

# Preparation and Characterization of Conductive Poly(Vinyl Alcohol)/Polyaniline Doped by Dodecyl Benzene Sulfonic Acid (PVA/PANDB) Blend Films

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Received 22 May 2006; accepted 5 July 2006

DOI 10.1002/app.25145

Published online in Wiley InterScience (www.interscience.wiley.com).

**ABSTRACT:** In this study a solution-blend method is adopted to prepare conductive poly(vinyl alcohol)/polyaniline doped by dodecyl benzene sulfonic acid (PVA/PANDB) blend films. Emeraldine base (EB)-type polyaniline (PANI) is dissolved in *N*-methyl-2-pyrrolidinone (NMP) and then blended with PVA/dodecyl benzene sulfonic acid (DBSA) solution by various amounts. It is found that the electrical conductivity and the thermal degradation onset temperature of the PVA/PANDB blend film are increased as the amount of EB-type PANI solution is increased. Fourier transform

infrared (FTIR) spectra show that the intensity of the characteristic peak of the functional groups in the blend film is significantly changed as the amount of EB-type PANI is changed. From optical microscopy examination, it indicates that the amount and size of green particles are increased with increasing the amount of EB-type PANI solution. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 103: 3415–3422, 2007

**Key words:** polyaniline (PANI); poly(vinyl alcohol) (PVA); dodecyl benzene sulfonic acid (DBSA); blend

## INTRODUCTION

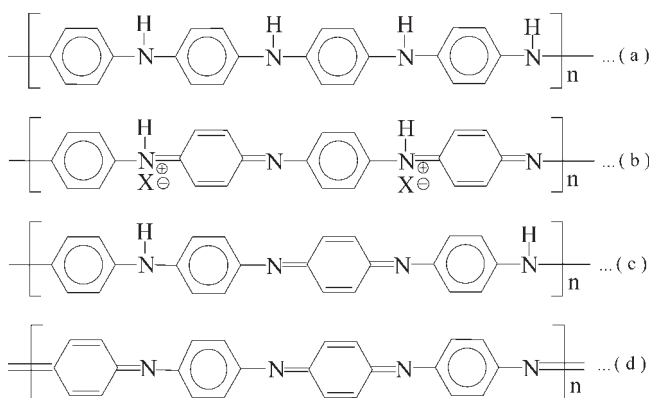
Currently, polyaniline has been an important member in the family of intrinsically conducting polymers (ICPs). Because of excellent environmental stability and unique electrochemical property, polyaniline (PANI) has been widely studied and applied, for example, in secondary batteries,<sup>1,2</sup> biosensors,<sup>3,4</sup> corrosion protections,<sup>5,6</sup> antistatic packaging materials,<sup>7</sup> and so forth.

Generally speaking, chemical oxidization and electrochemical syntheses are two main routes to prepare the PANI.<sup>8,9</sup> MacDiarmid et al.<sup>10</sup> have illustrated the chemical structure of PANI (Scheme 1). The emeraldine base (EB)-type PANI cannot be dissolved in common organic solvents. However, it can be dissolved in 1-methyl-2-pyrrolidinone (NMP). Then, freestanding EB-type PANI films can be cast from the NMP solution.<sup>11,12</sup> Moreover, the EB-type PANI can be doped in a strong protonic acid, such as HCl, HNO<sub>3</sub>, or H<sub>2</sub>SO<sub>4</sub>, and then transferred to emeraldine salt (ES)-type PANI with a moderately high electrical conductivity up to 10 ~ 100 S/cm. However, the PANI doped with strong protonic acids is intractable.

Recently, several methods have been developed to improve the processibility of PANI by increasing its solubility in various organic solvents.<sup>13–20</sup> There have been a number of attempts towards making conducting PANI processible by doping with various dopants. Since the PANI doped by DBSA (PANDB) becomes soluble in various organic solvents (e.g., chloroform and xylene), the most attractive and promising system is PANI doped with DBSA (PANDB). The chemical structure of the PANDB can be schematically represented<sup>20</sup> (Scheme 2).

Moreover, several papers have reported that the conducting polymer blends prepared with ES-type PANI and classic polymers (such as PMMA, PS, PI, etc.) in organic solvents exhibit good mechanical and electrical properties.<sup>21–24</sup> Due to the water solubility and good processibility of poly(vinyl alcohol) (PVA), there is considerable interest with regard to the blend of PANI with PVA.<sup>25,26</sup> There are two main routes to prepare the PVA/PANI blend film.<sup>27</sup> One is to chemically prepare the PVA/PANI blend film in the presence of PVA.<sup>25</sup> The other is to prepare the PVA/PANI blend film by mixing PANI with PVA solution in the aqueous system after the polymerization of PANI.<sup>28</sup> The blending of HCl-doped PANI nanoparticles with PVA has also been prepared by suspending HCl-doped PANI nanoparticles in the HCl aqueous solution.<sup>29,30</sup> Kuramoto et al. reported that a processible PANI doped and complexed with DBSA was pre-

