

Preparation of Conducting Nylon-6 Electrospun Fiber Webs by the *In Situ* Polymerization of Polyaniline

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ABSTRACT: For the preparation of conducting polyaniline (PANI)/nylon composites with high electrical conductivity as well as superior mechanical properties such as flexibility and lightness, PANI/nylon-6 composite nanofiber webs were prepared via the electrospinning process with a nylon-6/formic acid polymer solution, and then PANI on the surface of the nylon-6 electrospun nanofiber webs was chemically polymerized. The electrical conductivity measurements showed that the conductivity of the PANI/nylon-6 composite electrospun fiber webs was superior to that of PANI/nylon-6 plain-weave fabrics because of the high surface-area/volume ratios. On the other hand, the volume conductivities of the PANI/nylon-6 composite electrospun fiber webs increased from 0.5 to 1.5 S/cm as the diffusion time increased from 10 min to 4 h because of the even

distribution of PANI in the electrospun fiber webs. However, the surface conductivities of the PANI/nylon-6 composite electrospun fiber webs somewhat decreased from 0.22 to 0.14 S/cm as the diffusion time increased because of PANI contaminated with aniline monomers, aniline oligomers, and some alkyl chains, which served as electrical resistants. These results were confirmed with Fourier transform infrared, electron spectroscopy for chemical analysis, and morphology analysis. It was concluded that the diffusion time for the *in situ* polymerization of PANI in electrospun fiber webs was optimized at approximately 3 h. © 2005 Wiley Periodicals, Inc. *J Appl Polym Sci* 96: 983–991, 2005

Key words: conducting polymers; fibers; nylon

INTRODUCTION

Electrospinning is a fiber-spinning technique that produces polymer fibers with nanometer-to-micrometer diameters. Usually, a polymer solution or melt is placed in a syringe with a millimeter-size nozzle and is subjected to electric fields of several kilovolts. Under the applied electrostatic force, the polymer is ejected from the nozzle, whose diameter is reduced significantly as it is transported to and deposited on a collector, which also serves as the ground for the electrical charges. Such thin fibers provide unexpectedly high surface-area/volume ratios and are interesting for many applications, including textiles, composite reinforcements, sensors, biomaterials, and membranes.^{1,2}

Recently, the nanofiber fabrication of electrically conducting polymers has been demonstrated,^{3–7} and the preparation of conducting nanofibers through the addition of polyaniline (PANI) has attracted attention. Many researchers are studying conducting electro-

spun fiber webs mixed with PANI because, among organic conducting polymers, PANI is regarded as one of the most technologically promising conductive polymers. The monomer (aniline) is inexpensive, the polymer can be easily prepared by oxidative polymerization in a high yield, and the products are stable under ambient conditions.⁸

Therefore, when PANI is added to an electrospun fiber web, its electroactive nature (i.e., the electronic, magnetic, optical, and associated properties that respond to external influences) is enhanced for the high surface-area/volume ratio of the electrospun fiber substrate. Such conducting electrospun fiber webs could be useful in various industries not only because of their excellent electroactive nature but also because of their good mechanical properties, such as flexibility, toughness, and lightness. Reneker and Chun³ reported that PANI fibers could be successfully electrospun from a sulfuric acid solution into a coagulation bath; however, the conductivity value of the electrospun fibers was not reported. Norris et al.⁴ revealed that an electrospun fiber of PANI doped fully with camphorsulfonic acid (PANI-HCSA) was obtained as a blend with poly(ethylene oxide) (molecular weight = 900,000) through electrospinning. However, they stated that increasing the conductivity was limited because PANI-HCSA had very low solubility in chloroform. Also, Pinto et al.⁵ prepared the micrometer-

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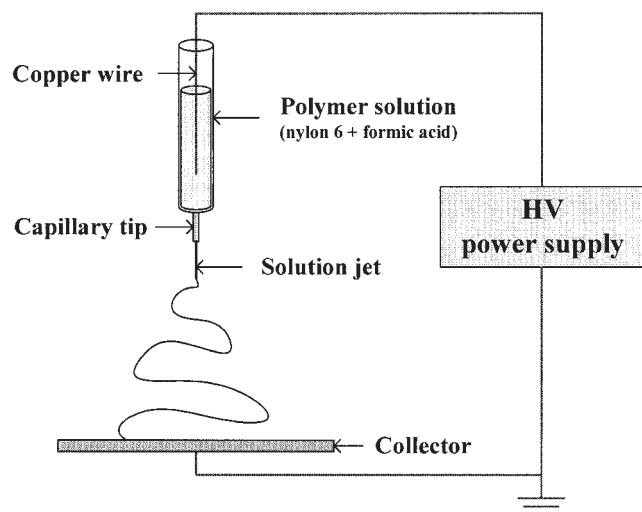


Figure 1 Diagram of the electrospinning system.

diameter fibers of 2-acrylamido-2-methyl-1-propane-sulfonic acid doped PANI via electrospinning, but the fiber diameters ranged from 2 to 10 μm , and the fibers had lower conductivities than cast films prepared from the same polymer solution.

