Iranian Journal of Materials Science and Engineering, Vol. 22, Number 4, December 2025
RESEARCH PAPER

Synthesis, Characterization and Electrical Properties of Poly 2-Amino-benzothiazole Doped by MWCNTS

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Received: February 2025 Revised: August 2025 Accepted: September 2025
DOI: 10.22068/ijmse.3928

Abstract: *Poly(2-aminobenzothiazole) (PAT) is a relatively new heterocyclic conducting polymer having a sulfur and nitrogen-rich chemical structure. During the past decade or so, there have been notable advances on the development of PAT. In particular, PAT and PAT-based composites have shown great potential for applications in photovoltaic cells, solar cells, and anti-corrosion organic coatings. In this study, 2-aminothiazole was successfully prepared as a pure polymer and as composite materials with multi-wall carbon nanotubes (MWCNTs). FTIR, X-ray diffraction and SEM images were investigated, showing that the composite of poly 2-aminobenzothiazole: MWCNTs was successfully synthesized. The electrical features of the pure polymer and the composite thin films were examined. The findings show that the conductivity of the pure polymer and composite thin films are about 1.67×10^{-6} (S/cm) and 4.1×10^{-2} (S/cm), respectively, exhibiting a significant enhancement by a factor of 2.5×10^{4} times as a result of doping the pure polymer with 1% wt MWCNTs.*

Keywords: *Poly 2-aminobenzothiazole, Poly 2-aminobenzothiazole: MWCNTs composite, Nanocomposite, Surface morphology, Electrical conductivity.*

1. INTRODUCTION

Organic conducting materials are referred to as ’synthetic metals’ [1] and are considered intrinsic and naturally conducting [2]. These materials are composed of π-conjugated molecules, which exhibit enhanced electro-optical properties when doped or substituted with other chemical groups [3]. These conductive polymers have huge applications in manty areas such as rechargeable batteries, [4] modified electrodes, [5] photovoltaic and solar cells, [6] sensors, [7, 8] electrochromic display devices, light-emitting diodes [9] bioelectronics [10] and bioimaging [11, 12]. The major of conjugated polymers has been improved, enabling their use in new-generation polymeric devices. Among electroactive polymers poly-2-amino benzothiazole is considered one of promising materials which have many applicable features like antitumor, anti-corrosive , antimicrobial , and electroactivity [13, 14]. Several studies have reported that chemically and electrochemically polymerised derivatives of thiazole exhibit conducting and electrochromic properties [15, 16]. Several studies reported that the conductivities of aminothiazole polymers can be improved through doping reactions [17]. Copolymers of thiazole with other conducting monomers have also been prepared and examined in the photovoltaic applications area[18].

Carbon nanotubes and other nanomaterials have attracted widespread interest in recent years due to their remarkable mechanical and electrical properties. Growing attention has been focused on the CNT surface modification, namely the interface between the CNT and the surrounding polymer matrix. Several methods of functionalization are applied, such as chemical, electrochemical, and plasma. [19] This functionalization is used to functionalize their nano surfaces and side chains. The potential and promising applications of nanotube compounds can be improved by making structural modifications. This modification can enhance the reactivity and improve the functional performance of the nano-network. Typically, oxidation of nanotubes, mainly inserting carboxyl and hydroxyl, has been found to promote the carbonic surface. The existence of oxygen-containing groups leads to an increase the solubility in polar media and improves the possibility for further groups functionalization [20]. The aim of this study is to prepare a poly(2-aminobenzothiazole) (PABT)/(MWCNT) nanocomposite via chemical polymerization, characterize its chemical structure and morphology by using spectral techniques, and evaluate and enhance its thermal and electrical properties. The nanocomposite has been prepared

[DOI: 10.22068/ijmse.3928]

by modification of (MWCNT) with carboxilic groups via treatment with suitable acidic medium and then has interaction with electroactive polyamionbenzothiazole to create a nano-composite.

2. EXPERIMENTAL PROCEDURES

2.1. Materials

Multi-walled Carbon Nanotubes (MWCNTs) with an outer diameter of 10-30 nm and a purity of approximately 90% are used in this study. 37% hydrochloric acid (HCl), 65% nitric acid (HNO_3), and 95% sulfuric acid (H_2SO_4) are purchased from Sigma-Aldrich. 2-aminobenzothiozol (Sigma Aldrich), deionized water, Dimethylformamide (DMF), ammonium persulfate($(NH_4)_2S_2O_8$, pre-patterned ITO substrates consisting of interdigitated ITO fingers are purchased from Ossila company.

2.1.1. Instruments and measurement systems

Nicholet FT-IR Spectrometer, X-ray Diffraction Measurements, Thermo gravimetric analysis TG, Scanning Electron Microscopy SEM, Energy Dispersive X-Ray(EDX), Centrifuge, Keithley 2400 source meter was used to measure I-V characteristics.

2.2. Samples Preparation

2.2.1. Modification of Multi-walled Carbon Nanotube

One of the most common MWCNT functionalization techniques is oxidation adding hydroxyl -OH, carboxyl -COOH, and carbonyl -CHO groups, which provide hydrophobicity and improve surface accessibility for further chemical modifications, using oxidizing acid solutions such as HNO_3, H_2SO_4:H_2O_2, or H_2SO_4:HNO_3 that can be used to oxidize MWCNTs. Chemical oxidation was carried out using a mixture of sulfuric acid and nitric acid 90 mL and 30 mL respectively in a ratio of 3:1 in solutions to reduce the destruction of the nanotubes for half an hour, after which 0.3 g of multi-walled nanocarbon was added. [21] The mixture was stirred magnetically for 6 hours at 50°C. It was washed several times with water using a centrifuge. Then it was dried at room temperature at 30°C for 12 hours, as shown in Fig. 1.

