

Dielectric properties of epoxy resin fly ash composite

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Abstract: Epoxy resin is widely used as an insulating material in high voltage applications. Ceramic fillers are always added to the polymer matrix to enhance its mechanical properties. But at the same time, filler materials decreases the electrical properties. So while making the fly ash epoxy composite, it is obvious to detect the effect of fly ash reinforcement on the dielectric nature of the material. In the present research work, fly ash is added to four different weight percentages compositions and post-curing has been done in the atmospheric condition, normal oven and micro oven. Tests were carried out on the developed polymer composite to measure its dielectric permittivity and tan delta value in a frequency range of 1 Hz - 1 MHz. The space charge behaviours were also observed by using the pulse electroacoustic (PEA) technique. The dielectric strength and losses are compared for different conditions.

1. Introduction

The definition of dielectric relates to the permittivity of the material. The permittivity is the ability of a material to polarize with respect to an applied field. Physically, it means the greater the polarization developed from the material in an applied field of given strength, the greater the dielectric constant will be. In earlier days, inorganic materials like mica and silica are used to prepare these dielectric materials. But with the advance of polymeric materials, polymeric materials have taken the place of inorganic materials. This is due to easy process ability, durability and low cost as compared to metallic materials. Inorganic and ceramic materials, have higher thermal properties as compared to polymer composites. Polymers are good insulators but cannot withstand high temperature [1]. Inorganic and ceramic materials have higher dielectric constant than polymeric materials, but when they are affected by the atmospheric moisture, their dielectric property changes drastically. Composites having a high dielectric constant and high dielectric breakdown strength are used for applications of capacitors and electric energy storage devices [2-4]. Pb (Mg^{1/3}Nb^{2/3}) O₃-PbTiO₃ (PMNPT), Pb (Zr, Ti) O₃ (PZT) and BaTiO₃ (BT) have been used as fillers in polymers by various researchers to achieve maximum dielectric and piezoelectric properties [5-11].

Materials having both high and low dielectric are useful in electrical and electronics industries. Low dielectric constants are required as insulators where as high dielectric is required for polarizable media in capacitors. Introduction of small amounts of impurities makes the insulating and nonpolar materials store large amount of energy with a small applied electric field. In the present study effort has been made to utilize industrial waste like fly ash, fabricate a low cost polymer composite and utilize it in electrical and electronics industry. The fabricated composite is cured in different conditions to



enhance the mechanical properties and simultaneously its dielectric properties have been evaluated at different frequencies and different treatment conditions.

2. Experimental details

2.1. Matrix and Filler material

Epoxy resin (LY 556), supplied by ATUL industries is used as the matrix material. It is a high viscous semisolid material whose chemical name is Bisphenol-A-Diglycidyl-ether. An amino group hardener (HY 951) is used for hardening the material. The thermosetting resin formed has high corrosion resistance, high thermal stability and good mechanical properties. Fly ash is collected from CPP-2 of Rourkela Steel Plant, Odisha, India. The SEM micrograph of fly ash show that these are spherical in nature which helps in proper bonding as the intermolecular gap is filled with a layer of epoxy material. A graph has been plotted between the particle size and the volume percentage of fly ash to determine the average particle size and it is found to be 27.26 μm as shown in figure 2. Being a ceramic particle, Si and Al are the main constituents present in the collected fly ash sample as revealed by the chemical compositional analysis as mentioned in Table 1.

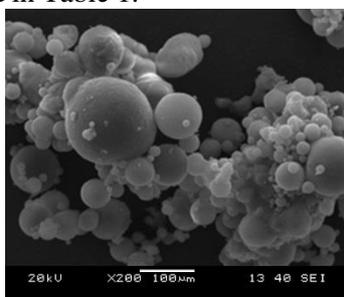


Figure 1. SEM micrographs of fly ash.

Table 1. Chemical composition of fly ash.

