Polyaniline–Nylon 6 Composite Nanowires Prepared by Emulsion Polymerization and Electrospinning Process

Kyung Hwa Hong, Tae Jin Kang

School of Materials Science and Engineering, Seoul National University, Seoul 151–742, Korea

ABSTRACT: Polyaniline (PANI) nanoparticles doped with the dodecylbenzene sulfonic acid (DBSA) were prepared and these nanoparticles were electrospun with nylon 6 as matrix material into fiber web. Depending on the contents and concentrations of PANI and nylon 6, either nylon 6 nanofibers (~96 nm) or PANI-nylon 6 composite nanofibers (~12 nm) were obtained. The electrical conductivity of PANI(DBSA)–nylon 6 electrospun fiber web was lower than that of PANI(DBSA)-nylon 6 film because of the low crystallinity of the PANI(DBSA)–nylon 6 composite electrospun fiber web. However, it showed that the PANI–nylon 6 composite nanofibers would have applications as the nanowires for connecting the microelectromechanical system (MEMS). © 2005 Wiley Periodicals, Inc. J Appl Polym Sci 99: 1277–1286, 2006

Key words: polyaniline (PANI); nylon 6; micellar system; dodecylbenzene sulfonic acid (DBSA); electrospinning; nanofiber; nanowire

INTRODUCTION

In the past few years, the design of composite materials of conducting and insulating phases in the nanometer range has flourished.[1] Particularly, nanofiber fabrication of electrically conducting polymers has recently been demonstrated[2–4] because the conducting nanofibers can be used as nanowires, which are among the key elements in fabrication and connecting the microelectromechanical system (MEMS). Among the conducting polymers, polyaniline (PANI) is regarded as one of the most technologically promising conductive polymer because, the monomer (aniline) is inexpensive, the polymer can be easily prepared by the oxidative polymerization with high yield, and products are stable under ambient conditions.[5]

On the other hand, the electrospinning is a simple fiber-spinning technique that produces polymer fibers with nanometer-to-micrometer size diameter. While the variation in the fiber diameter is large, the fibers prepared using this method typically have diameters much smaller than those attained using standard mechanical fiber-spinning technologies.[6] Therefore, if PANI was introduced into electrospun fibers, electrically conducting nanowires could be fabricated. Pinto et al.[7] prepared micron size diameter fibers of 2-acrylamido-2-methyl-1-propanesulfonic acid doped PANI via electrospinning, but the fiber diameters ranged from 2 to 10 μm and had lower conductivities than the cast films prepared from the same polymer solution. Reneker et al.[8] reported that PANI fibers could be successfully electrospun from sulfuric acid solution into a coagulation bath; however, the conductivity value and mechanical properties of the electrospun fiber were not reported. Also, through our previous study, a PANI-nylon 6 composite electrospun fiber web could be prepared by in situ polymerization of aniline on nylon 6 electrospun fiber web. However, uneven PANI coating was built up on the individual nylon 6 fibers and the PANI diffusion in nylon 6 electrospun fiber web showed ununiformity. For these problems, it has been difficult until now to prepare the conducting nanowire with high electrical conductivity and superior mechanical properties.

Therefore, to improve the electrical conductivity and mechanical properties of PANI composite nanofibers, it was prepared using PANI nanoparticles with anionic surfactants (dodecylbenzene sulfonic acid (DBSA)), and the nanoparticles (PANI(DBSA)) were added in the host polymer solution of nylon 6/formic acid. The nylon 6/formic acid solution dispersed with the PANI(DBSA) nanoparticles was placed in a syringe of electrospinning apparatus and then spun into nanofibers assembly form. We also prepared a free standing film with the same PANI(DBSA)-nylon 6 dispersions and investigated the morphology as well as electrical conductivity of PANI(DBSA)-nylon 6 composite electrospun fiber web comparing it with those of PANI-nylon 6 composite film.

EXPERIMENTAL

Materials and chemicals

Nylon 6 chips with molecular weight of ~23,000 were obtained from Hyosung Co. Ltd. (Anyang, Korea) and

Correspondence to: K. H. Hong (hkh713@snu.ac.kr).
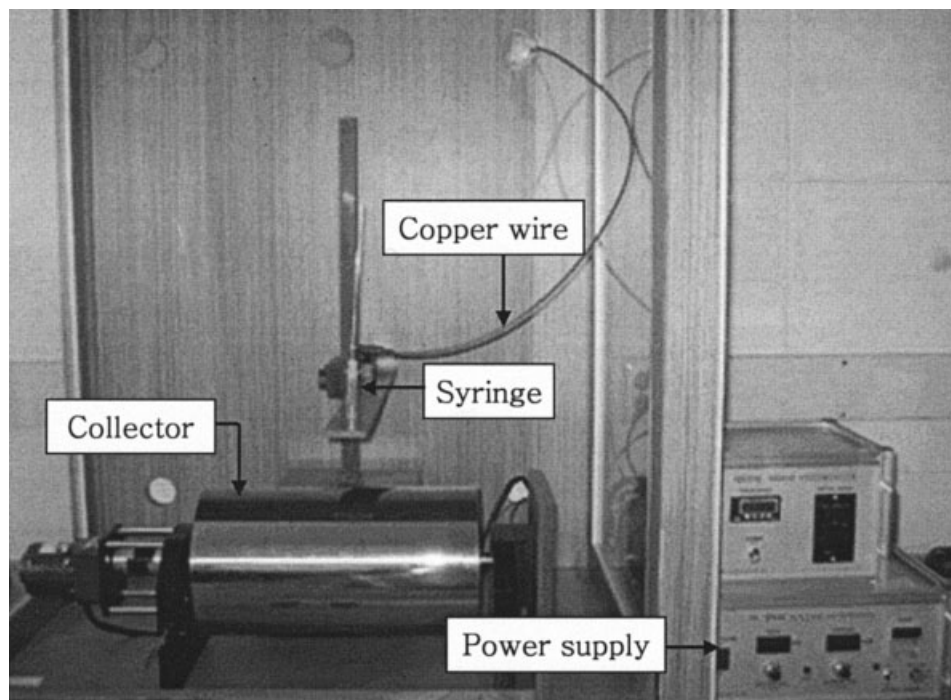
Figure 1 System setup for electrospinning process.

