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**Enhanced Electrical, Dielectric, and Optical Properties of Fe₂O₃-Decorated MWCNT/Epoxy
Nanocomposite Thin Films**

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Abstract

In this study, Fe₂O₃ nanoparticle-doped epoxy/multi-walled carbon nanotube (Fe₂O₃@CNT/EP) nanocomposite thin films were fabricated, and their structural, electrical, dielectric, and optical properties were investigated. EP/CNT nanocomposites containing different MWCNT concentrations (0.1, 0.5, 1.0, and 1.5 wt%) were prepared, followed by the incorporation of Fe₂O₃ nanoparticles into the EP/CNT matrix. The structural, chemical, and morphological characteristics of the prepared nanocomposites were investigated using X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM). The electrical and dielectric properties were evaluated using impedance spectroscopy over a broad frequency range, while the optical properties were investigated using UV–visible absorption and photoluminescence (PL) spectroscopy. The results demonstrated that the incorporation of MWCNTs significantly modified the electrical and dielectric properties of the epoxy matrix. Among the investigated CNT concentrations, the 1.0 wt% CNT-loaded EP/CNT nanocomposite exhibited a high capacitance of 8.1×10^{-9} F, dielectric constant of 71.5, and AC conductivity of 6.7×10^{-8} S m⁻¹. Further incorporation of Fe₂O₃ nanoparticles into the EP/CNT matrix resulted in a further enhancement of the electrical and dielectric responses. The optical absorption measurements revealed a decrease in the optical band gap from 4.15 eV for pristine EP to 3.86 eV for EP/CNT and 2.69 eV for Fe₂O₃@CNT/EP nanocomposites. The observed changes in the electrical, dielectric, and optical properties demonstrate the potential of these hybrid nanocomposite thin films for applications in electronic and optoelectronic devices.

Keywords: Nanocomposites; Thin films; AC conductivity; Dielectric constant; Band gap energy

Introduction.

Over the last few decades, researchers have devoted considerable effort to developing new materials with high strength- and stiffness-to-weight ratios [1, 2]. A major breakthrough in this field came with the introduction of nanocomposites, which consist of two or more different substances combined to obtain a novel set of properties compared with those of the individual constituents [3]. In general, nanocomposites consist of a matrix phase, which is usually a metal, ceramic, or polymer, and a reinforcement phase consisting of nanofillers. Polymer nanocomposites, in particular, comprise a polymer matrix and a dispersed nanomaterial phase, with at least one dimension of the dispersed material being less than 100 nm.

Polymer nanocomposites have been extensively investigated for a wide range of practical applications, including automobiles, biomedical devices, aerospace components, sporting goods, thermal and electrical insulators, sensors, and energy-harvesting systems [4, 5]. Common polymer matrices used in structural composites include epoxy resins, vinyl esters, and polyesters [6]. Among these, epoxy resin is a thermosetting polymer that has been extensively studied for a variety of real-world applications because of its high tensile strength, high stiffness, good thermal stability, and excellent chemical resistance. These properties make epoxy suitable for applications such as adhesives, coatings, semiconductor encapsulation, hardware components, electronic circuit boards, and composite matrices. Moreover, epoxy resins are widely used in the electronics industry for over-molding integrated circuits, transistors, and hybrid circuits, as well as for manufacturing printed circuit boards and diodes [7–9]. In the automobile industry, epoxy is also used as a structural adhesive, where it can partially replace conventional welding techniques [10].

However, the high crosslinking density and relatively low electrical conductivity of epoxy can limit its hardness, impact resistance, and functional performance, thereby restricting its applications in certain advanced technologies [11]. To overcome these limitations, various nanofillers, including metallic, ceramic, and carbon-based nanomaterials, have been incorporated into polymer matrices to improve their mechanical, thermal, electrical, optical, and flame-retardant properties while

maintaining acceptable processability [12]. A wide variety of nanofillers are available for the fabrication of polymer nanocomposites, including carbon nanotubes (CNTs), graphene, quantum dots, ZnO nanowires, and nanoparticles of gold, silver, and silicon [13].

Among these nanofillers, CNTs and their derivatives have attracted considerable attention because of their exceptional electrical, thermal, and mechanical properties [14]. Carbon nanotubes are one-dimensional nanomaterials with diameters typically ranging from approximately 1 to 100 nm [15]. They can be broadly classified into two types: single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs). An SWCNT consists of a single graphene sheet rolled into a cylindrical structure, whereas an MWCNT consists of multiple concentric graphene layers arranged coaxially around a central hollow core. The adjacent layers in MWCNTs are held together primarily by van der Waals forces. The electrical nature of SWCNTs, whether metallic or semiconducting, is determined by their chirality [16]. Owing to their outstanding mechanical, thermal, and electrical properties, as well as their high aspect ratio and large surface-to-volume ratio, CNTs are considered promising materials for a wide range of applications [17].

CNT-based nanocomposites have been employed in fuel cells, energy-conversion components, porous filters, and automotive components for nanocoating applications. In addition, these nanocomposites have been utilized in composite membranes for gas-separation applications, abrasion-resistant coatings, optical devices, power-tool housings, mobile phones, and microelectronic devices [18].

Despite the outstanding properties of CNT/epoxy (EP) nanocomposites, their practical applications are often limited by the non-uniform dispersion of carbon nanofillers within the polymer matrix and weak interfacial bonding between the CNTs and epoxy matrix. These factors can adversely affect the overall physical and functional properties of the resulting polymer nanocomposites [11]. To further enhance the performance of EP/CNT nanocomposites, metal oxide nanoparticles can be incorporated into the composite system [19, 20]. The addition of metal oxide nanoparticles can

improve desirable properties, including electrical, optical, and mechanical characteristics, as well as magnetic actuation [21, 22].

Among various metal oxide nanoparticles, iron oxide nanoparticles (Fe_2O_3 NPs) have attracted significant research interest because of their distinctive electrical and magnetic properties. These nanoparticles have numerous potential applications in diverse technological fields, including nanoscale electronic components, high-density information storage, and sensing devices [23]. However, to the best of our knowledge, Fe_2O_3 -doped EP/CNT nanocomposite thin films with varying Fe_2O_3 concentrations have not been systematically investigated to date.

Peng Cheng et al. [24] investigated the electrical properties of EP/CNT nanocomposites containing Ag nanoparticles and compared them with those of EP/CNT nanocomposites without Ag nanoparticles. They observed that the electrical conductivity of the composites containing Ag@CNTs was higher than that of the EP/CNT nanocomposites. Alkandari et al. [25] investigated the optical properties of epoxy/MWCNT nanocomposites. Their results demonstrated that increasing the MWCNT content in the epoxy matrix enhanced the absorption intensity of the epoxy resin. Abolfazl Alizadeh et al. [26] comprehensively investigated the effects of MWCNT concentration on the AC and DC electrical conductivity of epoxy nanocomposites at room temperature. Their study revealed that the electrical properties of the nanocomposites varied significantly with changes in CNT concentration. Rasha Al-Bayer et al. [27] investigated the AC electrical and optical properties of epoxy–alumina (Al_2O_3) composites. The experimental results indicated that both the optical and electrical properties changed with increasing alumina content in the epoxy matrix. Heinz et al. [28] investigated the sensing capabilities of epoxy/MWCNT nanocomposites over a temperature range of 160 °C. The resulting temperature sensor exhibited excellent stability and good sensitivity.

