

ELECTRICAL PROPERTIES ON COPOLYESTER INCORPORATED IN A POLYMER NANOCOMPOSITE MATRIX - A STUDY

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Abstract

This study aims at the application of the prepared Polymer Nanocomposite in the field of solid-state optoelectronic devices. Polymer Nanocomposite was prepared by incorporating iron nanoparticles, synthesized random Copolyester (BCTP), and Polypyrrole by Insitu chemical polymerization method. The application of the synthesized random Copolyester with wide biological activity due to bis chalcone moiety can also be extended to the optoelectronics field. The Copolyester (BCTP) was subjected to spectral analysis such as Fourier Transform Infrared Spectroscopy (FTIR) to confirm the chemical structural orientation. The Iron nanoparticles, Polypyrrole, and polymer nanocomposite were characterized by Fourier Transform Infrared Spectroscopy (FTIR) and powder X-ray Diffraction (PXRD). The FTIR spectroscopy confirms the formation of polymer nanocomposite by the prominent shift in stretching frequency from the starting materials taken. Powder X-ray Diffraction was used to determine the Iron Nanoparticles' average crystallite size. The structural morphology of iron nanoparticles, Polypyrrole, and polymer nanocomposite were identified by Scanning electron Microscopy. The SEM images gave the view of Iron nanoparticles, Polypyrrole was seated in the Polymer matrix. The specific capacitance for the newly synthesized Polymer Nanocomposite was found to be 58.88 F/g by Cyclic Voltammetric analysis. The electrochemical behavior of the random copolyester (BCPT), Iron nanoparticles, Polypyrrole, and polymer nanocomposite was carried out by Impedance analysis. This confirms the Polymer Nanocomposites were conducting nature with good ionic conductance of $6.89 \times 10^{-5} \text{ S cm}^{-1}$.

Keywords: Conducting; Impedance analysis; Nanocomposites; Nanoparticles

Introduction

In recent decades, Nanotechnology is an emerging field due to its promising developments in the universe. Supercapacitors, known as ultracapacitors are power delivery device that easily captures and releases with increased energy and reduced emission. Due to this, ultracapacitors

are used as excellent energy-storing devices (Joshi and Adak, 2019). Bischalcones are the class of chromophores with the molecular configuration (D- π -A- π -D) and having a large π -conjugation electronic system. (Nohut *et al.*, 2018; Shen *et al.*, 2022)

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Shiva *et al.* (2019) reported that the carbon Quantum - Dot -polypyrrole nanocomposites showed a 33 % increase in the specific capacitance as compared to the pure Polypyrrole. Sundara *et al.* (2020) reported that Fe_2O_3 -decorated N-doped CNT composites derived from Polypyrrole have 79% of capacitance retention, and good cycling stability of 84% capacitance loss after 10,000 cycles at 7mAcm^{-2} . Ananda *et al.* (2020) studied the electrical properties of Polypyrrole nanocomposites by using an Impedance analyzer. The AC-Conductivity of the Polypyrrole has a higher weight % of filler content in the nanocomposites and behaves like dielectrics and conductors, respectively (Ananda *et al.*, 2020).

Tarekegn *et al.* (2021) studied that zerovalent iron nanoparticles enhance sub-surface stability. They proved the removal activity of Pb, Cd, and Cu. Anita *et al.* (2021) reported that the Polypyrrole-coated iron oxide nanoparticles had enhanced conductivity and reduced resistance of the composite upon Fe_3O_4 , thereby securing the conductive properties. Zhang *et al.* (2021) investigated the impedance matching for a dual permeable matrix for the carbonyl iron @ SiO_2 @heterogeneous state Polypyrrole @ Paraffincomposites for electromagnetic wave absorption application. They reported the absorption performance at high frequency. (Zhang *et al.*, 2021).

