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Studies on Structural, Optical, Thermal and Dielectric Properties of Polyaniline/ZnO Nanocomposites Synthesized by In-Situ Polymerization

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Abstract

Objectives: To use the polymer that conducts Ammonium Persulphate (APS) as an oxidizing agent during the in-situ polymerization process to create polyaniline (PANI) and PANI-ZnO nanocomposites. **Methods:** PANI-ZnO nanocomposite optical band gap has been investigated at room temperature for varying weight percentages, including 10%, 20%, 30%, and 40%. The ZnO hexagonal wurtzite crystal structure was validated by the XRD pattern. The Debye-Scherrer formula was used to calculate the particle size, which came out to be 38.27 nm. ZnO's distinctive peak, which can be seen in the obtained absorption spectra at 375 nm, is present in all weight percentages of the nanocomposites. The presence of peaks at 400 and 300 nm indicates that polyaniline has formed in the nanocomposites. **Findings:** Thermo Gravimetric Analysis (TGA) thermographs support the idea that thermal stability rises as the ZnO nanoparticle weight percentage in the composite increases. At room temperature, the dielectric characteristics of PANI and PANI-ZnO nanocomposites, including dielectric loss, dielectric constant, and AC conductivity, are studied as a function of frequency between 40 Hz – 5 MHz. **Novelty:** Multiple characteristic properties such as structural, optical, thermal, and dielectric properties have been studied on the Polyaniline/ZnO nanocomposites which are synthesized by in-situ polymerization technique for the first time.

Keywords: Polyaniline; ZnO; Nanocomposites; XRD; Dielectric Properties; TGA; UV-Vis

1 Introduction

The promising and unique electrical properties of intrinsically conducting polymers, such as energy storage devices⁽¹⁾, gas sensors⁽²⁾, electromagnetic interference shielding⁽³⁾, and electrochromic materials⁽⁴⁾, have gained the attention of material science researchers. Because polymeric materials have great potential for solid state devices, there is a growing desire to learn about electrical transport in these materials. Similar to this, conducting polymer composites have attracted great attention recently due to their many applications in a wide variety of electrical and electronic devices. Desired properties are produced by conducting polymer composites that contain the right amounts of one or more insulating materials. Despite the fact that many conducting polymers have been created to date, polyaniline is the most significant conducting polymer because of its high conductivity, low cost, ease of preparation, and good thermal and environmental stability⁽⁵⁾. It was later demonstrated that doping these polymers causes a significant change in their conductivity. Polyaniline has many useful applications, such as electrochromic devices, electrochemical super capacitors, batteries, biosensors, and gas sensors, due to its appealing electrochemical and physio-chemical characteristics. At room temperature⁽⁶⁾, ZnO, a semiconducting material, has a large excitation binding energy (60meV) and a direct wide band gap (3.37eV). ZnO is an n-type semiconductor with good photocatalytic activity that is non toxic. Although, ZnO is an unusual substance with a variety of characteristics, such as pyroelectric, semiconducting, and piezoelectric ones. As a result, there is a growing need for ZnO in a variety of applications these days, active materials in batteries⁽⁷⁾, photo biosensors⁽⁸⁾, optoelectronics⁽⁹⁾, and gas sensors⁽¹⁰⁾.

In the current work, aniline monomer was in-situ polymerized with ZnO to create PANI/ZnO nanocomposites. The nanocomposites with varying weight percentages are described. Thermal analysis, TGA, UV-Visible, and X-ray diffraction (XRD) have all been used to examine these composites. It was also investigated how these composites' AC conductivity changed with temperature. The purpose of this research is to dope ZnO in PANI components in order to produce a new noble material that can be used for gas sensing applications as well as a useful compound with valuable physical properties.

2 Methodology

2.1 Materials

The present study employed only Analytical Reagent (AR) grade chemicals, all of which were utilized without additional purification. The materials used in this study were 99.5% Monomer Aniline (Merck), Ammonium Persulphate (APS) (Merck) as Oxidant, ZnO Nanomaterials (Adnano Technology Pvt Ltd) Hydrochloric acid (HCl)(Merck), Acetone and demineralized water (Thomas Baker).

