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Synthesis, characterization by AC conduction and antibacterial properties of polyaniline fibers

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ABSTRACT

The present paper aims at to study the preparation of Polyaniline fibers and characterize them. Polyaniline fibers were synthesized by chemical oxidation polymerization where ammonium per sulfate $[(NH_4)_2S_2O_8]$ was used as oxidant to oxidize Aniline to Polyaniline. The Polyaniline fibers were characterized by XRD, FTIR, TEM and UV-Visible Spectroscopy. The characteristic peaks in FTIR and UV-Visible spectra confirmed the expected structure of polymer as reported in the literature. Further, Transmission electron microscopy (TEM) confirmed the formation of Polyaniline fibers. The A.C. conductivity, dielectric constant ($\mathfrak{E}'(w)$) and DielectricLoss ($\mathfrak{E}''(w)$) of Polyaniline fiber were measured using impedance analyzer. The antibacterial activity was examined for this Polyaniline nano fibers and it was observed that PANI could be used as an antibacterial agent.

Keywords: A. C. conductivity, dielectric constant, dielectric Loss and antibacterial activity

INTRODUCTION

Conducting polymers have electronic properties of semiconductor and at the same time, the processing advantages and mechanical parameters of polymer. Conducting polymers are the novel class of synthetic metals with applications in the field of sensors, EMI shielding, polymer cells and organic LED. Among them, conducting Polyaniline (PANI)[1], [2] is one of the most studied and promising conducting polymers due to its environmental stability, low cost and good electrical conductivity. This polymer can be used for the metallic parts in components like capacitors, displays, light-emitting diodes etc. Measurements of dielectric properties are required in electronic applications.[3], [4]

Different methods are used to fabricate Polyaniline nano fibers, which include electro spinning ,ultrasonic irradiation, interfacial polymerization and rapid polymerization.[5]–[7] One-dimensional PANI nanostructures, including nanowires, nanorods, and nanotubes, have been studied. By varying the particle size, shape, composition and the extent of dispersion of Nanoparticles,[8] the properties can be changed and tuned to the desired applications [9]. Among these methods Polyaniline fibers can be produced with ease with rapid polymerization method.[10],[11]. In the present study Polyaniline fibers were synthesized by rapid polymerization technique. UV- Visible spectroscopy, FTIR, XRD, TEM, Dielectric measurements and antibacterial properties were done on Polyaniline fibers. The antibacterial effect of conducting Polyaniline has been investigated with different bacterial strains.[12]

MATERIALS AND METHODS

2.1 Materials

Aniline, Ammonium per sulphate and methanol were purchased from S.D. Fine Chem. Ltd. In all cases where solution of an acid or base in a specific concentration was needed, the solution was made by using deionized water.

2.2 Synthesis of polyaniline nano fibers

PANI nano fibers were synthesized by chemical oxidation polymerization where ammonium persulfate $[(NH_4)_2S_2O_8]$ was used as oxidant. At first, 2 ml aniline was dissolved in 1.5 M, 70 ml HCl solution in a 200 ml beaker. In another beaker, 4 g of $(NH_4)_2S_2O_8$ dissolved in 20 ml deionized water. This solution was rapidly poured into the above acidified aniline solution under constant stirring. The stirring continued at constant speed for 5 h and at a constant temperature of 25^oC. After leaving 24 hrs in unstirred condition, the whole solution was filtered, washed with deionized water followed by Acetone several times to get clear filtrate (pH~2), and dried in Oven at 70^oC to get green emeraldine salt form of PANI.

2.3 Characterisation

U.V visible absorbance spectra were recorded on Double beam UV-Spectrometer (Systronics – 2201). The wavelength range were observed from 200 - 1100nm. Wide Angle X-Ray powder diffraction is used to obtain information about the structure, composition, and state of Polyaniline materials. X-ray Diffractometer was used to characterize the samples with Cu K alpha ($1.5406 A_0$) radiation using philips PW 1530 Diffractometer. The diffraction patterns were collected at an diffraction angle 2theta from 0⁰ to 80⁰ at a scanning rate and step size of 1⁰/min and 0.05 respectively. Impedance analyzer measures the intrinsic electrical properties like capacitance, dissipation factor.. In the present work the measurements were done using Agilent 4980/ A impedance analyzer in the frequency range 20 Hz to 2 MHz. **3 Results and Discussions**

3.1 UV-Visible Spectra

The electronic absorption of conducting polymers is useful in investigating the oxidation and doping state of the polymer. The UV -Visible spectra of the synthesized PANI-is shown in figure 1. The peak in the wave length range of 250 to 290 nm is due to Π - Π * transition of Benzenoid ring. The peak at 340nm is due to Polaron – Π * transition. Broad band with absorption maximum at 620 nm corresponds to the transitions from localized benzene molecular orbital to a Quinonoid molecular orbital.

