

# Preparation and Characterization of Composites of Polyaniline Nanorods and Multiwalled Carbon Nanotubes Coated with Polyaniline

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**ABSTRACT:** Composites of polyaniline (PANI) nanorods and multiwalled carbon nanotubes (MWNTs) coated with PANI were prepared by *in situ* polymerization with perchloric acid as a dopant. Transmission electron microscopy images showed that the coexisting composites of PANI nanorods and MWNTs coated with PANI were formed at low MWNT contents. The interaction between MWNTs and PANI was proved by Fourier transform infrared and ultraviolet-visible spectra. The electrical conductivity of a dedoped PANI/MWNT composite with a 16.3 wt % concen-

tration of MWNTs reached  $3.0 \times 10^{-3}$  S/cm, which was 6 orders of magnitude higher than that of dedoped PANI nanorods. The results also showed that coexisting composites of PANI nanorods and MWNTs coated with PANI had high electrochemical activity and good cyclic stability. © 2007 Wiley Periodicals, Inc. *J Appl Polym Sci* 106: 4241–4247, 2007

**Key words:** composites; conducting polymers; electrochemistry; microstructure

## INTRODUCTION

Polyaniline (PANI) is one of the most important conducting polymers because of its relatively facile processability, electrical conductivity, and environmental stability,<sup>1,2</sup> and it has many potential applications in antistatic packaging, electromagnetic shielding, cathode materials for rechargeable lithium batteries, anticorrosion coatings, sensors, membranes for the separation of gas mixtures, and so forth.<sup>3</sup> Recently, considerable attention has been paid to nanostructured PANI (nanofibers, nanowires, and nanotubes) because of the highly specific surface area of nanostructured PANI and special physical and chemical properties. Template synthesis,<sup>4–6</sup> template-free self-assembly,<sup>7,8</sup> electron spinning,<sup>9</sup> a seeding method,<sup>10</sup> dilute polymerization,<sup>11</sup> and interfacial polymerization<sup>12–14</sup> have been developed to prepare PANI nanorods and nanofibers. However, PANI becomes gradually insulating at  $\text{pH} \geq 4$ . Therefore, it is necessary to introduce conducting fillers into the PANI matrix to improve the high stability of its conductivity toward environmental exposure.

Among all conducting fillers, multiwalled carbon nanotubes (MWNTs) possess unique structural and

excellent electronic properties, and the combination of MWNTs and PANI makes composites with excellent electrical and electrochemical properties.<sup>15</sup> Significant progress has been made in fabricating PANI/MWNT composites; examples include PANI/MWNT composites synthesized by *in situ* polymerization,<sup>16–19</sup> melt and solution mixing,<sup>20,21</sup> the grafting of macromolecules to MWNTs,<sup>22</sup> and electrochemical<sup>23</sup> and microemulsion polymerization.<sup>24</sup> Some related work has been done on composites of MWNTs coated with PANI. However, there is less reported on the preparation of a coexisting composite of carbon nanotubes coated with PANI and nanostructured PANI by *in situ* polymerization.

This article describes the preparation of coexisting composites of PANI nanorods and MWNTs coated with PANI produced by *in situ* polymerization with perchloric acid ( $\text{HClO}_4$ ) as a dopant. This method has the characteristics of easy control and simple processing. The prepared composites possess excellent electrochemical activity and high conductivity that is less affected by environmental exposure. It is expected that these composites can be used as cathode materials for lithium batteries and ultracapacitors.

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## EXPERIMENTAL

### Materials

Aniline (analytical-grade) was purchased from Shanghai Chemical Reagent Corp. (Shanghai, China) and distilled *in vacuo* before use. Ammonium persulfate

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**TABLE I**  
**Preparation Conditions and Chemical Components of the PANI/MWNT Composites**

Sample	Aniline/ MWNT weight ratio	Aniline/ APS molar ratio	N (%)	PANI (%)	MWNTs (%)
PANI/MWNT-1	64/1	1/4	14.2	94.3	5.7
PANI/MWNT-2	32/1	1/4	12.6	83.7	16.3
PANI/MWNT-3	16/1	1/4	10.9	72.4	27.6
PANI/MWNT-4	8/1	1/4	8.81	58.5	41.5
PANI/MWNT-5	4/1	1/4	6.92	46.0	54.0
PANI/MWNT-6	2/1	1/4	5.13	34.1	65.9
PANI/MWNT-7	1/1	1/4	3.10	20.6	79.4

