

# Effect of Composition on the Conductivity and Morphology of Ligno Sulfonic Acid Sodium Salt Doped Polyaniline

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**ABSTRACT:** A systematic approach is developed to study the ligno sulfonic acid sodium salt (LSA) protonation or doping process with polyaniline emeraldine base (Pani-EB) in organic solvents like dimethyl sulfoxide, and the influence of LSA-doping on the properties of polyaniline was investigated in detail. The composition of Pani-EB and LSA was varied in the weight ratio of 1:1 to 1:50 to investigate the effect of the dopant concentration on the conductivity and morphology. The doping process was confirmed by UV-vis and FTIR spectroscopies. The composition analysis indicates that only 50% of the LSA is used for the doping process irrespective of the weight ratio of LSA/Pani-EB in the feed. The four probe conductivity measurement suggests that the

conductivity of the doped samples are increasing with the increase in the ratio of Pani-EB/dopant composition, and the high conductivity of the doped material was obtained in the range of  $1.0 \times 10^{-2}$  S/cm. Scanning electron microscopy reveals that LSA induces a selective aggregation in the polyaniline chains to produce needlelike or rod-shape morphology of sizes having  $\sim 0.2 \mu\text{m}$  diameter and  $1 \mu\text{m}$  length. At very higher amount of LSA, the microrods are completely collapsed and form uniform continuous morphology. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 101: 2650–2655, 2006

**Key words:** polyaniline; ligno sulfonic acid; conducting polymers; morphology

## INTRODUCTION

Polyaniline (PANI) is unique among all conducting polymers in that its electrical properties can be reversibly controlled by simple protonation and deprotonation by reacting with acids and bases, respectively.<sup>1–9</sup> The availability of wide range of dopants, such as mineral, carboxylic, and sulfonic acids, made it easy to obtain electrically conducting polyaniline emeraldine salts<sup>10</sup> for various application in many high performance devices, such as rechargeable batteries,<sup>11</sup> chemical sensors,<sup>12</sup> electromagnetic shielding,<sup>13</sup> electrochemical and corrosion devices.<sup>14–16</sup> The interest in PANI-based devices arises from the relatively economical polymer synthesis and processing techniques combined with wide range of electronic, optical, chemical, and mechanical features.<sup>17</sup> Recently, we reported a systematic approach to produce ordered

(crystalline), submicron size and uniform morphology PANI-doped materials, using structurally different dopants in various polymerization methodologies, such as interfacial and emulsion.<sup>18</sup> Conducting polymers based on renewable resource materials are very attractive because of their wide availability and lower cost compared to petroleum-based products. The dopant materials used for the PANI are so far mostly based on petroleum-based dopants, and very few reports are known for renewable resource-based dopants. From our laboratory, Raji and Pillai have published a substantial amount of work on sulfonic acid dopants based on 3-pendadecyl phenol, a derivative of cardanol, which is the main component of cashew nut shell liquid.<sup>19–22</sup> Cellulose, starch, and lignin are natural available polymeric materials and they have been blended with PANI to improve the processability and thermal stability.<sup>23,24</sup> Cellulose derivatives were also used as stabilizers for dispersion polymerization to control the size and morphology of PANI particles.<sup>25</sup> Ligno sulfonic acid (LSA) is an attractive renewable resource-based dopant for PANI. Its solubility in water and its polymeric backbone makes it a very attractive dopant for PANI. Viswanathan and Berry have reported the preparation of ferromagnetic conducting PANI nanocomposites using LSA as dopant and ferromagnetic iron oxides.<sup>26</sup> The same research group has also reported the catalytic effect of selected

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transition metal ions in the synthesis of doped PANI.<sup>27</sup> Paterno et al.<sup>28,29</sup> and Nikolaidis et al.<sup>30,31</sup> reported separately the use of LSA for the preparation of self-assembled films of poly (*o*-ethoxyaniline) and exploration of PANI for biomedical application, respectively. Though the importance of PANI-LSA is recently very well documented, so far no effort has been taken to understand the doping process, the effect of composition of dopant/PANI on the morphology, conductivity, and etc., which is very crucial for the future development of molecular-electronics based on LSA-PANI.