Because of these problems, it has been difficult to prepare a conducting material with high electrical conductivity and superior mechanical properties. Therefore, to improve the conductivity and enhance the electroactive nature of PANI composite materials, we prepared nylon-6 nanofibers webs through the electrospinning process and used them as matrices for the preparation of conducting materials. We prepared PANI/nylon-6 composite electrospun fiber webs by *in situ* polymerizing aniline on the nylon-6 electrospun fiber webs. In our previous study,⁹ we found that *in situ* polymerization is a reasonable polymerization method for preparing PANI-coated materials because it does not require the destruction of the substrate and provides good conductivity. In particular, we believe that the diffusion step is important for improving the conductivity of PANI/nylon-6 composite electrospun fiber webs because electrospun fiber webs are comparatively voluminous materials. Therefore, in this study, the effect of the diffusion time on the conductivity of PANI/nylon-6 composite electrospun fiber webs was investigated. Also, PANI/nylon-6 composite plain-weave fabrics were prepared, and their conductivity was measured for comparison with that of PANI/nylon-6 composite electrospun fiber webs.

EXPERIMENTAL

Materials and chemicals

Nylon-6 chips for the preparation of nylon-6 nanofiber webs were obtained from Hyosung Co., Ltd. (Anyang,

Korea), and scoured nylon-6 plain-weave fabrics (70 \times 70 d, 43 \times 30/cm) were purchased from the Korea Apparel Testing and Research Institute (Seoul, Korea). Aniline (Ducksan Pure Chemicals Co., Ltd., Ansan, Korea) was distilled *in vacuo* before use, and reagent-grade ammonium peroxydisulfate [APS; $(\text{NH}_4)_2\text{S}_2\text{O}_8$] and hydrochloric acid (HCl) were purchased from Ducksan Pure Chemical Co., Ltd.

Preparation of nylon-6 nanofiber webs

Nylon-6 polymer solutions of various concentrations were prepared by the dissolution of nylon-6 chips in formic acid. The schematic diagram of the electrospinning apparatus is shown in Figure 1. The glass pipette used in these experiments had a capillary tip diameter of 1.2 mm. A positive potential was applied to the nylon-6 polymer solution through the insertion of a copper wire into the glass pipette, and the potential difference between the pipette and the counter electrode used to electrospin the polymer solution was 25 kV. A flat piece of aluminum foil, placed 10 cm below the capillary tip, was used to collect the electrospun fiber material.

Preparation of PANI/nylon-6 composite fabrics

The laboratory procedure was used to produce electrically conducting electrospun fiber webs, as shown in Figure 2. Freshly distilled aniline and APS were dissolved separately in 0.35M HCl aqueous solutions for diffusion and polymerization, respectively.

The nylon-6 electrospun fiber webs, fixed to glass frames for the retention of their original forms, were

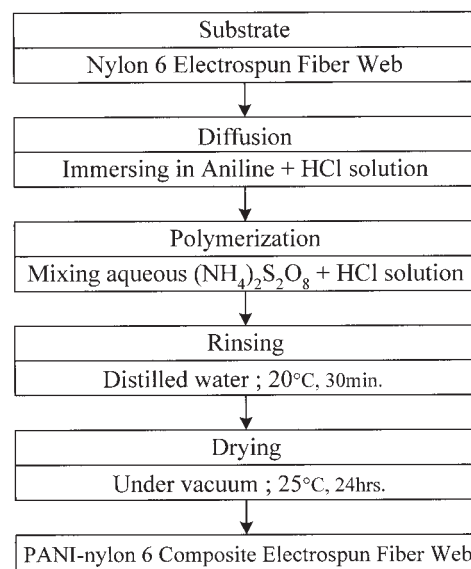


Figure 2 Schematic diagram of the PANI/nylon-6 composite fabric preparation process.

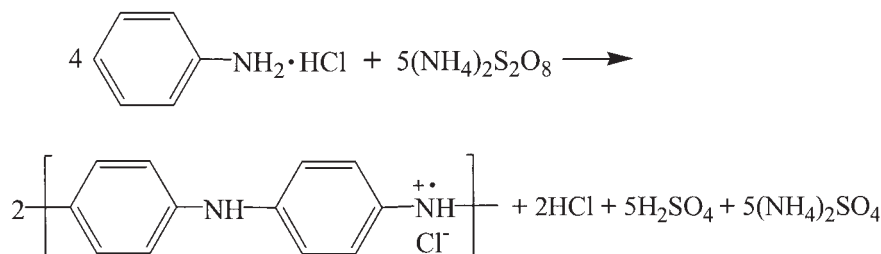


Figure 3 Mechanism of aniline oxidative polymerization and protonation with HCl.

immersed in an aqueous hydrochloride solution of aniline at 40°C for a specified time. Successive polymerization was then initiated by the mixing of the same volume of the oxidant solution containing APS and 0.35M HCl in the diffusion bath. Electrospun fiber webs were polymerized around 5°C for 1 h and then were washed with distilled water and dried. PANI (emeraldine salt form) was produced on the surface of the electrospun fiber webs through the oxidative polymerization of aniline and was protonated with the acid present in the reaction mixture. The chemical reaction mechanism is shown in Figure 3. For the comparison of the conductivities of PANI/nylon-6 composite electrospun fiber webs and PANI/nylon-6 composite plain-weave fabrics, plain-weave fabrics were also treated with the polymerization method mentioned previously.

Evaluation

The sorption property of PANI [add-on (%)] was measured via the weighing method on the basis of the weight changes of the fabric before and after treatment. The electrical volume conductivity of the composite fabric was measured by four straight lines and equidistant points, the thickness being taken as that of the fabric.¹⁰ A constant current source was used to pass a steady current through the two outer probes in the volume of a specimen, and the voltage drop across the inner two was measured. The conductivity of a sample was determined with the following formula:

$$\sigma(\text{S/cm}) = \frac{l}{dW} \times \frac{I}{V} \quad (1)$$

where σ is the electric conductivity, l is the spacing between probes (0.3 cm), d is the thickness of the sample, W is the length of the line probe (1 cm), I is the current passed through the outer probes, and V is the voltage drop across the inner probes.