(APS; analytical grade) was purchased from Shanghai Lingfeng Chemical Reagent Corp. (Shanghai, China) and was purified by recrystallization from ethanol. MWNTs (purity  $\geq 95\%$ , diameter = 10–20 nm) were obtained from Shenzhen Nanotech Port (Shenzhen, China). Before use, the MWNTs were ultrasonically treated with a 3 : 1 mixture of concentrated  $\text{H}_2\text{SO}_4$  and  $\text{HNO}_3$  at  $60^\circ\text{C}$  for 6 h to introduce carboxylic acid groups, which made them disperse well in aqueous solutions. Chloroform,  $\text{HClO}_4$ , and ammonium hydroxide ( $\text{NH}_4\text{OH}$ ) were analytical-grade and were used without further purification.

### Preparation

PANI nanorods were prepared by the oxidative polymerization of aniline with APS in an aqueous  $\text{HClO}_4$  solution. Aniline (4.47 g) was dissolved in a 100-mL 1.5 mol/L aqueous  $\text{HClO}_4$  solution. A 1.5 mol/L  $\text{HClO}_4$  solution (50 mL) containing 2.75 g of APS was slowly added dropwise to the solution. The mixture was continually stirred at room temperature for 24 h.

For the preparation of a series of composites, a similar aniline polymerization procedure was applied; that is, the composites were produced by the polymerization of aniline with APS as an oxidant in the presence of treated MWNTs. Typically, a solution of 1.5 mol/L  $\text{HClO}_4$  containing different quantities of treated MWNTs was sonicated at room temperature for 2 h. The aniline monomer (4.47 g) was added and then stirred for 30 min. APS (2.75 g) in the  $\text{HClO}_4$  solution was slowly added dropwise to the reaction mixture. The reaction was continued at room temperature for 24 h. The doped products were obtained by filtration and washed with deionized water several times. The dedoped PANI/MWNT composites were obtained by the treatment of the doped PANI/MWNT composites in 10 wt %  $\text{NH}_4\text{OH}$  for 24 h. The preparation conditions and chemical components of the PANI/MWNT composites are listed in Table I.

### Instrumentation

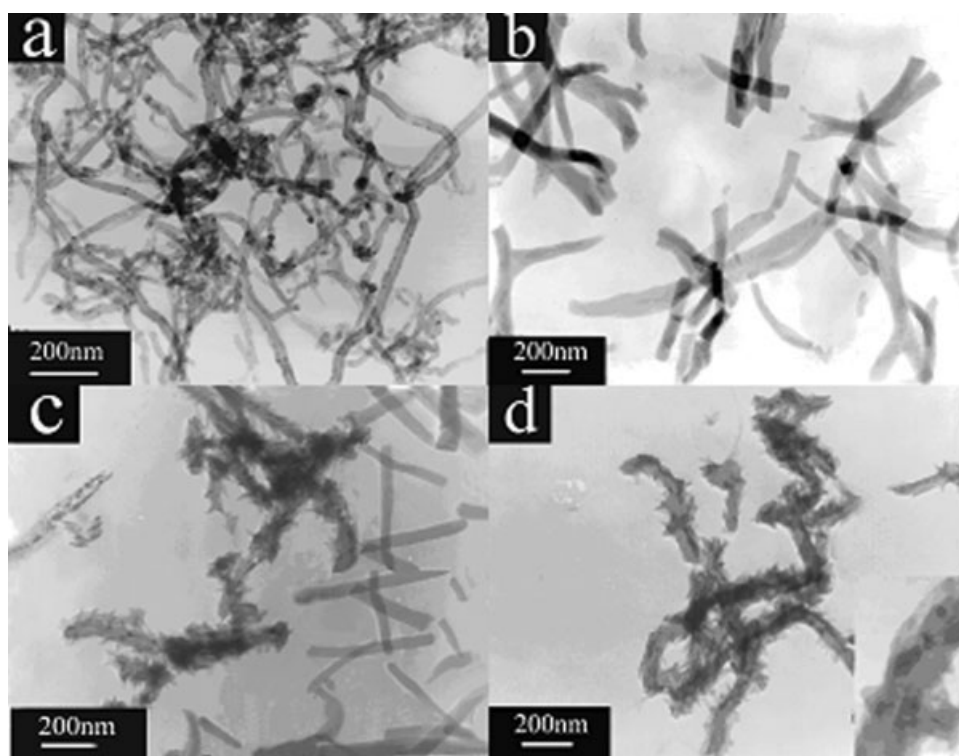
The morphology of the samples was investigated with transmission electron microscopy (TEM) on a JEM-100 CXII instrument (JEOL Ltd., Japan). The samples for

