

Optical and electrical properties of conducting polyaniline nanofibers synthesized by interfacial and rapid mixing polymerization

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ABSTRACT

One dimensional conducting polymer nanostructures have been the focus of quite extensive studies worldwide due to their high aspect ratio, high porosity apart from high surface area to volume ratio. Conducting polyaniline nanofibers can be synthesized by various methods. In this paper, we report the preparation of polyaniline nanofibers with an average diameter of 40–70 nm by two different simple approach rapid mixing and interfacial polymerization. The key to producing polyaniline nanofibers is to suppress secondary growth. Based on this, interfacial polymerization and rapidly mixed reactions have been developed that can readily produce nanofibers by slightly modifying the conventional chemical synthesis of polyaniline without the need for any template or structural directing agent. Synthesized polyaniline (PANI) nanofibers were characterized by FTIR spectroscopy, X-ray diffraction, transmission electron microscopy for their structural and UV-Vis absorption spectroscopy for optical properties. Direct and indirect transition energy gaps were determined from their Tauc plots. The absorption spectra show a linear fit for the transition. Electrical properties of the synthesized polyaniline nanofibers have been studied and the Arrhenius plots of electrical conductivity for the samples synthesized by rapid mixing and interfacial polymerization method show an approximate equal in their activation energy. The results obtained from optical and electrical properties are well compared, correlated and explained with respect to interfacial and rapid mixing polymerization techniques. Copyright © 2014 VBRI press.

Keywords: Conducting polymer; polyaniline; rapid mixing polymerization; interfacial polymerization; nanofibers.



by various techniques

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Introduction

Intrinsically conducting polymers (ICPs) have emerged very important class of materials worldwide because of their distinctive electrical, chemical and optical properties [1-3]. One can have remarkable scope for tuning of their electrical conductivity between semiconductor and metallic regime, when they are appropriate doped [4]. The unique properties of conducting polymers are the great scope of developing the new models to explain their observed properties, particularly various charge transport mechanism [5]. Among the various conducting polymers synthesized, polyaniline (PANI) occupies a prime position, because of its unique characteristics like inexpensiveness of the monomer, ease of processing and excellent stability. PANI in its pure and doped forms find extensive applications in making devices like polymer light emitting diodes, photovoltaics, sensors, batteries and super capacitors [6, 7]. Nanostructure conducting polymer polyaniline (PANI), due

to its high surface area, controllable conductivity, color responses with acid/base (doped/undoped) conditions, good environmental stability, and ease of preparation, has been widely studied for potential applications in the electric, optical, biologic, and sensor fields. In the past few years, several novel methodologies have been developed for the preparation of nanostructure PANI in the form of nanowires, nanofibers, and nanotubules [8, 9]. Among different dimensional nanostructure, one dimensional nanostructure PANI have been the focus of quite extensive studies worldwide due to their high aspect ratio, high porosity apart from high surface area to volume ratio. [10]. Properties of PANI Nanofibers have attracted even more interest due to which that make them possibly suitable for applications as sensors [11], batteries [12], molecular electronic devices [13] and separation membranes. There are Different methods used to fabricate polyaniline nanofibers include hard templates, soft templates [14], seeding, interfacial polymerization, rapidly mixing [15], and electrospinning [16]. Epstein et al found dilute polymerization of aniline is favorable for production of nanofibrous structures [17]. The preparation of PANI nanofibers received great development owing to the excellent work by Huang and Kaner et al [18-21]. They found that PANI naturally forms nanofibrillar morphology. The key to forming nanofibers is to suppress secondary growth that results in the agglomerated particles found in conventional PANI synthesis. In order to obtain pure PANI nanofibers, the secondary growth of the initially formed nanofibers must be suppressed and this can be achieved by two effective methods to suppress the secondary growth of the nanofibers, i.e., interfacial polymerization and rapidly mixing reaction.

