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# Synthesis and Dielectric Characterization of Nanographite Filled Polyglycerol Adipate (PGA) Nanocomposite

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## Abstract

Nanographite powder (0.5 wt%) filled polyglycerol adipate (NGPGA) nanocomposite was prepared using adipic acid and glycerol as the starting material without any solvent. Polycondensation between equal molar ratio of glycerol and adipic acid using dibutyltin dilaurate as catalyst resulted in a high molecular weight branched aliphatic polyester which is biodegradable. FTIR spectra confirmed the esterification of glycerol by the acid. Completely dried samples were used for frequency and temperature dependent dielectric measurements. It was observed that at ambient temperature both the samples did not show any frequency dispersion. However, nanographite filled nanocomposite showed a higher dielectric constant than unfilled PGA polymer. This was attributed to the increase in the number of microcapacitors due to addition of nanographite fillers. Temperature dependent dielectric behaviour showed no appreciable change in the magnitude of dielectric constant till 50-60ºC. These enhanced dielectric properties of nanocomposites, if combined with a high dielectric strength, render them as a potential material for energy storage devices like dielectric capacitors.

**Keywords**: Polyglycerol Adipate nanocomposite, dielectric, FTIR

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## 1.0 Introduction:

Glycerol is obtained as a by-product during the production of biodiesel from castor oil. Because future demand may increase manifold for bio-based fuel, it is expected to be available in abundance in future [1]. Since glycerol is a triol, it can act as a monomer for the synthesis of branched aromatic and aliphatic polyesters using carboxy diacids via enzymatic catalysis or metal catalysts at high temperature respectively. The three -OH groups of glycerol differ in their level of reactivity. Hence, firstly acid reacts vigorously with the primary -OH groups and later when primary OH groups exhaust, the esterification of secondary -OH groups lead to crosslinking between linear chains. The ester bonds being unstable they are susceptible to degradation. Thus, properties such as non-toxicity, low cost, biodegradability make biodegradable polyester including Polyglycerol adipate attractive for specific applications such as drug delivery, membranes, additives for polyurethanes etc. [2]. Dielectric applications of such biodegradable composites as protective layers or EMI shielding have also been reported [2,19]. Dielectric characterization of such composites assume significance because according to Maxwell's equations, material's response to electromagnetic wave is determined by electrical permittivity ($\varepsilon$), magnetic permeability ($\mu$), and electrical conductivity ($\sigma$) [2,21]. For low-conductivity materials, such as polymers and polymer composites, their behaviour can be evaluated by complex permittivity ($\varepsilon = \varepsilon'-j\varepsilon''$) and complex permeability ($\mu = \mu'-j\mu''$). The real parts of permittivity and permeability indicate the stored portion of the energy exchange between the fields and the material while the imaginary parts describe the energy dissipation which happens when the electromagnetic energy is absorbed by the material and converted to heat [21]. Loss of energy ($\varepsilon''$) also indicates the degree of dispersion in conductive fillers loaded polymer nanocomposites. An exhaustive literature survey on enhanced dielectric properties of polymers filled with carbon nanofillers can be found in ref. [23,24].

In this paper, we have reported the effect of nanographite filler on the temperature and frequency dependent dielectric behaviour of polyglycerol adipate polymer at low frequency (100 Hz to 100 kHz) and low temperature (<60°C).

## 2.1 Materials and method

Analytical grade (AR) Glycerol and Adipic Acid were procured from CDH, India while dibutyltin dilaurate was obtained from Sigma Aldrich. These chemicals were used in as received form without distillation. Slow evaporation of resinous solution was carried out in a furnace.

