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Research Paper

Characterization of Epoxy/Fe_2O_3 Nanocomposites with Enhanced Physical Properties

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Abstract

In this study, epoxy/iron oxide (EP/Fe_2O_3) nanocomposites were prepared at different weight percentages (0.1, 0.3, and 0.5 wt%) to investigate their structural, thermal, electrical, dielectric, and mechanical properties. Scanning electron microscopy (SEM) images showed that the Fe_2O_3 nanoparticles possessed a homogeneous morphology with irregular grain size and an average size of 60-70 nm, indicating their suitability for reinforcing polymer matrices. The addition of Fe_2O_3 nanoparticles significantly improved the thermal conductivity of the epoxy due to the formation of partial phonon transport pathways and enhanced interfacial interaction at higher addition percentages. Dielectric measurements showed an increase in the dielectric constant and dielectric loss with increasing Fe_2O_3 content, with both decreasing with increasing frequency due to interfacial polarization mechanisms. AC electrical conductivity results demonstrated frequency-dependent behavior with a marked improvement in conductivity upon nanoparticle addition. Furthermore, the Shore D hardness test results showed a gradual improvement with increasing Fe_2O_3 content, attributed to the restriction of epoxy chain movement and increased cross-linking density. These results confirm that Fe_2O_3 is an effective filler for enhancing the performance of multifunctional epoxy composites.

Keywords

Structural Properties, Thermal Properties, Dielectric Properties, Epoxy, Nanocomposite, Iron Oxide (Fe_2O_3)

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1. INTRODUCTION

Epoxy is considered the choice of the most sought-after technological applications primarily due to its exceptional physical and chemical features, high heat resilience, solvent resistance, simplicity of manufacture, and outstanding mechanical characteristics. The number of studies investigating polymer nanocomposites has expanded dramatically in recent decades as a consequence of the advancement of complex materials for potential applications (Frigione and Lettieri, 2020; Saharudin and Inam, 2020).

Epoxy and its composites are used in a broad range of sectors, particularly automobiles (Saharudin and Inam, 2020; Subadra et al., 2020; Campo et al., 2020; Wei et al., 2017a,b), aviation (Saharudin and Inam, 2020; Wei et al., 2017a,b; Yıldırım et al., 2021), electronic and electrical equipment (Wei et al., 2017b; de Oliveira et al., 2019; Wu et al., 2020; Yuan et al., 2020), and plenty more. Biomedical equipment, sensors, and other ordinary consumer products (Campo et al., 2020; Wei et al., 2017b; Glaskova-Kuzmina et al., 2020; Sanli et al., 2016; Spinelli et al., 2020). Regardless, extensive research is being conducted to increase the epoxy's mechanical properties, extreme environmental resistance, damage resistance, and high-temperature performance. Second-phase components can be dispersed in epoxy resin systems to increase performance (Balguri et al., 2021). Nanoparticles, or nanofillers, can increase mechanical qualities, as well as the barrier and anticorrosion properties of epoxy coatings (Frigione and Lettieri, 2020; Kausar, 2020; Jing et al., 2020; Saharudin and Inam, 2020). The nanocomposite is defined by two factors: (1) the type of matrix in which nanoparticles are disseminated, and (2) the type of nanofillers used. To optimize the qualities and features of epoxy resins, several nanoparticles have lately been utilized as fillers (Abdeen et al., 2019). In nanomaterials, nanofillers are defined as nanomaterials with at least one dimension between one and one hundred nanometers (Frigione and Lettieri, 2020). Nanofillers may be generated in four different configurations from zero to three dimensions (0D-to-3D).

Epoxy resins are widely used polymers in engineering and electrical applications. However, the limitations of some of their thermal, insulating, and mechanical properties necessitate their development to meet advanced performance requirements. In this context, reinforcing them with inorganic nanofillers represents a promising approach to enhancing their multifunctional properties. Iron(III) oxide (Fe_2O_3) is a suitable material for this purpose due to its thermal stability, excellent electrical properties, and low cost. The significance of this research lies in investigating the effect of low weight percentages of Fe_2O_3 nanofillers on the microstructure and thermal, electrical, and mechanical properties of epoxy composites. The aim is to understand the optimization mechanisms related to interfacial interaction, the formation of partial heat transfer pathways, and the restriction of polymer chain movement. The novelty of this work lies in providing an integrated analysis linking structural composition and functional performance, which contributes to the design of improved nanocomposite materials suitable for advanced engineering applications.