used for preparing nylon 6 nanofiber web through electrospinning process. Aniline (Ducksan Pure Chemical Co. Ltd., Ansan, Korea) was distilled under vacuum prior to use and the other chemicals including ammonium peroxydisulfate (APS) [$(NH_4)_2S_2O_8$], DBSA [$C_{12}H_{25}C_6H_4SO_3H$], and hydrochloric acid [HCl] were purchased from Ducksan Pure Chemical Co. Ltd. (Ansan, Korea) and used with reagent grade.

Preparation of PANI nanoparticles

DBSA aqueous micellar dispersion was prepared by introducing DBSA in 0.1M HCl aqueous solution (100 mL) with stirring. And then aniline monomer + 0.1M HCl aqueous solution (100 mL) was added to the DBSA + HCl aqueous solution. APS used as oxidant (initiator) was separately dissolved in 20 mL HCl (0.1M) aqueous solution, with stirring. After the monomer and surfactant micellar dispersion (aniline + DBSA in 0.1M HCl aqueous solution) was kept with stirring for 1 h, polymerization was performed by the addition of initiator solution (APS in 0.1M HCl aqueous solution) in the micellar solution. The molar ratio of APS to aniline was kept as 1. The color of polymerization solution changed with the polymerization step. In the initial stage, aniline/DBSA/APS/HCl solution was colorless, with high transparency. As the polymerization proceeded, the color of the solution changed to white through yellow, brown, and finally to green, which indicates the formation of PANI emeraldine salt. After the polymerization time of 15 h,

methanol was added to the mixture to stop the polymerization and wash the impurities. The result colloidal dispersion was immediately ultracentrifuged in a centrifuge machine (kontron CENTRIKON T-1180, Italy) at 40,000 rpm for 30 min and separated as deep green sediment. The supernatant was decanted and the sediment was rinsed several times with distilled water. These particles were then dried at 60°C in vacuum or dispersed in formic acid to prevent the aggregation of PANI nanoparticles.

Preparation of PANI-nylon 6 nanowires using electrospinning process

The PANI nanoparticles + nylon 6 dispersion solution was prepared by dissolving nylon 6 chips in formic acid and then blending it with the PANI nanoparticles. The electrospinning apparatus was shown in Figure 1. The hypodermic syringe used in these experiments had a capillary tip diameter of 20 gauge. A positive potential was applied to the PANI(DBSA)-nylon 6 polymer solution by connecting a copper wire to the metal capillary and the potential difference between the syringe and the counter electrode (collector) was 25 kV. A rotating drum covered with aluminum foil, placed 10 cm below the capillary tip, was used to collect the electrospun material.

Evaluation

The electrical volume conductivity of the composite fabric was measured by four probe method. A con-

stant 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. X-ray diffraction (XRD) measurements were performed at room temperature with Cu Kα X-rays (α = 1.5418 Å) with a Scintag XDS 2000 (USA). Theta–theta wide angle goniometer was used and the ultimate peak resolution was $2\theta = 0.025°$. Data were recorded from 5° to 35° in steps of 0.02°. UV absorption spectra were taken with Hitachi U-3300 (Japan) and Shimadzu UV-240 (Japan) in a wavelength range of 300–900 nm with a 1-cm quartz cell. The wavelength accuracy was ±0.3 nm with automatic wavelength correction, and photometric method was double-beam and direct ratio method. The longitudinal morphologies of PANI-nylon 6 composite fabrics were evaluated using a field emission scanning electron microscope (FE-SEM) (JOEL JSM-6340F) after they were coated with gold in vacuum.

RESULTS AND DISCUSSION

Preparation of DBSA doped PANI nanoparticles

To solve the processibility problems of conducting polymers, many research groups have tried to enhance their solubility and moldability by introducing side chains, controlling main-chain architecture, and even designing new monomer types. Other research groups hired various indirect methods such as doping techniques using functional dopants, blending and composite formation, and coating techniques using conducting polymer colloids.[10]

In this research, PANI nanoparticles were prepared in anionic surfactant (DBSA) solution to overcome the limited processibility of the PANI and make a PANI-nylon 6 composite electrospun fiber web through the electrospinning process. On the other hand, we used the HCl aqueous solution as the medium for polymerizing the PANI dispersed with DBSA (PANI(DBSA)) nanoparticles, because we intended to dope the PANI nanoparticles with dopant mixture (DBSA and HCl). Yin et al.[11] reported that the PANI doped with dopant mixture exhibited higher electrical conductivity than the PANI doped with mono dopant did because the PANI doped with dopant mixture had the optimum conjugation length; thereby, polaron and bipolaron in the PANI were more delocalized.

Figure 2 shows the morphologies of PANI(DBSA) nanoparticles prepared with various aniline concentrations when DBSA concentration was fixed at 0.1M. It was observed that the diameters of PANI(DBSA) nanoparticles increased with increasing the molar ratio of aniline to DBSA, as indicated in Figure 3. Also, the shape of the particles transformed from round phase to irregular form, so the flattening rates of the particles were increased with increasing the molar

ratio of aniline to DBSA (Fig. 3). It was supposed that as the molar ratio of aniline to DBSA increased in the polymerization bath, DBSA could not form the micellar system containing aniline monomer to a sufficiently spherical and small phase. Also, the micellar system, constructed by the relatively deficient amount of DBSA, was not firm and thus the shapes of the PANI(DBSA) nanoparticles were thought to be out of round phase. Especially, in case of no DBSA in the polymerization bath, the PANI particles were extremely aggregated and so the size of the clusters of the PANI particles was out of nano-scale level, as shown in Figure 4.