2.2 Polyaniline (PANI) Chemical Synthesis

The in-situ chemical oxidation polymerization technique was used to synthesize PANI. To create aniline hydrochloride, 0.1M aniline was combined with 1M hydrochloric acid and stirred for 15 minutes. Ammonium persulphate (0.1M), an oxidant, was added to this solution gradually drop-by-drop while being constantly stirred at 50°C for a full four hours to polymerize. To attain a consistent mass, the precipitate was filtered, cleaned in acetone and demineralized water, and then dried in an oven for a full day (24h). Polyaniline is synthesized in this manner.

2.3 Preparation of Polyaniline-ZnO nanocomposites

ZnO nanoparticles were present during the simple in-situ polymerization process used to create the PANI-ZnO nanocomposites. To create aniline hydrochloride, ZnO nanoparticles (10-40wt %) were added to 0.1M aniline that had been mixed with 1M HCl and stirred for 15 minutes. To maintain the ZnO uniformly suspended in the solution, ZnO nanoparticles of varying weight percentages were vigorously stirred in with the mass fraction of the previously mentioned solution. Ammonium persulphate (0.1M), an oxidant, was gradually added to this solution drop by drop while stirring constantly at 5°C for two hours. The polymerization was allowed to be processed for 4 to 6 hrs at temperatures 0°C to 5 °C. The reaction mixture was left in the dark room overnight to complete Polymerize and the precipitate was filtered. The nanocomposites were obtained by filtering and washing suspension with acetone and demineralized water repeatedly until the filtered solution became colorless and lastly, to attain a consistent mass, dried in an oven for a full day (24hrs). A fine powder was formed by grating the dried polymer. PANI-ZnO nanocomposites were created in this manner, containing different weight percentages of ZnO (10-40 wt %) in polyaniline.

2.4 Preparation of Pellets

Using an agate mortar pestle, the polyaniline powder, PANI-ZnO nanocomposites, and fine powder were crushed and ground very finely using acetone medium. After that, the powder was compressed into pellets with a 10 mm diameter and a thickness of between 1.8 and 2 mm using a hydraulic pressure of 100 MPa, and they were dried. After that, silver paste was applied to both pellet's faces to create electrical contact.

2.5 Characterization

The chemically synthesized samples underwent X-ray Diffraction (XRD) studies using a Rigaku Smart Lab SE, diffractometer using $\text{Cu-K}\alpha$ as radiation source of Wavelength $\lambda=1.54\text{\AA}$ in the range of 2° to $150^\circ(2\theta)$. Pellet preparation using KBr press MODEL M-15.Thermo-gravimetric Analyzer (TGA) studies using (Perkin Elmer- STA8000), UV-VIS Spectrophotometer studies using Labindia-UV3200. Dielectric studies were conducted utilizing an Agilent 4294A Precision Impedance Analyzer within the frequency range of 40Hz-5MHz.

3 Results and Discussion

3.1 Analysis of X-Ray Diffraction (XRD)

Figure 1 a & b display the XRD patterns of pure zinc oxide, PANI-ZnO (10-40 wt%) composites, and pure PANI, respectively. Using a Rigaku Smart Lab SE diffractometer and a $\text{Cu-K}\alpha$ radiation source, the synthesized sample's X-ray diffraction patterns were obtained. The wavelengths were measured in the range of 2° to $150^\circ(2\theta)$, with $\lambda=1.5460\text{\AA}$. Between 10° and 100° , the diffractograms were measured and reported in terms of 2θ . The characteristic peak, $2\theta=25.47^\circ$, is where the broad peak occurred, and it explains pure PANI is amorphous in nature. All of the tested samples' PANI peak positions matched the JCPDS Card No.00-053-1891 exactly. The dispersion from the periodicity at right angles to PANI chains was identified as the source of the broad diffraction peak along 25.47° , while the peaks prior to this angle can be regarded as periodically analogous to PANI chains. The presence of PANI and ZnO nanoparticles was validated by the XRD patterns of the composite samples (10–40 wt %). The polymeric nature of PANI and PANI/ZnO composites is confirmed by the broad peak that appears in all samples and is centered at $2\theta = 25.47^\circ$ with the plane of (003) (due to relatively less addition of ZnO nanoparticles into the PANI matrix). The high-intensity peak of PANI (1 0 1) in the composites was noticeably condensed, as shown in Figure 1, and the ZnO peaks in the composites sample became more intense as more ZnO nanoparticles were added. The PANI/ZnO peak positions were somewhat different from those of pure PANI; this shows that the composite was formed and that the addition of ZnO nanoparticles only caused a conformational change in the polymer backbone rather than a structural alteration. Using XRD patterns the ZnO crystallite size and lattice parameters are calculated using the Debye Scherer formula is 38.27 nm. In contrast, increasing the crystallite size increases the unit cell volume of the PANI, whereas increasing the crystallite size typically decreases the unit cell volumes of the metal oxides (ZnO).

