

# Electrochemical Characteristics and Catalytic Activity of Polyaniline Doped with Ferrocene Perchlorate

Zhang Wen-Zhi, Kan Xian-Wen, Jiao Shou-Feng, Sun Jin-Gao, Yang Dong-Sheng, Fang Bin

School of Chemistry and Materials Science, Anhui Normal University, Wuhu 241000, People's Republic of China

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**ABSTRACT:** A new-doped method based on electrochemical polymerization of aniline in the presence of ferrocene perchlorate and perchloric acid was presented. Polyanilines (PAn and PAnFc) were polymerized in the solution of perchloric acid and the solution containing ferrocene perchlorate and perchloric acid, respectively. Both flame atomic absorption spectrometry and infrared spectrum indicated that ferrocene perchlorate was assured to be doped into the polyaniline. We presumed that ferrocene perchlorate was combined via electrostatic adsorption onto the chain of polyaniline. The effect of pH on the electrochemical characteristics of PAn and PAnFc was studied by cycle voltammetry. Compared with PAn prepared in the absence of ferrocene perchlorate, PAnFc prepared in the presence of ferrocene

perchlorate has a rather high electrochemical activity at pH > 4. The electrochemical oxidation of ascorbic acid (AA) on both PAn film and PAnFc film modified Pt electrode was also performed. The results indicated that the catalytic activity of the PAnFc film to AA was better than that of the PAn film. The anodic peak currents of AA (measured by constant potential amperometry) increased linearly with the concentration of AA in the range of  $8.0 \times 10^{-5}$ – $1.0 \times 10^{-2}$  mol/L. The detection limit (S/N = 3) obtained was  $1.0 \times 10^{-5}$  mol/L. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 102: 5633–5639, 2006

**Key words:** polyaniline; ferrocene perchlorate; doped; electrochemical properties; catalyze; ascorbic acid

## INTRODUCTION

Polyaniline, an inherently conducting polymer, is an important conjugated polymer, since it has good environmental stability and the relative ease in preparation.<sup>1,2</sup> Its conductivity can be reversibly controlled either electrochemical (by oxidation/reduction) or chemical (by protonation/deprotonation).

However, the energy density and the catalytic activity of PAn are limited by the pH value, since PAn has little electrochemical activity at pH > 4; the potential range of the electroactivity for PAn decreases with increasing pH value,<sup>3</sup> and its redox peaks disappear in the cyclic voltammetry for pH > 5. Thus, in general, the redox potential of species to be oxidized and reduced by PAn is within the potential range in which PAn itself is electroactive, restricts its application in bioelectrochemistry, and normally requires a neutral pH environment. To improve the chemical and physical properties of PAn, acidic groups (normally sulfo or carboxyl groups) were introduced into the PAn

chain, to form a so-called "self-doped" PAn, which can maintain its electrochemical activity in neutral or even basic solutions.<sup>4–9</sup> In this case, the inserted ionic groups change the micro-environment of the nitrogen atoms in the PAn chain, thus shifting the local pH.

Ferrocene can be oxidized and reduced reversibly, so it is widely used as an electron transfer mediator. Recently, Mu shaolin's group reported that PAn doped with ferrocene sulfonic<sup>10</sup> acid and ferrocene phosphonic acid<sup>11</sup> by electrochemical polymerization, could shift its electroactivity to pH > 4 and its catalytic effect was much better than that of PAn.

In despite of these great advances in the polymerization of aniline, improving properties of PAn and the study on ferrocene-doped,<sup>12</sup> little is known at present about the effect of ferrocene perchlorate on the electrochemical polymerization of aniline and the characteristics of PAn. Ferrocene perchlorate is a kind of salt. There are few studies about the effect of salt on the electrochemical polymerization of aniline. Our studies have shown that although ferrocene perchlorate, an organic electrolyte,<sup>13</sup> is different from ferrocene sulfonic acid and ferrocene phosphoric acid, it can be also doped within PAn. Ferrocene perchlorate played an important role in increasing the electrochemical polymerization rate of aniline. The result will provide with some significant facts for a further study on the electrochemical polymerization mechanism. In our article, the research emphasized on the

Correspondence to: F. Bin (binfang\_47@yahoo.com.cn).

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application of PAN at electrochemical area. The results indicated that PANFc synthesized in the solution containing ferrocene perchlorate and perchloric acid had a better electrochemical activity at pH 5.6, compared with PAN synthesized in the solution free of ferrocene perchlorate. PANFc synthesized in this manner could effectively catalyze the electrochemical oxidation of AA at pH 6.4. Although the catalytic activity came from PAN itself, ferrocene perchlorate played an important role in enhancing the catalytic activity of polyaniline. The anodic peak currents of AA (measured by constant potential amperometry) increased linearly with the concentration of AA in the range of  $8.0 \times 10^{-5}$ – $1.0 \times 10^{-2}$  mol/L. The detection limit ( $S/N = 3$ ) obtained was  $1.0 \times 10^{-5}$  mol/L.

