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Synthesis and Characterization of Conductive Copolymer/MWCNT Nanocomposite via Chemical and Interfacial Polymerization

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Abstract:

Various monomers were employed to synthesize copolymers, followed by the fabrication of nanocomposites through the incorporation of 1% carboxyl-functionalized multi-walled carbon nanotubes (MWCNTs–COOH). These nanotubes were modified in the laboratory to enhance the interaction between the polymer matrix and the nanotubes. The nano-doping has led to improve the electrical and thermal performance of the producte materials. The FTIR, XRD, TGA, SEM, and EDX were utilized to characterized composites, while electrical conductivity was measured using the pellet method. The results revealed that the incorporation of functionalized MWCNTs led to significant improvements in electrical conductivity, thermal stability, and morphological structure of the copolymer systems, highlighting their potential for advanced electronic and optoelectronic applications.

Keywords: Nanocomposites, Copolymers**,** Carbon Nanotubes (MWCNTs–COOH)**,** Electrical Conductivity**,** pellet method

1.Introduction:

In recent decades, the technology of industrial materials has witnessed remarkable growth, with nanocomposites especially those integrating polymers and carbon nanotubesstanding out as a key area of innovation. This is due to the unique combination of organic polymer flexibility with the exceptional characteristics of carbon nanotubes[1], such as high electrical conductivity, mechanical strength[2], and thermal stability[3]. One of the main challenges in producing efficient polymer–nanotube systems lies in enhancing the interfacial interaction between the two components. To improve dispersion and compatibility, carbon nanotubes are commonly functionalized through acid treatments that introduce reactive groups like carboxyls, promoting stronger bonding with polymer chains[4]. In this study, a series of copolymers were synthesized using two distinct techniques: chemical polymerization and interfacial polymerization. Multiwalled carbon nanotubes (MWCNTs), previously functionalized by acid treatment, were incorporated to fabricate nanocomposite materials with improved electrical conductivity, thermal stability, and surface morphology. This work represents a step forward in the design of multifunctional materials suitable for advanced applications such as sensors, smart coatings, and flexible electronics.

2.Experimental Section:

2.1. Materials:

1,8-naphthalendiamine (Merck), Carbazole and Ammonium peroxydisulfate (BDH, England), 2-aminophenol , Indole (Aldrich), multiwall carbon nanotube (MWNT) with outer diameter of 10-30 nm, hydrochloric acid (HCl), Nitric acid(HNO_3) , Perchloric Acid($HClO_4$) and Sulfuric acid (H_2SO_4) are purchased from Sigma – Aldrich, 8- Hydroxy quinolin.

2.2.Instruments and measurement systems:

FT-IR Spectrometer, X- ray Diffraction Measurements, (Rigaku UItima iv "japan", Thermo gravimetric analysis TG(TG-50A Shimadzu, Japan) Scanning Electron Microscopy(SEM)(TESCAN mira3) "Japan ", Energy Dispersive X- Ray(EDX) , Keithley 2400 source meter was used to measure I-V Characteristics.

2.3 samples preparation:

2.3.1. Chemical Functionalization of Multi-walled Carbon Nanotubes:

The chemical oxidation of multi-walled carbon nanotubes (MWCNTs) was carried out using a mixture of sulfuric acid and nitric acid in a 3:1 volume ratio, specifically 90 mL of sulfuric acid and 30 mL of nitric acid. he acids were prepared as a solution to minimize structural damage to the nanotubes during treatment. After 30 minutes, 0.3 grams of MWCNTs were added to the acid mixture[5]. The resulting suspension was magnetically stirred for six hours at 50°C[6]. Following the reaction, the mixture was washed several times with distilled water using a centrifuge to remove residual acids. The final product was then dried at room temperature (30°C) for 12 hours[7] .

2.3.2. poly (2-aminophenol and 8- Hydroxy quinolin):

The copolymer was synthesized by dissolving 1.1612 g (8 mmol) of 8-hydroxyquinoline in 30 mL of0.8730 g (8 mmol) of 2-aminophenol was dissolved in 50 mL of perchloric acid-acidified water (containing 1 mL of $HClO_4$). The two monomer solutions were mixed together, followed by the gradual addition of the oxidizing agent prepared by dissolving 3.6512 g (16 mmol) of ammonium persulfate in 15 mL of deionized water. The oxidant was added dropwise using a dropping funnel. The reaction mixture was stirred continuously for 48 hours using a magnetic stirrer to ensure complete polymerization. After completion of the reaction, the mixture was filtered, thoroughly washed with deionized water, and dried at 50 °C. The resulting precipitate appeared as a dark brown solid with a melting point above 300 °C[8]. Figure (1) illustrates the proposed reaction pathway for the formation of the[9]

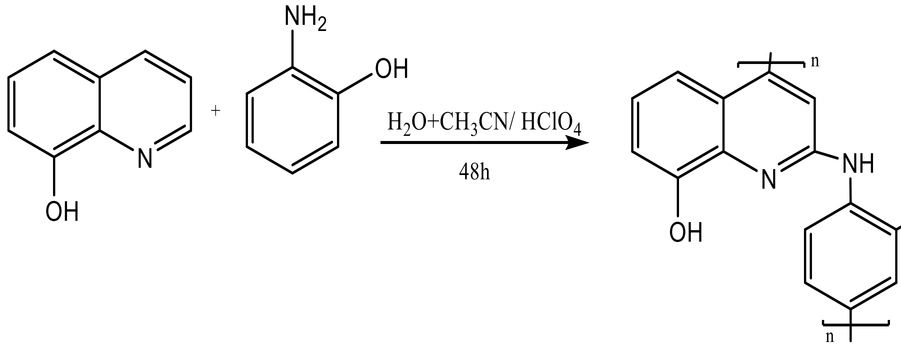


Figure 1: illustrates the reaction equation for the synthesis of the copolymer poly(2-aminophenol-co-8-hydroxyquinoline)