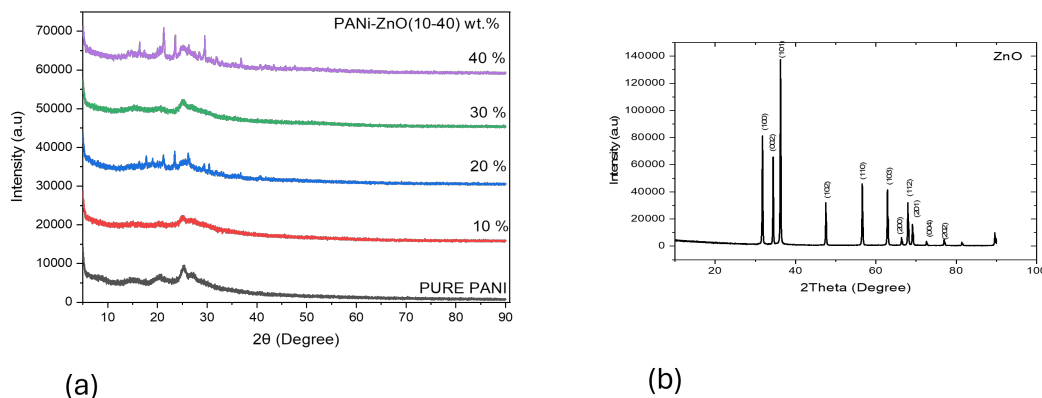


Fig 1. (a) PANI-ZnO nanocomposite of 10-40 wt% & (b) Pure ZnO nanoparticle XRD Pattern

It is clear from the above figure that the presence of ZnO nanoparticles in the composite sample is what causes the increase in crystallite size to be correlated with a decrease in unit cell volume. Thus, it is clear that ZnO nanoparticles have been well incorporated into the polymer matrix⁽¹¹⁾.

3.2 UV-Vis ible Spectroscopy Analysis

3.2.1 Absorbance

The prepared samples were subjected to UV-Visible absorption studies in this paper using a Shimadzu UV-Visible Spectrophotometer (Model 1800) and DMSO₄ (Dimethyl sulfoxide) as a solvent. From the **Tauc plot** energy gap of the prepared samples was determined.

Tauc relation is given by,

$$\alpha h\nu = A(h\nu - E_g)^n \quad (1)$$

where, “ E_g ” is the energy band gap, “ A ” Absorbance of the sample “ t ” i.e., $\alpha = A/t$.

“ α ” is called as Absorption coefficient and it depends on the value of absorbance

“ n ” depends on the procedure in which transition happens and for direct band gap semiconducting material $n=2$.

