Electrochemical Polymerization of *p*-Terphenyl in Mixed Electrolyte of Boron Trifluoride Diethyl Etherate and CH₂Cl₂

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ABSTRACT: High quality poly(*p*-phenylene) (PPP) film with conductivity of 0.015 S cm^{-1} was synthesized electrochemically by direct anodic oxidation of *p*-terphenyl (PP) oligomers in boron trifluoride diethyl etherate (BFEE) containing 37.5% CH$_2$Cl$_2$ (v/v). The oxidation onset potential of PP in this medium was measured to be only 1.23 V vs. saturated calomel electrode (SCE), which was lower than that determined in CH$_2$Cl$_2$ + 0.1 mol L^{-1} Bu$_4$NBF$_4$ (1.87 V vs. SCE). As-formed PPP films showed good electrochemical behavior, good electrochromic property and good thermal stability. The structures and morphology of doped and dedoped PPP were investigated by UV-vis, FTIR, and Scanning electron micrographs. The infrared spectroscopic measurements for the estimation of chain lengths revealed that PPP was composed of about 10 phenyl rings. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 117: 2688–2694, 2010

Key words: conducting polymers; boron trifluoride diethyl etherate; electrochemical polymerization; *p*-terphenyl; poly(*p*-phenylene)

INTRODUCTION

Inherently conducting polymers have attracted great attention due to their wide fundamental interest and potential industrial applications.[1–4] Among them, poly(*p*-phenylene) (PPP) films have been widely investigated for applications of the active layers in electroluminescence because they have superior mechanical properties, nongenerated ground state, moderate electrical conductivity, high thermal stability.[5–8] And they have been proposed as an electrode material for rechargeable batteries.[9,10]

PPP has been synthesized from benzene and its derivatives by using various methods.[10–22] In the early stages of research, however, it was not easy to make a PPP film for its characterization and application because PPP powders synthesized chemically were not soluble in most organic solvents, and electrochemical synthesis of PPP using benzene as a monomer also required harsh experimental conditions.[13–15] Although there were some reports on the electrochemical preparations of PPP under mild experimental conditions by using longer chain oligomers as starting reactant,[10–12,19–21] the properties of the PPP films were greatly dependent on the experimental conditions such as monomers, solvents, supporting electrolytes, and applied potentials and their ranges. As one of the benzene oligomers, there were several reports on the electrochemical polymerization of *p*-terphenyl (PP) in a rigorously dried CH$_2$Cl$_2$ solution with tetrabutylammonium tetrafluoroborate as an electrolyte.[19,21] However, the main disadvantage of these media was that the oxidation onset potential of PP was too high [about 1.87 V vs. saturated calomel electrode (SCE)], which was not useful for the preparation of high quality PPP from PP as monomer. Up to now, there was no report on the low-potential electrochemical polymerization of PP.

Boron trifluoride diethyl etherate (BFEE) was found to be a good electrolyte for electrochemical polymerization of aromatic compounds, such as thiophene and its derivatives.[4,23–28] Utilizing BFEE, the oxidation onset potentials of aromatic monomers could be considerably lowered. Under these circumstances, BFEE served not only as the solvent but also

as the supporting electrolyte and no other supporting electrolyte was needed. And the decrease of the oxidation potential during the electrochemical polymerization also had many advantages as following: (1) It could increase the activity of α-position of monomer, decrease the possibility of side reaction, avoid the over-oxidation of polymer and decrease the cross-linking between chains. It was useful to improve the conductivity and polymeric degree of polymer. (2) It could decrease the activity of cation radical duing the polymerization, which made the polymerization occur favorably under a kindly condition. (3) It could reduce cost because many metals (such as aluminium, zinc, stainless steel, etc.), which were partial oxidation dissolution at high potential, might be used as anode material at low-potential in the electro-polymerization experiment. The PPP fibers with the best quality could be obtained from benzene as monomer only by using the BFEE solution containing about 5 mM water as the electrolyte.[29] Moreover, BFEE containing additional sulfuric acid or trifluoroacetic acid was used for the electrochemical polymerization of benzene and high quality free-standing PPP films with high tensile strength could be also easily produced.[30,31] As far as we know, there were no reports on the electro-polymerization of PP in BFEE based electrolytes.