Therefore, we have used these methods to prepare PANI nanofibers. In this paper, we report PANI nanofibers prepared by interfacial polymerization and rapidly mixing reaction and mainly focused on the systematic optical and thermal band gap studies along with the comparative study of structural properties of PANI nanofibers by FTIR spectroscopy, X-ray diffraction and transmission electron microscopy.

Experimental

Materials

All the chemicals including aniline (99.5%), hydrochloric acid (99.5%) and ammonium persulfate (APS) (99.5%) were purchased from Sigma-Aldrich and used as received except aniline which was purified through distillation under reduced pressure and stored refrigerated before use. In the study of interfacial polymerization, the reaction was placed in a heterogeneous biphasic system in which, toluene ($C_6H_5CH_3$) (99.5%) was employed as one of the immiscible phases. De-ionized water was used for the preparation and dilution of solutions.

Methods

X-ray diffraction (XRD) was carried on Philips PW1710 automatic X-ray diffractometer with Cu-K α radiation ($\lambda=1.5404\text{\AA}$), with a scanning speed of $10^\circ \text{ min}^{-1}$. FTIR spectra were performed on Shimadzu FTIR-8101A

Spectrophotometer in the wavelength range of 400–4000 cm^{-1} . Transmission electron microscope images were obtained using PHILIPS-CM200 TEM with resolution 2.4 \AA . The optical absorption spectra were carried out using UV-1800 double beam UV-VIS spectrophotometer. The electrical conductivity of the compressed pellets of PANI nanofibers was determined by four probe resistivity technique. The pellets were prepared with the help of hydraulic press (Kimaya Engineers, India) by applying a pressure of 5000 kg/cm^2 .

Synthesis of PANI nanofibers by interfacial polymerization

The reaction was placed in a heterogeneous biphasic system, where the polymerization occurs primarily at the interface. Since the as-made PANI product was synthesized in its hydrophilic emeraldine salt form, it diffuses away from the reactive interface into the water layer. This made more reaction sites available at the interface and avoids further overgrowth. In a typical process, 1.86 g (3.2 mmol) of aniline was dissolved in 100 ml of organic solvent as toluene in a beaker so as to form organic phase and 4.56 g (0.8 mmol) of Ammonium per sulfate (APS) in 100 ml of hydrochloric acid (1.0 M) in another beaker so as to form aqueous phase. Then the organic phase was carefully added along the side of beaker to the aqueous phase to produce a clear interface of aqueous–organic solutions. As the reaction proceeds, the color of the organic phase becomes darker and finally stops changing, indicating reaction completion. An overnight reaction time is generally sufficient. Upon completion we had vials with toluene organic solvent of polyaniline nanofibers. The synthesis of polyaniline at low temperature (avoid 0°C) favors the formation of longer chains length (and hence, high molecular weight), under this conditions a less amount of structural defect in the polymer can be expected. The dark green precipitate resulting from the polymerization reaction had washed repeatedly with dilute HCl solution and de-ionized water until the filtrate became transparent. This precipitate was then dried in vacuum oven at 60°C for 24 hours. Solid sample was powdered in a mortar and finally compressed to form pellets.

Synthesis of PANI nanofibers by rapid mixing polymerization

The nanofibers form of polyaniline was obtained by the chemical oxidative polymerization process in a rapidly mixed reaction. In this method when the reaction was started, the initiator molecules were consumed rapidly during polymerization and depleted after nanofibers formation. Therefore, the overgrowth of polyaniline was suppressed due to lack of initiator molecules. In a typical process, aniline (0.5M) was dissolved in 100 ml of 1M HCl to form aniline solution and then APS (0.5M) was dissolved in 100 ml of 1M HCl to form oxidant solution. The oxidant solution was then quickly poured into the aniline solution at room temperature followed by immediately magnetic stirring (stirring speed: 500 rpm) for 2 hour. Polymerization can be observed when green color of PANI emeraldine salt became visible. The stirring was stopped after 2 hour and left undisturbed to react overnight.