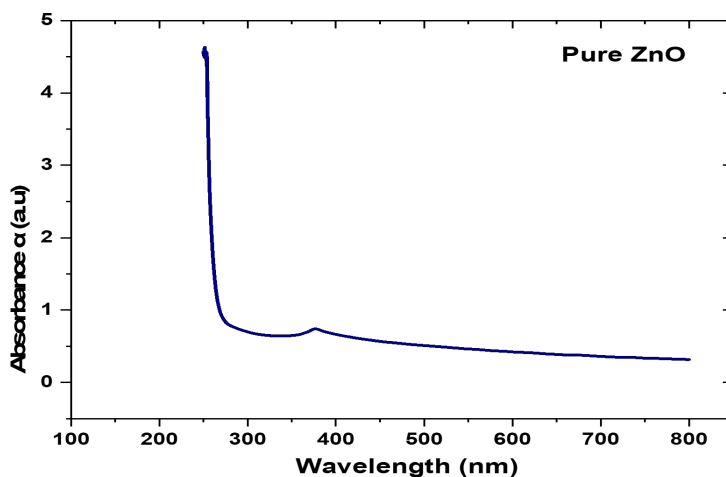


Fig 2. ZnO nanoparticles UV-Visible Absorbance Spectrum

The spectra of UV absorption of pure ZnO nanoparticles are displayed in Figure 2. The UV and visible spectrum (100-800 nm) were used to measure the absorption spectrum. In the UV-visible range, pure ZnO nanoparticles are transparent to optical transitions. The range of 300–400 nm contains the maximum absorption peak. The ZnO nanoparticles are found to have a higher excitation binding energy, which is consistent with the findings of Bouaine et al⁽¹²⁾. The direct transition of electrons in ZnO nanocrystals is responsible for a narrow absorption band that is visible at the characteristic wavelength of roughly 375 nm.

It can be seen from the UV-VIS spectra shown in Figure 3. That the neat PANI has wavelength peaks at 371, 432, and 609 nm. The peak at 371 nm wavelength, or the p-p* electronic transition, is caused by an electron moving from the polymeric chain's benzenoid's HOMO (Highest Occupied Molecular Orbit) to the quinoid's LUMO (Lowest Unoccupied Molecular Orbit). The excitation transition from p polaron quinoid to polaron benzenoid p* is responsible for the peaks at 432 and 609 nm in wavelength. With a very slight red shift, the PANI-ZnO nanocomposite material showed nearly identical peaks or bands. This slight change is the result of the interaction between pure PANI and ZnO, which is consistent with the findings of Manawwer Alam et al.⁽¹³⁾.

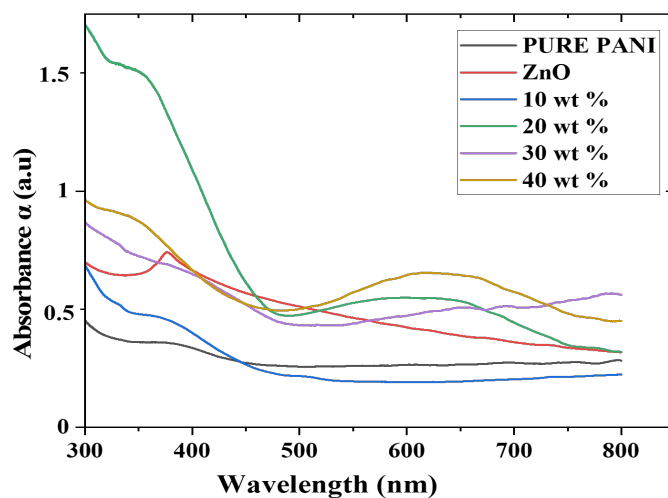


Fig 3. The UV-visible absorption spectra of ZnO and PANI-ZnO nanocomposites after preparation

3.2.2 Optical Absorption Coefficient and Optical Band Gap

Using Equation (1), the optical absorption coefficient α was calculated from the spectra. The absorbance (A) was used to calculate the absorption coefficient (α). The absorption coefficient (α) was calculated using the relation after reflection was considered.

$$\alpha(\nu) = 2.303 A/d \quad (2)$$

where d is the sample thickness and A is the absorbance

Solid state band structure can be understood through the study of optical absorption.

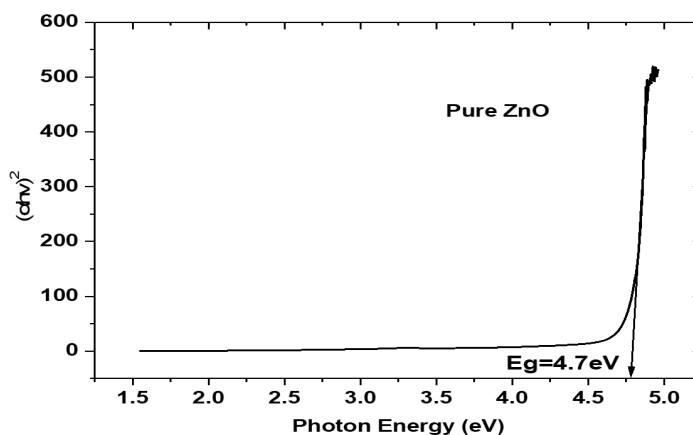


Fig 4. Pure ZnO nanoparticles Tauc plot

Figure 4 shows the plot of $(\alpha h\nu)^2$ versus Photon Energy (eV). The energy of the optical band gap of pure ZnO nanoparticles is found using an energy plot known as a Tauc plot. The intermediate linear region is displayed on the Tauc plot, and the energy band gap value can be obtained by extrapolating this region until it intersects the x-axis. Based on the figure, the energy band gap of pure ZnO nanoparticles is determined to be 4.7 eV.