Constituents	Vol %
Fe ₂ O ₃	8.1
MgO	1.14
Al ₂ O ₃	24.98
SiO ₂	55.85
P ₂ O ₅	0.15
SO ₃	1.16
K ₂ O	0.85
CaO	2.54
Na ₂ O	0.2
TiO ₂	1.75
CO ₂	1.56

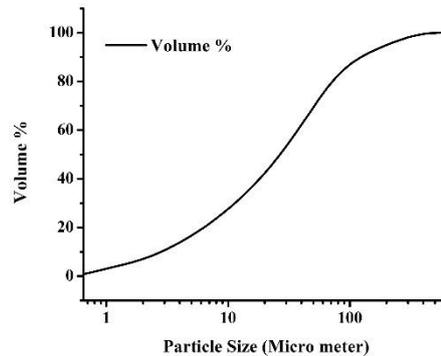


Figure 2. Particle size analysis of fly ash.

2.2. Composite fabrication

Epoxy resin and fly ash are mixed in four different weight percentages and put under an ultrasonic sonicator for 30 min with a pulse time of 5 Sec. Once it is complete, 10wt % of hardener is added to it and hand stirred gently to avoid trapping of bubbles and poured into plastic pipes of 15 mm diameter and 1inch height and allowed to cure at room temperature for 24 hrs. Once it is hardened, the plastic pipes were cut to remove the solidified cylindrical samples. Temperature, pressure, strain rate and even the environment may make an enormous difference in the mechanical response of polymers. So, controlled atmosphere is maintained while making of samples for uniformity. The cylindrical samples are post cured in oven and micro oven in predetermined conditions to enhance its glass transition temperature. The long cylindrical samples after curing are cut into thin slices with polished surfaces for dielectric test.

2.3. Thermal Analysis

Alternating low temperature differential scanning calorimetry is a thermo analytical technique in which the difference in the amount of heat required to increase the temperature of a given sample and that of a reference sample is measured as a function of temperature. It is normally used to find out the glass transition temperature of the samples. Mettler Toledo model no DSC 822 having a temperature range of -100°C to $+400^{\circ}\text{C}$ is used in this experiment. Tg (Glass Transition Temperature), shows the amount of curing due to the post curing treatments and changes in crystallinity of the material.

2.4. Dielectric Analysis

Solartron 1296 Dielectric Interface Instrument is used to measure the dielectric constant of the material. The measurement temperature range varies from 30°C to 150°C and frequency from 100Hz to 1 MHz. To make the sample conductor both sides are coated with silver. The sample is placed between two parallel electrodes. Electric field produces polarization effect in the sample. As an alternating current is passed, it oscillates at the same frequency as that of the applied electric field, but has a phase angle shift δ .

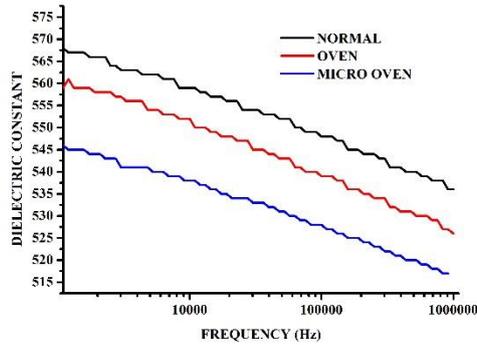
Let C_p = Measured capacitance
 C =capacitance= $\epsilon_0 [A/d]$
 $\epsilon_0=8.8542 \times 10^{-12}\text{F/m}$ = Dielectric constant of vacuum
 ϵ_r =Relative permittivity
 A =Area
 D =Diameter=15mm

$$\text{Dielectric constant} = K = C_p / C$$

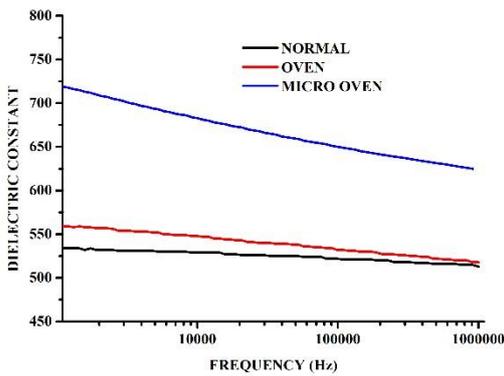
3. Result and Discussion:

3.1. Effect of frequency at different curing conditions on different curing conditions

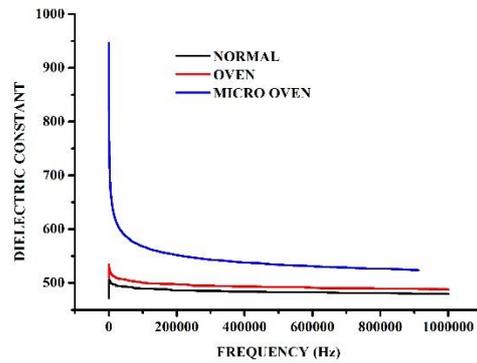
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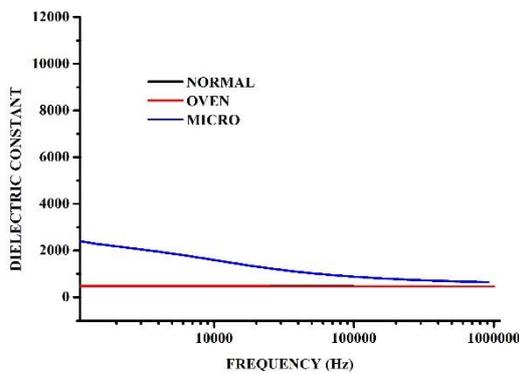
10% FA



20% FA



30% FA



40% FA

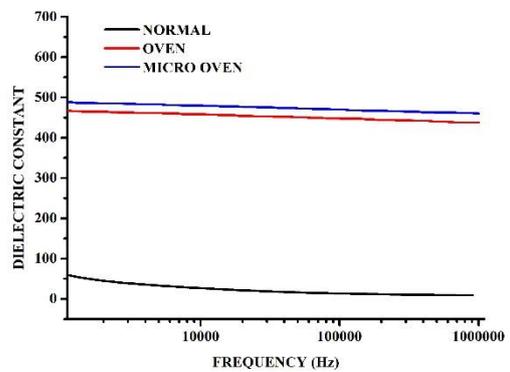


Figure 3. Variation of dielectric constant w.r.t frequency.

Dielectric constant with frequency have been plotted in figure 3 for different percentages of fly ash epoxy resin. In each figure into three different treatment conditions have been mentioned. From the figure, it is clear that the dielectric constant decreases with increasing frequency as it is the expected behaviour for most of the materials. As the frequency increases, charges become more random and start to oscillate out of phase with the applied voltage and will contribute to the alternating current causing decrease in K value. It is also seen that the dielectric constant is in decreasing order for normal, oven treatment and micro oven treatment only for 0% fly ash i.e. only epoxy resin condition. Once the fly ash has been added there is reverse of the order.

It may also be due to the dielectric relaxation which is the cause of anomalous dispersion. At higher frequencies the orientation of polar molecules along the direction of applied field is disturbed. The dielectric properties of a polymer are controlled by the charge conveyance. The polarization of a dielectric is contributed by ionic, electronic and dipole polarization. The electronic polarization happens amid a short interim of time of request of 10-20 sec, however more than for electronic polarization.

Free volume is additionally imperative for deciding the dielectric quality of the material. Free volume is resolved as the space that is not possessed by the polymeric material. The free volume connected with one mole of reshaped units of the polymer may be evaluated by subtracting the involved molar volume of reshaped unit [12].

