

# Processable Conductive Blends of Polyaniline/Polyimide

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**ABSTRACT:** By using camphorsulfonic acid (CSA) to protonate polyaniline (PANI), the counterion enabled the PANI-CSA complex processable as a solution phase. So camphorsulfonic acid (CSA)-doped polyaniline/polyimide (PANI/PI) blend films were prepared by the solvent casting method using *N*-methylpyrrolidinone (NMP) as a cosolvent followed by thermal imidization. The conductivity of the PANI-CSA/PAA (50 wt % PANI content) is greater than that of the pure PANI sample at room temperature. As the thermal imidization proceeded, molecular order of polymer chain structure was improved in the resulting PANI-CSA/PI film due to the annealing effect of PANI chain, and this PANI-CSA/PI film showed higher conductivity than PANI-CSA and PANI-CSA/PAA film. PANI-CSA/PI blend films had a good thermal stability of conductivity at high temperature. © 1998 John Wiley & Sons, Inc. *J Appl Polym Sci* **67**: 1863–1870, 1998

**Key words:** polyaniline; polyamic acid; polyimide; blends; conductivity

## INTRODUCTION

Polyaniline (PANI) has been generally categorized as an intractable polymer because of the difficulty in thermal or solution processing. However, the emeraldine base form of polyaniline has been found to be soluble in aqueous acetic acid, formic acid, dimethylsulfoxide, dimethylformamide, *N*-methylpyrrolidinone (NMP), concentrated sulfuric acid, and other strong acids,<sup>1–3</sup> but their solubilities are very low. Many researchers have attempted to solve this problem. They reported that substitution of aromatic ring of polyaniline with  $-\text{CH}_3$ ,  $-\text{OCH}_3$ ,  $-\text{SO}_3$ , or long alkyl chain led to higher solubility in organic solvents because of lessening of polymer chain stiffness and interaction between polymer chains; consequently, it made the improvement of processibility.<sup>4–8</sup> Still, it has been impossible to dissolve PANI in the conducting emeraldine salt form. In addition to this problem, steric hindrance

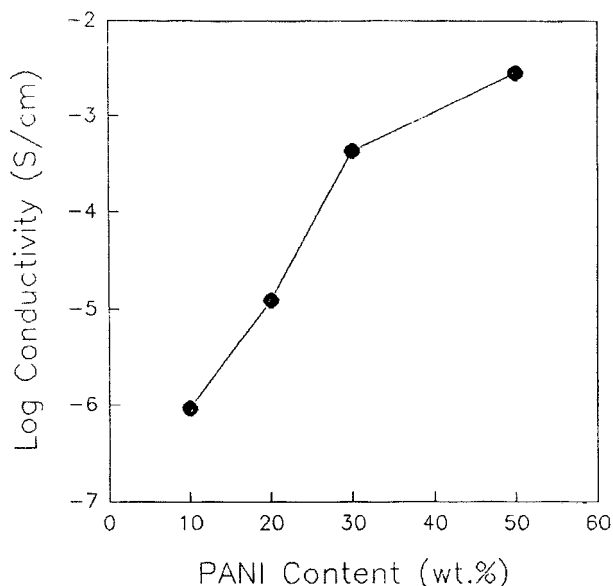
results from the substituents induced, the increase in interchain length, and the decrease in conjugation length, all of which caused the conductivity decrement.

However, recently, Cao et al.<sup>9,10</sup> reported the use of functionalized protonic acid both to dope PANI and, simultaneously, to render the resulting PANI complex soluble, in the conducting emeraldine salt form, in common nonpolar or weakly polar organic solvents in which most commercial polymers can be codissolved (counter-ion induced processability). Thus, processing of emeraldine salt of PANI in common organic solvents is enabled by doping with functionalized protonic acids like camphorsulfonic acid (CSA) and dodecylbenzenesulfonic acid (DBSA), etc.

The ability to achieve conducting polyaniline complexes that are soluble in specific common organic solvents enables the creation of a variety of conducting polyblends in which polyaniline is blended with bulk polymers. The fundamental requirement for creating conducting polyblends is the need for a solvent in which both the conducting polyaniline complex and desired bulk polymers are cosoluble. Given such a solvent, conduct-

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**Figure 1** Conductivity of PANI–CSA/PAA blends with PANI content.

ing polyblends can be made by codissolving the PANI complex and the bulk polymer at concentrations such that when cast from solution, the resulting blend will have the desired ratio of conducting PANI complex to bulk polymer. Therefore, the conducting polyblend material can be a solution-processable material.

