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Research Article

**PREPARATION AND CHARACTERIZATION OF
POLYTHIOPHENE DOPED V₂O₅ THIN FILMS**

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Abstract:

Polymeric thin films of polythiophene and with different %w/vV₂O₅ were fabricated by chemical bath deposition method. Using Fecl₃ as an oxidant and chloroform as a solvent .the effect of dopant on the properties of polythiophene thin film was studied The complexation of the polymer was confirmed by Fourier transform infrared (FTIR) .the surface morphology was influenced by dopant.SEM images shows agglomerated nanoparticles . XRD analysis sample were confirmed noncrystalline and crystal structure has been modified after doping by V₂O₅. TGA-DTA result indicates composites of polythiophene are found to be more thermally stable than pure polythiophene.

Keywords: Polythiophene ,V₂O₅, chemical bath deposition, chemical structure etc

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1. INTRODUCTION:

The macromolecules that propagate charge along their structural format are Conducting polymers. Conductive polymers characterised by controllable conductivity and have special electrical and optical properties comparable to those of metal and inorganic semiconductors. These polymers are of substantial interest due to their electronic properties and their potential technological application [1]. Among the conducting polymers polythiophene is one of the promising material. Polythiophene (PTh) it is a heterocyclic polymer that has particular interest due to its properties that can be used in various applications, such as solar and photovoltaic cells [2, 3] biosensors, tissue engineering scaffolds [4], antistatic coatings. There are several routes for the synthesis of polythiophene like electrochemical and chemical; Chemical oxidative polymerization gives higher yield and hence is a better route for the polymerization of polythiophene [5]. The synthesis conditions and the nature of the dopant anion play a vital role in the polymerization process as they strongly affect the various properties of the material [6].

Polymer thin films are formed by incorporating a metal into highly polar polymer complexes. During the last few decades, the utilization of polymer light emitting diode in various applications has led to intensive interest in these materials [7-9]. Among various other methods of film deposition, chemical bath deposition (CBD) method appears most suitable for the integration in large scale fabrication process. The main advantages of the CBD method are its low cost, no requirement of sophisticated instrument, low processing temperature and non-polluting properties [10]. In the present article the properties of polythiophene and 0.5% w/v and 1% w/v V_2O_5 doped thin films prepared by chemical bath deposition are reported.

2. EXPERIMENTAL:

2.1 Sample preparation-

Thiophene (AR grade Merck), Iron chloride $FeCl_3$ (Sd-Fine), V_2O_5 , chloroform (CH_3Cl), methanol (CH_3OH), acetone were used for the preparation of polythiophene thin films. Micro slides of glass with dimensions 2.5 cm \times 7.5 cm were used as substrate. Initially, the substrate were washed with deionized water, boiled in chromic acid and washed with detergent, rinsed in acetone before deposition of thin film. Monomer solution was prepared by dissolving 0.1 M of thiophene in chloroform, the oxidant solution was prepared in a glass beaker with 0.5 M concentration of $FeCl_3$ in chloroform the ratio of monomer to oxidant was kept 1:5. In case of PTh

+ V_2O_5 composite material dissolved in chloroform and modified with different %w of V_2O_5 . The substrates were immersed in the bath at room temperature at constant stirring. The monomer solution was added drop wise in the oxidant solution the reaction being carried out at room temperature. During the precipitation, heterogeneous reaction occurred and the deposition of polythiophene took place on the substrate. The thickness of the polymer layer was controlled with help constant deposition time. The substrates coated with polythiophene thin films were removed after a time interval of 1 h from the bath, washed with chloroform followed by methanol and acetone repeatedly to remove residual oxidant and unreacted monomers, dried in air and preserved in an airtight container in the same way polythiophene doped with V_2O_5 thin film prepared by adding 0.5% w/ and 1% w V_2O_5 . For structural investigation, samples are characterized using different techniques such as elemental analysis, FTIR analysis, scanning electron microscope, XRD, Thermal analysis using TGA-DTA technique has also been carried out.

2.2 Analytical Techniques

In the present study

FTIR: Bruker Germany spectrometer at SAIF IIT Mumbai.

XRD: Rigaku MiniFlex 300/600 instrument at Vidyabharti college, Amravati.

TGA-DTA: Rigaku thermos plus EV02 instrument at GVISH College, Amravati.

SEM: JSM-6380 Instrument VNIT College, Nagpur.