## EXPERIMENTAL

### Apparatus

A flame atomic absorption spectrometer (WFX-IF2, BSOIF Model) was used to determine iron using an air/acetylene flame; the analytical wavelength of iron is 248.3 nm. FTIR spectra of PAN were measured on pressed pellets with KBr using a Perkin-Elmer 2000 FTIR spectrometer. All electrochemical experiments were performed with a computer-controlled CHI660 electrochemical analyzer (USA) in a conventional three-electrode electrochemical cell using platinum foil ( $4 \times 4 \text{ mm}^2$ ) as the working electrode, platinum wire as the auxiliary electrode, and the saturated calomel electrode as the reference electrode.

### Chemicals and solutions

Aniline (99.5%, Beijing Chemical Regent Company) was distilled before use and stored under highly puri-

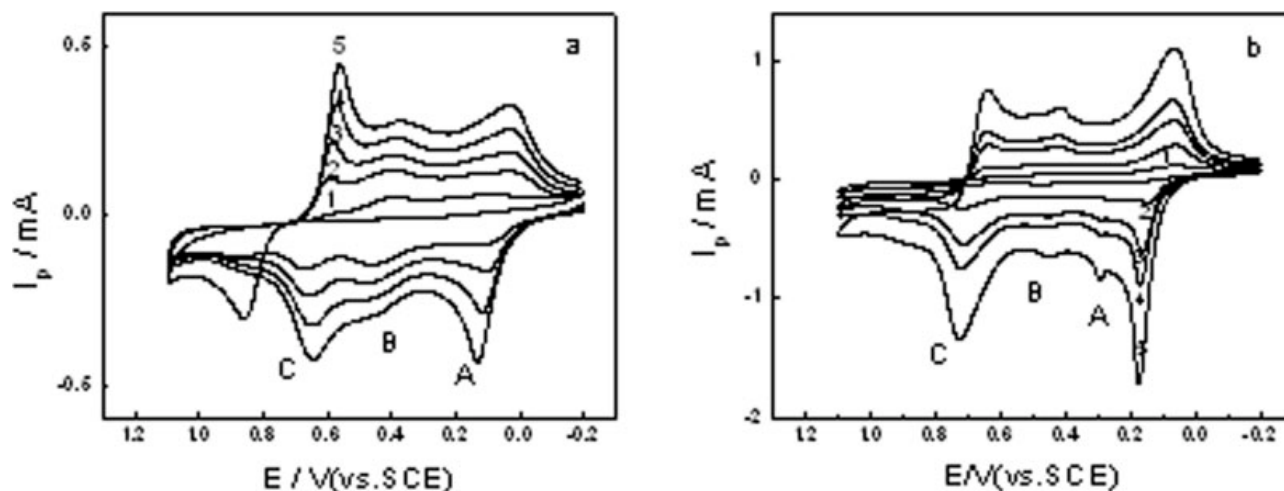
fied nitrogen. Ferrocene perchlorate was obtained from the Organic Chemistry Institute of Anhui Normal University. Ascorbic acid was purchased from Sigma. All other chemicals used in this investigation were of analytical grade. Double distilled water was used to prepare solutions.

All experiments were made at room temperature. The solutions were thoroughly deoxygenated by highly bubbling purified nitrogen and a nitrogen atmosphere was maintained over the solutions.

## RESULTS AND DISCUSSION

### Electrochemical polymerization of aniline in the presence of ferrocene perchlorate

Figure 1(a) shows the cyclic voltammograms of PAN film growth during electrolysis of the solution containing 0.2 mol/L aniline and 0.3 mol/L perchloric acid. The scan potential range is controlled from  $-0.2$  to  $1.0$  V. We can see that an anodic peak for the first scan appears at  $0.86$  V, compared with the result ( $0.91$  V) reported by the literature.<sup>10</sup> Aniline is much easier to be oxidized in this manner. This is likely to be caused by the type and the concentration of protonic acid and aniline. There are three couples of typical redox peak in the voltammogram in the following scans. The polymerization current of aniline increased with increasing the number of potential cycles. This suggested that the films were conductive and also electroactive. According to the literature,<sup>1</sup> the oxidation peaks A and C can be assigned to the oxidation state of PAN in the leucoemeraldine oxidation state to the emeraldine oxidation state, and the further oxidation of the emeraldine oxidation state, respectively. The interpretation of the middle peaks B/B' corresponding to the benzo/hydroquinone (BQ/HQ) is coupled.<sup>14</sup>



**Figure 1** Film growth of PAN (a) and PANFc (b) during electrolysis of the solution (a) 0.2 mol/L aniline and 0.3 mol/L perchloric acid (b) 0.2 mol/L aniline, 0.3 mol/L perchloric acid and 0.7 mol/L ferrocene perchlorate. Curve: (1) first cycle, (2) second cycle, (3) third cycle, (4) fourth cycle, and (5) fifth cycle.