2. METHOD AND MATERIALS

2.1 Materials Used

2.1.1 Matrix

Epoxy polymer matrix is prepared by mixing 3:1 parts by volume of epoxy resin (Nitofill, EPLV with hardener (amine) supplied by the company Fosroc Jordan. Epoxy resin contains epoxide groups that serve as cross-linking points. The resin reacts with the hardener to form long chains of cross-linked polymer.

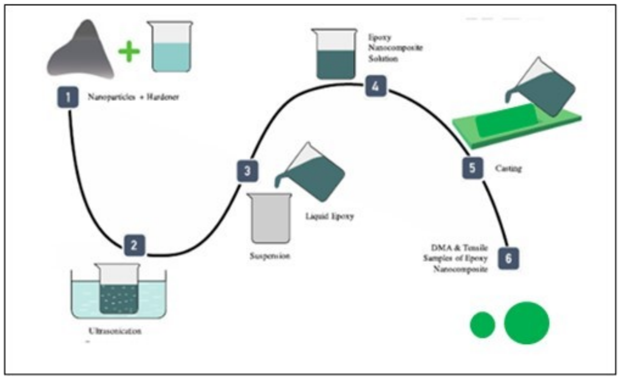

Figure 1. Illustrative Diagram of Sample Preparation

2.1.2 Filler

In this study, nanomaterial was used to prepare nanocomposites (Fe_2O_3 particle size 60–70 nm supplied by a company Nanjing Nano Technology of purity 99.9%, with weight ratios (0.3, 0.6, 0.9) wt%.

2.2 Experimental Part

The first step of sample preparation is to prepare the composite matrix as shown in Figure 1. The matrix in this work was prepared from an epoxy polymer resin (Nitofill, EPLV) and a hardener (amine) in a mixing ratio of [3:1]. The materials were supplied by Fosroc Jordan. Epoxy resin has epoxide groups in it and acts as a crosslinking point; however, these long chains of cross-linked polymer are formed when the resin reacts with the hardener. The reinforced material used was Fe_2O_3 nanomaterial with a particle size ranging from 60 to 70 nm, and a purity of. (99.9%) supplied from US Research Nanomaterial Ins. and weight ratios of. Three mixing ratios (0.1, 0.3 and 0.5) wt% were used to study the effect of Fe_2O_3 on the properties of epoxy. However, weighing of the nano powder was weighed and gently mixed with the hardener for 5 seconds, and then the mixture was placed in a bath sonicator for about 30 minutes at room temperature to ensure proper mixing. After cooling to room temperature, the solution was mixed with the liquid epoxy in a mixing ratio of [3:1] for 5 minutes using vigorous hand mixing, cast into molds, and allowed to dry to room temperature for 7 days.

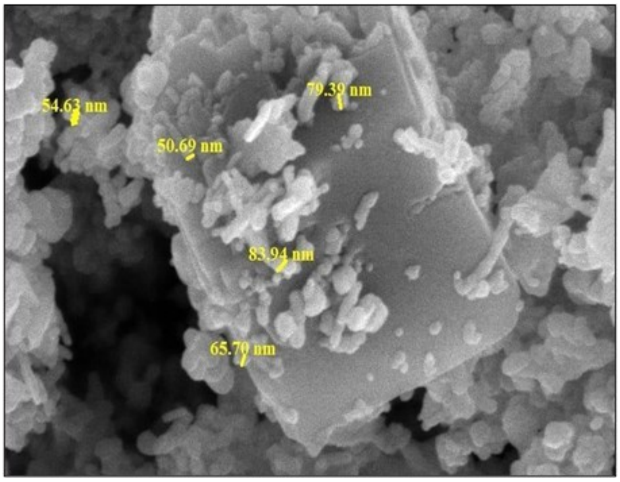

Figure 2. SEM of Fe_2O_3 Nanopowder

In general, the samples used in the tests and examinations were prepared according to the standard system specifications, as follows:

(a) Thermal conductivity: Diameter (40 mm), calculated using Lee's disc method (Griffen & George).
(b) Dielectric properties: Diameter (20 mm), measured using an Agilent impedance analyzer (model 4294A, Taiwan).
(c) Hardness test: Specimen size (25 mm × 25 mm), measured using a Shore D durometer (model Checkline DD-100, USA).

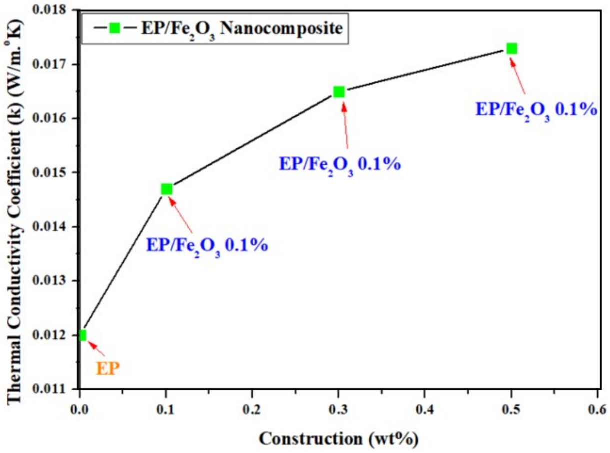

Figure 3. Thermal Conductivity as a Function of Fe_2O_3 Weight Ratios

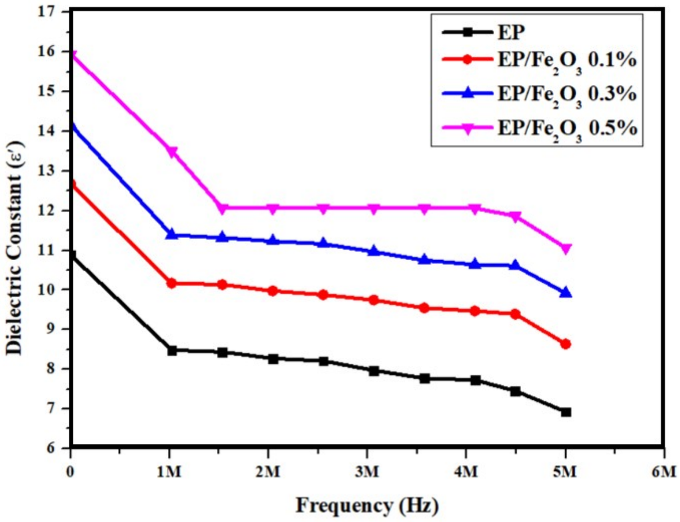

Figure 4. The Dielectric Constant of (EP/Fe_2O_3) Composite Varies with Frequency

3. RESULTS AND DISCUSSION

This section discusses the experimental measurements that were obtained as follows. Morphology of the nanopowder (SEM), the surface morphology of Fe_2O_3 nanopowder was visualized by scanning electron microscopy, as shown in Figure 2. The particle morphology has homogenous irregular grains. The island growth of the tightly packed spherical arrangement was clearly observed. In some regions, the big nanoparticles were surrounded by smaller nanoparticles. As may be seen, the mean particle size of Fe_2O_3 nanoparticles is about 60-70 nm.

