

SUMMARY OF THE THESIS

The present thesis deals with the “**Synthesis and Characterization of Conjugated polymers**”. Conjugated polymers are the most recent generation of polymers [1]. They are called “*fourth generation of materials*”. Polymeric materials in the form of wood, bone, skin, and fibers have been used by man since prehistoric time. Although organic chemistry as a science dates back to the eighteenth century, polymer science on a molecular basis is a development of the twentieth century. The saturated polymers studied by Staudinger, Flory, Ziegler, and Natta were insulators, and were viewed as uninteresting from the point of view of electronic materials. However, A. J. Heeger, Alan Mac Diarmid and H. Shirakawa have changed this view with their discovery of conjugated polyacetylene that can be made conductive almost like a metal. In saturated polymers all of the four valence electrons of carbon are used up in covalent bonds but in conjugated polymers the electronic configuration is fundamentally different. The idea of utilizing the electrically conducting properties of polymers was not proposed until the 1960’s [2] but since then polymers have been used as active components in a variety of applications. In 1977, it was discovered that an alternating-bond conjugated polymers—trans polyacetylene, could be doped and thereby transformed into a good electrical conductor [3]. This discovery led to the Nobel Prize in chemistry 2000, awarded to Prof. A. J. Heeger, A. G. Macdiarmid, and H. Shirakawa. Early on, most of the research effort was concentrated to the conducting properties of the doped alternating-bond conjugated polymers [4] and these polymers were often referred to as “conducting polymers”. After the discovery of polyacetylene, various conjugated polymers have been discovered. e.g. polyaniline [5], poly phenylene vinylene[6], poly para phenylene [7]. The discovery of conducting polymers has become even more significant in hindsight as this class of materials has proven to be not only of intrinsic scientific interest but also of great technological promise. Conducting polymers have been put to use in such niche applications as electromagnetic shielding, antistatic coatings on photographic films, and windows with changeable optical properties. And the undoped polymers, which are semiconducting and sometimes electroluminescent, have led to even more exciting

possibilities, such as transistors, light-emitting diodes (LEDs) [8], polymer solar cells [9, 10] and photodetectors.

Conjugated polymers have a framework of alternating single and double carbon–carbon (sometimes carbon–nitrogen) bonds. The chemical bonding leads to one unpaired electron (the π -electron) per carbon atom. Moreover, π -bonding, in which the carbon orbitals are in the sp^2pz configuration and in which the orbitals of successive carbon atoms along the backbone overlap, leads to electron delocalization along the backbone of the polymer [11]. This electronic delocalization provides the “highway” for charge mobility along the backbone of the polymer chain. As a result, therefore, the electronic structure in conducting polymers is determined by the chain symmetry (i.e., the number and kind of atoms within the repeat unit), with the result that such polymers can exhibit semiconducting or even metallic properties. It is the delocalized pi-electron system that allows the polymers to support negative and positive charge carriers along the polymer chain. However, conjugation is not enough to make polymer conductive. Doping of conjugated polymers involves random dispersion or aggregation of dopants in molar concentrations in the disordered structure of entangled chain and fibrils. Dopant increases the number of charge carriers in the form of extra electrons or holes to be injected in the material. Doped conjugated polymers show high electrical conductivities, whereas conjugated polymers in undoped states are either semiconductor or insulators.

Electrical conductivity is one of the important parameter to understand the electrical transport in conjugated polymers. Conjugated polymers are amorphous in nature with short conjugation lengths. Therefore it has been suggested that electrical conduction take place by charge hopping between polymeric chains. The electrical conductivity in homogeneous systems can be well explained by quasi particles such as polaron, bipolaron and solitons [12]. In heterogeneous systems the structure is not uniform but rather a more disordered or branched one. In this type of system the charge transport along the polymer chains take place by hopping or tunneling process.

Conjugated polymers can be divided into two types on the basis of the ground states, i.e. *degenerate* and *non-degenerate*. In non degenerate polymers, the alternating single and double bonds lead to electronic structures of varying energy levels and has no two identical structures in ground state [13]. Polyaniline (PANI), and derivative of PPV e.g.

poly [2-methoxy (5, 2'-ethyl hexyloxy) 1, 4 phenylene vinylene)] (MEH-PPV) belong to this class. We have categorized PANI and MEH-PPV into two different groups on the basis of the applications. Keeping in view of importance of the subject, and applications of conjugated polymers we have selected the studies of both types of conjugated polymers i.e. *polyaniline* and *MEH-PPV*.