In this research, solution blending was employed with a PANI–CSA complex and polyimide (PI) based on the properties of polyimides, which are high-temperature stable polymers that have a great applications in the microelectronics due to their excellent thermal stability, high chemical resistance, superior mechanical properties, and easy processability. Furthermore, NMP, which is the traditional solvent of PANI, is a good solvent of polyamic acid, which is a precursor polymer of polyimide. Molecular orientation property of polyimide is possible to induce chain orientation in the blend system, hence, resulting in increasing conductivity of the blends. Therefore, we report here our observation of electrical properties of polyaniline–polyimide blend films with pyromellitic dianhydride (PMDA) and 4,4'-oxydianiline (ODA) as a polyamic acid material and PANI–CSA as a conducting material.

## MEASUREMENTS

The electrical conductivity of the blend films was measured with the standard four-probe method;

for measuring the conductivity variation with temperature, the four-probe cell was inserted in a glass tube, and the heating rate was 3°C/min. The ultraviolet–visible (UV–vis) absorption spectra of the blend films were recorded with UNICAM 8700 series UV–vis spectrophotometer. Wide angle X-ray diffraction (WAXD) patterns were taken with Ni-filtered CuK $\alpha$  radiation using Rigaku Denki RAD-C 4037A1. Fourier transform infrared (FTIR) analysis was done using Prospect-IR (MIDAC) with NaCl cell. Thermogravimetric analysis (TGA) thermograms were measured by Perkin–Elmer TGA-7. Temperature range was from 30–800°C with heating of 10°C/min.

## EXPERIMENTAL

### Chemicals

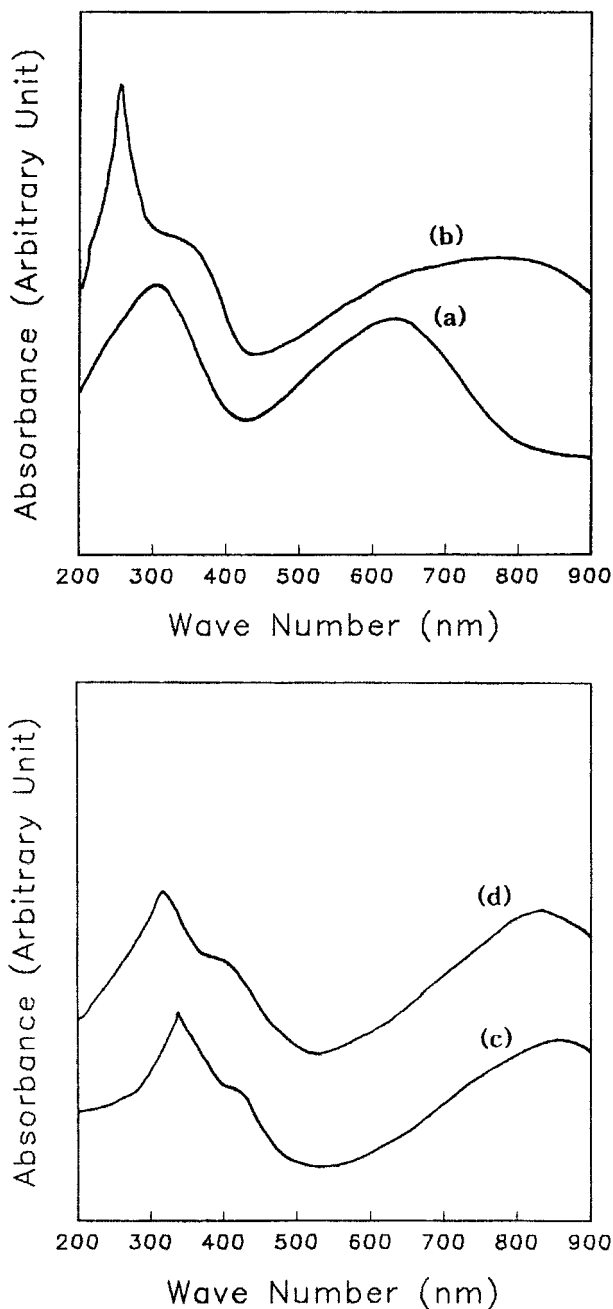
Aniline (Aldrich) was purified by vacuum distillation before use and ammonium peroxydisulfate [(NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub>], HCl, NH<sub>4</sub>OH, and (1S)-(+)-10-camphorsulfonic acid (CSA, from Aldrich) were used as received. 4,4'-oxydianiline (ODA, from TCI) and pyromellitic dianhydride (PMDA, from TCI) were recrystallized. *N*-methyl-2-pyrrolidone (NMP, from Aldrich) was used as a cosolvent.

