

Eureka Journal of Physical and Chemical Research (EJPCR)

ISSN 2760-490X (Online)

Volume 2, Issue 4, April 2026



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IMPACT OF TiO₂ NANOPARTICLES ON THE STRUCTURAL AND ELECTRICAL PROPERTIES OF NANOCOMPOSITE POLY (VINYL ALCOHOL)/ POLY(ACRYLAMIDE)/ POLYACRYLONITRILE

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Abstract

The following procedure is applied to synthesize the hybrid polymer materials. The polymer solutions of PVA, PAAm, and PAN were independently prepared. Thus, a solution of 0.4 g PVA was dissolved in 30 mL UPW under magnetic stirring at 500 rpm during 1 h. Similarly, a solution of 0.4 g of PAAm was obtained in 30 mL of UPW. Then, the two solutions were combined and further stirred during 1 h to obtain a homogeneous mixture. After that, 0.2 g of PAN was introduced to the polymer mixture; the mixture was stirred until total dissolution. Finally, four samples from the synthesized polymer matrix were made by doping TiO₂ nanoparticles in different weight percent (0%, 2%, 4%, and 6%). The doping process was

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performed in the same solvent without heating. The samples were additionally treated under ultrasonication for 2 min at 80°C to achieve a uniform distribution of the TiO₂ nanoparticles. The FT-IR data indicate clear interactions between the functional groups on the surface of the nanoparticles and polymer matrix. According to XRD, the prepared material can be considered semicrystalline. TEM imaging reveals a core-shell structure of the obtained hybrid material. The optical measurements show a remarkable increase in both real and imaginary components of the dielectric constant compared to the undoped polymer matrix.

Keywords: PVA- PAAm- PAN /TiO₂ nanocomposites; optical properties; electrical conductivity; antibacterial activity.

Introduction

Over the past two decades, interest in polymer nanocomposites has increased significantly due to their distinct properties, making them a key focus in many industrial, technological, and biomedical applications [1–10]. This interest is attributed to their diverse physical and chemical properties, such as high mechanical strength, enhanced optical and electrical properties, as well as their low cost, ease of fabrication, and flexibility, which make them suitable for use in multiple fields [11–17]. When combined with nanoparticles, polymer matrices exhibit strong interfacial interactions that contribute to enhancing their functional performance and improving their structural properties [18]. Such nanocomposites are usually manufactured by mixing the specified amount of nanofillers into the matrix of the polymer compound. In particular, polymers which are the most widely utilized for manufacturing nanocomposites include polyvinyl alcohol (PVA) and polyacrylamide

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(PAAm), due to their high biocompatibility, biodegradability, and aqueous solubility [19–21]. Additionally, the materials possess high optical transparency in the visible light spectrum. Hence, the mentioned polymers have a wide range of applications in the field of optics, microelectronics, and protective coatings [7,22]. Besides, they have high spectral band gap providing special optical and electronic properties for the improvement of systems' performance [19]. Another polymer which can be used to produce nanocomposites is polyacrylonitrile (PAN), which is a semi-crystalline synthetic polymer possessing an interrelated structure consisting of carbon chains with double bonds (C=C) and polar nitriles (-C≡N). Such chemical structure provides excellent mechanical strength, high chemical resistance, great thermal stability and flexibility [23]. Due to the great number of unique properties possessed by PAN, this polymer has been widely studied in different fields of science and technology. For example, PAN is one of the main raw materials for manufacturing of carbon fibers and microporous membranes, as well as used to obtain functional polymeric materials after chemical modifications such as amination or in situ grafting producing electron- and ion-conducting polymers [24]. Furthermore, PAN modified by incorporation of TiO₂ nanoparticles provides the following additional properties, namely improved optical and electrical performance, high antibacterial and antioxidant activity. The abovementioned advantages are determined by specific photo-catalytic activity of TiO₂ nanoparticles and its ability to change optical band gap and electrical conductivity of nanocomposite. In this connection, this study is designed to develop novel polymer nanocomposite consisting of three main polymers, namely PVA, PAAm, and PAN, supplemented by the introduction of TiO₂ nanoparticles. The goal is to design multifunctional nanofilms combining such properties as

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improved mechanical strength, optical and electrical performance, antibacterial and antioxidant activity. To achieve the goal, the study will focus on assessing the interaction between components of the proposed polymer system.