### 2.1.1 Synthesis of Polyglycerol Adipate polymer

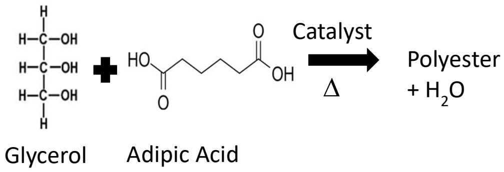


**Figure 1.** Synthesis of PGA polyester

Glycerol is the common name given to 1,2,3-propanetriol (IUPAC name), the simplest polyol, bearing three hydroxyl groups as shown in Fig. 1. PGA polymer was prepared from the polycondensation between glycerol and adipic acid using dibutyltin dilaurate as catalyst. Equal molar ratio of adipic acid and glycerol was used for bulk polymerization. Known quantity of Adipic Acid was dissolved in Glycerol at 100°C on a magnetic stirrer under constant mild stirring till a clear solution was obtained. Small amount of catalyst was added and the temperature of the solution was raised to 150-180°C. Water was distilled out during the process. The solution was further stirred for six hours under vacuum in a rotary evaporator unit. Solution was then poured in a glass petridish and allowed to dry. The solution was then completely dried for at 180°C for 2-3 days. Final polymer was obtained as a thick film. As reported in the literature, a synthesis route (molar ratio of glycerol/ Adipic Acid = 1:1; Processing temperature 130-140°C; catalyst: dibutyltin dilaurate; Processing time: 12 hours) resulted in a branched polymer with an approximate molecular weight of 14000 as shown in Fig. 2 unlike enzymatic route which produces a linear polymer **[3,4].**

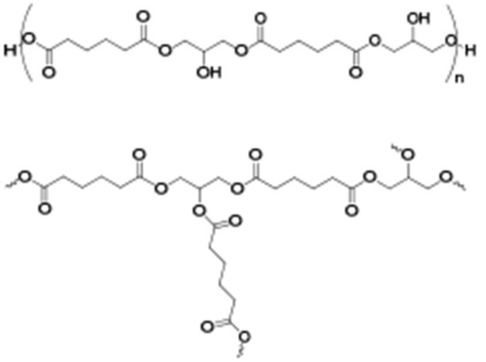


**Figure 2.** Synthesis of linear and branched Polyglycerol Adipate polymer based on catalytic reaction [3,4]

### 2.1.2 Synthesis of nanographite filled Polyglycerol Adipate polymer nanocomposite

Very small quantity of nanographite powder (0.5 wt%) was sonicated in a known quantity of glycerol for 1 min at ambient temperature. A known quantity of adipic acid was added to this solution and heated at 100°C under constant mild stirring till the acid completely dissolved. Small amount of catalyst was added and the temperature of the solution was raised to 150-180°C. Water was distilled out during the process. The solution was further stirred for six hours under vacuum in a rotary evaporator unit. Solution was then poured in a petri dish and allowed to dry. Final polymer nanocomposite was obtained as a thick film.

### 2.2 Structural characterization

FT-IR spectra of the two polymeric system (PGA) and NPGA were obtained to confirm the esterification process. The transmission spectra were obtained in the wavelength range of 400-4000 nm.

### 2.3 Dielectric measurements

Completely dried samples were used for frequency and temperature dependent dielectric measurements. Samples (1 cm x 1 cm x 0.1 cm) were cut from the sheets and electroded using conductive silver. Capacitance (C) and Dissipation factor (loss tangent) were measured on Agilent LCR meter. Dielectric constant was calculated using following formulae

$$\varepsilon = C/C_o; \quad C_o = \varepsilon_o A/d$$

(A is the area of electrode, d is the thickness of sample, $\varepsilon_o$ is the permittivity of free space)

## 3.0 Results and discussion

The FT-IR spectra of the polyesters synthesised in the present work are shown in Fig. 3. The esterification of glycerol is confirmed by the presence of the bands at 1728 $cm^{-1}$ which can be assigned to the ester group. The band at 1712 $cm^{-1}$ indicate unreacted carboxylic group [5,6]. The assignment of the absorption bands is summarized in Table 1. This is in conformation with the reported data [1].