The electrical conductivities of the PANI(DBSA) nanoparticles were investigated by measuring the pellets made of the PANI(DBSA) nanoparticles with the Jandel four point probe. Figure 5 shows the changes of the electrical conductivity and yield of the PANI(DBSA) nanoparticles, which were polymerized in 0.1M HCl + 0.1M DBSA + 0.01M aniline + 0.01M APS aqueous solution at 25°C, with various polymerization times. The reasonable electrical conductivity was obtained at about 4 h polymerization time and the highest conductivity was achieved at about 15 h. However, if the polymerization time was over the 15 h, the electrical conductivity somewhat decreased. On the other hand, the yield was increased steeply until 15 h polymerization time and after then it showed level-off. Therefore, it was concluded that the polymerization time was optimized at about 15 h and excessively prolonged polymerization time presumably made the PANI be over oxidized.

Figure 6 shows that the electrical conductivities of the PANI(DBSA) nanoparticles increases with increasing the molar ratio of aniline to DBSA. However, if the aniline and APS concentrations were same in the polymerization bath, the conductivity of PANI(DBSA) nanoparticle pellet was superior to that of no-DBSA PANI pellet (Fig. 6). It was assumed that with polymerization of anilinium cations into the core of micelle, adjacent DBSA could easily dope together into polymerizing anilinium cations. When the aniline content was decreased, anilinium cations became deficient in comparison with dodecylbenzene sulfate anions. Hence, the competition effect or molecular attraction between the adjacent DBSA molecules made it difficult to perform doping to polymerizing aniline into the micelle, which could be the cause of relatively low doping level of the PANI particles.[12]

On the other hand, we decided that the PANI(DBSA) nanoparticles for preparing PANI-blended nylon 6 electrospun fiber web would be polymerized in 0.1M HCl + 0.1M DBSA + 0.01M aniline + 0.01M APS aqueous solution bath for 15 h at 25°C. Because although the electrical conductivity of the PANI(DBSA) nanoparticles were not most superior to those of other particles prepared in other concentration solutions
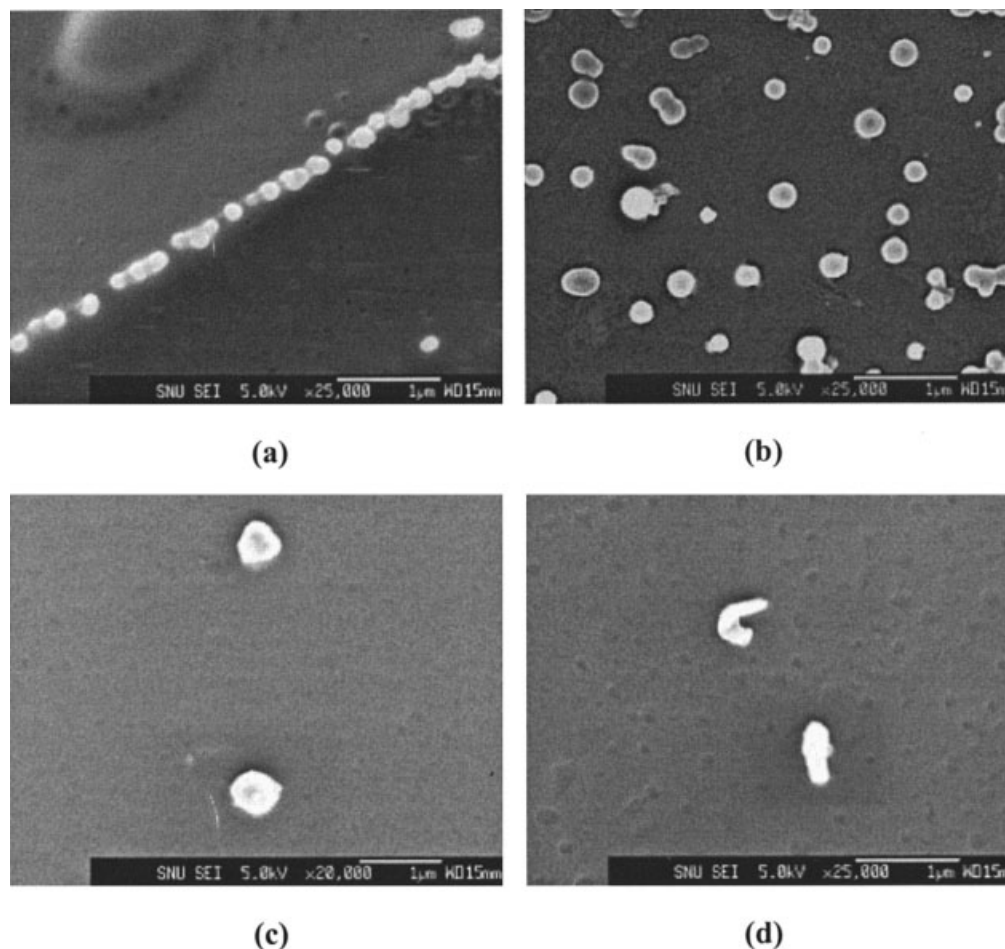
Figure 2 SEM surface photographs of PANI(DBSA) particles with various aniline concentrations. DBSA concentration: 0.1 *M*; polymerization temperature/time: 25°C/15 h; molar ratio of aniline : oxidant = 1 : 1; aniline concentration: (a) 0.01*M*, (b) 0.025*M*, (c) 0.07*M*, (d) 0.10*M*.

(Fig. 6), the conductivity level of the PANI(DBSA) nanoparticles were similar to those of other particles, with the exception of the particles prepared in 0.1*M* HCl + 0.1*M* DBSA + 0.1*M* aniline + 0.1*M* APS. In particular, the PANI(DBSA) nanoparticles (prepared in 0.1*M* HCl + 0.1*M* DBSA + 0.01*M* aniline + 0.01*M* APS aqueous solution) showed small and spherical phase and thus, we thought that the particles were suitable to prepare the PANI nanoparticles imbedded nylon 6 electrospun nanofibers.