In this work, the electrolyte of BFEE + 37.5% CH_2Cl_2 was used for the electrochemical polymerization of PP. And high quality PPP films could be easily electrodeposited by direct anodic oxidation of PP on stainless steel electrode. The electrochemical properties, spectroscopic properties, thermal stability and morphology of as-formed PPP films were studied in detail.

EXPERIMENTAL

Materials

P-terphenyl (PP, Alfa Aesar) was used directly without further purification. Boron trifluoride diethyl etherate (BFEE, Beijing Changyang Chemical Plant) was distilled and stored at −20°C before use. Bu$_4$NBF$_4$ (Acros Organics, 95 %) was dried in vacuum at 60°C for 24 h before use. CH_2Cl_2 and 25% ammonia (made by Jinan Chemical Reagent Company) were used as received without further purification.

Electrosyntheses of PPP films

Electrochemical syntheses and examinations were performed in a one-compartment cell with the use of LK2006 potentiostat-galvanostat (Tianjin Lanlike Chemical Electronic Technology, China) under computer control. For electrochemical examinations, the working and counter electrodes were Pt wire with a diameter of 0.5 mm and stainless steel wire with diameter of 1 mm, respectively, and the two electrodes were placed 5 mm apart. To obtain a sufficient amount of polymer for characterization, stainless steel sheets with a surface area of 2 and 2.5 cm^2 each were employed as the working and counter electrodes, respectively. Electrodes mentioned above were carefully polished with abrasive paper (1500 mesh), and cleaned by water and acetone successively before each examination. All potentials were referred to a SCE.

The typical electrolytic solution was BFEE + 37.5% CH_2Cl_2 and 5 mmol L^{-1} PP. All solutions were deaerated by a dry argon stream and maintained at a slight argon overpressure during experiments. The amount of polymer deposited on the electrode was controlled by the integrated charge passed through the cell. To remove the electrolyte and monomer, the electropolymerized films were rinsed by CH_2Cl_2 and water. As-formed PPP film was in doped state and metallic dark in color. The polymers were deposited on an indium-tin-oxide (ITO) coated glass for UV-visible spectral measurements. For IR spectral analysis, the polymer was dedoped with 25% ammonia for 3 days then washed repeatedly with water. Finally, it was dried under vacuum at 60°C for 24 h.

Characterizations

The conductivity of polymer film was measured by conventional four-probe technique with pressed pellets of fresh samples and the pellets thicknesses were determined with a TT260 (Time Group, China) thickness monitor. UV-visible spectra were taken by using Perkin–Elmer Lambda 900 UV-vis-NIR spectrophotometer. Infrared spectra were recorded using Bruker Vertex 70 FTIR spectrometer with KBr pellets. The thermogravimetric analysis (TGA) was performed with a thermal analyzer of TA Q50. Scanning electron micrographs (SEM) were taken out by the use of S-4700 (Japan) scanning electron microscope.

RESULTS AND DISCUSSION

Electrochemical syntheses of PPP films

Generally, high quality conducting polymer films could be electrodeposited from pure BFEE. Thus, pure BFEE was the first choice as the electrolyte. However, the solubility of PP in pure BFEE was very poor. On the other hand, CH_2Cl_2 was tested to be a fairly good solvent for the PP. It was also reported that the free-standing stable film was synthesized via potentiostatic and potentiodynamic methods in CH_2Cl_2 + BFEE solvent mixture.[32]

Therefore, BFEE + CH_2Cl_2 were tested as the solvent and supporting electrolyte for the electro-polymerization of PP. It was found that the 5 mmol L^{-1} PP can be thoroughly soluble in BFEE + 37.5% CH_2Cl_2 and high quality polymer films could be electrodeposited during the experiment.

