Volume 17, Number 2, 2024. pp. 245-252

Jordan Journal of Physics

ARTICLE

Dielectric Properties $ZnFe_2O_4$ Nanofiller on the Commercial Epoxy Composites

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Doi: **https://doi.org/10.47011/17.2.11**

Received on: 10/07/2023; *Accepted on: 09/10/2023*

Abstract: Epoxy is an eco-friendly polymer with excellent insulation properties that can be utilized to encapsulate and protect electronic components. In this analysis, the $ZnFe_2O_4$ nanoparticle was successfully synthesized by a simple physical method taking ZnO and Fe_2O_4 as precursors. The pure epoxy sheet was formed by the solution casting method and subsequently, nanocomposite sheets were produced. Fourier transform infrared of pure epoxy reveals the presence of polymeric groups and using a similar approach the other composite samples were analyzed. The spectrum of composite samples has a slight shift in the absorption band due to the $ZnFe_2O_4$ nanofiller. The dielectric loss, dielectric constant, and AC conductivity values show the influence of metal-based nanoparticle $ZnFe_2O_4$ incorporation compared to the pure sample and its activity corresponding to temperature and frequency. The results have proven that the prepared nanocomposite can accumulate electrical energy and can be utilized as a dielectric material.

Keywords: Epoxy polymer, FTIR, Dielectric constant, Dielectric loss, Polymer nanocomposites.

1. Introduction

A polymer is a large molecule that consists of repeated structural units linked by covalent bonds. Polymer composites are commercially produced for a wide range of applications, including flooring, sporting goods, aerospace components, automobiles, and so on [1]. Polymers can be classified based on their origin into natural polymers, synthetic polymers, and an intermediary group known as semi-synthetic polymers [2]. Polymer and composite materials are significantly lighter than traditional metals. In chemically harsh environments, polymer materials perform far better than metals enhancing the longevity of aircraft and reducing repair costs associated with corroding metallic components [3]. Polymers have low specific gravity and specific strength, low coefficient of friction, high corrosion resistance, low density, low cost, low mechanical behavior, poor temperature resistance, low tensile strength, and the ability to be produced transparently or in various colors [4]. Epoxy is a type of thermosetting polymer that is often used as an adhesive or coating material. It is created by mixing two components: a resin and a hardener. When these two components are combined, a

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chemical reaction occurs, resulting in a rigid and durable material. Epoxy resins, also known as polyepoxides, are a type of reactive prepolymers and polymers containing epoxide groups [5]. Due to their excellent adhesive properties, high strength, and resistance to heat, chemicals, and moisture, epoxy or epoxy-based composite materials have a wide range of load-bearing applications. Metals, plastics, wood, glass, and concrete can all be bonded together with it. Because of its low cost and eco-friendliness, epoxy is widely used in construction, manufacturing, automotive, aerospace, marine, as well as DIY projects [6].

Since the advent of nano-fillers, which show significant improvements over micrometer-sized fillers in epoxy systems, nano-based nanodielectric systems have been the subject of increased research attention for their electrical properties. Unlike standard epoxy compounds, only a small amount of nanoparticle dispersant is needed to improve the properties of epoxy composites, making them suitable for aerospace applications [7]. The interesting dielectric properties of epoxy-based nanostructures are associated with a large volume fraction of functionalities in the material bulk and the resulting interactions between polarized nanoparticle surfaces and epoxy chains. Polymer (epoxy) nanocomposites have potential uses in energy storage systems, particularly when high dielectric constants are required [8]. When conductive fillers are dispersed in an epoxy nanocomposite system, variations in electrical conductivities and low diffusion thresholds are seen. The smaller the particle size, the more particles can be dispersed in the epoxy matrix, which influences the polymer's properties through the incorporation of metal nanoparticles. The suggestive applications of zinc and iron in epoxy polymers include insulation materials and coatings for electronic devices. The production methodology for polymer nanocomposites represents a straightforward approach for bulk material production, resulting in enhanced stability and reusability. Ferrite, as a magnetic material, finds applications in magnetic devices, adsorbents, batteries, etc., while zinc is used in paints, batteries, cosmetics, and electronic devices. In this work, the epoxy/$ZnFe_2O_4$ nanocomposite is fabricated to improve its dielectric properties which can be used in high-voltage insulation.