The green-colored PANI(DBSA) colloidal dispersion had a good long-term colloidal stability and existed as stable solution, without precipitations over several months. The doping states of the PANI(DBSA) dispersion in aqueous solution with various pH values were observed by UV spectra, as shown in Figure 7. Figure 7(a) shows the UV-VIS spectra of the solution prepared by dispersing the PANI(DBSA) colloidal particles in aqueous solution having pH 1. The absorption peaks at 350, 430, and 742 nm could be assigned as the π–π* transition of benzenoid segment, polaron band, and localized polaron band of doped PANI,

respectively.[12] Figure 7(c) shows the UV-VIS spectra of the solution prepared by dispersing the PANI(D-BSA) colloidal particles in aqueous solution having pH 14, and the absorption peaks at 325 and 630 nm agreed with the undoped PANI.[13] However, when the PANI(DBSA) colloidal particles were dispersed in pH 7 aqueous solution [(Fig. 7(b)], the π–π* transition of benzenoid peak and the polaron peaks still existed. This result showed that the transition from emeraldine salt to emeraldine base form occurred above pH 7 in this PANI(DBSA) micellar system. However, it was reported that the metal-insulator transition of conventional PANI was at about pH 4.[14] Hence, it was thought that when the PANI was doped and complexed with DBSA, the anionic environmental effect might lead to the higher transition pH from doping state to dedoping state.[15] Therefore, it has been found that the PANI(DBSA) colloidal dispersion was doped state in an acidic solution and the dedoping reaction of the PANI(DBSA) colloidal dispersion successfully occurred in a strongly alkali solution. However, the doping state of PANI(DBSA) colloids was relatively stable
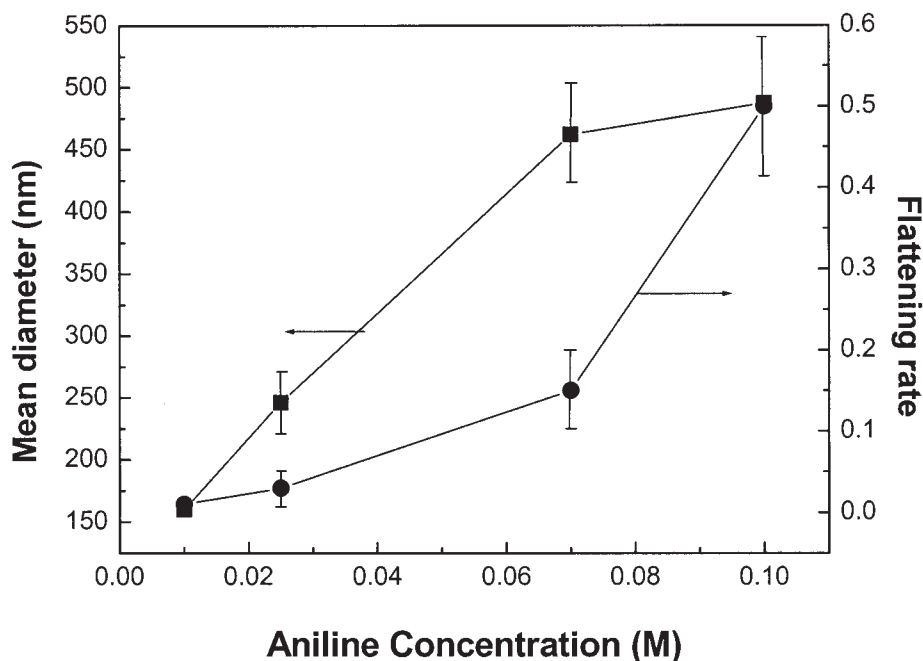
Figure 3 Mean diameter and flattening rate of PANI(DBSA) particles with various aniline concentrations. DBSA concentration: 0.1M; polymerization temperature/time: 25°C/15 h; molar ratio of aniline : oxidant = 1 : 1.

in a neutral solution and even in a weak alkali solution. From this result, it could be concluded that the PANI(DBSA) colloidal form would show the excellent environment stability when compared with that of the conventional PANI.

Preparation of PANI-nylon 6 electrospun fiber web

In recent years, nano-scale devices have been attracting considerable attention because of the demands for ultra-large-scale integration technology. Conducting

Figure 4 SEM surface photograph of PANI particles with no surfactant added. Polymerization temperature/time : 25°C/15 h; molar ratio of aniline : oxidant = 1 : 1; aniline concentration : 0.01M.

nanowires are among the key elements in fabricating and connecting the devices. Therefore, nanowires have been studied intensively, and particularly, those associated with carbon nanotubes and conducting polymers. Among conducting polymers, PANI has many advantages, as mentioned earlier. However, the PANI prepared by routine methods usually shows a granular morphology or fibers entangled together. Therefore, in this experiment, a novel conducting PANI nanofiber network was prepared through electrospinning process with a DBSA doped PANI + nylon 6 chips/formic acid blending (PANI(DBSA)-nylon 6) solution.