An extensive analysis of the relevant literature indicates that the electrical, optical, and sensing properties of EP/CNT nanocomposites have been widely investigated. However, the combined effects of Fe_2O_3 incorporation into EP/CNT nanocomposites, particularly in thin-film form, remain

insufficiently explored. Therefore, in the present study, EP/CNT and $\text{Fe}_2\text{O}_3@\text{CNT}/\text{EP}$ nanocomposites were synthesized, and their corresponding thin films were fabricated. The AC electrical and dielectric properties of the prepared EP/CNT and $\text{Fe}_2\text{O}_3@\text{CNT}/\text{EP}$ nanocomposite thin films were investigated using impedance spectroscopy. In addition, their optical properties were studied by determining the optical band gap using UV–visible spectroscopy. The obtained optical results were further compared with photoluminescence (PL) measurements to gain a better understanding of the electronic and optical characteristics of the prepared nanocomposite thin films.

2. Experimental

2.1. Materials and reagents

Multi-Walled Carbon Nanotubes ($\geq 99.99\%$) with outer diameter < 15 nm and length of 10-20 μm , Iron Oxide (Fe_2O_3) NPs ($\geq 95.95\%$), and Ethanol ($\geq 99.45\%$), were purchased from Sigma Aldrich. Diglycidyle ether of bisphenol A epoxy (DGEBA, EPON 828 Miller-Stephenson Chemical Co. Inc.) was used as the matrix.

2.2 Preparation of EP/CNTs and Fe_2O_3 @CNTs/EP Composites

Solutions of neat epoxy (EP) and epoxy/carbon nanotube (EP/CNTs) composites with different CNT concentrations (0.1 wt%, 0.5 wt%, 1.0 wt%, and 1.5 wt%) were prepared separately for the synthesis of EP/CNTs-based nanocomposites. The required amount of neat EP was dissolved in ethanol and stirred for 30 min at 100°C and 600 rpm. A few drops of the surfactant dimethylformamide (DMF) were added to the mixture to facilitate the breakdown and dispersion of the epoxy polymer chains. Similarly, CNT suspensions with different concentrations were prepared separately in ethanol. Each CNT suspension was subjected to sonication for 8 h, followed by stirring for 2 h at 60°C and 600 rpm, to achieve a homogeneous suspension and ensure the uniform dispersion of CNTs. Each CNT suspension was then individually mixed with the EP solution to obtain the desired EP/CNTs compositions. The resulting mixtures were stirred for 2 h at 80°C and 600 rpm to ensure proper mixing and dispersion. The Fe_2O_3 nanoparticle suspension was prepared using a similar procedure. Finally, an appropriate amount of Fe_2O_3 nanoparticles was incorporated into the EP/CNTs mixture to prepare the Fe_2O_3 @CNTs/EP nanocomposite solution. A comprehensive schematic representation of the synthesis process for Fe_2O_3 @CNTs/EP and EP/CNTs nanocomposites is presented in Fig. 1.

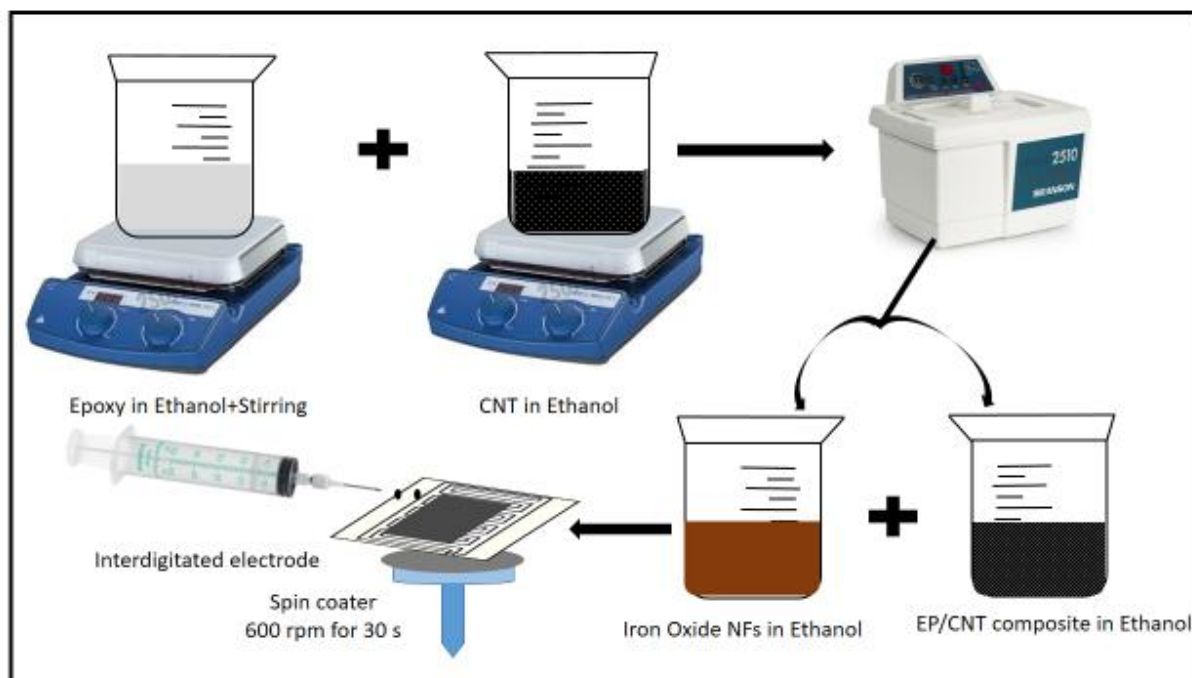


Fig. 1. Schematic illustration of the fabrication of EP, EP/CNT and Fe@CNT nanocomposites thin films.

2.3. Characterization

The samples were characterized using a PANalytical Pro X-ray diffractometer equipped with Cu-K α radiation ($\lambda = 1.541 \text{ \AA}$) over a 2θ scanning range of 10° – 80° at a scanning rate of $1^\circ/\text{min}$ for structural analysis. Fourier-transform infrared (FTIR) spectroscopy was performed using a PerkinElmer Spectrum 2 FTIR spectrometer equipped with a UATR-2 accessory in the wavenumber range of 400 – 4000 cm^{-1} to investigate the different vibrational modes present in the composites. The surface morphology of the fabricated samples was examined using a scanning electron microscope (SEM; JEOL JSM-5910, PerkinElmer, USA). Photoluminescence (PL) spectroscopy was carried out using a PerkinElmer LS45 photoluminescence spectrometer. A UV–visible spectrometer (PerkinElmer Lambda 11) was used to investigate the changes in the absorbance of the composite solutions.

2.4. Thin film device fabrication

A clean ceramic substrate measuring $14 \times 7 \text{ mm}^2$ was used to fabricate thin-film devices based on pure EP, EP/CNTs, and Fe₂O₃@CNT/EP nanocomposites. Interdigitated electrodes (IDEs) were

fabricated on the substrate. As shown in Fig. 2(a, b), each electrode had an approximate width of 0.2 mm and consisted of five interdigitated fingers. A known amount of each sample was dissolved or dispersed in the appropriate solvent to prepare the required solution or suspension for electrical characterization. After preparing the solution or suspension for each sample, approximately 50 μL was deposited onto the interdigitated electrode using a spin coater operated at a rotational speed of 600 rpm. The fabricated devices were subsequently dried in an oven at 80 $^{\circ}\text{C}$ for 1 h to ensure good adhesion and electrical contact while removing the solvent from the deposited film.