TEM were prepared by the ultrasonic dispersion of the powder in ethanol and then the coating of the dispersing solution onto wholly carbon-coated copper grids. Fourier transform infrared (FTIR) spectra were recorded from KBr sample pellets with a Nicolet 5700 spectrometer (Thermo Electron Co.). The ultraviolet–visible (UV–vis) spectra were obtained with a Shimadzu UV-250 spectrophotometer (Shimadzu Co., Japan) in the wave-number range of 200–1000 nm. The electrical conductivity of the samples was determined with an SX 1934 four-probe instrument. Cyclic voltammetry experiments were carried out with a CH760C apparatus (Shanghai Instruments, Ltd., Shanghai, China). A conventional three-electrode system was used that consisted of a working electrode, a platinum wire auxiliary electrode, and an Ag/AgCl reference electrode. The working electrode was made by the same quantity of a sample being coated onto a platinum disk (2 mm in diameter) in every experiment. After the evaporation of the solvent, the working electrode was further dried *in vacuo* at  $100^\circ\text{C}$  for 4 h before being placed into the glovebox. A 1 mol/L  $\text{LiPF}_6$  solution in dimethyl carbonate/ethyl methyl carbonate/ethylene carbonate (1/1/1 w/w) was used as the supporting electrolyte. The experiments were carried out at room temperature under an inert atmosphere.

## RESULTS AND DISCUSSION

### Morphology

Figure 1 shows TEM images of MWNTs, PANI nanorods, and PANI/MWNT composites. The MWNTs are clearly evident in the TEM image [Fig. 1(a)] as tangled, hollow ropes with a smooth surface. Their diameters are estimated to be in the range of 10–20 nm. As shown in Figure 1(b), PANI nanorods with a diameter of 70–110 nm have been prepared in the absence of MWNTs. For the PANI/MWNT composites, the changes in the morphology are remarkable. At a low MWNT content, we have found that the prepared products include both PANI nanorods and MWNTs coated with PANI (the rough part), as shown in Figure 1(c) (PANI/MWNT-2). However, at a high MWNT content, only MWNTs coated with PANI are formed, and no PANI nanorods are generated independently



**Figure 1** TEM images of (a) MWNTs, (b) PANI nanorods, (c) PANI/MWNT-2, and (d) PANI/MWNT-5 (the inset shows a magnified TEM image of a single MWNT coated with PANI).

[Fig. 1(d)]. The inset in Figure 1(d) shows a magnified TEM image for a single MWNT coated with PANI, in which the hollow structure between the two sides of the walls indicates the presence of the MWNT. The very thick diameter of the MWNTs and the very rough surface of the MWNTs indicate MWNTs coated with PANI.

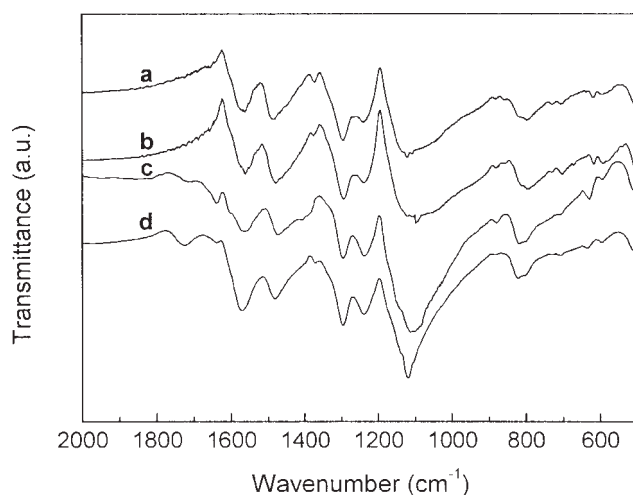
The formation of PANI nanorods is probably related to the oxidative polymerization process of aniline and the linear nature of PANI chains.<sup>13</sup> In the initial stage of polymerization, the PANI that forms initially exists in a fibrillar form. This fibrillar PANI will likely become the nucleation centers for further polymerization of aniline.<sup>11</sup> In this experiment, a low molar ratio of APS to aniline (1/4) has been adopted, which reduces the density of the initial nucleation centers and prevents secondary aggregation of the fibrillar PANI. Moreover, the HClO<sub>4</sub> medium is employed in this system. Because of the steric effect of ClO<sub>4</sub><sup>-</sup> ions of a large size, the polymerization rate of aniline decreases, and this also restrains the fast conglomeration of PANI and gives the generating PANI enough time to self-assemble into PANI nanorods. As the MWNTs are introduced into the reaction system, the  $\pi$ - $\pi^*$  electron interaction between the MWNTs and the aniline monomer,<sup>25</sup> as well as the hydrogen-bond interaction between the carboxyl groups of MWNTs and the amino groups of aniline monomers, causes the MWNTs to serve as the core for the polymeriza-