After completion of the reaction the polymer was separated and washed with distilled water and hydrochloric acid several times until the filtrate was colorless. This precipitate was then dried in vacuum oven at 60° C for 24 hours. Solid sample was powdered in a mortar and finally compressed to form pellets.

Results and discussion

Fig. 1 shows the FTIR spectra of polyaniline nanofibers synthesized by interfacial polymerization (I-PANI) and rapid polymerization (R-PANI). The vibration band around 3300 cm^{-1} is attributed to the N-H stretching vibrations. The band at 1610 cm^{-1} is a signature of the N-H bending vibration of PANI present in both the samples. The strong band observed at 1140 cm^{-1} and the band at 1200 cm^{-1} are due to the C-C stretching and C-C twisting of the alkyl chain. The C-Cl stretching vibration observed around 600 cm^{-1} confirms the fact that the PANI nanofibers are in doped states. The peaks at 1400 and 1460 cm^{-1} are assigned to the C=C stretching vibrations of the benzenoid and quinoid units of PANI, respectively [22, 23]. All the above observed characteristics bands confirmed the formation of PANI.

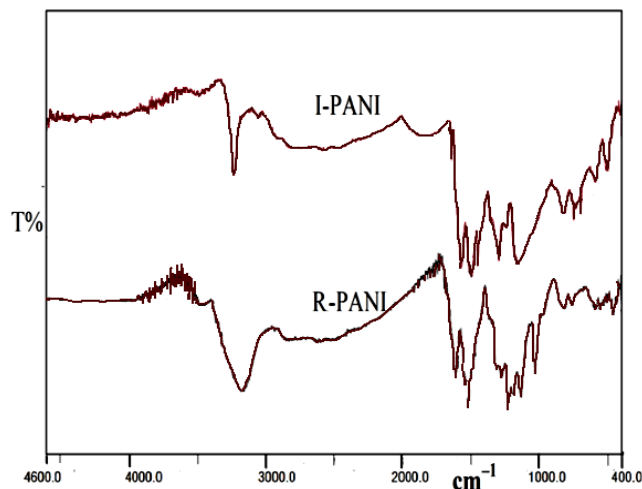


Fig. 1. FTIR spectra of I-PANI and R-PANI nanofibres.

Fig. 2 (a) and **(b)** show the TEM images of polyaniline nanofibers synthesized by interfacial polymerization (I-PANI) and rapid polymerization (R-PANI) respectively. The uniform nanofibers morphology can be observed with the length in microns and diameter in the range of 40-70 nm. The average diameter of I- PANI and R-PANI nanofibers was found to be 70 nm and 40 nm respectively.

The XRD pattern of the polymer generally differs because of uncertainty in the polymeric chains. It depends on the synthetic routes, solvent and ionized state [24]. **Fig. 3** shows the XRD pattern of polyaniline nanofibers synthesized by interfacial polymerization (I-PANI) and rapid polymerization (R-PANI). The pattern shows sharp and well defined peaks, which indicate semi-crystalline nature of both the PANI nanofibers. The diffraction pattern for I-PANI nanofibers shows the peaks at $2\theta = 19.05^\circ$, 17.86° , 32.26° and 25.75° indicating more crystalline due to size effect as compared to that of R-PANI for which the peaks observed at $2\theta = 20.24^\circ$ and 22.53° supports the

results of TEM images. The plains of benzenoid and quinoid rings of PANI chain are responsible for crystalline structure [21]. The average diameter of the synthesized PANI nanofibers was found to be 60 nm and 30 nm for I-PANI and R-PANI respectively determined by using Scherer formula [23], quite consistent with TEM results of I-PANI and R-PANI.