2. Experimental Details

2.1. Materials

Epoxy resin LY556 and hardener HY951 from Araldite were commercially purchased for the experiment. The metal mold of rectangular shape was utilized for the production of pure/composite sheets. High-purity zinc oxide and ferrite oxide nanoparticles were purchased for the synthesis of the zinc ferrite nanoparticle.

2.2. Synthesis of $ZnFe_2O_4$ Nanoparticle

The zinc ferrite nanoparticle ($ZnFe_2O_4$) was prepared using a simple chemical method. High-purity ZnO (99.5%) and Fe_2O_4 (99.5%) powders were carefully weighted in stoichiometric proportions and thoroughly hand-grounded in an agate mortar and pestle for about 1 hour. The dry mixture powder was calcined at the temperature of 700˚C for 2 hours. The calcined $ZnFe_2O_4$ nanoparticle was again ground to a fine powder.

2.3. Preparation of Pure Epoxy Sheet

The epoxy resin and hardener taken in a ratio of 10:1 were stirred for 10 minutes by a mechanical stirrer at low speed to reduce air molecules. The mixed solution was kept in the vacuum desiccator to eliminate the air bubbles. Finally, the clean solution was poured into a metal mold and left undisturbed for 24 hours at room temperature. The cured sheet inside the mold was kept in a hot air oven at 100 °C for 2 hours. Once the mold cooled to room temperature, the neat epoxy sheet was separated.

2.4. Nanocomposite Sample Preparation

Nanocomposite sheets were also prepared using a solution-casting method. The synthesized zinc ferrite ($ZnFe_2O_4$) nanoparticles were vacuum dried to avoid lumps in the sample. Initially, epoxy resin and hardener were taken in two different beakers in a ratio of 10:1 and both were degassed at 40˚C for 2 hours. In the mechanical stirrer, the nanoparticles were mixed with the epoxy at a speed of 700 rpm for 8-10 hours for uniform dispersion/mixing. After complete mixing the beaker was sonicated for 60 minutes at room temperature. Finally, the appropriate amount of hardener was added and mixed vigorously for a few minutes. The mixture was kept in a vacuum desiccator for a few minutes to remove air and poured into the mold. The mold was left for 24 hours, and the next day it was kept in an oven at 100˚C for 2 hours for curing. Likewise, sheets were prepared for 3wt.% and 5wt.% nanocomposites. A photograph of the prepared sheets of pure epoxy and $ZnFe_2O_4$ nanofiller-embedded epoxy composite is shown in Fig. 1.

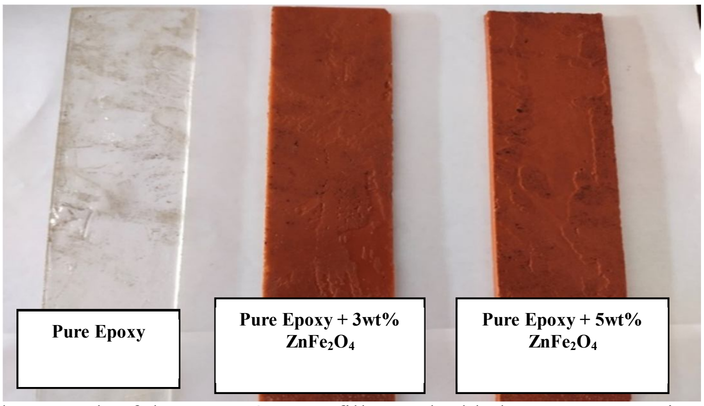

FIG.1. Photograph of the $ZnFe_2O_4$ nanofiller-embedded epoxy composite samples.

3. Results and Discussion

3.1. Fourier Transform Infrared Spectroscopy

Fourier transform infrared spectroscopy was carried out to characterize the functional groups of prepared pure epoxy and epoxy-$ZnFe_2O_4$ nanocomposite samples. The qualitative FTIR analysis was performed using the SHIMADZU IR Affinity–1 (FTIR spectrophotometer) spectrometer in transmittance mode. The study was conducted in the range of 4000 cm^{-1} to 400 cm^{-1} with a resolution value of 4. The obtained sheet was scrapped to achieve powder which was pressed into pellets with a KBr mixture. The FTIR spectra for all prepared samples are shown in Figs. 2-4.