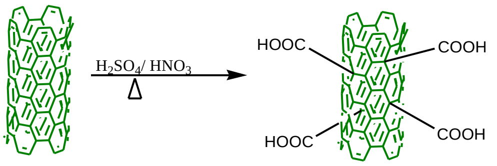

Fig. 1. Modification of multi-walled carbon nanotube

2.2.2. Syntheses of polymer 2-Aminobenzothiazole

The oxidative condensation (OP) reactions of 2-aminobenzothiozole were carried out in an aqueous acidic medium, as suggested for other aromatic amines [22]. An aqueous solution of ammonium persulfate $(NH_4)_2S_2O_8$ was chosen as a common oxidant for the oxidative condensation reactions of phenolic compounds. Ammonium persulfate $(NH_4)_2S_2O_8$ has a high oxidation potential and is inexpensive, and is therefore preferred for oxidative condensation reactions. The synthesis methods are summarized in Fig. 2. The synthesis procedures were as follows: 1 g (7 mmol) of 2-aminobenzothiozole monomer was dissolved separately in 50 mL aqueous solution containing 1 mL of concentrated hydrochloric acid. The solutions were placed in 250 cm^3 three-necked round-bottom flasks equipped with a condenser, a thermometer, and a magnetic stirrer. The reaction mixtures were heated to 70°C and the $(NH_4)_2S_2O_8$ solution (prepared by dissolving (1.5974 g (7 mmol) ammonium persulfate) in 8 mL deionized water) was added to the reaction medium dropwise. The reactions were refluxed for 6 h, and the solutions turned dark brown. The heat was then turned off, and stirring was continued for 72 h to complete the polymerisation. Dark brown solids were obtained and were filtered and dried at 38°C. The equation for the preparation of 2-aminobenzothiozole polymer is shown in Fig. 2.

2.2.3. Preparation of 2-aminobenzothiazole with MWCNTs composites

Poly 2-Aminobenzothiozole composites were prepared from Multi-walled Carbon Nanotubes (MWCNTs) using a weight percentage (1%) composite material. In general, the reaction conditions for preparing 1% Multi-walled Carbon Nanotubes (MWCNTs) polymer composite were given as follows:

1 g (7 mmol) of 2-aminobenzothiozole monomer was dissolved separately in 50 mL of aqueous solution containing 1 mL of concentrated hydrochloric acid. The solutions were placed in 250 cm^3 three-neck round-bottom flasks equipped with a condenser, a thermometer, and a magnetic stirrer. The reaction mixtures were heated to 70°C, and 0.01 g of multi-walled carbon nanotubes (MWCNTs) was added to the mixture, which was then ultrasonicated at 25°C for 3 h in an ultrasonic bath (7 mmol).

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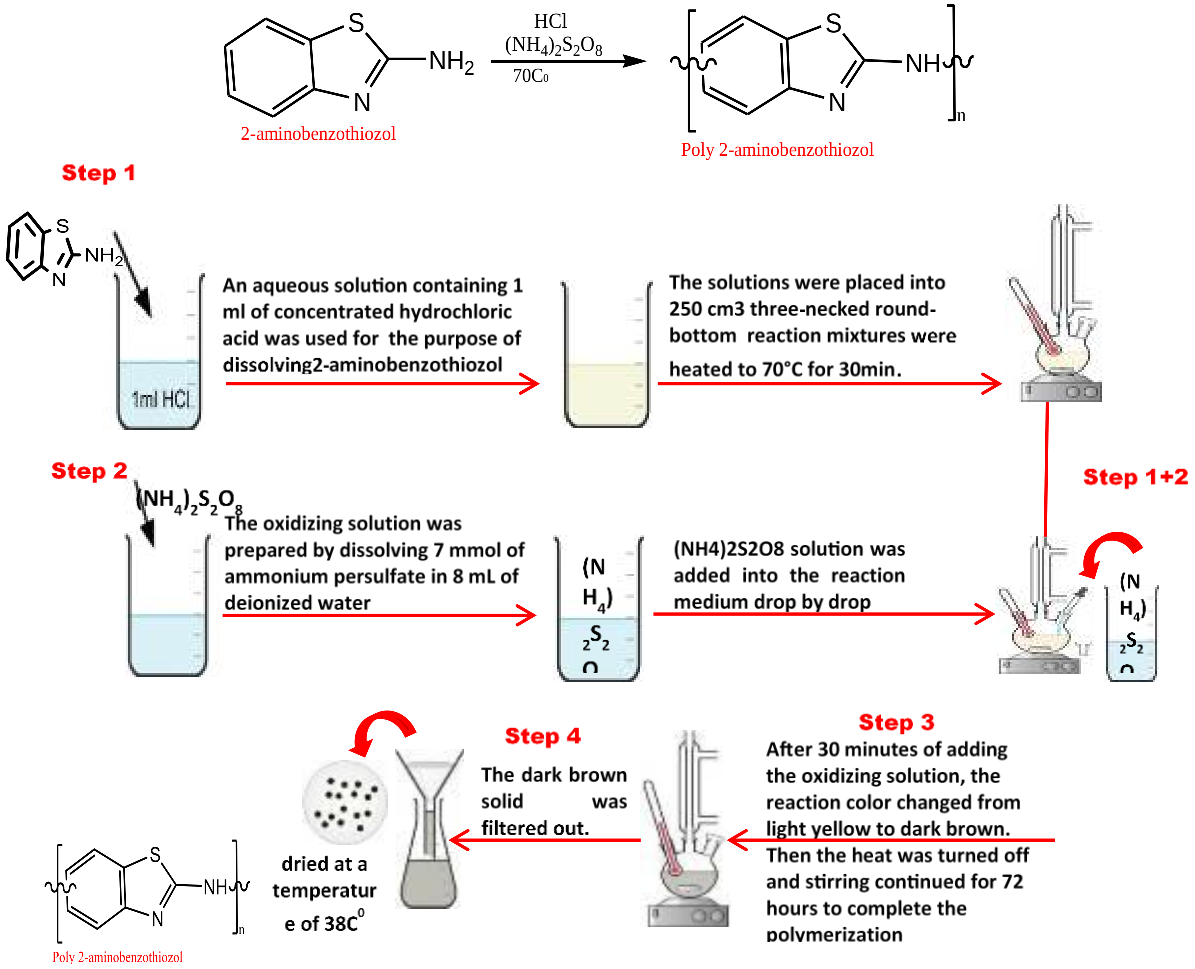