Fourier transform infrared (FTIR) spectroscopy was performed with a PerkinElmer Spectrum 2000 FTIR spectrometer with a resolution of 4 cm⁻¹, and the measurements were carried out with a KBr support. The X-ray photoelectron spectroscopy (XPS)

measurements were performed with an Aries ARSC 10MCD 150 (VSW, United Kingdom) spectrometer from Surface Science Instruments. This spectrometer was equipped with an Mg K α X-ray source at the power level of 10 kV and 15 mA. The pressure in the analysis chamber was maintained at 10⁻⁹ Torr or lower during the measurement. All core-level spectra were obtained at a photoelectron takeoff angle of 55° with respect to the sample surface. To compensate for the surface charging effect, all binding energies were referenced to a C_{1s} neutral carbon peak at 284.5 eV. In the peak analysis, the line widths (the width at half-maximum) of the Gaussian peaks were kept constant for the components in a particular spectrum. The surface elemental compositions were determined from the peak area ratios and were accurate to within 1%. A JEOL JSM-6340F field emission scanning electron microscope and an Olympus BH-2 optical microscope were used for high-magnification observations.

RESULTS AND DISCUSSION

Preparation of nylon-6 electrospun fiber webs

The processing parameters of electrospinning include the polymer concentration, deposition distance, and applied field strength. The discontinuous thin webs created by this process are composed of shaped elements such as fibers or beads. The aspects of the web structure affected by change in the electrospinning process parameters include the element shape, element size, and overall web thickness.^{11,12} Therefore, we examined the diameter variations at different nylon-6 concentrations in polymer solutions. All the other parameters were kept constant at 25 kV and a 10-cm deposition distance.

Figure 4 shows a series of scanning electron microscopy (SEM) micrographs depicting the morphology changing from a filamentous and beaded web stage [Fig. 4(a)] to filamentous web stages [Fig. 4(b–d)]. Buchko et al.¹² observed that in a monomeric fluid, a lower viscosity results in the breakup of an electrically driven jet into droplets. Therefore, as shown in Figure 4(a), with a lower nylon concen-

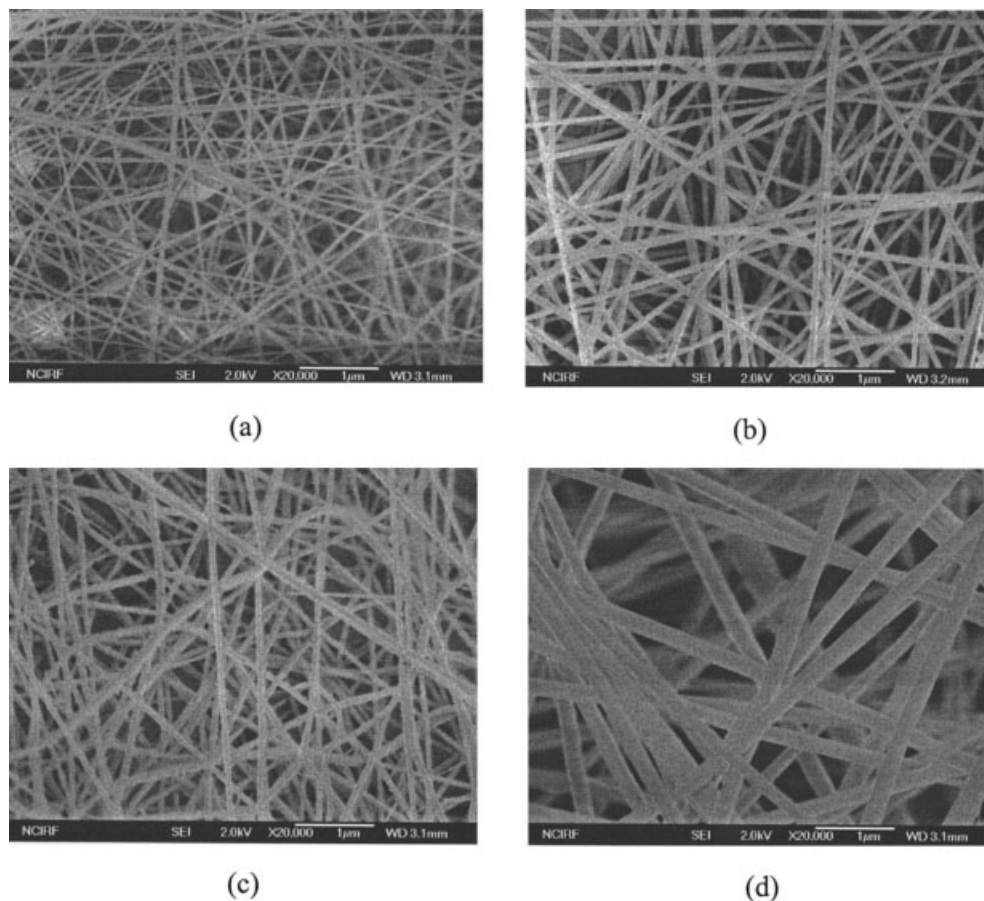


Figure 4 SEM photographs of nylon-6 nanofibers with the following nylon-6 concentrations in formic acid: (a) 14, (b) 18, (c) 20, and (d) 25 wt % (tip-to-collector distance = 10 cm, applied electric power = 25 kV).