The present work emphasized on a systematic approach, for the first time, to produce LSA sodium salt doped PANI in solution. The weight ratio of the polyaniline emeraldine base (Pani-EB) and dopant was varied from 1:1 to 1:50 to investigate the effect of the dopant concentration on the structure-property of doped materials. The doping processes were confirmed by FTIR spectroscopy, UV-vis spectroscopy, and conductivity measurements. The solid state ordering (crystalline) and morphology were analyzed by wide-angle X-ray diffraction (WXR) and scanning electron microscopy (SEM). The thermal stability of the doped materials is analyzed by thermogravimetric analysis.

## EXPERIMENTAL

### Materials

LSA sodium salt was purchased from Aldrich and used without further purification. Aniline, ammonium persulfate (APS), concentrated sulfuric and hydrochloric acids, aqueous ammonia, and solvents were purchased locally and purified.

### Measurements

For conductivity measurements, the polymer samples were pressed into a 10-mm diameter disc using an IR pelletizer and analyzed using a four probe conductivity instruments by applying a constant current. The resistivity of the samples was measured at five positions, and at least two pellets were measured for each sample: the average of 10 readings was used for conductivity calculations. Infrared spectra of the polymers were recorded using an Impact 400 D Nicolet FTIR spectrophotometer in the range of 4000–400  $\text{cm}^{-1}$ . For SEM measurements, polymer samples used for the conductivity measurements were further subjected for thin gold coating using JOEL JFC-1200 fine coater. The probing side was inserted into JEOL JSM-5600 LV scanning electron microscope for taking photographs. Wide angle X-ray diffractions of the finely powdered polymer samples were recorded by Philips Analytical diffractometer using  $\text{CuK}\alpha$  emission. The

spectra were recorded in the range of  $2\theta = 0\text{--}50$  and analyzed using X' Pert software. UV-vis spectra of the drop cast PANI samples on glass plate are recorded using PerkinElmer Lambda 35 Spectrophotometer. The thermal stability of the polymers was determined using TGA-50 Shimadzu thermogravimetric analyzer at a heating rate of  $10^\circ\text{C}/\text{min}$  in nitrogen.

### Synthesis of polyaniline

Aniline (10 mL, 0.11 mol) was polymerized in the acidic medium using ammonium persulfate (31.95 g, 0.14 mol) as oxidant. The resultant polyaniline emeraldine salt was filtered, washed with water, and was converted into its corresponding emeraldine base by treating with aqueous  $\text{NH}_3$  (450 mL, 15%  $\text{NH}_3$  in water) by following the reported procedure.<sup>32</sup> The dried polymer weighed 8.1 g (yield = 89%). FTIR (KBr,  $\text{cm}^{-1}$ ): 1589 (C=C, quinoid), 1508 (C=C, benzoid), 1315 (C—N), 1163, 831 (C—H).

### Doping of PANI-EB with sodium salt of lignosulfonic acid

Polyaniline emeraldine base (Pani-EB) (1 g, mol) was taken in 75 mL dry dimethylsulfoxide (DMSO) and stirred at  $90^\circ\text{C}$  for 1 h. It was cooled to  $25^\circ\text{C}$ , and 10 g (for P-4) of sodium salt of LSA was added and heated to  $90^\circ\text{C}$  and stirred for additional 2 h. The green colored slurry was cooled and poured into excess of water (or chloroform for higher composition) and filtered. The residue was successively washed with methanol and acetone and dried in a vacuum oven at  $80^\circ\text{C}$  for 24 h (0.1 mmHg). Yield = 5.6 g (51%). FTIR (KBr): 3278, 2941, 1597 (C=C, quinoid) 1508 (C=C, benzoid), 1460, 1423, 1350, 1211 (S=O), 1039 ( $\text{NH}^+\text{SO}_3^-$ ), 952, 815 (C—H), 704 (C—S), 650 (S—O), and 530  $\text{cm}^{-1}$ .