We successfully obtained the PANI(DBSA)-nylon 6 nanofibers, using electrospinning technique. Figure 8 shows the photographs of PANI(DBSA)-nylon 6 electrospun fiber web taken with various PANI and nylon contents in formic acid solution. The solids concentrations in all PANI(DBSA)-nylon 6 polymers were kept at 25 wt %. When the concentration of PANI(DBSA) nanoparticles was from 2 to 8 wt %, the PANI(DBSA)-nylon 6 electrospun nanofibers were composed of two kinds of phases. It was thought that one part was PANI(DBSA) nanofibers (few tens nm size diameter) and the other part was pristine nylon 6 nanofibers (~100 nm size diameter). When the concentration of PANI(DBSA) nanoparticles was over 12 wt %, the PANI(DBSA)-nylon 6 electrospun nanofibers had only one-type phase, which was PANI(DBSA)-nylon 6 composite nanofibers; however, the fibers were severed into the length of few micrometers and many
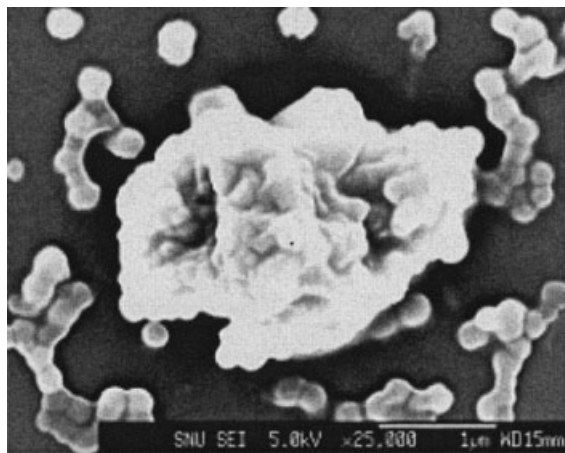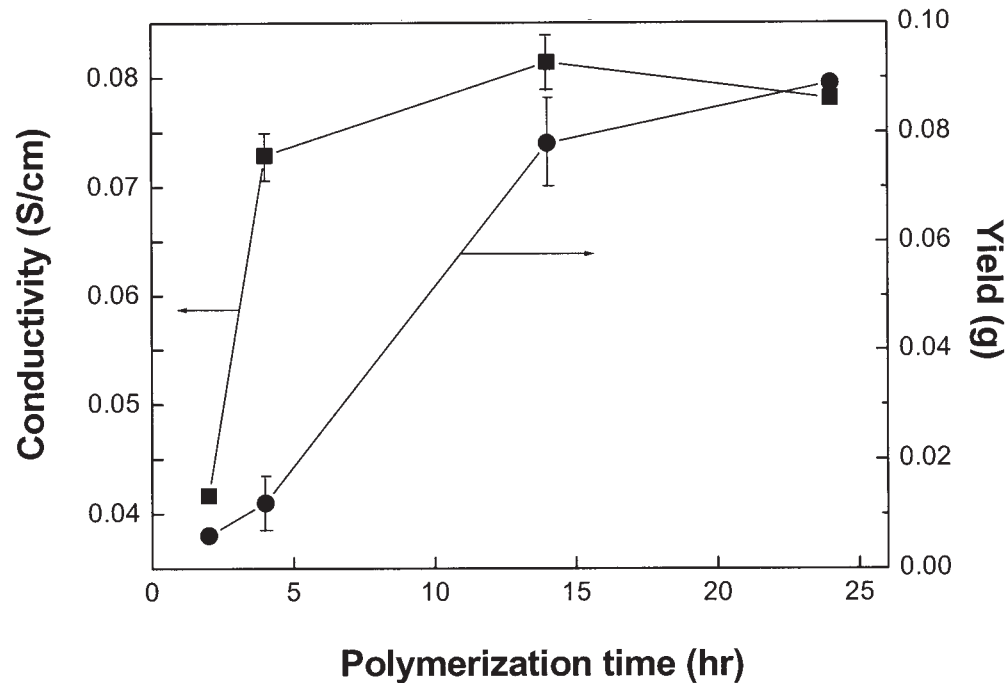
Figure 5 Effect of polymerization time on the electrical conductivity and yield of PANI(DBSA) powder pellets. DBSA concentration: 0.10*M*; aniline concentration: 0.01*M*, polymerization temperature: 25°C; molar ratio of aniline : oxidant = 1 : 1.

disconnected parts were observed as shown in Figure 8(c). It was assumed that the excess amount of PANI(DBSA) nanoparticles in nylon 6/formic acid solution caused the fibers' defects.

Figure 8(b) and Figure 9 show photographs of a PANI(DBSA)-nylon 6 electrospun fiber web (prepared from 8 wt % + 17 wt % nylon 6 in formic acid solution) taken with various magnifications. However, Figure
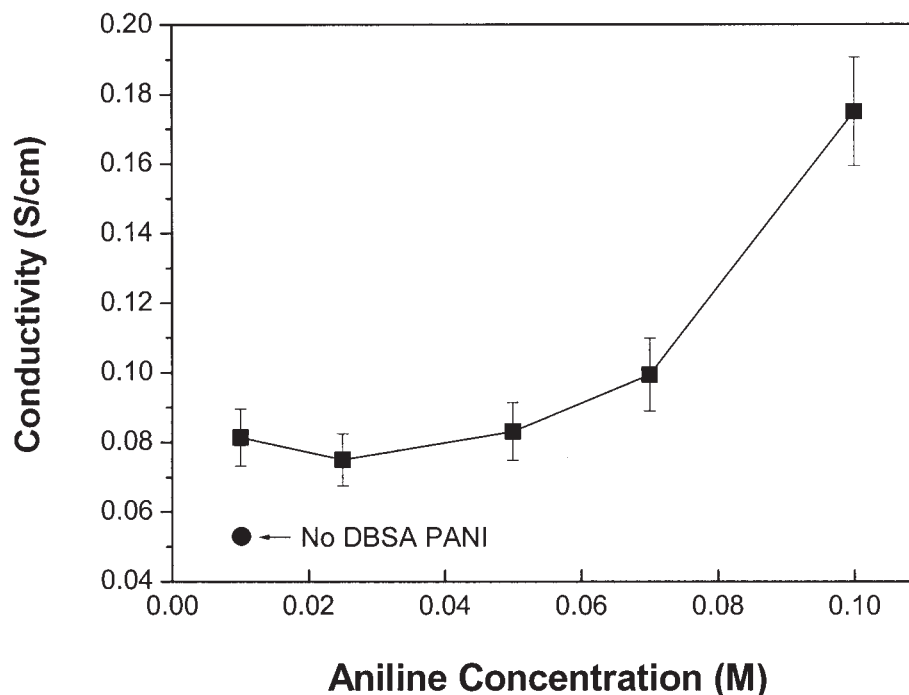