Figure 1(b) shows the cyclic voltammograms of PAN film growth during electrolysis of the solution consisting of 0.2 mol/L aniline, 0.3 mol/L perchloric acid, and 0.7 mol/L ferrocene perchlorate. Compared with Figure 1(a), there are many differences in the shapes of I-E curves. First, at the same first scan cycle, an anodic peak appeared at 0.86 V in Figure 1(a), on the other hand, an anodic and a cathodic peak appeared at 0.80 and 0.20 V accordingly in Figure 1(b). This difference was mainly caused by the catalytic effect of ferrocene perchlorate to the polymerization of aniline. Second, the wave current in Figure 1(b) is much stronger than that in Figure 1(a) from the second scan cycle. This means that the PANFc film grows very quickly in the presence of ferrocene perchlorate. Third, in Figure 1(b), there is a pair of redox peaks about at 0.18 V and the current increases with the number of potential cycles remarkably and sharply. To prove the attribution of this pair of redox peaks, a separate experiment was carried out in the solution containing 0.5 mol/L perchloric acid and 0.7 mol/L ferrocene perchlorate. The cyclic voltammograms obtained showed that a pair of redox peaks also appears around 0.19 V, but the peak potentials and the peak currents were independent of the number of scan cycles (omitted here). As a result, this pair of redox peaks around 0.18 V in Figure 1(b) should be attributed to the redox of ferrocene perchlorate itself. The peak location is very similar to the result reported by the literature.<sup>13</sup>

The above results fully indicated that ferrocene perchlorate was assuredly doped into the PAN and ferrocene perchlorate displayed a very important role in increasing the electrochemical polymerization rate and electroactivity of aniline. We presume that the phenomenon was caused by ferrocene perchlorate which played an important role in the electron transfer. The presence of ferrocene perchlorate in aniline solution accelerated the electrochemical polymerization of aniline. In the Figure 1(b), for the peak around 0.18 V, the anodic peak current was higher than the corresponding cathodic current every scan cycle, consistent with the oxidation of an adsorbed deposit. This result was in good agreement with the literature.<sup>13</sup>

#### Effect of concentration of perchloric acid on electrochemical polymerization of aniline

When the aniline concentration was kept constant and the perchloric acid concentration increased from 0.1 to 0.5 mol/L, and with increasing perchloric acid concentration from 0.1 to 0.3 mol/L, the number of electroactive centers increased. Hence the conductivity of polyaniline increased. But with increasing perchloric acid concentration from 0.3 to 0.5 mol/L, on the contrary, the conductivity of polyaniline decreased. This implied that a saturated conductivity was obtained.

So we chose 0.3 mol/L perchloric acid as medium during the polymerization of aniline.

#### Flame atomic absorption spectrometric determination of iron element in the PANFc film

After dissolving and dealing with the electrode product using a freshly prepared 15% HNO<sub>3</sub>, the following optimum analytical parameters were used for the determination of iron element in PANFc film: wavelength (nm), 248.3; slit width (nm), 0.2; lamp current (mA), 2.5; flame gases: air (L/min), 5.5; acetylene (L/min), 1.5. It was found that the electrode product had a very strong absorption at 248.3 nm. This result indicated that the electrode product contained iron element and ferrocene perchlorate was doped into PAN film.

#### Infrared spectra

The IR spectra of PAN and PANFc were given in Figure 2. In Figure 2(a), we found the characteristic absorption peaks of polyaniline. The presence of sharp peaks at  $\sim 1485\text{ cm}^{-1}$  and  $\sim 1567\text{ cm}^{-1}$  can be attributed to benzenoid ring stretching band and quinoid ring stretching band, respectively. Strong peaks at  $\sim 1301\text{ cm}^{-1}$  for secondary (C—N) stretching band and at  $\sim 1116\text{ cm}^{-1}$  for N=Ar=N mode vibration were also found. Approximately  $814\text{ cm}^{-1}$  can be attributed to out of plane C—H bending vibration.

