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Preparation, characterization, and property evaluation of PS+Fe_3O_4 nanocomposites

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Abstract

PS+Fe_3O_4-based nanocomposites were fabricated by combining solution casting and hot pressing methods. SEM image revealed the presence of microvoids and pores on the volume of the nanocomposites. This microstructure indicates that the incorporation of Fe_3O_4 nanoparticles altered the morphology of the polymer matrix. Dielectric measurements performed in the 50 kHz–1 MHz frequency range showed that the addition of Fe_3O_4 increased the polarity of the composites and modified their relaxation behavior. In particular, the PS+10%Fe_3O_4 nanocomposite exhibited a weak frequency dependence of dielectric permittivity, while the dielectric loss tangent of all filled samples was higher than that of neat PS. These results demonstrate that Fe_3O_4 nanoparticles strongly influence the interfacial polarization and dielectric response of PS-based nanocomposites.

Keywords: Nanocomposite, magnetite, dielectric properties, dielectric polarization
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1. Introduction

Polymer-based nanocomposites have attracted considerable attention in recent years. They combine the lightweight nature, processability, and chemical stability of polymers. Inorganic nanoparticles possess several functional properties [1]. Fe_3O_4 nanoparticles are of particular interest as fillers due to their unique magnetic behavior, relatively low cost, chemical stability. When incorporated into polymer

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matrices, Fe_3O_4 nanoparticles can significantly modify the structural, dielectric, and interfacial characteristics of the nanocomposites. Polystyrene is a widely used thermoplastic polymer with good processability, low density, and favorable electrical insulation properties [2]. The phenyl groups in PS's macromolecular structure lead to the observation of characteristic polarization behavior, which makes it an attractive matrix for studying the effect of magnetic Nano fillers on dielectric response. The introduction of Fe_3O_4 nanoparticles into a polystyrene matrix may lead not only to changes in morphology and interfacial interactions, but also to improvements in functional performance [3].

Huang et al. (2022) fabricated a superhydrophobic polystyrene and Fe_3O_4-based sponge using a high internal phase emulsion templating method. The obtained material combined superhydrophobicity with superoleophilicity. These materials exhibit water and oil contact angles of 155° and 0°, respectively. With the addition of Fe_3O_4, the sponge could be remotely manipulated under a magnetic field [4]. The environmental relevance of Polystyrene and Fe_3O_4 - based composites has also been emphasized by Chen et al. (2022). They developed magnetic PS@Fe_3O_4 microparticles for improving biological nitrogen removal in wastewater treatment. Their study showed that the incorporation of such particles into activated sludge systems accelerated sludge settling to 0.76m/h. It also improved ammonium nitrogen removal, total nitrogen removal, and simultaneous nitrification–denitrification by 7.08%, 24.27%, 19.53% respectively[5]. Guo et al. (2006) investigated the coating of Fe_3O_4 particles with polystyrene by dispersion polymerization and demonstrated that the use of a cross-linking reagent played a crucial role in forming a continuous and dense polymer shell on the particle surface. Their results suggested that the coating mechanism proceeded through the adsorption and coalescence of polymer sols, leading to the formation of a relatively thick polymer layer [6]. Similarly, Yan et al. (2008) prepared Fe_3O_4/polystyrene composite particles by miniemulsion polymerization and emphasized the importance of nanoparticle surface modification. They found that Fe_3O_4 nanoparticles modified with a monolayer of oleic acid exhibited better hydrophobicity and dispersibility in styrene than those coated with a multilayer, thereby enabling the formation of composite particles with more defined structure and controllable magnetite content [7].

In addition to binary PS-Fe_3O_4 systems, more complex hybrid structures have also been explored. Abdullah et al. (2021) reported polystyrene–Fe_3O_4–MWCNT nanocomposites for the removal of toluene from water. In their work, the optimum adsorption parameters were found to be 60 min, 2 mg adsorbent dose, pH 5, 35 °C, 50 mL solution volume, 50 mg/L toluene concentration, and a shaking speed of 240 rpm [8].

Despite the reported results, the role of Fe_3O_4 nanoparticles on the structure and dielectric behavior of polystyrene-based nanocomposites still needs additional investigation. Therefore, the aim of the present work is to prepare PS/Fe_3O_4 nanocomposites, to characterize their morphology, and to evaluate their dielectric properties as a function of Fe_3O_4 content and frequency.

2. Experimental Part

2.1. Materials

The chemicals that were used in this study: High impact polystyrene (HIPS 7240) was purchased from Tabriz Petrochemical Company (Tabriz, Iran) with a melt flow index of 4.5 g/10 min tested at 200 ◦C using a load of 5 kg.