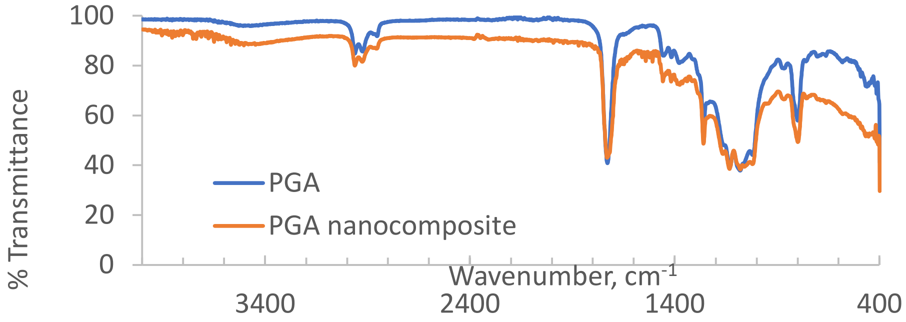
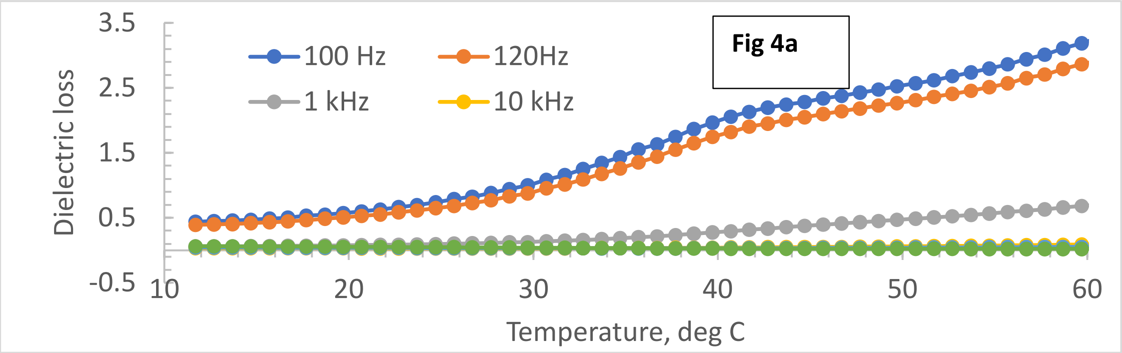


**Figure. 3:** FT-IR spectra of PGA and NPGA nanocomposites

**Table 1:** FT-IR assignment peaks [1]

| Wavenumber ($cm^{-1}$) [Ref 1] | Peak Assignment ($\nu$-stretching; $\delta$-bending) | Wavenumber ($cm^{-1}$) (present work) |
|---|---|---|
| **3300-2500** | $\nu(O\text{-}H)_{\text{alcohol, carboxylic acid}}$ | 3400-2500 |
| **2950-2932-2855** | $\nu(C\text{-}H)_{\text{aliphatic}}$ | 2960-2922, -2852 |
| **1720** | $\nu\ (C{=}O)_{\text{ester}}$ | 1728 |
| **1711** | $\nu\ (C{=}O)_{\text{carboxylic acid dimmer,}}$ free adipic acid | 1712 |
| **1439-1371** | $\delta(C\text{-}H)_{\text{methyl}}$ | 1458-1375 |
| **1215** | $\delta\ (C\text{-}C)_{\text{ester}}$ | 1259 |