An aqueous solution containing 1 ml of concentrated hydrochloric acid was used for the purpose of dissolving2-aminobenzothiozol

The solutions were placed into 250 cm3 three-necked round-bottom reaction mixtures were heated to 70°C for 30min.

The oxidizing solution was prepared by dissolving 7 mmol of ammonium persulfate in 8 mL of deionized water

(NH4)2S2O8 solution was added into the reaction medium drop by drop

After 30 minutes of adding the oxidizing solution, the reaction color changed from light yellow to dark brown. Then the heat was turned off and stirring continued for 72 hours to complete the polymerization

The dark brown solid was filtered out.

dried at a temperature of 38C0

Fig. 2. Schematic diagram of polymer 2-Aminobenzothiazole preparation steps

The oxidizing solution was added. (prepared by dissolving (1.5974 g (7 mmol) ammonium sulfate in 8 mL of deionized water) was added to the reaction medium drop-wise. The reactions continued for 6 h under the reversal, and the solution turned dark brown. The heat was then turned off, and stirring continued for 72 h to complete the polymerisation. The solid fraction was collected by centrifugation, washed with deionised water, and dried at 38°C. The preparation steps of 2-aminobenzothiozol: MWCNTs composite were illustrated in Fig. 3, and the hydrogen bonding of 2-aminobenzothiozol polymer with multi-walled carbon nanotube (MWCNTs) was shown in Fig. 4.

2.2.4. Preparation of 2-aminobenzothiazole and 2-aminobenzothiozole: MWCNTs thin films

The pure polymer and composite thin films were fabricated as follows: 0.02 g of 2-Aminobenzothiozole and 0.02 g of the 2-Aminobenzothiozole: MWCNTs composite were separately dissolved in 2 mL of DMF. Both solutions were dispersed in an ultrasonic bath at 40°C for 10 min. Then each solution was cast on interdigitated ITO substrates at 40°C for 3 hours [22].

3. RESULTS AND DISCUSSION

3.1. Structural Analysis

3.1.1. FTIR spectroscopy

It is essential to understand our synthesized compounds. In this study, FT-IR was used to examine the structures of various compounds. Fig. 5(a) shows the FTIR spectra of the monomer compound obtained from Fluka. We will list and assign some main peaks. A medium absorption band at 3338 cm^{-1} and a band at 3273 cm^{-1} are observed in the case of the monomer, which could be due to the NH_2 stretching vibration of the primary amine. A medium absorption band appearing at 3055 cm^{-1} in the case of monomer is attributed to the C-H stretching vibration of the benzene ring.