### Preparation of Polyaniline–Polyimide (PANI–PI) Blends

Polyaniline emeraldine base (PANI–EB) powder was prepared by conventional chemical method.<sup>11</sup> Inherent viscosity of 0.1 wt % PANI–EB in NMP at 25°C was 0.26 dL/g. Emeraldine base powder

**Table I** Conductivity Data of Various Materials

Materials	Conductivity (S/cm)
PANI–CSA powder	$2.65 \times 10^0$
PANI–CSA film	$1.23 \times 10^{-3}$
PANI–CSA/PAA (10% PANI content)	$9.26 \times 10^{-7}$
PANI–CSA/PAA (20% PANI content)	$1.25 \times 10^{-5}$
PANI–CSA/PAA (30% PANI content)	$4.33 \times 10^{-4}$
PANI–CSA/PAA (50% PANI content)	$2.82 \times 10^{-3}$
PANI–CSA/PI (10% PANI content)	$1.46 \times 10^{-7}$
PANI–CSA/PI (20% PANI content)	$8.62 \times 10^{-6}$
PANI–CSA/PI (30% PANI content)	$4.01 \times 10^{-4}$
PANI–CSA/PI (50% PANI content)	$5.05 \times 10^{-3}$



**Figure 2** UV-vis spectra: (a) PANI-EB, (b) PANI-HCl, (c) PANI-CSA, and (d) PANI-CSA/PAA.

was mixed with CSA in a molar ratio of 0.5 CSA to polymer repeat unit phenylnitrogen. The resulting mixture was dissolved in NMP and then treated in an ultrasonic bath. The solubility of PANI-CSA to NMP was only about 2–3 wt %.

Polyamic acid was polymerized by following method.<sup>12</sup> First, 3.804 g of ODA (0.019 mol) was dissolved in NMP (24.27 mL) under N<sub>2</sub> purging at room temperature with stirring. 4.36 g of PMDA

(0.02 mol) was added into the ODA solution. After 12 h, polyamic acid (PAA) was prepared, and PANI-CSA/PAA solution was prepared by blending PANI-CSA solution with PAA solution at various PANI contents (10, 20, 30, and 50 wt %). PANI-CSA/PAA films were prepared by solvent casting at 50°C under vacuum. These PANI-CSA/PAA and pure PAA films were converted to polyimide by thermal imidization process.

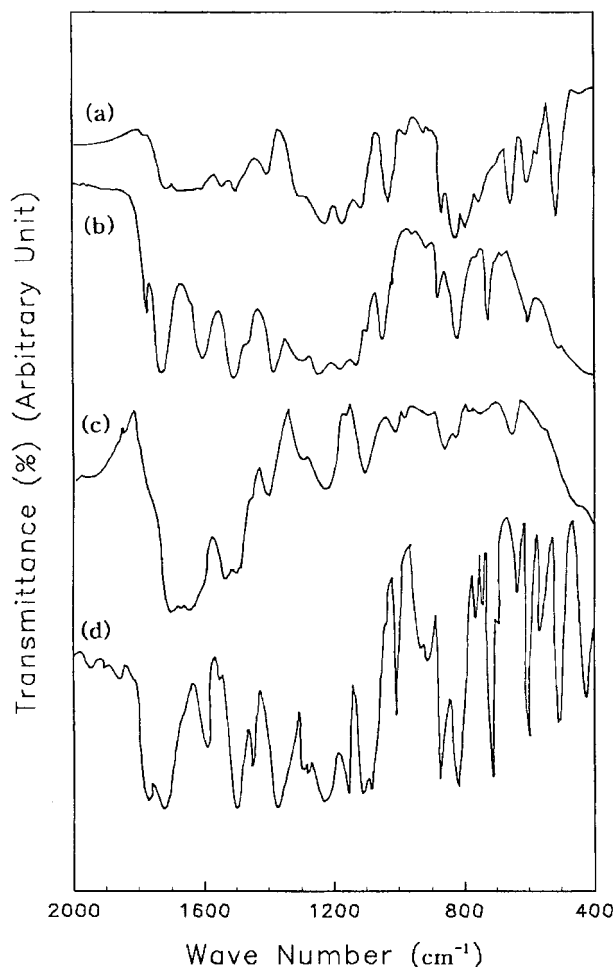
PANI-CSA film was prepared by solvent casting the PANI-CSA solution in the same way as PANI-CSA/PAA.