The basic properties expected from a material to be used as a dielectric are low dielectric losses, low leakage currents and a high dielectric strength [7,8]. A detailed review is available in the literature on the dielectric behaviour of nanocomposites of biopolyester reinforced with carbon nanotubes [9]. Figures 4-6 show the temperature and frequency dependent dielectric behaviour of PGA and NPGA nanocomposite. Figure 4 (a,b) shows the variation of dielectric loss and dielectric constant respectively as a function of frequency for nanographite filled polyglycerol adipate nanocomposite. Figure 5 (a,b) shows the variation of dielectric loss and dielectric constant respectively as a function of frequency respectively for unfilled polyglycerol adipate nanocomposite. A quick look at these figures shows that with incorporation of nanographite as a filler, both dielectric constant and dielectric loss have increased at all frequencies and at all temperatures. However, the uneven curves at low frequencies (100 and 120 Hz) are attributed to the differences among permittivities and conductivities of continuous (matrix) and discontinuous (filler) phases. Similarly, in unfilled PGA it can be attributed to impurities and unreacted residues during chemical synthesis [10,11]. At a frequency of 1 kHz and above, remarkably higher dielectric constants are observed for nanographite filled PGA compared to unfilled PGA composite at temperatures. This is explained in terms of a build-up of a network of micro-capacitors wherein nanofillers are separated by a thin dielectric polymer layer [12,13]. For a higher filler content, the number of micro-capacitors increases and the insulation distance between them decreases, so that the capacitance of a single micro-capacitor increases. The relative permittivity is about $\varepsilon_r = 50$ for nanocomposite as compared to 8-9.5 in unfilled PGA (Fig. 6). These results are in accordance with the reported increase in the dielectric constant of biodegradable PHBV/PBAT blend with increase in the concentration of MWCNTs, at low frequencies [14], similar to those reported for binary polyvinylidene fluoride/barium titanate nanocomposites [22]. Figure 6 shows the frequency dependent dielectric constant of nanographite filled PGA and unfilled PGA polymer. It is seen that the relative permittivity decreases with frequency because for larger dipoles orienting themselves according to the applied field is difficult and they do not contribute to polarization. The dipoles in the nanocomposites are not able to follow the field variations due to inertia. The polarization due to the charge accumulation will decrease, leading to a decrease in permittivity [15,16]. To summarise, firstly the increase in dielectric constant can be attributed to the homogeneously and uniformly distributed conductive fillers (which acted as electrodes for thin layer of insulating polymer), secondly polarization of matrix due to segmental motion of molecules (dipoles) and matrix/filler interfacial polarization together contributed to the final permittivity of the composite [17,18]. The results obtained in the present work are in accordance with the reported literature albeit it has been done for their studies on EMI shielding which is conducted at frequencies ~ GHz. For instance, it has been reported that dispersing conductive particles into a polymeric matrix changes its response to electromagnetic radiation via altering the EM properties of the polymer. Both magnetic permeability (μ) and electric permittivity (ε), as well, of PLA and PBAT nanocomposites loaded with graphene nanoplatelets (GNP) show different responses. While permeability remained unaffected by addition of GNPs, permittivity increased with GNP loading [19]. Consequently, the intrinsic impedance of the matrix decreases, resulting in higher impedance mismatch at the interface and hence higher reflection of the electromagnetic power. In a similar manner, wave velocity in the prepared nanocomposites decreases as the permittivity increases. This improves the radiation absorption capacity of the nanocomposites [19]. For epoxy/ZnO nanocomposites, it was observed that when the filler concentration was high, ε′ and ε″ were higher. However, when the dispersion was inhomogeneous and non-uniform, ε″ was higher [20]. Based on this finding, authors have concluded that lower values of ε″ indicated better dispersion of GNPs in PBAT compared to PLA [19].



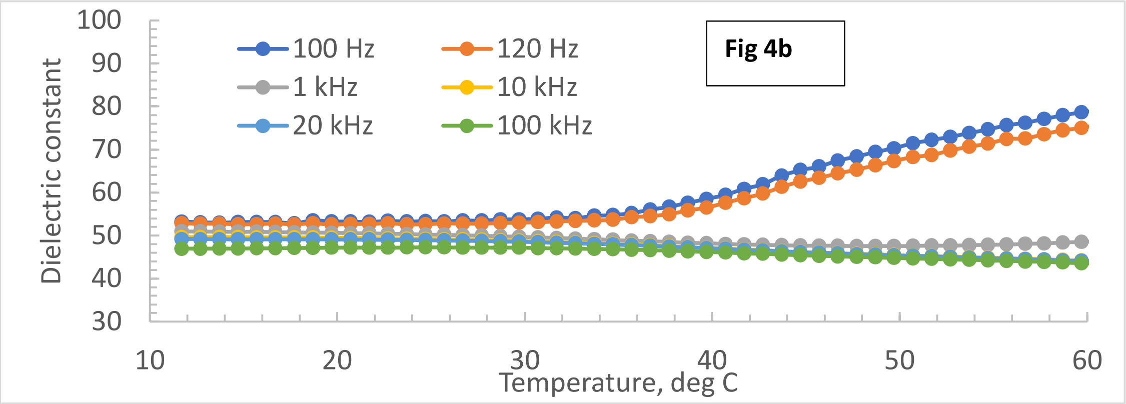
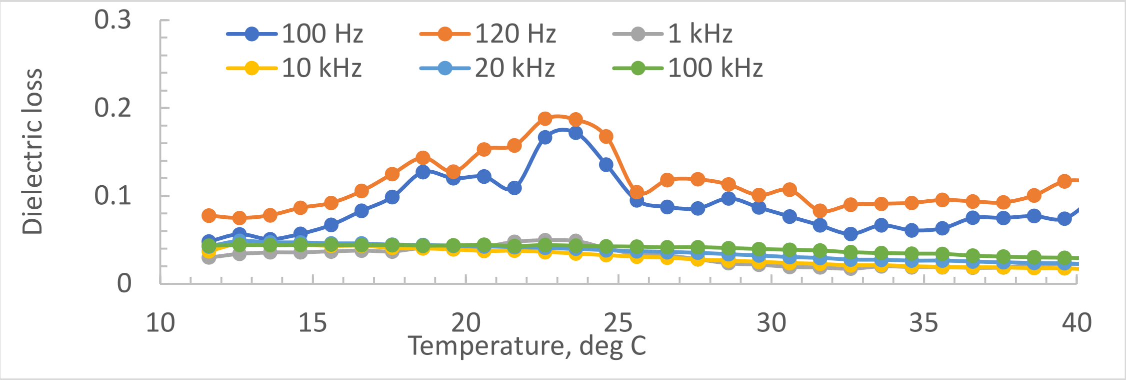