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**Scheme 1** Four forms of PANI: (a) leucoemeraldine base (LEB); (b) metallic emeraldine salt (ES); (c) emeraldine base (EB); (d) pernigraniline base (PNB).

pared by one-step chemical oxidation polymerization in the presence of DBSA.<sup>31</sup> They also prepared and characterized PVA/PANDB blend films with various molar ratios by suspending PANDB particles in an aqueous system.<sup>27</sup>

Because NMP is a polar basic solvent that easily interacts with acids, the competition mechanism between PANI and NMP in the acid environment can be given by



which refers to doped PANI, that is, ES-type, and



that is, EB-type PANI. The competition results in coexistence and equilibrium of the doped and de-doped PANI. With diluting of the solution, the equilibrium was shifted to the right-hand side of the reaction (2) and then PANI was de-doped.<sup>32</sup>

In this study, PVA/PANDB blend films are prepared using a solution-blend method. First, EB-type PANI powder is prepared as the method indicated in a previous paper.<sup>33</sup> In order to prevent the dissociation between PANI and organic acid, two homogeneous solutions, solutions A and B, are then prepared. Solution A is a PVA aqueous solution homogeneously

mixed with DBSA. Solution B is an EB-type PANI dissolved in *N*-methyl-2-pyrrolidinone (NMP). Finally, solutions A and B are blended by different amounts to prepare electrically conductive PVA/PANDB blend films.

The electrical conductivity of the PVA/PANDB blend films is characterized by the four-probe method. Meanwhile, the thermal degradation property of the PVA/PANDB blend films is analyzed using a thermogravimetric analyzer (TGA). The characteristics of the weight loss ( $\Delta Y$ ) and thermal degradation onset temperature of the blend films are studied. The changes of characteristic peak intensity and frequency of the functional groups in the blend films are also illustrated by the Fourier transform infrared (FTIR) spectra.

## EXPERIMENTAL

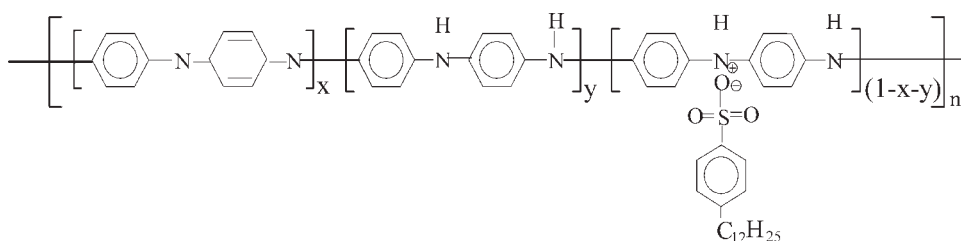
### Materials

The molecular weight of the EB-type PANI prepared by our laboratory was about 100,000 g/mol. DBSA, NMP, and PVA was purchased from Merck Ltd., Germany. The molecular weight of PVA was about 72,000 g/mol. All reagents were used without further purification.

### Preparation of PVA/PANDB blend films

DBSA (1 g) and PVA (4 g) were added into a beaker containing 45 g distilled water. This mixture was then heated while stirring to form a homogenous 10 wt % PVA-DBSA (abbreviation: PD) solution (also called solution A).

EB-type PANI (0.5 g) was added into a beaker that contains 49.5 g NMP. This mixture was then stirred for 3 h and vibrated by ultrasonic for additional 3 h. Then, filtration was executed and the filtrate (solution B) was collected. Wet filtrate cake was dried and then weighed. The amount of EB-type PANI dissolved in 49.5 g NMP can be calculated as 0.5 g minus the weight of dried cake. In this study, the dried cake was about 0.35 g. Thus, the EB-type PANI dissolved in NMP was about 0.15 g, that is, the NMP solution contains about 0.3 wt % of EB-type PANI.



**Scheme 2** Chemical structure of the PANDB.

**TABLE I**  
**Abbreviations and Blend Weight Ratios for PVA-DBSA Solution (Solution A) and EB-Type PANI Solution (Solution B)**

Abbreviation	Blend weight ratio	Amount of EB-type PANI (wt %)
PD10	10 g solution A	0.000
PD9.5	9.5 g solution A + 0.5 g solution B	0.015
PD9	9 g solution A + 1 g solution B	0.030
PD8.5	8.5 g solution A + 1.5 g solution B	0.045
PD8	8 g solution A + 2 g solution B	0.060

Total weight of 10 g of solutions A and B was used as a base. PVA/PANDB blend films were prepared by a solution-blend method. The amount of solutions A and B used in the experiments and the weight percent of EB-type PANI are both indicated in Table I. The mixture containing solutions A and B was stirred for 30 min for the purpose of homogeneous. After stirring, this mixture was naturally dried for three days in the hood. After that, it was put in an oven set at 60°C for additional drying of 6 h. Then, the PVA/PANDB blend film was obtained.