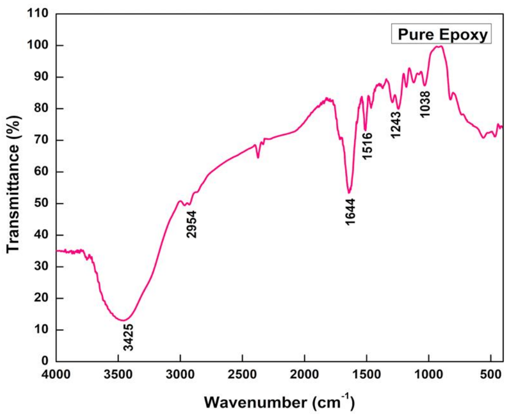

FIG. 2. FTIR spectrum of pure epoxy.

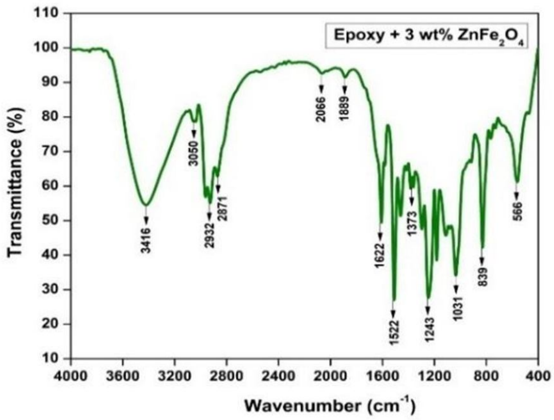

FIG. 3. FTIR spectrum of epoxy+3wt% $ZnFe_2O_4$ nanocomposite.

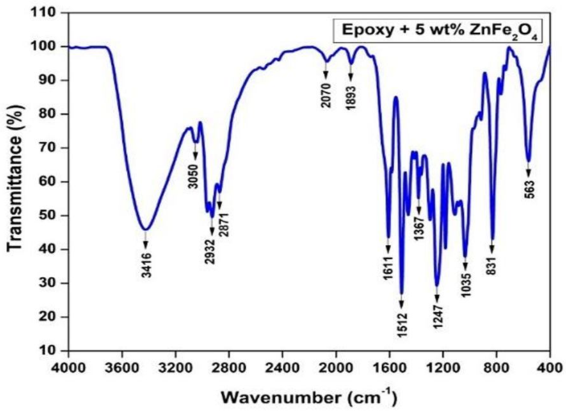

FIG. 4. FTIR spectrum of epoxy +5wt% $ZnFe_2O_4$ nanocomposite.

The FTIR data of all three prepared sheets are given in Table 1. In the FTIR spectrum of pure epoxy the band at 3425 cm^{-1} corresponds to the vibration of the hydroxyl group, revealing the existence of linkers or species with a high molecular weight. The band at 2954 cm^{-1} corresponds to the asymmetric C-H stretching of the CH_3 group [10]. The band at 1882 cm^{-1} corresponds to the overtone band in the vibration spectrum. The band at 1644 cm^{-1} corresponds to the C=O stretching of aromatic rings [9]. The appearance of the band at 1243 cm^{-1} indicates C-N amine stretching. In the FTIR spectrum of the epoxy nanocomposite with $ZnFe_2O_4$ nanofiller, similar bands to those in pure epoxy are observed, along with additional bands found due to the influence of nanoparticles [10]. Some secondary amine groups in the range 1500 cm^{-1} are observed at lower wave numbers. A slight shift in absorption bands is observed in the $ZnFe_2O_4$ nanofiller-added epoxy systems. This is due to the strong attraction of $ZnFe_2O_4$ nanoparticles with epoxy [11]. The homogeneous mixture of all weight percentages of nanofillers is observed from the obtained spectrum.

TABLE 1. Frequency assignments for pure epoxy, epoxy + 3wt.% $ZnFe_2O_4$, and epoxy + 5wt.% $ZnFe_2O_4$.

Wave Number (cm^{-1})			Assignments
Pure Epoxy	Epoxy + 3wt% $ZnFe_2O_4$	Epoxy + 5wt% $ZnFe_2O_4$	
3425	3416	3416	O-H stretching
-	3050	3050	Stretching of C-H of the oxirane ring
2954	2932	2932	Asymmetric C-H stretching of CH_3 group
-	2871	2871	Asymmetric C-H stretching of CH_2 group
-	2066	2070	N=C=S stretching
-	1899	1893	Overtone
1644	1622	1611	C=O stretching of aromatic rings
1516	1522	1512	C-C stretching vibration in aromatic
-	1373	1367	O-H bending
1243	1243	1247	C-N stretching in amine
1038	1031	1035	Symmetrical C-O-C esther
-	839	831	C-H out of plane deformation in aromatic
-	560	563	Bending vibration of C-H