**Figure 4:** Temperature and frequency dependent dielectric behaviour of nanographite powder filled polyglycerol Adipate composite (a) Dielectric loss (b) Dielectric constant



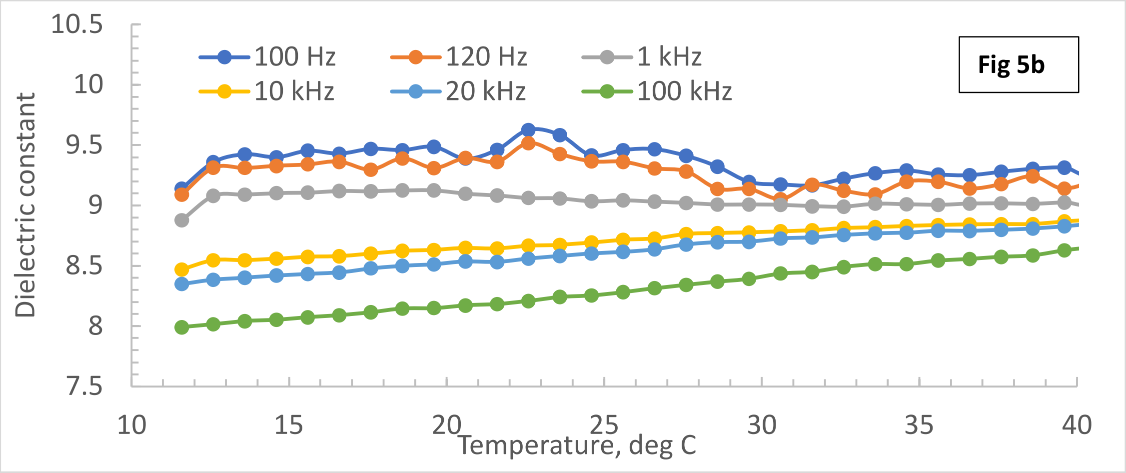


**Figure 5:** Temperature and Frequency dependent dielectric behaviour of unfilled polyglycerol Adipate polymer (a) Dielectric loss (b) Dielectric constant

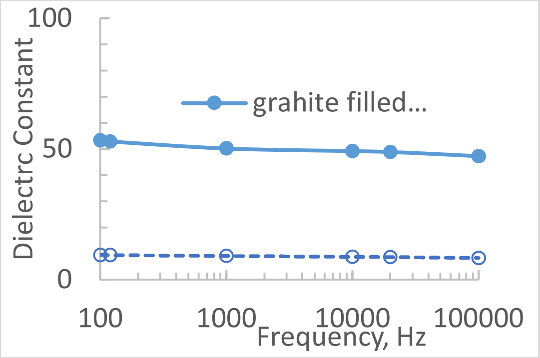


**Figure 6:** Frequency dependent dielectric behaviour of unfilled and nanographite powder filled polyglycerol Adipate composite at room temperature (25°C)

## 4.0 Conclusions

Solvent free synthesis of biodegradable polyglycerol adipate polymer is non-toxic and cost-effective. Dielectric characterization of these composites shows that permittivity of biodegradable polyglycerol adipate increases by adding very small amount of nanographite powder. It has been attributed to the fact that the incorporation of conductive nanographite might have increased the number of micro-capacitors and decreased the thickness of insulation between them. Consequently, the capacitance of a single micro-capacitor increased. Since, energy storage devices such as dielectric capacitors require thermal stability, mechanical integrity and good dielectric properties including high dielectric strength, the nanocomposites explored in the present study seem a promising material for such applications.

## Conflict of Interest

Authors declare No conflict of interest

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