3. RESULTS AND DISCUSSION:

3.1 FTIR Analysis

Fourier Transform Infra-Red Spectroscopy of undoped Polythiophene and doped V_2O_5 thin films by using chemical bath deposition method. FTIR spectroscopy is an important investigation of polymer structure that provides information about the complexation and interactions between the various constituents in the polymeric films. Each type of bond has a different natural frequency of vibration, so the identification of an absorption peak in the vibration portion of the infrared region will give a specific type of bonding. The FTIR spectra for pure polythiophene and different %w/v of V_2O_5 doped thin films are shown in Figure 1, 1(a) and 1(b). The absorption band in region 2920.03 cm^{-1} is due to aromatic C=C-H stretching frequency of pure polythiophene which is shifted to the ranges 2920.37 cm^{-1} and 2919.58 cm^{-1} in PTh/ V_2O_5 composite. The absorption band in the region 1625.13 cm^{-1} is due to the aromatic C=C stretching frequency of Pure PTh which is shifted to the ranges 1625.17 cm^{-1} , 1625.96

cm⁻¹.the peak at 1318.17 cm⁻¹for Pth is assign for C-C stretching frequency are shifted to 1317.90 cm⁻¹, 1320.84 cm⁻¹.The absorption band in the region 780.91 cm⁻¹is due to the C-S stretching frequency of Pure PTh which is shifted to the ranges 780.42 cm⁻¹,

785.58 cm⁻¹.In PTh/ V₂O₅composite.The absorption peak at lower frequency 578.19 and 579.58 cm⁻¹ observed for V-O-V stretching which is absent in undopedpolythiophene.

Table 1: Enclosed the observed vibrational frequencies

Material	Frequency	In cm ⁻¹			Stretching
	Ar-C=C-H	Ar C=C	C-C	C-S	V-O-V
UndopedPTh	2920.03	1625.13	1318.17	780.91	
PTh+0.5w% V ₂ O ₅	2920.37	1625.17	1317.90	780.42	578.19
PTh+1w% V ₂ O ₅	2919.58	1625.96	1320.84	785.58	579.58

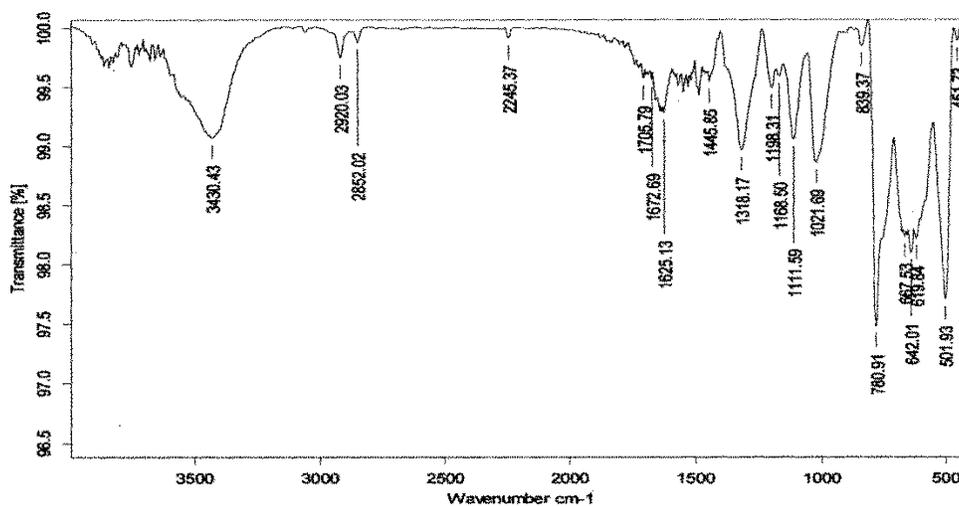


Fig.1: FTIR spectra of undopedpolythiophenethin films.