Adopting the same procedure, the doping of Pani-EB was done by varying the compositions of Pani-EB/LSA sodium salt as 1:1, 1:1.5, 1:7.5, 1:10, 1:15, 1:20, 1:30, 1:40, and 1:50 for producing doped polymers P-1 to P-8.

## RESULTS AND DISCUSSIONS

Pani-EB was synthesized from aniline and ammonium persulfate by following the reported procedure.<sup>32</sup> The Pani-EB was doped by heating with the powdered sample of LSA sodium salt in organic solvents. Various solvents like *N*-methylpyrrolidinone, tetrahydrofuran, toluene, *N,N*-dimethylformamide, *N,N*-dimethylacetamide, *N,N*-dimethyl sulfoxide (DMSO), and water were investigated for the doping process. Among all, DMSO was found to be a very good solvent for producing green color emeraldine salt. The following procedure is typically used for the doping

TABLE I  
Composition, Yield, and Color of LSA Doped Samples

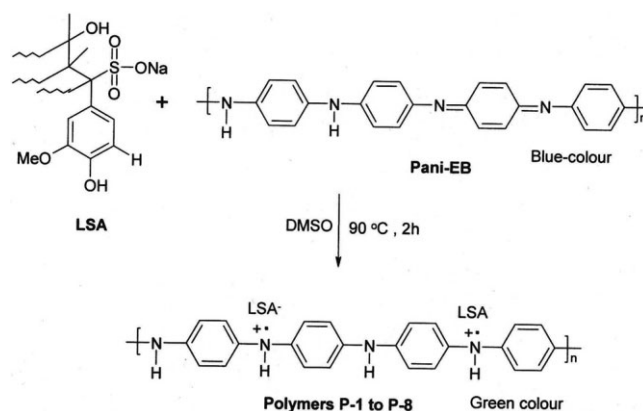
Sample	Pani-EB/LSA in feed (g)	Yield (g)	Pani-EB/LSA in doped (g)	Yield (%)	Conductivity (S/cm)	Color
P-1	1:1	1.2	0	52	$1 \times 10^{-8}$	Blue
P-2	1:1.5	1.3	0	32	$1 \times 10^{-8}$	Blue
P-3	1:7.5	2.2	0	26	$3 \times 10^{-7}$	Bluish green
P-4	1:10	5.6	1:4.6	51	$3.9 \times 10^{-6}$	Green
P-5	1:20	13.5	1:12.5	66	$4 \times 10^{-3}$	Green
P-6	1:30	20.8	1:19.8	67	$4.64 \times 10^{-3}$	Green
P-7	1:40	21.3	1:20.3	52	$4.6 \times 10^{-3}$	Green
P-8	1:50	26.4	1:25.4	52	$1 \times 10^{-2}$	Green

process. Finely powdered Pani-EB was taken in DMSO and stirred at 90°C using magnetic stirrer under nitrogen atmosphere. After cooling to room temperature, required amount of sodium salt of LSA was added to the blue colored Pani-EB solution in DMSO and heated for 2 h at 90°C. The resultant viscous liquid was cooled and poured into excess of water, filtered, and washed with large amount of water, methanol, acetone, and chloroform. The doped samples were dried in vacuum oven at 90°C for 24–48 h (0.1 mmHg). The compositions of Pani-EB/LSA was varied in the following weight ratio of 1:1, 1:1.5, 1:7.5, 1:10, 1:15, 1:20, 1:30, 1:40, and 1:50 to produce doped PANI **P-1** to **P-8**. It was found that for lower compositions the DMSO solution as well as the purified doped materials is in blue color, which indicates the insufficient doping. In the preparation of **P-3**, though the DMSO solution appeared to be blue in color, the purified doped materials was obtained as bluish green solid. For all other higher compositions (**P-4** to **P-8**), green colored polyaniline emeraldine salt was produced. The composition, yield, and color of the doped materials are summarized in Table I. The LSA was also prepared by treating its sodium salt with 10M HCl. The sulfonic acid was highly hygroscopic and its doped PANI materials were found to be similar in conductivity values compared to that of LSA sodium salt. Therefore, for the present investigation, the LSA was employed as dopant, and DMSO was used as a doping solvent. The structures of the LSA and its doped materials are shown in Scheme 1.