Materials and Methods

Materials

The polyvinyl acetate (PVA; $(C_2H_4O)_n$) of 85,000 g/mol molecular weight was obtained from Himedia (India). The polyacrylamide (PAAm) having 5×10^5 g/mol molecular weight and 99.99% purity was also used. This was manufactured by BDH (British Pharma). Polyacrylonitrile (PAN) was purchased from Beijing Chemical Co., Ltd. (China), available in powder form having a molecular weight of 1.50×10^5 g/mol and dissolved in DMF solvent with a purity greater than 90%. It is obtained from Alpha Chemica (India). The nano material titanium dioxide (TiO_2) nanoparticles having 10 to 35 nm size, 47.867 g/mol molecular weight, and 99.6% purity were also used. They have been obtained from Sigma-Aldrich. These highly pure materials ensure stable chemical reaction and reliable results for the determination of physical and optical characteristics of the resulting nanocomposite polymers.

Preparation and combination of samples

Prior to the preparation process, polyvinyl acetate (PVA) was dissolved in distilled water at a moderate temperature. The temperature was increased gradually while continuously stirring to achieve total dissolution due to the high molecular weight of the composite. PVA and PAAm were dissolved in distilled water, followed by mixing with PAN in a ratio of 40:40:20 for each polymer. The TiO_2 nanoparticles were then incorporated into the mixture in different weight fractions (2%, 4%, and 6%) to form the PVA-PAAm-

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PAN/TiO₂ nanocomposite.. The resulting samples were distributed in clean Petri dishes and left to dry at room temperature for five days to ensure complete evaporation of the solvent and the formation of homogeneous and stable nanocomposite polymer films.

Characterization

X-ray diffraction (XRD) analysis was performed using a Philips Model PW1730 (USA) to determine the degree of crystallinity and characterize the crystal structure of the prepared samples. Field scanning electron microscopy (FESEM) analysis was performed using a TESCAN Mira3 equipped with an EDAX elemental analysis module, manufactured by TESCAN in the Czech Republic, to study the surface morphology and distribution of nanoparticles in the samples. Optical measurements were performed using a Vertex 701 UV–Vis dual-beam spectrophotometer, operating in the wavelength range (190–1100 nm), manufactured by Philips, Japan. For electrical measurements, an AC Electrical Hi Tester (LCR Meter) from Hioki, Japan, was used to accurately evaluate the electrical properties of the prepared samples.

Findings and conversation

The structure of the crystals in the nanofibers was determined using an X-ray diffraction technique (XRD). X-ray diffraction studies were performed using a Philips X'Pert High Score diffractometer (PANalytical, Almelo, The Netherlands) with Cu K α radiation ($\lambda = 0.1542$ nm) at 40 kV. The samples were scanned in a range of 2θ from 10° to 80° in order to determine the structural form of the crystalline components in the matrix. According to the results shown in Figure 1, broad peaks appeared at $2\theta = 19.75^\circ$ and 23.65° in the XRD pattern. This indicated the presence of an amorphous polymer matrix

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in the mixture of the three types of polymers. When TiO₂ nanoparticles were added into this mixture, several new peaks appeared. This indicated the crystalline nature of the matrix. Specifically, the diffraction peaks corresponded to the (110), (200), (111), (211), (002), (311), and (202) planes for TiO₂. For the rutile phase, the peaks appeared at $2\theta = 27.43^\circ$, 39.18° , 41.23° , 54.31° , 62.75° , 72.41° , and 76.52° , which correspond to tetragonal crystals of TiO₂ according to the JCPDS card No. 72-1148. Overall, TiO₂ nanoparticles were confirmed to be successfully incorporated into the polymer matrix, and results obtained were supported by previous studies