In the present thesis, a comprehensive study has been conducted to investigate the electrical, thermal and optical properties of conjugated polymers for the use in optoelectronic devices such as organic solar cells and LED. In the present thesis we have studied the two conjugated polymers i.e. *Polyaniline* and *MEH-PPV*.

PANI comes in the first group which utilizes their conductivity as its main property. It can be used as electrostatic materials, conducting adhesives, electromagnetic shielding, printed circuit boards, artificial nerves, antistatic clothing, piezoceramics, active electronics (diodes, transistors), aircraft structures and recordable optical disks [14]. *PANI* is used because of its diverse, but unique properties. The unique reversible proton dopability, excellent redox recyclability, environmental stability, low cost, easy to synthesize and variable electrical conductivity allowing its potential application in various fields. We have taken Tellurium (Te), for the doping of *PANI*. Te is one of the important chalcogen, and shows greater conductivity in certain directions, depending on the alignment of the atoms. It is used for production of thermo junctions, photocells and thermocouple to measure low temperature. Te alloys are used in phase-change recording materials [15].

MEH-PPV comes in the second group of applications, which utilizes the electroactivity character property of the materials. *MEH-PPV* has good environmental stability, thermal stability up to 400 °C, easy conductivity control, cheap production in large quantities and solubility in common organic solvents. It can be used in molecular electronics, electrical displays, chemical, biochemical and thermal sensors, rechargeable batteries and solid electrolytes, drug release systems, optical computers, ion exchange membranes, electromechanical actuators, 'smart' structures, switches, light emitting diodes, solar cells. *MEH-PPV* is a light emitting polymer, and attracts great interest because of their electroluminescent applications. In order to improve the dissociation of excitons and charge transport to the electrodes, *MEH-PPV* has been associated to electron acceptors

like bulkminister fullerene (C_{60}) and organic dyes like perylene and coumarin. This donor-acceptor network leads to an extremely rapid electron transfer from polymer to the dye with a very low back transfer that induces a higher dissociation rate from quenching of MEH-PPV photoluminescence. Thus dye doping can be used to enlarge the spectral response of photovoltaic devices.

This MEH-PPV polymer was doped with different organic dyes. Two organic dyes have been used for the doping of MEH-PPV. To our knowledge, these dyes were never used for doping of MEH-PPV. The first dye is Acridine orange (Fluorescent dye) and the other one is Nile blue (laser dye). These dyes are chosen as emissive material for MEH-PPV (Donor material) and they behave like as an acceptor because MEH-PPV act like as p-type semiconductor.

Thus the scientific interest in this field is rapidly expanding (growing) and a lot of work on electrical, optical, thermal and structural properties is available in the literature. However, many questions and queries have yet to be explained satisfactorily, viz

- (i) The mechanism of electrical transport.
- (ii) The exact nature and distribution of the localized state arising due to intrinsic defects in these materials.
- (iii) The structure and chemical bonding in conjugated polymers.

To explain these fundamental questions we have proposed to study the electrical transport properties in bulk sample of PANI and MEH-PPV doped films. Hence, in the present thesis work, we have undertaken the following:

- ❖ Chemical synthesis, doping of polyaniline with tellurium and its characterization by different techniques such as electrical, thermal and spectroscopic (FTIR spectroscopy).
- ❖ The doping of different organic dyes (Acridine orange, Nile Blue) in MEH-PPV, which makes them suitable material for polymer photovoltaics (PVs) and polymer light emitting diodes (PLEDs). Organic dye doped MEH-PPV films have been characterized by electrical (dc, photoconductivity, I-V measurements), photoluminescence and FTIR studies.

The present research work describes the electrical, thermal and optical properties of PANI and MEH-PPV. Attempts are being made to prepare the doped PANI and MEH-PPV.

We have divided our research work into two parts. In the first part, we have studied the spectroscopic, thermal and electrical properties of PANI doped with the Te.