All reagents used for the synthesis of Fe_3O_4 nanoparticles were obtained from Sky Spring and were of high purity

2.2. Preparation PS+Fe_3O_4 nanocomposites

Firstly, Fe_3O_4 nanoparticles were synthesized by the co-precipitation method. Specifically, $FeCl_3 \cdot 6H_2O$ and $FeSO_4 \cdot 7H_2O$ were introduced into the reaction medium in amounts of 0.008 and 0.015 mol, respectively. Subsequently, ammonium hydroxide ($NH_3 \cdot H_2O$) was added dropwise until the pH of the solution reached 11–12. After completion of the reaction, the mixture was allowed to cool to room temperature, and the obtained precipitate was washed six times with ethanol and dried under vacuum at 45 °C. As a result, Fe_3O_4 nanoparticles were successfully obtained.

Nanocomposite films were fabricated using a combination of solution mixing and hot pressing. First, polystyrene was dissolved in toluene under continuous stirring with a magnetic stirrer at 120 °C for approximately 30 min. The previously synthesized Fe_3O_4 nanoparticles were then added to the polymer solution, and the mixture was stirred for 2 h to ensure homogeneous dispersion of the filler within the polymer matrix. Afterward, the solvent was removed, and the resulting mixture was subjected to hot pressing to obtain the final nanocomposite films. The hot-pressing process was carried out at 160 °C under a pressure of 10 MPa for 5 min.

2.3. Characterization of nanocomposites

2.3.1. X-ray analysis

Rigaku Mini Flex 600 XRD diffractometer (CuKα radiation from Cu X-ray tube, run at 15 mA and 30 kV) was used for X-ray diffraction analysis at ambient temperature. The nanocomposite films were scanned in the 2θ angle range from 100 to 100°.

2.3.2. Scanning electron microscopy (SEM) analysis

SEM analysis of nanocomposites was performed on an FEI Quanta Inspect microscope (FEI, Hillsboro, OR, U.S.A.)

2.3.3. Dielectric measurement

Dielectric permittivity of the nanocomposites was determined using a broadband immittance analyzer (MNIPI E7-20). Frequency-dependent measurements were conducted in the range of 25 Hz to 1 MHz at room temperature.

3. Experimental results and their discussion

The morphology of the polymer nanocomposite was investigated using scanning electron microscopy. The micrograph of the sample is shown in Figure1. It reveals the presence of distinct microvoids and pores in the structure of the polymer nanocomposite, confirming that the incorporation of these nanoparticles induces significant changes in the polymer morphology.

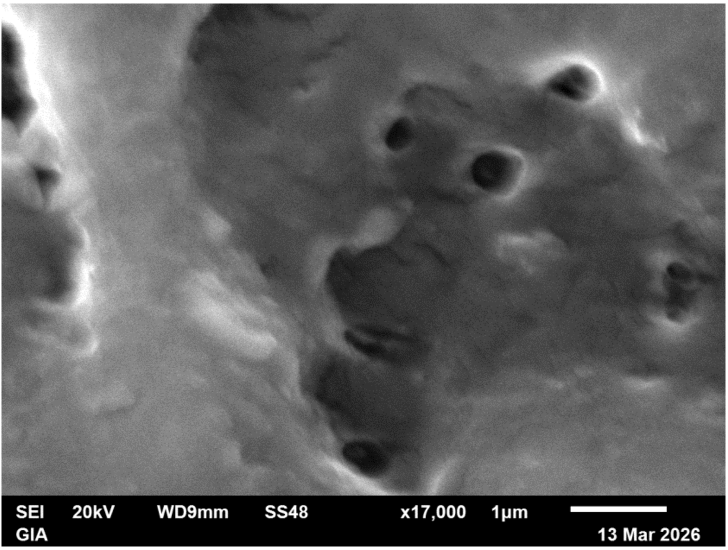

Fig. 1. SEM micrograph of PS+Fe_3O_4 nanocomposites

Figure 2 illustrates the frequency dependence of the dielectric permittivity of PS+Fe_3O_4-based nanocomposites. The results indicate that the incorporation of Fe_3O_4 nanoparticles enhances the polarity of the material. In polystyrene, a small dipole moment is generally assumed due to the weak asymmetry induced by the phenyl ring. Therefore, at higher Fe_3O_4 contents, the probability of charge accumulation at the polymer matrix–nanoparticle interface increases, leading to the development of interfacial polarization of the Maxwell–Wagner type. As a result, the weak frequency dependence of ε′ observed for the PS+10%Fe_3O_4 nanocomposite can be attributed both to the additional electronic polarization associated with the phenyl ring and to the enhanced interfacial polarization arising from the higher filler concentration [9].