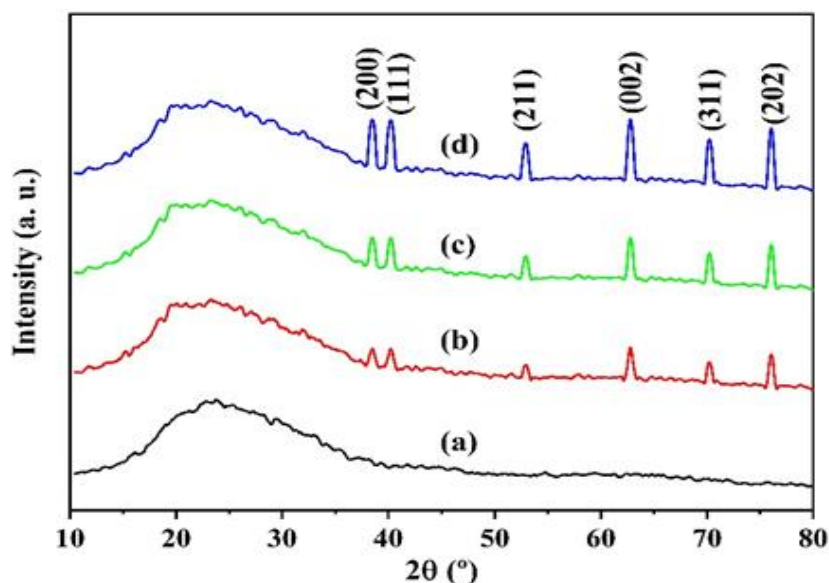


Fig.1. XRD patterns of the PVA- PAAm- PAN /TiO₂ nanocomposites: (a) blend , (b) 2 wt.%, (c) 4 wt.%, and (d) 6wt.%.

Field-emission scanning electron microscopy (FESEM) was used to analyze the surface structure and morphology of both the host sample (PVA–PAAm–

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PAN) and the titanium dioxide nanocomposites (TiO₂ PNCs). Images taken at 100× magnification (Figure 2) demonstrate the distinct variation in surface morphology between the different samples. Images of the base sample (PVA–PAAm–PAN) revealed a smooth and homogeneous surface, with no dark clusters indicating the presence of nanoparticle agglomerates, confirming the purity of the matrix prior to doping. For the samples loaded with TiO₂ particles, the images showed a relatively homogeneous distribution of nanoparticles within the polymer matrix, with the formation of fine, evenly distributed agglomerates. The size and number of agglomerates gradually increased as the TiO₂ content increased from 0.5% to 10% by weight, with the agglomerates becoming more pronounced and dense at higher concentrations. This behavior indicates an increased probability of coalescence of nanoparticles at high concentrations due to interparticle attractive forces. These observations are consistent with the results of Abdel Salam's research group [27], which indicated a similar behavior when incorporating SiO₂/In₂O₃ particles into a polystyrene polymer matrix, where higher nanoparticle concentrations lead to increased agglomeration and improved structural homogeneity of the composite.