3.2. Dielectric Analysis

3.2.1 Dielectric Constant

Dielectric spectroscopy is based on the phenomena of electrical polarization and electrical conduction in materials. In the present work, the relative permittivity and the loss tangents (tan δ) are determined from dielectric measurements using the HIOKI 3532-50 LCR HiTESTER, over a frequency range of 10^2 – 10^6 Hz, at a temperatures from 150°C to 40°C. For testing, the sample was cut into dimensions of 7.5 × 6 × 4 mm. The applied voltage was set to 1 V and during all the measurements, room temperature was maintained. The data of dielectric constant at various frequencies are given in Table 2.

TABLE 2. Dielectric constant at various frequencies.

Temperature (°C)	Dielectric constant of pure epoxy				Dielectric Constant of epoxy + 3Wt% $ZnFe_2O_4$				Dielectric Constant of epoxy + 5Wt% $ZnFe_2O$			
	1KHz	10KHz	100KHz	1MHz	1KHz	10KHz	100KHz	1MHz	1KHz	10KHz	100KHz	1MHZ
150	0.8511	0.5156	2.2149	1.6585	2.8590	1.6583	2.2145	2.5563	3.6883	1.5302	2.7901	3.8956
140	0.7373	0.4634	0.5422	1.6155	2.8345	1.6537	2.1860	2.5453	3.3929	1.5229	2.7453	3.6453
130	0.6282	0.3708	0.5091	1.9135	2.7221	1.6040	2.1551	2.5348	3.0452	1.4832	2.7351	3.4204
120	0.6224	0.3299	0.4801	1.8698	2.6659	1.4166	2.1405	2.5303	2.9226	1.4321	2.6906	3.3843
110	0.5116	0.3148	0.4768	1.8520	2.5406	1.3768	2.1226	2.5275	2.7456	1.3429	2.6714	3.2844
100	0.4313	0.2755	0.4465	1.8406	2.3447	1.3045	2.1089	2.5184	2.7351	1.2983	2.6314	2.9215
90	0.3717	0.2387	0.4287	1.8183	2.2859	1.1959	2.0981	2.5007	2.5439	1.1483	2.4500	2.8635
80	0.3513	0.2257	0.4101	1.8029	2.1396	1.0599	2.0135	2.4569	2.3425	0.9423	2.3162	2.8083
70	0.2737	0.1943	0.4117	1.7870	1.8817	0.0952	1.9810	2.4412	1.9423	0.7948	2.2274	2.7756
60	0.1996	0.1786	0.3641	1.7674	1.7796	0.8572	1.9542	2.4309	1.7924	0.5243	2.1927	2.7395
50	0.1035	0.1032	0.334	1.7739	1.4873	0.5181	1.9364	2.4111	1.6428	0.3481	2.1796	2.6686
40	0.0334	0.0753	0.308	1.7257	1.3753	0.4832	1.9059	2.3154	1.5496	0.1178	2.0733	2.6036

The graph of dielectric constant corresponding to temperature and frequency is given in Figs. 5 and 6, respectively. The dielectric constant of unfilled epoxy varies from that of nano-filled epoxy concerning both temperature and frequency. As can be seen in Fig. 5, the dielectric constant increases with the increase in temperature for all tested nanocomposites. Pure epoxy has a low dielectric constant; however, the addition of metal oxide fillers significantly increases the dielectric constant. As the frequency increases, the Er value for $ZnFe_2O_4$, also increases, displaying strong ionic polarization, and achieving a maximum value of dielectric constant [12]. Another interesting observation from this study is that 3wt.% and 5wt.% $ZnFe_2O_4$ nanofiller-added epoxy systems have a higher dielectric constant relative to temperature and frequency. This indicates that the metal impacts the dielectric constant when bonded with epoxy. The increase in dielectric constant with the increase of metal is due to interfacial polarization. A mild fluctuation is observed at lower frequencies and temperatures; otherwise, the 5wt.% metal-loaded composite reaches the maximum dielectric constant.