Compared with the undoped polymer, the electron cloud's density degraded in the molecular chain of polymer doped with ferrocene perchlorate, resulting in the decrease of the force constant among atoms and shifting towards a higher wave number for every adsorption peaks. In Figure 2(b), there were characteristic absorption peaks at  $\sim 480\text{ cm}^{-1}$  and  $\sim 820\text{ cm}^{-1}$  for ferrocene. However, there were no distinct characteristic peaks for ferrocene at  $\sim 1000\text{ cm}^{-1}$  and  $\sim 1100$

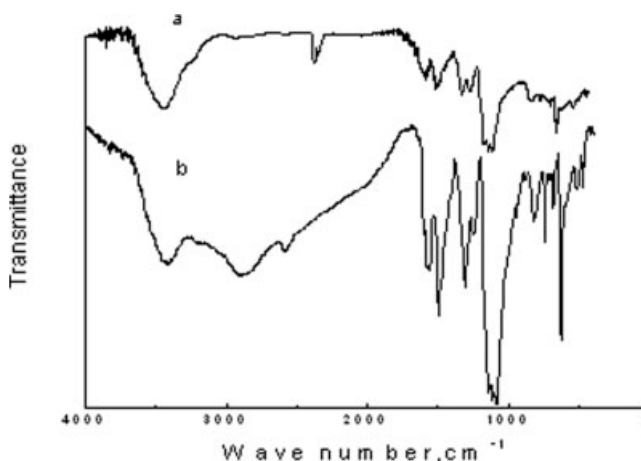


Figure 2 IR spectra of (a) PAN (b) PanFc.

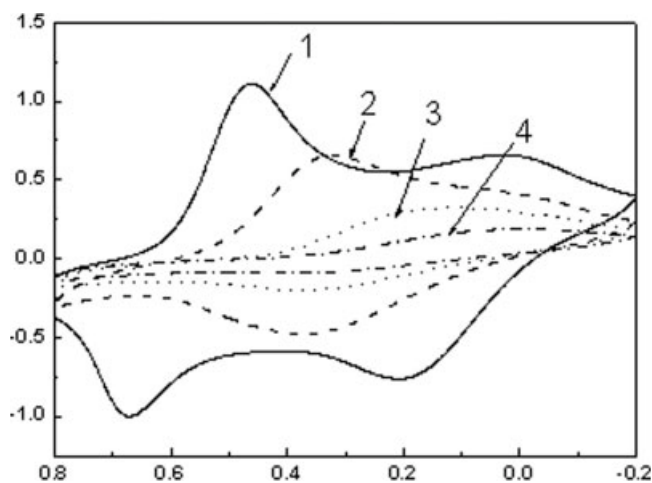
$\text{cm}^{-1}$ . It was likely to be caused by the superposition for aniline and ferrocene at the location. In other words the differences between two spectra lines indicated that ferrocene perchlorate was doped into PAN.

#### Effect of pH on the cyclic voltammograms of PAN and PAnFc film

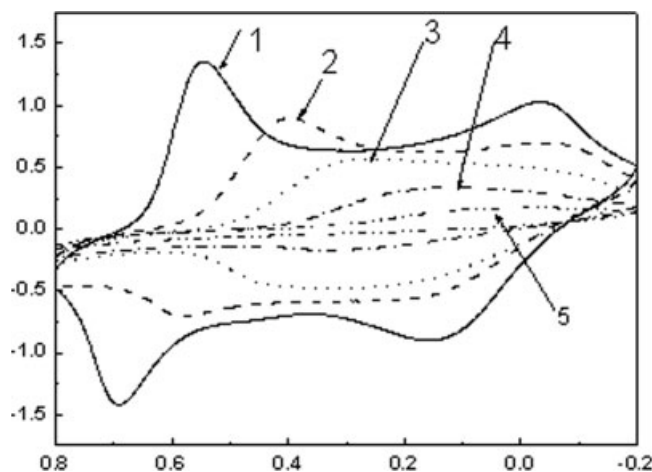
Both PAN and PAnFc films modified Pt electrode were washed thoroughly using perchloric acid solution at pH 3 to remove unreacted aniline and then using doubly distilled water before use in following experiments.

Figure 3 showed the cyclic voltammograms of PAN modified Pt electrode in 0.5 mol/L  $\text{NaClO}_4$  solution under different pH values. The scan rate was set at 60 mV/s. Lines 1, 2, 3, and 4 showed the cyclic voltammograms of PAN at pH 1.0, 2.0, 3.0, and 4.0, respectively. There were two anodic peaks at 0.19 and 0.67 V and two cathodic peaks at 0.027 and 0.46 V on Line 1. When pH value increased from 1.0 to 2.0, there was a pair of redox peak. Compared with Line 1, their peak potentials shifted to the negative direction. This result was in good agreement with the normal polyaniline.<sup>15</sup> At pH 4.0, only a cathodic peak remained on Line 4, and its area of the cyclic voltammogram was much smaller than those of other pH values. This indicated that the electrochemical activity of PAN became very low at pH 4.0.