### Characterization and analyses

The four-point measurement method was used to examine the electrical conductivity  $\sigma$  (S/cm) of the PVA/PANDB blend film at room temperature. A HP 4338B low-resistance meter was used.

A thermogravimetric analyzer (TGA, Perkin Elmer, Model TGA 7) was conducted to measure the thermal weight loss of the PVA/PANDB blend films. Under a nitrogen stream, the temperature was raised from 50 to 700°C with a heating rate of 10°C/min. The curves of TGA and DTG are recorded and analyzed.

The characteristic absorption peaks of the chemical structure of the PVA/PANDB blend films are identified using a FTIR spectrometer (Spectron One, Perkin Elmer). The wavenumber range of 650–4000  $\text{cm}^{-1}$  with 32 scans per second was adopted for the analysis.

An optical microscope (Olympus BH2-UMA) was used to examine the morphological change of the PVA/PANDB blend films of various amounts.

## RESULTS AND DISCUSSION

### Preparation of PVA/PANDB blend films

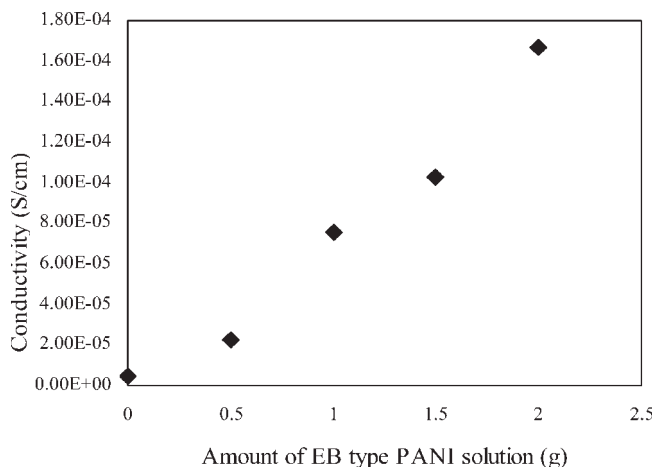
Dark-blue EB-type PANI solution and clear PVA-DBSA solution were blended by different amounts. Since EB-type PANI can be doped by DBSA to form ES-type PANI (PANDB), the colors of the blend films were changed during the doping process. The color of the mixture solution was changed from dark blue to light green, green, or dark green, depending on the amount of EB-type PANI solution. When the amount of EB-type PANI solution was increased, the color of

the PVA/PANDB blend films was changed from colorless transparent (PD10, no addition of EB-type PANI solution), light green semi-transparent (PD9.5, containing 0.5 g EB-type PANI solution), green semi-transparent (PD9, containing 1 g EB-type PANI solution), to dark green semi-transparent (PD8.5 and PD8, containing 1.5 g and 2.0 g EB-type PANI solution, respectively). The amounts of PANDB in PD9.5, PD9, PD8.5, and PD8 were 0.015, 0.03, 0.045, and 0.06 wt %, respectively.

### Electrical conductivity measurement

Since the EB-type PANI (electrical insulator) can be doped with the DBSA and form ES-type PANI (electrical conductor), the electrical conductivity of PVA/PANDB blend film is improved with an increasing amount of EB-type PANI solution. Figure 1 illustrates the influence of the weight of EB-type PANI solution on the electrical conductivity of PVA/PANDB blend films. The electrical conductivities of PD9.5, PD9, PD8.5, and PD8 are  $2.3 \times 10^{-5}$  S/cm,  $7.5 \times 10^{-5}$  S/cm,  $1.0 \times 10^{-4}$  S/cm, and  $1.7 \times 10^{-4}$  S/cm, respectively.

In this study, in order to prevent the dissociation between PANI and organic acid, two solutions (solutions A and B) were prepared. Solution A was PVA



**Figure 1** Influence of the amount of EB-type PANI solution on the electrical conductivity of PVA/PANDB blend films.

TABLE II  
Thermal Weight Losses and Degradation Onset Temperatures of the PVA/PANDB  
Blend Films Prepared at Various Amounts

	$\Delta Y_1$ (wt %)	$T_{onset-1}$ (°C)	$\Delta Y_2$ (wt %)	$T_{onset-2}$ (°C)	$\Delta Y_3$ (wt %)	$T_{onset-PANI}$ (°C)
PD10	33.09	161.42	8.74	312.57	32.98	—
PD9.5	35.4	179.05	8.615	320.05	32.02	449.56
PD9	37.67	180.43	7.791	322.39	28.09	450.55
PD8.5	41.1	184.69	6.875	334.99	25.39	456.62
PD8	43.66	191.75	4.851	335.74	21.56	456.88

