

# Synthesis of Dendritic Polyaniline Nanofibers by Using Soft Template of Sodium Alginate

Biplab Bhowmick,<sup>1</sup> Mrinal Kanti Bain,<sup>1</sup> Dipanwita Maity,<sup>1</sup> Nirmal Kumar Bera,<sup>1</sup> Dibyendu Mondal,<sup>1</sup> Md. Masud Rahaman Mollick,<sup>1</sup> Prabir Kumar Maiti,<sup>2</sup> Dipankar Chattopadhyay<sup>1</sup>

<sup>1</sup>Department of Polymer Science & Technology, University of Calcutta, Kolkata-700 009, India

<sup>2</sup>Department of Chemical Technology (Ceramic Engineering), University of Calcutta, Kolkata-700 009, West Bengal, India

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**ABSTRACT:** Highly crystallized polyaniline (PANI) nanofibers were synthesized by oxidative polymerization of aniline in the presence of sodium alginate as a soft template in HCl and ammonium peroxydisulfate (APS) acting as an oxidizing agent. Sodium alginate, in presence of a protonic acid like HCl, formed hydrogen bonds with anilinium ions or oligomers. The formed hydrogen bonds provide the driving force to form PANI nanofibers. The nanofibers were separated from the alginate gel by degelling with ammonium hydroxide and during degelling emeraldine salt was converted into emeraldine base form. The polymerized PANI was characterized using ultraviolet (UV)–visible spectroscopy, Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and scanning

electron microscopy (SEM). UV and FTIR spectra showed that the presence of sodium alginate had no effect on the electronic state and backbone structures of the resulting PANI products. It was evident from the XRD analysis that the obtained PANI nanofibers exhibit higher crystalline order. SEM micrographs showed that PANI nanofibers were just like a mat of interwoven twisted nanofibers. After magnification of the SEM image, it was found that most of the nanofibers were interconnected to form ramose structures rather than isolated nanofibers. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 123: 1630–1635, 2012

**Key words:** soft template; alginate gel; PANI nanofibers; ramose structures

## INTRODUCTION

Nowadays, various nanophase materials like nanospheres,<sup>1</sup> nanowires,<sup>2,3</sup> nanoribbons,<sup>3,4</sup> helical structures,<sup>5,6</sup> nanotubes,<sup>7–10</sup> nanofibers<sup>11</sup> etc., with regular and specified morphology have attracted considerable attention owing to their different potential applications ranging from molecular electronics,<sup>12</sup> biomedical sciences,<sup>13–15</sup> optoelectronics, and memory devices.<sup>16,17</sup> In the field of intrinsically conductive polymers, for instance, polypyrrole, polythiophene, polyaniline (PANI) etc., the PANI is unique because of its relatively facile processability, excellent environmental stability, tunable conductivity switching between insulating and semiconducting materials, easy acid/base doping/dedoping behavior, reversible redox property,<sup>18</sup> and low cost. Fast transfer of electrons in nanoscaled PANI,<sup>19–25</sup> having fascinating morphologies and architectures offers the possibility of improved performance.

In the recent years, several techniques for synthesizing PANI nanostructures have been developed including template synthesis,<sup>26</sup> template-free synthesis,<sup>27</sup> seeding polymerization,<sup>28,29</sup> interfacial polymerization,<sup>30–32</sup> rapid mixing polymerization,<sup>33</sup> electrospinning,<sup>34</sup> and electrochemical polymerization.<sup>35,36</sup> Template method introduces “structural directors” in the polymerization bath that dictates the PANI nanostructures morphology. These structural directors include “soft templates” such as surfactants,<sup>37–42</sup> inorganic acid,<sup>43</sup> organic acids,<sup>44,45</sup> or polyelectrolytes that assists in the self-assembly process, or “hard templates” such as zeolites,<sup>46</sup> anodic aluminum oxide,<sup>9</sup> and porous alumina membranes<sup>23</sup> where the 1D nanochannels promote the growth of nanotubes or nanowires.

Among the PANI nanomaterials such as nanoparticles,<sup>47–54</sup> nanorods,<sup>55</sup> nanospheres,<sup>55</sup> nanowhiskers,<sup>56</sup> PANI nanofibers have attracted a great attention because of its large surface area. PANI nanofibers have several novel behaviors like enhanced sensitivity for gas sensors as compared with conventional PANI films<sup>11</sup> and the irradiation of randomly deposited PANI nanofibers with camera flash can be transformed into a smooth shiny film, exhibiting a unique technology in processing polymers into useful forms.<sup>57</sup>

Recently, polyaniline nanofibers have been synthesized using interfacial polymerization in an aqueous/

Correspondence to: D. Chattopadhyay (dipankar.chattopadhyay@gmail.com).