tion and growth of the aniline monomer to form MWNTs coated by PANI. At a low MWNT content, the polymerization of the partial aniline monomers first happens on the MWNT surface. The superfluous aniline monomer in the solution polymerizes to form PANI nanorods. With increasing MWNT content, a majority of the aniline monomer polymerizes on the surface of the MWNTs to form MWNTs coated with PANI, and PANI nanorods do not occur. This indicates that aniline preferentially polymerizes on the surface of MWNTs.

#### FTIR spectra

Figure 2 shows the FTIR spectra of the doped PANI nanorods and doped PANI/MWNT composites. The PANI nanorods and PANI/MWNT composites show similar IR absorption bands. The absorption bands at about 1560 and 1480 cm<sup>-1</sup> correspond to C=C stretching of quinoid and benzenoid rings, respectively.<sup>18,26</sup> The strong characteristic band appearing at 1121 cm<sup>-1</sup> has been described by MacDiarmid et al.<sup>27</sup> as an electron-like band that is considered to be a measure of the delocalization of electrons.<sup>27</sup>

However, there are several differences among the FTIR spectra of the PANI/MWNT composites and the PANI nanorods. As observed for the PANI nanorods and PANI/MWNT composites with low MWNT contents, the benzenoid band at about 1480 cm<sup>-1</sup> is

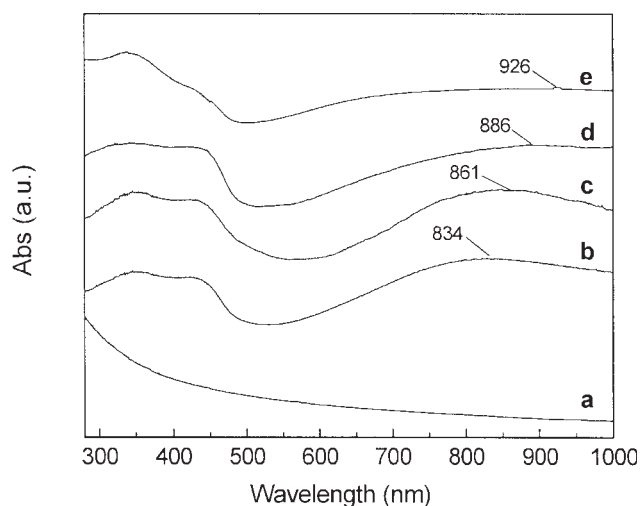


**Figure 2** FTIR spectra of (a) doped PANI nanorods, (b) doped PANI/MWNT-2, (c) doped PANI/MWNT-5, and (d) doped PANI/MWNT-7.

stronger than that of the quinoid band at about  $1560\text{ cm}^{-1}$  [Fig. 2(a,b)]. With increasing MWNT content in the PANI/MWNT composites, the intensity ratio of the absorption band at  $1560\text{ cm}^{-1}$  to that at  $1480\text{ cm}^{-1}$  ( $I_{1560}/I_{1480}$ ) increases [Fig. 2(b–d)], and finally, the spectrum of the composite shows an inverse intensity ratio of  $I_{1560}/I_{1580}$  [Fig. 2(d)]. The data reveal that the quinoid units of PANI synthesized at high MWNT contents are richer than those of the pure PANI nanorods and PANI/MWNT composites formed at low MWNT contents. The reason is that the  $\pi$ -bonded surface of the MWNTs interacts strongly with the conjugated structure of PANI, especially through the quinoid ring, just as it is known that the aromatic structures interact strongly with the basal plane of the graphitic surface via  $\pi$  stacking.<sup>28</sup> At the same time, the intensity of the peak at  $1120\text{ cm}^{-1}$  becomes stronger with increasing MWNT content in the composites. The change is possible because the interaction between PANI and MWNTs facilitates the charge-transfer process between the components of the system and increases the effective degree of electron delocalization and thus enhances the conductivity of the polymer chains.<sup>29</sup>