2.3.3. poly (1,8-naphthalendhamine and Indol):

The copolymer was synthesized via interfacial polymerization at room temperature. A solution of 0.158 g (1mmol) of 1,8-naphthalenediamine was prepared by dissolving it in 40 mL of dichloromethane. In a separate beaker, 0.234 g (2 mmol) of indole was dissolved in 40 mL of dichloromethane. The two monomer solutions were then mixed together in 80 mL of deionized water containing 5 mL of hydrochloric acid (HCl). This oxidizing mixture was added along the wall of the reaction vessel to initiate the polymerization. The reaction was left undisturbed for three days at room temperature to ensure complete polymerization. After the polymerization process, the reaction mixture was filtered, washed thoroughly with deionized water, and dried at 50 °C. The resulting precipitate appeared black in color and exhibited a melting point above 300 °C[10][11] .

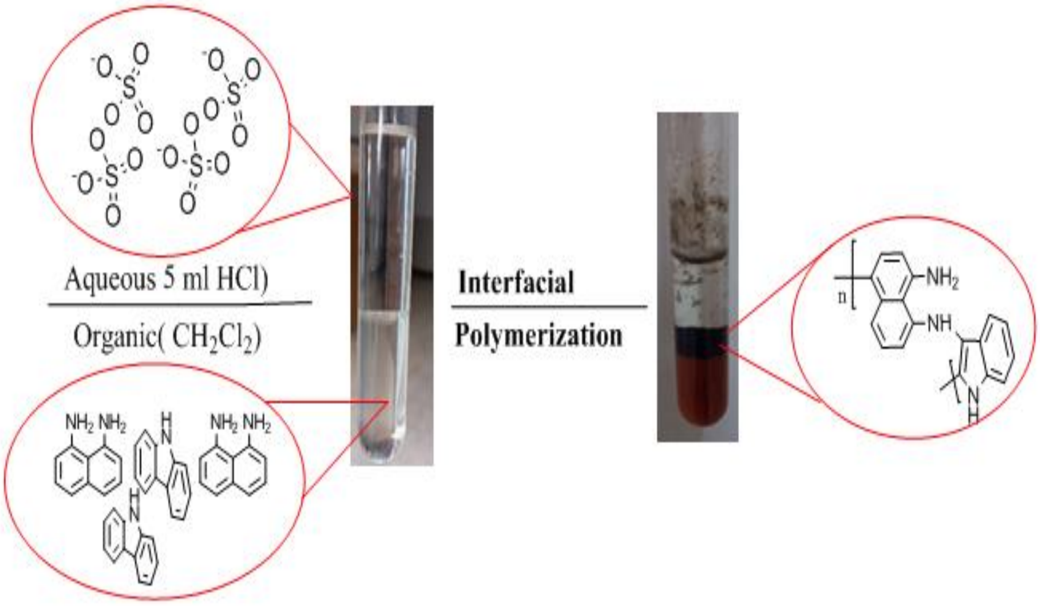


Figure (2) illustrates the reaction equation for the synthesis of the copolymer poly(1,8-naphthalenediamine-co-indole).

2.3.4.Synthesis of poly, (2-aminophenol and 8- Hydroxy quinolin), (1,8-naphthalendhamine and Indol) Carboxylated CNT Nanocomposite:

Separately, 0.003 g of functionalized multi-walled carbon nanotubes (MWCNTs) was dispersed using an ultrasonic bath to ensure uniform dispersion. The dispersed nanotubes were then added to the polymer solution, and the mixture was stirred continuously for 24 hours to allow proper interaction and integration. The resulting Nanocomposite was subsequently dried at 50 °C.