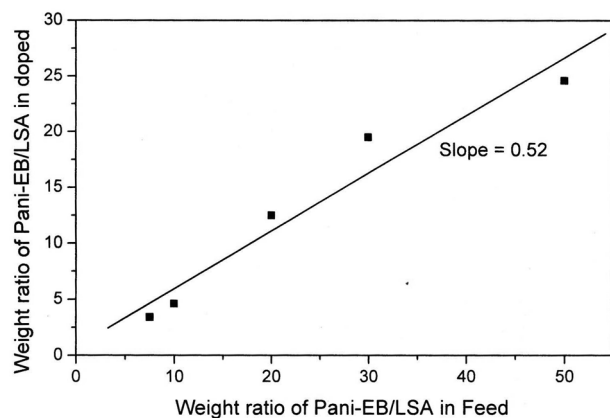
The amount of the LSA incorporated in the doped material is calculated using the following empirical relation. Pani-EB (1 g) is reacted with LSA ( $y$ -gram) to produce the resultant Pani-EB-LSA complex ( $1 + y$  g). Since Pani-EB is not soluble in the solvents like water, methanol, and chloroform, the reduction in the isolated yield is mainly due to the loss of unreacted LSA (LSA is soluble) and not due to Pani-EB. Under these assumption, the actual amount of LSA reacted during the doping process can be directly calculated by subtracting the yield by the amount of Pani-EB (i.e., 1 g in the present case). The incorporation of LSA in the

doping process for **P-4** to **P-8** is calculated by the above method and reported in the Table I. For lower compositions (**P-1** to **P-3**), the empirical relationship is not valid because of the insufficient doping. There are many factors such as unavailability of sulfonic functionalities buried in the LSA polymer chains, electrostatic interactions and steric hindrance, etc., may influence on the poor protonation ability of LSA at lower compositions (**P-1** to **P-3**). In the present investigation, it is very difficult to find which factor is more predominant, and therefore, more detail analysis are further required to understand the doping process in the lower composition range. The amount of LSA in the feed is plotted against the actual incorporation for **P-4** to **P-8** and shown in Figure 1. A straight line is fitted using  $y = mx + C$  and the slope of the resultant plot is found to 0.52 ( $\sim 0.5$ ). The slope value of 0.5 indicates that only 50% of the LSA is used in the current doping process irrespective of the weight ratio of Pani-EB/LSA in the feed. The increasing trend indicates that by applying more amount of LSA one can produce higher amount of doped materials.

The photophysical optical properties of the doped materials were studied by UV-vis spectroscopy to understand the doping process (Fig. 2). The UV-vis spectrum of PANI-EB was recorded for film caste from



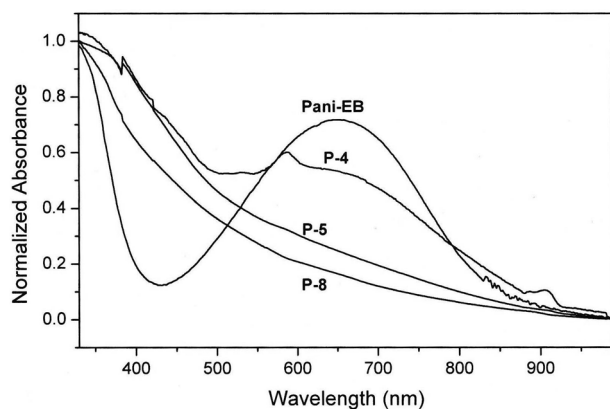
Scheme 1 Synthesis of LSA-doped polyaniline.