### UV-vis spectra

Figure 3 shows the UV-vis spectra of the MWNTs, doped PANI nanorods, and doped PANI/MWNT composites dispersed in deionized water. No absorption band can be observed for MWNTs in the 300–1000-nm region [Fig. 3(a)]. The PANI nanorods present three absorption peaks. The peak at 350 nm corresponds to a  $\pi$ - $\pi^*$  transition of the benzenoid ring of PANI. Two characteristic peaks at 430 and 834 nm are assigned to the localized polarons<sup>30</sup> [Fig. 3(b)]. For the

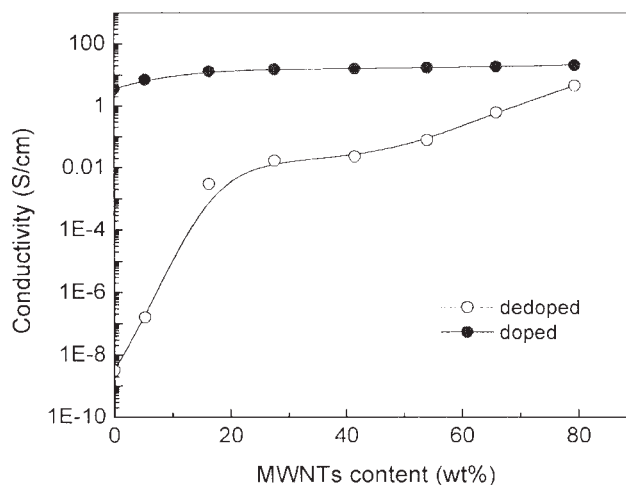


**Figure 3** UV-vis spectra of (a) MWNTs, (b) doped PANI nanorods, (c) doped PANI/MWNT-2, (d) doped PANI/MWNT-5, and (e) doped PANI/MWNT-7.

PANI/MWNT composites, three characteristic peaks of PANI can also be observed. With an increase in the MWNT content, the characteristic peak at about 830 nm redshifts gradually, and its free carrier tail becomes much broader; this indicates that the interaction between the functionalized MWNTs and PANI layer facilitates electron delocalization among the composites, and this results in enhanced conductivity of the polymer chains.<sup>31</sup> The results are also supported by the data observed from FTIR.

### Conductivity

Figure 4 shows the effect of the MWNT content on the conductivity of PANI/MWNT composites with PANI in the doped form and the dedoped form. The effects of the MWNT content on the conductivity of the com-



**Figure 4** Influence of the MWNT content on the conductivity of the PANI/MWNT composites.

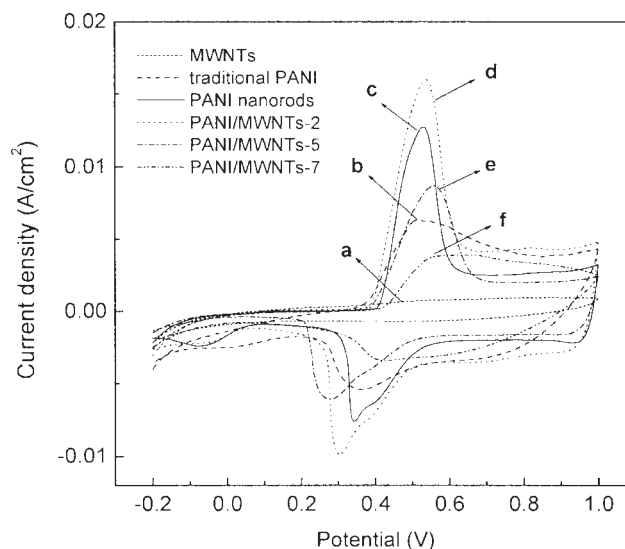


posites of both forms are obviously different. The conductivity of the doped PANI nanorods is 3.5 S/cm. For the doped PANI/MWNT composites ( $\text{pH} < 4$ ), the effect of the MWNT content on the conductivity is very small. At low MWNT contents, the conductivity of the composites is mainly determined by doped PANI nanorods. When the MWNT content reaches 10 wt %, the conductivity reaches a stable value and does not change with the MWNT content. Finally, the conductivity reaches 21.2 S/cm when the MWNT content is 79.4 wt %, which is a value approaching that of pure MWNTs (23.2 S/cm). The reason for the high conductivity is that the  $\pi$ - $\pi^*$  interaction between the surface of the MWNTs and the quinoid ring of the PANI chain effectively improves the degree of electron delocalization between the two components, as proved by FTIR and UV-vis spectra. The MWNTs serve as a conducting bridge to link doped PANI to improve the conductivity of the composites.