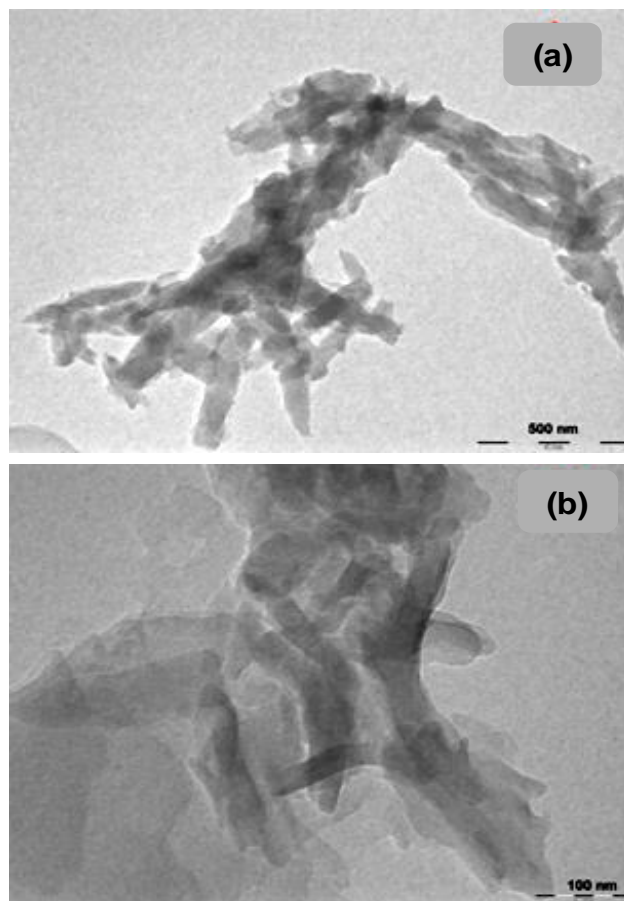


Fig. 2. TEM images of (a) I-PANI and (b) R-PANI nanofibers.

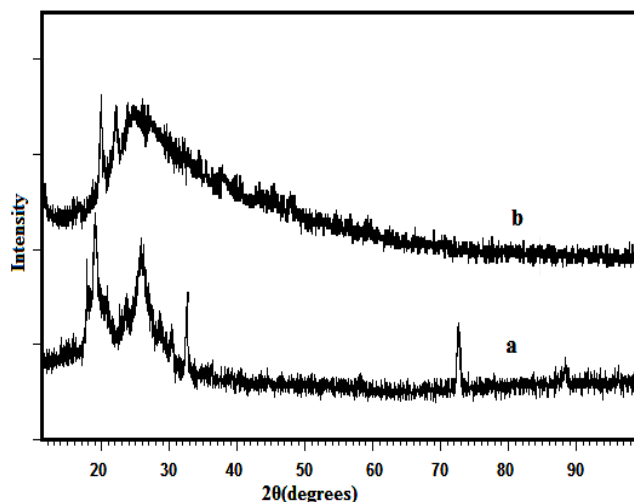


Fig. 3. XRD pattern of (a) I-PANI (b) R-PANI nanofibers.

Fig. 4 demonstrates UV – visible absorption spectra of I-PANI and R-PANI nanofibers. The peak in wavelength

range 306-324 nm is due to the π - π^* transition of benzenoid ring of PANI [25]. The peak at 402-420 nm is attributed to the protonation of PANI (polaron - π^*), indicative of the conducting state [13] and the peak in wavelength range 828-835 nm is due to π - polaron transition. The peak at 320 nm is due to the π - π^* transition which is related to the extent of conjugation between the adjacent phenylene rings in the polymeric chain. These peaks have been assigned to transition from valance band to conduction band at 320 nm and charge transfer between benzenoid and quinonoid rings due to free nonbonding electrons that can absorb relatively low energy radiation and these are well agreed with the results of Yang et al [25] and Jing et al [26]. The valance band or highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) bands are separated by an energy gap is called band gap which is of fundamental important, because the energy gap determines the electrical conductivity and optical absorption character of the polyaniline.