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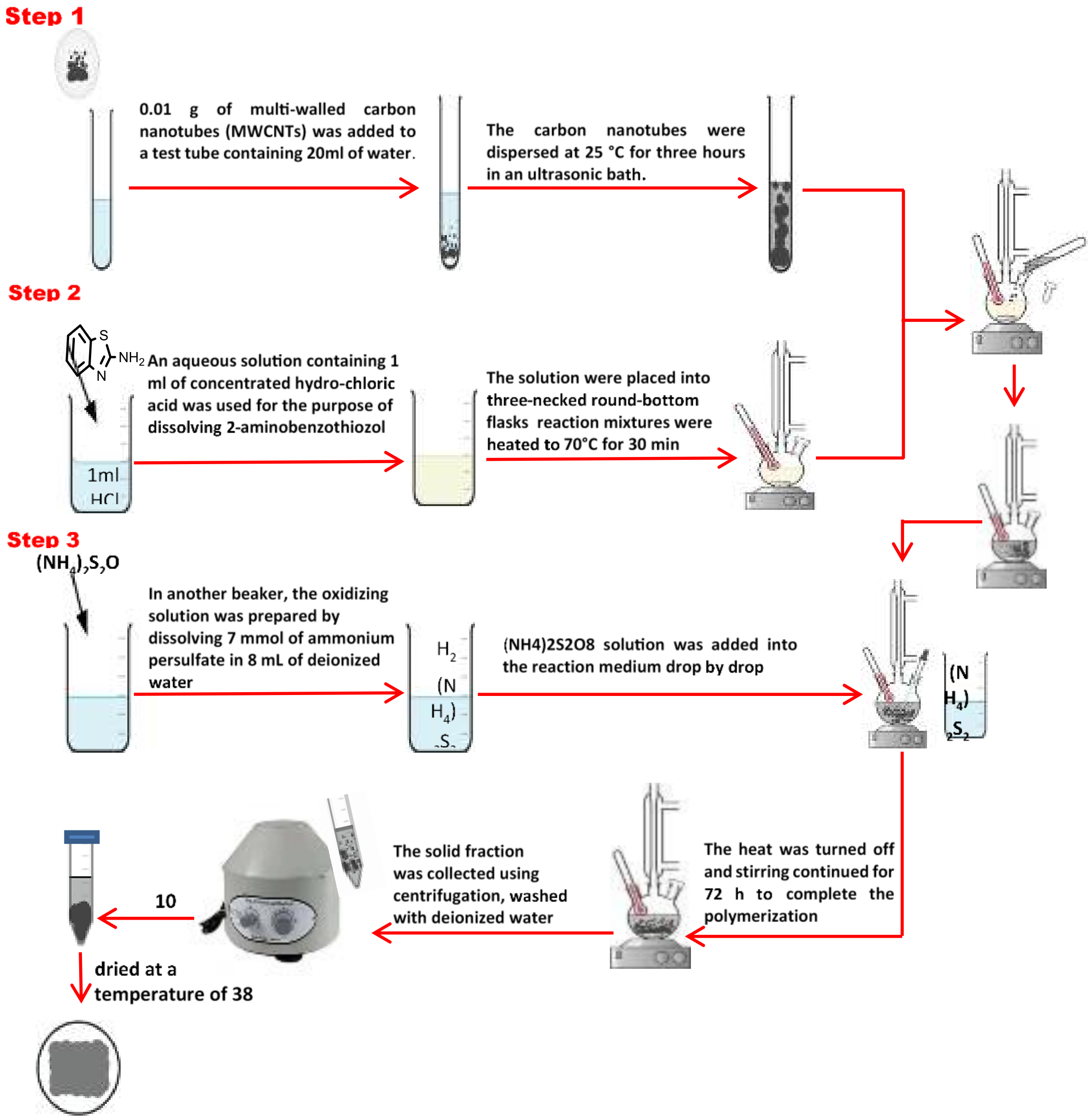

0.01 g of multi-walled carbon nanotubes (MWCNTs) was added to a test tube containing 20ml of water.

The carbon nanotubes were dispersed at 25 °C for three hours in an ultrasonic bath.

An aqueous solution containing 1 ml of concentrated hydro-chloric acid was used for the purpose of dissolving 2-aminobenzothiozol

The solution were placed into three-necked round-bottom flasks reaction mixtures were heated to 70°C for 30 min

In another beaker, the oxidizing solution was prepared by dissolving 7 mmol of ammonium persulfate in 8 mL of deionized water

(NH4)2S2O8 solution was added into the reaction medium drop by drop

The heat was turned off and stirring continued for 72 h to complete the polymerization

The solid fraction was collected using centrifugation, washed with deionized water

dried at a temperature of 38

Fig. 3. Schematic diagram of 2-Aminobenzothiazole: MWCNTs composite preparation steps

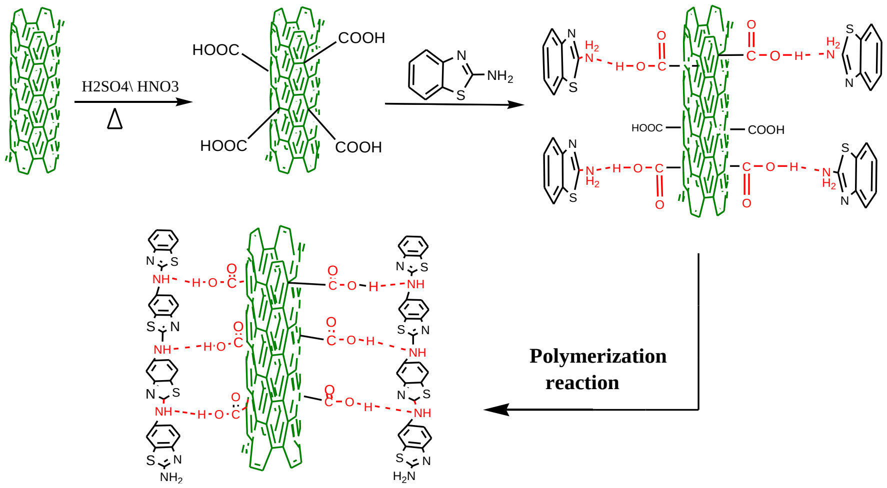

Fig. 4. The hydrogen bonding of 2-aminobenzothiozole polymer with multi-walled carbon nanotube (MWCNTs)

The peak at 1643 cm^{-1} relates to the C=C stretch of benzene. The C=N stretch has a peak at 1589 cm^{-1}. The peaks at about 1120 to 1107 cm^{-1} corresponded to the C-H out-of-plane bending vibration. The medium absorption band appears at 626.8 cm^{-1}, which could be attributed to the C–S symmetric stretching vibration. Fig. 5(b) shows the FTIR spectra for CNTs-COOH. The IR spectrum for the CNTs-COOH shows a new adsorption band with a characteristic –COOH peak at 3358 cm^{-1}. Another peak appears at 2924 cm^{-1}, which is attributed to symmetric and asymmetric CH_2 stretching, while 1695 cm^{-1} is assigned to carboxylic C=O stretching for the acidic group. Moreover, a sharp carboxylic peak of C=O stretch attributed to the –COOH group was also observed at 1745 cm^{-1} [24].