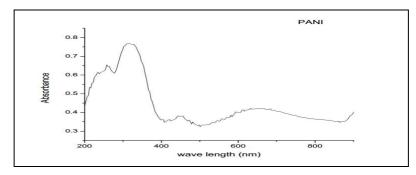


Figure 1. UV spectra of PANI

3.2 FTIR studies

The FTIR spectra of PANI is shown in figure 2. The IR vibrational frequencies for the PANI samples given in the literature are N-H stretching vibration of Benzenoid ring at -3380-3480 cm⁻¹, Qunonoid and Benzenoid ring units at 1603 cm⁻¹ and 1495 cm⁻¹, C-N stretching at -1240-1380 cm⁻¹ and Aromatic C-H in plane bending modes (quinonoid and benzenoid rings) at -1060-1150 cm⁻¹. In the present study, IR spectra were taken for the PANI samples. The spectra show the presence of relatively sharp peaks 1545cm⁻¹, 1483cm⁻¹, 1319cm⁻¹ (conducted protonated form with C-N. stretching mode of imine), 1113cm⁻¹ and 1065cm⁻¹ (broad band in plane aromatic C-H bending) in PANI. [13]

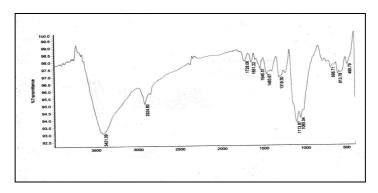


Figure 2. FTIR spectra of PANI

3.3 XRD studies

Figure 3 shows the XRD spectrum of a PANI .The XRD profile of PANI do not show sharp peaks thus suggest semi crystalline nature of polymer samples. Some peaks were observed at 7^0 , 20.5 0 to 26 0 corresponding to (011),(020) and (200) planes of PANI.

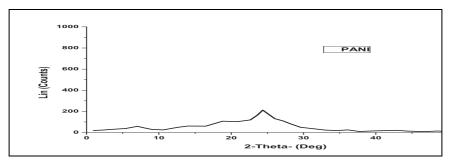


Figure 3 . XRD pattern of PANI

3.4 Transmission electron microscopy

Morphology of PANI was imaged using transmission electron microscope. TEM images are shown in Figure 4. PANI nano fibers are observed in the sample. The images reveal agglomeration of nano fibers.

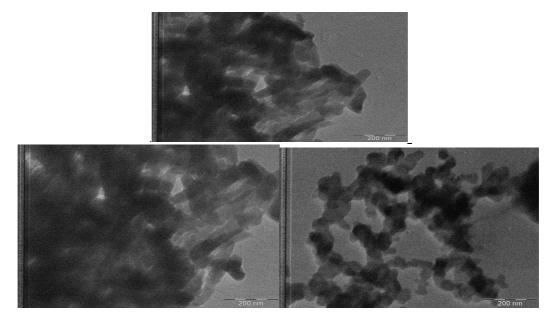


Figure 4. TEM images of PANI

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3.5Electrical investigation

Electrical investigation is one of the most important characteristics of a conducting polymer, especially to explore their use in electrical devices. An attempt was made to measure a.c conductivity and dielectric measurements. For electrical properties the powder was made in to pellets by uniaxial pressing,

3.5.1 D.C. Conductivity

D.C. Conductivity measurements for pressed pellet were measured. Resistance of the sample was found at room temperature . DC Conductivity was measured by the formula

 $\sigma dc = (L / R^* A) \qquad -----(1)$

where R is the resistance, L is the thickness of the pellet and A is the area of cross section of the pellet. The D.C conductivity of PANI nano fiber was found to be 0.588 S/m.

3.5.2 Dielectric measurement

For dielectric measurement the capacitance (C) and the dissipation factor (D) for the sample was measured using an Agilent 4980/A impedance analyzer in the frequency range of 20Hz – 2M Hz. Figure 5 represents the variation of dielectric constant (ϵ ') and loss tangent or dielectric loss (ϵ '') with logarithmic frequency respectively for PANI sample. The high dielectric constant at low frequencies reveals that the system exhibit strong interfacial polarization. [14], [15]