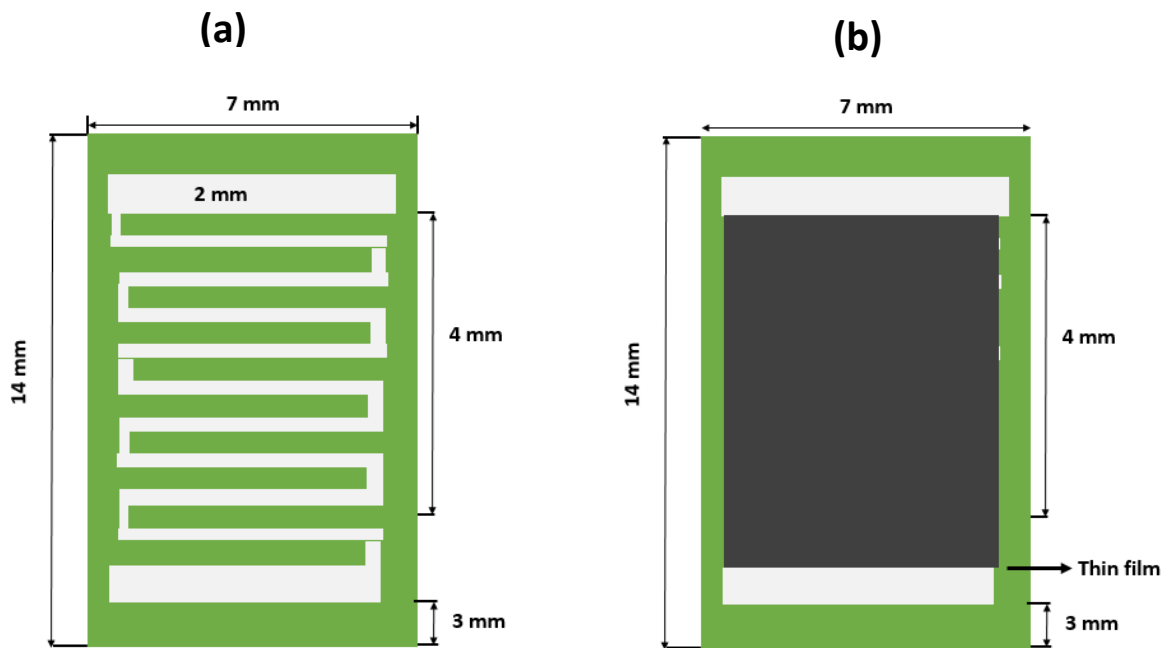


Fig. 2 (a, b). Schematic representation of the device structure.

3. Results and Discussion

3.1. XRD Analysis

X-ray diffractometer (PANalytical—X'Pert³) with Cu-K α radiation ($\lambda = 1.541 \text{ \AA}$) was used to examine the crystallinity of the fabricated samples within a 2θ scan range of 10° – 80° at scanning rate of $1^\circ/\text{min}$. Fig. 3 (a, b) depicts the XRD pattern of Pure EP and pristine MWCNTs. Since there was no discernible peak in the case of EP, the matrix was amorphous. The two intense peaks at about angle $2\theta = 26.3^\circ$, and 42.2° reflection of (002) (100) crystal planes respectively and well consistent with standard JCPDF card# 41-1487, confirmed the single phase of pristine MWCNTs. Likewise, the intense peaks at $2\theta = 31.2^\circ$ (2 2 0), 35.5° (3 1 1), 43.2° (4 0 0), 57.3° (4 2 2), and 63.01° (4 2 1) show in Fig. 3 (c), confirms the crystalline γ -phase of Fe_2O_3 NPs which matched with JCPDF card# 39-1346. Fig. 3(d) illustrates the XRD pattern of Fe@CNT/EP nanocomposites which demonstrates that the crystallinity of the prepared nanocomposites don't disturbed during the preparation.

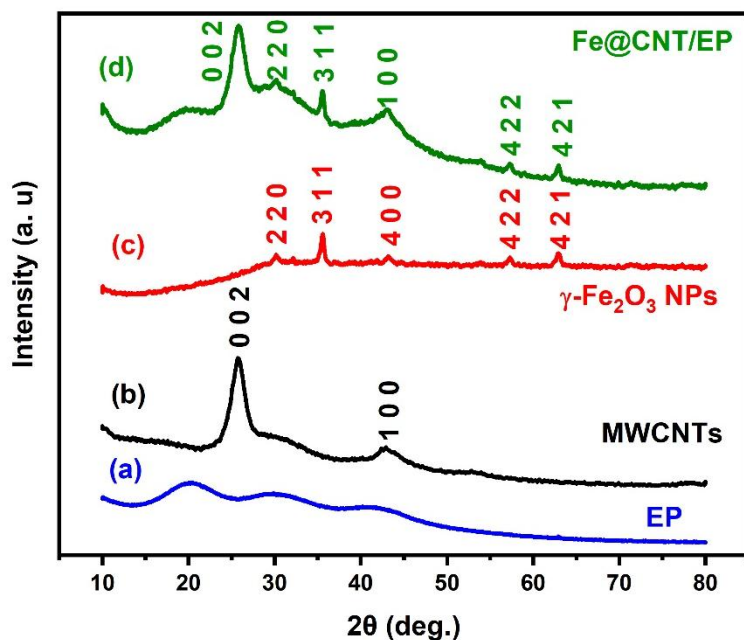


Fig. 3. XRD pattern of (a) EP (b) MWCNTs (c) Fe_2O_3 NPs and (d) Fe@CNT/EP nanocomposites.

3.2. FTIR Analysis

Fourier-transform infrared (FTIR) spectroscopy (PerkinElmer Spectrum 2 FTIR spectrometer equipped with a UATR-2 accessory) was used to investigate the presence of various chemical bonds and vibrational modes in the synthesized materials. The FTIR spectra of Fe₂O₃ nanoparticles (NPs), EP, EP/CNTs, and Fe₂O₃@CNTs/EP nanocomposites are presented in Fig. 4(a–d). FTIR analysis was performed to identify the functional groups and investigate changes in the bonding environment associated with the incorporation of CNTs and Fe₂O₃ into the epoxy matrix. When the material is exposed to infrared radiation, molecular vibrations associated with changes in dipole moments occur, resulting in characteristic absorption bands in the FTIR spectrum. These absorption bands can therefore be used to identify the chemical bonds and functional groups present in the material. As shown in Fig. 4(a, b), the FTIR spectra of pristine EP and EP/CNTs exhibit similar behavior, with nearly identical characteristic absorption peaks. The absorption peak observed at 2920 cm⁻¹ is attributed to the C–H stretching vibration of the methylene groups. The absorption band at 1725 cm⁻¹ corresponds to the stretching vibration of carbonyl (C=O) groups. The absorption peaks observed at 1446 and 1264 cm⁻¹ are associated with C–H vibrations of the aromatic ring. Furthermore, the bands observed at 1013 and 874 cm⁻¹ can be attributed to the stretching vibration of C–O groups associated with ether bonds and the epoxide ring, respectively. Finally, the absorption peak at 700 cm⁻¹ is assigned to the out-of-plane C–H bending vibration of the substituted aromatic ring. The similarity and overlapping of the FTIR spectra of pristine EP and EP/CNTs, together with the XRD results, indicate the successful incorporation of CNTs into the epoxy matrix without significant alteration of the characteristic chemical structure [29]. The two absorption peaks observed at 1103 and 543 cm⁻¹ correspond to metal–oxygen vibrational modes, confirming the presence of the γ -Fe₂O₃ spinel structure, as shown in Fig. 4(c) [30]. Figure 4(d) presents the FTIR spectrum of the Fe₂O₃@CNTs/EP nanocomposite, which exhibits the characteristic absorption bands associated with the epoxy matrix, CNTs, and Fe₂O₃ nanoparticles. The presence of these characteristic peaks confirms the successful incorporation of Fe₂O₃@CNTs into the epoxy matrix.