3. Results and discussion:

3.1.1. FTIR Fourier Transform Infrared Spectroscopy:

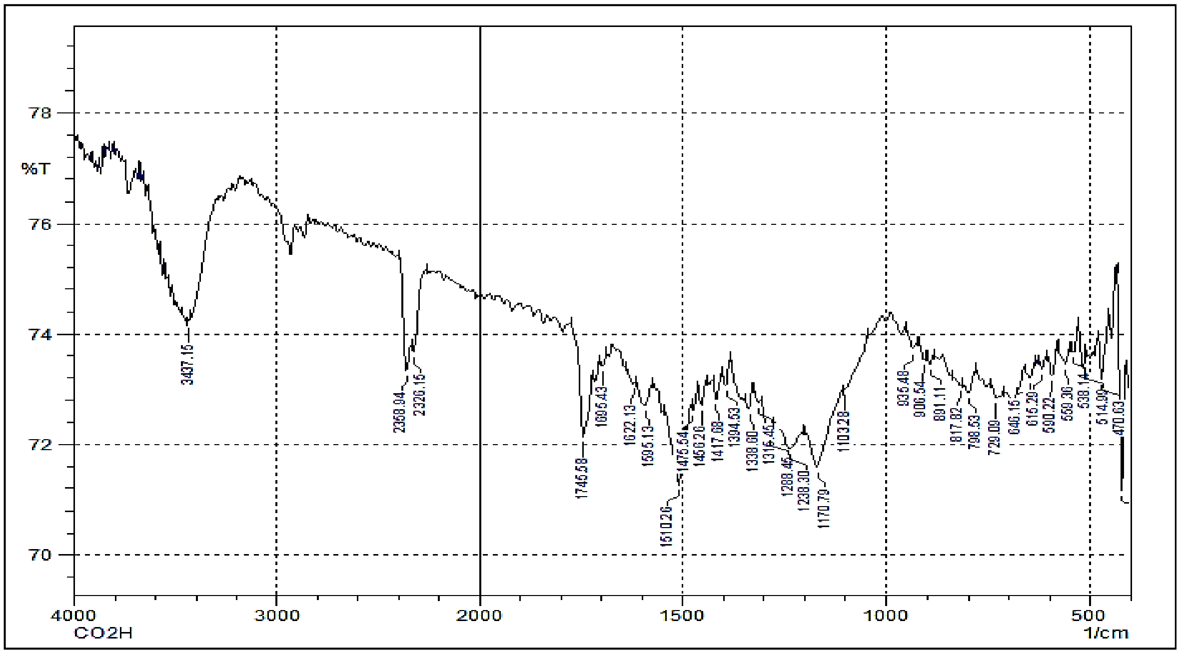
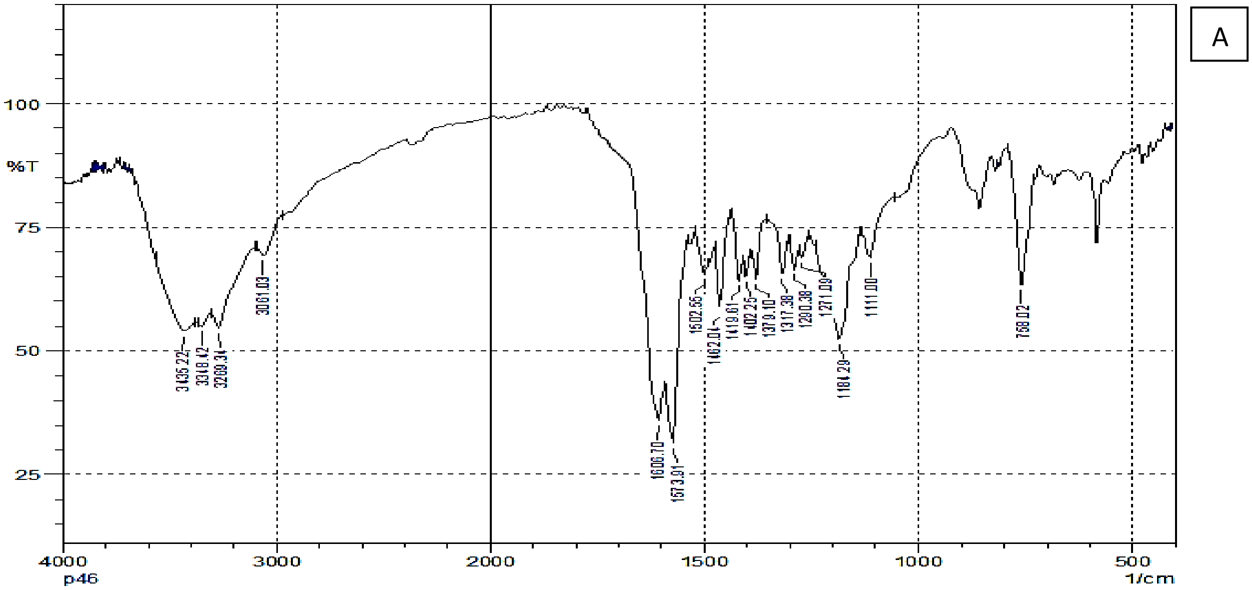