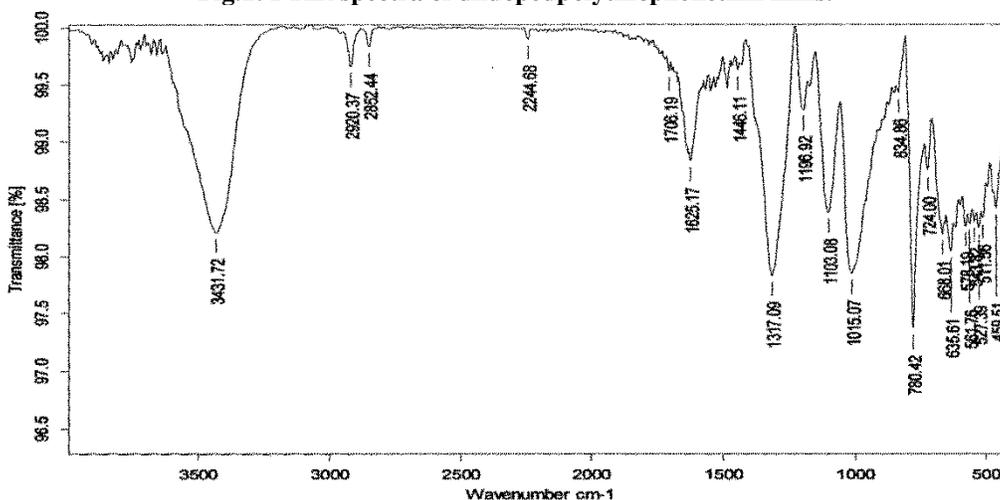


Fig.1(a): FTIR spectra of PTh /0.5w% V₂O₅composite thin. Films

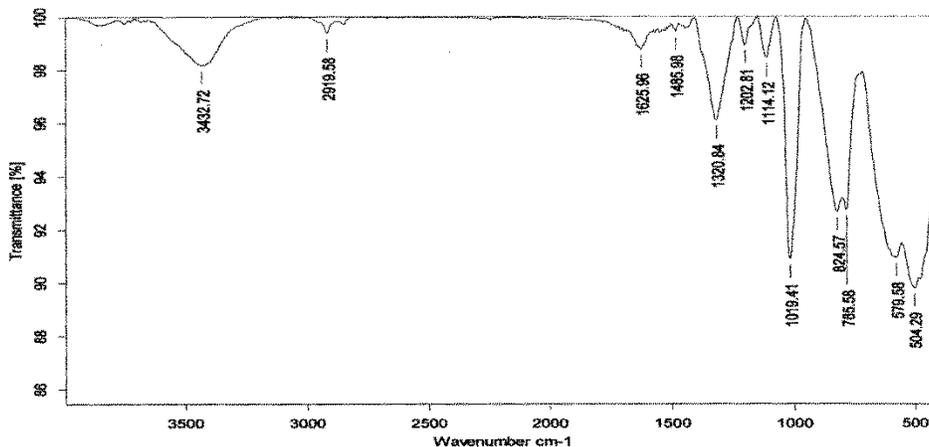


Fig.1(b): FTIR spectra of PTh /1 w% V₂O₅ composite thin films.

3.2 XRD Analysis

The XRD profiles of pure polythiophene and doped V₂O₅ with deferent %w/v are shown in Figure2,2(a) and 2(b).In case of polythiophene no sharp peak observed indicating the polymer under investigation is nanocrystalline in nature In XRD analysis of 0.5

%wV₂O₅ doped polythiophene shows one peak at 2 theta =26.27 and 1% wV₂O₅doped polythiophene shows peaks at 2 theta 20.523 22.01 26.404,31.272,32.63,33.56 ,34.608,45.64..which does not found in pure polythiophenesuggesting the increase in degree of crystallinity of the complex.

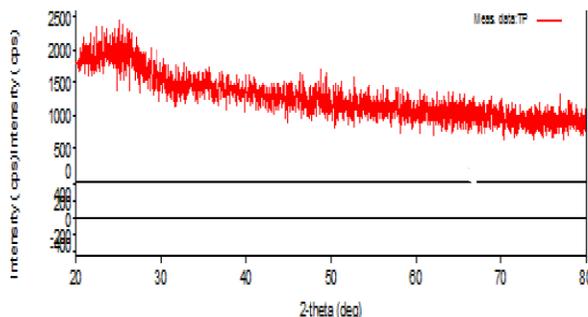


Fig.2: XRD patterns of pure Pth and0.5% WV₂0doped composite

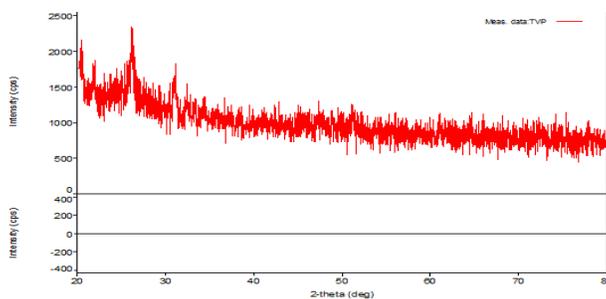


Fig.2(a):XRD patterns of polythiophene 0.5% w/v V₂0s doped composite.