Fig. 6(a) shows the FTIR spectra of the prepared polymer compound. We will list and assign some main peaks. A broad absorption peak is observed from 3600 to 2000 cm^{-1} in the polymer spectrum, attributed to electronic transitions from the valence band to the conduction band in the polymer film. This peak is usually broad and can obscure other peaks across the region from 3400 to 2000 cm^{-1}. The 3402 cm^{-1} peak, which appears as a broad absorption in the polymer, may be due to a strong hydrogen-bonded group in H_2O molecules of hydration in the polymer. It is noted that the NH_2 band has disappeared in the spectrum.[16] This indicates that the polymerisation is formed via NH_2. The medium absorption band, which appears at 3325 cm^{-1}, could be due to the NH stretching vibration of the amine. A medium absorption band appearing at 3055 cm^{-1} in the case of monomer is attributed to the C-H stretching vibration of the benzene ring. The peak at 1622 cm^{-1} relates to the C=C stretch of benzene. The C=N stretch has a peak at 1589 cm^{-1}. The peaks at about 1120 to 1107 cm^{-1} corresponded to the C-H out-of-plane bending vibration. The medium absorption band appears at 626.8 cm^{-1}, which could be attributed to the C–S symmetric stretching vibration.

Fig. 6(b) shows the FTIR spectra of the prepared polymer composite. A broad absorption peak is observed from 3600 to 2100 cm^{-1} in the composite spectrum, attributed to electronic transitions from the valence band to the conduction band in the polymer film and to hydrogen bonds between NH groups in the polymer and the COOH groups in the nanotube.

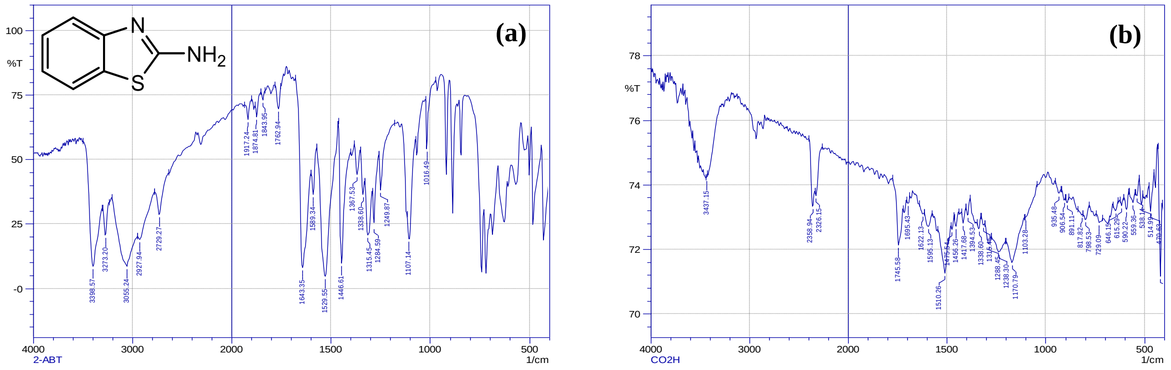

Fig. 5. FTIR spectra of a) monomer compound and b) MWCNTs-COOH

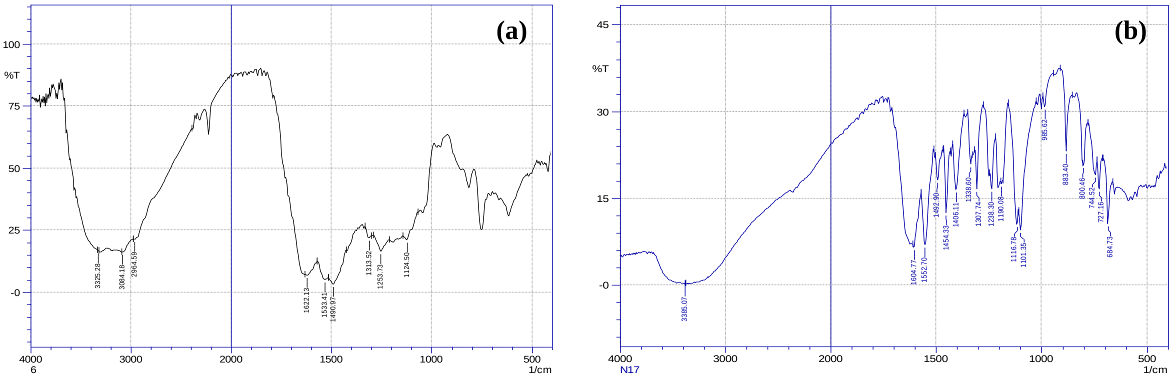

Fig. 6. FTIR spectra of a) 2-Aminobenzothiozole and b) 2-aminobenzothiozole: MWCNTs composite

[DOI: 10.22068/ijmse.3928]

This peak is broad and can obscure other peaks in the 3400-2000 cm^{-1} region. A medium absorption band is observed at 3325 cm^{-1}, which may be due to the NH stretching vibration of the amine and may overlap with the broad peak of hydrogen bonds. The peak at 1622 cm^{-1} relates to the C=C stretch of the aromatic ring. The C=N stretch has a peak at 1589 cm^{-1}. The peaks at about 1120 to 1107 cm^{-1} corresponded to the C-H out-of-plane bending vibration. The medium absorption band appears at 625 cm^{-1},which could be attributed to C–S symmetric stretching vibration [24].