aqueous solution mixed with DBSA. Solution B was EB-type PANI dissolved in NMP. Finally, solutions A and B were blended by different amounts to prepare electrically conductive PVA/PANDB blend films. The electrical conductivity of PD8 (containing 0.06 wt % PANDB) was  $1.7 \times 10^{-4}$  S/cm. Kuramoto et al. also prepared and characterized PVA/PANDB blend films with various amounts by suspending PANDB particles in aqueous system.<sup>27</sup> They found the PVA/PANDB blend film with appropriate conductivity of  $5.5 \times 10^{-3}$  S/cm for the low weight fraction of PANDB at 6.9 wt %. Wan et al. prepared PANI doped by  $\beta$ -naphthalene sulfonic acid ( $\beta$ -NSA) (PANI- $\beta$ -NSA).<sup>34</sup> They prepared the PVA/PANI- $\beta$ -NSA blend films by simple mixing of PANI- $\beta$ -NSA dispersion in water with aqueous PVA solution. They found the PVA/PANI- $\beta$ -NSA blend film with an electrical conductivity of  $7.0 \times 10^{-6}$  S/cm for the weight fraction of PANI- $\beta$ -NSA at 10.0 wt %.

Comparing the result of this study with those of Kuramoto et al. and Wan et al., it can be concluded that there are three important factors that affect the electrical conductivity of the blend film. The first important factor is definitely the amount of electrical conducting material. Generally speaking, the electrical conductivity of the blend film is increased when the amount of electrical conducting material is increased. This is due to the formation of more conductive paths through the blend film for higher loading of electrical conducting material. The second important factor is the kind of electrical conducting material. For example, the same polymer matrix (for instance, PVA) is blended with the same amount of electrical conducting material (for instance, PANDB or PANI- $\beta$ -NSA), resulting in blend films with different electrical conductivities. The third important factor is the blend process. For example, the same polymer matrix (for instance, PVA) is blended with the same amount of electrical conducting material (for instance, PANDB), resulting in PVA/PANDB blend films with different electrical conductivities.

### TGA analysis

TGA characteristics of PVA/PANDB blend films are summarized in Table II. TGA curves of PD10 (no

addition of EB-type PANI solution), PD9 (containing 1 g EB-type PANI solution), and PD8 (containing 2 g EB-type PANI solution) are shown in Figure 2. Three significant weight losses are observed. The first thermal weight loss, 50–200°C, is due to the vaporization of moisture and NMP.  $\Delta Y_1$  is used to represent the first thermal weight loss and  $T_{onset-1}$  stands for the first thermal degradation onset temperature. The second thermal weight loss, starting at around 300°C, represents the evaporation and degradation of DBSA.  $\Delta Y_2$  is used to represent the second thermal weight loss and  $T_{onset-2}$  is the thermal degradation onset temperature of DBSA. According to the findings from the previous study, the weight loss of pure DBSA starts at around 200°C. Besides, the weight loss of the DBSA doped on the backbone of PANI starts at around 250°C.<sup>20</sup> Therefore, this illustrates that not only the coulomb attraction between DBSA and the backbone of PANI is destroyed, as well as the DBSA starting evaporation and degradation, but also the molecular interaction (e.g., hydrogen bonding) among DBSA, PVA, and PANI is destroyed.

The third thermal weight loss, 400–510°C, results from the total thermal weight loss of PVA and its by-products that are formed during the TGA thermal degradation process. According to Hay's report, in the molten state, thermal degradation can lead to the

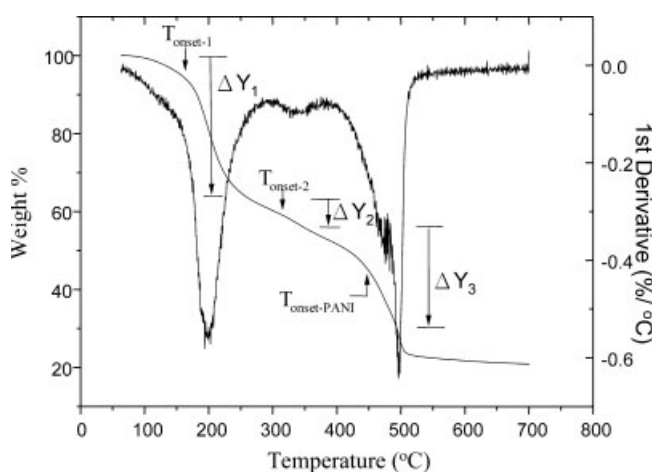
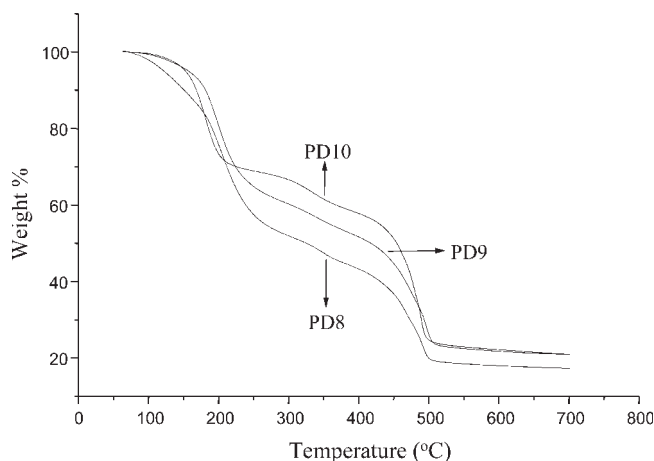


Figure 2 TGA and DTG analysis of PD9 blend films (heating rate of 10°C/min under a nitrogen stream).