Figure 1 showed the anodic polymerization curves of PP in BFEE + 37.5% CH_2Cl_2 (A) and in CH_2Cl_2 + 0.1 mol L^{-1} Bu_4NBF_4 (B) on Pt electrode at potential scan rates of 20 mV s^{-1}. The oxidation onset potential of PP was initiated at 1.23 V vs. SCE in BFEE + 37.5% CH_2Cl_2 [Fig. 1(A)], which was much lower than that of PP in CH_2Cl_2 + 0.1 mol L^{-1} Bu_4NBF_4 [1.87 V vs. SCE, Fig. 1(B)]. This result indicated the oxidation of PP in BFEE + 37.5% CH_2Cl_2 was much easier than that in CH_2Cl_2 + 0.1 mol L^{-1} Bu_4NBF_4. It should be noted here that mixed electrolyte was electrochemically inert in the whole potential range.[23]

The successive cyclic voltammograms (CVs) of 5 mmol L^{-1} PP in BFEE + 37.5% CH_2Cl_2 on a Pt wire electrode was shown in Figure 2(A). As seen from this Figure, the CVs of PP in BFEE + 37.5% CH_2Cl_2 showed characteristic features as other conducting polymers such as polythiophene, polypyrrole during potentiodynamic syntheses. There were two obvious reduction peaks near 1.07 V and 0.85 V in the first cycle for the CVs [Fig. 2(A)]. In the second cycle, an inconspicuous peak near 1.10 V in oxidized cycle formed and the two reduction peaks shifted negatively to 0.75 V and 0.97 V, respectively. This pair of peaks was assigned to the transformation from quinoid structure to aromatic state.[33] As the CV scan continues, a polymer film was also formed on the working electrode surface. In BFEE + 37.5% CH_2Cl_2, PPP could be reduced and oxidized between 0.5 and 1.3 V vs. SCE [Fig. 2(A)]. The increase in the redox wave currents implied that the amount of the polymer on the electrode was increasing. The potential shift of the wave current maximum provides information about the increase of the electrical resistance in the polymer film and the over-potential needed to overcome the resistance.[34]

On the other hand, the successive CVs of PP in CH_2Cl_2 + 0.1 mol L^{-1} Bu_4NBF_4 was not very successful and only a trace amount of the polymer can be formed on the working electrode [Fig. 2(B)]. The PPP film prepared from CH_2Cl_2 could be reduced and oxidized between 1.6 and 2.0 V. The high redox potential generally resulted in the conductivities of PPP films unstable.[18] From this point of view, the introduction of BFEE into CH_2Cl_2 was useful to the electrochemical polymerization of PP.

Electrochemistry of PPP films

To get a deeper insight into the electrochemical and environmental stabilities of as-formed PPP films, the
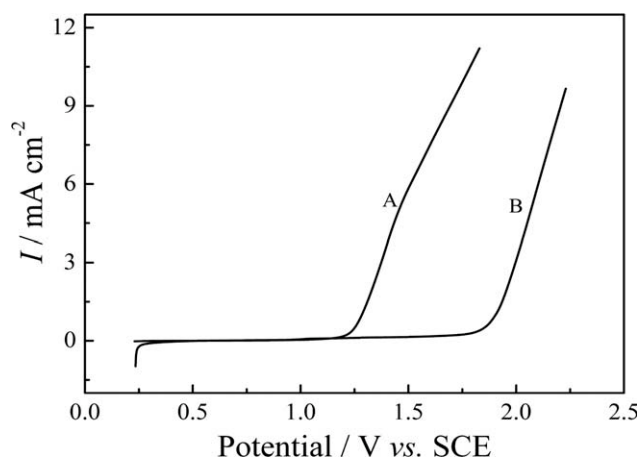