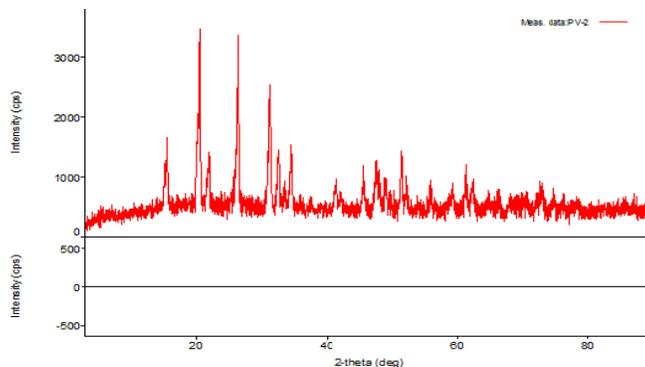


Fig.2(b): XRD patterns of Polythiophene 1% W/v V₂O₅ doped composite.

SEM studies

The SEM images of the pure polythiophene and pth/V₂O₅ composites thin film are shown in figure the film shows different surface morphology due to different weight % of v₂o₅doping.Figure 3,3(a),3(b)

shows is observed in pure polythiophene thin film.the grains are highly agglomerated highly irregular shape some grains are irregular in structure some of them are elongated and some are spherical in shape .

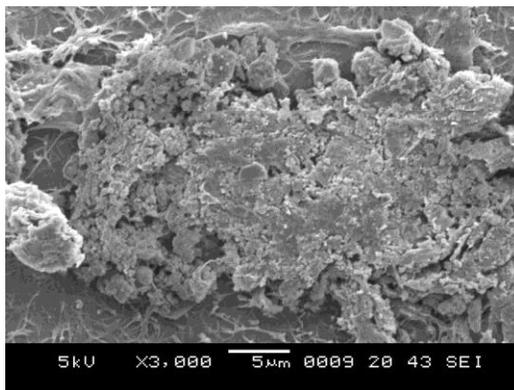


Fig.3: SEM image of undoped polythiophene thin film

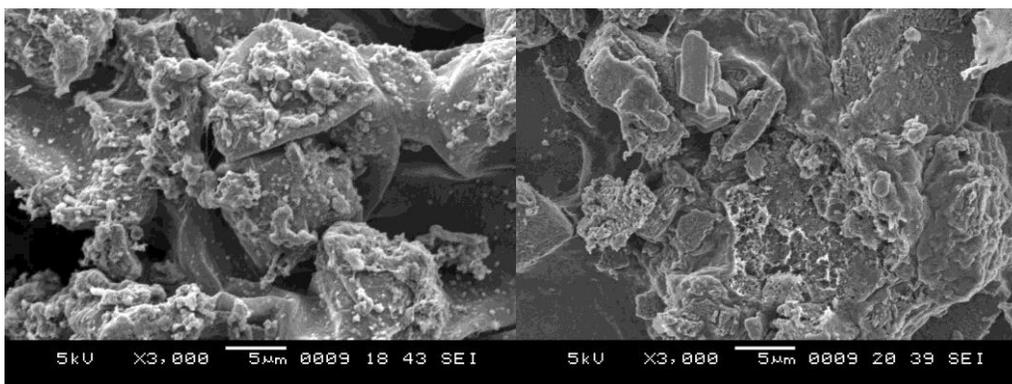


Fig.3(a): SEM image of 0.5%w/vV₂O₅ doped polythiophene thin film

Fig.3(b): SEM image of 1%w/v V₂O₅ doped polythiophene thin film.

TG/DTA analysis-

Thermogravimetric and differential thermal analysis of Polythiophene and different %w/v of V₂O₅ composite was carried out in air atmosphere .TGA was performed on Rigaku TG8121 thermal analyser with pt pan in the temperature range 0-550°C.the thermogram of polythiophene and composite as shown in figure. Three major weight losses are observed one around 50-80⁰ weight loss about 1-3%

due to elimination of moisture, evaporation of solvent as well as unreacted monomer .second weight loss around 280-300⁰ is due to loss of dopant component of polythiophene. Third major drop in weight observed at at 350-400⁰ and beyond the range is due to degradation of polythiophene itself.