tration (14 wt %), the polymer solution was deposited as filaments with beads or hairy beads. However, there were evidently sufficient molecular chain entanglements in a greater than 18 wt % nylon polymer solution to prevent the breakup of an electrically driven jet and to allow the electrostatic stresses to further elongate the jet. On the other hand, when the nylon-6 concentration was increased, the fiber diameters were significantly increased from about 56 nm (at 14 wt %) to about 240 nm (at 25 wt %), as shown in Figure 4. It is assumed that as the nylon-6 concentration in the polymer solution increased, the amount of the nylon-6 polymer in the jet stream increased.¹³

We chose an electrospun fiber web, which was produced at 25 kV, at a 10-cm tip-to-collector distance, from a 25 wt % nylon polymer solution, as a matrix for preparing PANI/nylon-6 composite electrospun fiber webs not only because of the polymer fibers with nanometer-size diameters but also because of the filamentous morphology and the sufficient amount of filament ejection per unit of time.

In situ polymerization of PANI

Effect of the diffusion time and concentration

Diffusion can be defined as the random walk of an ensemble of particles from regions of high concentration to regions of lower concentration. According to Fick's first law [eq. (2)], a concentration gradient of particles gives rise to a particle flux (J_x), which is proportional to the diffusion coefficient (D) and the slope of the molecular concentration ($\partial c/\partial x$):¹⁴

$$J_x = -D\partial c/\partial x \quad (2)$$

In Fick's second law [eq. (3)], a time concept (c_t) is also included in the diffusion factors:¹⁴

$$\partial c_t/\partial t = D(\partial^2 c_t/\partial x^2) \quad (3)$$

From these theories, we know that the time and molecular concentration are important variables that affect the aniline diffusion behavior during the diffusion step. Therefore, we examined the effect of the diffusion time and monomer concentration on the electrical

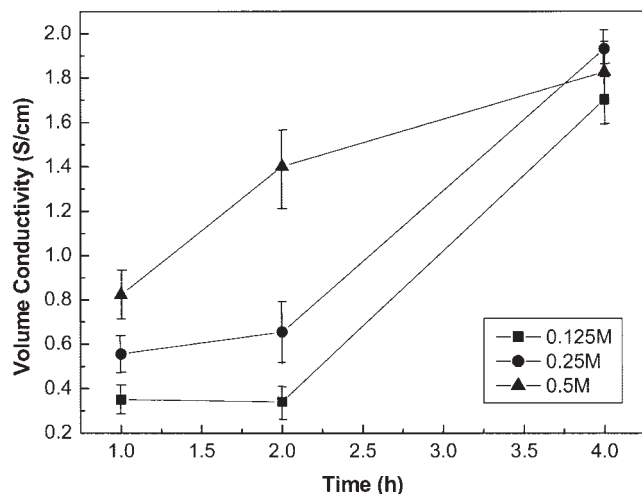


Figure 5 Effect of the diffusion time on the volume conductivity of PANI/nylon-6 composite electrospun fiber webs with the aniline and $(\text{NH}_4)_2\text{S}_2\text{O}_8$ concentrations (diffusion temperature = 40° , diffusion time = 1 h, polymerization temperature = $0-5^\circ\text{C}$, polymerization time = 1 h, aniline/oxidant molar ratio = 1:1).

conductivity of PANI/nylon-6 composite electrospun fiber webs with the optimum diffusion temperature (40°C) and with a ratio of the monomer to oxidant (aniline/APS = 1:1) selected from a previous study.¹⁵

Figure 5 shows that when the diffusion time was short (1–2 h), the volume conductivity increased with increasing aniline concentration, but the gap of the volume conductivities at different aniline concentrations was diminished with increasing diffusion time. On the other hand, Figure 6 shows that the add-on (%)

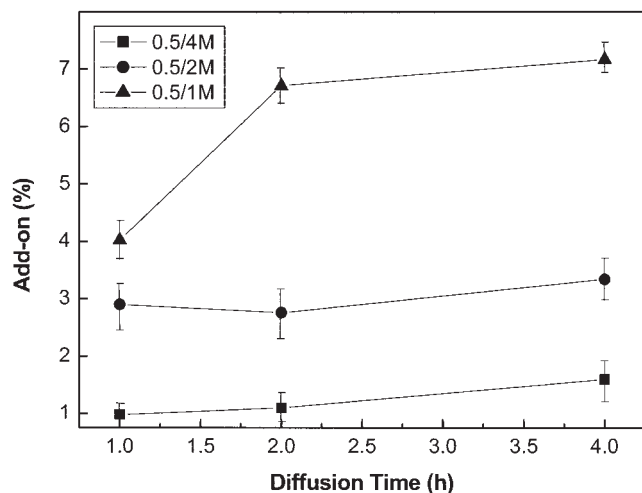
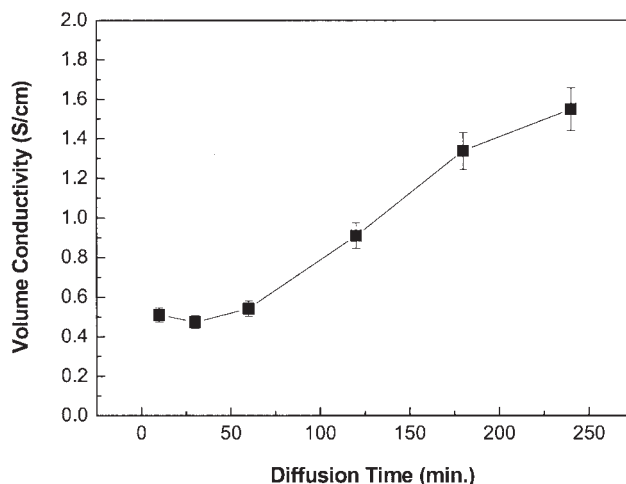
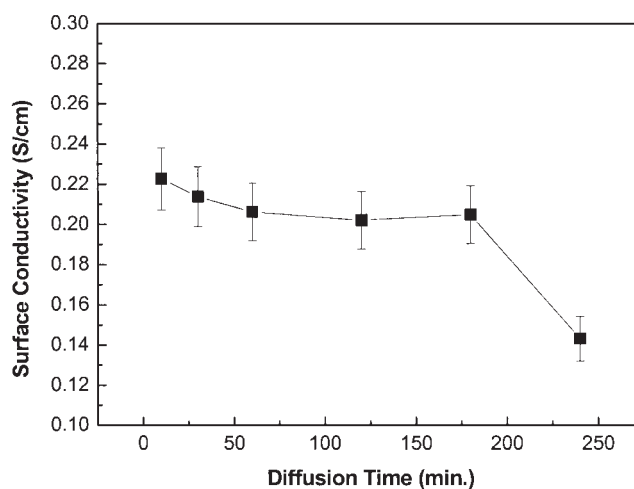