Xu *et al.* (2021) examined the iron - doped alpha - manganese dioxide coated with polypyrrole nanocomposites has a specific capacity of 270mAhg^{-1} at 100mA g^{-1} and high diffusion coefficient of 10^{-10} to $10^{-14}\text{cm}^2\text{s}^{-1}$ are also observed. Yan *et al.* (2021) studied the polypyrrole-based nanocomposite for electromagnetic wave absorption. Vorobiov *et al.* (2021) reported that the $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles in polypyrrole-based hydrogel have a strong pseudocapacitance from 239Fg^{-1} to 486Fg^{-1} . The $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles in polypyrrole hydrogel have a capacitance retention of about 75% after 3000 charge-discharge cycles, respectively (Rashtbari *et al.*, 2022).

Lin *et al.* (2022) studied the electrical conductivity of PPY/ SMPP composites and showed the MA performance value of -56.3 dB at 8.04 GHz was achieved for PPy / SMPP composites. Pasha *et al.* (2022) reported that the electrically conductive PDMS was decorated with Polypyrrole / Manganese-iron oxide nanocomposites for high-performance EMI shielding applications. They also studied the flexibility, stretchability, high conductivity, and superior strain sensing performance of PPy/ Manganese-iron oxide nanocomposites (Pasha *et al.*, 2022).

Many researchers have reported the synthesis of nanocomposites, but the insitu chemical Polymerization method is relatively easier, and easy to synthesize, producing a high yield.

In this study, we report an in situ chemical polymerization method to synthesize a novel Polymer Nanocomposite and investigate the specific conductance, and ionic capacitance for polyester, Iron Nanoparticles, Polypyrrole, and Polymer Nanocomposite by Cyclic Voltammetry and Impedance Analysis.

Highlights

- The Polymer Nanocomposites are synthesized by incorporating Copolyesters [BCTP] and iron Nanoparticles in the Polypyrrole Composite Matrix by in situ Chemical Polymerization method.
- The Polymer Nanocomposites were characterized by FTIR, XRD, SEM, Cyclic Voltammetry, and Impedance Analysis.
- In FTIR, XRD analytical results predict that the Polymer Nanocomposite spectral results show the shifting of peaks due to the interaction between the H- bonding and π - π stacking.
- The specific capacitance for the newly synthesized Polymer Nanocomposite was found to be 58.88F/g by Cyclic Voltammetric analysis.
- The Polymer Nanocomposites were conducting nature with good ionic conductance of $6.89 \times 10^{-5}\text{S cm}^{-1}$.

Materials and Methods

The chemicals which are used in this study are of analytical grade (AR). Sodium Dodecylbenzene Sulfonate, pyrrole monomer, sodium borohydride, and ferrous sulfate were purchased from Merck.

A. Synthesis of Polyester

[BCTP-BC-Bischalcone; T-Terephthaloyl Chloride; P-Polymer] [Bichalcone-Monomer diol, Terephthaloyl Chloride-acid chloride]

The monomer diol was prepared by the base-catalyzed Claisen-Schmidt Condensation method. The random Copolyester (BCTP) was also synthesized by the polycondensation method. (Bahraeian, *et al.*, 2013; Aswini, *et al.*, 2022) [% yield - 75%]

B. Synthesis of Iron Nanoparticles

Iron Nanoparticles were prepared by the chemical reduction method (Zhang and Wang,

1997). 0.1 M solution of ferrous sulfate was prepared by using distilled water, and it is stirred for 10 mins. Then add a 0.75 M solution of sodium borohydride in a dropwise addition with continuous stirring for 30 mins. The black color particles were filtered using Whatman filter paper [% yield - 70%].

C. Synthesis of Polypyrrole

4 g of Sodium Dodecylbenzene Sulfonate was dissolved in 100 mL of distilled water. 28 g of ferric chloride was dissolved in 100 mL of distilled water. Both solutions were mixed and stirred for 15 mins. 0.15 mole of pyrrole monomer was added to the above solution and stirred for 20 mins. It is filtered and washed with distilled water several times. It is then dried in an oven at 60°C for 8 hours [% yield - 79%].