3.1 Thermal Conductivity

Several factors, including the architecture, volume of loading, dispersion, and surface qualities of inorganic fillers, and even the thermal impedance of the interface among fillers and the polymer matrix, all have an impact on the thermal conductivity of inorganic-organic composites (Wang et al., 2017). Ep/Fe_2O_3 nanocomposites with different filler weight ratios are examined in terms of their thermal conductivity at room temperature. Figure 3, purified epoxy has a thermal conductivity of (0.012 W/m.°K) at room temperature, which is considered to be low by modern standards. The formation of partial connections or networks between Fe_2O_3 particles occurs at a higher filler loading amount, which is advantageous for phonon transmission and thus capable of significantly increasing the thermal conductivity of Fe_2O_3/epoxy nanocomposites when increasing the Fe_2O_3 filler loading amounts from 0.1 wt% to 0.3 wt% (Najmi and Hu, 2023). The findings suggest that Fe_2O_3 particles could be used as a viable filler material for augmenting the epoxy polymer matrix whilst preserving thermal stability. It is therefore possible to boost the interface interactions involving Fe_2O_3 fillers and epoxy by treating the surface of the Fe_2O_3 particles. It has been shown that the formation of covalent connections between Fe_2O_3 particles and epoxy matrix can reduce the mobility of local matrix material surrounding the filler particles, leading to enhanced thermal stability, though at extreme temperatures (Sahu et al., 2017).

3.2 Dielectric Constant (ϵ')

The Figure 4 shows the frequency dependence of the dielectric constant (ϵ') for pure epoxy (EP) and EP/Fe_2O_3 nanocomposites at different addition percentages (0.1, 0.3, and 0.5 wt%). Pure epoxy exhibits the lowest dielectric constant values across the studied frequency range, while the addition of Fe_2O_3 nanoparticles leads to a significant increase in the dielectric constant, with this increase increasing with the addition percentage. The composite at 0.5 wt% Fe_2O_3 shows the highest dielectric constant compared to the other samples. The figure also demonstrates that the dielectric constant gradually decreases with increasing frequency for all samples. This behavior is attributed to the inability of the electrical dipoles and interpolar polarization mechanisms (Maxwell–Wagner–Sillars) to keep pace with the changing electric field at high frequencies, and the reason for this could be due to the heterogeneous distribution of nanocomposite materials within the substrate, or perhaps the increase in the molecular weight of the polymeric chains as a result Mohammed (2022) and Hajiyeva and Ramazanov (2025).

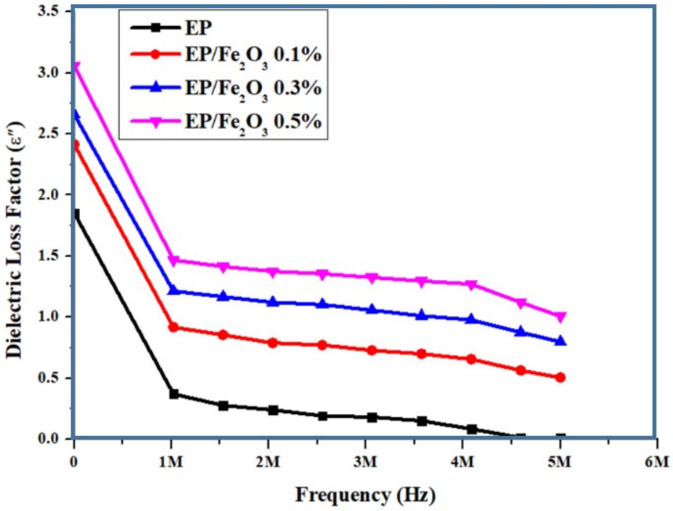

Figure 5. The Dielectric Loss Factor of (EP/Fe_2O_3) Composite Varies with Frequency

3.3 Dielectric Loss Factor

The Figure 5 shows the change in dielectric loss coefficient (ϵ'') with frequency for pure epoxy (EP) and EP/Fe_2O_3 composites at different addition percentages (0.1, 0.3, and 0.5 wt%). The dielectric loss coefficient is observed to be high at low frequencies and then gradually decreases with increasing frequency for all samples. This behavior is attributed to the dominance of interpolar polarization mechanisms and charge transfer at low frequencies, while their contribution is reduced at high frequencies due to the inability of dipoles to keep pace with rapid changes in the