3.2. Thermal Characterization

TG–DTA curves are shown in Fig. 7. According to the results, PAT and its composite thermally degrade in three distinct steps. Materials start to decompose at 54°C and 63°C, respectively. Compared to the first degradation temperatures, the composite was more stable than the polymer.

In general, aromatic thiazole-based polymers exhibit high thermal stability due to their stable resonance structures. 5–11% weight losses between 20 and 140°C are due to losses of moisture, adsorbed solvent or monomer. The first steps could indicate the degradation of polymer chains at their bonds, forming small subunits; the other thermal degradation is due to the degradation of those small units. [25] According to the thermal curve, the polymer and its composite have finally converted to different subunits, including NH_3, HCN, CS_2 and carbon residue.

The overall weight loss to the original weight of PAT polymer at the end of decomposition was found to be 64% at 600°C for the pure PAT, whereas the overall weight loss of the nano-composite was lower than that of the pure polymer (30% at 600°C). These findings indicate that the thermal stability of the nano-composite has been enhanced by the interaction between the nanotube and PAT, compared to that of the homopolymer (PAT).

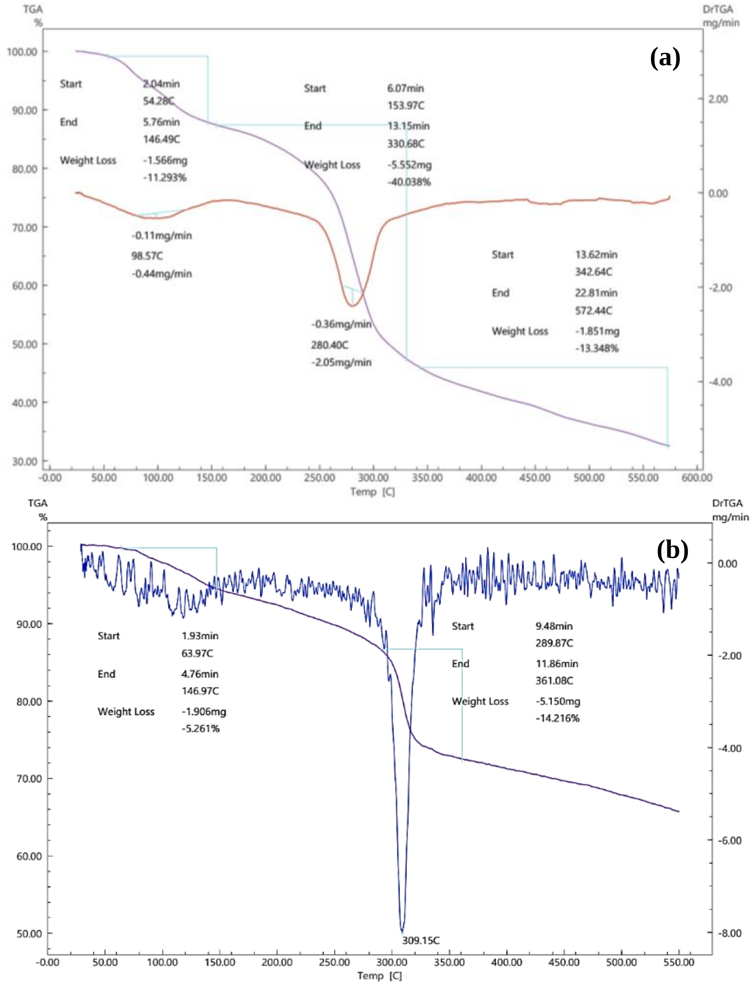

Fig. 7. TG curve of a) poly 2-Aminobenzothiozole and b) poly 2-aminobenzothiozole: MWCNTs composite

[DOI: 10.22068/ijmse.3928]

3.3. SEM measurements

Fig. 8 shows the scanning electron microscopy (SEM) images of the synthesised materials and fabricated thin films. From the SEM images in Figs. 8(b1) and 7(b2), it is clearly observed the MWCNTs are located within the structure of the polymer, proving that the polymer: MWCNTs composite was successfully prepared. Further, typical SEM images of the prepared polymer show many pieces with dimensions of hundreds of nanometers. Furthermore, as can be seen from the SEM images of the fabricated thin films, the surface morphology of the pure polymer thin film (see Fig. 8(c1) and 8(c2)) is smooth and uniform compared with that of the 2-aminobenzothiozole: MWCNTs films where a significant change in the surface topography was observed with high visibility of MWCNTs in the structure of the doped film as illustrated in Figs. 8(d1) and 8(d2) [26].

3.4. XRD measurements

XRD patterns of the poly2-aminobenzothiozol and poly2-aminobenzothiozol: MWCNTs composite were shown in Fig. 9. For the pure PAT, the broad reflection centered at a 2θ value around xx is merit of the amorphous polymer. Further, the poly(2-aminobenzothiazole): MWCNT composite shows two additional peaks at 2θ=23° and 42°, corresponding to reflections of the MWCNT compound in the polymer matrix. The mass fraction of MWCNTs in poly2-aminobenzothiozol: MWCNTs composite is sufficiently tiny that the MWCNTs diffraction peaks can hardly be noted for 1 wt % MWCNTs-containing poly2-aminobenzothiozol: MWCNTs composites. However, traces of diffraction peaks at 2θ=23° and 42° were observed, indicating that the MWCNTs had partially interacted with poly(2-aminobenzothiazole) molecules [27].