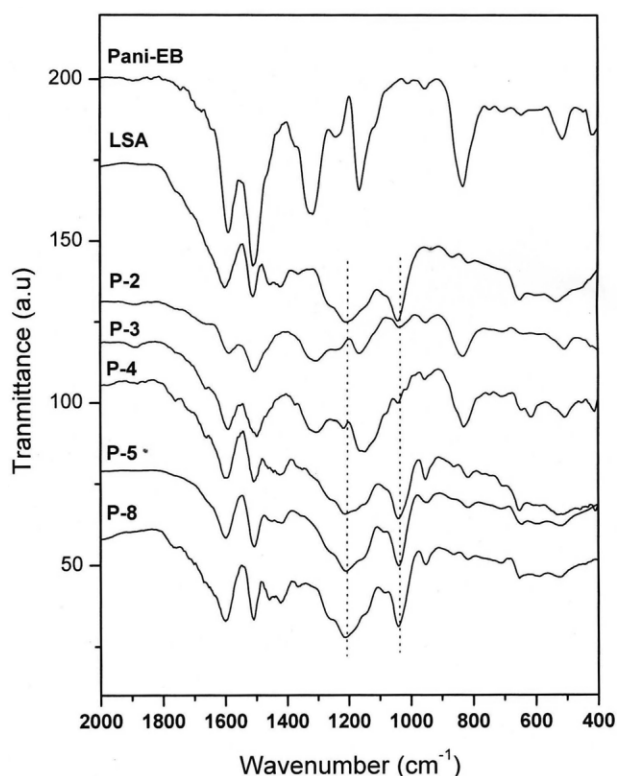
**Figure 1** Plot of weight ratio of Pani-EB/LSA in feed versus in doped materials.

NMP and it has two distinguish absorbance peaks at peak maximum at 326 and 650 nm, which are assigned to  $\pi$ - $\pi^*$  transitions corresponding to the benzenoid and quinoid units.<sup>33</sup> To study the doping efficiency of LSA, solid state UV-vis spectra of the doped samples are recorded for drop caste film from NMP. The UV-vis spectra of **P-4**, **P-5**, and **P-8** are shown along with **PANI-EB** in Figure 2. It is very clear from the spectra that upon doing with LSA the peak at 650 nm is getting vanished, which confirm that LSA is very effective dopant for PANI like other sulfonic acids.<sup>18</sup> Interestingly, in **P-4** and **P-5** have still relatively small absorbance at 650 nm, which suggests that the insufficient for doping. However, at higher LSA amount (for **P-8**), the peak at 650 nm is completely vanished indicating the higher degree of doping.

FTIR spectroscopy is a powerful tool to analyze the structures of doped PANI samples. FTIR spectra of **PANI-EB**, **LSA** and **LSA** doped samples are shown in Figure 3. The five peaks in **PANI-EB** at 1587, 1485, 1317, 1155, and 831  $\text{cm}^{-1}$  are in accordance with earlier reports.<sup>34</sup> The two peaks at 1587 and 1485 are corresponding to the quinoid and benzenoid ring



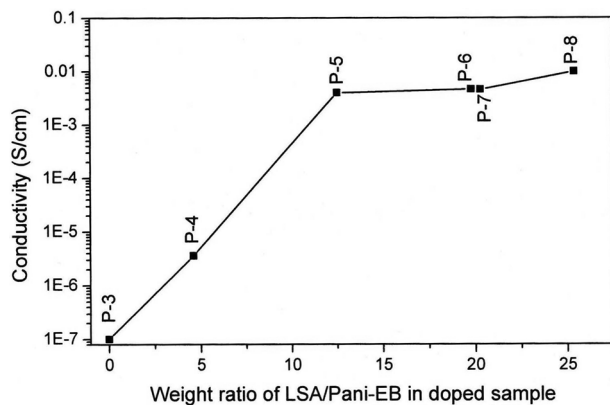
**Figure 2** UV-vis spectra of Pani-EB and doped materials.



**Figure 3** FTIR spectra of Pani-EB and doped samples.

$\text{C}=\text{C}$  stretching ring deformations, respectively. The peak at 1317 and 830 are corresponding to  $\text{C}-\text{N}$  stretching and  $\text{C}-\text{H}$  out-of-plane vibrations of 1, 4-disubstituted benzene ring, respectively. The appearance of new peaks in the LSA doped samples at 1211 and 670  $\text{cm}^{-1}$  are attributed to symmetric and unsymmetric stretching vibrations of  $\text{O}=\text{S}=\text{O}$  and  $\text{S}-\text{O}$  groups.<sup>18</sup> The peak at 1039  $\text{cm}^{-1}$  is corresponding to  $\text{NH}^+ \dots \text{SO}_3^-$  interactions between the polymers chain and the LSA. The comparison of two peaks at 1211 and 1039  $\text{cm}^{-1}$  in **P-3** and **P-4** to **P-8** suggest that the intensity are more stronger for the latter case with respect to high degree of doping by LSA. This observation is very well in accordance with the UV-vis spectra in Figure 1.