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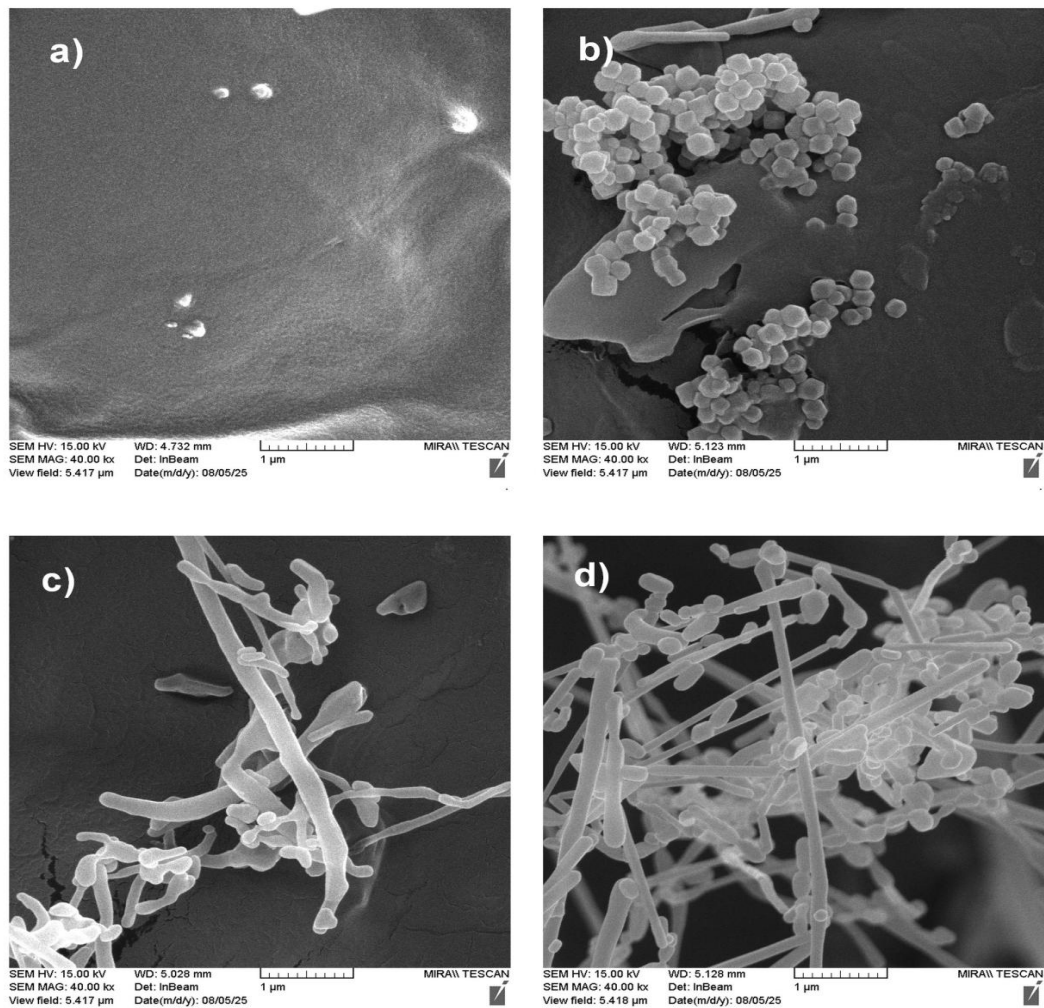


Fig. 2. FESEM images of (a) PVA- PAAm- PAN blend and (a)–(d) TiO₂ NPs (2, 4 and 6)wt.%

The A.C electrical Characteristics of PVA- PAAm- PAN /TiO₂ nanocomposites are investigated as well, including dielectric constant, dielectric loss and A.C electrical conductivity in frequency range (100 –

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5×10^6) Hz at room temperature. The dielectric constant (ϵ') of nanocomposites were calculated from equation (1) [28]:

$$\epsilon' = \frac{C_p}{C_0} \quad (1)$$

Where C_p is parallel capacitance and C_0 is a vacuum capacitor.