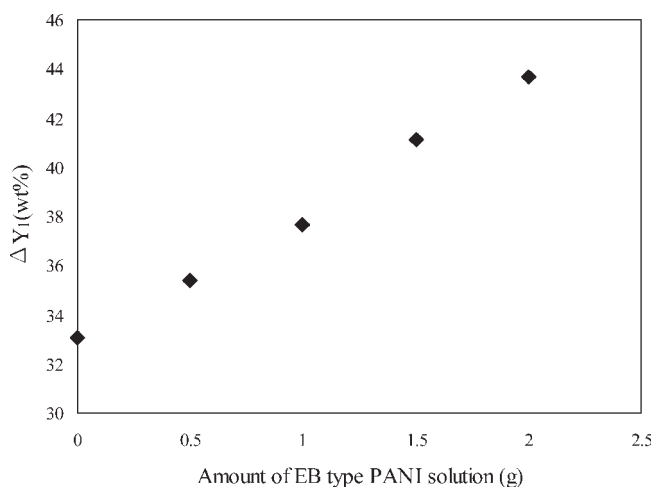




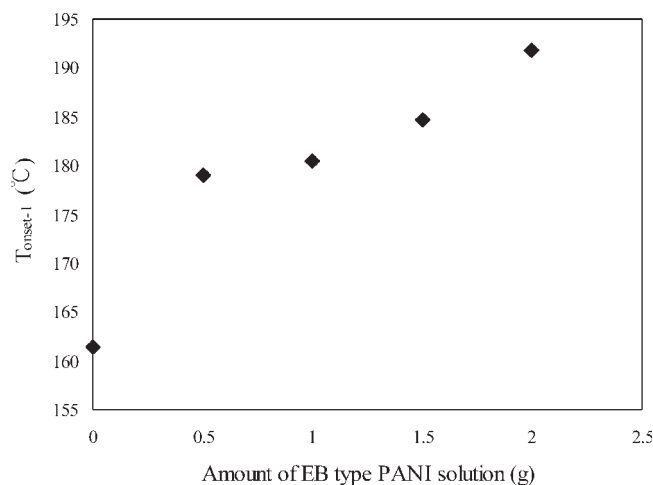
**Figure 3** TGA analysis of PD10, PD9, and PD8 blend films.

production of aldehyde and alkene end-groups and the formation of a vinyl ester by the rearrangement.<sup>35</sup>  $\Delta Y_3$  is used to represent the total thermal weight loss of PVA and its by-products that are formed during the TGA thermal degradation process. Around 440–460°C, an extra thermal degradation onset temperature,  $T_{onset-PANI}$ , is observed from the first derivative thermogravimetric (DTG) analysis curves of PD9.5, PD9, PD8.5, and PD8 blend films. The results indicate that  $T_{onset-PANI}$  becomes much more obvious when the amount of EB-type PANI solution is increased. The phenomena imply that  $T_{onset-PANI}$  results from the thermal degradation of PANI main molecular chain. Moreover,  $T_{onset-PANI}$  increases with an increasing amount of EB-type PANI solution. This illustrates that the polymer molecular interaction between PVA and PANI exists.

Figure 3 shows the TGA analyses curves of PD10, PD9, and PD8 blend films. Figure 4 shows the influ-



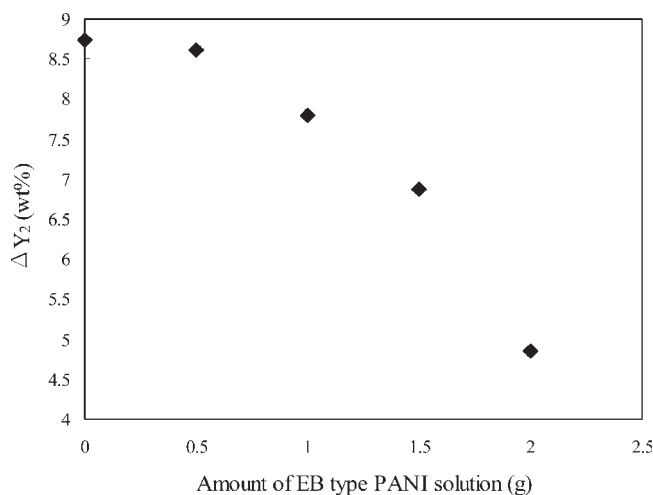
**Figure 4** Influence of the amount of EB-type PANI solution on the thermal weight loss ( $\Delta Y_1$ ) in the PVA/PANDB blend films.