In the second part, we have studied the electrical transport and optical properties of organic dyes (Acridine orange & Nile Blue) doped MEH-PPV films. The experimental techniques and results of all the studies are given below:

1. Electrical, thermal and spectroscopic studies of Pani doped with Te:

In the present research work, PANI has been synthesized by chemical oxidative polymerization method using ammonium persulphate as an oxidant suggested by Macdiarmid et al. The synthesized polyaniline was doped with different concentration of Te, which was dissolved in con. H_2SO_4 . The dopant solution of different concentration such as 0.25%, 0.5%, 1.00%, 1.25%, 2.5%, 10%, 25% (w/v) was added to same amount of swollen polyaniline (swollen by THF) and was kept for 24 h. We have also made a separate solution of PANI in H_2SO_4 to see its effect on PANI chain. After doping the dried polyaniline was then grounded to make fine powder and used for making the samples (pellets). Bulk samples in the form of round pellets were obtained by finely grinding the polyaniline powder and then compressing the powder under a pressure $\cong 6.6$ ton. The doped PANI was characterized in bulk form (pellets) by different characterization methods. Electrical, thermal and spectroscopic studies have been investigated for Te doped Pani and we got fruitful results in Te doped PANI.

Electrical dc conductivity was measured in a specially designed metallic sample holder [16] using two-probe technique in the temperature range from 300-400 K. Voltage of 1.5 volts (from a dry cell) was applied across the pellet and the resulting current was measured with a Keithley electrometer (Model 617).

Thermal analysis is carried out by Differential Scanning Calorimeter (Model-DSC plus, Rheometric Scientific Co. U.K.). DSC scans were obtained for undoped and all tellurium doped PANI samples by heating accurate weighed sample (4 to 7 mg) at a constant heating rate of $10^{\circ}C/min$. DSC scans were also taken for undoped and 0.5 % (w/v) Te

doped PANI samples at four different heating rates i.e. 5,10, 15, 20 °C/min. The temperature range covered in DSC was from room temperature to 300 °C.

FT-IR spectra of polyaniline-KBr pellets were obtained by using Perkin-Elmer FTIR spectrometer in the range from 4000-400 cm⁻¹ at a resolution of 4 cm⁻¹. The chemical structure of PANI, sulphuric acid doped PANI and all tellurium doped PANI samples were characterized by FTIR spectroscopy.

We found that the dc conductivity of doped PANI increases by six orders of magnitude after incorporation of Te. The conductivity increases and activation energy decreases after doping of tellurium in pure samples of PANI. An increase of the σ_{dc} after doping can be explained in terms of increased hopping conduction in impurity induced systems [17]. The activation energy (ΔE) alone does not provide any information whether the conduction takes place in extended states or by hopping in the localized states. These two conduction mechanisms can be distinguished on the basis of the pre-exponential factor (σ_0). It was suggested by Mott [18] that the pre-exponential factor (σ_0) for conduction in the localized states should be two or three order smaller in the magnitude than for conduction in the extended states, and should become still smaller for conduction in the localized states near Fermi level. The observed value of σ_0 is still smaller for undoped and doped PANI sample. Therefore, in PANI samples conduction takes place by hopping process in localized state near to Fermi level.

The conductivity can also be explained on the basis of the formation of polarons and bipolarons. The most commonly accepted model is that of polaron formation upon oxidation and combination of polarons gives bipolarons. The bipolarons are then free to move along the polymer chain, which gives rise to the electronic conductivity. So, these Te doped samples can be used for optical storage media and in other application.

In thermal studies, it has been investigated that glass transition temperature shifts towards higher temperature on increasing the heating rate. The increase in glass transition temperature (T_g) could be attributed to the increase in molecular weight of polymer with increasing tellurium concentration or to the increase in concentration of PANI-Te chain. The calculated enthalpy (ΔH) increases with increasing heating rate.

FTIR spectra corroborate the structural changes of PANI after doping of tellurium and also confirm the incorporation of tellurium in polymeric chain. Results have been published in *Current Applied Physics [Volume7 (2007)68-75]*.