Figure 4 showed the cyclic voltammograms of PAnFc in 0.5 mol/L  $\text{NaClO}_4$  solution with different pH values from 1.0 to 5.6. The scan rate was also controlled at 60 mV/s. The shape of I-E curve at pH 1.0 was very similar to that Line 1 in Figure 3. However, at pH 2.0, there were still two pairs of redox peaks (Line 2 in Fig. 4). This was much different from the



**Figure 3** Cyclic voltammograms of PAN modified Pt electrode in 0.5 mol/L  $\text{NaClO}_4$  with different pH values. Scan rate: 60 mV/s (1) pH 1.0, (2) pH 2.0, (3) pH 3.0, and (4) pH 4.0.



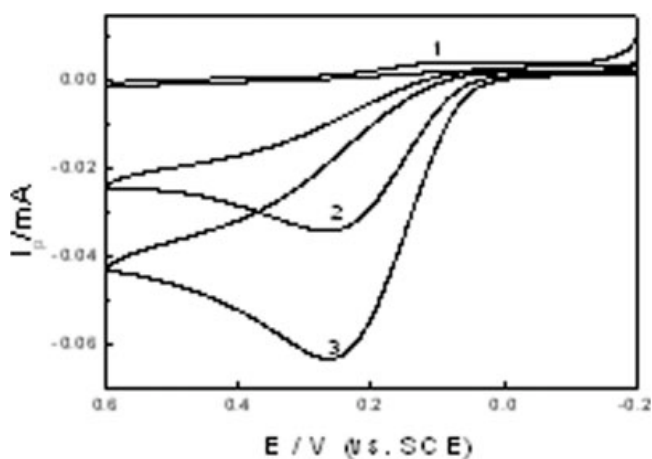
**Figure 4** Cyclic voltammograms of PAnFc modified Pt electrode in 0.5 mol/L  $\text{NaClO}_4$  solution with different pH values. The scan rate: 60 mV/s (1) pH 1.0, (2) pH 2.0, (3) pH 3.0, (4) pH 4.0, and (5) pH 5.6.

cyclic voltammograms of PAN film at pH 2.0 (Line 2 in Fig. 3). There was a pair of redox peak at pH 3.0 and 4.0, respectively, for PAnFc film modified Pt electrode. Although the case was the same as that of PAN, the areas of cyclic voltammograms of PAnFc were much bigger than that of PAN film. Also, at pH 5.6, there was still a cathodic peak for PAnFc film. This indicated that PAnFc still had a rather high electrochemical activity at pH 5.6. The results obtained by us were comparable with the result obtained by Mu shaolin's group.<sup>10</sup> Thus, the electrochemical activity of PAnFc at higher pH values was improved compared with PAN. This was caused by ferrocene perchlorate doped into polyaniline. In a word, this study demonstrated that the doping of ferrocene perchlorate into PAN via an electrochemical procedure could extend the electroactivity to pH 5.6 of PAN and widen a more broad application range of PAN in electrochemical area.

The three kinds of solutions, i.e. solution A–C, were used in the following electrochemical experiments. Solution A was 0.2 mol/L acetate buffer solution; Solution B consisted of  $2 \times 10^{-3}$  mol/L AA and 0.2 mol/L acetate buffer solution; Solution C consisted of  $4 \times 10^{-3}$  mol/L AA and 0.2 mol/L acetate buffer solution.

#### Electrochemical oxidation of AA at the bare Pt electrode

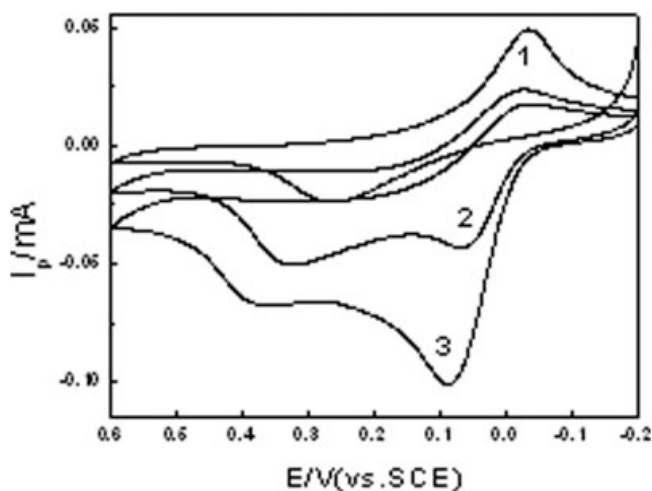
Lines 1–3 in Figure 5 showed the cyclic voltammograms of solution A–C, respectively, at the bare Pt electrode. The pH value of the solution was 6.4. The scan rate was set at 6 mV/s. It was clear that the anodic peak current increased with increasing the concentration of AA. Therefore, the anodic peak at 0.28 V on Lines 2 and 3 were caused by the electrochemical oxidation of AA.