Figure 1 Anodic polarization curves of 5 mmol L^{-1} PP in BFEE + 37.5% CH_2Cl_2 (A), and in CH_2Cl_2 + 0.1 mol L^{-1} Bu_4NBF_4 (B) on Pt electrode. Potential scan rates: 20 mV s^{-1}.

electrochemical behavior of the PPP films deposited by potentiodynamic method at potential of 0–1.53 V vs. SCE on Pt wire electrode from BFEE + 37.5% CH_2Cl_2 was studied in pure BFEE (Fig. 3) and in concentrated sulfuric acid (Fig. 4), respectively. Similar to the results in the literature,[35] the steady-state cyclic voltammograms represented broad anodic and cathodic peaks. The peak current densities, I_{pa} and I_{pc}, were both proportional to the scan rates, which indicated that a well adhered film on the electrode surface and the process was not diffusion controlled (inset of Figs. 3 and 4). Furthermore, PPP films could be cycled repeatedly between the conducting (oxidized) and insulating (neutral) states without significant decomposition of the materials, indicating high stability of the polymer. The polymer film obtained from BFEE + 37.5% CH_2Cl_2 could be oxidized and reduced from 1.31 V (Ea) to 1.02 V (Ec) in monomer-free BFEE at a potential scan rate of 20 mV s^{-1}. With the scan rates increasing, the potentials needed to oxidize and reduce the polymer film shifted positively and negatively to about 1.50V (Ea) and 0.71V (Ec), respectively. This could be mainly ascribed to the slow transfer rate of large doping anions and solvent molecules in BFEE, such as $(EtO)_3BF_3^-$, especially at higher scan rates. PPP films obtained from BFEE + 37.5% CH_2Cl_2 showed almost similar behavior in concentrated sulfuric acid (Fig. 4), which can be oxidized and reduced from 0.80 V (Ea) to 0.74 V (Ec) at a potential scan rate of 25 mV s^{-1}. The difference of (Ea–Ec) related to the kinetics of the doping–dedoping reactions was equal to 0.06 V in concentrated sulfuric acid at a potential scan rate of 25 mV s^{-1}. This implied that the redox properties of PPP films were fairly good in concentrated sulfuric acid. All these results indicated that as-formed PPP films had good redox properties and stabilities.
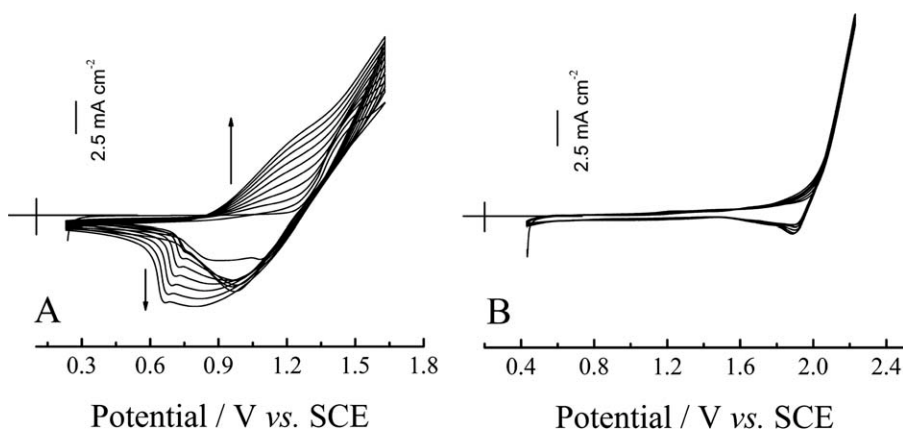
Figure 2 Cyclic voltammograms of 5 mmol L^{-1} PP in BFEE + 37.5% CH_2Cl_2 (A), and in CH_2Cl_2 + 0.1 mol L^{-1} Bu_4NBF_4 (B) on Pt electrode. Potential scan rates: 100 mV s^{-1}.