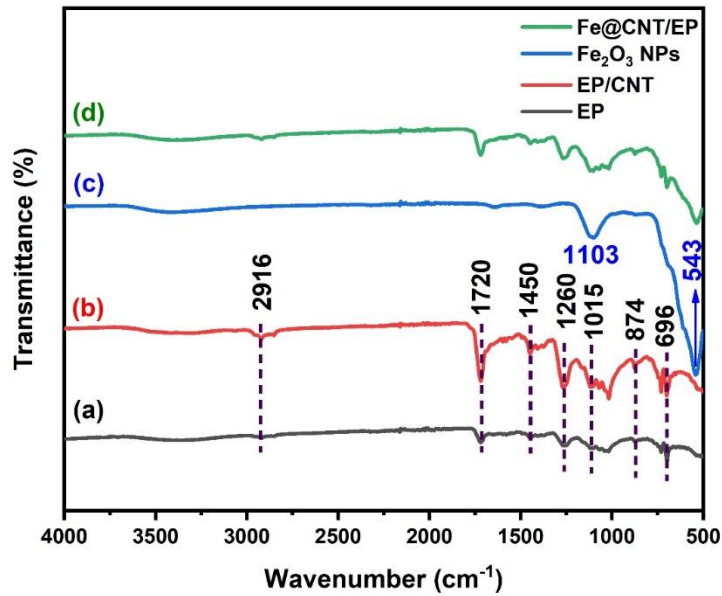


Fig. 4. FTIR spectra of (a) EP, (b) EP/CNT, (c) Fe₂O₃ NPs and (d) Fe@CNT/EP nanocomposites.

3.3 Morphological analysis

A scanning electron microscope (SEM; JEOL JSM-5910) was used to investigate the surface morphology of the synthesized materials. Figure 4.5(a–f) presents the SEM micrographs of neat EP, EP/CNT nanocomposites containing the optimum CNT concentration (1.0 wt%), and Fe₂O₃-doped EP/CNT nanocomposites at low and high magnifications. The SEM analysis was performed to investigate the microscopic morphology and surface interactions in the EP/CNT and Fe₂O₃-doped EP/CNT nanocomposites. Figures 4.5(a, b) show the relatively smooth surface morphology of neat EP. Figures 4.5(c, d) present the surface morphology of the EP/CNT nanocomposites. It can be observed that the CNTs form clusters and create fractured and rough surface features, which may be beneficial for sensing applications. Figures 4.5(e, f) show the SEM micrographs of the Fe₂O₃-doped EP/CNT nanocomposites. The observations indicate that the MWCNTs are intertwined with Fe₂O₃ nanoparticles, which may provide conductive pathways through the composite. In addition, this interconnected structure can contribute to conduction losses and enhance the reflection and scattering of electromagnetic radiation [31]. It is evident that the Fe₂O₃ nanoparticles exhibit a

predominantly spherical morphology with a non-uniform size distribution ranging from approximately 80 to 200 nm. Compared with neat EP and EP/CNT nanocomposites, the Fe₂O₃-doped EP/CNT nanocomposites exhibit a more interconnected and heterogeneous surface morphology. These characteristics suggest that Fe₂O₃-doped EP/CNT nanocomposites may be promising candidates for sensing and electrical applications.

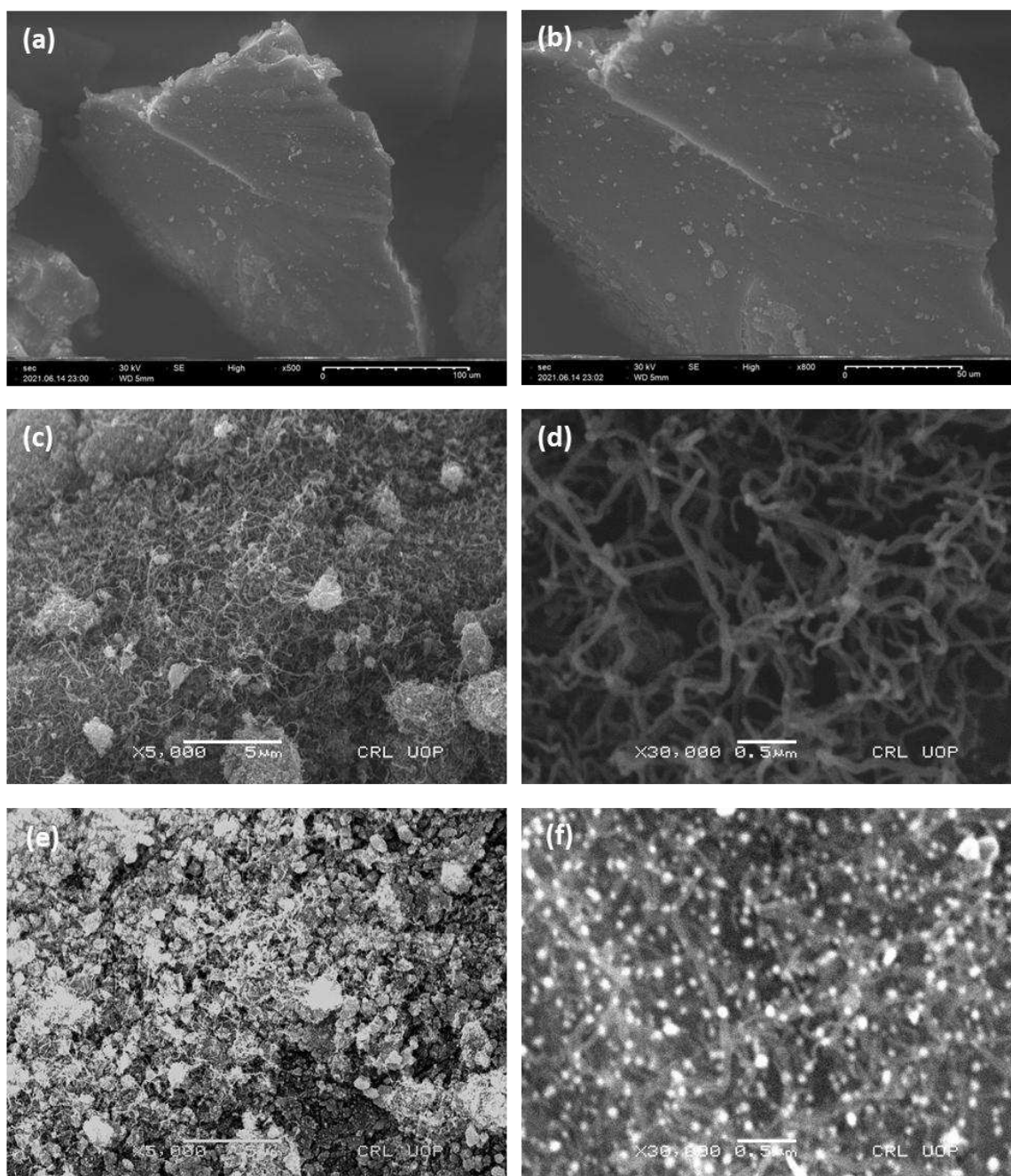


Fig. 5. SEM micrographs of (a, b) EP, (c, d) EP/CNT nanocomposites and, (e, f) Fe@CNT/EP nanocomposites.

