



# Structural, photoconductivity, and dielectric studies of polythiophene-tin oxide nanocomposites



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## ABSTRACT

Polythiophene-tin oxide (PT-SnO<sub>2</sub>) nanocomposites were prepared by in situ chemical oxidative polymerization, in the presence of various concentrations of SnO<sub>2</sub> nanoparticles. Samples were characterized by X-ray diffraction, Fourier-transform infrared spectroscopy, thermogravimetric analysis, X-ray photoelectron spectroscopy and Zeta potential measurements. Morphologies and elemental compositions were investigated by transmission electron microscopy, field-emission scanning electron microscopy and energy-dispersive X-ray spectroscopy. The photoconductivity of the nanocomposites was studied by field-dependent dark and photo conductivity measurements. Their dielectric properties were investigated using dielectric spectroscopy, in the frequency range of 1 kHz–1 MHz. The results indicated that the SnO<sub>2</sub> nanoparticles in the PT-SnO<sub>2</sub> nanocomposite were responsible for its enhanced dielectric performance.

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## 1. Introduction

Conducting polymers and their nanocomposites have been an important research area since their discovery in the mid-1970s. Conducting polymer-metal oxide nanocomposites have been extensively investigated, because of their interesting physiochemical properties and potential application in nanodevices [1–5]. Polymer nanocomposites have been synthesized by various physical and chemical methods, including in situ chemical oxidation, electrochemical polymerization, and melt processing [6–8]. Those with high dielectric constants are used in charge-storage devices, telecommunications, electromagnetic interference shielding, integral capacitor technology, and electromechanical applications [9–12]. Polythiophene (PT) is a promising conducting polymer, because of its high and controllable electrical conductivity [13–15]. Its low solubility and poor processability limit its application, so PT has been incorporated with metal oxide nanoparticles to overcome these problems. Incorporating metal oxide nanoparticles can enhance the electrical properties of conjugated polymers. PT composites containing Fe<sub>3</sub>O<sub>4</sub>, Al<sub>2</sub>O<sub>3</sub>, and TiO<sub>2</sub> have all been thoroughly studied [16–18]. Nanoscale

metal oxide particles are of particular interest, because of their size-dependent physical and chemical properties.

SnO<sub>2</sub> is a wide band gap (3–6 eV) n-type semiconductor used in many applications, including electrode materials for Libatteries, gas sensors and antistatic coatings [19–21]. Optical charge generation and transport in photoconducting PT derivatives is receiving much current interest [22]. Photoconductivity is the enhancement of a material's electrical conductivity by absorbing photons of a suitable energy. Polyfluorene, its copolymers including poly(*para*-phenylene vinylene), and substituted PT derivatives are all widely used in this field [23]. Photoconducting substituted PTs have applications in electronic devices including electro-optic modulators, optical signal processors, photoreceptors, solar cells, and optical frequency doublers [24]. Photoconducting devices based on polymer composites with internal donor/acceptor heterojunctions have also been investigated [25]. However, there are no reported studies investigating the photoconductivity of PT-SnO<sub>2</sub> nanocomposites.

PT and SnO<sub>2</sub> are both widely applied in technology. Thus, PT-SnO<sub>2</sub> nanocomposites were thought likely to possess interesting properties, which could potentially be useful when designing and developing polymer electronic devices. While there are some reported syntheses and morphological studies of PT-metal oxide nanocomposites [26–28], to the best of our knowledge none have probed the electric behavior of the PT-SnO<sub>2</sub> nanocomposites. In

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addition to requiring good photoconductivity and dielectric performance, other properties of the material are also important when developing novel polymer electronic materials. These include structural order (i.e., morphology yielding optimum efficient charge transport), thermal stability, and surface charge.

In the current study, we report the synthesis, structural characterization, zeta potential and photoconductivity measurements of PT-SnO<sub>2</sub> nanocomposites. The dielectric behavior of PT-SnO<sub>2</sub> nanocomposites containing 10, 20, 30, and 40 wt.% SnO<sub>2</sub> was investigated over the frequency range of 1kHz–1 MHz.

## 2. Experimental

### 2.1. Materials

All chemicals used were of analytic reagent (AR) grade. PT was prepared from freshly double distilled thiophene (Aldrich). Anhydrous ferric chloride (FeCl<sub>3</sub>) was purchased from Fluka. Stannouschloride dihydrate (SnCl<sub>2</sub>·2H<sub>2</sub>O), chloroform and ammonia solution were purchased from Merck. Deionized water was used in all reactions.

### 2.2. Synthesis of SnO<sub>2</sub> nanoparticles

SnO<sub>2</sub> nanoparticles were synthesized by coprecipitation, using SnCl<sub>2</sub> as a Sn source. 4 g (0.2 mol) of SnCl<sub>2</sub>·2H<sub>2</sub>O was dissolved in 200 ml of water. 6 ml of ammonia solution was added, and the mixture was stirred vigorously at room temperature for 1.5 h to form a precipitate. The white gel precipitate was allowed to settle for 14 h, collected by filtration and thoroughly rinsed with distilled water. The precipitate was heated at 110 °C until dry, and then calcined at 500 °C for 5 h to yield SnO<sub>2</sub> nanoparticles.