Structural characterizations

Conducting polymer films could be prepared by potentiostatic, galvanostatic, or potentiodynamic methods. Usually, high quality polymer films could be prepared by the potentiostatic method, such as polythiophenes,[23] polyindole and its derivatives,[36–38] etc. Previous studies indicated that a constant applied potential was higher (0.2–0.3 V) than the oxidation onset potential of monomers by potentiostatic method could lead to high quality polymer film formation.[29] Therefore, PPP films were electrosynthesized by potentiostatic approach. Through this method, PPP film was formed on ITO or stainless steel electrode surface through potentiostatic at 1.43 V vs. SCE in BFEE + 37.5% CH_2Cl_2.

The PPP films were insoluble in general solvent. Therefore, the UV-visible spectra of PP (A) in CH_2Cl_2, doped (B) and dedoped PPP (C) prepared potentiostatically at 1.43 V vs. SCE from electrolyte of BFEE + 37.5% CH_2Cl_2 on ITO were examined, as shown in Figure 5. As seen from Figure 5, the spectra of polymer showed a broader absorption band than that of the monomer. The longer wavelength in the spectra usually indicated a longer polymer sequence. Therefore, these spectral results confirmed a conjugated polymer formation with a broad molar mass distribution.[30] The absorption spectrum of doped PPP [Fig. 5(B)] showed a strong band at 304, a shoulder at about 380 nm, and a strong broad absorption at ca. 836 nm, which could be assigned to the absorption of polaron on the main backbone of PPP in the doped state.[39,40] For the spectrum of dedoped PPP [Fig. 5(C)], the dedoped PPP showed only one peak at 318 nm due to the π–π* transition. On the other hand, doped PPP was red-brown,
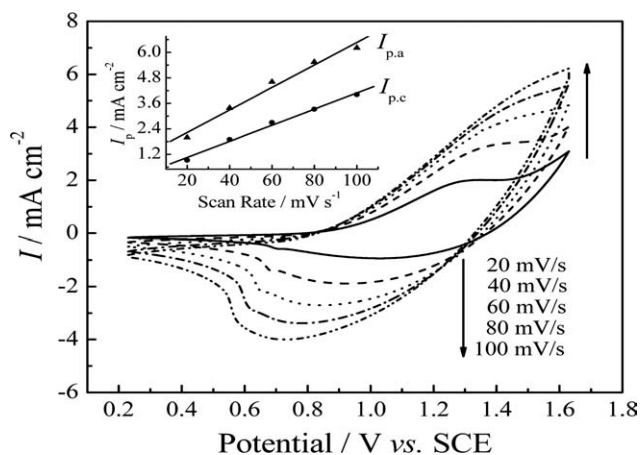

Figure 3 Cyclic voltammograms of a PPP film prepared by potentiodynamic method at potential of 0–1.53 V vs. SCE on Pt electrode in monomer-free BFEE at potential scan rates of 20 mV s^{-1}, 40 mV s^{-1}, 60 mV s^{-1}, 80 mV s^{-1}, 100 mV s^{-1}, respectively. Inset: Plot of anodic (I_{pa}) and cathodic (I_{pc}) peak current vs scan rate for PPP.
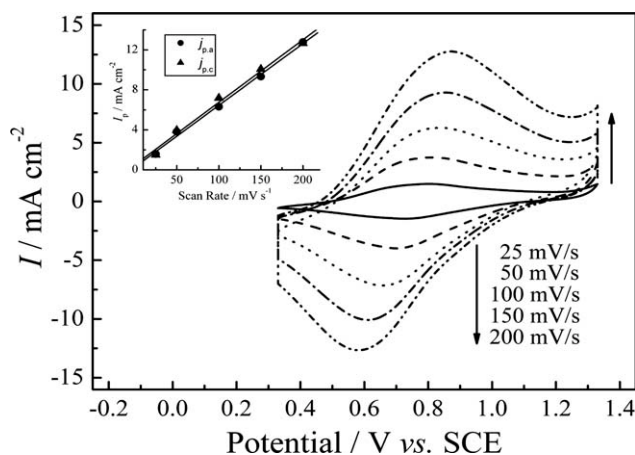