3.3 Thermo Gravimetric Analysis (TGA)

3.3.1 Thermal Stability Analysis of PANI and PANI-ZnO Nanocomposites

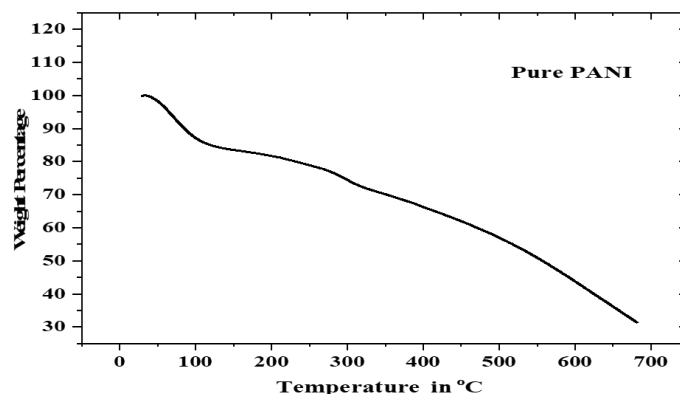


Fig 5. TG analysis of Pure PANI

The polyaniline (PANI) thermal behavior is depicted in Figure 5. In the PANI, there were three phases for losing weight. Within the temperature range of 30°C to 140°C, the experiment's first stage causes a 20 wt% loss of the sample total from the PANI chain. The breakdown of HCl acid and the simultaneous evolution of water in the temperature range of 140 °C to 286 °C are the causes of the PANI's second step weight loss. Approximately 10 wt % of the sample's total weight decomposes at this point. The third stage of PANI chain degradation occurs between 286 °C and 700 °C in temperature.

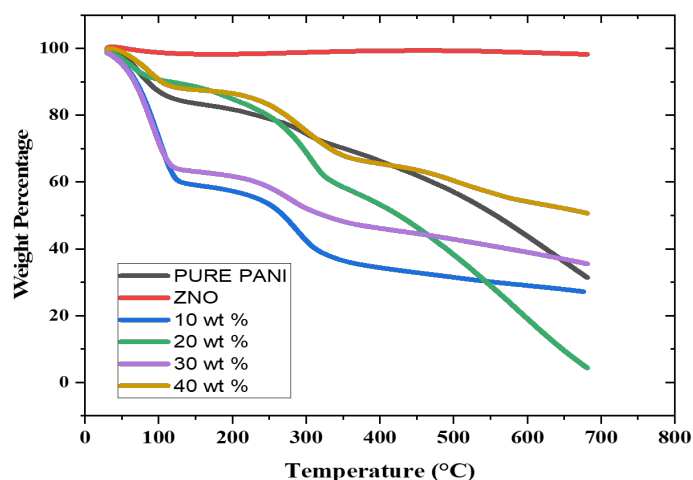


Fig 6. TG analysis of PANI-ZnO nanocomposites

Figure 6 shows the thermal behavior of PANI and PANI-ZnO nanocomposites. When compared to the composite material, the PANI has less thermal stability. As ZnO nanoparticles' weight percentage in the composite rises, so does its thermal stability. As seen in the figure, PANI-ZnO nanocomposites exhibit a more gradual breakdown than PANI alone. According to these findings, PANI-ZnO nanocomposites exhibit a notable enhancement in thermal stability. It is observed that ZnO nanoparticles exhibit robust thermal stability within the 80°C to 700°C temperature range. The weight percentages of ZnO nanoparticles in the composite material are confirmed by the TGA plot.

3.4 Dielectric Properties

The dielectric properties of all PANI and PANI-ZnO nanocomposites have been studied as a function of frequency at room temperature.