Figure 6 Effect of the diffusion time on the add-on (%) of PANI/nylon-6 composite electrospun fiber webs with the aniline and $(\text{NH}_4)_2\text{S}_2\text{O}_8$ concentrations (diffusion temperature = 40° , diffusion time = 1 h, polymerization temperature = $0-5^\circ\text{C}$, polymerization time = 1 h, aniline/oxidant molar ratio = 1:1).



(a)



(b)

Figure 7 Effect of the diffusion time on (a) the volume conductivity and (b) the surface conductivity of PANI/nylon-6 composite electrospun fiber webs prepared at a 0.25M monomer concentration (diffusion temperature = 40° , polymerization temperature = $0-5^\circ\text{C}$, polymerization time = 1 h, aniline/oxidant molar ratio = 1:1).

of PANI did not change significantly with the diffusion time, with the exception of the early stages with a 0.5M aniline concentration. From these results, we concluded that the volume conductivity of PANI/nylon-6 composite electrospun fiber webs was not affected by just the PANI content in the electrospun fiber webs.

We also compared the volume conductivity and surface conductivity of the PANI/nylon-6 composite electrospun fiber webs with the aniline diffusion time,

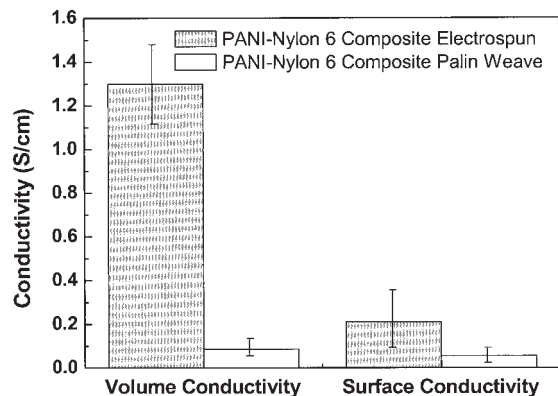


Figure 8 Comparison of the volume and surface conductivities with the matrix structure.

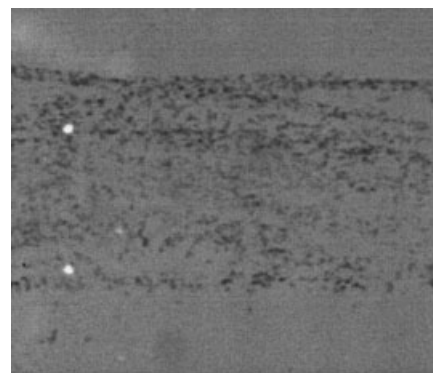
as shown in Figure 7. The volume conductivity increased [Fig. 7(a)], but the surface conductivity was somewhat reduced [Fig. 7(b)], as the diffusion time increased. It was assumed that as the diffusion time increased, aniline monomers were diffused into the electrospun fiber webs more extensively and thus formed PANI deep inside the electrospun fiber webs. Therefore, the extensive diffusion of PANI caused the increase in the volume conductivity of the PANI/nylon-6 composite electrospun fiber webs. However, on the surface of the PANI/nylon-6 composite electrospun fiber webs, an excessive amount of adhesive aniline caused the phase separation of aniline and an aqueous oxidant solution and the gelation of concentrated aniline initiated by contact with the oxidant at the aniline/oxidant solution interface in the polymerization bath.⁹ Also, it was presumed that as the diffusion time increased, the nylon-6 electrospun fiber web matrix was damaged by the acidity in the diffusion solution, and so some alkyl chain oligomers detached from the nylon-6 polymers came off to the surface of the nylon-6 electrospun fiber webs. Therefore, the oligomers acted as impurities or resistants and caused the reduction of the surface conductivity of PANI/nylon-6 composite electrospun fiber webs. On the basis of these results, we concluded that the optimum diffusion time for preparing PANI/nylon-6 composite electrospun fiber webs was approximately 3 h.