### 2.3. Preparation of PT-SnO<sub>2</sub> nanocomposite

The PT-SnO<sub>2</sub> nanocomposite was prepared by in situ chemical oxidative polymerization of thiophene monomer, in the presence of SnO<sub>2</sub> nanoparticles. In a typical synthesis, 0.2 g of SnO<sub>2</sub> nanoparticles and 0.05 mol of thiophene were dispersed in 100 ml of chloroform and stirred for several minutes. 0.25 mol of anhydrous FeCl<sub>3</sub> in chloroform was added under vigorous

stirring. The reaction mixture was stirred with a magnetic stirrer bar at room temperature for 4 h, during which its color changed from grey to black. The precipitate was collected by filtration, and washed thoroughly with methanol and doubly distilled water to remove unreacted oxidants and monomer. The resulting powder was dried under vacuum at 70 °C for 8 h. PT-SnO<sub>2</sub> nanocomposites were synthesized containing 10, 20, 30, and 40 wt.% of SnO<sub>2</sub> nanoparticles. PT was prepared similarly, but in the absence of SnO<sub>2</sub> nanoparticles.

### 2.4. Characterization

X-ray diffraction (XRD) patterns were collected at 2θ of 10–80° using a diffractometer (XRD-Smart Lab, Rigaku, Japan). XRD patterns were analyzed by indexing observed peaks with standard JCPDS values. Fourier transform infrared (FTIR) spectra were obtained at 4000–400 cm<sup>-1</sup> using a FTIR spectrometer (Spectrum RX1, Perkin Elmer, MA, USA). Sample morphologies were observed by transmission electron microscopy (TEM; JEOL 3010, Japan) and field-emission scanning electron microscopy (FESEM; SUPRA 55, Carl Zeiss, Germany). Energy-dispersive X-ray analysis (EDAX) was used to determine the elemental compositions of PT and the PT-SnO<sub>2</sub> nanocomposites. Thermal stabilities of the samples were investigated by thermo gravimetric analysis (TGA; TG/DTA 6200), at temperatures from 30 to 900 °C at a heating rate of 10 °C/min in a nitrogen atmosphere.

X-ray photoelectron spectroscopy (XPS) (AXIS ULTRA from AXIS 165) was used to obtain information about the chemical states of samples. Zeta potential measurements (Horiba) were used to determine the surface charge and isoelectric points of samples in water. Photoconductivity measurements were conducted using a Keithley Picoammeter 6485. PT and PT-SnO<sub>2</sub> powders were finely ground with an agate mortar. Pellets of ~10 mm in diameter and 2 mm in thickness were prepared by hydraulically pressing powders at 5.5 t. Silver paste was then coated on each face to create contact with two electrodes. The pellet was then placed into sample holder. Dielectric measurements were carried out using a LCR HiTester apparatus (HIOKI 3532-50 LCR HiTester, Japan), over the frequency range 1kHz–1 MHz.

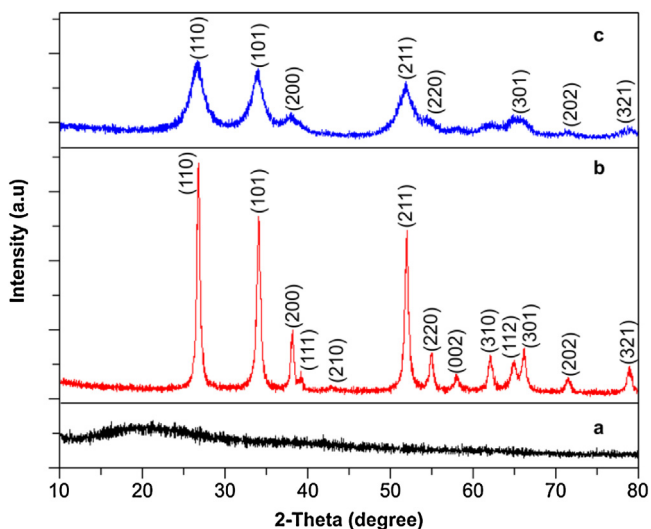


Fig. 1. XRD patterns of (a) PT, (b) SnO<sub>2</sub> and (C) the PT-SnO<sub>2</sub> nanocomposite containing 10 wt.% SnO<sub>2</sub>.

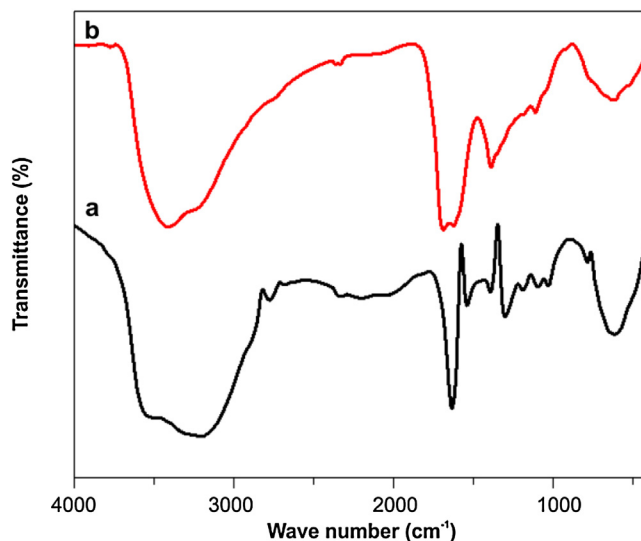


Fig. 2. FTIR spectra of (a) PT and (b) the PT-SnO<sub>2</sub> nanocomposite containing 10 wt.% SnO<sub>2</sub>.

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