D. Preparation of Polymer Nanocomposite [0.5% formulation]

It is a novel method for synthesizing Polymer Nanocomposite by in situ Polymerization method.

28 g. of ferric chloride was dissolved in 100 mL. of distilled water and 4 g. of Sodium Dodecylbenzene Sulfonate was dissolved in 100 mL. of distilled water. 20 mL. of ferric chloride and Sodium Dodecylbenzene Sulfonate were added and stirred for 20 mins. Then, add 0.0040 g. of polyester (BCTP) and 0.0045 mg of Iron nanoparticles with continuous stirring for 5 mins. 0.15 mole of pyrrole monomer was added to the above solution and stirred for 20 mins. It is then filtered and dried at room temperature for 24 hours [% yield - 82%]

Characterization

Fourier Transform Infrared Spectroscopy was performed by Shimadzu Instrument with the range of 4000 to 400 cm^{-1} . The FTIR spectrum shows the presence of a functional group. PXRD analysis was performed by Powder X-ray Diffractometer-PANalytical, Xpert3 instrument. It is used to predict the average crystallite size for the sample. Scanning Electron Microscopy was performed by Desktop Mini-SEM (SNE-3200, SEC SEM). SEM analysis is used to predict the magnification images of a sample's surface topography. The electrochemical behavior of polymer nanocomposite was studied by Cyclic Voltammetry analysis. It is performed by Princeton Applied Research with Versa STAT MC.

Results and Discussion

A. Fourier Transform Infrared Spectroscopy

The FTIR spectra of Copolyester (BCTP) are shown in Figure 1. The characteristic absorption peaks at 3444 cm^{-1} , 2549 cm^{-1} , 1767 cm^{-1} , 1702 cm^{-1} , 788 cm^{-1} , 735 cm^{-1} , and 508 cm^{-1} correspond to the O-H stretching frequency, C-H stretching vibration, C=O stretching frequency, C=C bending vibration, and =C-H bending vibration, respectively. The presence of ester carbonyl stretching frequency (1767 cm^{-1}) confirms the formation of copolyesters (Chudgar *et al.*, 1995; Aswini *et al.*, 2022).

The Iron Nanoparticles were confirmed by the FTIR spectrum and are shown in Figure 2. The absorption peaks at 3398 cm^{-1} correspond to the O-H stretching vibration frequency, 2932 cm^{-1} correspond to the C-O bending vibrations, and 629 cm^{-1} corresponds to the presence of iron-oxygen (Fe-O), which confirms the formation of iron nanoparticles (Bhatia *et al.*, 2018; Seliem *et al.*, 2020; Tarekegn *et al.*, 2021; Rashtbari *et al.*, 2022).

In Figure 3. the absorption peak at 3441 cm^{-1} corresponds to the N-H Stretching vibration of the pyrrole ring and the C-N Stretching vibration at 1459 cm^{-1} . The C-H and N-H in-plane deformation

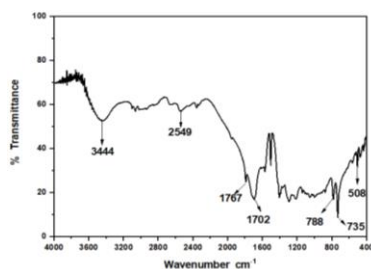


Figure 1. FTIR Spectrum of Copolyester (BCTP)