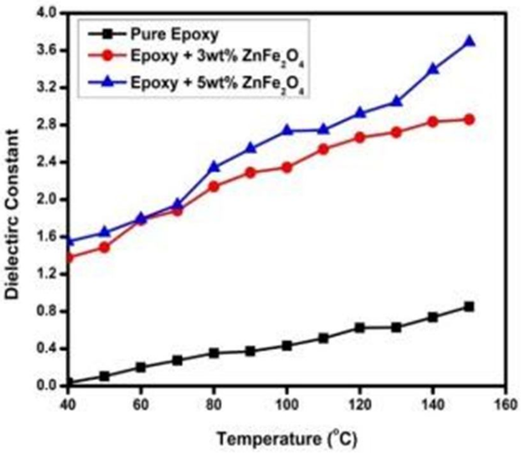

FIG. 5. Dielectric constant vs. temperature at 1KHz.

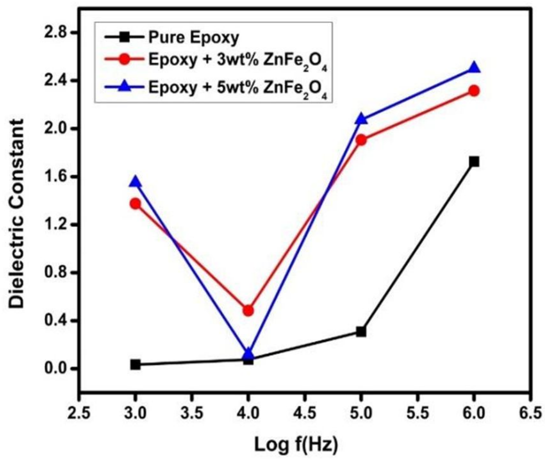

FIG. 6. Dielectric constant vs. frequency at 40˚C.

3.2.2 Dielectric Loss

The dielectric loss depends on the electrical conductivity, which in turn varies with the quantity of charge carriers in the bulk of the material, the relaxation time of the charge carriers, and the frequency of the applied electric field. The dielectric loss values at various frequencies within the temperature range of 40˚C-150˚C are given in Table 3, with the corresponding graphs shown in Figs. 7 and 8.

TABLE 3. Dielectric loss values of epoxy and nanocomposites.

Temperature	Pure Epoxy				Epoxy + 3% $ZnFe_2O_4$				Epoxy + 5% $ZnFe_2O_4$			
(˚C)	1 KHz	10KHz	100KHz	1MHz	1KHz	10KHz	100KHz	1MHz	1KHz	10KHz	100KHz	1MHz
150	0.0982	0.0487	0.0869	0.0087	0.1047	0.1921	0.0713	0.0247	0.2843	0.0548	0.0145	0.0249
140	0.0877	0.0474	0.0828	0.0086	0.0988	0.0973	0.0704	0.0235	0.2596	0.0523	0.0139	0.0239
130	0.0763	0.0453	0.0782	0.0071	0.0773	0.0773	0.0593	0.0228	0.1874	0.0479	0.0103	0.0232
120	0.0753	0.0427	0.0754	0.0068	0.0754	0.6768	0.0573	0.0227	0.1827	0.0454	0.0101	0.0294
110	0.0724	0.0395	0.0672	0.0064	0.0721	0.0716	0.0492	0.0226	0.1663	0.0382	0.0100	0.0251
100	0.0668	0.0382	0.0634	0.0060	0.0675	0.0674	0.0475	0.0225	0.1642	0.0299	0.0054	0.0240
90	0.0571	0.0369	0.0593	0.0059	0.0557	0.0473	0.0462	0.0217	0.1620	0.0190	0.0025	0.0230
80	0.0442	0.0352	0.0493	0.0056	0.047	0.0392	0.0354	0.0187	0.1483	0.0185	0.0022	0.0195
70	0.0323	0.0339	0.0489	0.0043	0.0449	0.0221	0.0279	0.0157	0.1398	0.0172	0.0021	0.0173
60	0.0301	0.0316	0.0477	0.0040	0.0400	0.0198	0.0175	0.0153	0.1368	0.0045	0.0020	0.0108
50	0.0289	0.0297	0.0465	0.0037	0.0398	0.0171	0.0153	0.0135	0.1283	0.0023	0.0019	0.0098
40	0.0271	0.0258	0.0458	0.0032	0.0286	0.0021	0.0112	0.0112	0.1148	0.0019	0.0019	0.0086