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organic biphasic system without templates or functional dopants.<sup>2,58</sup> Huang and Kaner<sup>2</sup> fabricated the PANI nanofibers using interfacial polymerization and they showed that the diameter of nanofibers alters with changing dopants from HCl to perchloric acid. The average diameter of nanofibers is 30 nm, 50 nm, and 120 nm in the case of HCl, camphorsulfonic acid, and perchloric acid, respectively. PANI nanofibers have been synthesized in a large scale by interfacial polymerization. They proposed that the interfacial polymerization is insensitive to the polymer concentration as well as polymerization temperature but the uniformity of the PANI nanofibers depends on the acid concentration. Epstein and co-worker<sup>59</sup> also reported the preparation of PANI nanofibers by dilute polymerization. A nanofiber seeding method has been developed to synthesize PANI nanofibers without organic dopants, surfactants, and insoluble templates.<sup>60</sup> Another piece of research has been done through chemical oxidation polymerization of aniline in a surfactant gel, which is formed by a mixture of hexadecyltrimethylammonium chloride, acetic acid, aniline, and water at  $-7^{\circ}\text{C}$ .<sup>61</sup> The synthesized dendritic PANI nanofibers have the diameter of 60–90 nm and the length of 1–2  $\mu\text{m}$ . Wang and coworker<sup>62</sup> reported the oligomer-assisted path to fabricate the polyaniline nanofibers and the resultant PANI nanofibers are in the diameter range of 20–40 nm with several micrometer length. Conducting  $\text{HClO}_4$ -doped polyaniline nanofibers are prepared on a large scale through a modified spraying technique and these nanofibers are promising in the application of lithium/polymer rechargeable batteries.<sup>63</sup> Qiang et al.<sup>64</sup> chemically synthesized PANI nanofibers by rapid mixing polymerization with very low aniline concentration in the presence of ammonium peroxydisulfate (APS) and they showed that the yield of PANI nanofibers are much higher than the yield in the case of interfacial polymerization. Mandal and coworkers<sup>65</sup> demonstrated a novel route to synthesize fine and uniform polyaniline nanofibers at room temperature by reverse microemulsion and they showed nanofibers are highly conducting and thermally more stable. In addition, one-phase surfactant-assisted chemical method has been utilized to synthesize PANI nanofibers, which is doped with CSA and 2-acrylamido-2-methyl-1-propanesulfonic acid, in large quantities.<sup>66</sup> Xue and coworkers<sup>67</sup> prepared PANI nanofibers using aniline-SA complex as a template. They polymerized aniline in the presence of aqueous solution of APS and produced uniform nanofibers of average diameter of 40–100 nm. Qiu et al.<sup>68</sup> successfully synthesized highly crystallized PANI nanofibers in the presence of sucrose octaacetate and they proposed that this sucrose octaacetate acted not only as *in situ* seed but also as “soft template.” Hydrogen

bonds formed between sucrose octaacetate and anilinium ions or oligomer molecules supply a driving force to form PANI nanofibers.

In this research, we endeavor a new approach to synthesize PANI nanofibers in the presence of sodium alginate, acting as a soft template. Sodium alginate, in the presence of a protonic acid like HCl, transforms into gels. The gel interstices provide the driving force to form PANI nanofibers. The characterization of synthesized PANI nanofibers using UV-Visible spectroscopy, Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and scanning electron microscopy (SEM) are also reported in this article.

## EXPERIMENTAL

### Materials

All materials were used as received. Aniline and ammonium peroxydisulfate extra pure were purchased from MERCK, was stored in a sealed container in a refrigerator. Sodium alginate purchased from Central Drug House (P) has been used for template synthesis.

### Synthesis of polyaniline

Aniline was doubly distilled under vacuum to eliminate the impurities. Two percent of alginate solution was prepared with normal double distilled water. Aniline (0.25 mL) was mixed with alginate solution by vigorous mixing and in the presence of 1M HCl the whole solution transformed into gel. To synthesize the PANI nanofibers, finally APS (0.6252 g) was added to the gel and the polymerization was started instantly. The oxidation of aniline was indicated by the formation of blue pernigraniline at the first stage but slowly this blue color disappeared and converted to a green protonated emeraldine form at the end of the polymerization. To complete the polymerization, the whole system was kept at  $4^{\circ}\text{C}$  for 24 h. The synthesized PANI nanofibers were easily separated from the template and above all a mass production of PANI nanofibers by this process within a short time is possible.