3.4. AC electrical measurements

The electrical and dielectric properties of neat EP, EP/CNT nanocomposites, and Fe₂O₃@CNT/EP nanocomposites were investigated using an Agilent LCR meter (E4980A) at an applied voltage of 1 V. The AC signal frequency was varied from 25 Hz to 2 MHz. Figure 6 illustrates the AC conductivity spectra of neat EP, EP/CNT nanocomposites containing different CNT concentrations (0.1 wt%, 0.5 wt%, 1.0 wt%, and 1.5 wt%), and Fe₂O₃@CNT/EP nanocomposites. The AC electrical conductivity of all the prepared composite samples increased with increasing frequency. This behavior can be attributed to the formation of conductive networks between the CNTs and the EP matrix, which provide more effective pathways for charge transport throughout the polymer matrix. The migration of charge carriers may occur through electron tunneling and hopping mechanisms [32]. In addition, the conductivity increased with increasing frequency because the charge carriers have sufficient mobility to respond to the alternating electric field. The AC conductivity is directly related to the applied frequency and the imaginary part of the dielectric permittivity, as expressed by Eq. (1) [27]:

$$\sigma_{AC} = 2\pi f \epsilon_0 \epsilon'' \quad (1)$$

where ϵ_0 is the permittivity of free space, ϵ'' is the imaginary part of the relative dielectric permittivity, and f is the applied frequency.

It is evident from Fig. 6 that the AC conductivity increased for all composite samples compared with the neat EP matrix. Furthermore, the incorporation of CNTs into the EP matrix resulted in a progressive increase in electrical conductivity until the electrical percolation threshold was reached. The CNT concentration at which a continuous conductive network is established throughout the thin film is referred to as the percolation threshold. Below the critical concentration, the probability of forming a continuous conductive pathway is very low, whereas above the percolation threshold, the probability increases significantly. In the present study, the optimum CNT concentration was found to be 1.0 wt%, at which the composite exhibited the highest conductivity before the

subsequent decrease observed at higher CNT concentrations. At higher CNT loadings, excessive aggregation of CNTs can occur, which may disrupt the formation of an efficient conductive network and consequently reduce the electrical conductivity. Therefore, selecting an appropriate CNT concentration is crucial for achieving optimum electrical performance. Ounaies et al. [33] reported non-ohmic behavior between CNT loading and electrical conductivity beyond the percolation threshold, which was attributed to changes in the conductive network and charge-transport mechanisms. Electron transport can occur through hopping or tunneling from one nanotube to an adjacent nanotube when the inter-tube distance is sufficiently small, even when the nanotubes are not in direct physical contact. In contrast, classical percolation theory assumes the formation of a continuous network through interconnected conductive elements. Therefore, both tunneling/hopping and physical contact between CNTs can contribute to charge transport in CNT/polymer nanocomposites. To further enhance the AC electrical conductivity, Fe_2O_3 nanoparticles were incorporated into the EP/CNT nanocomposites containing the optimum CNT concentration of 1.0 wt%. As evident from Fig. 6, the electrical conductivity increased further following the incorporation of Fe_2O_3 nanoparticles. This enhancement may be attributed to the synergistic interaction between the Fe_2O_3 nanoparticles and CNTs, which facilitates charge transport within the composite [19]. The AC conductivity values of the fabricated samples are summarized in table 1.

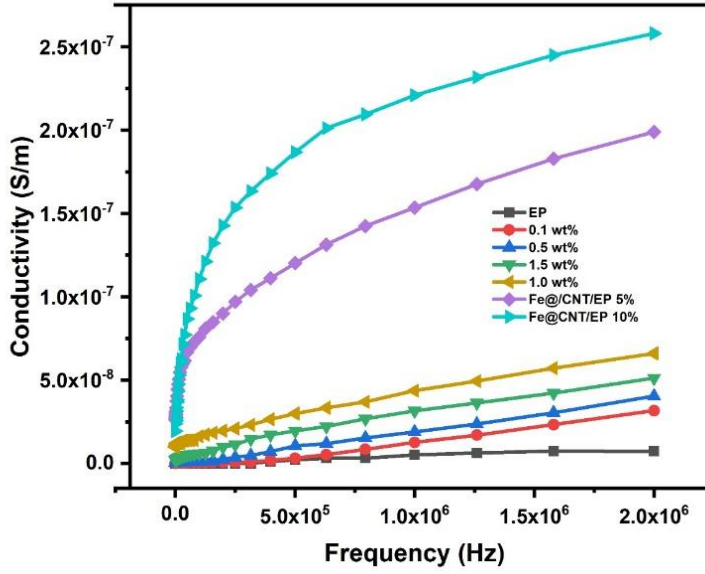


Fig. 6. AC conductivity spectra of EP, EP/CNTs and Fe@CNT/EP.

3.5. Dielectric properties of EP/CNT and Fe@CNTs/EP nanocomposites thin films

3.5.1 Real (ϵ') and imaginary parts (ϵ'') of dielectric constant (ϵ_r)

The real component (ϵ') of the complex dielectric constant represents the ability of a material to store electrical energy and is closely related to its degree of polarization [32, 34]. The imaginary component (ϵ'') of the complex dielectric constant represents the energy dissipation or loss within the material and is associated with dielectric loss [35]. Figures 7(a) and 7(b) show the real (ϵ') and imaginary (ϵ'') components, respectively, of the prepared samples as a function of frequency. The real dielectric constant was found to exhibit a relatively high value at low frequencies and gradually decrease with increasing frequency. Similarly, the imaginary dielectric constant exhibited a high value at low frequencies and decreased with increasing frequency. Figure 7(c) shows the variation of the dielectric constant of the prepared samples.

Dielectric materials generally exhibit four different polarization mechanisms, which occur over different frequency ranges and significantly influence their dielectric properties [36, 37]. These mechanisms include electronic, ionic, dipolar, and interfacial polarization. Space-charge polarization, also referred to as interfacial polarization, is predominantly observed at low

frequencies and generally occurs at surfaces, interfaces, and interphase boundaries. Ionic (or atomic) polarization generally occurs at relatively high frequencies, typically above 10^6 Hz, whereas dipolar (or orientational) polarization occurs at intermediate frequencies, typically around 10^4 Hz, in materials containing permanent dipole moments [38].

The dielectric constant is relatively high at low frequencies because of the accumulation of charges at interfaces and interphase boundaries. These accumulated charges require a relatively long relaxation time to respond to the applied electric field, resulting in enhanced polarization. However, at higher frequencies, the dielectric constant decreases because the electric dipoles do not have sufficient time to reorient themselves in response to the rapidly alternating electric field.

In the case of EP/CNT nanocomposites, the significant difference in electrical conductivity between the insulating EP matrix and the highly conductive CNTs promotes interfacial polarization. The mobile charge carriers accumulate at the interfaces between the EP matrix and CNTs, resulting in enhanced space-charge polarization. In addition, the incorporation of CNTs into the EP matrix can lead to the formation of nanocapacitors, in which adjacent conducting MWCNTs are separated by a thin insulating layer of the polymer matrix. The formation of these nanocapacitive structures contributes to an increase in capacitance, real permittivity, and dielectric constant.

To further enhance these dielectric properties, Fe_2O_3 nanoparticles were incorporated into the EP/CNT nanocomposites. The resulting $\text{Fe}_2\text{O}_3@\text{CNTs}/\text{EP}$ nanocomposites exhibited improved dielectric characteristics. This enhancement can be attributed to the reduction in the effective distance between conductive CNT layers and the resulting increase in capacitance. Since the dielectric constant is directly related to the capacitance, as expressed by Eq. (2) [39], an increase in capacitance leads to an increase in the dielectric constant:

$$\varepsilon = Cd/(A\varepsilon_0) \quad (2)$$

where C is the capacitance, d is the thickness of the thin film, ε_0 is the permittivity of free space, and A is the effective area of the sample under investigation.