Figure 6 Effect of aniline concentration on the electrical conductivity of PANI(DBSA) powder pellets. DBSA concentration: 0.10*M*; polymerization temperature/time: 25°C/15 h; molar ratio of aniline:oxidant = 1 : 1.
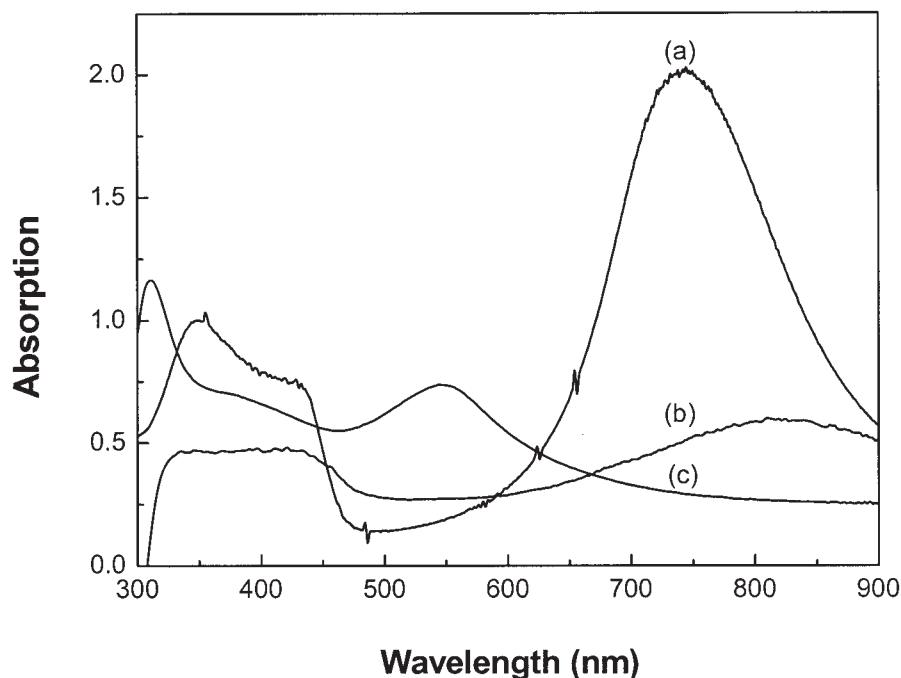
Figure 7 UV-VIS spectra of PANI(DBSA) colloidal solution. DBSA concentration: 0.1 0M; aniline concentration: 0.01M; polymerization temperature/time: 25°C/16 h; molar ratio of aniline : oxidant = 1 : 1; dispersed in aqueous solution having pH 7(a), dispersed in aqueous solution having pH 7 (b), dispersed in aqueous solution having pH 14 (c).

9(a,b) taken from different places in the sample display different morphologies, which indicate that the sample is not uniform. The overall morphology of the PANI(DBSA)-nylon 6 electrospun fiber web was very interesting. It consists of two parts; one was thick (~96 nm) and smooth surface filaments part, which was supposed as pure nylon 6 filaments. And the other was thin (~12 nm) and three-dimensional network filaments web, which was assumed the PANI(DBSA)-nylon 6 nanofibers. Especially, the fine filaments had spherical nodes and the PANI(DBSA) nanoparticles shown in Figure 2(a) were presumably divided into even smaller size by electric power. On the other hand, the morphology of Figure 9(b) is separated into PANI balls 40–60 nm in size; this means that the PANI nanoparticles were agglomerated. That is, if the solvent evaporation rate of nylon 6/formic acid solution was much slower than aggregation rate of PANI(DBSA), the clusters of PANI(DBSA) colloidal particles were formed [Fig. 9(b)]. On the contrary, if the solvent evaporation rate of nylon 6/formic acid solution was much faster than the aggregation rate of PANI(DBSA) nanoparticles, nylon 6 solution formed the thick and smooth surface filaments, as if the nanofibers web had been electrospun from pure nylon 6/formic acid solution. It showed the possibility that if these two speeds were comparable, three-dimensional networks with thin diameter and spherical nodes were produced. This result was similar to that of He et al.[16] They reported that as controlling the crystalline growth rate

of surfactant matrix or aggregation rate of PANI(anionic surfactant), various morphologies of PANI(anionic surfactant) could be obtained.