To characterize the PANI nanofibers, first of all the synthesized PANI nanofibers had been separated from alginate gel. Acid medium was responsible for the gelation of sodium alginate and *vice versa*. The nanofibers were separated from the alginate gel by degelling with ammonium hydroxide and during degelling emeraldine salt was converted into emeraldine base form. The alginate was washed out with double distilled water and the nanofibers were centrifuged. The emeraldine base form of PANI nanofibers were converted to salt form for characterization.

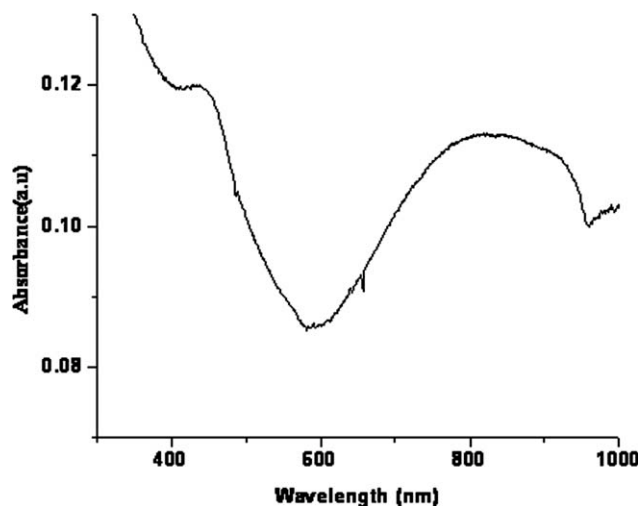


Figure 1 UV-vis spectra of the PANI nanofibers.

### Characterization

#### Ultraviolet-visible spectroscopy

Absorbance spectra from 200 to 1000 nm were obtained on a UV-Vis spectroscopy system (Agilent 8453 Spectrophotometer) using quartz cuvettes (1 cm path length). A few drops of the separated emeraldine base form of nanofibers were added to 1M HCl to produce a pale green dispersion of emeraldine salt form of nanofibers. Then UV spectra of the nanofibers were taken.

#### Fourier transform infrared spectroscopy

The FTIR spectra of salt form of the synthesized PANI nanofibers were obtained using Shimadzu FTIR-8400S spectrometer in the range of 400–4000  $\text{cm}^{-1}$ .

#### X-ray diffraction spectroscopy

The XRD of PANI nanofibers were performed with a Scifert XRD 3000P diffractometer. Powder sample was taken for the structural characterization. Microscopic glass slides were cut into segments of approximately 2 cm  $\times$  1.6 cm, rinsed with methanol and then dried in air. The emeraldine salt form of polyaniline was added on the substrate to cover the entire surface. Then the sample was dried in air at room temperature. This process was repeated several times as needed to increase the thickness of the sample and then the XRD analysis was carried out.

#### Scanning electron microscopy

HITACHI- SC400N SEM was used at an accelerating voltage of 15 kV. One drop of the dispersion of emeraldine salt form of PANI nanofibers was deposited on small segment of microscopic glass slide. The sol-

vent was evaporated by air and kept in vacuum desiccator for 1.5 h; then the sample was attached to aluminum sample stubs using double-sided carbon tape for SEM examination. Average nanofiber diameters were measured on at least 25 randomly chosen fibers at several regions of the sample.

## RESULTS AND DISCUSSION

The UV-vis spectrum of the PANI nanofibers is shown in Figure 1. The PANI nanofibers exhibit two characteristics peaks at 410 nm and 810 nm along with a broad absorption tail. The absorption peak at 410 and 810 nm are due to the  $\Lambda$ - $\Lambda^*$ (benzenoid) and  $n$ - $n^*$ (quinoid) transitions, respectively. The broad band at 810 nm with a long tail corresponds to the polaron transitions.<sup>67</sup> A free carrier tail from 810 nm onwards to the IR region is characteristics of metallic conductive materials.<sup>69</sup> Thus, it can be concluded that the presence of sodium alginate does not alter the electronic state of the resulting PANI products.