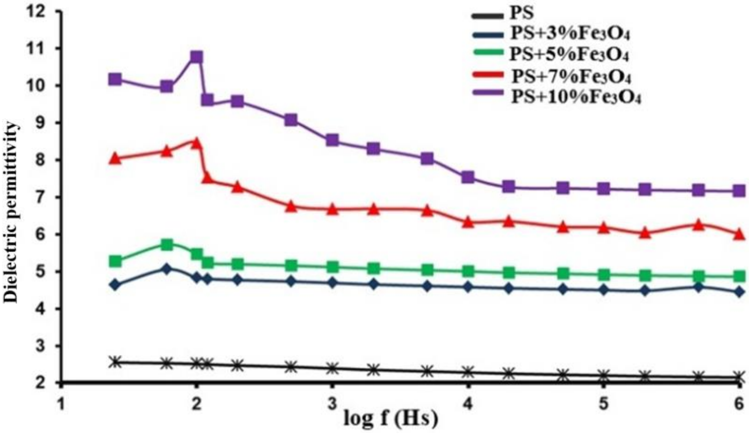

Fig. 2. Frequency dependence of the dielectric permittivity of PS+Fe_3O_4-based nanocomposites

Figure 3 showing the dependence of the dielectric loss tangent (tan δ) on the logarithmic value of frequency in the 50 kHz–1 MHz frequency range for PS+Fe_3O_4-based nanocomposite samples with different concentrations is presented in the figure. The incorporation of Fe_3O_4 nanoparticles leads to noticeable changes in the relaxation processes. In particular, unlike neat polystyrene, no second resonance maximum is observed in the high-frequency region, and for all nanocomposite concentrations, the values of the dielectric loss tangent are higher than those of pure polystyrene[10]. It is well known that the dielectric loss tangent characterizes the energy dissipation occurring in the material. The increase in dielectric losses in the high-frequency region after the introduction of the nanosized filler can be explained by the fact that the incorporation of magnetite nanoparticles alters the internal field of the polymer. As a result, at high frequencies, the resonance maximum associated with the response of the polymer to variations in the external field disappears, while the magnitude of dielectric losses increases [11]. The analysis of the dielectric loss tangent as a function of the logarithm of frequency also indicates that the morphological structure of the nanoclusters and their distribution within the polymer matrix significantly affect not only the dielectric constant but also the energy losses.

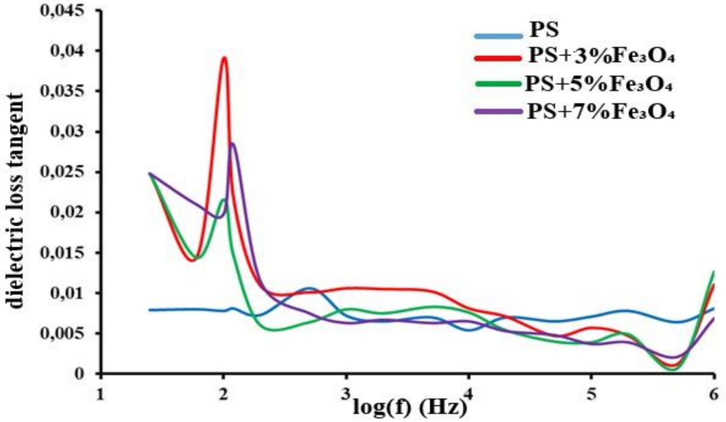

Fig. 3. Frequency dependence of the dielectric loss tangent of PS+Fe_3O_4-based nanocomposites

4. Conclusion

In this study, PS+ Fe_3O_4-based nanocomposites were successfully prepared and their morphological and dielectric properties were investigated. SEM analysis revealed the presence of distinct microvoids and pores in the nanocomposite structure. This fact indicate that the incorporation of Fe_3O_4 nanoparticles leads to significant modifications in the morphology of the polymer matrix. These structural changes confirm that the nanoparticles affect not only the microstructure of the material but also the interfacial interactions within the composite system. The dielectric measurements showed that the addition of Fe_3O_4 nanoparticles enhances the polarity of the nanocomposites and significantly influences their frequency-dependent dielectric behavior. In particular, the weak frequency dependence of dielectric permittivity observed for the PS+10%Fe_3O_4 sample can be explained by the combined effect of electronic polarization associated with the phenyl groups of polystyrene and the increased interfacial Maxwell–Wagner polarization at higher filler contents. Furthermore, the dielectric loss analysis demonstrated that the incorporation of Fe_3O_4 alters the relaxation behavior of the polymer, suppresses the second resonance maximum in the high-frequency region, and increases the dielectric loss tangent compared with neat polystyrene. Overall, the results indicate that both the concentration of Fe_3O_4 nanoparticles and their distribution in the polymer matrix play a key role in determining the dielectric permittivity and energy dissipation characteristics of PS-based nanocomposites.

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