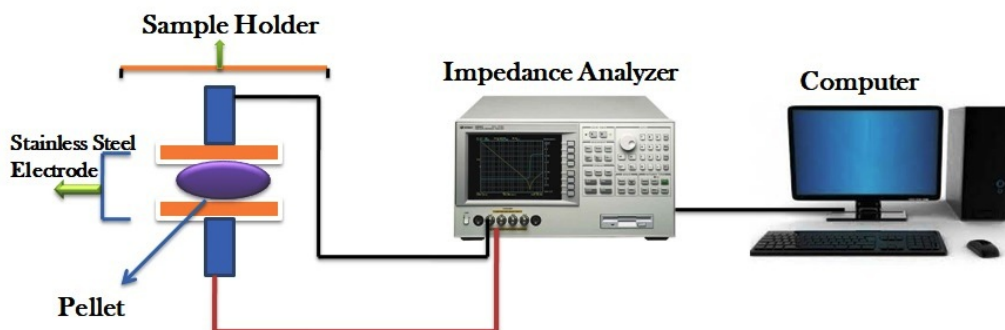


Fig 7. Electrical Properties Measuring Apparatus

3.4.1 Dielectric Constant

Using an Agilent 4294 A precision impedance analyzer, the parallel plate capacitance (C_p) and the dielectric loss tangent ($\tan \delta$) are measured for the pellet samples within the frequency range of 40Hz to 5 MHz. Equation (3) is used to calculate the dielectric constant (ϵ') and dielectric loss (ϵ'') from Equation (4) for both pure and doped nanocomposites⁽¹⁴⁾.

$$\epsilon' = \frac{Cd}{\epsilon_0 A} \quad (3)$$

$$\epsilon'' = \tan \delta \epsilon' \quad (4)$$

where ϵ_0 is the free space permittivity ($8.854 \times 10^{-12} \text{ F/m}$), d is the thickness of the sample, A is the area of cross section, and $\tan \delta$ is the measured dielectric loss tangent. Equation (5) is used for the calculation of AC conductivity.

$$\sigma_{AC} = (\epsilon' \epsilon_0 \omega \tan \delta) \quad (5)$$

' ω ' is the angular frequency.

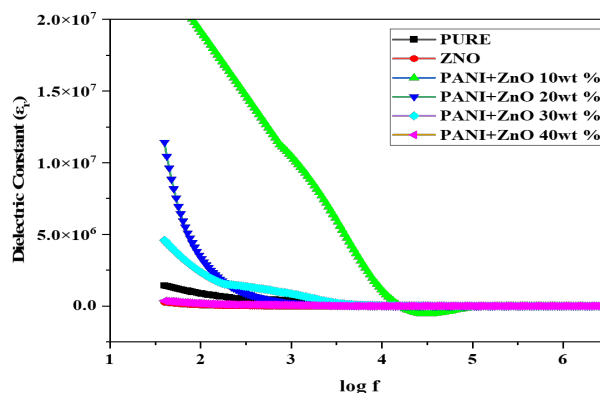


Fig 8. Dielectric constant ϵ' for PANI and PANI-ZnO nanocomposites with frequency

The dielectric constant ϵ' varies with frequency for various weight percentages of polyaniline-ZnO nanocomposites, as depicted in Figure 10. The dielectric constant is found to be relatively high at low frequencies in all cases except 30 wt %, and it further decreases as the applied frequency increases. The dielectric constant for 10 and 20 weight percent is fairly high at low frequencies and decreases linearly with increasing frequency. The observed behavior might be the result of a relaxation mechanism akin to Debye's occurring in these materials.

3.4.2 Dielectric loss

Figure 9 displays the frequency-dependent dielectric loss for PANI and PANI-ZNO nanocomposites with different wt %. Equation (6) is used to calculate the dielectric loss using experiment data on the dissipation factor and dielectric constant values.

$$\epsilon'' = \tan \delta \epsilon' \quad (6)$$

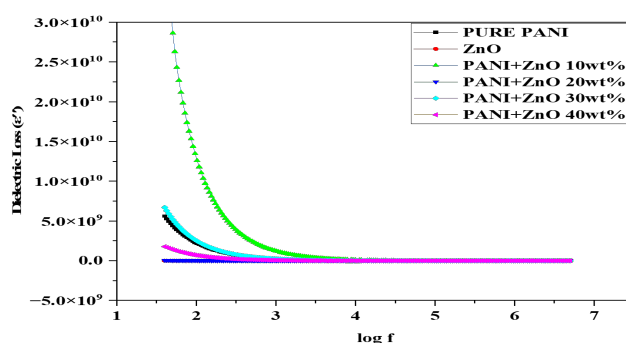


Fig 9. Dielectric loss (ϵ'') with frequency $\log(f)$ for PANI and PANI-ZnO nanocomposite