The conductivity of the Pani-EB-LSA doped samples was determined by four probe conductivity measurement unit by applying a constant current. The samples were compressed to 10 mm diameter and 0.5–0.8 mm thickness pellets for the measurements. The conductivity values for the doped samples are summarized in Table I, and the conductivity values are plotted against the actual LSA incorporation and shown in Figure 4. It is very clear from the plot that the conductivity of the Pani-LSA complex increases with the increase in the amount of LSA used for the doping. The high conductivity is attained a plateau-region at **P-5**, and the values are almost invariant with further increase in the LSA amount (in **P-6** to **P-8**). It



**Figure 4** Plot of conductivity versus weight ratio of Pani-EB/LSA.

suggests that LSA is very good dopant for PANI, and more than 10 weight equivalents are needed in the doped material to attain a conductivity in the range of 0.01 S/cm. The FTIR and UV-visible studies are also confirmed similar trend for variation with the Pani-EB/dopant composition.

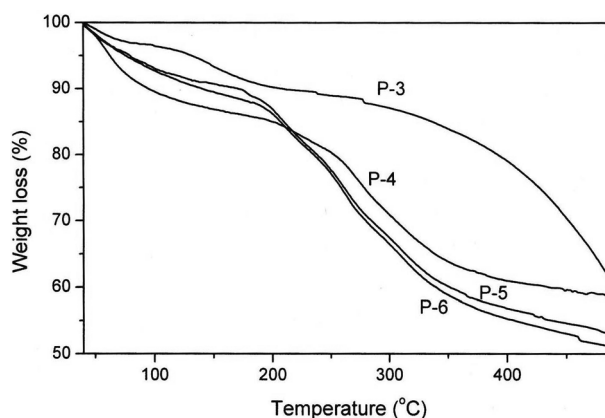
The thermal stability of the LSA-doped samples were determined by thermogravimetric analysis (TGA), and the representative TGA plots are shown in Figure 5. It is very clear from the plots that the samples are only stable up to 225°C. The low temperature stability of the doped materials arise from the poor thermal stability of the LSA. It suggests that the LSA-doped PANI materials are limited to low temperature applications below 200°C. All the polymer samples were subjected to wide angle X-ray diffraction using Cu- $\alpha$  radiation source and scanned from  $2\theta$  values 0–50. A broad diffraction pattern was obtained for PANI-EB due its typical amorphous nature. The WXR patterns (not shown) of LSA-doped samples are similar to that of pure LSA, and no peaks for highly ordered PANI chains are observed. It may be due to that in the doped samples (P-4 to P-8) the amount of LSA is very large compared with that of Pani-EB, which may lead to highly amorphous nature for the entire material.

The morphology of the LSA-doped materials was analyzed by SEM and their photographs are shown in Figure 6. The sample preparation was done by compressing the polymer powders into 1-mm-thick pellets, followed by depositing gold on the probing side by electroplating technique. It is very clearly observable from the photographs that there is a significant difference in the morphology of the materials. Pani-EB exhibits [Fig. 6(a)] a disordered porous morphology with larger particle size distribution in the range of 0.5  $\mu\text{m}$ .<sup>18</sup> A significant improvement in the porosity of the morphology is noticed for LSA-doped samples [Figs. 6(b)–6(d)]. Interestingly, in P-3, the PANI chains are undergoing selective types of aggregation process to