3.5.2 Dielectric loss ($\tan\delta$)

Dielectric loss, commonly expressed in terms of the dissipation factor ($\tan\delta$), is an important parameter in energy-storage applications because it determines the amount of electrical energy dissipated within a material [40]. Figure 6(e) shows the variation of $\tan\delta$ for neat EP, EP/CNT nanocomposites with different CNT concentrations, and $\text{Fe}_2\text{O}_3@\text{CNTs}/\text{EP}$ nanocomposites. It was observed that $\tan\delta$ exhibited a relatively high value at a CNT concentration of 1.0 wt% and increased further with the incorporation of Fe_2O_3 nanoparticles into the EP/CNT nanocomposites.

Because $\tan\delta$ is directly related to the real and imaginary components of the complex dielectric constant, as expressed by Eq. (3), a similar trend was observed for the imaginary component of the dielectric constant. The graph shows that $\tan\delta$ exhibits relatively high values at low frequencies because of the predominant polarization effects. At higher frequencies, $\tan\delta$ becomes approximately constant, which can be attributed to the reduced contribution of polarization mechanisms at higher frequencies [41]. Generally, $\tan\delta$ is associated with dielectric and ohmic losses resulting from the movement of charge carriers within the material. Therefore, a higher dissipation factor is generally associated with increased electrical conductivity and greater energy loss. The maximum $\tan\delta$ value observed for the $\text{Fe}_2\text{O}_3@\text{CNTs}/\text{EP}$ nanocomposite may be attributed to the formation of an interconnected network between the Fe_2O_3 nanoparticles and CNTs within the EP matrix. This interconnected network can provide additional pathways for charge transport through the composite, thereby increasing its electrical losses.

The dissipation factor can be determined using Eq. (3)

$$\tan\delta = \frac{\varepsilon''}{\varepsilon'} \quad (3)$$

Where ε'' is the imaginary part and ε' real part of the complex dielectric constant.

3.5.3. Capacitance (Cp)

The capacitance of fabricated samples was measured at voltage 1 v through LCR meter as illustrated in fig. 7 (d). It is observed that the capacitance of all nanocomposites increases at low frequencies and becomes low at higher frequencies. This behavior was also observed in Fig. 7 (c) because capacitance is directly related with the dielectric constant as represented by eq. (4).

$$C = \frac{A\epsilon_0\epsilon}{d} \quad (4)$$

The capacitance values of EP, EP/CNT and Fe@CNT are given in table 1.

3.5.4. Complex Impedance spectroscopy (CIS) of Epoxy/CNT nanocomposites

Complex Impedance spectroscopy (CIS) is a versatile technique used for characterizing the electrical properties of materials and investigating their interface with electrodes ⁴². Plotting the resulting response on a complex plane shows a series of semicircles that correspond to the contributions from the bulk, grain boundaries, and interfaces ^{43, 44}. Complex parameters such as complex permittivity (ϵ^*), complex modulus (M^*), complex admittance (Y^*), complex impedance (Z^*), and dielectric loss ($\tan \delta$) are frequently used to describe the electrical characteristics of a material. The following relations establish the relationship between these frequency-dependent characteristics.

$$Z^*(\omega) = Z' - jZ'' = R_s - \frac{j}{\omega C_s} = \frac{1}{j\omega C_0 \epsilon^*} \quad (5)$$

$$Y^*(\omega) = Y' - jY'' = \frac{1}{\omega R_p} - j\omega C_p = j\omega C_0 \epsilon^* = \frac{1}{Z^*}, \quad (6)$$

$$M^*(\omega) = M' - jM'' = \frac{1}{\epsilon^*} = j\omega C_0 Z^* : (j = \sqrt{-1}), \quad (7)$$

$$\epsilon^*(\omega) = \epsilon' - j\epsilon'', \quad (8)$$

$$\tan(\delta) = \frac{Z'}{Z''} = \frac{Y'}{Y''} = \frac{M''}{M'} = \frac{\epsilon''}{\epsilon'}, \quad (9)$$

Where R_s and R_p represent the series and parallel resistances respectively, C_p and C_s are the parallel and series capacitances respectively, C_0 denote the geometrical capacitance, Z' , M' , Y' , ϵ'

and Z'' , M'' , Y'' , ε'' , Z'' show the real and imaginary parts of the impedance, modulus, admittance, and permittivity respectively. Fig. 7 (f) and (g) depict the spectroscopic plots of the real and imaginary components of the complex impedance (i.e. $\log f$ vs. Z' and $\log f$ vs. Z''). Fig. 7 (f) shows the variation of Z' with $\log$ of frequency for pure EP, EP/CNT at selected CNT concentrations, and Fe@CNT/EP (5 wt% and 10 wt%). It is cleared that Z' decreases for all CNT concentrations as well as for iron oxide NPs doped EP/CNT nanocomposites with the increase in frequency which indicates that AC conductivity rises with an increase in frequency. In addition the Z' values coincide at higher frequencies for all samples, which suggests that space charges may be released and decrease the materials barrier properties. Fig. 7 (g) shows the spectroscopic plots of the imaginary part of the complex impedance Z'' i.e. (Z'' vs. $\log f$). These plots provide information regarding the most resistive region in the sample. From these plots, R and C can be measured by taking the maximum value of Z'' using peak height in accordance with the relation, $R_{gb} = 2Z''$ and $(2\pi f_{max})RC = 1$, respectively. The plots show that the values of Z''_{max} shifts towards higher frequencies with increasing CNT concentrations and by incorporation of iron oxide NPs into the EP matrix. The corresponding observations suggested the occurrence of relaxation phenomenon in the EP/CNT nanocomposites and Fe@CNT/EP nanocomposites. Fig. 7 (h) depicts the Nyquist or Cole-Cole plots ($Z' vs Z''$) taken over a wide range of frequency (10 Hz to 1 MHz) of Pure EP, EP/CNTs at various CNT concentrations (0.1 wt%, 0.5 wt%, 1 wt% and 1.5 wt%) and Fe@CNT/EP (5 wt% and 10 wt%). These plots consist of arcs or semicircles representing an electro-active region with a related relaxation time, $\tau=RC$, where, R, C, and τ are the resistance, capacitance, and relaxation time of the charge carriers, respectively. The arrow indicates the increase in ω . The depressed semicircle represents the bulk nature while the enlarged semicircle represents the grain boundary nature in sample ⁴⁵. This result suggests, that grain boundary is more resistive as compared to grain. The diameter of each arc exhibited a decreasing trend with changing composition, showing that the resistivity of the sample decreases with increasing the percentage of CNTs in the compositions which corroborated with Fig. 6. This behavior arise due to the

incorporation of CNTs content in EP matrix up to the percolation limit which provide different path-ways for charge carriers. Furthermore, by addition of iron oxide NPs in to EP/CNTs composite reduces the resistance (Z'). On the other hand the capacitance increases because of the inverse relation with resistance. It is observed that the optimum CNTs concentration found to be 1 wt% which is the percolation threshold in the present study. The percolation threshold is already defined in the ac measurements section. From impedance spectroscopy measurements, it is possible to define the percolation threshold of the nanocomposites^{46, 47}.

