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Dielectric spectroscopy of Mixed-Nanoparticle loaded EpoxyResin

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ABSTRACT:

In present work, the dielectric measurements of mixed nanoparticle loaded Bisphenol A-(epichlorhydrin); epoxy resin with hardener N(3-dimethylaminopropyl)-1,3-propylenediamine were carried out at room temperature. Mixer of highly pure grades of commercially available uncoated nanoparticle size compound of SiO_2 (APS-20 nm), and of ZnO (APS-30nm) were used as a filler and sample of the neat epoxy resin and nanoparticle loaded epoxy resin in the form of disc were prepared of different weight fraction (i.e 0.5 wt%,0.7 wt%,1 wt%,1.5 wt%,1.7 wt%,2 wt%). Complex permittivity of the prepared samples was measured using Agilent E4980A precision LCR meter in frequency range of 10^3 Hz to 2 MHz. The dependency of dielectric behavior on concentration of mixed nanoparticleand comparative analyses of dielectric properties of all the samples in considered frequency range are discussed in detail.

KEY WORDS: Epoxy Resin,Nanocomposite,Dielectrics, Nanoparticle.

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INTRODUCTION:

Epoxy resin is a one of the most commonly used thermosetting epoxide polymer that contains two or moreepoxide groups. Because of its important impact on mechanical, chemical and electrical properties, epoxy resin become essential materials for industrial applications and widely used such as dielectricmaterials, anti-corrosion materialsand manufacture of composite materials. In recent time,nanoreinforced technology - method of loading nanoparticle to improve properties of epoxy resin become prominent for epoxy resin modification. Normally 0.1% to 5% nanoparticle loaded polymer nanocomposites have been found toexhibit enhanced thermal property, mechanical property, physical property and dielectric property compared to the traditional polymer materials and that tooat low nano-filler concentrations.[1-4]Since the concept of nano-dielectrics based on polymer nanocomposite technology came to be known, limited researcher reported dielectric characteristics of such systems for a better understanding of the behaviorsdepending on the type and concentration of nanofiller. Clear understanding of thepermittivity behaviors in nanoparticle loaded epoxy systems is yet missing and hence this study tries to throw more knowledge into this perspective. [5-8]

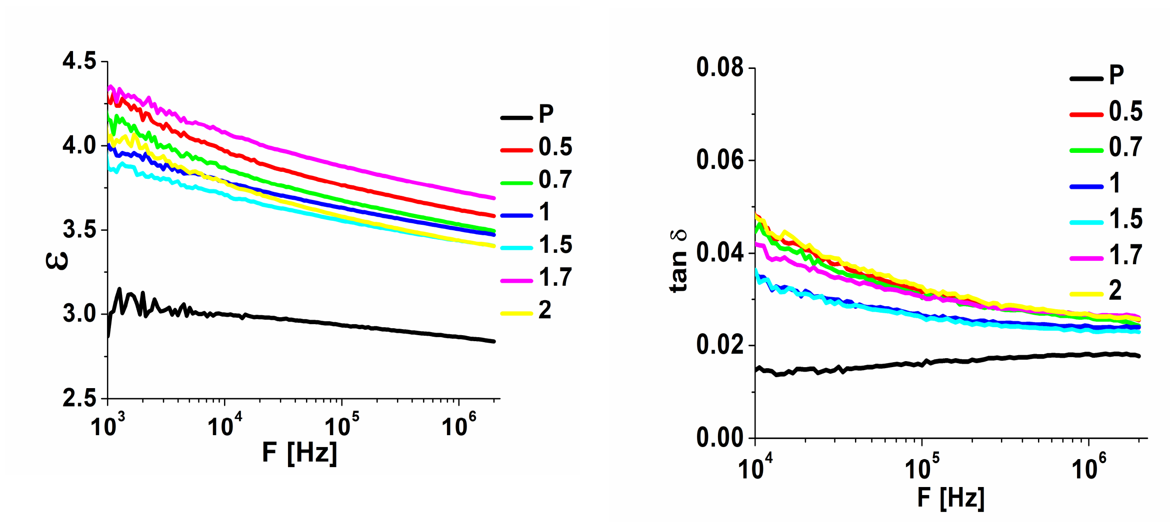
In our previous research paper on epoxy nanocomposites we added $SiO_2$and ZnO nanoparticle distinctly in an epoxy matrix with 0.5 to 2 wt. % concentration in an attempt to study dielectric behavior over a frequency range of 20 Hz – 2MHz at room temperatures and have seen that the relative permittivity for all the weight percentage is higher than unfilled epoxy resin, for epoxy ZnO composite and for epoxy SiO_2composite for 0.5,1 and 1.7 wt. % the dielectric permittivity is lower than the unfilled epoxy resin. This indicated that the presence of nanofillers can lead to higher or lower relative permittivity values compare to the neat epoxy depending on the type and concentration of nanoparticles. [9]

