycles.

b) Cyclic voltammograms of 1 x 10^{-2} M $[Fe(CN)_6]^{3-}$ in sulphate mixture, pH 0.5, on Pt-coated polyaniline films grown using the PSM upto different number of cycles : (1) 30, (2) 50, (3) 70, (4) 90 and (5) 110 cycles.

Fig. 4 : Variation of the cathodic peak current of $[Fe(CN)_6]^{3-}$ reduction with the polymer film thickness. films prepared using the PSM and films prepared using the PPM.

Fig. 5 : Variation of the cathodic peak current of $[Fe(CN)_6]^{3-}$ reduction with the square root of scan rate. films prepared using the PSM and films prepared using the PPM.

Fig. 6 : Dependence of the ratio I_{pc}/I_{pa} on the bulk concentration of $[Fe(CN)_6]^{3-}$. films prepared using the PSM and films prepared using the PPM.

Fig. 7 : Dependence of the cathodic peak current, Ipc, on the bulk concentration of $[Fe(CN)_6]^{3-}$. films prepared using the PSM and films prepared using the PPM.

قطب البلاتين المغطى بالبولى انيسلين الخواص والاستجابة الكهروكيميائية

ممدوح عبد الرحيم ، أمل على ، وحيد خليل
قسم الكيمياء – كلية العلوم – جامعة القاهرة – الجيزة – مصر

ملخص

تم بلمرة البولى انيسلين على أقطاب البلاتين فى محلول مائى من حمض الكبرتيك ٥ر٠ مولار وتم عمل البلمرة بطريقة جهد المسح وجهد النبض وقد وجد أن أفلام البوليمر المتكونة بهاتين الطريقتين تكون اكثر ثباتا بنقعها فى مخلوط من ١ر٠ مولار كبرتيات الصوديوم + ٥ر٠ مولار حمض كبرتيك ، ولهما تقريباً نفس الخواص الكهروكيميائية التى درست باستخدام الفولتا مترية الدائرية . وقد تم دراست دينامكبية تفاعل الاكسدة – الاختزال لحديدى سيايند على هذه الأقطاب فى محلول من الحمض والكبرتيات معا وقد وجد أن الفيلم المحضر بطريقة النبض أفضل بكثير من المحضر بالطريقة الأخرى .

والتفاعلات على هذه الأفلام وجدت أنها تسير بطريقة انعكاسية تحت تحكم الانتشار . وقد تم تقدير تراكيز حتى ١ × ١٠$^{-٤}$ مولار من حديدى سيا يند على هذه الأفلام .