**Figure 5** Cyclic voltammograms of different concentration of AA at bare Pt electrode, pH 6.4. The scan rate, 6 mV/s. (1) 0 mol/L, (2)  $2 \times 10^{-3}$  mol/L, (3)  $4 \times 10^{-3}$  mol/L.

#### Electrochemical oxidation of AA at PAN film modified Pt electrode

To investigate the electrochemical oxidation of AA on PAN film modified Pt electrode, the cyclic voltammetry was performed under the same solution condition (pH 6.4) as that in Figure 5. But the redox peak of PAN wasn't found. This was because there wasn't electrochemical activity for PAN at the pH 6.4, consistent with the previous result in the article. So the pH 5.0 acetate buffer solution was chosen to study the oxidation of AA on the PAN film modified Pt electrode. Lines 1–3 in Figure 6 were the cyclic voltammograms of solutions A–C, respectively, at the PAN modified Pt electrode. The pH and scan rate were 5.0 and 6 mV/s, respectively. A shoulder peak at about 0.3 V in Figure 6 was attributed to the oxidation peak of polyaniline. The anodic peak current at 0.08 V on Lines 2 and 3 in



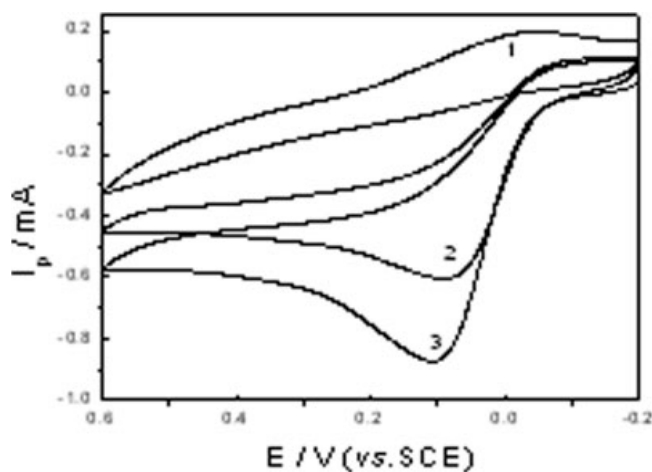
**Figure 6** Cyclic voltammograms of different concentration of AA at PAN modified Pt electrode, pH 5.0. The scan rate, 6 mV/s. (1) 0 mol/L, (2)  $2 \times 10^{-3}$  mol/L, (3)  $4 \times 10^{-3}$  mol/L.

Figure 6 increased with increasing the concentration of AA, so the anodic peak should be caused by the oxidation of AA. These currents were two times as high as that on the bare Pt electrode, and the oxidation potential of AA at the PAN film electrode was lower about 200 mV than that on the bare Pt electrode at pH 6.4. These were strong evidences for the electrocatalytic oxidation of AA.

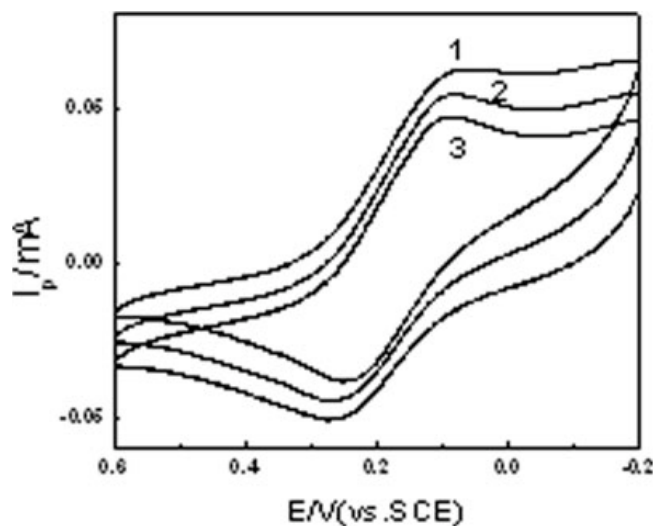
#### Electrochemical oxidation of AA at PANFc film modified Pt electrode