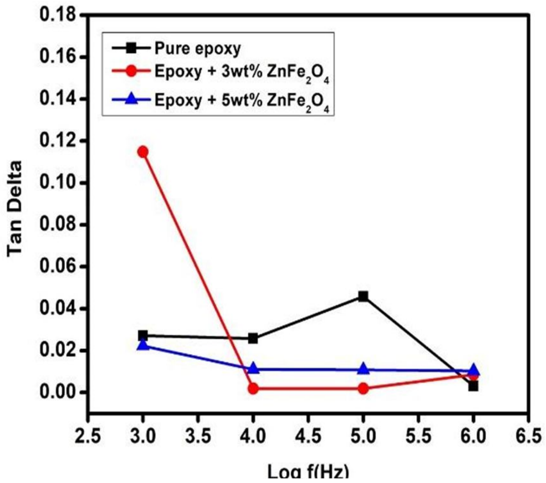

FIG. 7. Variation of Tan delta vs. temperature at 1KHz.

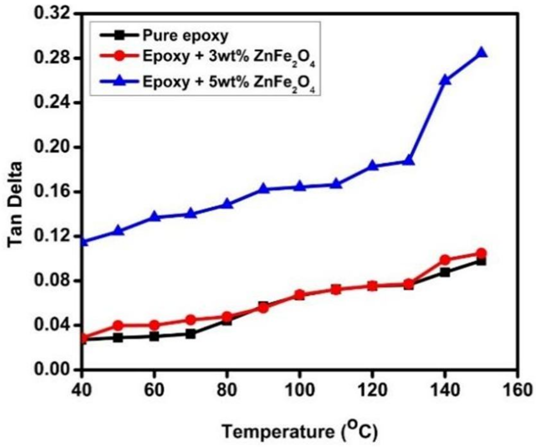

FIG. 8. Variation of Tan delta vs. frequency at 40˚C.

The dielectric loss increases with an increase in temperature and decreases with an increase in frequency for all the tested samples. The dielectric loss values of pure epoxy initially increase and then decrease after 5Hz [13]. The dielectric loss of pure epoxy and minimum quantity $ZnFe_2O_4$ loaded samples shows only slight variations in values relative to temperature. However, the 5 wt.% filler gives greater dielectric loss, which increases with temperature. This observation is most likely due to the presence of a large number of nanoparticles in the system, which affects the electrical conductivity mechanism in nanocomposites. The dielectric loss graph (Fig. 8) shows a greater difference owing to the weight percentage of nanofiller, as the quantity of $ZnFe_2O_4$ is higher. The frequency dependence graph also reveals that a greater filler weight percentage leads to a decrease in dielectric loss relative to an increase in frequency.

3.3 AC Conductivity

The AC conductivity values for pure and $ZnFe_2O_4$-added epoxy nanocomposites are tabulated in Table 4. AC conductivity increases with an increase in temperature, especially particularly for nanocomposites with higher filler percentages. The graphs shown in Figs. 9 and 10 reveal the effect of metal loading over the epoxy concerning temperature and frequency, respectively. The as-taken filler metal $ZnFe_2O_4$ is known for its good conductivity, and retains its properties when combined with epoxy. Thus, dielectric spectroscopy results of pure epoxy and epoxy $ZnFe_2O_4$ nanocomposites show different dielectric behaviors depending on the frequency and the filler concentration [14].

From Fig. 10, it is evident that AC conductivity increases with frequency for all tested nanocomposites. Hence, using a low content of these fillers in epoxy neither improves or worsens the dielectric behavior. For better dielectric values, a higher concentration of metal filler should be included. Adding filler in higher concentration results in increased AC conductivity, as the metal-based compound enhances the conductive mechanism when binding with the insulating polymer [15]. The conductivity graph shows that AC values increase with the increase in temperature and frequency. Higher metal wt.% in epoxy leads to higher conductivity values due to the flawless incorporation of metal and its conductive properties.

In conclusion, the applied electric field, filler permittivity, and the number of nanoparticles all influence the charge transfer mechanisms of the AC electrical absorption process in nanocomposites [16].

TABLE 4. AC conductivity values for pure and $ZnFe_2O_4$ nanofiller added epoxy.