On the other hand, the volume conductivity and surface conductivity of PANI/nylon-6 composite electrospun fiber webs, prepared under optimum polymerization conditions, reached up to 1.3 and 0.21 S/cm, respectively, as shown in Figure 8. However, the volume conductivity and surface conductivity of PANI/nylon-6 composite plain-weave fabrics prepared under optimum polymerization conditions were approximately 8.6×10^{-2} and 5.4×10^{-2} S/cm, respectively. The results verified the assumption that the PANI/nylon-6 composite electrospun fiber webs would exhibit excellent electrical conductivities in

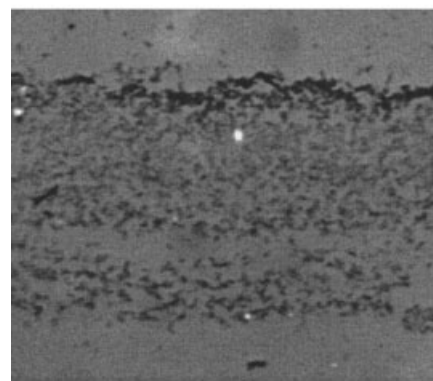
comparison with the other forms of PANI-coated materials because of the high surface-area/volume ratios.

Morphologies of PANI/nylon-6 composite electrospun fiber webs

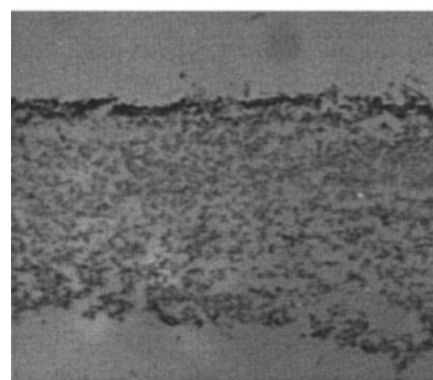
We examined the cross-sectional views of PANI/nylon-6 composite electrospun fiber webs and also ex-



(a)



(b)



(c)

Figure 9 Cross section of PANI/nylon-6 composite electrospun fiber webs prepared at a 0.25M monomer concentration as a function of the diffusion time: (a) 1, (b) 2, and (c) 4 h (diffusion temperature = 40°, polymerization temperature = 0–5°C, polymerization time = 1 h, aniline/oxidant molar ratio = 1:1).

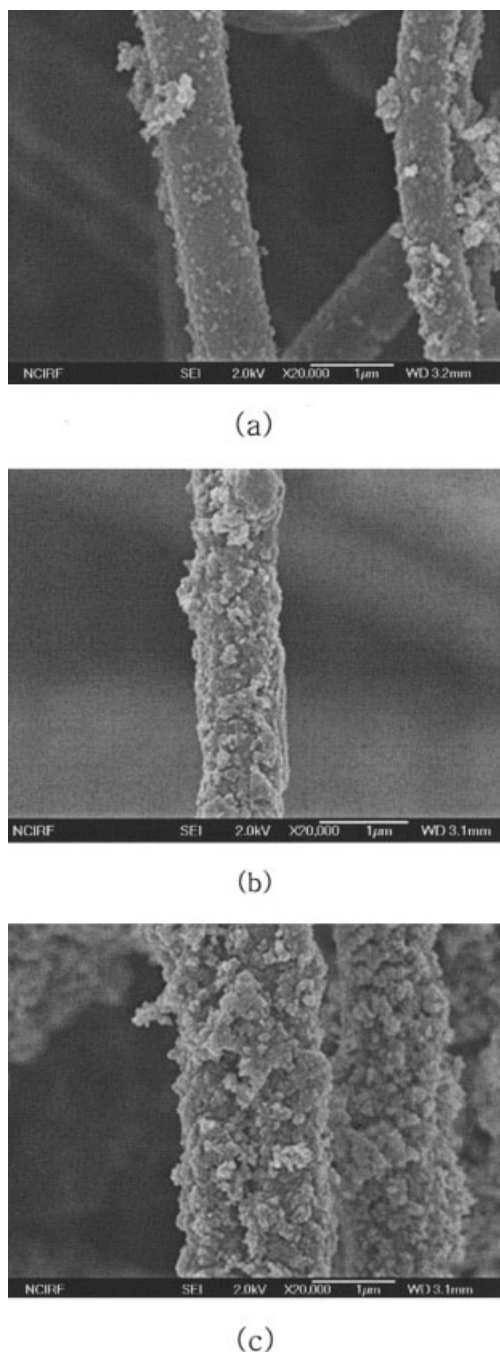


Figure 10 Longitudinal surface view of SEM surface photographs of PANI/nylon-6 composite electrospun fiber webs prepared at a 0.25M monomer concentration as a function of the diffusion time: (a) 1, (b) 2, and (c) 4 h (diffusion temperature = 40°, polymerization temperature = 0–5°C, polymerization time = 1 h, aniline/oxidant molar ratio = 1:1).

amined the longitudinal views of PANI/nylon-6 composite nanofilaments to investigate the sources affecting the volume and surface conductivities of PANI/nylon-6 composite electrospun fiber webs.

With a prolonged diffusion time, deeper penetration and an even distribution of PANI within the

composite electrospun fiber webs were observed, as shown in Figure 9. As the diffusion time increased, the flow amount of the aniline monomers into the electrospun fiber webs increased during the early stage of diffusion. However, with a prolonged time of diffusion, the inflow amount of aniline was reduced, and alternatively the aniline monomers, which were already exhibited in the electrospun fiber webs, migrated and were evenly distributed at the inside of the electrospun fiber webs. Therefore, the views further confirm that the growth of the volume conductivity with increasing diffusion time could be attributed to the diffusion degree and even distribution state of PANI in the PANI/nylon-6 composite electrospun fiber webs.