3.5. Electrical Measurements

Current-voltage (I-V) characteristics of the pure polymer and 2-aminobenzothiazole: MWCNTs composite thin films under dark conditions. The measurements were carried out at room temperature over an applied voltage range from 0.025 V to 10 V, and the results were plotted in Fig. 10. It was found that when the voltage increases, the current of the fabricated thin films increases gradually, showing a linear behavior (Ohmic low) for pure polymer thin film and a nonlinear behavior for composite thin film. The conductivity σ of the two samples was determined from the following equation [28]:

$$\sigma = \frac{I}{V}\frac{d}{LtN} \qquad (1)$$

Where L is the length of fingers, d is the distance between the fingers electrodes, N is the number of fingers, and t is the thickness of the thin films. I and V are the electrical current and voltage, respectively. The resistivity was also calculated from the formula:

$$\rho = 1/\sigma \qquad (2)$$

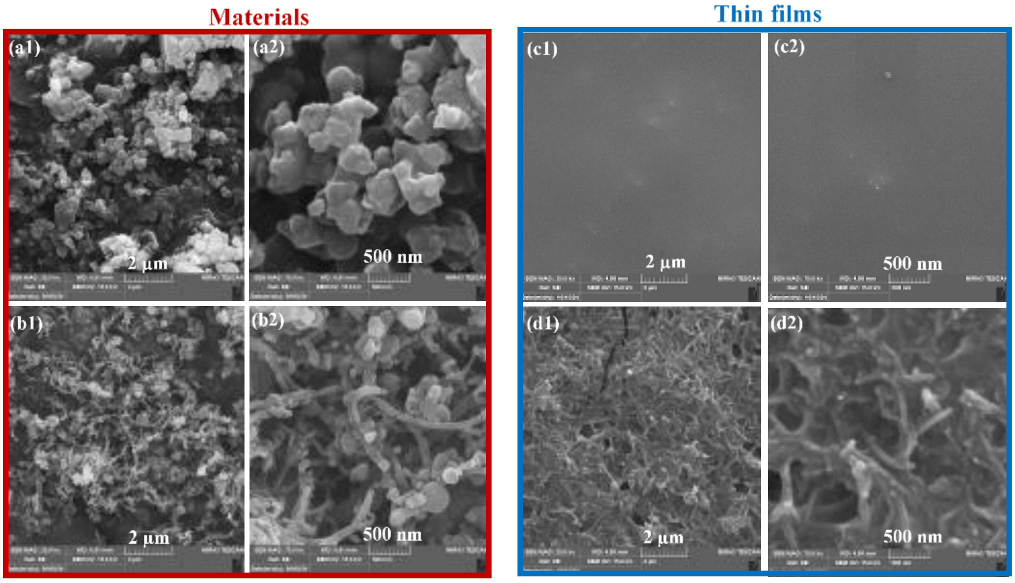

Fig. 8. Fig.8. Scan electron microscopy (SEM) images of (a1) poly2-Aminobenzothiozol, (b1) poly 2-aminobenzothiozol: MWCNTs composite, (c1) poly 2-Aminobenzothiozol thin film, and (d1) poly 2-aminobenzothiozol: MWCNTs composite thin film.

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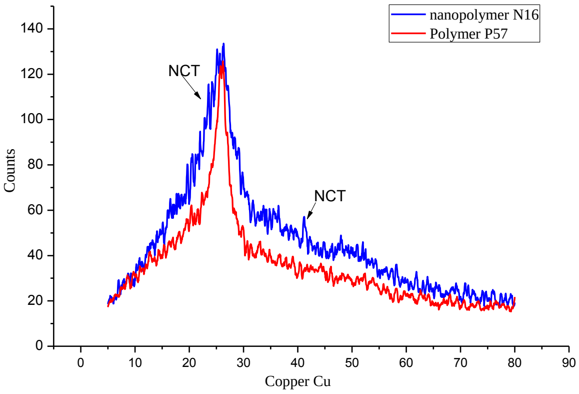

Fig. 9. X-ray diffraction (XRD) curve of poly 2-Aminobenzothiozol (red curve), poly 2-aminobenzothiozol: MWCNTs composite

The conductivity and resistivity of the fabricated thin films were evaluated and listed in Table 1. As shown in Fig. 10 and Table 1, the conductivity of the pure polymer thin film increases from 1.7×10^{-6} S/cm to 4.1×10^{-2} S/cm as a result of doping by 1 %wt of MWCNTs, showing a significant enhancement by a factor of 24000x. In contracts, the resistivity of the pure polymer reduces from 0.68 MΩ.cm to 24.5 Ω.cm [29].

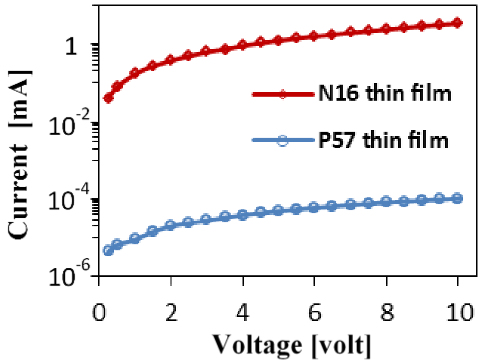

Fig. 10. Current-Voltage (I-V) characteristics of the pure polymer (P57) and 2-aminobenzothiozole: MWCNTs composite (N16) thin films under dark conditions

The temperature dependence of the current, conductivity, and resistivity of the fabricated thin films was also investigated, as illustrated in Fig. 11.