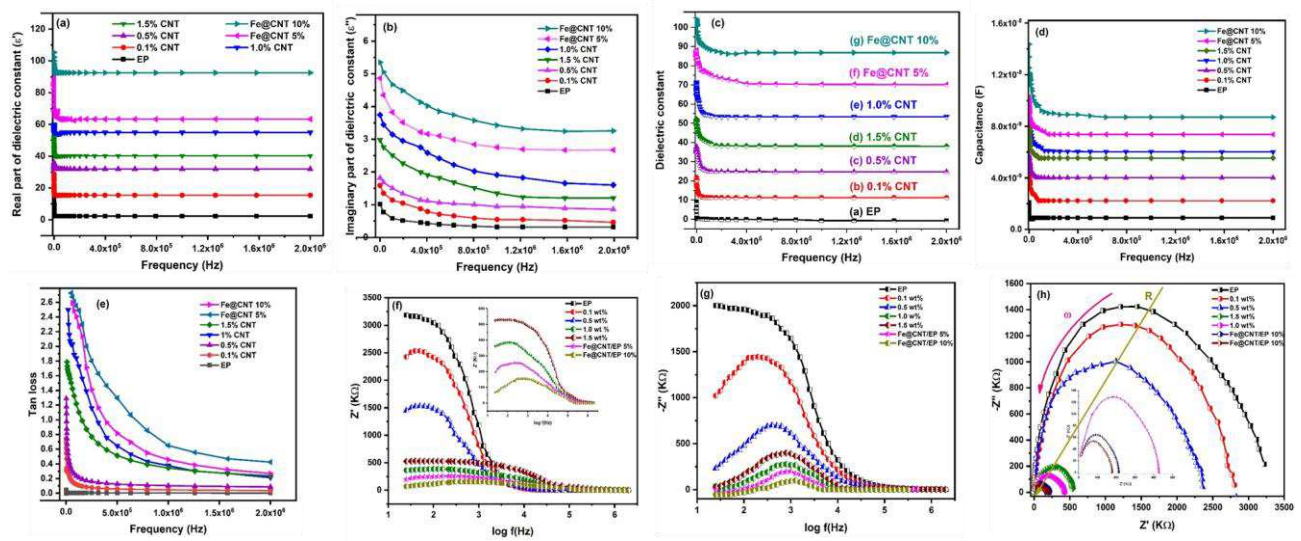


Fig. 7. (a) Real part of Complex dielectric constant (b) Imaginary part of dielectric constant (c) dielectric constant (d) Tan loss (e) capacitance (f) variation of real part as a function of frequency (g) variation of the imaginary part as a function of frequency (h) Nyquist or Cole-Cole plots of EP, EP/CNTs and Fe@CNT nanocomposites.

3.6. UV Visible spectroscopy

The absorption spectra of EP and EP/CNT nanocomposites containing different MWCNT concentrations, i.e., 0.1 wt%, 0.5 wt%, 1.0 wt%, and 1.5 wt%, in the wavelength range of 250–800 nm are shown in Fig. 8(a). It is observed that all the samples absorb UV light in the wavelength range of 250–300 nm, indicating that UV radiation can induce electronic transitions from the valence band to the conduction band. The cut-off wavelength, or absorption edge, of each spectrum was determined from the intersection of the perpendicular line drawn from the absorption peak with

the wavelength axis. It is clearly observed that the absorption intensity increases with increasing CNT concentration from 0.1 wt% to 1.0 wt% in the host matrix, accompanied by a red shift in the absorption edge. This behavior may be attributed to electronic interactions between the EP matrix and CNTs [48]. The absorption edge also shifted toward longer wavelengths following the incorporation of Fe₂O₃ nanoparticles into the EP/CNT composite containing the optimum CNT concentration of 1.0 wt%. This shift may be attributed to the quantum confinement effect [22]. The optical band gap is related to the photon energy ($h\nu$) and absorbance, as given by Eq. (10) [49].

$$\alpha h\nu = \beta (h\nu - E_g)^n \quad (10)$$

Where " α " is the absorption coefficient, β is a constant, E_g is the optical band gap and the exponent n shows the nature of electronic transition ($n=2$ for direct band semiconductor $n=1/2$ for indirect semiconductor). For the given samples best fitting on $n=1/2$. Further, the Lambert's formula (eq. 11) was used to calculate " α "⁵⁰.

$$I = I_0 \exp(-\alpha x) \quad (11)$$

$$\alpha = \frac{2.303}{x} \log \left(\frac{I}{I_0} \right) = \frac{2.303}{x} A \quad (12)$$

where I and I_0 are the transmitted and incident intensities respectively, and x is the sample thickness. In order to calculate the energy gap E_g , $(\alpha h\nu)^{1/n}$ is calculated and the plots of $(\alpha h\nu)^{1/n}$ versus incident, photon energy $h\nu$ is plotted for EP, EP/CNTs (0.1 -1.5 wt%), and Fe@CNT and is shown in Fig. 8 (b). The linear regions were obtained by these plots and extrapolating this linear region to the energy axis yields the energy band gap [48]. The energy band gap of the prepared samples is listed in Table 1. It is observed that the band gap of the composites decreases along with CNTs concentrations and by the incorporation of Fe NPs. This variation in band gap may arise due to the block or delays of the motion of charge carriers in host matrix [49]. The study revealed that EP/CNTs and Fe@CNT nanocomposites could be considered potential candidates for organic optoelectronic devices.

Table1. Comparison of various parameters of the present work with previously reported work

Samples	E_g (eV)	$\sigma_{ac}(Sm^{-1})$	$C_p(F)$	ϵ_r	$\tan\delta$	Ref.
Epoxy/CdS	2.63	-----	-----	-----	-----	²²
Epoxy/Al ₂ O ₃	3.45	125×10^{-6}	-----	5.5	0.03	²⁷
Al-doped ZnO/EP	3.75	-----	-----	-----	-----	⁵¹
MWCNTs/PANI/Fe ₃ O ₄	3.46	-----	-----	-----	-----	⁵²
CNT/PE/Cr ₂ O ₃	4.01	-----	-----	-----	-----	⁵³
MWNT/PDMS	-----	$5.25 \times 10^{-11} - 1.83 \times 10^{-8}$	$5.25 \times 10^{-11} - 1.83 \times 10^{-8}$	-----	-----	⁵⁴
EP	4.15	$0.1 \times 10^{-10} - 7.8 \times 10^{-9}$	$2.2 \times 10^{-9} - 7.5 \times 10^{-10}$	9.6-4.8	0.06	Present work
0.1 wt%	3.93	$1.2 \times 10^{-10} - 3.2 \times 10^{-8}$	$4.1 \times 10^{-9} - 2.1 \times 10^{-9}$	22.2-11.7	0.7	
0.5 wt%	3.92	$9.1 \times 10^{-10} - 5.2 \times 10^{-8}$	$5.8 \times 10^{-9} - 4.08 \times 10^{-9}$	37.5-25.3	1.2	
1.0 wt%	3.86	$1.2 \times 10^{-8} - 6.7 \times 10^{-8}$	$8.1 \times 10^{-9} - 6.1 \times 10^{-9}$	71.5-54.6	2.4	

1.5 wt%	3.92	$5.1 \times 10^{-9} - 5.2 \times 10^{-8}$	$7.9 \times 10^{-9} - 5.4 \times 10^{-9}$	52.4-38.4	1.7
Fe@CNT/E P 5%	2.69	$2.5 \times 10^{-8} - 1.99 \times 10^{-7}$	$1.1 \times 10^{-8} - 7.4 \times 10^{-9}$	88.2-70.5	2.5
Fe@CNT/E P10%	----	$2.6 \times 10^{-8} - 2.5 \times 10^{-7}$	$1.4 \times 10^{-8} - 8.8 \times 10^{-9}$	105.3-86	2.7

Table1. Comparison of various parameters of the present work with previously reported work