On the other hand, Figure 10 shows that with increasing diffusion time, the PANI coating became thicker and the surface was less uniform. It is thought that the distinctive changes of the PANI/nylon-6 composite fiber longitudinal morphologies, varying with the diffusion time, mainly occurred in the most external surface of the PANI/nylon-6 composite electrospun fiber webs because the add-on (%) of PANI in the composite electrospun fiber webs did not significantly increase with the diffusion time, as shown in Figure 6. In light of the results of the surface conductivity change with the diffusion time [Fig. 7(b)], the overcoating material adhering on the individual fibers was presumed to be not pure PANI but instead PANI contaminated with some alkyl chains detached from the nylon matrix, aniline monomers, and aniline oligomers, which served as electrical resistants.

Characterization of PANI/nylon-6 composite electrospun fiber webs

For FTIR analysis, we cut and pulverized the PANI/nylon-6 composite electrospun fiber webs into fine

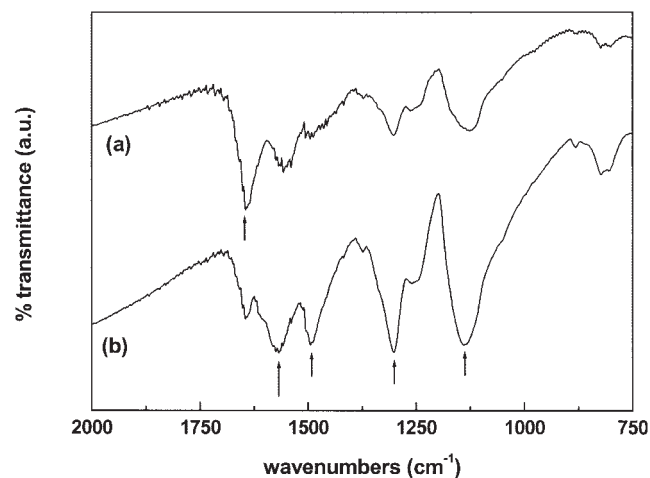


Figure 11 FTIR spectra of PANI/nylon-6 composite electrospun fiber webs with diffusion times of (a) 1 and (b) 4 h.

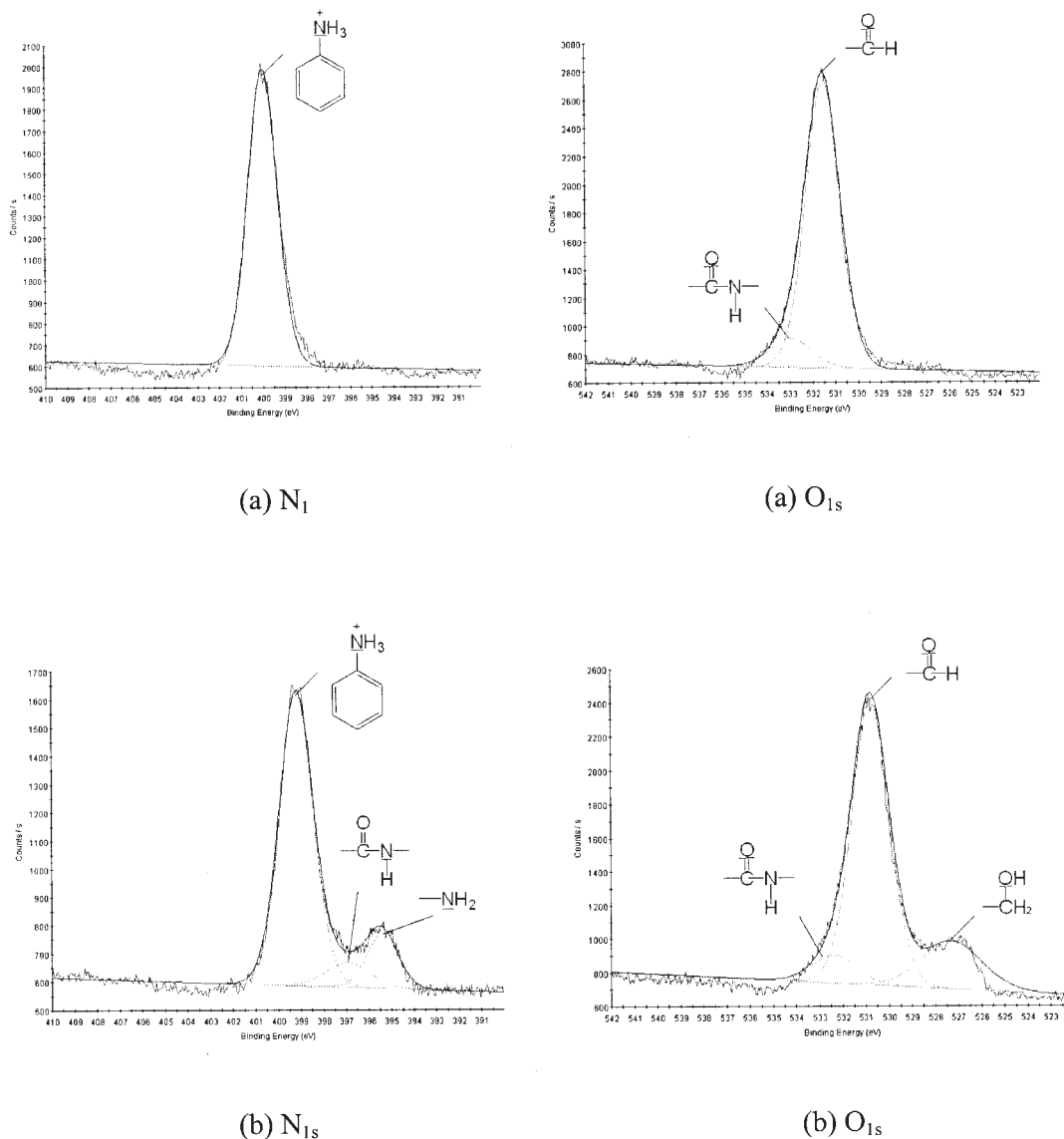


Figure 12 XPS spectra for aniline-diffused nylon-6 electrospun fiber webs with diffusion times of (a) 1 and (b) 4 h.