The FTIR spectra of synthesized PANI nanofibers after removal of sodium alginate are shown in Figure 2. The IR spectrum of PANI nanofibers shows main peaks at 1591, 1504, 1315, 1161, and 831  $\text{cm}^{-1}$ . The peaks at 1591 and 1504  $\text{cm}^{-1}$  indicate the presence of quinonoid and benzenoid moiety of PANI, respectively.<sup>70-72</sup> The two peaks at 1315 and 1161  $\text{cm}^{-1}$  are assigned to C-N and C=N stretching vibrations.<sup>67</sup> The peaks at 1130 and 831  $\text{cm}^{-1}$  can be attributed to the in-plane and out-of-plane C-H bending modes, respectively.<sup>69</sup> 3427  $\text{cm}^{-1}$  is ascribed to the stretching of N-H bond and the peak at 1635  $\text{cm}^{-1}$  assigning to the C=O group of quinone obtained by the oxidation of quinonoid form.<sup>73</sup>

As there is no difference in the FTIR spectra of the synthesized PANI nanofibers with the reported

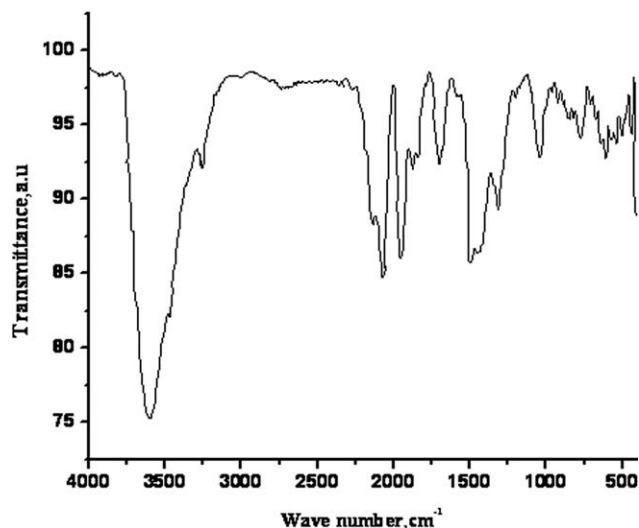
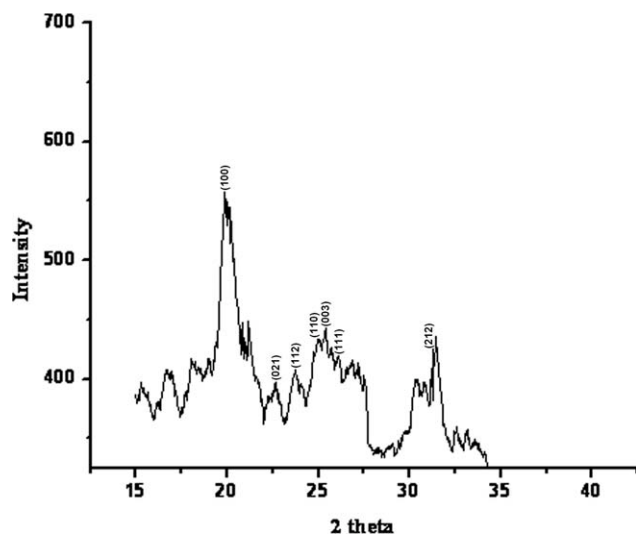


Figure 2 FTIR spectra of the PANI nanofibers.



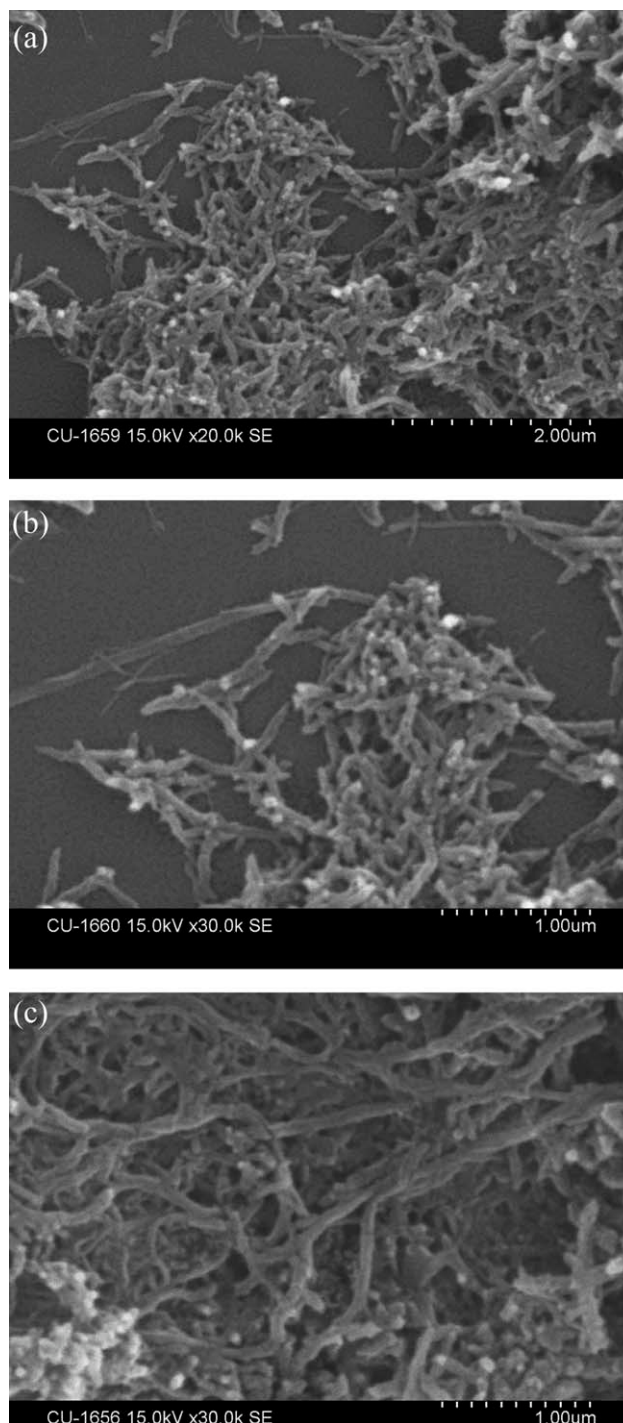
**Figure 3** X-ray diffraction patterns of polyaniline nanofibers.