Figure (3) shows the FTIR spectrum of carboxylated carbon nanotubes



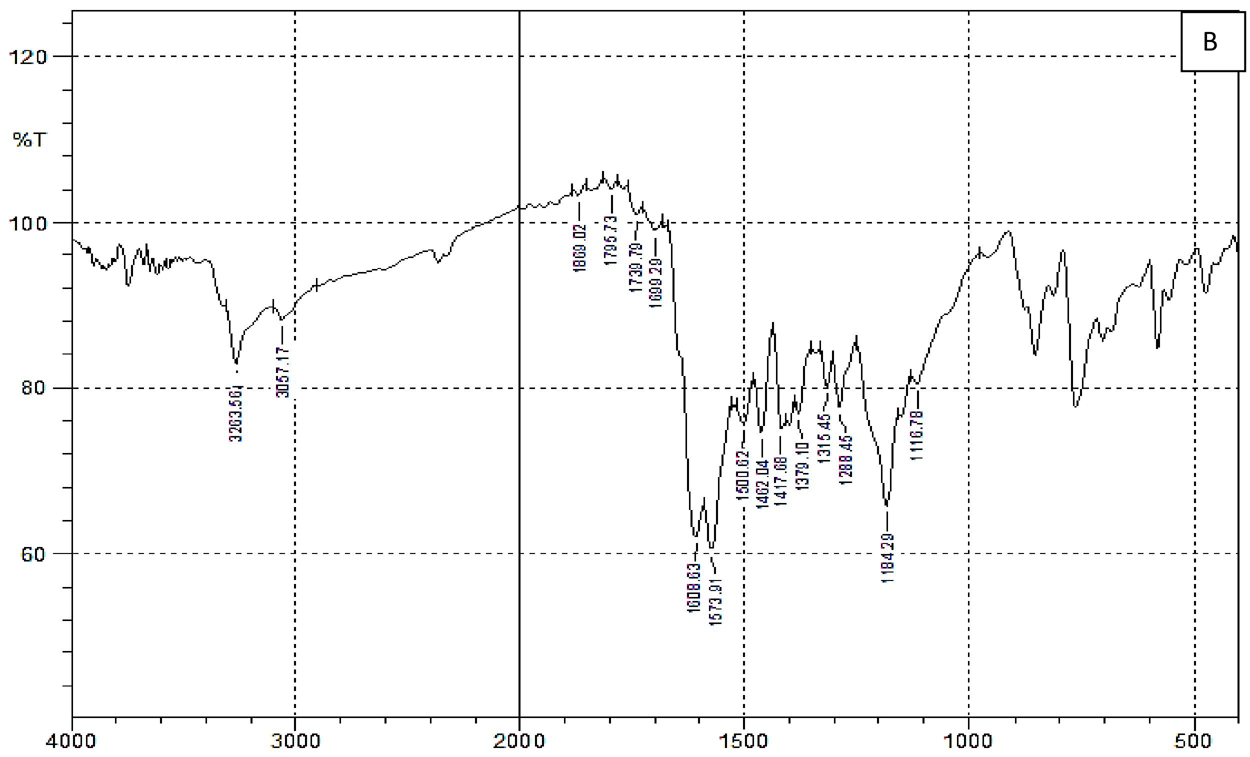
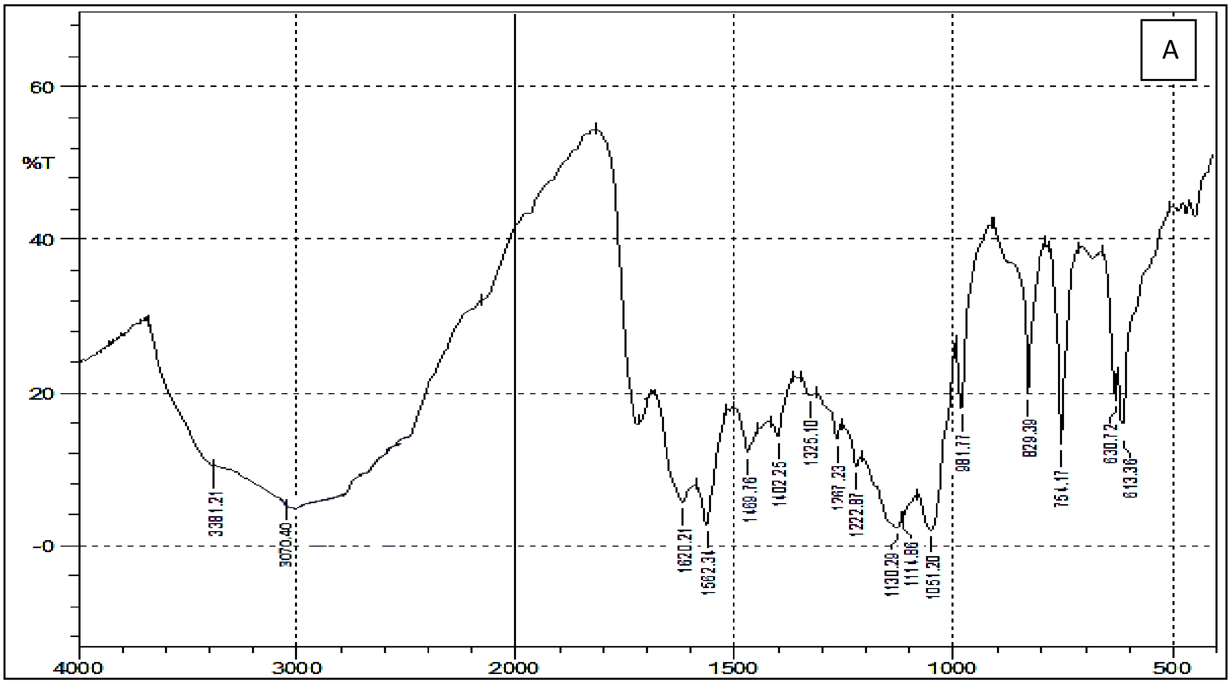
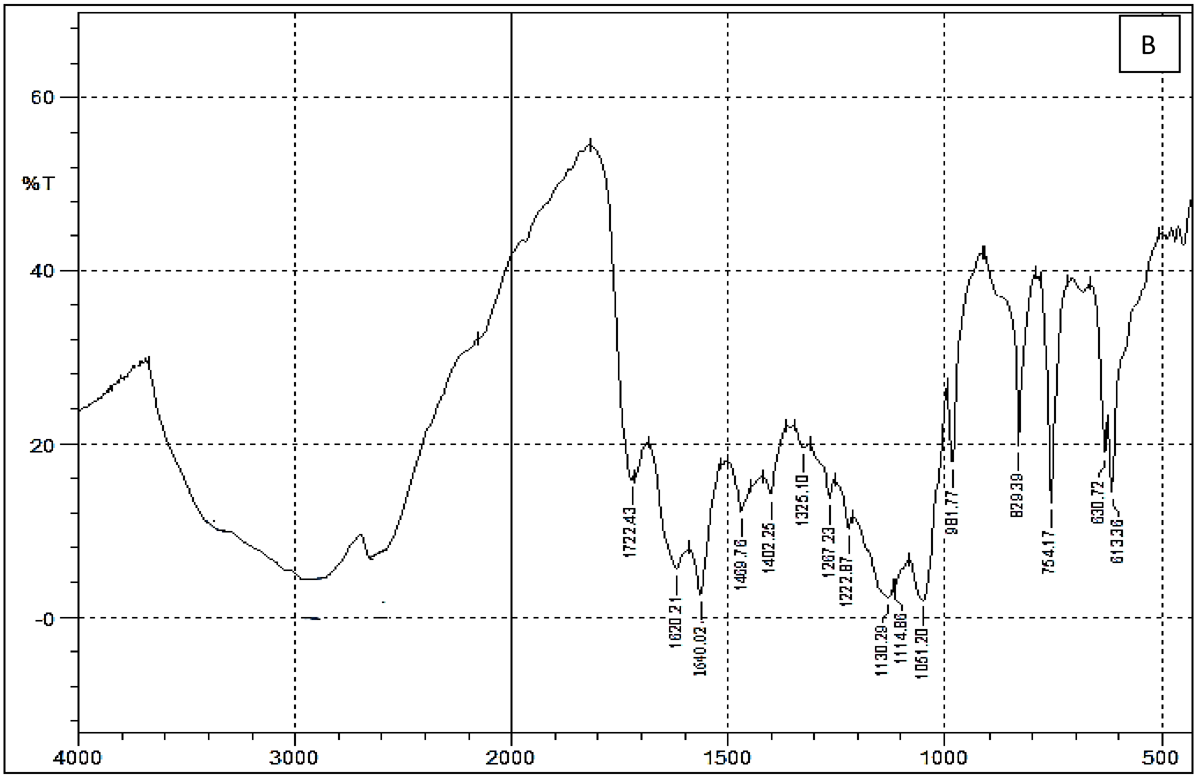