Figure 9 displays the variation of dielectric loss as a function of frequency for different PANI-ZnO nanocomposites weight percentages. The maximum dielectric loss occurs at 10% weight percentage and is consistent with the pure polyaniline trend. There is a minimum dielectric loss for the other weight percent, which is 20, 30, and 40. For all PANI-ZnO composites, the dielectric loss progressively reduced as frequency increased to higher values. The migration of ions in the materials results in a decrease in the dielectric loss ϵ'' (15).

3.4.3 AC Conductivity Studies

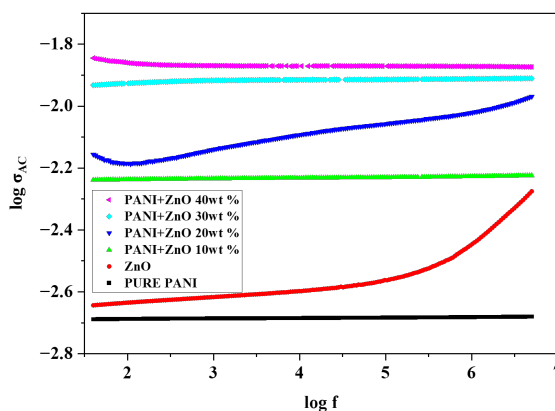


Fig 10. AC conductivity for PANI and PANI-ZnO nanocomposites with frequency

The AC conductivity for PANI and PANI-ZnO nanocomposites at room temperature with different weight percentages is shown as a function of frequency in Figure 10. Equation (5) has been used to determine the Electrical Property of AC Conductivity (σ_{AC}) as a function of frequency based on dielectric data.

Figure 10 shows that for PANI and PANI-ZnO nanocomposites, σ_{AC} (AC Conductivity) stays constant up to 7.5 Hz. Afterwards, it is raised for higher frequencies because interfacial polarization causes an increase in the AC conductivity as the weight percentages of ZnO in increase in the polyaniline matrix. 20 wt% polymer nanocomposites have a high percentage. However, dipole polarization causes the AC conductivity to drop by 10, 30, and 40% weight percentage of PANI-ZnO nanocomposite. These results are well matched with similar studies by others^(16,17).

4 Conclusion

In summary, Polyaniline-ZnO composites have been Synthesized by in-situ oxidative polymerizing aniline hydrochloride with varying weight percentages of ZnO and ammonium persulphate acting as an oxidant. XRD, TGA, UV-Visible, Dielectric Properties, and AC conductivity were used to thoroughly characterize the composites. The amorphous nature of the composites is demonstrated by XRD analysis. The crystalline nature of the zinc oxide nanoparticle is confirmed by its X-ray diffraction pattern. Additionally, the particle size of 38.27 nm was determined using the Debye-Scherer equation. TGA shows that composites are more thermally stable than PANI. Numerous kinetic parameters are computed, and their changes with varying ZnO compositions in PANI are examined. The presence of polyaniline in polymer nanocomposites is revealed by the characteristic peaks that are seen at 375 nm in the optical properties studies. It was also discovered that a pure ZnO nanoparticle had a band gap of 4.7 eV. PANI-ZnO nanocomposites of different weight percentages had their dielectric characteristics examined in the 40 Hz–5 MHz frequency range. PANI-ZnO dielectric behavior exhibits almost a Debye-type relaxation; as a result, with an increase in frequency, the dielectric constant decreases. These composites' dielectric loss indicates that it decreases with increasing ZnO weight percentage in PANI. These composites have AC conductivity, which arises with frequency and increases with the composite disorder, contributing to a 20 wt % increase in conductivity of ZnO in PANI. One of these composites may be tested for various applications, such as capacitor, gas sensing, humidity sensing, and the field of electric optoelectronic devices, according to the PANI and PANI-ZnO nanocomposites observed characteristics.

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