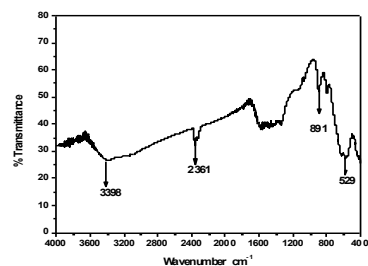


Figure 2. FTIR Spectrum of Iron Nanoparticles

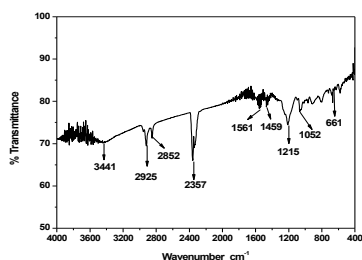


Figure 3. FTIR Spectrum of Polypyrrole

was observed at 2826 cm^{-1} , 1052 cm^{-1} . The above peaks confirm the formation of Polypyrrole (Chitte *et al.*, 2011; Moorthy *et al.*, 2018; Shiva *et al.*, 2019).

In Figure 4. FTIR spectrum for Polymer nanocomposite. The N-H stretching vibration frequency was shifted from 3441 cm^{-1} to 3409 cm^{-1} . The ester carbonyl stretching frequency was shifted from 1767 cm^{-1} to 1725 cm^{-1} . The iron nanoparticles were also shifted from 529 cm^{-1} to 523 cm^{-1} . This may be due to the H-bonding interactions and π - π stacking can lead to the formation of a Copolyester, Iron Nanoparticles incorporated in the Polypyrrole Nanocomposite matrix.

B. Powder X-ray Diffraction Analysis

PXRD patterns for the Polypyrrole, Iron Nanoparticles, and Polymer Nanocomposite have recorded the $\text{CuK}\alpha$ radiation with the diffraction angle between 10° and 80° . The average crystallite size D was calculated from the Sherrer equation. The average crystallite size D was calculated by $D = K\lambda / \beta \cos \theta$.

The synthesized iron nanoparticles (Figure 5) are in the cubic system with the lattice constant of

$a=b=c= 2.82\text{ \AA}$. The hkl values for iron nanoparticles are (110) and (200) planes (JCPDS File 87-0721), respectively. The diffraction peak is around 45° , which corresponds to the iron nanoparticles (Ansari, *et al.*, 2012; Sravanthi, *et al.*, 2018; Saffari, 2018). The average crystallite size for iron nanoparticles was found to be 25 nm.

Figure 6. shows the XRD pattern for Polypyrrole. The broad peak in the region of 21° indicates the amorphous nature of Polypyrrole (Pal *et al.*, 2016).

The XRD pattern for Polymer Nanocomposite is shown in Figure 7. It shows the halo pattern in the diffraction angle is shifted from 21° to 28° , confirming the formation of nanocomposite in an amorphous nature.

C. Scanning Electron Microscopy (SEM)

The Surface topography was analyzed by Scanning electron Microscopy. Figure 8 (a) represents the SEM images for Iron Nanoparticles, which seem to be agglomerate. The SEM images of PPy are shown in Figure 8 (b), and it is observed that the Iron Nanoparticles have a minute gap in the surface level.