The purpose of the present paper is to study the effects of mixed nano-size fillers on dielectric behaviors of nano epoxy composite. SiO_2 and ZnOmixed nano fillers are used in this study. In this paper we reported complex permittivity of neat epoxy resin and SiO_2 –ZnO mixed nanoparticle loaded epoxy resin in the frequency range of 10^3 Hz to 2 MHz. Influence of filer concentration variation between 0.5 to 2 wt. % on the dielectric behavior of the nanocomposites is studied.

EXPERIMENTAL PROCEDURE:

In the present study we used, bisphenol A-(epichlorhydrin); epoxy resin with hardener N(3-dimethylaminopropyl)-1,3-propylenediamine. (Supplied by Hindustan Ciba Geigy Ltd., Mumbai, India). We procured highly pure grades of commercially available uncoated nanoparticle size compound of SiO_2(APS-20 nm,Product of Otto chemical, India), and ZnO(APS-30nm, Product of SRL chemical, India). For processing a pure epoxy resin sample 100 parts by weight of the resin is mixed homogenously with 80 parts by weight of hardener. And for processing a nanocomposite, the epoxy resin and hardener are taken in two different beakers and then required quantity of mixed SiO_2 -ZnO nanoparticle are mixed to the hardener vigorously with hand for few minutes and then resin and nanoparticle filled hardener was mixed by normal hand stirring method for 15 minutes Finally, the uniformly mixed dough is then slowly decanted into the plastic molds, coated beforehand with wax. The composites were cast in this mold to get disc type specimens. Then they are kept under vacuum desiccation. It was found that a good dispersion could be obtained with this method. [10] The dielectric data of all samples in the frequency range of 10^3 Hz to 2 MHzusing precision LCR meter were obtained. Dielectric measurements were achieved using an Agilent 4980A LCR meter with solid dielectric test fixture Agilent 16451B. [11, 12]

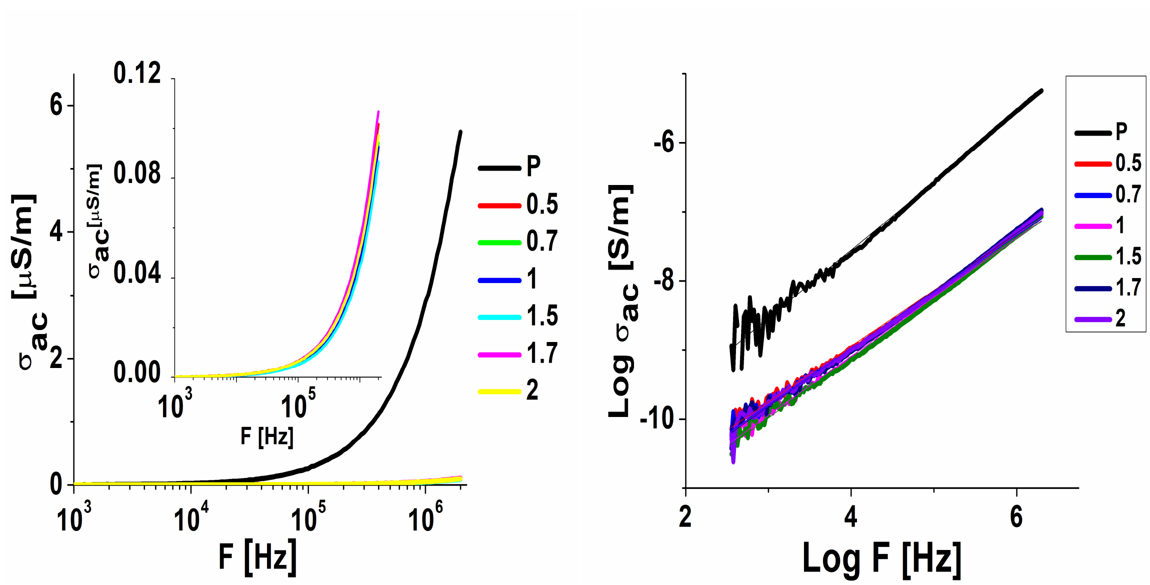
RESULT & DISCUSSION:



(a) (b)

Figure 1- (a) Variations of relative permittivity and (b) Variations of tan δ loss in SiO_2-ZnOmixed nanoparticle loaded epoxy composite in the frequency range of 10^3 Hz to 2 MHz.

For epoxy resin loaded with different weight concentration of mixed SiO_2-ZnO nanoparticles, the variation of dielectric permittivity as a function of frequency is shown in Figure 1 (a). It is clearly seen in figure that effective permittivity decreases with increases in frequency. The same kind of behavior was observed in our earlier study in which SiO_2 and ZnO nanoparticle were mixed separately in epoxy resin. [9]. The values of dielectric constantfor an epoxy resin loaded with mixed nanoparticle for all concentrations is higher than the pure epoxy resin. This result can be attributed to the increase in the polarity of all blends due to the increase of filler concentration, which in turn leads to an increase in the orientation polarization and to the presence of interfacial polarization. [11, 13]Compare to the SiO_2 nanoparticle loaded epoxy resin, SiO_2-ZnO mixed nanoparticle epoxy resin system have high dielectric constant. This is may be due to law dielectric constant of pure SiO_2(about3.9 [14]) than that of ZnO(about 9 at 2 MHz frequency[15]). It is shown in figure 1(b) that for pure epoxy resin tan δ slightly increase with increase in frequency where as in mixed nanoparticle loaded epoxy resin tan δ value decrease with the increase in frequency with no evident of relaxation peak. This kind of behavior suggesting low frequency dispersion.[16,17]



. (a) (b)

FIGURE 2- (a) Variations of ac conductivity in SiO_2-ZnO mixed nanoparticle loaded epoxy composite in the frequency range of 10^3 Hz to 2 MHz.and (b) log [σ_{ac}] versus log[F]

As shown in figure 2 (a) the value of ac conductivity is highest for unfilled epoxy resin in complete frequency range compare to nanoparticle loaded epoxy resin. Increase in ac conductivity in higher frequency region indicates that the charge carries are sufficiently free to follow the change in electric field.The ac conductivity patterns show a frequency independent plateau in the low frequency region and exhibits dispersion at higher frequencies. This behavior obeys the universal power law,