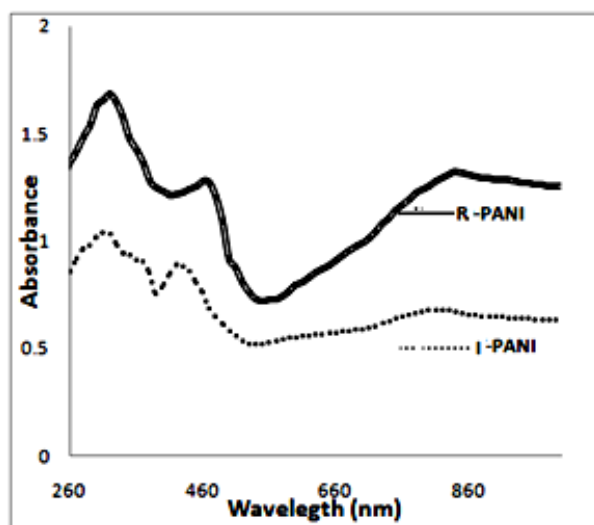


Fig. 4. UV-Vis spectra of I-PANI and R-PANI nanofibers.

The photon absorption in many amorphous materials is found to obey the Tauc relation [25], which is of the form

$$\alpha h\nu = B(h\nu - E_g)^n$$

Where, α is the absorption coefficient, $h\nu$ is the photon energy, B is the band gap tailing parameter, E_g is a characteristic energy which is termed as optical band gap and n is the transition probability index, which has discrete values $n = 1/2, 3/2, 2$ and 3 for direct allowed, direct forbidden, indirect allowed and indirect forbidden electronic transitions, respectively [13]. The absorption coefficient (α), at the corresponding wavelengths is calculated using the Beer-lambert's relation.

$$\alpha = \frac{2.303A}{l}$$

Where, l is the path length and A is the absorbance [27].

The index $n = 1/2$ represents the direct allowed transition energy gap. To determine the direct transition

energy gaps, plot of $(\alpha h\nu)^2$ versus $h\nu$ is linear function existence of direct allowed band transition in PANI samples. Extrapolating of linear dependence of the relation to abscissa yields the corresponding direct allowed band gap. The value of the optical direct transition energies are 2.54 eV and 2.85 eV for I-PANI and R-PANI respectively obtained from Fig. 5(a, b) and this is due to π - π^* transition from valance band to conduction band nearly at 330 nm and the formation of polaron approximately at 830 nm. In order to determine the indirect transition energy gap, plots of $(\alpha h\nu)^{1/2}$ as a function of photon energy versus $h\nu$ was plotted and shown in Fig. 5 (c, d). The linear portion of the plots at the absorption edge confirms that the material has an indirect optical band gap [28]. The intercept of these curves on the photon energy axis gives the indirect transition energy gap. The value of the optical indirect transition energies was found to be 1.25 eV and 1.69 eV for I-PANI and R-PANI respectively.

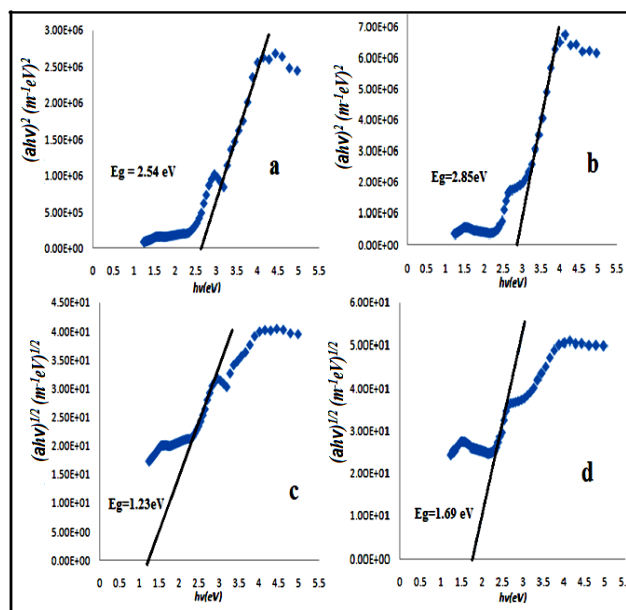


Fig. 5. Direct allowed transition energy of (a) I-PANI and (b) R-PANI. Indirect allowed transition energies of (c) I-PANI and (d) R-PANI.