Figure (4) FTIR spectrum of (A) the copolymer of 8-hydroxyquinoline and 2-aminophenol (B) the copolymer of 8-hydroxyquinoline and 2-aminophenol with MWNTs-COOH





"**Figure (5)** shows the FTIR spectra of (A) the copolymer of 1,8-naphthalene diamine and indole, and (B) the copolymer of 1,8-naphthalene diamine and indole combined with MWNTs-COOH

Figure (3)the beaks at 3435 cm⁻¹ is attributed to the stretching vibration of acidic hydroxyl (–OH) groups A band observed at 2975 cm⁻¹ corresponds to aliphatic C–H stretching vibrations [169]. Additionally, the band at 1745 cm⁻¹ is assigned to the stretching vibration of the carbonyl (C=O) group(1745)[12][13]. Figure (4) shows absorption bands at 3435–3269 cm⁻¹ due to –OH and –NH stretching, indicating strong hydrogen bonding[14] .The 3061 cm⁻¹ peak corresponds to aromatic C–H, confirming aromatic structure retention. Peaks at 1606–1573 cm⁻¹ are related to C=C and C=N stretching, while 1290–1317 cm⁻¹ indicates C–N–C linkages. The 758 cm⁻¹ band suggests out-of-plane C–H bending, supporting the presence of substituted benzene rings[9]. Figure (4B) shows a broad band at 3400–3000 cm⁻¹, attributed to overlapping –NH, –OH, and –COOH stretching, indicating hydrogen bonding between the copolymer and MWCNTs. The 3067 cm⁻¹ peak corresponds to aromatic C–H, and the distinct 1715 cm⁻¹ band indicates C=O stretching from CNT surface carbonyls. Sharp peaks at 1609 and 1573 cm⁻¹ are assigned to aromatic C=C stretching. Figure (5) shows a broad band at ~3300 cm⁻¹ due to –NH/–NH_2 stretching, indicating aromatic amines in the polymer. Peaks at 1618 and 1562 cm⁻¹ confirm the presence of aromatic C=C and C=N, while the 1336 cm⁻¹ band evidences C–N bond formation between indole and 1,8-naphthalenediamine[15][16]. Figure (5B), confirms the retention of structural features of both the copolymer and CNT–COOH[17], with a slight N–H peak shift suggesting possible hydrogen bonding or π–π interactions between the two components.

3.1.2. thermal analysis(TG)