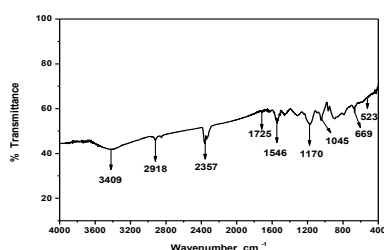


Figure 4. FTIR Spectrum of Polymer Nanocomposite

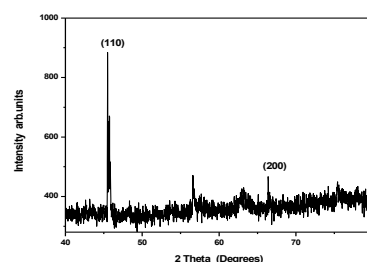


Figure 5. XRD pattern for Iron Nanoparticles

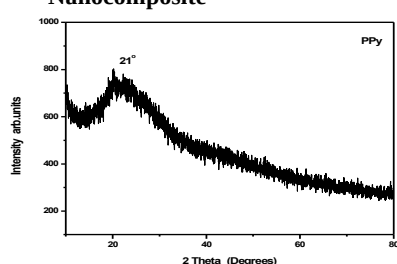


Figure 6. XRD pattern for Polypyrrole

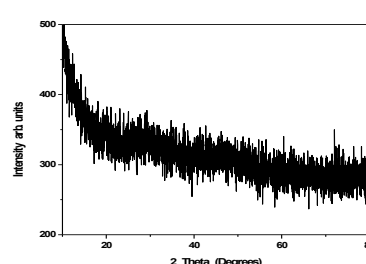
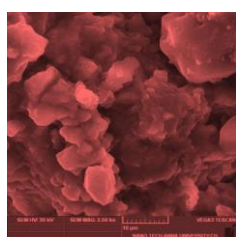
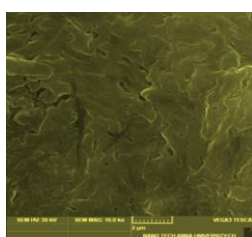


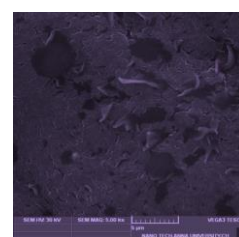
Figure 7. XRD pattern for Polymer Nanocomposite



(a)



(b)



(c)

Figure 8. (a), (b), (c) SEM images of Iron Nanoparticles, PPy, and Polymer Nanocomposite, respectively

Figure 8 (c) shows the SEM images of the Polymer Nanocomposite. The Iron Nanoparticles and copolyester are distributed to fill the gap at the PPy matrix resulting in the amorphous nature of Polymer nanocomposite. (Shahenoor, *et al.*, 2017; Seliem, 2020)

D. Cyclic Voltammetry

Cyclic Voltammetry is an important technique to find out the electrochemical behavior. The Cyclic Voltammogram was recorded at a single scan rate. The Potential ranges from -10 to +10 V, with a scan rate of 10 mVs⁻¹.

The Specific Capacitance is calculated by the formula:

$$C_p = A / 2mk (V_2-V_1)$$

Where; C_p Specific Capacitance in F/g
 A Area inside the CV curve having units in AV
 m a mass of active material
 k Scan rate of CV in mV/s
 (V_2-V_1) A potential window of CV (total voltage range)

Figure 9, represents the redox potential curve for PPy, copolyester, Iron Nanoparticles, and Polymer Nanocomposite. Table 1 shows the Specific Capacitance value for the Polymer Nanocomposite.

On comparing the Specific Capacitance of PPy, Copolyester, Iron Nanoparticles, and Polymer Nanocomposite. The Polymer Nanocomposite shows a specific capacitance of 58.88 F/g. This is due to the incorporation of copolyester and iron Nanoparticles in the PPy matrix. This newly synthesized Polymer Nanocomposite may be a good candidate for the Supercapacitor application.

E. Electrochemical Impedance Spectroscopy

The impedance analysis is an important technique to find out the ionic conductivity of Polyester, Iron nanoparticles, Polypyrrole, and Polymer Nanocomposite. The Conductivity (σ) of a sample was calculated by the formula:

$$\sigma = t / A \times R$$

where; t Thickness of the sample
 A Contact Area of the sample
 R Resistance of the sample

Table 1. The Specific Capacitance value for the Polymer Nanocomposite

S. No	Sample Code	Specific Capacitance (F/g)
1	PPy	48.02
2	BCTP	25.64
3	Iron Nanoparticles	24
4	Polymer Nanocomposite	58.88