The dielectric loss (ϵ'') for PVA- PAAM- PAN /TiO₂ nanocomposites was calculated as following [28,29] :

$$\epsilon'' = \epsilon' D \quad (2)$$

Where: D is the dispersion factor. The A.C electrical conductivity of nanocomposites is estimated by using equation (3) [29]:

$$\sigma_{A.C} = \omega \epsilon_0 \epsilon'' \quad (3)$$

Figure (3) shows the behavior of the dielectric constant of the PVA–PAAM–PAN/TiO₂ nanocomposites as the frequency changes. The results showed that the dielectric constant gradually decreased with increasing frequency. This decrease is attributed to the limited ability of the electric dipoles to respond quickly to electric field changes at high frequencies. At low frequencies, several mechanisms contribute to the polarization process, including ionic and electronic polarization, and interface polarization (Maxwell–Wagner), resulting from the difference in dielectric constant between the polymer components and the nanoparticles. As the frequency increases, the contribution of spatial charges and interface polarization decreases due to their inability to adapt to the accelerating field changes. The results also showed that the incorporation of TiO₂ nanoparticles into the ternary polymer

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matrix led to a significant improvement in dielectric properties at low frequencies. This decrease is attributed to the effective interfacial interaction between the nanoparticles and the polymer chains, which enhanced the interface polarization. In addition, the nanocomposites exhibited low energy loss, indicating their high electrical response efficiency and thermal stability. Based on these results, it can be concluded that the (PVA–PAAm–PAN/TiO₂) nanocomposites possess excellent electrical insulation properties that qualify them for use in microelectronic components and pressure sensors, in line with previous scientific reports [28, 30].

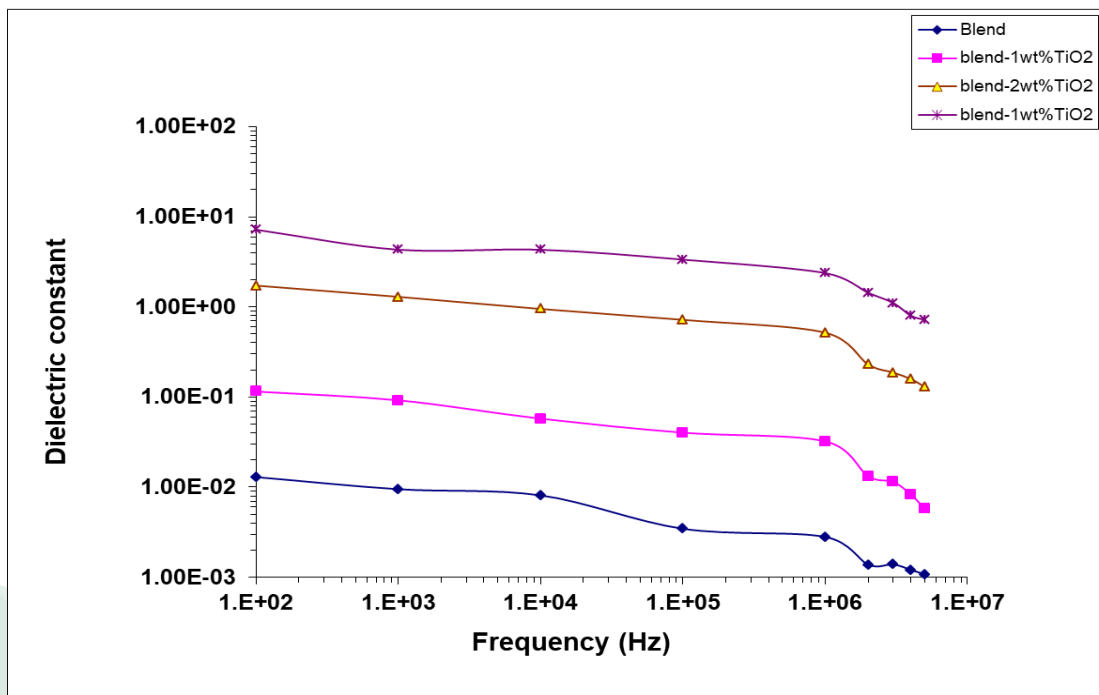


Fig. 3: Variation of dielectric constant for PVA- PAAm- PAN /TiO₂ nanocomposites with frequency