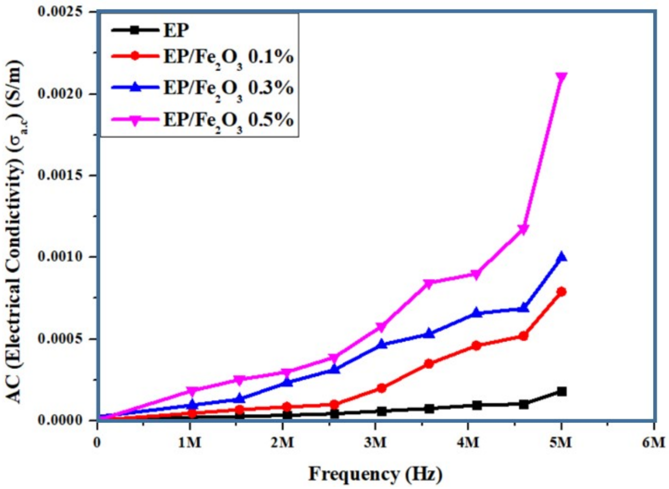

Figure 6. AC Conductivity of (EP/Fe_2O_3) Composite Varies with Frequency

electric field. The results also show that the addition of Fe_2O_3 nanoparticles leads to an increase in the dielectric loss coefficient compared to pure epoxy, and this increase increases with the addition percentage. The composite at 0.5 wt% Fe_2O_3 exhibits the highest value for the loss coefficient, indicating increased electrical energy loss within the material due to increased interpolar polarization and the elevated partial conductivity resulting from the presence of the nanoparticles, which is due to an increase in the number of polarized units originating from the interface between the matrix and the reinforcement material (Luo et al., 2022).

3.4 Electrical Conductivity ($\sigma_{a.c}$)

Figure 6 shows the frequency dependence of alternating current conductivity (AC, ($\sigma_{a.c}$)) for pure epoxy (EP) and EP/Fe_2O_3 nanocomposites at different addition percentages (0.1, 0.3, and 0.5 wt%). The conductivity increases progressively with increasing frequency for all samples, a common behavior in polymers and nanocomposites. This is attributed to the activation of charge transport mechanisms and hopping between local states at higher frequencies. The results also show that the addition of Fe_2O_3 nanoparticles leads to a significant increase in conductivity compared to pure epoxy, with this increase increasing with the addition percentage. The composite at 0.5 wt% Fe_2O_3 exhibits the highest conductivity values across the studied frequency range, resulting from increased charge carrier density and improved conductivity pathways within the polymer matrix. At high frequencies, a sharp increase in ($\sigma_{a.c}$) values is observed, especially for samples with higher addition ratios. This is attributed to the enhancement of interfacial polarization and interference of nanoparticles, which reduces barriers to charge movement. The effect of Fe_2O_3 concentration on the percolation effect was shown in the figure. Around Fe_2O_3, a highly immobile epoxy nano-layer developed. As a result, epoxy chains near the immobile epoxy Nano layer surface will prevent charges from traveling through chains and interface zones. The area of the interfacial surface and the diameter of Fe_2O_3 determine the high immobile epoxy nano layer (Chen et al., 2018).