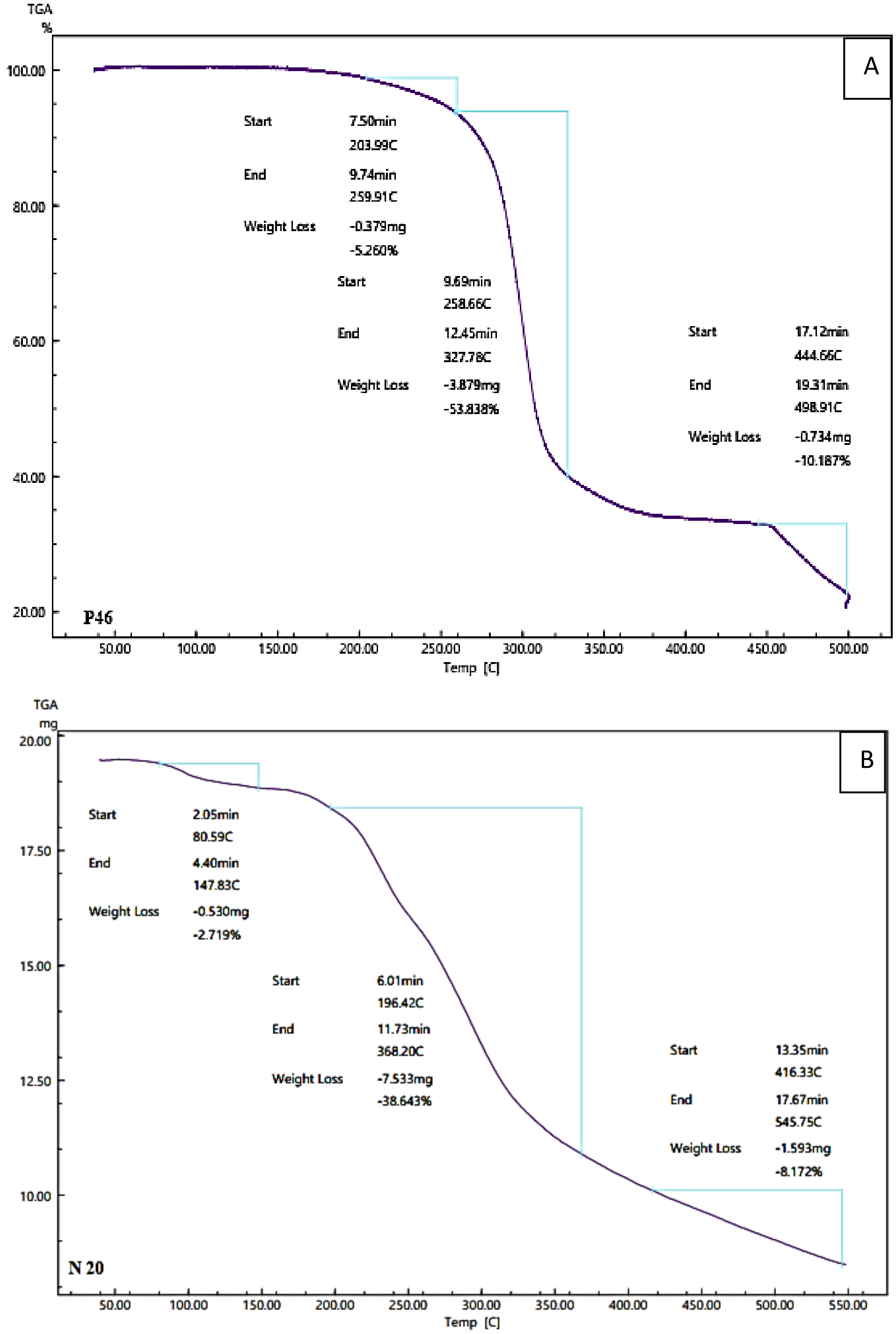


Figure (6) presents the thermogravimetric analysis (TGA) of (A) the copolymer of 2-aminophenol and 8- hydroxyquinoline, and (B) the copolymer of 2-aminophenol and 8-hydroxyquinoline incorporating MWNTs-COOH

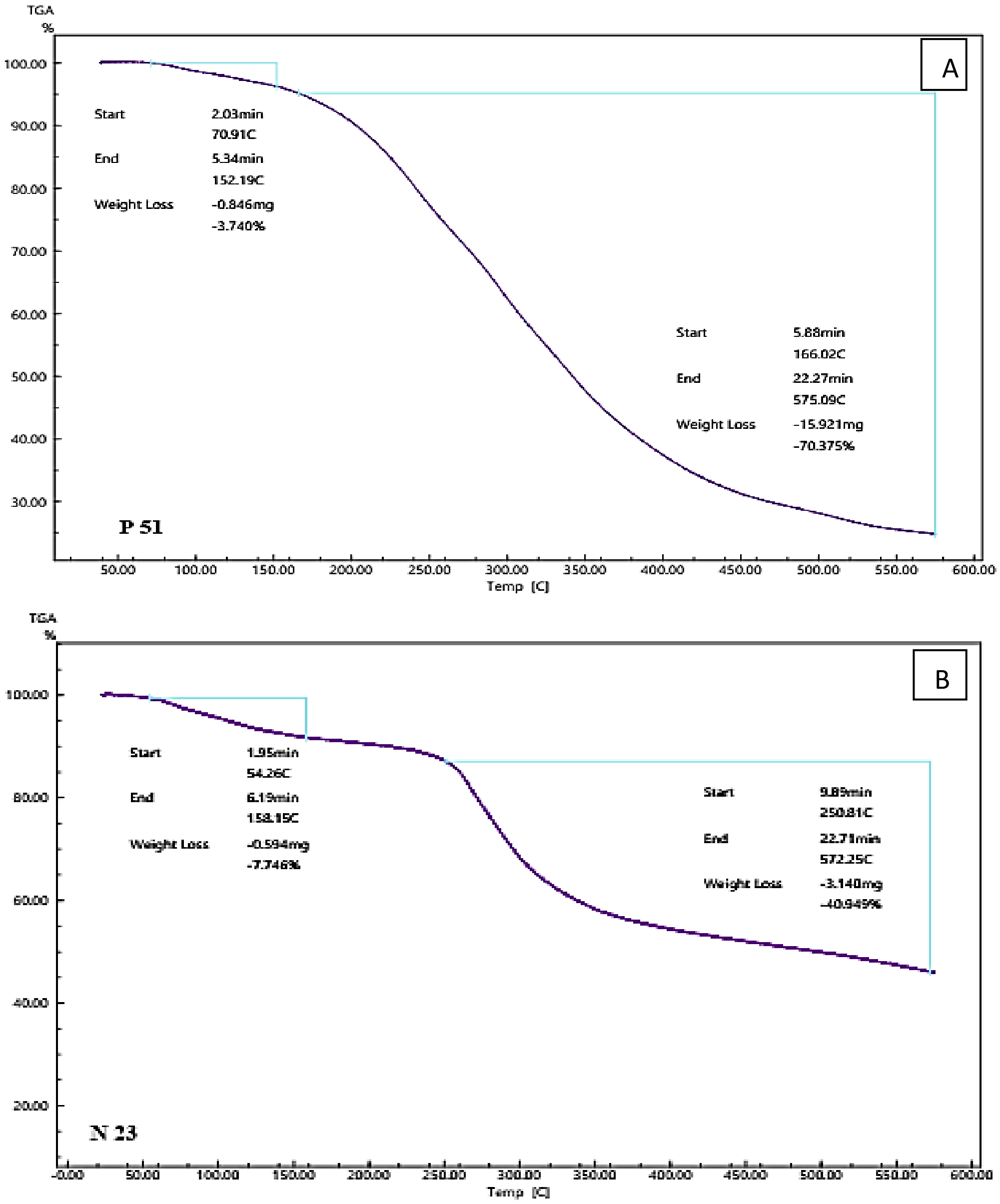


Figure (7) shows the thermogravimetric analysis (TGA) of (A) the copolymer of indole and 1,8-naphthalene diamine, and (B) the copolymer of 1,8-naphthalene diamine and indole combined with MWNTs-COOH

Figure (6)TGA analysis revealed that the copolymer of 2-aminophenol and 8-hydroxyquinoline undergoes multi-step degradation with a total weight loss of 70% at 600 °C. In contrast, the nanocomposite with MWCNTs-COOH showed improved thermal stability, with a reduced total weight loss of 48%, indicating enhanced resistance due to nanotube incorporation. Figure (7) presents TGA data for the indole/1,8-naphthalenediamine copolymer and its nanocompositewith MWCNTs-COOH. Both samples showed two-step weight loss. The copolymer exhibited an initial 3.7% loss (70–152 °C) due to moisture and oligomer removal, followed by a major 75% loss (166–570 °C) attributed to decomposition of unreacted monomers and polymer chains. In contrast, the nanocomposite demonstrated enhanced thermal stability, with only 47% total weight loss. The delayed degradation and reduced loss are ascribed to the stabilizing interaction between the nanotubes and the polymer matrix.