The electrical conductivity of PANI(DBSA) particles pellet (~4.27 × 10^{-2} S/cm) was the highest and then PANI(DBSA) nylon 6 film (~1.68 × 10^{-4} S/cm) > PANI(DBSA)-nylon 6 electrospun fiber web (~6.19 × 10^{-7} S/cm) in decreasing order, as shown in Figure 10. Therefore, the electrical conductivity of PANI(DBSA)-nylon 6 electrospun fiber web almost belonged to the range of semiconductor, which might be a level for antistatic clothing materials. However, sometimes the electrical conductivity of the PANI(DBSA)-nylon 6 electrospun fiber web could not be measured because the PANI(DBSA)-nylon 6 electrospun fiber web was not uniform, as shown in Figure 9. Also, we thought that it is not suitable to measure the electrical conductivity of the PANI(DBSA)-nylon 6 composite electrospun nanofibers respectively, by using the existing test method. Therefore, if it were possible to measure the electrical conductivity of the only PANI(DBSA)-nylon 6 composite filaments piece by piece, it would be expected that the electrical conductivity value of the PANI(DBSA)-nylon 6 composite electrospun nanofibers would be improved. And also, it was assumed that the low degree of PANI crystallinity in the PANI-nylon 6 composites affected the low electrical conductivity of PANI(DBSA)-nylon 6 composite electrospun fiber web, because Chaudhari and Kelkar[16] reported that as increasing the crystallinity of PANI sample, its
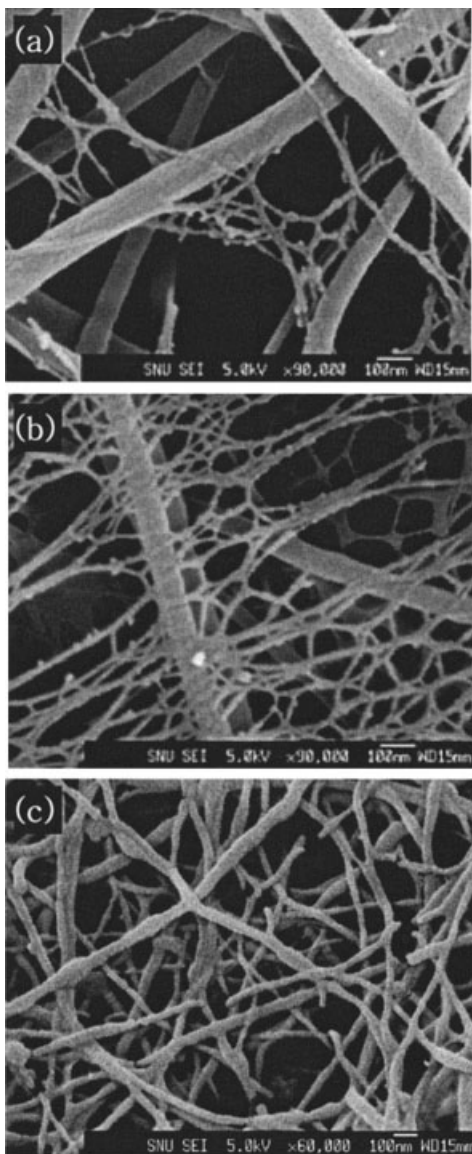
Figure 8 SEM surface photographs of DBSA doped PAM-nylon 6 blending electrospun fiber web, tip to collector distance: 10 cm, electric power: 25 kV; polymer solution: 2 wt % PANI + 23 wt % nylon 6 (a), S wt % PANI + l7 wt % nylon 6 (b), and l2 wt % PANI + l3 wt % nylon 6 (c) in formic acid solution.

electrical conductivity was improved. The XRD patterns of PANI(DBSA)-nylon 6 film [Fig. 11(a)], PANI(DBSA)-nylon 6 electrospun fiber web [Fig. 11(b)], and PANI(DBSA) powder [Fig. 11(c)] were obtained, as shown in Figure 11. Figure 11(c) reveals that the PANI has degree of crystallinity; three broad peaks appear in the region of $2\theta = 5°–30°$, with a maximum around 25° for PANI chains at interplanar spacing.[18] When the PANI(DBSA)-nylon 6 composite solution was electrospun by electric power, the overall crystallinity of the composite polymer decreased [Fig. 11(b)] and thus, the PANI crystallinity would be less. This could be attributed to the rapid evaporation of the

solvent during electrospinning process, resulting in an amorphous rather than a crystalline structure in the fiber as could have been the case in the cast film, where the solvent evaporated slowly compared with that of the electrospun leading to larger crystalline regions.[19]

CONCLUSIONS

The DBSA doped PANI-nylon 6 blending (PANI(DBSA)-nylon 6) electrospun fiber web was prepared. The PANI(DBSA) nanoparticles were polymerized in $0.1M$ HCl + $0.1M$ DBSA + $0.01M$ aniline + $0.01M$ APS aqueous solution bath for 15 h, with stirring. When the contents and concentrations of the PANI and nylon 6 were compatible (PANI: 8 wt %, nylon 6.17 wt % in formic acid), the obtained PANI(DBSA)-nylon 6 electrospun fiber web showed two different morphologies; One was the pure nylon 6 nanofibers (~96 nm) and the other was PANI-nylon 6 composite nanofibers (~12 nm), which can be used as nanowires for con-
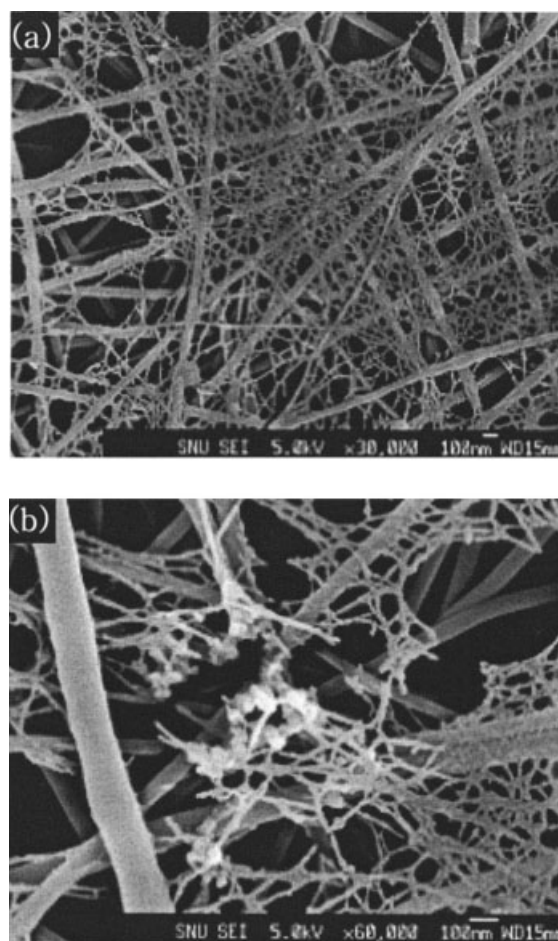