2 Electrical properties of MEH-PPV doped with acridine orange (AO) and Nile blue (NB) dye.

(a) Electrical properties of AO dye

The electrical properties of poly [2-methoxy-5 (2'-ethyl hexyloxy)-1,4-phenylene vinylene] (MEH-PPV) films were studied [19] at different concentration of acridine orange (AO) dye in the temperature range (293-416 K). MEH-PPV was dissolved in n-butyl acetate and was doped with different concentration of acridine orange (AO) dye solution. The MEH-PPV films were prepared by spin coating technique with the speed of 1000 rpm. For conductivity measurements, Cu electrode contacts in planar configuration were made on the top surface of the film by RF sputtering. The dc conductivity and steady-state photoconductivity measurements were performed on these spin coated thin films. A specially designed metallic cryostat was used to measure the dark conductivity and photoconductivity of the films. The measurements were conducted in the vacuum of about 10^{-3} Torr. For photoconductivity measurement, white light of a tungsten lamp (100 W) was used to irradiate the films. Before measuring the dc conductivity and photoconductivity, the films were annealed at 350 K for 30 min.

The temperature dependence of dc conductivity for various MEH-PPV films doped with AO dye increases exponentially with single activation energy in the temperature range (293-416K), which indicates that conduction in these polymer films occurs through an activated process. On incorporation of AO dye, the dc conductivity increases whereas the ΔE and σ_0 decreases, which indicates that conduction is taking place through the wide range of localized state near the Fermi level. From the value of pre-exponential factor (σ_0), it has been noticed that as the concentration of dye increases, the conduction process changes from extended states to the localized states. In doped MEH-PPV films the dark conductivity as well as photoconductivity increases by two orders of magnitude to that of pure MEH-PPV film. Photoconductivity measurements are useful in determining the nature of optical charge generation and transport in conjugated polymers. The value of T_0

(measurement of disorderness) is in good agreement with the XRD spectra, which depicts the amorphous nature of the pure MEH-PPV film. The value of T_0 decreases with an increase in the concentration of AO dye, which confirms partially crystalline nature of the dye-doped MEH-PPV films.

Photocurrent dependence on incident radiation has also been observed which follows the power law ($I_{ph} \propto F^\gamma$). This dependence shows that the recombination with in the localized state is predominant. We have observed low photosensitivity in the doped MEH-PPV films at 30 mW/cm^2 for white light. The values of γ for different samples for MEH-PPV films are in between 0.5 and 1, which shows the existence of continuous distribution of localized states in the mobility gap of these materials. Steady state photoconductivity measurements show that the dependence of σ_{ph} on intensity is governed by the localized recombination. These results have been published in *Physica B [Volume 393(2007) 310-315]*.

The Current-Voltage characteristics of AO dye doped MEH-PPV films prepared by spin-coating technique on ITO coated glass substrate have been determined [20] as a function of temperature. The temperature of the sample could be varied from 295-395 K. Voltage of 30 volts from a regulated DC power supply was applied across the films and the resulting current was measured with a digital Pico-Ammeter (Model DPA-111). For the cathode contact, Aluminum was deposited using RF sputtering technique so that the current is predominantly due to holes. The current densities increase exponentially with the increased forward bias voltage for different AO dye doped MEH-PPV films. The conductivity increases by two orders of magnitude as the AO dye concentration increases to 2.65 molL^{-1} . These results have been accepted for publication in *Solar Energy Materials and Solar cells (2007)*.

(b) Electrical properties of NB dye

The J-V characteristics of the MEH-PPV films doped with different concentrations of NB dye were studied in dark and in illumination corresponds to the measurement of the current produced due to the application of varying electric field (voltage) both in forward and in reverse direction. The current is measured as a function of voltage at room

temperature (295 K). We have calculated conductivity in dark from these J-V measurements at 3volts.

The current density increases with increasing voltage for different NB dye doped MEH-PPV films. The conductivity decreases as the NB dye concentration increases. It can be noticed from the J-V curve that in MEH-0NB sample the structure shows ohmic behavior. On doping with Nile blue (NB) dye, It has been observed that the structure shows non-ohmic (diode) characteristics, which make it suitable for photovoltaic applications.