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The dielectric losses of PVA-PAAm-PAN/TiO₂ nanocomposites decrease as the frequency increases, as shown in Figure 4. The reason for such a trend is that when frequencies become lower, there is a decline in space charge polarization, leading to an increase in dielectric losses. However, as the frequencies become higher, the dielectric losses decrease because there is enough time for the electric dipoles to orient themselves to the applied field before the change in the direction. Moreover, the dielectric losses increase when the TiO₂ concentrations increase due to an increase in charge carriers. [31,32].

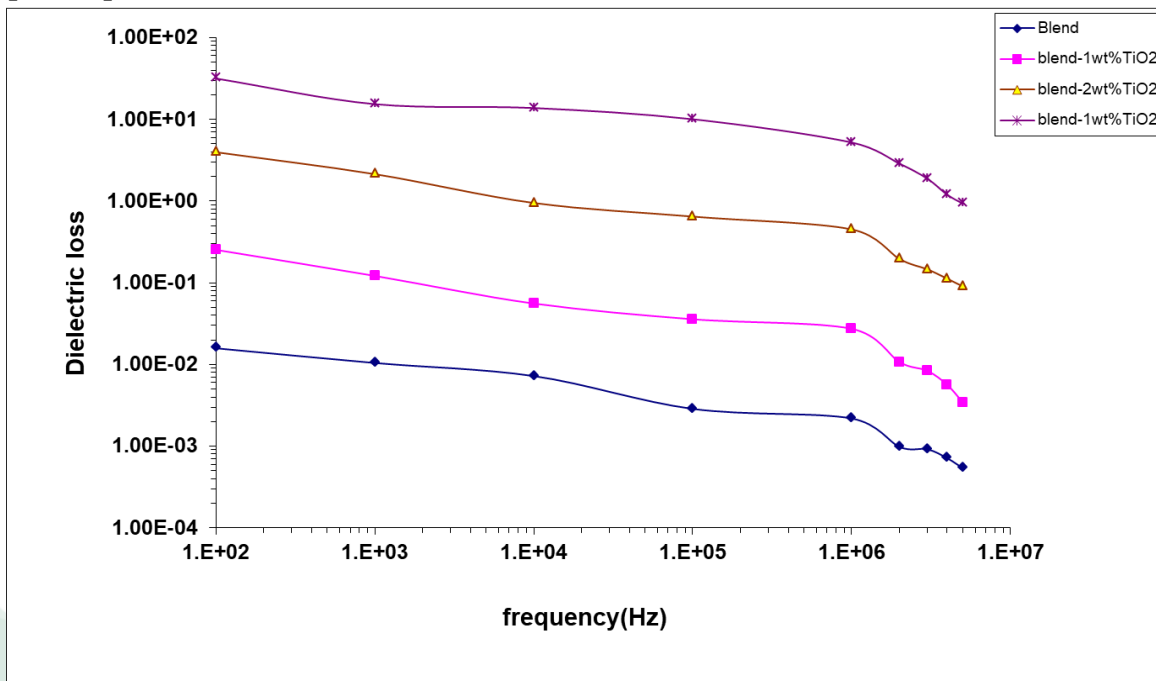


Fig. 4: Variation of dielectric loss for PVA- PAAm- PAN /TiO₂ nanocomposites with frequency

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It is noted that the TiO₂ nanoparticles contribute to the development of a percolating path network in the nanocomposite due to their higher concentrations in the polymeric blend comprising PVA, PAAm, and PAN. The dependence of AC conductivity on the frequency for the case of PVA-PAAm-PAN nanocomposites is illustrated in Figure 5. The increase in AC conductivity in terms of frequency is associated with space-charge polarization that occurs at lower frequencies and with better charge carrier mobility through the hopping mechanism. Increased charge carrier mobility is achieved in the high-frequency domain, and hence, the conductivity of PVA-PAAm-PAN/TiO₂ nanocomposites grows with an increase in the frequency. [31-33].