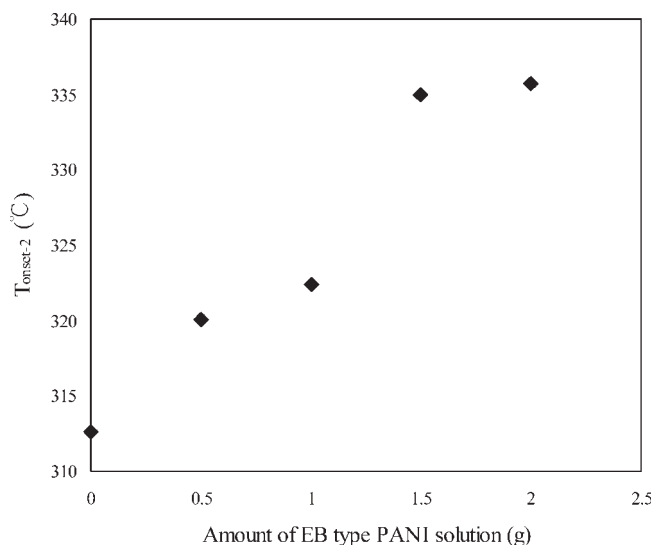
**Figure 5** Influence of the amount of EB-type PANI solution on the  $T_{onset-1}$  of PVA/PANDB blend films.

ence of the amount of EB-type PANI solution on the first thermal weight loss ( $\Delta Y_1$ ) in the PVA/PANDB blend films. Figure 5 indicates the influence of the amount of EB-type PANI solution on the first thermal degradation onset temperature ( $T_{onset-1}$ ) in the PVA/PANDB blend films. From Figures 4 and 5, it is found that  $\Delta Y_1$  and  $T_{onset-1}$  increases significantly as the amount of EB-type PANI solution is increased. This also indicates that the amount of NMP in the PVA/PANDB blend films increases as the weight of EB-type PANI solution is increased. Since NMP is a good solvent for EB-type PANI and can act as a plasticizer for PANI, it is not easy to remove NMP from the blend films during the drying process.

Figure 6 demonstrates the influence of the weight of EB-type PANI solution on the second thermal weight loss ( $\Delta Y_2$ ) in the PVA/PANDB blend films. Figure 7 shows the influence of the weight of EB-type PANI so-



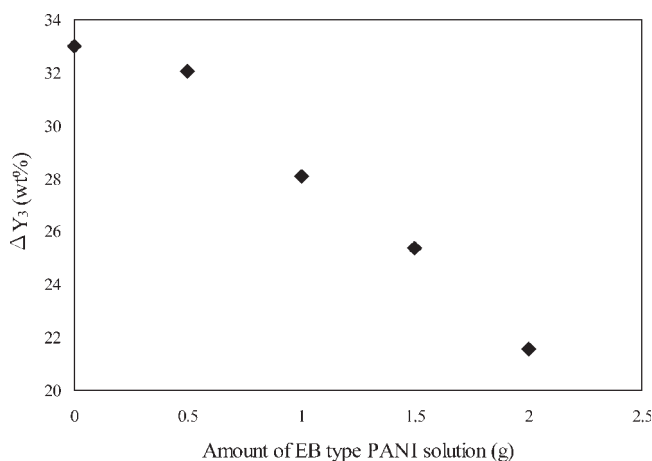
**Figure 6** Influence of the amount of EB-type PANI solution on the thermal weight loss ( $\Delta Y_2$ ) of PVA/PANDB blend films.



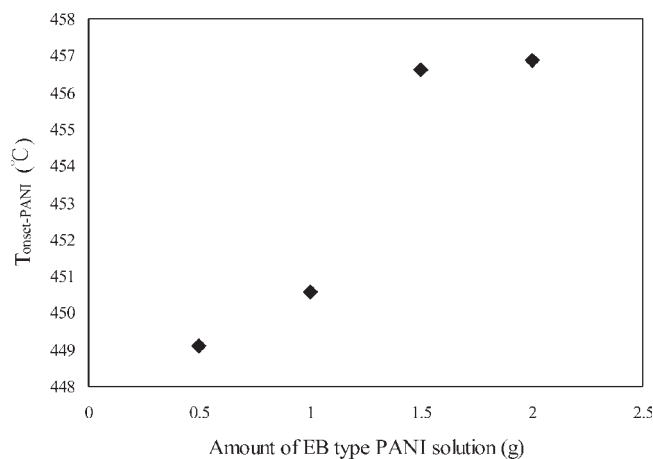
**Figure 7** Influence of the amount of EB-type PANI solution on the second thermal degradation onset temperature ( $T_{onset-2}$ ) of PVA/PANDB blend films.

lution on the second thermal degradation onset temperature ( $T_{onset-2}$ ) in the PVA/PANDB blend films. From Figure 6, it is found that  $\Delta Y_2$  is decreased as the amount of EB-type PANI solution is increased. This is due to the amount of DBSA that decreases as the amount of EB-type PANI solution is increased. Besides, from Figure 7,  $T_{onset-2}$  increases as the amount of EB-type PANI solution is increased. As mentioned previously, this tendency indicates both the molecular interaction among DBSA, PVA, and PANI and the coulomb attraction between DBSA and the backbone of PANI increase when the amount of EB-type PANI solution is increased.

