

Structural and Optical Study of Chemically Synthesized Polyaniline

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ABSTRACT

Polyaniline (PANI) is synthesized by chemical oxidative polymerization method. The, characterization were made using XRD (X-ray diffraction), FT-IR (Fourier transform spectroscopy), UV -vis (ultra-violet visible spectrophotometer) technique which confirms the synthesis of the Polyaniline. The surface morphology of Polyaniline was studied with scanning electron microscope (SEM).

Keywords: Polyaniline, XRD, SEM, FTIR

1. INTRODUCTION

Polymers are generally insulators and to exhibit electrical conductivity they must possess, ordered conjugation with extended (π) electrons and large carrier concentrations. Conjugated polymers are the organic compounds that have an extended (π) orbital system and conjugated carbon system [1-3]. It is possible to reduce the structural disorder in doped conducting polymers by choosing optimum parameters during synthesis[2-5]. It is worthwhile to mention that the nanostructured intrinsically conducting polymers (NSICP) offer reduced structural disorder which consequently helps in increasing the electronic conductivity of the polymers (Bianchi et al. 1999 [6-11]).

In this study, PANI was prepared by chemical oxidation polymerization method. The diameter of the pellet was 13 mm. The structural characterization is made by using X-ray diffraction and optical characterizations are made by using Fourier transform spectroscopy, Ultraviolet-visible spectrophotometer techniques. The surface morphology of these nanocomposites is studied with scanning electron micrograph.

2. EXPERIMENTAL

2.1. Sample Preparation

Aniline hydrochloride (2.59 g) was dissolved in distilled water in a volumetric flask to make 50 mL solution. Ammonium peroxydisulfate (5.71 g) was dissolved in water also to make 50 mL of solution. Both the solutions were kept for 1 hour at room temperature. They were then mixed with a brief stirring and left at rest to polymerize. The solution turned to dark green within few minutes. Next day PANI Precipitate was collected on a filter paper, washed three times with 100 mL portions of 0.2M HCl to remove the un-reacted aniline and its oligomers from the precipitate. After this process, precipitate was washed three times with 100 mL portions of acetone to absorb the water molecules. The precipitate was firstly dried in air for 30 min and then in oven for 3 hours at 60 °C.

2.2. Measurements

The XRD spectrum of the sample is recorded by PANalytical, X'pert PRO diffractometer using CuK_α radiation ($\lambda=1.54056 \text{ \AA}$) for structural analysis of the sample. The SEM image of the sample is taken by scanning electron microscope (Model-430, LEO

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Cambridge, England). FTIR spectrum of the sample is recorded on the Bruker Alpha spectrometer to determine the formation of Polyaniline. To record absorbance spectrum, 0.02 g of sample is dissolved in 5 mL of m-cresol. The absorption spectrum of the solution is recorded with UV-vis spectrophotometer (Model No. V-670 JASCO).

3. RESULT AND DISCUSSION

3.1. X-ray diffraction

XRD spectra show weak crystalline quality because there is no sharp peak in the spectra besides only two small peaks at 12.98° and 15.53° as shown in figure 1 (a).

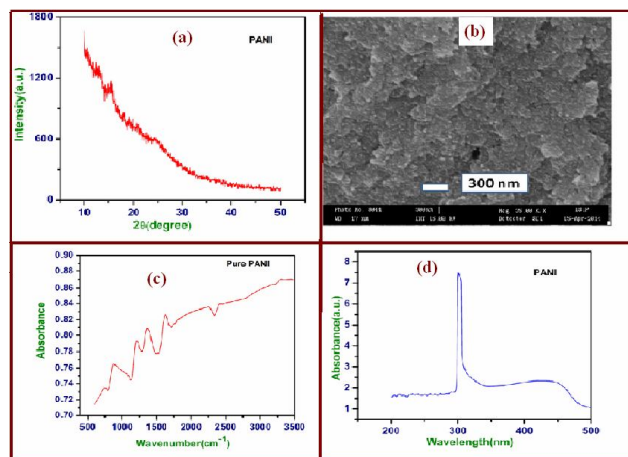


Fig. 1: Images of Polyaniline (a) X-ray diffraction Spectra (b) Image of SEM (c) FTIR spectra (d) UV-visible spectra

3.2. Scanning Electron Microscopy

The morphology of Polyaniline was studied using scanning electron microscopy (SEM). SEM image of pure Polyaniline shows jelly like structures as shown in figure 1 (b).

3.3. FTIR Spectroscopy

FTIR spectrum of the PANI was obtained in the range $400\text{--}4000\text{ cm}^{-1}$ as shown in Figure 1 (c). The FTIR spectrum of PANI shows main peaks at 749, 876, 1211, 1372, 1632, 3293 cm^{-1} . FTIR spectrum of PANI shows strong absorption bands, in the region

$1100\text{--}1700\text{ cm}^{-1}$, which correspond to the characteristics of conducting Polyaniline. The observed peak at frequency 749 and 876 cm^{-1} , that is because of NH Wagging, position of the band depends on the degree of the H bonding. The observed peak at the frequency 1211 cm^{-1} due to C-N stretching vibration, which is unconjugated C-N linkage and coupled with the stretching of adjacent bands. The absorption peak at the frequency 1372 cm^{-1} is due to C-N stretching for quinoid ring. The observed peak at the frequency 1632 cm^{-1} , which is the characteristic of the C=C stretching of quinoid ring. The absorption peaks at frequency 3293 cm^{-1} which is characteristics of N-H stretching. Peaks at 749, 876, 1211, 1372, 1632, 3293 cm^{-1} belonging to the conducting emeraldine salt form of PANI proves that PANI have an electrical conductivity, similar peaks are reported by Ersel Ozkazan et al [9].

3.4. UV-Visible Spectroscopy

The UV-vis absorption spectrum of the Polyaniline is recorded at room temperature by using a spectrophotometer between the wavelength range $200\text{--}800\text{ nm}$. Optical spectroscopy is an important technique to understand the conducting states corresponding to the absorption bands of inter and intra gap states of conducting polymers [10]. Figure 1 (d) illustrates the absorption peaks at 300 and 440 nm. The observed intense band at 300 nm is due to the $\pi\text{--}\pi^*$ transition of benzenoid ring which is related to the extent of conjugation between the adjacent Phenylene rings in the polymeric chain and the forced planarization of π -system induced by aggregation [11]. It leads to increased conjugation and thus lowers the band gap [12]. The transition of $\pi\text{--}\pi^*$ of benzenoid ring and the formation of polaron band in the nanocomposites are responsible for increase of the electrical conductivity of the nanocomposites [13]. The peak at 440 nm is due

to polaron-transition and shift of electron from benzenoid ring to quinonoid ring [14].

4. CONCLUSIONS

X-ray diffraction spectrum shows that chemically synthesized Polyaniline is inherently amorphous and hence there are no sharp peaks. SEM image of Polyaniline tends to aggregate in jelly like structures. FTIR spectrum of PANI shows strong absorption bands, in the region 1100–1700 cm^{-1} , which correspond to the characteristics of conducting Polyaniline. In the UV-vis spectrum shows two major absorption peaks at 300 and 440 nm. The observed intense band at 300 nm is due to the π - π^* transition of benzenoid ring.

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