## RESULTS AND DISCUSSIONS

The electrical conductivity of the blends versus the weight fraction of polyaniline is plotted in Figure 1. The percolation threshold seems to be around 20 wt % PANI content. Less than 10 wt %, the conductivity of the blends was not measurable. The conductivity of the blends increases as the PANI content increases from 10 up to 30 wt %, but at more than 30%, it shows similar or somewhat higher conductivity than that of pure PANI-CSA film prepared from NMP solvent and shows low slope of the increase of the conductivity as the PANI content increased. Detailed conductivity data of all samples are listed in Table I.

The powder sample has much higher conductivity than the film sample, because the PANI-CSA powder has a more ordered structure than PANI-CSA film made using NMP. The PANI-CSA film shows compact coil-like conformation caused by poor interaction between the polymer and NMP. On the other hand, PANI-CSA solution in *m*-cresol revealed higher inherent viscosity than in NMP about 1.5 times and exhibited higher conductivity over five orders of magnitude. Based on these results, NMP was an unfavorable solvent for our blend system, but when we used better solvents, like *m*-cresol, the resulting blends precipitated. Therefore, although NMP was an unfavorable solvent, it was used as a cosolvent of both PANI and PAA in our system. Meanwhile, PANI-CSA film had the highest conductivity when the molar ratio of H<sup>+</sup> to PhN was 0.5.

Interestingly, PANI-CSA/PAA film (50% PANI content) has higher conductivity than the pure PANI-CSA film, and this PANI-CSA/PAA film was converted into PANI-CSA/PI by thermal imidization at curing temperatures of 80, 100, 120, and 150°C, respectively, for 30 min by stage. Conductivity has the tendency to decrease, except



**Figure 3** FTIR spectra: (a) PANI-CSA/PAA, (b) PANI-CSA/PI, (c) polyamic acid, and (d) polyimide.

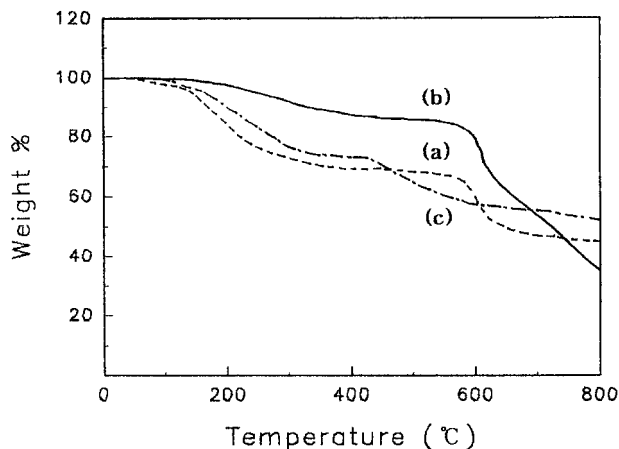
in case of 50% PANI content after imidization, which also showed higher conductivity than pure PANI-CSA film. This decrease of conductivity seems to be attributed to the volatilization of a small quantity of dopant CSA.

The UV-vis spectra of (a) polyaniline emeraldine base (EB), (b) polyaniline emeraldine salt doped with HCl, (c) polyaniline emeraldine salt doped with CSA, and (d) PANI-CSA/PAA blends are shown in Figure 2. Spectra of (a) and (b) are very similar to that previously reported<sup>13</sup> in NMP. In PANI-EB, absorption peaks at 292 and 637 nm, which are attributed to the  $\pi-\pi^*$  transition and the exciton band of the quinoid ring, were shown. When this sample was doped with HCl (b), the absorption peak at 436 nm and polaron bands at 268 and 825 nm appeared. In the case of PANI solution doped with CSA, three distinct peaks at 341, 420, and 863 nm for the solutions in NMP are consistent with a coil-like conformation and localized polaron structure. When a different solvent was used, like *m*-cresol, the PANI-CSA solution showed free carrier tail commencing at 1000 nm. This very intense free carrier tail was consistent with delocalization of electrons in the polaron band promoted by a straightening out of the polymer chain as its coil-like structure becomes more expanded with concomitant reduction in  $\pi$ -defects caused by ring twisting.<sup>14</sup> This may be due to the interaction between the dopant and solvents, but the polymer chain remains coil-like conformation in NMP solution, and the delocalized polaron was not seen. PANI-CSA/PAA shows similar phase as PANI-CSA, so we could conclude that when PANI-CSA and PAA were blended, their blending process did not affect on the doping condition of polyaniline chain.