Figure 4 Cyclic voltammograms of a PPP film prepared by potentiodynamic method at potential of 0–1.53 V vs. SCE on Pt electrode in concentrated sulfuric acid at potential scan rates of 25 mV s^{-1}, 50 mV s^{-1}, 100 mV s^{-1}, 150 mV s^{-1}, 200 mV s^{-1}, respectively. Inset: Plot of anodic (I_{pa}) and cathodic (I_{pc}) peak current vs. scan rate for PPP.
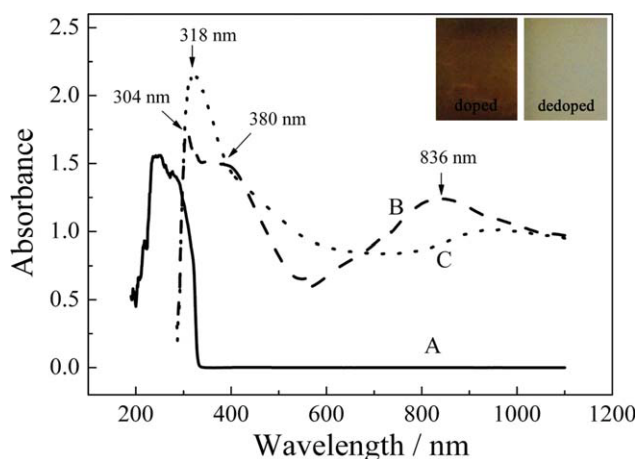
Figure 5 UV-visible spectra of PP (A) in CH_2Cl_2, doped PPP (B) and dedoped PPP (C) prepared potentiostatically at 1.43 V vs. SCE from electrolyte of BFEE + 37.5% CH_2Cl_2 on ITO. Inset: the electrochromic of PPP films prepared from PP electrochemically from BFEE + 37.5% CH_2Cl_2 on ITO. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

while the color of dedoped PPP changed almost to achromatism (Inset of Fig. 5). The color of the doped PPP changed slowly to dark with the increase of thickness. This indicated that the PPP film was a good electrochromic polymer.

The typical transmittance IR spectra of PP, dedoped and doped PPP film were tested. In the spectra of doped PPP, the band at around 1085 cm^{-1} was due to the existence of the dopant $(EtO)_3BF_3^-$ counter-anion on the main backbone of polymer in the doped state, which was disappeared in dedoped PPP film. The characteristic absorptions of PPP were located around 650–900 cm^{-1}. The band at 816 cm^{-1} was characteristic of the C—H out-of-plane vibration for adjacent hydrogen atoms on *para*-disubstituted rings, which was smaller than that of PP oligomers (839 cm^{-1}). This was due to the C—H out-of-plane vibration of *para*-disubstituted polyphenylenes shifted towards lower wave numbers and its intensity increased when the number of phenyl groups increased in the PPP chain.[11] The bands located between 693 and 762 cm^{-1} were typical of C—H out-of-plane deformation vibrations of the mono-substituted benzene ring (the terminal phenyl ring).[11] The band at 1001 cm^{-1} was attributed to the aromatic hydrogen in-plane bending vibration, which also indicated a *para*-substitution.[41] The bands located at 1603, 1481 cm^{-1} were assigned to the stretching and shrinking modes of the C=C and C—C. Furthermore, in this spectrum no strong absorptions at 840 and 860 cm^{-1} were detected, excluding trisubstituted and tetrasubstituted benzene respectively. These results agreed well with those reported for chemically or electrochemically synthesized poly(*para*-phenylene),[29,42,43] and indicated that the principal