3.1.3.X-ray diffraction patterns:

The XRD pattern of Figure (8), representing the copolymer of 2-aminophenol and 8-hydroxyquinoline, shows a main diffraction peak at 2θ ≈ 27.48°, along with weaker peaks at 2θ ≈ 24.64° and 32.24°. These reflections suggest a semi-crystalline nature of the copolymes n the nanocomposite, a distinct peak appears at 2θ ≈ 26.76°[18], corresponding to multi-walled carbon nanotubes (MWCNTs). Additionally, variations in peak intensity are observed, indicating potential interactions between the polymer matrix and the nanostructures. Figure (9), The XRD pattern of the indole/1,8-naphthalenediamine copolymer reveals a semi-crystalline structure with a dominant peak at 2θ = 27°, indicating a well-ordered crystalline arrangement. Secondary peaks at 21°, 24°, and 30° 2θ reflect minor crystalline domains. The use of interfacial polymerization promotes higher structural organization, explaining the observed crystallinity.

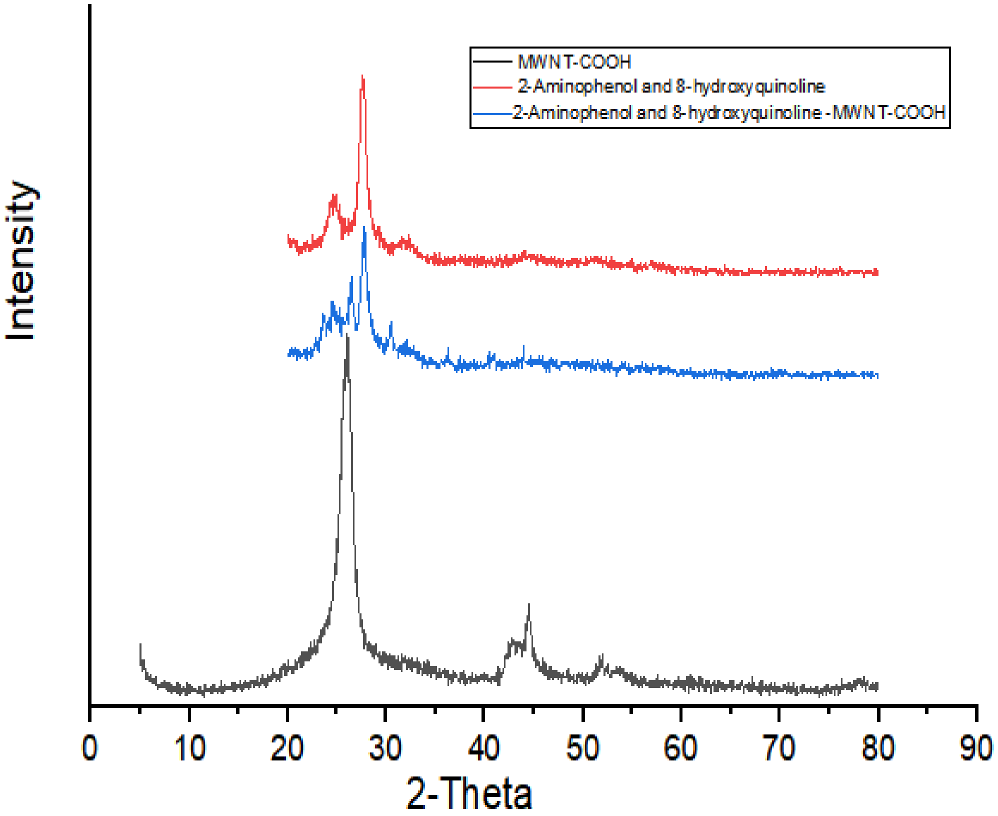


Figure (8) XRD2-Amino phenol and 8-hydroxyquinline : 2-Amino phenol and 8-hydroxyquinline- MWNT-COOH

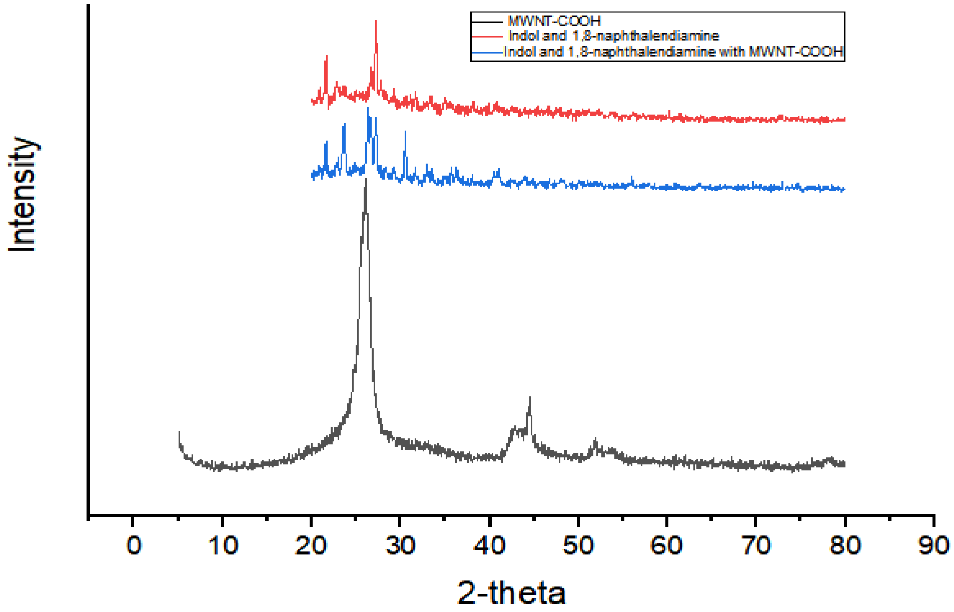


Figure (9) XRD1,8- Naphthalendiamine and indol : 1,8-naphthalendiamine and Indol- MWNT-COOH