Figure 3 reveals the FTIR spectra of PAA, PI, PANI-CSA/PAA, and PANI-CSA/PI. IR analysis<sup>15</sup> has been used frequently in studies of kinetics of the thermal imidization of polyamic acids. Despite widespread use of this method, there is no clear consensus as to which of the various imide bands should be followed or what the mea-

**Table II** Degree of Imidization and Conductivity as Imidization Temperature

Temperature (°C)	Degree of Imidization (%)	Conductivity (S/cm)	Conductivity (S/cm)
		PANI-CSA	PANI-CSA/PAA
Room temperature	—	$1.22 \times 10^3$	$2.80 \times 10^3$
80	32.4	$3.43 \times 10^{-4}$	$9.79 \times 10^{-4}$
100	38.3	$8.43 \times 10^{-4}$	$1.21 \times 10^{-3}$
120	51.7	$8.93 \times 10^{-4}$	$2.64 \times 10^{-3}$
150	76.4	$1.59 \times 10^{-3}$	$5.05 \times 10^{-3}$
200	87.7	—	$3.51 \times 10^{-3}$



**Figure 4** TGA thermograms: (a) polyamic acid, (b) polyimide, and (c) PANI-CSA/PI (imidized  $\sim 150^{\circ}\text{C}$ ).

sured changes in each band mean in terms of the changes occurring in the polymers. So we will compare peaks between PAA and PI, and this will indicate the process of conversion of PAA into PI. In this case, polyimide state was obtained by thermal imidization up to  $300^{\circ}\text{C}$ ; but practical imidization temperature was up to  $150^{\circ}\text{C}$  (the degree of imidization was 76.4%; see Table II) because, over this temperature, detrimental reduction of conductivity depends on the volatilization of dopant CSA occurred. In the FTIR spectra for polyamic acid, NMP hindered the characteristic peak; but as the imidization proceeded, characteristic peaks could be seen. First, in case (c) and (d), the peak at  $1776\text{ cm}^{-1}$  is not found in PAA, which is C=O in-phase peak that is usually seen only in the polyimide. We can also see that C=O out-of-phase and C—N stretching depend on  $\text{CN}(\text{OC})_2\text{NC}$  of polyimide, respectively, at  $1725$  and  $1381\text{ cm}^{-1}$ . An aromatic band at  $821.7\text{ cm}^{-1}$  and imide (IV) benzene ring at  $752.1\text{ cm}^{-1}$  are rapidly grown as imidization proceeds. Although a small number of the main vibrational bands of polyimide and polyaniline are absent in the case of (a) and (b), C=O in-phase, C=O out-of-phase, and  $\text{CN}(\text{OC})_2\text{NC}$  peak, respectively, at  $1776.6$ ,  $1728$ , and  $1379.2\text{ cm}^{-1}$  could be seen in (b). With the imidization proceeding, the C=O in-phase peak grows sharply. Also, the  $\text{SO}_3^-$  peak at  $1168.9$ – $1095.6\text{ cm}^{-1}$  in (a) is reduced in (b) due to the volatilization of dopant CSA. C=C stretching in the benzenoid and quinoid rings, which are attributed to polyaniline, are also seen at  $1502.6$  and  $1599.1\text{ cm}^{-1}$ . In these results, we could well understand that as the thermal curing

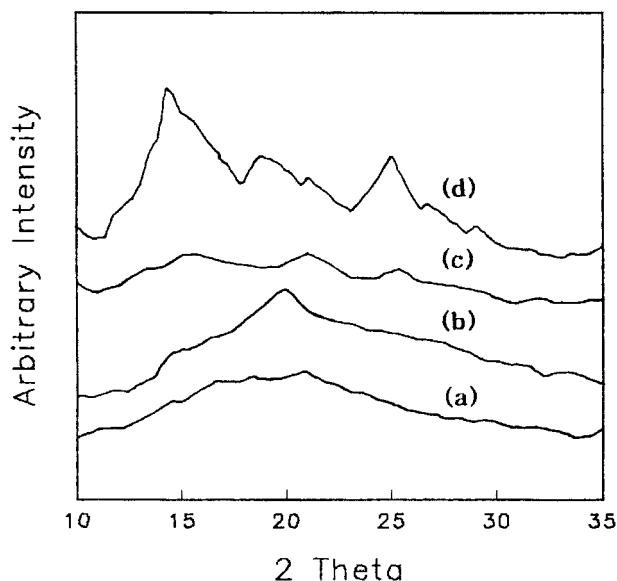
proceeded, both PAA and PANI-CSA/PAA were developed into the PI state.