structure of the PPP polymer was not cross-linking. In conclusion, the PPP films obtained in this medium had a high regularity (linear structure). On the other hand, the chain lengths of PPP could be estimated by IR spectroscopy.[11,18,21] PPP prepared in this paper had an average chain length of about 10.
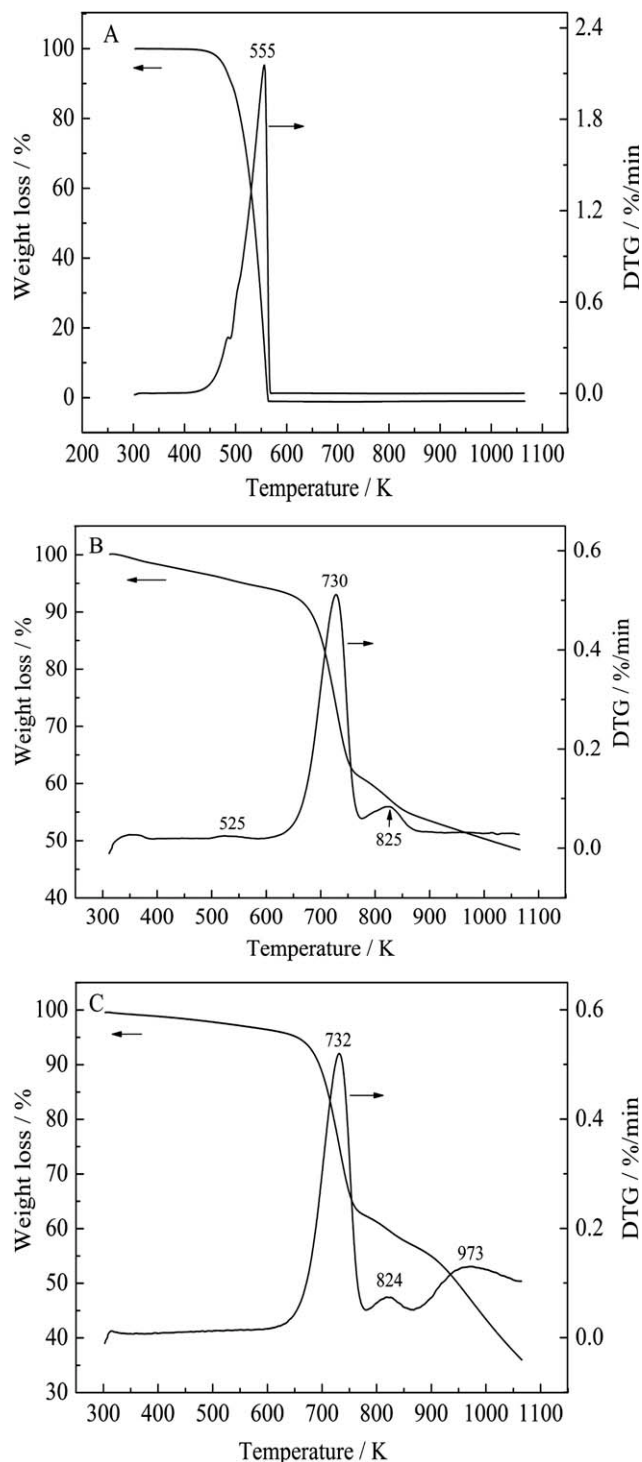