3.2. Effect of reinforcement of fly ash on dielectric constant

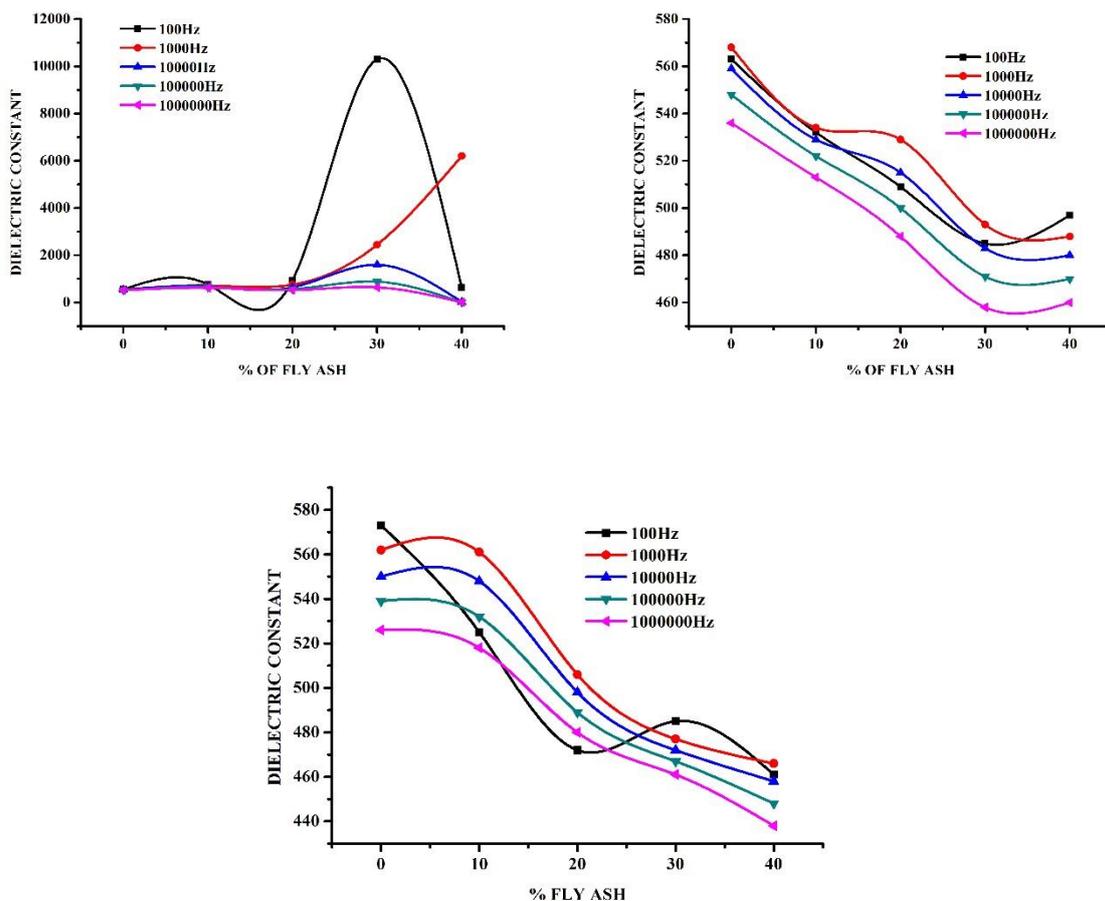


Figure 4. Variation of dielectric constant w.r.t. % of fly ash.

From the Figure 4, it is clear that dielectric constant is less with increase in frequency for a particular percentage of fly ash. The line drawn for 100 Hz frequency only deviate from following a trend. It may be due to the following reason.

Polymers are known to be a blend of shapeless and crystalline districts. In crystalline districts, the chains are methodical organized. Charge transporters are caught at the crystalline-formless interfaces make a huge commitment to the dielectric parameters at lower frequencies.

3.3. Effect of post curing on T_g

The glass transition temperature is the temperature where the material is converted into a glassy state material and its properties are changed. Graphs have been plotted between temperature and amount of heat absorbed. The DSC results are dependent on the heating rate and mass of the sample [13-15].