Lines 1–3 in Figure 7 were the cyclic voltammograms of solution A–C, respectively, at PANFc film modified Pt electrode. The pH value of solution was 6.4. It appeared an anodic peak on Lines 2 and 3, respectively. The peak potential was 0.09 V on Line 2 and 0.098 V on Line 3. The peak current on Line 2 is higher than that on Line 1, and increases with increasing the concentration of AA. So the anodic peaks on Lines 2 and 3 were caused by the electrochemical oxidation of AA on the PANFc film electrode. The oxidation current of AA on the PANFc electrode was 15.3–16.7 times as high as those on the bare Pt electrode in the same solution, respectively. Compared with the PAN film electrode, the location of oxidation potential of AA was similar to that on PANFc film modified Pt electrode. However, the currents of AA on the PANFc film electrode were higher than those on the PAN film electrode at the same condition. Thus, the PANFc film electrode can more effectively catalyze the electrochemical oxidation of AA than the PAN film electrode.

According to above experimental results, we found that both PAN film electrode and PANFc film electrode can effectively catalyze the oxidation of AA. The question was whether the catalytic effect arose from PAN or ferrocene perchlorate, which was doped into the



**Figure 7** Cyclic voltammograms of different concentration of AA at PANFc modified Pt electrode, pH 6.4. The scan rate, 6 mV/s. (1) 0 mol/L, (2)  $2 \times 10^{-3}$  mol/L, (3)  $4 \times 10^{-3}$  mol/L.



**Figure 8** Cyclic voltammograms of different concentration of AA at bare Pt electrode in 0.2 mol/L acetate, pH 6.4. The scan rate, 6 mV/s. (1)  $5 \times 10^{-3}$  mol/L ferrocene perchlorate, (2)  $5 \times 10^{-3}$  mol/L ferrocene perchlorate and  $2 \times 10^{-3}$  mol/L AA, (3)  $5 \times 10^{-3}$  mol/L ferrocene perchlorate and  $4 \times 10^{-3}$  mol/L AA.

PAnFc film, since both of them can catalyze some species. For this purpose, a separate experiment was carried out using a bare Pt electrode in the acetate buffer solution containing ferrocene perchlorate and AA. The pH and scan rate were 6.4 and 6 mV/s, respectively.

Lines 1–3 in Figure 8 showed the cyclic voltammograms of the bare Pt electrode in the solution consisting of  $0.2 \times 10^{-3}$  mol/L acetate buffer solution and  $5 \times 10^{-3}$  mol/L ferrocene perchlorate, the same solution with 2 and  $4 \times 10^{-3}$  mol/L AA, respectively. There were an anodic peak at 0.22 V and a cathodic peak at 0.03 V on Line 1 in Figure 8. It was clear that this pair

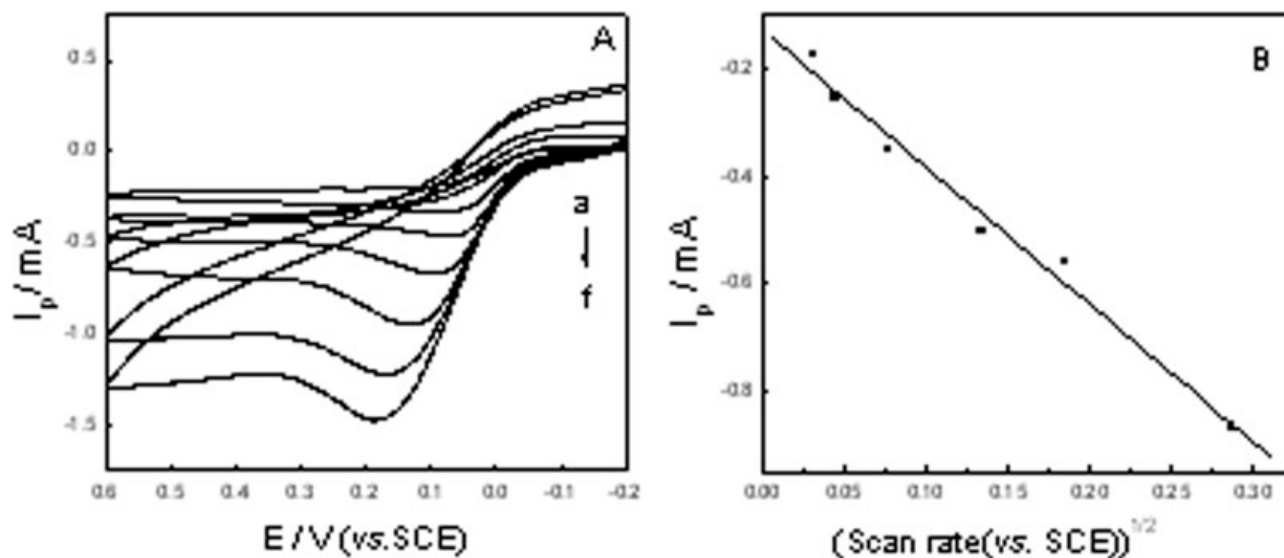
of redox peak was attributed to ferrocene perchlorate. After adding to  $2 \times 10^{-3}$ – $4 \times 10^{-3}$  mol/L AA, however, the oxidation peak of AA wasn't observed (Lines 2 and 3). Compared with Line 1, there wasn't an obvious increase along with the concentration increase of AA for the redox current on lines 2 and 3. So it concluded that ferrocene perchlorate had no catalytic effect to the oxidation of AA. Thus, the catalytic effect to the oxidation of AA arose from PAN itself, and ferrocene perchlorate doped into PAN only played a role in enhancing the electrochemical and catalytic activities of PAN.