Figure 8 shows the influence of the amount of EB-type PANI solution on the third thermal weight loss ( $\Delta Y_3$ ) in the PVA/PANDB blend films. This illustrates



**Figure 8** Influence of the amount of EB-type PANI solution on the thermal weight loss ( $\Delta Y_3$ ) of PVA/PANDB blend films.



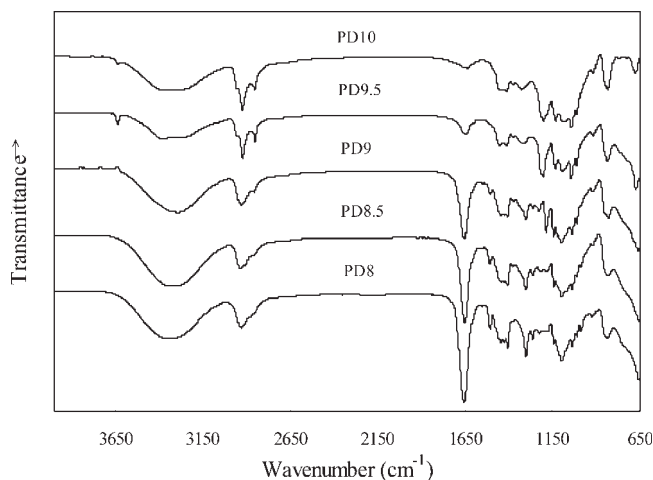
**Figure 9** Influence of the amount of EB-type PANI solution on the  $T_{onset-PANI}$  of PVA/PANDB blend films.

that  $\Delta Y_3$  decreases as the amount of EB-type PANI solution is increased. This is due to the weight content of PVA in the blend film decreasing as the weight of EB-type PANI solution is increased.

Figure 9 shows the influence of the amount of EB-type PANI solution on the thermal degradation onset temperature ( $T_{onset-PANI}$ ) of PANI in the blend films. This illustrates that  $T_{onset-PANI}$  is increased as the amount of EB-type PANI solution is increased.

### FTIR analysis

The FTIR spectra of the PVA/PANDB blend films are shown on Figure 10. The characteristic peak of the  $-\text{CH}-$  stretching vibration is observed at around  $2900\text{--}2950\text{ cm}^{-1}$ . This peak is shifted to the higher wavelength ( $\text{cm}^{-1}$ ) when the amount of EB-type PANI is increased. The characteristic peak of the stretching vibrations of  $\text{C}=\text{C}$  and  $\text{C}-\text{N}$  are observed at around  $1640\text{--}1667\text{ cm}^{-1}$  and  $1300\text{ cm}^{-1}$ , respec-



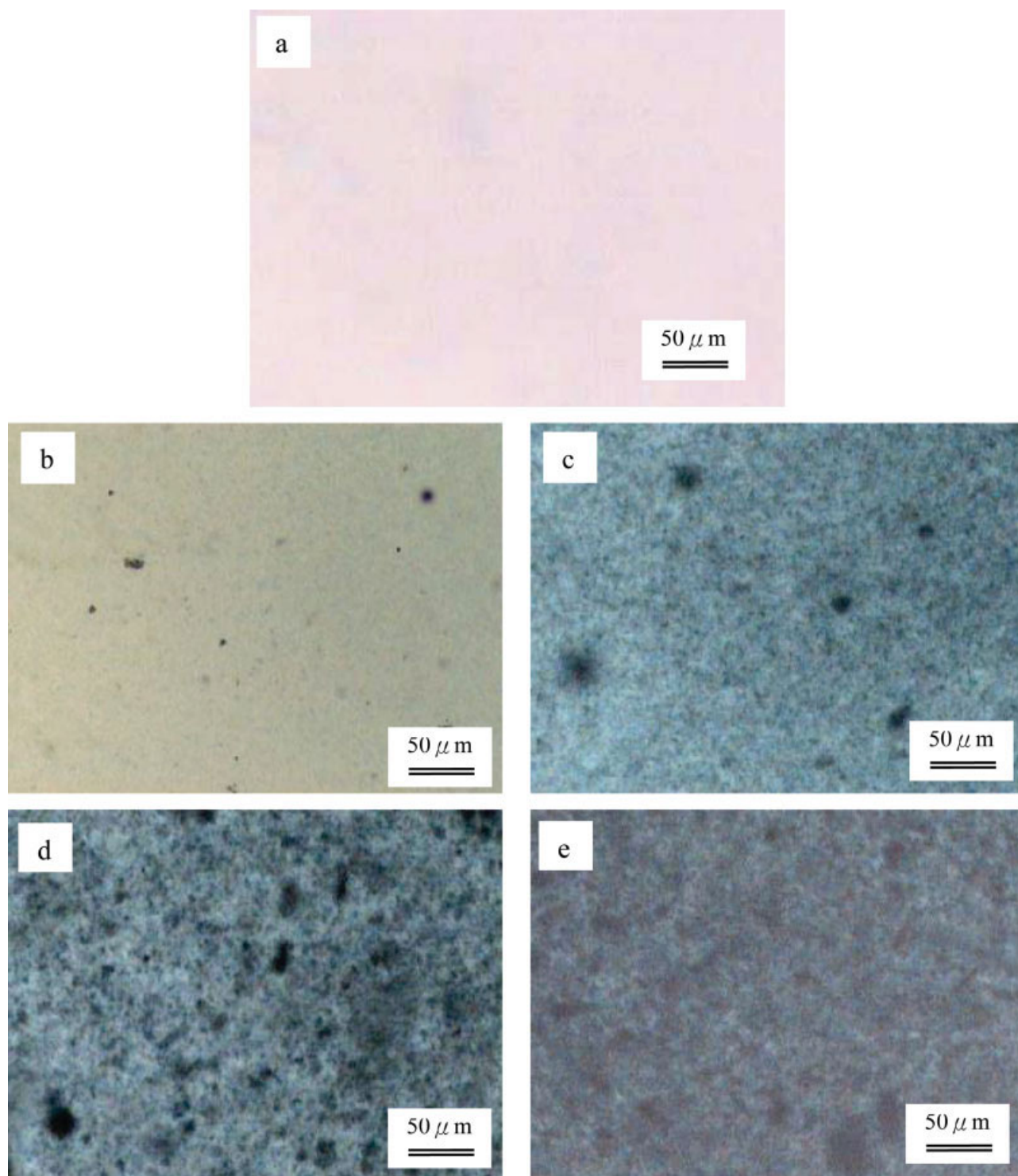
**Figure 10** FTIR spectra of PD10, PD9.5, PD9, PD8.5, and PD8 blend films.