Figure 9 SEM surface photographs of DBSA doped PANI-nylon 6 blending electrospun fiber web. Tip to collector distance: 10 cm, electric power: 25 kV; polymer solution: S wt % PAM + l7 wt % nylon 6 in formic acid solution
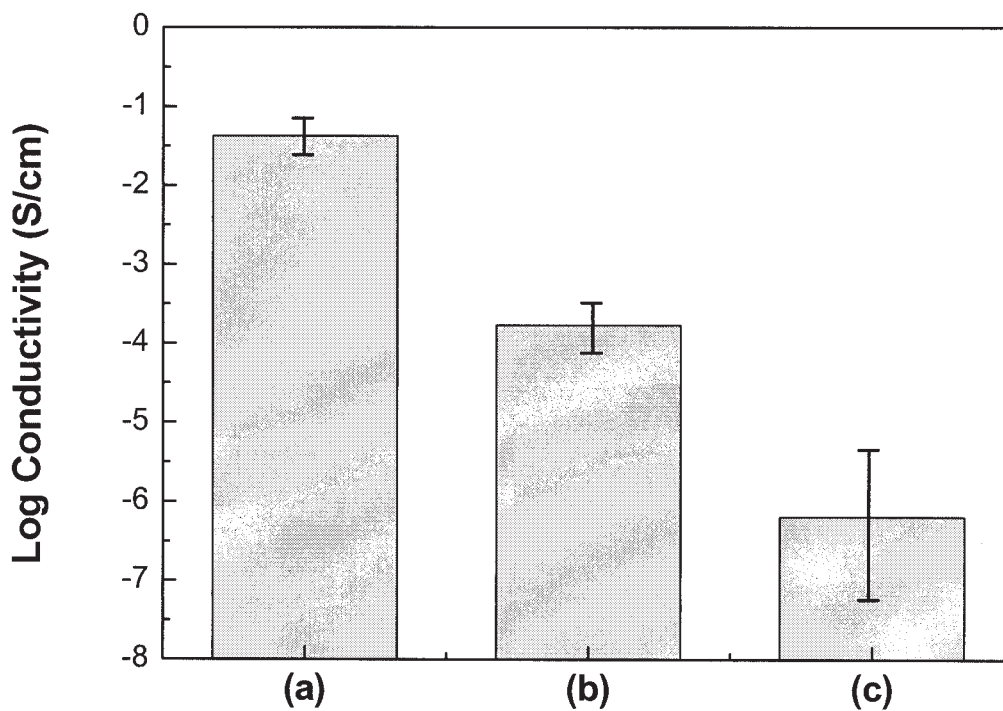
Figure 10 Log conductivity (S/cm) of PANI(DBSA) pellet (a), PANI(DBSA)-nylon 6 film (b), and PANI(DBSA)-nylon 6 electrospun fiber web (c). Tip to collector distance: 10 cm, electric power: 25kV; polymer solution: 8 wt % PANT + l7 wt % nylon 6 in formic acid solution.
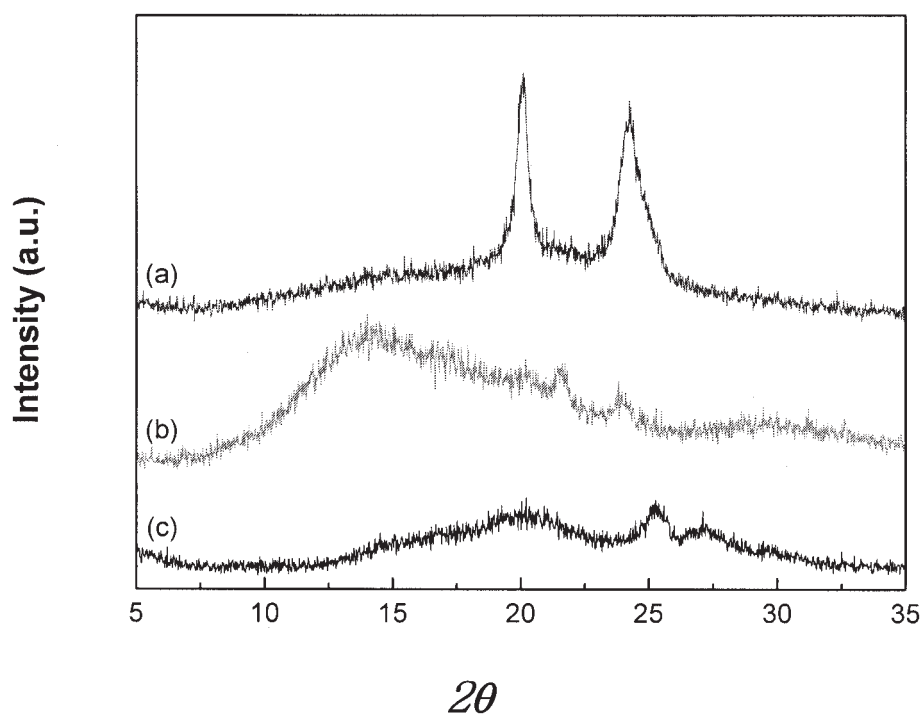

Figure 11 XRD patterns of PANI(DBSA)-nyton 6 film (a), PANI(DBSA)-nylon 6 electrospun fiber web (b), and PANI(DBSA) powder (c). Tip to collector distance; 10 cm, electric power: 25 kV; polymer solution: 8 wt % PANI + l7 wt % nylon 6 in formic acid solution.

necting the MEMS. However, the electrical conductivity of PANI(DBSA)-nylon 6 electrospun fiber web was inferior to that of PANI-nylon 6 cast film. It was assumed that the existing method for measuring electrical conductivity was not suitable to measure the electrical conductivity of the nanofilaments piece by piece, and also, the crystallinity of whole PANI(DBSA)-nylon 6 electrospun fiber web was lower than that of PANI(DBSA)-nylon 6 film. This could be attributed to the rapid evaporation of the solvent during electrospinning process, resulting in an amorphous rather than a crystalline structure in the fiber.

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