The degree of imidization as well as the conductivity of PANI-CSA and PANI-CSA/PAA blends as a function of imidization process are shown in Table II. The degree of imidization was calculated from following way with FTIR spectra<sup>15</sup>:

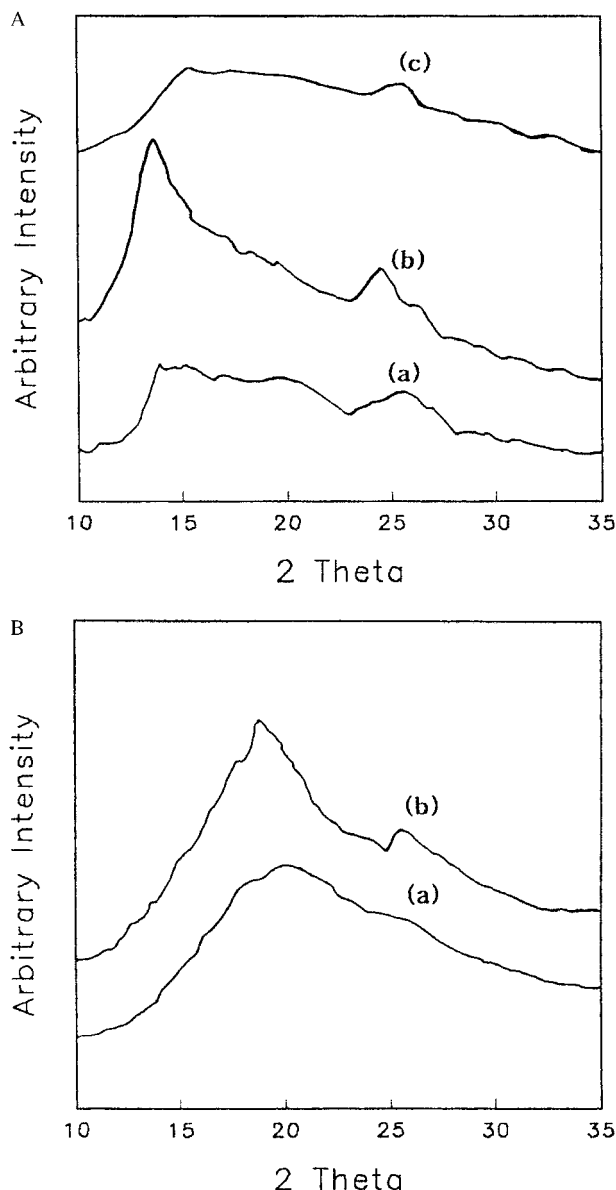
$$\text{Degree of imidization} = (A_{1381}/A_{1500})_{\text{sample}} / (A_{1381}/A_{1500})_{\text{completely imidized sample}} \times 100$$

where  $A$  is the peak area.

A completely imidized sample was obtained by curing PAA up to  $300^{\circ}\text{C}$ . The peak at  $1500\text{ cm}^{-1}$  corresponds to the C—C band of parasubstituted benzene, which is almost unchanged with the degree of imidization; it thus can be a good internal reference for calculating the degree of imidization. The band at  $1381\text{ cm}^{-1}$  is C—N stretching of the imide group. Imidization is proceeded at  $80$ ,  $100$ ,  $120$ ,  $150$ , and  $200^{\circ}\text{C}$  for each 30 min and at  $300^{\circ}\text{C}$  for 1 h. The temperature term in Table II indicates that the imidization process is finished. As the temperature increases, the degree of imidization of PANI-CSA/PAA film also increases. As the imidization proceeded, conductivity also increased up to  $150^{\circ}\text{C}$ , except at  $80^{\circ}\text{C}$ . This was caused by the increase in the crystallinity of the polymer chain. A slight decrease of conductivity



**Figure 5** WAXD spectra: (a) PANI-CSA/PAA (20% PANI content) film, (b) PANI-CSA/PI (20% PANI content) film, (c) PANI-CSA/PAA (50% PANI content) film, and (d) PANI-CSA/PI (50% PANI content) film.