tively. These two characteristic absorption peaks became much more obvious when the amount of EB-type PANI is increased. All the FTIR spectra demonstrate the existence of the  $-\text{SO}_3\text{H}$  functional group,  $1040\text{--}1180\text{ cm}^{-1}$ . This indicates that the EB-type PANI has been doped by DBSA to form a conductive ES-type PANI (PANDB). Meanwhile, the  $-\text{SO}_3\text{H}$  absorp-

tion peak becomes much significantly when the amount of EB-type PANI is increased.

#### Optical microscopy examination

Figure 11(a–e) shows the optical morphological change of the PVA/PANDB blend films with various



**Figure 11** Optical micrographs of PVA/PANDB blend films at  $200\times$  magnitude: (a) PD10; (b) PD9.5; (c) PD9; (d) PD8.5; (e) PD8. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

amounts. As mentioned previously, the color of the blend films is changed due to the EB-type PANI can be doped by DBSA to form ES-type PANI (PANDB). The color of the mixture solution (solution A mixing with solution B) is changed from dark blue to light green, green, or dark green, depending on the amount of EB-type PANI solution. When the amount of EB-type PANI solution is increased, the colors of the PVA/PANDB blend films are also changed from colorless transparent (PD10, no addition of EB-type PANI solution), light green semi-transparent (PD9.5, containing 0.5 g EB-type PANI solution), green semi-transparent (PD9, containing 1 g EB-type PANI solution), to dark green semi-transparent (PD8.5 and PD8.0, containing 1.5 and 2.0 g EB-type PANI solution, respectively). In Figure 11, it is found that the amount and size of green particles increase with an increasing amount of EB-type PANI. These green particles are ES-type PANI (PANDB) that result from the EB-type PANI doped by DBSA. Moreover, as the amount of EB-type PANI increases, some of the green PANDB particles are agglomerated, as shown in Figure 11(b–11e).

### CONCLUSIONS

A solution-blend method has been successfully conducted in order to prepare conductive PVA/PANDB blend films by adopting various amounts in this research. The color of the blend films changes by increasing the amount of EB-type PANI. This is due to the EB-type PANI being doped by DBSA to form the ES-type PANI (PANDB). The electrical conductivity of the PVA/PANDB blend films increases with an increasing amount of EB-type PANI. Three significant weight losses are observed in the TGA curves. The thermal degradation onset temperature is increased when the amount of EB-type PANI is increased. All the FTIR spectra show the existence of the  $-\text{SO}_3\text{H}$  functional group at  $1040\text{--}1180\text{ cm}^{-1}$ . Meanwhile, the  $-\text{SO}_3\text{H}$  characteristic absorption peak becomes much more significant when the amount of EB-type PANI is increased. The characteristic peaks of the stretching vibrations of  $\text{C}=\text{C}$  and  $\text{C}-\text{N}$  are observed at around  $1640\text{--}1667\text{ cm}^{-1}$  and  $1300\text{ cm}^{-1}$ , respectively. Also, these two characteristic absorption peaks became much more obvious when the amount of EB-type PANI was increased. Optical microscopy examination indicated that the amount and size of the green particles increase when the amount of EB-type PANI is increased. Moreover, as the amount of EB-type PANI increases, some green PANDB particles are agglomerated.

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