The conductivity of the dedoped PANI nanorods is very low ( $3.2 \times 10^{-9}$  S/cm). For the dedoped PANI/MWNT composites ( $\text{pH} > 7$ ), the conductivity of the composites increases first quickly. The conductivity of the composite with a 16.3 wt % concentration of MWNTs reaches  $3.0 \times 10^{-3}$  S/cm, which is 6 orders of magnitude higher than that of dedoped PANI nanorods. Thereafter, the increase in the conductivity becomes slow. The lower conductivity at low MWNT contents results from the surface of MWNTs being coated with insulating dedoped PANI, which increases the contact resistance between MWNTs. With increasing MWNT content, the PANI insulation layer becomes thinner, and this reduces the contact resistance of MWNTs and makes the charge-carrier transfer over short distances between the neighboring MWNTs easier. As a result, the PANI/MWNT composites with a concentration of MWNTs over 16 wt % possess highly stable conductivity even in a neutral environment, and this is suitable for the cathode materials of lithium batteries.

### Electrochemical properties

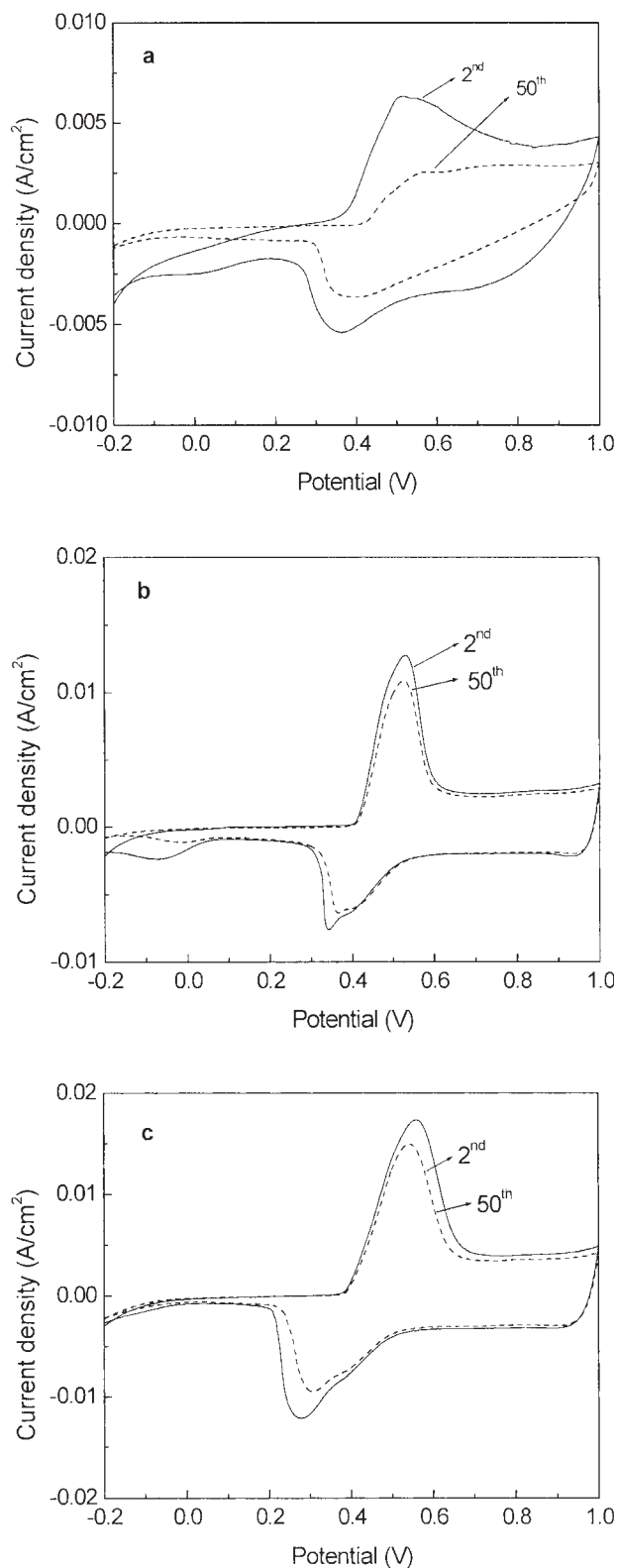
To investigate the electrochemical activity of the PANI/MWNT composites as a cathode material for lithium batteries, the MWNTs, traditional PANI (aggregated PANI), PANI nanorods, and PANI/MWNT composites with different MWNT contents have been chosen for comparison, and their cyclic voltammograms (CVs) have been measured (Fig. 5). No redox peaks can be observed for MWNTs in the CV curve [Fig. 5(a)], and this indicates that the MWNTs have no electrochemical activity. The CVs of traditional PANI exhibit one oxidation peak at 0.53 V and one reduction peak at 0.34 V [Fig. 5(b)]. The CV of PANI nanorods also shows a pair of redox peaks at similar potentials. Compared with that of