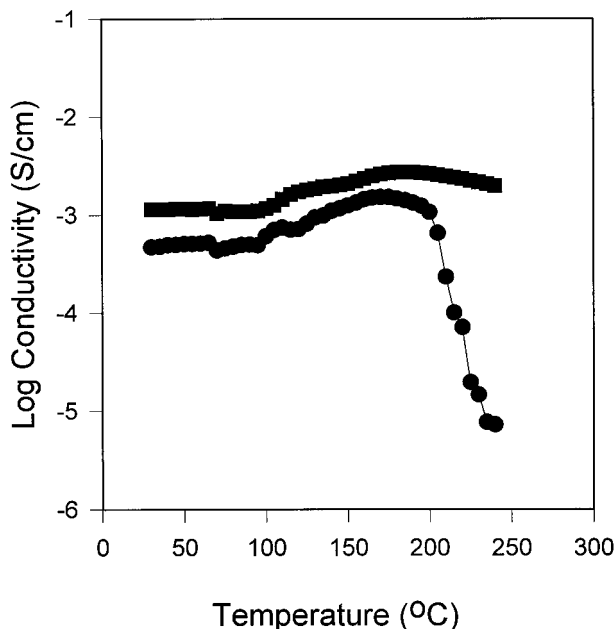
**Figure 6** (A) WAXD spectra: (a) PANI-CSA (room temperature), (b) PANI-CSA (after thermal treatment at 150°C), and (c) PANI-CSA (after thermal treatment at 200°C). (B) WAXD spectra: (a) polyamic acid and (b) polyimide.

of PANI-CSA/PAA at 200°C can be compensated by a high degree of imidization. But the conductivity was abruptly decreased at 200°C, especially the case in PANI-CSA because of the volatilization of dopant, which could not be measured due to extremely low conductivity. This conductivity decrease deviation between PANI-CSA and PANI-CSA/PAA blends can be attributed to their

difference in thermal stability of conductivity, while thermal treatment at higher temperatures (PANI-CSA, over 200°C and PANI-CSA/PAA, over about 230°C) bring about an irreversible conductivity decrease. That is, after thermal treatment, the redoping process in 1M CSA aqueous solution has no effect on conductivity, which can be attributed to thermal crosslinking in PANI chain.

Figure 4 shows TGA curves of PAA, PI, and PANI-CSA/PI (curing up to 150°C) blends under an N<sub>2</sub> atmosphere. Both PAA and PI have good thermal stability; therefore, the apparent weight loss can be observed over 550°C, which is attributed to the polymer chain degradation. In PAA, the decomplexation of NMP, which leads to massive weight loss (about 28%), can be observed from about 170°C. In PANI-CSA/PI, massive weight loss (about 20%) seems to be depend on that of dopant CSA from about 185°C, which looks higher than pure PANI-CSA (about 169°C), and the polymer chain decomposed from about 420°C. This temperature difference of the elimination of dopant from the polymer chain between PANI-CSA and PANI-CSA/PI leads to the difference of conductivity stability (Table II). Because the polyimide, together with polyaniline, have good thermal stability, blend films also show good thermal stability. And nondecomposed material of about 40% remained due to the stiffness and rigidity of molecular chain.

Figure 5 shows WAXD patterns of PANI-CSA/PAA blends (PANI content of 20 and 50%) and their imidization state by curing (up to 150°C). When in the PAA condition [(a) and (c)], all patterns show low intensity; but after the imidization process by curing, the growth of the crystalline peak can be seen. There are no pronounced diffraction peaks in the patterns of low PANI content blend. No major change of crystal phase was detected except the increase in intensity in the case of low PANI content blend films like (a) and (b), in which a broad amorphous reflection at  $2\theta = 19.5^\circ$  was only seen; however, in high PANI content blends [(c) and (d)], the crystalline peaks exist. Another remarkable result is that (b) is like WAXD patterns of nonoriented PI and (d) is like those of PANI-CSA film (Fig. 7). In (b), only (002) reflection of polyimide is shown at  $2\theta = 19.5^\circ$ ,<sup>16</sup> but there exists (010) reflection at  $2\theta = 14.4^\circ$ , (110) reflection at  $2\theta = 25^\circ$ , and (100) reflection at  $2\theta = 21^\circ$ <sup>17</sup> in (d), which are the characteristic peaks of PANI-CSA film produced from good solvents like *m*-cresol. So it can be concluded,



**Figure 7** The temperature dependence of conductivity at elevated temperature: PANI-CSA (●) and PANI-CSA/PAA (■).

as follows, that the PANI content is one of the major factor of chain structure changes of blend film, such as crystallinity, size of crystalline domain, degree of chain orientation, interchain order, etc. The reason for the increase in conductivity of 50% PANI content PANI-CSA/PI is explained well by this phenomena. It seems that when the blend system has a large content of PANI, the imidization process plays a key role of annealing effect of PANI, hence leading to an increase in crystallinity and conductivity.