#### Effect of scan rate on the peak currents of AA on PAnFc film modified Pt electrode

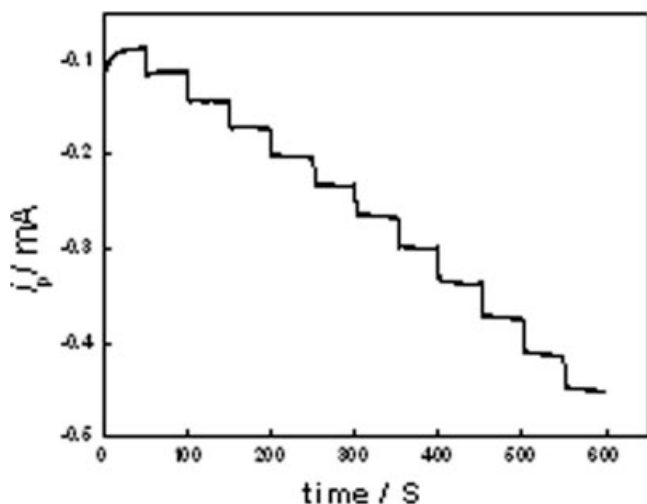
The dependence of oxidation peak currents of AA on the scan rate was showed in Figure 9. It can be observed that the peak current ( $i_p$ ) increased with scan rate ( $v^{1/2}$ ) from 0.01 to 0.10 V/s. According to the  $i_p$ - $v^{1/2}$  curve, the current increased linearly with square root of the scan rate, from which we deduced that the electrode reaction was diffusion controlled.<sup>16</sup>

#### The determination of AA at PAnFc film modified Pt electrode

To examine the response character of modified electrode to AA, we have undertaken the detection of AA. Figure 10 showed a typical amperogram obtained at the modified electrode, which obviously showed an increase in current measured with each addition of  $2 \times 10^{-4}$  mol/L of AA. According to this experiment, a linear relationship between anodic current of AA and its concentration was obtained in the range of  $8.0 \times 10^{-5}$ – $1.0 \times 10^{-2}$  mol/L. The regression equation was



**Figure 9** (A) Cyclic voltammograms of PAnFc modified Pt electrode at various scan rates in pH 6.4 acetate: (a) 0.001, (b) 0.002, (c) 0.006, (d) 0.018, (e) 0.034, (f) 0.082 (V/s). (B) Dependence of anodic currents on the square root of the scan rate.



**Figure 10** Amperometric detection of AA at PANFc film modified Pt electrode in pH 6.4 acetate, each addition of  $0.5 \times 10^{-3}$  mol/L (alternating current was measured at constant potential of 0.19 V modulated with pulse 50 mV in time of 0.5 s in a stirred system).

$I_{pa} = 0.056 C + 1.857 (I_{pa}, \text{mA}, C, \text{mol/L})$ , with the correlation coefficient of 0.9982. The detection limit (three times the blank/slope) was  $1.0 \times 10^{-5}$  mol/L. The electrode modified with PANFc film has a potential application to detect AA.

The stability of the PANFc film modified Pt electrode was also tested. When the electrode was stored in pH 6.4 acetate buffer solution at 4°C for 1 month, the peak current and potential were stable. Even if the electrode was dried and stored in air, its peak only slightly decreases during 1 week.

## CONCLUSIONS

This work demonstrates the electrochemical characteristics of polyaniline prepared by electrochemical

polymerization in the absence and presence of ferrocene perchlorate. Flame atomic absorption spectrometry and FTIR spectrum indicated that ferrocene perchlorate was doped into PAN. We presumed that ferrocene perchlorate was doped into PAN via electrostatic adsorption. The cyclic voltammograms displayed that PANFc film had a better electrochemical activity in high pH value than PAN. PANFc film modified platinum electrode can effectively catalyze the oxidation of AA and be applied to determine AA. Ferrocene perchlorate plays an important role in enhancing the electrochemical and catalytic activity.

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