3.1.4.SEM measurements:

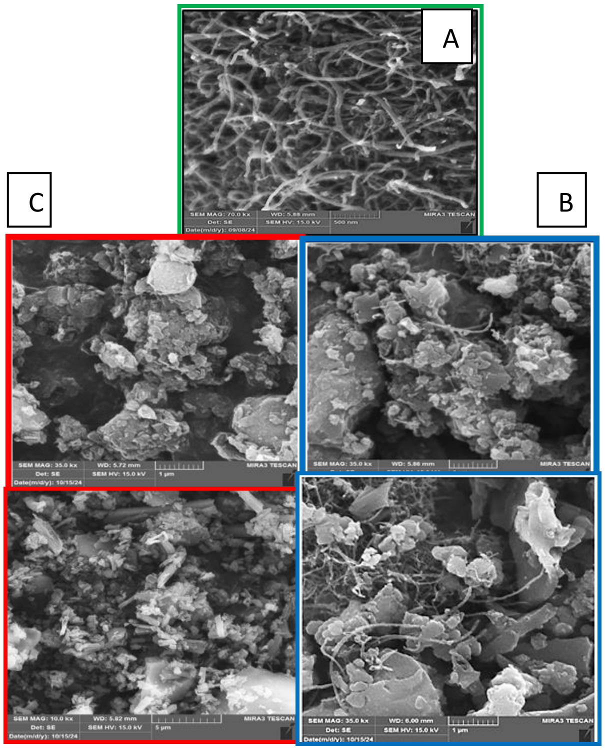


Figure (10): SEM images of (A) carbon nanotubes, (B) copolymer of 2-aminophenol with 8-hydroxyquinoline and (C) copolymer of indole with 1,8-naphthalenediamine

Figure (10) SEM analysis confirmed that the nanotubes preserved their cylindrical morphology after HNO_3/H_2SO_4 acid treatment, with no evidence of structural damage. This indicates effective surface functionalization while maintaining mechanical integrity, highlighting their suitability for chemically modified applications[19][20] . SEM (B) images revealed that the copolymer of 2-aminophenol and 8-hydroxyquinoline forms irregular aggregates with porous regions, indicating a polymeric network. Upon incorporation of carbon nanotubes, clear interaction and entanglement with the copolymer matrix were observed. SEM (C) analysis of the poly(1,8-naphthalenediamine-co-indole) copolymer revealed dense aggregates with sharp, rod- or needle-like crystalline structures. Upon incorporation of carbon nanotubes, clear interaction with the copolymer matrix was observed.

3.1.5. Electrical properties:

The I–V measurements of the polymer discs and their nanocomposites (1% MWCNTs-COOH) showed that the nanocomposites exhibited linear ohmic behavior, while some pure polymers showed non-linear characteristics. The improved conductivity in nanocomposites is attributed to the presence of MWCNTs enhancing charge transport.

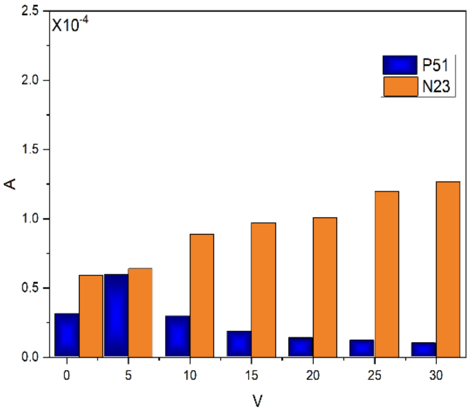


Figure (12): Current–voltage (I–V) characteristics of the copolymer (indole and 1,8-naphthalenediamine) (P51) and its nanocomposite with MWCNTs-COOH (N23).

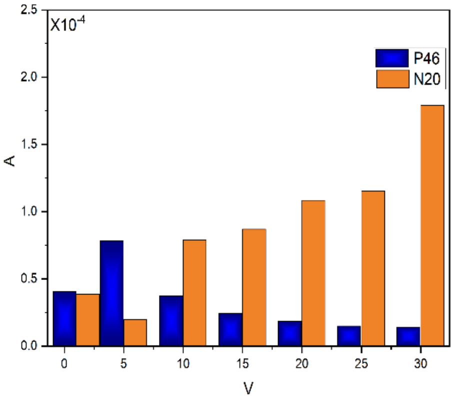


figure (11): Current–voltage (I–V) characteristics of the copolymer (2-aminophenol and 8-hydroxyquinoline) (P46) and its nanocomposite with MWCNTs-

4.Conclusion:

In this study, nanocomposite materials were successfully prepared by incorporating multi-walled carbon nanotubes functionalized with carboxyl groups (MWCNTs–COOH) into various copolymer matrices. The results demonstrated significant improvements in electrical conductivity, thermal stability, and surface morphology. These enhancements confirm that the functionalized carbon nanotubes effectively reinforce the polymer systems, making them promising candidates for advanced electronic and optoelectronic applications. Future studies are recommended to explore the effects of varying nanotube concentrations and to evaluate the performance of these materials under different environmental and operational conditions

Conflict of Interest:

The authors declare no conflict of interest.

Authors' Declaration

The authors hereby declare that the work presented in this article is original and that any liability for claims relating to the content of this article will be borne by them

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Conflicts of Interest Statement………………………………………………

Synthesis and Characterization of Conductive Copolymer/MWCNT Nanocomposite via Chemical and Interfacial Polymerization

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