Temperature (°C)	AC conductivity ($\sigma_{a.c}10^{-6}$) mho m^{-1}											
	Pure epoxy				Epoxy + 3wt% $ZnFe_2O_4$				Epoxy + 5wt% $ZnFe_2O_4$			
	1 KHz	10 KHz	100 KHz	1 MHz	1KHz	10 KHz	100 KHz	1MHz	1KHz	10 KHz	100 KHz	1MHz
150	0.0046	0.0139	0.2839	0.8047	0.0173	0.1815	0.8821	3.5581	0.0206	0.0202	0.5918	2.1620
140	0.0036	0.0123	0.2495	0.7695	0.0148	0.0902	0.8577	3.3654	0.0193	0.0192	0.4503	1.6508
130	0.0027	0.0093	0.2211	0.7519	0.0113	0.0710	0.7153	3.2275	0.0175	0.0173	0.4149	1.5901
120	0.0026	0.0078	0.2011	0.7025	0.0107	0.0682	0.6805	3.1881	0.0172	0.0172	0.3786	1.5547
110	0.0021	0.0069	0.1782	0.6633	0.0094	0.0565	0.5804	3.1747	0.16	0.0166	0.3309	0.8849
100	0.0018	0.0058	0.1572	0.6148	0.0086	0.0519	0.5567	3.1506	0.0161	0.0161	0.3301	0.4187
90	0.0016	0.0049	0.1419	0.5912	0.0066	0.0347	0.5387	3.0215	0.0158	0.0157	0.3205	0.3509
80	0.0009	0.0044	0.1129	0.5601	0.0049	0.0252	0.3995	2.5658	0.0125	0.0125	0.2934	0.3131
70	0.0005	0.0036	0.1117	0.4291	0.0044	0.0130	0.3049	2.1247	0.0105	0.0111	0.2571	0.2703
60	0.0003	0.0031	0.0966	0.3939	0.0033	0.0105	0.1933	2.0698	0.0096	0.0100	0.1928	0.2475
50	0.0002	0.0017	0.0863	0.3677	0.0031	0.0085	0.1679	1.8037	0.0073	0.0100	0.1327	0.2362
40	0.00005	0.0011	0.0784	0.3098	0.0020	0.0061	0.1186	1.4451	0.0042	0.0086	0.0849	0.2192

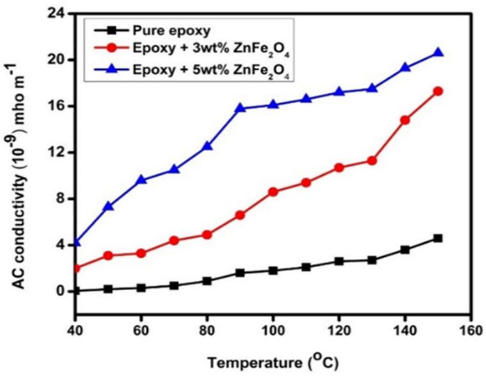

FIG. 9. Variation of AC conductivity vs. temperature at 1KHz.

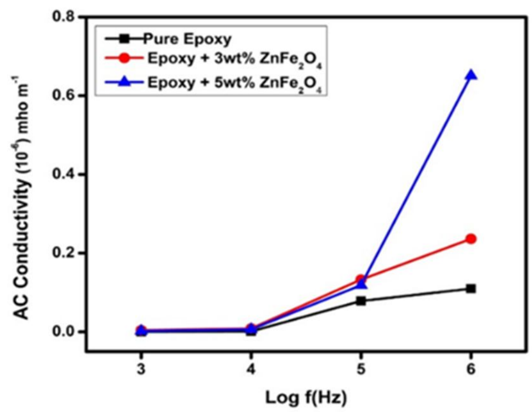

FIG. 10. Variation of AC conductivity vs. frequency at 40˚C.

3. Conclusion

In this work, pure epoxy, epoxy +3wt.% $ZnFe_2O_4$, and epoxy +5wt.% $ZnFe_2O_4$ nanocomposites were successfully prepared using the solution casting method. The dimensions of the developed sheets are approximately 180 × 50 × 3 mm. Fourier transform infrared analysis of samples confirmed the occurrence of epoxy and amine hardeners, as well as their interaction with $ZnFe_2O_4$ nanoparticles. A slight shift in the absorption bands of the nanocomposites was observed for the $ZnFe_2O_4$ nanofiller added to the epoxy system, which is attributed to the strong attraction between $ZnFe_2O_4$ nanoparticles and epoxy. The orientational mode cannot contribute to polarization at low temperatures, leading to a lower dielectric constant. The presence of a significant number of zinc ferrite nanoparticles in the system influences the electrical conductivity mechanism. The AC conductivity increases significantly with an increase in frequency. Dielectric investigation of the produced samples shows that the dielectric constant, dielectric loss, and AC conductivity are frequency and temperature dependent parameters.

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