**Figure 5** CVs of (a) MWNTs, (b) traditional PANI, (c) PANI nanorods, (d) PANI/MWNT-2, (e) PANI/MWNT-5, and (f) PANI/MWNT-7 [scan rate ( $v$ ) = 20 mV/s; electrolytic solution: 1 mol/L  $\text{LiPF}_6$  in 1/1/1 (w/w) dimethyl carbonate/ethyl methyl carbonate/ethylene carbonate].

traditional PANI, the intensity of these peaks for PANI nanorods is obviously stronger [Fig. 5(c)]. This indicates that the PANI nanorods have better electrochemical activity because of the larger specific surface area of the PANI nanorods. For the coexisting composites of PANI nanorods and MWNTs coated with PANI (at low MWNT contents), the intensity of the redox peaks is higher than that of PANI nanorods because of the highly specific surface area of the PANI nanorods and the  $\pi$ - $\pi^*$  interaction between PANI and MWNTs [Fig. 5(d)]. This indicates that the coexisting composites of PANI nanorods and MWNTs coated with PANI possess high electrochemical activity. However, for the composites of MWNTs coated with PANI (at high MWNT contents), the intensity of the redox peaks is lower than that of the PANI nanorods because of the decrease in the PANI component [Fig. 5(e,f)].

Figure 6 shows the cyclic stability of the traditional PANI, PANI nanorods, and PANI/MWNT composites. The CVs of the 2nd and 50th cycles are presented for comparison. As shown in Figure 6, the PANI nanorods and PANI/MWNT composites are more stable than traditional PANI. For traditional PANI, the doping/dedoping of the counteranions in the electrochemical cycles produces a structural variety of PANI, resulting in electrochemical instability for the traditional PANI.<sup>32</sup> The cause of the stabilization of the electrochemical response for the PANI nanorods and PANI/MWNT composites can be explained as follows. During electrochemical cycles, more doping/dedoping of counteranions occurs on the surface of the composites in comparison with the traditional



**Figure 6** Stability measurements of (a) traditional PANI, (b) PANI nanorods, and (c) PANI/MWNT-2 [ $v = 20$  mV/s; electrolytic solution: 1 mol/L LiPF<sub>6</sub> in 1/1/1 (w/w) dimethyl carbonate/ethyl methyl carbonate/ethylene carbonate].

PANI because of the highly specific surface area of the PANI nanorods and PANI/MWNT composites; this results in less change in the PANI structures.

## CONCLUSIONS

PANI/MWNT composites have been prepared by *in situ* polymerization with HClO<sub>4</sub> as a dopant. At low MWNT contents, coexisting composites of PANI nanorods and MWNTs coated with PANI have been formed. However, composites of only MWNTs coated with PANI have been produced at high MWNT contents. FTIR and UV-vis spectra illustrate that the interaction between MWNTs and PANI occurs through the  $\pi$ -bonded surface of the MWNTs and the quinoid ring of the PANI chain. The conductivity of the dedoped composites has been improved by the addition of MWNTs as conducting bridges, and the conductivity has reached  $3.0 \times 10^{-3}$  S/cm at an MWNT concentration of 16.3 wt %; this indicates high stability toward environmental exposure. Cyclic voltammetry measurements have shown that the coexisting composites of PANI nanorods and MWNTs coated with PANI have high electrochemical activity and good cyclic stability. The materials are expected to be applied as cathode materials for lithium batteries and ultracapacitors.

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