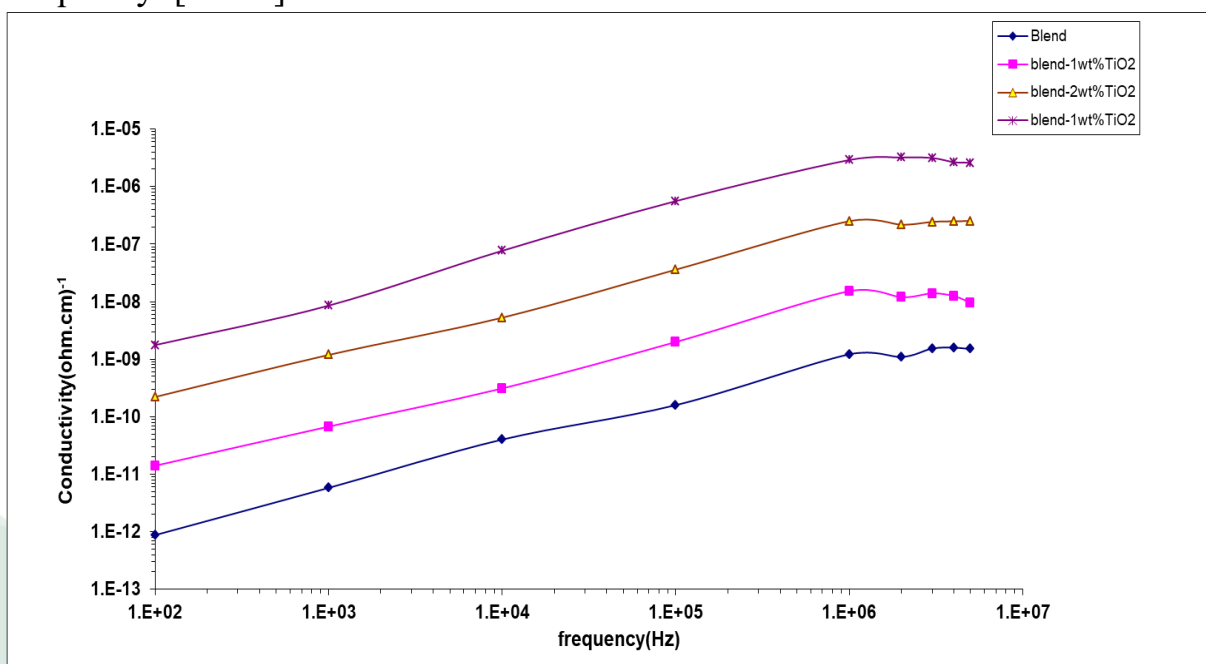


Fig.5 :Variation of A.C electrical conductivity for PVA- PAAm- PAN /TiO₂ nanocomposites

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Conclusions

This study demonstrated clear success in designing and characterizing hybrid nanocomposites based on a PVA–PAAm–PAN polymer matrix reinforced with TiO₂ nanoparticles, in response to the growing interest in developing innovative and sustainable materials with advanced electrical and functional properties. The results revealed that the addition of TiO₂ to the polymer matrix significantly improved the electrical performance. AC electrical conductivity gradually increased with the increase in the nanoparticle content, and dielectric values improved due to the effective interaction between the components at the nanoscale. However, the PVA–PAAm–PAN/TiO₂ nanocomposites exhibit multifunctional behavior, making them useful for numerous applications like biomedical devices, flexible electronics, and intelligent packaging due to their remarkable stability in terms of both mechanical and electrical behavior. It is suggested in the study that further work should be conducted to find out the optimal dispersal of nanoparticles in the polymer matrix in higher concentrations, as well as the in vivo behavior of the nanocomposites.

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