In case of DTA shows two exothermic maxima at near about 400⁰ and 450⁰

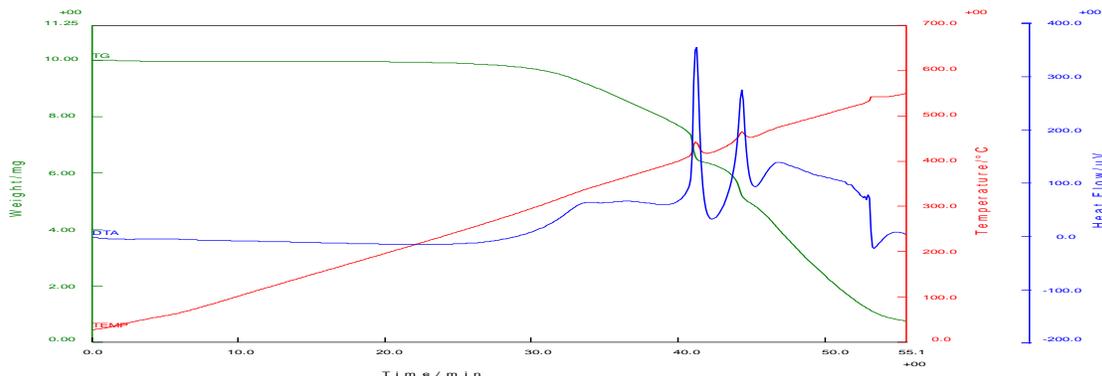


Fig. 4: TG-DTA of undoped polythiophene.

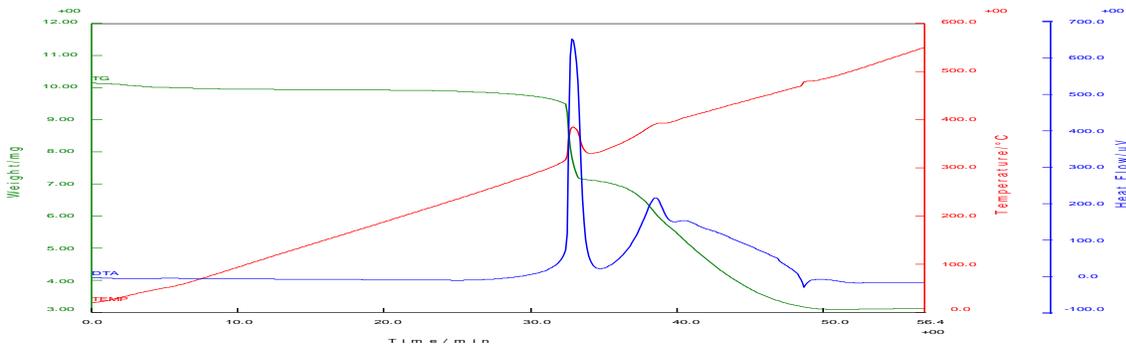


Fig.4(a): TG-DTA of polythiophene 0.5% w/v V₂O₅ doped .

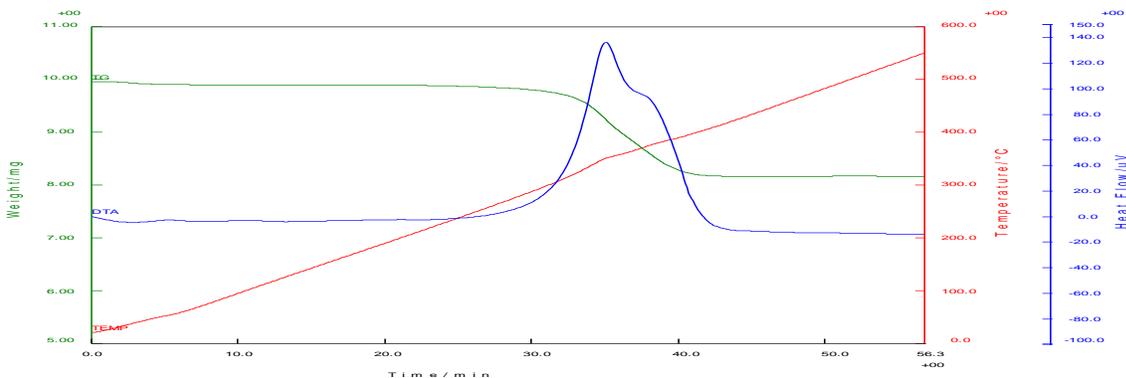


Fig.4(b): TG-DTA of polythiophene 1% w/v V₂O₅ doped .

5. CONCLUSIONS:

We have demonstrated the successful synthesis of nanostructure polythiophene thin film by chemical bath deposition technique. Polythiophene can be doped by V₂O₅ in chloroform with the formation of complex. The result of FTIR proved the formation of polythiophene. Morphology of thin film were analysed by SEM shows change in morphology after doping. XRD shows modification from amorphous to the well developed crystalline structure. Thermal properties of nanocomposites were investigated by TG-DTA analysis shows composites of polythiophene with V₂O₅ are found to be more thermally stable than undoped polythiophene.

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