On comparing dark and illumination J-V characteristics for MEH-2.39 NB device, It is observed that on illumination the J-V shifts to the fourth quadrant which indicate that this device now generating power. Various important photovoltaic parameters of the film were evaluated by these characteristics. This indicates a short circuit current (J_{sc}) density of $2.21 \times 10^{-5} \text{ A/cm}^2$ and opens circuit voltage (V_{oc}) of 4.8 Volts. The calculated fill factor is 0.44. An energy conversion efficiency of 0.046 % has been obtained for the NB dye doped MEH-PPV device.

3. Optical properties of MEH-PPV doped with acridine orange (AO) and Nile blue (NB) dye.

(a) Optical properties of AO dye

The AO dye concentration has been optimized on the basis of absorption spectra and PL studies carried over the films particularly for photovoltaic applications. The UV-visible absorption spectra of pure and AO dye doped MEH-PPV films, were obtained using Perkin Elmer lambda spectrophotometer in the wavelength range of 200-800 nm. The photoluminescence spectra of films were recorded in the wavelength range of 500-700 nm using a Perkin Elmer LS55 luminescence spectrometer. The excitation wavelength was 465 nm.

The absorption studies show that the addition of AO dye does not significantly change (reduce) the absorption intensity, which indicates the suitability of photon absorption. The photoluminescence (PL) spectra were recorded for MEH-PPV films, which become narrow on increasing the AO dye concentration. The PL studies indicate that up to certain concentration ($1.98 \times 10^{-6} \text{ molL}^{-1}$) of AO dye in MEH-PPV films, PL intensity decreases as compare to that for undoped MEH-PPV film. All these three parameters are favorable

for good photovoltaic devices based on MEH-PPV films doped with AO dye. The increase in conductivity is expected to give the better fill factor and hence, higher efficiency of photovoltaic devices. It is concluded that we optimized the AO dye concentration for a typical MEH-PPV based photovoltaic devices.

The combined effect of increase in conductivity by two orders, decrease in PL intensity and no significant change in absorption makes the material suitable for photovoltaic applications.

However, it may be pointed out that if the concentration of AO dye increases further then the material is no more useful for photovoltaic applications and starts becoming more favorable for LED application as indicated by increase in PL intensity. As it is clear that after incorporation of dye, an optimized concentration (2.65×10^{-6} mol/L) shows an increase of photoluminescence significantly indicating more radiative transitions. This condition is favorable for LED application and the EL emission efficiency is likely to increase with the incorporation of dye with optimized concentration. These results have been accepted for publication in *Solar Energy Materials and Solar cells* (2007).

(b) Optical properties of NB dye

The UV-visible absorption spectra of pure and NB dye doped MEH-PPV films were obtained using Shimadzu (UV-2550) spectrophotometer in the wavelength range of 300-800 nm. The photoluminescence spectra for films were recorded in the wavelength range of 500-700 nm using a Jobin Yvon, Horiba spectrofluorometer. The excitation wavelength was 500 nm.

The optical properties of doped MEH-PPV films were carried out by absorption and fluorescence spectra. The absorption spectra of NB dye doped MEH-PPV films show the absorption edge at 370 nm and absorption peak at 500 nm. The absorption edge at 370 nm corresponds to the $n-\pi^*$ transition while the absorption peak at 500 nm corresponds to the $\pi-\pi^*$ transition. The absorption spectra become broad after incorporation of dye, which may be due to the distribution of MEH-PPV polymer chain length over the wide range. After incorporation of NB dye the peak intensity in doped MEH-PPV films increases significantly. It reveals that the incident energy absorbed by the dye leads to photo carrier generation rather than photoluminescence.

The PL of undoped MEH-PPV (MPV-0 NB) shows only one peak at 590 nm and while in spectra of NB dye doped films there is no significance peak. It is observed from photoluminescence spectra that the intensity of MEH-PPV films decreases very much as the NB dye concentration increases, which suggests that the dye separates the charges, photo generated by the MEH-PPV before radiative recombination occurs. At high dye concentrations, in PL, singlet excitons are captured and immediately decay both on the dye, which explains the slight reduction of PL intensity. Exciton dissociation is favorable when the difference in electron affinity and ionization potential between the polymer and blended compound is large i.e. larger than the exciton binding energy. Photoluminescence quenching is used as an indication for efficient charge transfer. The effective PL quenching is also observed in MDMO-PPV, DE 69 and DE 21 polymers in the presence of PCBM, which indicates an efficient charge transfer from these polymers to the acceptors PCBM [21, 22]. The optical band gap (E_g) was also measured for pure and NB dye doped MEH-PPV films and there is very small increase in E_g after doping. The small band gap differences between the polymer and dye favour charge transfer, and hence the use in efficient photovoltaic devices [23]. This research work has been communicated for publication in *Journal of Materials Science (Materials in Electronics)* (2007).