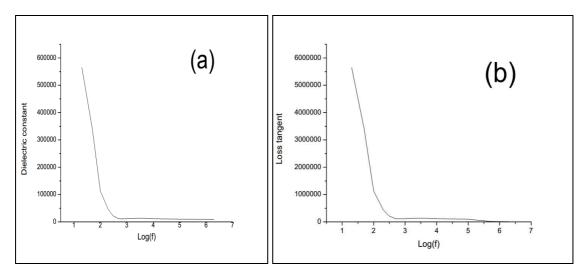


Figure 5. (a) Dielectric constant of PANI as a function of frequency (b) Loss tangent of PANI as a function of frequency

3.5.3 A.C. Conductivity

The frequency dependent conductivity of PANI is shown in Figure 6. Using the values of capacitance (C) and dissipation factor (D) recorded by impedance analyzer, AC conductivity is calculated by following equations

 $\mathbf{C} = \mathbf{C}'(\mathbf{w}) \mathbf{C} \mathbf{A} / \mathbf{d}$ -----(2)

Here A is the cross section of the area of the capacitor, d is the separation between the plates and $\epsilon_0 = 8.85 \times 10^{-12}$ F/m is the absolute permittivity of free space. The other measured quantity, the dissipation factor given by

 $D = \notin "(w) / \notin (w)$ -----(3)

and can be used to extract the imaginary part of the dielectric function $\notin ``(w)$. The a.c. conductivity (σ ac) is obtained from using the relation

 $\sigma ac = \varepsilon 0 \in (w) \omega D$ -----(4)

where $\omega(=2\pi f)$ is the angular frequency. Thus, σac depends strongly on the frequency of the applied field. At low

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frequencies AC conductivity is almost constant and increases with frequency. This increase can be attributed to the presence of polarons and bipolarons in PANI.[16], [17]

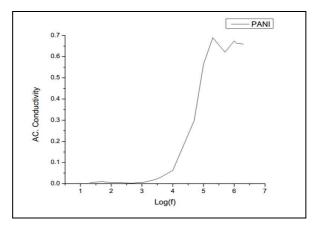


Figure 6. A.C conductivity of PANI as a function of frequency

3.6 Antimicrobial Activity

3.6.1 Materials and Methods:

The Antimicrobial activity of the samples was tested against Klebsiella sps and Staphylococcus sps by Agar Well Diffusion method. Agar diffusion method is a means of measuring the effect of an antimicrobial agent against bacteria. 100μ l of the culture was inoculated over the dried surface of nutrient agar. Wells bored in the agar were impregnated with a concentration of 2mg, 3mg and 5mg of PANI. The compound diffuses from the well into the agar. The concentration of the compound will be highest near the well and will decrease as distance from the well increases. If the compound is effective against bacteria at a certain concentration, no colonies will grow where the concentration in the agar is greater than or equal to the effective concentration creating a zone of inhibition. Thus, the size of the zone of inhibition is a measure of the compound's effectiveness: the larger the clear area around the well, the more effective is the compound.



Figure 7. Photographic image of zones of inhibition at various concentrations of PANI on Staphylococcus sps and Klebsiella sps

RESULTS AND DISCUSSION

TABLE I INHIBITION ZONES AT VARIOUS CONCENTRATIONS OF PANI ON KLEBSIELLA SPS AND STAPHYLOCOCCUS SPS

Organism	PANI		
	2mg	3mg	5mg
Klebsiella	15mm	18mm	18mm
Staphylococcus	18mm	19mm	19mm

From Table 1 it is evident that PANI nano fibers is effective against *Klebsiella sps and* Staphylococcus sps. The Photographic image of zones of inhibition of PANI on Klebsiella sps and Staphylococcus sps is shown in Fig.7 .Our present study shows that polyaniline matrix exhibited antibacterial activity against the bacterial species tested. Pol-

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yaniline was found to contain antibacterial activity due to various factors such as surface hydrophilicity, length of the polymer chain, low molecular weight [18], electrostatic adsorption between the Polyaniline and bacteria [19], direct contact between material and bacterial cells and the presence of amino groups[20], [21].

CONCLUSION

PANI fibers were synthesized by using ammonium persulphate as oxidizer. The synthesized PANI was found to be in emeraldine Salt form as indicated by the green The characterization methods employed i.e. UV - Visible, FTIR, and XRD studies confirmed the expected structure of polymer as reported in the literature. TEM images confirmed the formation of PANI fibers. Conductivity and dielectric properties such as dielectric constant and dielectric loss have been measured. It is observed that there is an increase in ac conductivity for the PANI sample at higher frequencies. It was evident that PANI nano fibers is effective against *Klebsiella sps and* Staphylococcus sps. We anticipate that PANI would be good candidate for fabrication of Bio medical and biosensor devices.

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