produce needle morphology with  $\sim 0.2 \mu\text{m}$  diameter and 1  $\mu\text{m}$  length. The microrods or needle formations are in higher length and well organized in P-6 compared to P-3. It suggests that with increasing amount of LSA in doped samples, the microrod formation is also significantly increasing. At very higher amount of LSA [for P-7, Fig. 6(d)], the microrods are completely collapsed and we get a very uniform morphology for the doped samples. It indicates that the microstructures formation in LSA-doped PANI is very critical to the amount of LSA sample present in the doped matrix. The present investigation reveals that the effect of composition of Pani-EB/LSA weight ratio on the properties of PANI, such as conductivity and morphology, is very significant. The combination of UV-vis, FTIR, conductivity, and SEM analysis indicate that, with the increase in the weight ratio of Pani-EB/dopant composition, the doped materials are undergoing a selective aggregation process to produce higher electrically conducting materials. It can be very clearly understood by comparing the conductivities of P-3 and P-6 that latter has better morphological features, which contributes to the higher conductivity of almost  $10^3$  times, compared to P-3.

## CONCLUSIONS

We have investigated the effect of LSA doping on the conductivity and morphology of PANI in detail. The present investigation clearly demonstrate that the success of producing highly conducting PANI based on renewable resource based dopants like LSA is highly depended on composition of Pani-EB/dopant. Dimethyl sulfoxide is found to be the best solvent suited for the LSA doping with PANI, and the minimum 20 times more weight of LSA is required in the feed compared to PANI to produce conductivity in the range of 0.01 S/cm. The LSA induce a selective aggregation process in the PANI to produce microrod structures, which is highly sensitive to the amount of LSA



**Figure 5** TGA plots of doped samples.

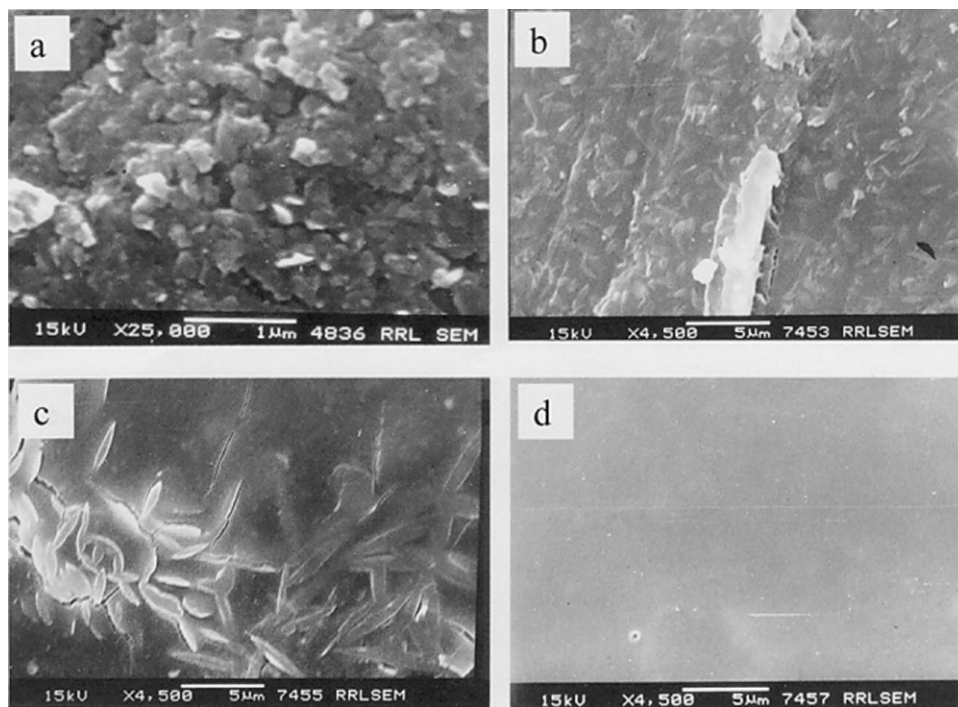


Figure 6 SEM photographs of Pani-EB (a), P-3 (b), P-6 (c), and P-8 (d)

in the doped materials. Since P-3 to P-6 has microrod structures with conductivity in the range of 0.01 S/cm, they will be attractive for many applications in optoelectronic and biomedical applications.

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