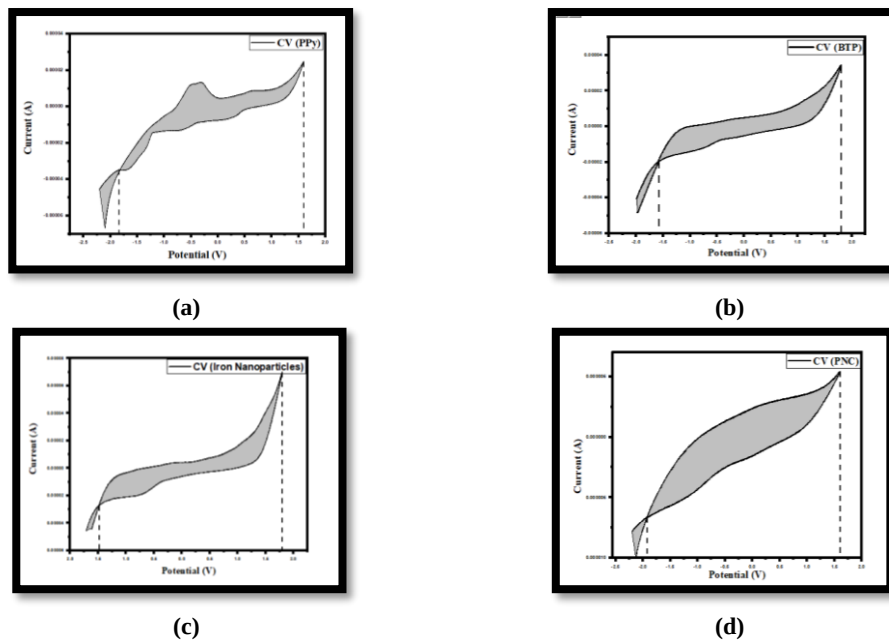


Figure 9. (a). Cyclic Voltammogram for PPy, (b). Cyclic Voltammogram for Copolyester (BTP), (c). Cyclic Voltammogram for Iron Nanoparticles, and (d). Cyclic Voltammogram for Polymer Nanocomposite

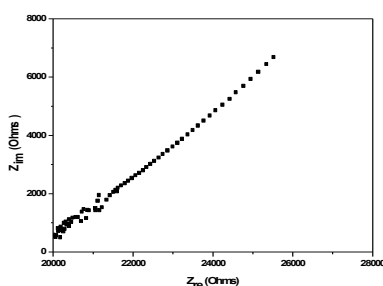


Figure 10. Nyquist Plot for Polymer Nanocomposite

Table 2. Represents the ionic Conductivity of a sample

S.No	Sample Code	Ionic Conductivity
1	PEBT	1.27×10^{-4}
2	Fe NPs	5.50×10^{-4}
3	PPy	1.21×10^{-5}
4	PolymerNanocomposite	6.89×10^{-5}

The Table 2 shows the ionic Conductivity of a sample

The highest ionic conductivity value for polymer nanocomposite is due to the presence of polyester and iron nanoparticles in the composite matrix. It enhances the conductivity behavior of polymer nanocomposite. Shown in Figure 10.

Conclusions

A novel Polymer Nanocomposite was synthesized by incorporating Polyester (BCTP) and iron nanoparticles in the Polypyrrole matrix. The Bischalcone moiety in the copolyester backbone was confirmed by FTIR. The X-ray diffraction study predicts the amorphous nature of Polymer Nanocomposite. The morphology of the Iron Nanoparticles, Polypyrrole, and Polymer Nanocomposite was studied by Scanning Electron Microscopy analysis. The Cyclic Voltammetric analysis was carried out to find out the specific conductance of Copolyester (BCTP), Iron Nanoparticles, Polypyrrole, and Polymer Nanocomposite. The specific capacitance for the newly synthesized Polymer Nanocomposite was found to be 58.88 F/g by Cyclic Voltammetric analysis. The electrochemical impedance analysis was carried out to determine the Polymer Nanocomposite's conducting nature. Hence, the newly synthesized Polymer Nanocomposite was found to have ionic conductivity of $6.89 \times 10^{-5} \text{ S cm}^{-1}$. In the future study, the application of the Polymer Nanocomposite will be studied as a solid-state electronic device.

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