Chemical Synthesis of Polypyrrole Thin Films using Ferric nitrate as an Oxidant

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Abstract. In the present study, we deposited Polypyrrole thin films in the temperature range between 280-290 K, by simple, inexpensive and low cost successive ionic layer adsorption and reaction (SILAR) method. The chemically synthesized Polypyrrole thin films were further characterized by physico-chemical characterization. The formation of Polypyrrole thin film was confirmed by Fourier transform infrared (FTIR) spectroscopy. The surface wettability study was made by contact angle meter. The surface wettability study showed that the Polypyrrole thin films are in hydrophilic in nature. Also the optical study was made by measuring optical absorption with spectrophotometer.

Keywords: Polypyrrole, successive ionic layer adsorption and reaction (SILAR) method, Fourier transform infrared (FTIR) spectroscopy etc.

1. INTRODUCTION

Generally, conjugated polymers are prepared by either chemical oxidation method or electrochemical oxidation methods. One of the major advantages of chemical oxidation method is no requirement of the sofisticated instruments. However, electrochemical oxidation requires only conducting substrates. In chemical oxidation methods, the pyrrole monomer oxidized with oxidants. There are variety of oxidants are available for polymerization such as ammonium per sulphate, ferric cloride and ferric nitrate etc. In the present case we choose ferric nitrate as an oxidant for the chemical polymerization of pyrrole at 280-290 K.

2. EXPERIMENTAL

The materials such as Pyrrole were purchased from Spectrochem, P.V.T., LTD. Mumbai (India). Ferric nitrate was purchased from SD-fine chemical limited. Polypyrrole thin films were prepared by SILAR method. SILAR method consists of two precursor's bath. One contains 0.1 M pyrrole with electrolyte and another contains 0.1 M ferric nitrate with electrolyte. The whole system was kept in the bath of ice and NaCl salt for mainting the required temperature range 280-290 K. The schematic presentation of the experimental process is shown in figure 1. The dipping time in pyrrole solution and reaction time in ferric nitrate solution is same.

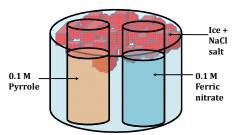


FIGURE 1: Schematic presentation of SILAR method

FT-IR spectra measurements were conducted using a Perkin ElmerRX1 FT-IR spectrometer. Polypyrrole films were peeled off from the substrate surface and their pellets were prepared with KBr. The absorption spectra measurements were performed with a Shimadzu UV-3600 UV-vis

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spectrophotometer in the wavelength range 300–800 nm. The surface wettability of the films was tested by measuring the contact angle (θ) of a water droplet of 1µL placed on the film surface using the contact angle meter equipped with a CCD camera (Ramehart Instrument Co., USA) at ambient temperature (~27 °C).

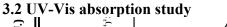
3 RESULTS AND DISCUSSIONS

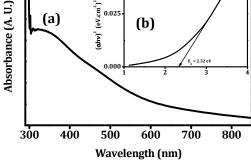
3.1 FTIR spectroscopy:

The FTIR spectroscopy of chemically synthesized Polypyrrole thin films showed that the different bonding between the elements.

FIGURE 2: FTIR spectroscopy of polypyrrole film.

The fig. 2 shows that characteristic absorption peaks of conducting polypyrrole thin films. A strong broad absorption band is observed at around 3443 cm⁻¹ corresponding to the N–H stretching vibrations. The band at about 1460 cm⁻¹ reflects the C–N stretching vibration in the ring. The strong peak at 782cm⁻¹, characteristic of a five-member aromatic ring was present [1].





From the UV-Vis absorption spectra, the absorption peak was observed at 350 nm due to π - π * transition [2], which confirms that the chemically synthesized polypyrrole thin films are conducting forms. The band gap energy was estimated from the $(\alpha hv)^2$ vs hv plot, the extrapolation to the enrgy axis at zero absorbance gives the direct band gap energy of polypyrrole thin film and it is found to be 2.32 eV.This results were matches with the earlier reported values[3].

3.3 Contact angle measurement:

Surface wettability measuremets were carried out with the help of contact angle measurement. Figure 4 shows the polypyrrole thin films are hydrophilic in nature. The water contact angle for the Polypyrrole thin film is 33°. Such hydrophilic nature of polypyrrole thin film is important for the supercapacitive applications.



FIGURE 4: Contact angle image of Ppy thin film.

4. CONCLUSION

The Ferric nitrate is a suitable oxidizing agent for preparation of good quality polypyrrole thin films, such as; conjugated form, semiconducting nature with band gap of 2.32 eV and hydrophilic with contact angle 33°.

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FIGURE 3: (a) UV-Vis absorption spectra and (b) Band gap energy plot.

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