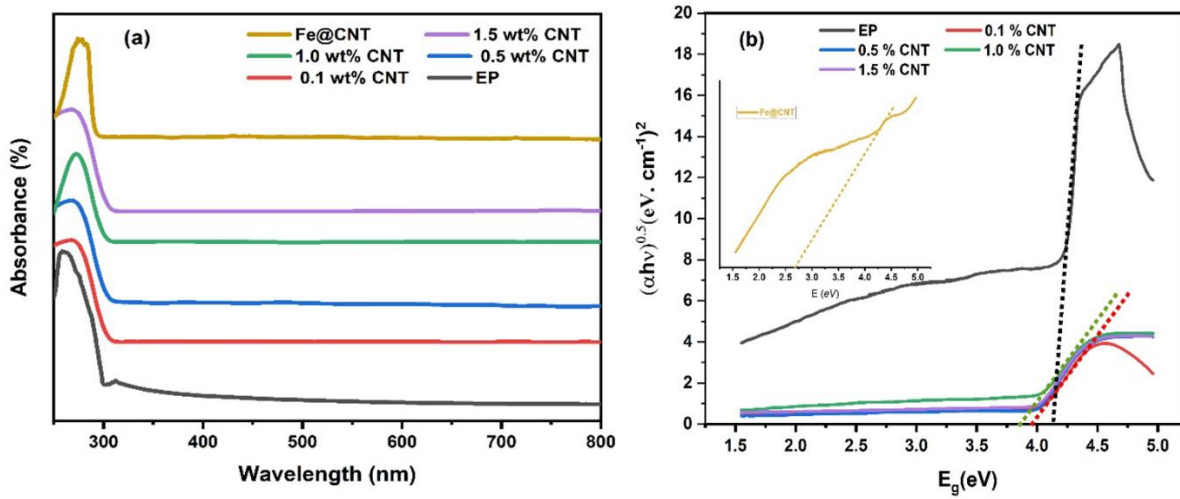


Fig. 8. (a) Optical absorption spectra and (b) Tauc's plots of EP, EP/CNTs and Fe@CNT.

3.7. Photoluminescence spectroscopy

Fig. 9 depicts the photoluminescence emission spectra of EP, EP/CNT with different CNT weight percent and Fe@CNT nanocomposites in the wavelength range from (250 nm-550 nm). The graph revealed qualitatively that increasing the CNT concentrations' emission intensity increases. In addition, the PL peak tends to shift longer wavelength along with increasing CNT content (0.1 to 1 wt%). Furthermore, more shifts toward longer wavelengths are obtained by the incorporation of Fe NPs in EP/CNT nanocomposites. The band gap energy E_g can also measure from PL spectra by using eq. (13).

$$E_g = h\nu = \frac{hc}{\lambda} = \frac{1240}{\lambda (nm)} eV \quad (13)$$

where h is plank's constant, c speed of light and λ is a wavelength at which maximum absorption occurs. The E_g was calculated as 4.09 eV, 3.98 eV, 3.97 eV, 4.03 eV, 3.96 eV and 3.2 eV for EP, 0.1 % CNT, 0.5 % CNT, 1.0 % CNT, 1.5 % CNT and Fe@CNT respectively. Our PL results, however, verified the absorbance measurements qualitatively.

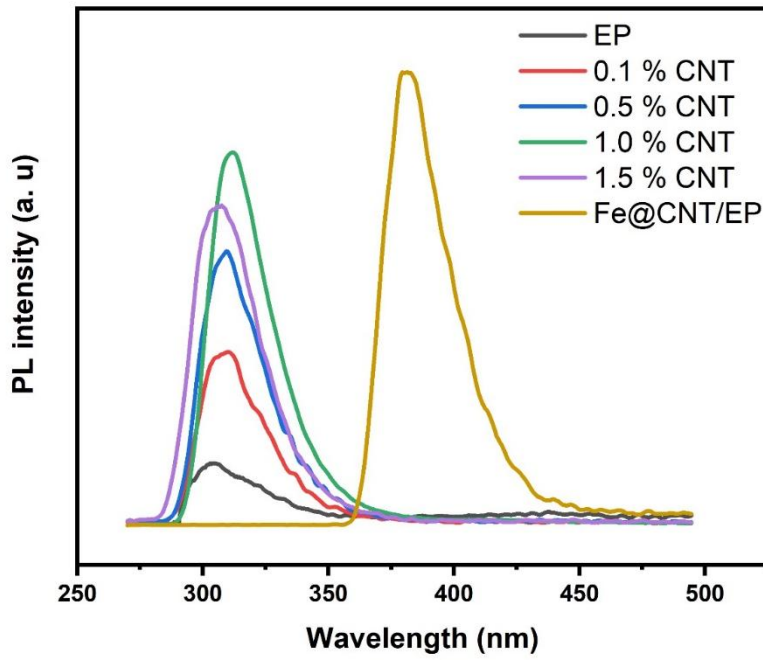


Fig. 9. PL emission spectra of EP, EP/CNTs and Fe@CNT nanocomposites.

3.8. Comparison of efficiency of the fabricated thin films

Table 1 compares and summarizes the results obtained in the present investigation with those reported in the published literature. The pure polymer matrix exhibits relatively poor optical and electrical properties. However, the incorporation of a small amount of CNT nanofillers significantly enhances these properties, which is in good agreement with previously reported results [reference]. Furthermore, the AC conductivity, dielectric constant, capacitance, and optical band gap of the Fe₂O₃@CNT/EP nanocomposites were measured and found to be significantly altered compared with those of the pristine polymer matrix. To the best of our knowledge, such a combination of

materials has not been previously reported in the literature. These results indicate that the developed nanocomposites have promising potential for applications in organic optoelectronic devices.

4. Conclusions

Pure EP, EP/CNT nanocomposite thin films containing different CNT concentrations (0.1 wt%, 0.5 wt%, 1.0 wt%, and 1.5 wt%), and Fe₂O₃@CNT/EP nanocomposite thin films were successfully fabricated using the spin-coating method. The fabricated samples were subsequently characterized using various characterization techniques. The XRD patterns revealed the polycrystalline nature of the nanocomposites, while FTIR analysis was used to investigate the crystallinity and vibrational modes of the prepared samples. The electrical, dielectric, and optical properties of the nanocomposites were also systematically investigated. It was observed that the electrical conductivity increased with increasing CNT concentration and further increased with the incorporation of a small amount of Fe₂O₃ nanoparticles. The optimum CNT concentration was found to be 1.0 wt%, which exhibited high AC conductivity ($\sigma_{AC} = 6.7 \times 10^{-8} \text{ S m}^{-1}$), dielectric constant ($\epsilon_r = 71.5$), and capacitance ($C_p = 8.1 \text{ nF}$). Furthermore, small amounts of Fe₂O₃ nanoparticles (5 wt% and 10 wt%) were incorporated into the optimized EP/CNT nanocomposite, resulting in further enhancement of the corresponding properties, with $\sigma_{AC} = 2.5 \times 10^{-7} \text{ S m}^{-1}$, $\epsilon_r = 105.3$, and $C_p = 14 \text{ nF}$. The impedance spectroscopy results showed that the resistance decreased with increasing CNT concentration and with the incorporation of Fe₂O₃ nanoparticles. These findings are consistent with the observed increases in AC conductivity, capacitance, and dielectric constant. In addition, the optical band-gap energies of pure EP, EP/CNT nanocomposites containing 0.1 wt%, 0.5 wt%, 1.0 wt%, and 1.5 wt% CNTs, and the Fe₂O₃@CNT/EP nanocomposite were found to be 4.15, 3.93, 3.92, 3.86, 3.92, and 2.69 eV, respectively. Overall, the results demonstrate that EP/CNT and Fe₂O₃@CNT/EP nanocomposites are promising materials for potential applications in microelectronic and optoelectronic devices.

CRedit authorship contribution statement:

Yousaf Ali Shah: Conceptualization, Methodology, Software, Data curation, Writing- Original draft preparation

Mutabar Shah: Supervision.

Afzal Khan: Visualization, Investigation.

Muhammad Habib: Software, Validation.

Khan Malook: Reviewing, Discussion, Editing.

Declaration of Competing Interest:

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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