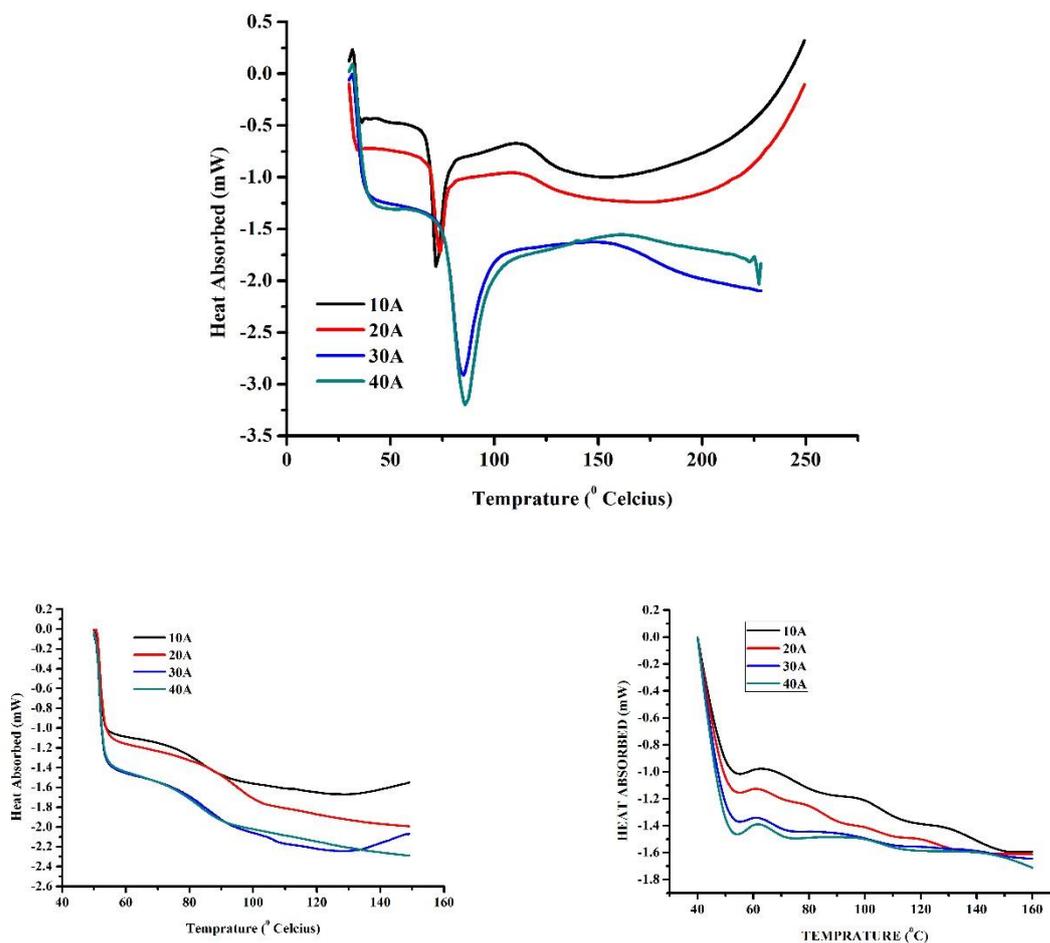


Figure 5. Variation of T_g for different curing condition.

Figure 5 show the T_g and the enthalpy of the material. The curves show the amount of energy required for the particular material to be completely cured. From the above graphs it is clearly evident that the T_g increases after treatment in oven and micro oven. As the T_g increases, the amount of crystallinity increases for the material which is proportional to the dielectric of the material. Figure 5 (a, b, c) shows the DSC curves for different percentages of fly ash for different curing conditions. In all the cases, it follows the trend of decreasing with increase in fly ash content. This may be due to the uniform

coating of epoxy material over each particle of fly ash. As the matrix becomes continuous, the crystallinity, glass transition and the strength increases.

4. Conclusion

Polymers have been widely used in electrical and electronic industries due to its high value of flexural modulus in place of ceramic materials. Ceramic filler like fly ash increases the mechanical properties having good dielectric strength may be good choice of using it in electrical industries. Some of the results obtained from the experiment has been discussed as follows-

1. Post curing increases the crystallinity of the composite which increases the mechanical strength of the material.
2. Dielectric constant of fly ash epoxy composite decreases with increase in frequency.
3. Dielectric strength increases with post curing conditions.
4. Dielectric constant decreases with increase in % reinforcement of fly ash.
5. Fly ash epoxy polymer composite can be used in electrical and electronics industries as a good dielectric material.

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