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LIST OF PUBLICATIONS

PUBLISHED IN INTERNATIONAL JOURNALS /INTERNATIONAL CONFERENCES:

- i. **Electrical, Thermal and Spectroscopic studies of Te doped Polyaniline.**
Samrana Kazim, V. Ali, M. Zulfequar, M. Mazharul Haq, M. Husain
Current Applied Physics, 7/1 68-75 (2007)
- ii. **Electrical transport properties of MEH-PPV thin films doped with Acridine orange dye.**
Samrana Kazim, V. Ali, M. Zulfequar, M. Mazharul Haq, M. Husain
Physica B 393(2007)310-315.
- iii. **Electrical and optical properties of thin films based on poly [2-methoxy-5 (2'-ethyl hexyloxy)-1,4-phenylene vinylene] doped with acridine orange dye with possible photovoltaic applications.**
Samrana Kazim, M. Zulfequar, M. M. Haq, P. K. Bhatnagar, M. Husain.
(Solar energy materials and solar cells)(2007) Published online
- iv. **Electrical and optical properties of thin films based on poly [2-methoxy-5 (2'-ethyl hexyloxy)-1, 4-phenylene vinylene] doped with acridine orange dye.**
Samrana Kazim, X. Mathew, M. Zulfequar, M. Mazharul Haq, P. K. Bhatnagar, M. Husain.
Proceedings of XV International Material Research congress, Cancun, Mexico, 20-24 August (2006).
- v. **Spectroscopic studies of Nile blue dye doped poly [2-methoxy-5 (2'-ethyl hexyloxy)-1, 4-phenylene vinylene] thin films".**
Samrana Kazim, M. Husain
4th International Conference on Materials for Advanced Technologies 2007 (ICMAT 2007) 1 to 6 July 2007, Singapore.

COMMUNICATED:

- i. **Electrical and optical characterization of thin films based on poly [2-methoxy-5 (2'- ethyl hexyloxy)-1,4-phenylene vinylene] doped with Nile Blue dye.**
Samrana Kazim, M. Zulfequar, M. Mazharul Haq, M. Husain.
Journal of Material Science (Materials in electronics)
- ii. **Optical studies of Nile blue dye doped poly [2-methoxy-5 (2'-ethyl hexyloxy)-1, 4-phenylene vinylene] films for photovoltaic applications”.**
Samrana Kazim, M. Husain
Proceedings of XVI International Material Research congress, Cancun, Mexico, 19-23 August (2007).

PRESENTATIONS:

- i. **Synthesis and Characterization of Te doped polyaniline.**
Samrana Kazim, Vazid Ali, M.Zulfequar, M. Mazharul Haq, M.Husain
Proceeding of International workshop on the physics of semiconductor Devices, NPL, Delhi, 13-17 December 2 (2005) 948-951.
- ii. **Dark and photoconductivity measurements of poly [2-methoxy-5 (2'-ethyl hexyloxy)-1, 4-phenylene vinylene] thin films doped with Acridine orange dye.**
Samrana Kazim M. Zulfequar, M. M. Haq, M. Husain
National conference on recent advances in molecular structures (NCMS-2007) July 19th -20th July 2007, Kancheepuram dt., Tamilnadu

WORKSHOPS AND CONFERENCES ATTENDED:

- ⊕ Workshop on nano-materials, Jamia Millia Islamia, New Delhi (March 11, 2003).
- ⊕ User awareness programme at NPL New Delhi.10-15 th January, 2004.
- ⊕ International Conference on Nano-technology, Career Institute of Technology and Management (CITM), Surajkund, Faridabad, (May 9-10, 2005).
- ⊕ One day seminar on “Applications of Conducting Polymers & Nanomaterials for Science & Technology”, Sept.-19, 2006, Jamia Millia Islamia, Delhi.