The measurement of dc conductivity was accomplished through use of the four probe technique. I-V characteristics of doped PANI were recorded at room temperature and are found to be linear; the values of electrical conductivity at room temperature of doped I-PANI and R-PANI nanofibers were found to be 0.993 and 2.25 (S/cm) respectively. Fig. 6 shows thermal dependence dc electrical conductivity (σ) of I-PANI and R-PANI nanofiber respectively. We observed that the temperature dependence for each polymer behaves in the usual activated manner indicating semiconducting property. The increase in conductivity with increase in temperature is the characteristic of "thermal activated behavior" [29]. The increase in conductivity could be due to increase of efficiency of charge transfer between the polymer chains and the dopant with increase in temperature [30]. It is also possible that the thermal curing affects the chain alignment of the polymer, which leads to the increase of conjugation length and brings about the increase of conductivity. There also had to be molecular rearrangement on heating, which made the molecular conformation

favorable for electron delocalization [31]. The value of electrical conductivity of the synthesized I-PANI is slightly decreased than that of R-PANI nanofiber. This difference in the values of conductivity is attributed to the effect of toluene on treatment in interfacial polymerization, which shows that organic solvent as toluene in polymerization of aniline using interfacial method is immiscible with water may disturb the polymerization when reaction time is prolonged, which result decrease in conductivity [32].

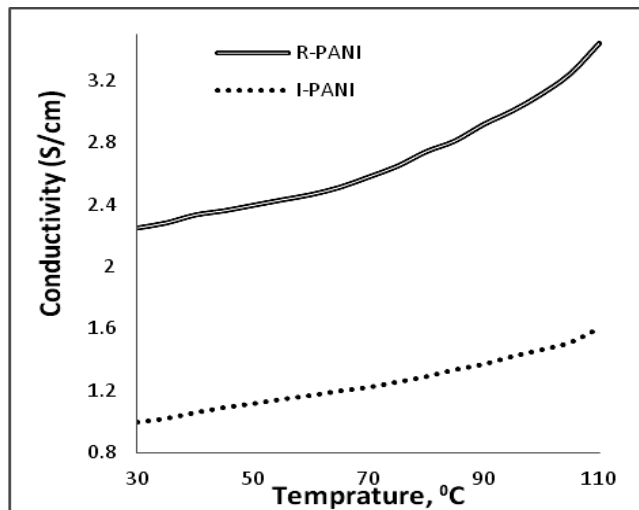


Fig. 6. Conductivity of I-PANI and R-PANI nanofibers.

The electrical conductivity in temperature range 300 to 400 K was found to follow the Arrhenius model [33]. Plots of $\log(\sigma)$ vs $1/T$, gives activation energy for R-PANI and I-PANI, which was found to be 0.099 eV and 0.113 eV respectively. The values did not differ significantly from the activation energy of PANI reported [34-36].

Conclusion

PANI nanofibers have been successfully synthesized by chemical oxidation of aniline using interfacial and rapid methods. The XRD study reveals semi-crystalline nature and TEM results the formation of PANI nanofiber of diameter between 40 – 70 nm. Tauc plots used to find direct and indirect transition energies of PANI nanofibers. Arrhenius plot provided activation energy in the range 0.099 – 0.113 eV from their thermally activated behavior. While comparing crystallinity and transition energies of PANI nanofibers, interfacial polymerization technique was found to be good as compared to rapid mixing technique.

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