Figure 6 TGA and DTG curves of PP (A), doped (B), and dedoped (C) PPP films obtained potentiostatically at 1.43 V vs. SCE from BFEE + 37.5% CH_2Cl_2 on SS electrode.
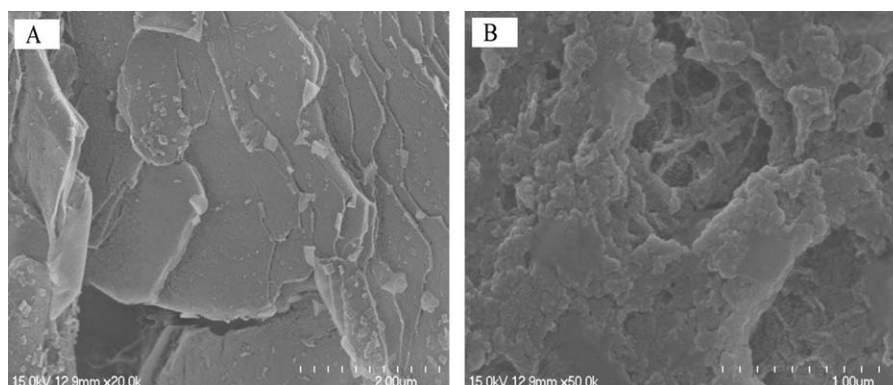
Figure 7 SEM micrographs of a doped PPP (A) and dedoped PPP (B) film deposited on the electrode surface from BFEE + 37.5% CH$_2$Cl$_2$ at a constant applied potential of 1.43 V vs. SCE.

Thermal analysis

The thermal stability of conducting polymers is very important for their application. TGA is a significant dynamic way to detect the degradation behaviors. The weight loss of a polymer sample is measured continuously, whereas the temperature is changed at a constant rate. Figure 6 showed the TGA and DTG curves of PP (A), doped (B) and dedoped (C) PPP. As shown in Figure 6(A), TGA curve of PP showed single weight loss at 555 K. The TGA curve for doped PPP showed three transitions. The first transition at about 525 K was due to the presence of moisture and dopant anions in the polymer. The prominent second transition at about 730 K and the third transition at about 825 K were attributed to the rupture of the polymer backbone. In the Figure 6(C), it could be seen that when the temperature was 732, 824, and 973 K, the weightloss rate of PPP films was fastest. Moreover, the char residue of doped PPP and dedoped PPP was 48.4% and 36% after 1065 K, respectively [Fig. 6(B,C)]. However, PP disappeared completely after 566 K. These results indicated very good thermal stability for the PPP films obtained from BFEE + 37.5% CH$_2$Cl$_2$.

Morphology and conductivity

As-formed PPP films prepared in BFEE + 37.5% CH$_2$Cl$_2$ could not be peeled off from the electrode into free-standing state. The SEM images of a PPP film were shown in Figure 7. Macroscopically, in both the doped and the dedoped states, as-formed PPP films on ITO electrodes were flat, homogeneous and compact. Microscopically, the doped PPP film consisted of the ordered, smooth, and compact pieces. This structure was extremely beneficial to improve their electrical conductivity and electron transfer capability. However, after dedoping at −0.2 V, the surface of the PPP film became very rough and appeared some hollows, which was different from that of the doped polymer. This difference between the doped and dedoped PPP was mainly due to the migration of counter-anionout-of the polymer film.

The electrical conductivity of the PPP film obtained from BFEE + 37.5% CH$_2$Cl$_2$ was measured to be 0.015 S cm^{-1}. These semiconducting properties will be greatly helpful for the applications of PPP films.

CONCLUSIONS

In this article, high quality PPP films with conductivity of 0.015 S cm^{-1} were synthesized electrochemically on stainless steel electrode by direct anodic oxidation of PP in BFEE + 37.5% CH$_2$Cl$_2$. In this medium, the oxidation potential onset of PP was measured to be only 1.23 V vs. SCE, which was much lower than that in CH$_2$Cl$_2$ + 0.1 mol L^{-1} Bu$_4$NBF$_4$ (1.87 V vs SCE). PPP films obtained from this medium showed good redox activity and stability in BFEE. TGA results indicated good thermal stability for the PPP films obtained from BFEE + 37.5% CH$_2$Cl$_2$. The infrared spectroscopic measurements for an estimation of chain lengths reveal that PPP was composed of an average of about 10 phenyl rings when synthesized from PP oxidation.

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