$$\sigma_{ac} = \sigma_0 + Af^n$$

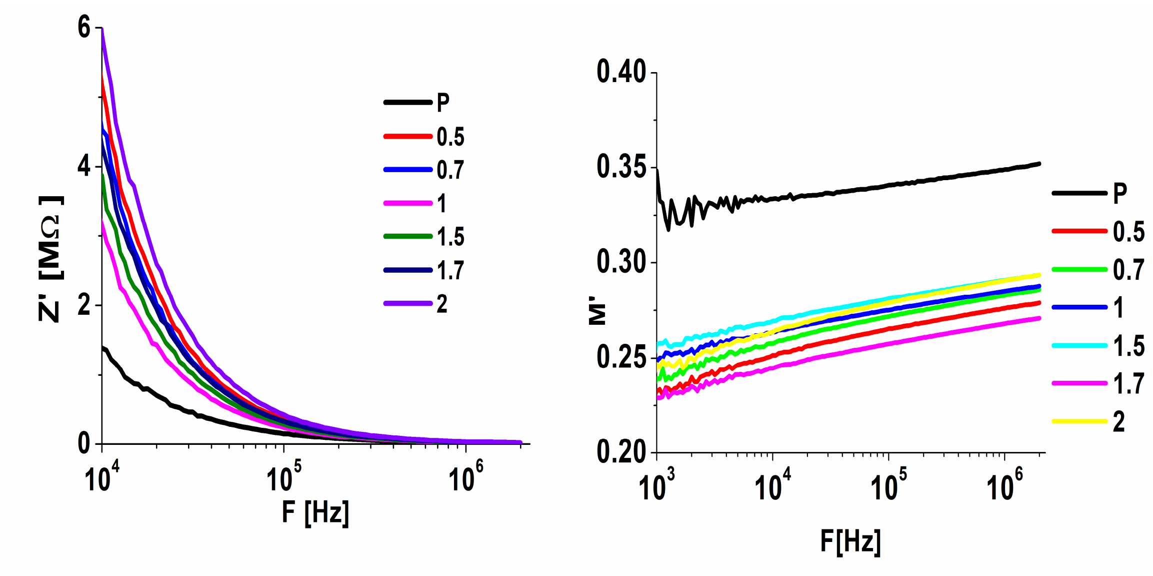
Analyzing the ac conductivity by plotting log [σ_{ac}] versus log[F] (figure2 (b)) under the condition,σ_0 much less than that σ_{ac}, equation can be simplified as

$$\sigma_{ac} = Af^n$$

The values of coefficient A and n were obtained by fitting the σ_{ac} is tabulated in Table 1 and it was found that the value of n is near 1.[18,19,20]

TABLE No. 1. "A and n values for composites with different concentration of nanoparticle"

Sr. No.	Nano particle Weight % in epoxy Composites	Fitting parameter	
		n	A
1	Neat Epoxy	0.9937	-11.535
2	0.5 wt%	0.8092	-12.18404
3	0.7 wt%	0.81786	-12.25134
4	1 wt%	0.85496	-12.51077
5	1.5 wt%	0.86222	-12.56701
6	1.7 wt%	0.83649	-12.32883
7	2 wt%	0.82563	-12.463



(a) (b)

Figure3-(a) Variations of Real part of complex impedance (b) Variations of Electric modules in SiO_2-ZnO mixed nanoparticle loaded epoxy composite in the frequency range of 10^3 Hz to 2 MHz.

Figure 3(a) represents the dependence of the real part of complex impedance on the frequency. The figure shows the exponential decrease in the real part of complex impedance with the increase in frequency for all filler weight fractions. This indicates that the material become more conductive. This behavior indicate the cross link between the SiO_2 - ZnO nanoparticle and the epoxy system. [21-22]It can be clearly seen from figure 3(b) that values of electric modulus M' increased with increases in frequency. It is Because of at higher frequency dipolar groups are not able to orient themselves hence polarization become ineffective at higher frequency. [18]

CONCLUSION:

The effective dielectric permittivity decreases with increase in frequency and the values of dielectric constants for all weight percentage of SiO_2-ZnO mixed nanoparticle loaded epoxy resin is higher than neat epoxy resin, which is due the increase of filler concentration, which in turn leads to an increase in the orientation polarization and to the presence of interfacial polarization. Effective Tan δ value decreases with the increase in frequency with no evident of relaxation peak suggesting "low frequency dispersion". The ac conductivity was found to be higher in neat epoxy compare to SiO_2-ZnO nanoparticle loaded epoxy resin and this ac conductivity behavior obey universal power law model.For all weight percentage of SiO_2-ZnO mixed nanoparticle loaded epoxy resin, thereal part of complex impedance were found to be frequencydependent. The values of electric modules isincreases with increase in frequency, indicate ineffectiveness of polarization at higher frequency.

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