Table 1. The conductivity and resistivity values of the pure polymer and the composite thin films at room temperature

Sample	Conductivity	Resistivity	Enhancement
2-aminobenzothiozol	1.7×10^{-6} S/cm	0.68 MΩ.cm	-
2-aminobenzothiozol:MWCNTs N16	4.1×10^{-2} S/cm	24.5 Ω.cm	24000x

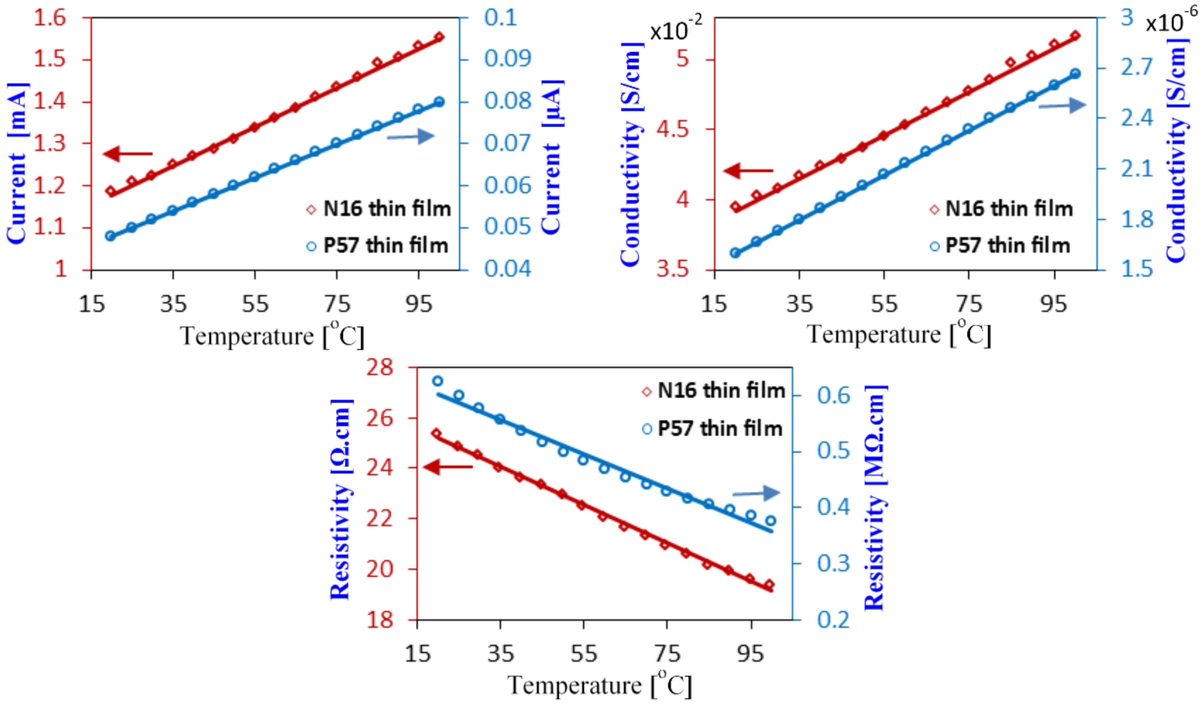

Fig. 11. Temperature dependence of the current, conductivity and resistivity for the pure polymer (P57) and 2-aminobenzothiazole: MWCNTs composite (N16) thin films under dark conditions

The measurements were carried out over a temperature range of 20ºC to 110ºC at an applied voltage of 5 V. The results revealed that the electrical parameters, current and conductivity, increase gradually with increasing temperature, while resistivity decreases. As shown in Fig. 11, when the temperature increases from 20ºC to 110ºC, the conductivity of the pure polymer thin film increases from 1.6×10^{-6} S/cm to 2.8×10^{-6} S/cm, while the resistivity decreases from 0.625 MΩ to 0.356 MΩ. For composite thin film, the conductivity increases from 4.1×10^{-2} S/cm at 20ºC to 5.3×10^{-2} S/cm at 20ºC, and the resistivity reduces from 25.35 Ω.cm at 20ºC to 18.85 Ω.cm at 110ºC. The optimisation in conductivity and resistivity is attributed to the generation of carriers via thermal excitation. It was also found that the temperature coefficients of the DC conductivity for the fabricated thin films are positive, indicating that the two samples are semiconductors [30].

4. CONCLUSIONS

In this study, Poly(aminobenzothiazole) (PAT) was successfully synthesized by chemical-oxidative polymerization technique. Then, a composite material with modified carboxyllic multi-wall carbon nanotubes (MWCNTs) was prepared. The structure of the polymer matrix and its composite was characterized by several techniques including FT-IR, SEM, and TG techniques. The various peaks observed in the FTIR spectra confirm the functionalization of CNTs, with a new peak arising from oxidation. In addition, other peaks appeared in the FTIR spectra of the prepared polymer and the interaction between CNTs and polymer chains. The TG findings indicated that the thermal stability of the PAT polymer improved upon MWCNT doping. The overall weight loss relative to the original weight of the PAT polymer was 64%, whereas that of the nanocomposite was 30%. These findings indicate that the nanocomposite's thermal stability has improved due to interactions between the nanotubes and the polymer chains. The SEM images confirm that the pure polymer was successfully doped with MWCNTs, with several MWCNTs overlapping with the pure polymer's structure. Thus, the carrier concentration of the doped polymer could be increased by increasing the dopant energy levels. Consequently, the findings show that the conductivity of composite thin films reaches 4.1×10^{-2} (S/cm), exhibiting a significant enhancement by a factor of 24000 due to doping the polymer with 1% wt. MWCNTs.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the Department of Chemistry and Department of Physics, College of Education for Pure Science, for their assistance.

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[DOI: 10.22068/ijmse.3928]