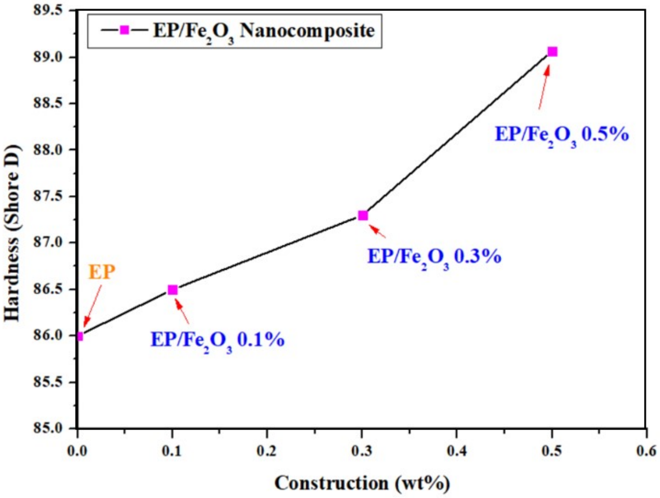

Figure 7. Hardness as a Function of Fe_2O_3 Weight Ratios

3.5 Hardness

Figure 7 shows the effect of adding iron oxide nanoparticles (Fe_2O_3) to the epoxy (EP) matrix on Shore D hardness values. The results show a gradual and consistent increase in hardness with increasing Fe_2O_3 content, compared to pure epoxy. Unreinforced epoxy exhibited the lowest hardness value, while the introduction of a low Fe_2O_3 content (0.1 wt%) resulted in a significant improvement in hardness, indicating the beginning of the restorative effect of the nanoparticles within the polymer matrix. This improvement is attributed to the nanoparticles' ability to restrict the movement of the epoxy chains and reduce localized deformation under load (Kaminsky, 2018). As the Fe_2O_3 content increased to 0.3 wt%, the hardness continued to rise more pronouncedly, reflecting improved stress transfer between the polymer matrix and the nanoparticles, as well as increased interfacial density resulting from the more efficient distribution of the nanofiller. This behavior indicates good compatibility between Fe_2O_3 and epoxy, enhancing the mechanical properties of the composite. At the highest studied percentage (0.5 wt%), the composite achieved the highest hardness value, indicating effective strengthening without any apparent negative effects from nanoparticle agglomeration within this range of additives. This improvement is attributed to the robust role of the Fe_2O_3 nanoparticles in increasing surface resistance to penetration and deformation, as well as enhancing the rigidity of the cross-linked polymer network. When adding iron (III) oxide particles into the epoxy resin matrix, the oxygen molecule from the particle is getting replaced by the OH functional group from the epoxy resin because the Fe-OH bond is more stable than the Fe-O bond. Since the valence of the OH functional group is one, each 1 set of iron particles gets surrounded by six OH functional groups.

Due to this action, epoxy polymer chains come closer and form a cross-linked network. From the six OH, three water molecules are formed, and they fled away in the form of bubbles. Now again the iron particle consists of three oxygen which have a link between the iron and CH, which is in the primary polymer chain (Hardiman et al., 2015). When adding the curing catalyst (TETA), the CH_2-CH_2 functional group from amine reacts with the oxygen and forms cross link, and also hydrogen from amine reacts with the epoxide group and forms another cross-link; hence, the amount of cross links are high. The highest cross-link density is the reason for the high flexural modulus of epoxy after adding iron (III) oxide particles (Prakash and Rajadurai, 2016).

4. CONCLUSIONS

Scanning electron microscopy (SEM) results showed that iron oxide nanoparticles (Fe_2O_3) possess a homogeneous morphology with irregular grains and an average size ranging from 60 to 70 nm, providing suitable conditions for reinforcing the epoxy matrix. The incorporation of Fe_2O_3 nanoparticles into the epoxy resulted in a significant improvement in thermal conductivity. This is attributed to the formation of partial thermal pathways and enhanced phonon transmission with increasing Fe_2O_3 content, without negatively impacting the material's thermal stability. The dielectric properties of the composites showed a marked increase in dielectric constant and dielectric loss with increasing Fe_2O_3 content, exhibiting a strong frequency dependence. This is due to interfacial polarization mechanisms (Maxwell–Wagner–Sillars) and the inability of dipoles to follow the electric field at high frequencies. The electrical conductivity ($\sigma_{a.c}$) results showed a gradual improvement with increasing Fe_2O_3 content, indicating enhanced charge transport mechanisms and the formation of efficient conduction pathways within the polymer matrix, with a bonding threshold effect emerging at higher percentages. The Shore D hardness test results recorded a consistent increase in hardness with increasing Fe_2O_3 content. This is attributed to the restriction of epoxy chain movement, increased interfacial crosslinking density, and improved stress transfer between the matrix and the nanoparticles. The study confirmed that adding low percentages of Fe_2O_3 nanoparticles (up to 0.5 wt%) is an effective way to improve the thermal, electrical, insulating, and mechanical properties of epoxy, making EP/Fe_2O_3 composites strong candidates for advanced multifunctional engineering applications.

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