powders and then made them into KBr pellets. The FTIR spectra for the PANI/nylon-6 composite electrospun fiber webs are shown in Figure 11. The peaks at 1490 and 1568 cm^{-1} were assigned to $C=C$ stretching of the benzenoid and quinoid rings, respectively. The peak at 1301 cm^{-1} was assigned to $C-N$ stretching of the secondary amine of the PANI backbone, and the band at 1139 cm^{-1} was assigned to an in-plane bending vibration of imino-1,4-phenylene. Comparing these spectra with the spectrum of the PANI powder (emeraldine salt form) in the reference,¹⁶ we observed that the peak positions of PANI in the PANI/nylon-6 composite electrospun fiber webs shifted to lower wave numbers. We assumed that PANI formed some composite with nylon 6, and this also indicated that hydrogen bonding was formed between PANI and nylon 6 and may

have resulted in a weakening of the bond strength in PANI and a reduction of the electric conductivity.¹⁶ In particular, because the intensive bending peak of amide $N-H$ in nylon-6 existed around 1550 cm^{-1} , it overlapped with the $C=C$ stretching peak of PANI quinoid rings, and thus the peak near 1570 cm^{-1} was shown to be broad. The peak at 1645 cm^{-1} , assigned to $C=O$ of amide, was also observed because of the nylon-6 electrospun fiber webs. On the other hand, the peaks related to PANI were generally intensified with increasing diffusion time [Fig. 11(b)]; however, the peak of amide $C=O$ stretching that originated from nylon 6 decreased with decreasing diffusion time [Fig. 11(a)]. From these results, we concluded that the amount of PANI formed in nylon-6 electrospun fiber webs increased with increasing diffusion time.

The surface composition of aniline-diffused nylon-6 composite electrospun fiber webs was determined by XPS analysis. Figure 12 presents the XPS curve fitting of N_{1s} and O_{1s} core-level spectra of the nylon-6 electrospun fiber webs, which were just diffused by an aniline/HCl aqueous solution for 1 [Fig. 12(a)] and 4 h [Fig. 12(b)], respectively. When the diffusion time was 1 h, the N_{1s} core-level spectrum [Fig. 12(a)] showed only one peak at 400.0 eV, which corresponded to N^+ in the aniline cation. When the diffusion time was increased to 4 h, the N_{1s} core-level spectrum [Fig. 12(b)] could be deconvoluted into three components: 399.2, 397.2, and 395.6 eV. These peaks were due to the presence of the aniline cation, amide, and inactive amine groups. When the diffusion time was excessive, the nylon matrix was damaged by the acidity of the diffusion solution, and the decomposed fragments of the nylon-6 polymer (some alkyl chains) came off to the surface of the electrospun fiber webs; therefore, inactive $-NH_2$ was detected on the surface of the nylon-6 electrospun fiber webs because of the breakup of the amide group in the nylon-6 electrospun fiber webs. Also, when the diffusion time was 4 h, the O_{1s} spectrum [Fig. 12(b)] could be deconvoluted into three peaks at 532.8, 530.8, and 527.3 eV, respectively. These peaks were due to the presence of amide $C=O$, aldehyde $C=O$, and hydroxy $-OH$, and in particular, the aldehyde $C=O$ and hydroxy $-OH$ could be associated with the decomposition sign of the nylon-6 matrix.

CONCLUSIONS

PANI/nylon-6 composite nanofiber webs (electrospun fiber webs) were prepared via the electrospinning process with a nylon-6/formic acid polymer solution, and then PANI on the surface of the nylon-6 electrospun fiber webs was chemically polymerized. The conductivities of the PANI/nylon-6 composite electrospun fiber webs were superior to those of the PANI/nylon-6 plain-weave fabrics because of their high surface-area/volume ratios. When the electrospinning process parameters were kept at 25 kV and 10-cm deposition, nylon-6 filamentous nanowebs were obtained in greater than 18 wt % polymer solutions. The volume conductivity of the PANI/nylon-6 composite electrospun fiber webs increased, but the surface conductivity decreased, with increasing diffusion time. It was assumed that as the diffusion time increased, a greater diffusion degree and an even distribution state

of PANI in the composite electrospun fiber webs were achieved, and so the volume conductivity was increased. However, the surface conductivity decreased with the diffusion time because the overcoating material, piled up on the surface of the composite electrospun fiber webs, was composed of not only PANI but also unpolymerized aniline monomer, oligomer, and alkyl chains that originated from the damaged nylon matrix. The impurities served as electrical resistants and so interrupted the flux of electrical conductivity. Therefore, for preparing PANI/nylon-6 composite electrospun fiber webs, we found that a 3-h diffusion time was optimum for the aniline monomer diffusion step. On the other hand, from the results of FTIR and XPS analysis, we observed that the PANI content in the composite electrospun fiber webs increased with increasing diffusion time, but when the diffusion time was excessive, the nylon matrix was damaged by the acidity in the diffusion solution, and so the nylon oligomer seemed to flow out to the surface of the electrospun fiber webs.

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