The fact that after thermal treatment the chain structure of blends can be converted to be more ordered structure like PANI-CSA film produced from *m*-cresol is important. MacDiarmid and Epstein<sup>18</sup> reported that the polymer chains of PANI-CSA in different solvents may have different conformations and therefore different conjugation lengths. For example, fully doped PANI-CSA has a more expanded coil-like conformation and hence a longer conjugation length in solvents such as *m*-cresol, *p*-cresol, and 2-fluorophenol than in solvents such as chloroform, NMP, DMF, and benzylalcohol. That is, better polymer-solvent interactions results in conformational change from a compact-coil to expanded-coil structure. In our system, chain structure conversion from coil-like to more ordered structure seems to occur due to thermal treatment. Kobayashi and coworkers<sup>19</sup>

also showed that exposure to elevated temperatures could have an annealing effect on chemically prepared polyaniline. This result suggests that the blend film can be changed to be a more ordered structure mainly due to PANI-CSA during the imidization process. So we can conclude that the concomitant increase in crystallinity due to the annealing effect is expected to contribute to increase in bulk conductivity in the intermolecular component of the blend film (PANI content 50%) in spite of the volatilization of small quantity of dopant. This annealing effect will be considered in detail in Figure 6.

Figure 6 presents the WAXD spectra of PANI-CSA and PAA, respectively, as the thermal treatment. In regard to PANI-CSA film, after thermal treatment up to 150°C, two peaks ( $2\theta = 14.4$  and 25) grow more sharply. So, thermal treatment during the imidization process played an important part in annealing effect and then caused to be converted to more ordered structure. In case it was of PAA, the structure was changed from amorphous to a little crystalline. As the temperature increases more, the X-ray diffraction pattern is unchanged from Figure 6(B), peak (b), except a slight increase in intensity. But, over 150°C, the crystalline structure of PANI [Fig. 6(A)] is destroyed. The peaks at  $2\theta = 14.4$  and 25 grew sharper as the thermal treatment temperature increased until 150°C; but above this temperature, rapid chain destruction happened that seemed to result from volatilization of dopant or/and some irreversible structural changes of polymer chain, i.e., crosslinking between polymer chain.

Variations of conductivity of the PANI-CSA and PANI-CSA/PAA film as the temperature changes are shown in Figure 7. The temperature was raised 3°C/min. The conductivity of PANI-CSA increases slowly with temperature below 185°C but decreases rapidly above this temperature. However, in the case of PANI-CSA/PAA, the conductivity increases slowly until 195°C, and after this temperature, it decreases mildly as the temperature increases. The increase of conductivity until the critical point is attributed to polymer chain mobility and activation of dopant CSA and the decrease of conductivity can be attributed to the volatilization of dopant CSA, followed by structural change.<sup>20</sup> This shows that PANI-CSA/PAA have higher conductivity stability than PANI-CSA and the conductivity decline of PANI-CSA in Table II is in strong accordance with this.

## CONCLUSIONS

Polyaniline/polyamic acid blend films could be prepared with CSA-doped polyaniline solution and polyamic acid solution by solvent casting using NMP as a cosolvent. The PANI-CSA/PAA blend solution showed a coil-like conformation and a localized polaron structure (shorter conjugation length). The conductivity of films increased as the polyaniline content increased and had higher conductivity than pure CSA-doped polyaniline film when the polyaniline content reaches 50 wt %. After thermal treatment, PANI-CSA/PAA films converted to PANI-CSA/PI films, which had slightly higher conductivity than PANI-CSA/PAA. This increase of conductivity was assumed to be caused by an increase in conductivity in the intermolecular component of the blend because of the annealing effect originated from thermal treatment. PANI-CSA/PAA film had a good thermal stability of conductivity at relatively high temperature and showed higher conductivity than room temperature.

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