literature, so it can be concluded that the use of sodium alginate has no effect on the backbone structures of PANI.

Figure 3 shows the XRD patterns of the resultant polyaniline nanostructures in its doped form. It was reported that emeraldine salt was partly crystalline whereas emeraldine base was essentially an amorphous polymer.<sup>74</sup> The diffraction pattern shows broad diffraction peaks centered at  $2\theta = 20.3^\circ$ ,  $25.3^\circ$ , and  $31.4^\circ$ . The first two peaks, showing that the resulting doped PANI was partly amorphous and partly crystalline, are ascribed to the periodicity parallel and perpendicular to the PANI polymer chain.<sup>65</sup> This indicates that the resulting polymer is in the form of highly doped emeraldine salt and has good crystallinity. The diffraction peak at  $2\theta = 25.3^\circ$  implies that the densely packed phenyl rings gives rise to a planar conformation and, thus, to an extensive interchain  $\pi \rightarrow \pi^*$  orbitals overlap. The peaks at  $2\theta = 20.3^\circ$ ,  $22.7^\circ$ ,  $23.6^\circ$ ,  $25.3^\circ$ ,  $25.6^\circ$ ,  $26.5^\circ$ ,  $31.4^\circ$  belong to the crystal plane (100),<sup>75</sup> (021),<sup>76</sup> (112),<sup>76</sup> (110),<sup>75</sup> (003),<sup>76</sup> (111),<sup>75</sup> and (212)<sup>76</sup> respectively. So, it is evident from the XRD analysis that the obtained PANI nanofibers exhibit higher crystalline order.

SEM micrographs are shown in the Figure 4(A–C). It is clear from SEM pictures that emeraldine salts nanofibers are just like a mat of interwoven twisted nanofibers. However, the intention of this experiment is to synthesize PANI nanofibers, but some short length fibers and small particles are present on the surface of nanofibers. SEM image with higher magnification shows that [Fig.4(A)] most of the nanofibers are interconnected to form ramose structures rather than isolated nanofibers [Fig. 4(B)]. Fiber dimensions are measured in the regions where they are less in numbers preferably near the edge of the

mats of nanofibers. The average diameter of the doped polyaniline nanofibers is about 30 nm with a length of several microns. According to Manohar and coworker<sup>66</sup> the synthesized polyaniline nanofibers using surfactants or soft templates are chemically



**Figure 4** A: Scanning electron microscopy images of polyaniline nanofibers synthesized with sodium alginate soft template. B: Magnified SEM image of polyaniline nanofibers synthesized with sodium alginate soft template. C: Scanning electron microscopy images of polyaniline nanofibers after repetitive doping and dedoping.

robust and retain their fibrillar morphology even after repeated doping and dedoping using aqueous acids and bases. Figure 4(C) gives the image of densely packed polyaniline nanofibers after repetitive doping and dedoping.

## CONCLUSIONS

PANI nanofibers were synthesized by oxidative polymerization of aniline in the presence of sodium alginate as a soft template in HCl and APS acting as an oxidizing agent. UV and FTIR spectra showed that the presence of sodium alginate had no effect on the electronic state and backbone structures of the resulting PANI products. It was evident from the XRD analysis that the obtained PANI nanofibers exhibit higher crystalline order. SEM micrographs showed that PANI nanofibers were just like a mat of interwoven twisted nanofibers. After magnification of the SEM image, it is found that most of the nanofibers are interconnected to form ramose structures rather than isolated nanofibers.

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