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A new low dielectric constant barium titanate – poly (methyl methacrylate) nanocomposite films

Ravindra H. Upadhyay and Rajendra R. Deshmukh*

Department of Physics, Institute of Chemical Technology, Matunga, Mumbai-400019, India

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Abstract. In the present investigation, nanocomposite films with poly(methyl methacrylate) (PMMA) as a polymer matrix and barium titanate as a filler were prepared by solution casting method. Barium titanate nano particles were prepared using Ti(IV) triethanolaminato isopropoxide and hydrated barium hydroxide as precursors and tetra methyl ammonium hydroxide (TMAH) as a base. The nanocomposite films were characterized using XRD, FTIR, SEM and dielectric spectroscopy techniques. Dielectric measurements were performed in the frequency range 100 Hz-10 MHz. Dielectric constant of nanocomposites were found to depend on the frequency, the temperature and the filler fraction. Dissipation factors were also influenced by the frequency and the temperature but not much influenced by the filler fractions. The 10 wt% of BT-PMMA nanocomposite had the lowest dielectric constant of 3.58 and dielectric loss tangent of 0.024 at 1MHz and 25^0C. The dielectric mixing model of Modified Lichtenecker showed the close fit to the experimental data.

Keywords: BT-PMMA nanocomposite; XRD; SEM; FTIR; dielectric constant

1. Introduction

A technology in which a novel artificial material is fabricated by combining, for instance, ceramic and polymer materials is called composite technology (Sebastien and Jantunen 2010). Ceramic–polymer composites, especially type 0-3, are a potential material group suitable for producing demanding and functional packages that combine the electrical properties of ceramics and the mechanical flexibility, chemical stability and processing possibilities of polymers (Newnham *et al*. 1978, Skinner *et al*. 1978). In polymer nanocomposites nanosized fillers typically less than100 nm are dispersed in an ordered manner or just by mixing (Ajayan *et al*. 2003). Nanocomposites have been found to exhibit enhanced physical, electrical, thermal and optical properties specially at low nano-filler concentrations of 1 to10% as compared to the conventional polymer materials (Messersmith and Giannelis 1994, Gensler *et al.* 2002).

These nanocomposite materials occasionally show the way to unexpected new properties, which are often not exhibited by individual materials and open a new avenue for physicists, chemists and material scientists. They are slowly emerging as excellent functional materials for dielectrics and electrical insulation application and the term “nanodielectrics” for such materials is

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∗Corresponding author, Dr., E-mail: rr.deshmukh@ictmumbai.edu.in

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increasingly becoming popular (Jordan *et al.* 2005, Paul and Robeson 2008, Jancar *et al.* 2010, Bai *et al.* 2000).

Poly(methyl methacrylate) (PMMA) has been considered as a good host material since it is an important amorphous thermoplastic with desirable properties such as transparency, chemical resistance, dimensional stability, good weatherability, protection against ultraviolet radiation and good moldability. PMMA has good resistance to both acidic and alkaline solutions and it is a widely studied polymer for composite fabrications (Mark 1985, Bamfor and Tipper 1975, Gross *et al.* 2007, Htoo 1989). Barium titanate ($BaTiO_3$, BT) is a ceramic material with a perovskite crystal structure characterized by excellent ferroelectric/paraelectric properties. In recent years BT has been utilized for composite preparation in conjunction with various polymers, including PMMA. Besides, it is found to be useful in Multilayered Ceramic Capacitors (MLCC), Micro-Electro-Mechanical Systems (MEMS) and Dynamic Random Access Memory (DRAM) (Setter and Waser 2000) and other applications such as waveguides, ferroelectrics sensors, and luminescent materials (Popielarz *et al.* 2001, Singh and Chandra 2003, Kobayashi *et al.* 2005).

Recently a new method for the preparation of 24 nm particle size with paraelectric cubic structure and having dielectric constant 31 at 10 kHz frequency at room temperature have been reported (Upadhyay *et al.* 2011). Ferroelectric BT has been widely used for nanocomposites.

However, paraelectric nanosized BT has not been reported for polymer nanocomposites. The ceramic nanocomposites with low dielectric constant and low dissipation factor have found applications in microelectronics (George *et al.* 2010). BT with low dielectric constant in microelectronics applications has rarely been reported. Therefore, this work has been undertaken. It was found that at 1 MHz, nanocomposite of 10 wt% BT-PMMA has dielectric constant 3.58 and dissipation factor 0.024 at 25^0C.

2. Experimental details

2.1 Materials

All the chemicals and solvents used were of A. R. grade. $Ba(OH)_2.8H_2O$ and Formic acid of S. D. Fine Chem. Ltd., Mumbai (India), Ti (IV) triethanolaminato isopropoxide of Aldrich, U.S.A., Tetra Methyl Ammonium Hydroxide (TMAH) of SRL, Mumbai (India), Methanol of Merck, Mumbai (India) and Chloroform of Merck, Mumbai (India) were purchased from the local dealer. PMMA having M_W= 82264 was used as the polymer matrix.

2.2 Preparation of BT nanoparticles

BT particles were prepared in the laboratory by sol-precipitation method using Ti(IV) triethanolaminato isopropoxide and hydrated barium hydroxide in alkaline media as reported elsewhere (Upadhyay *et al.* 2011).

2.3 Preparation of PMMA - BT film

PMMA-BT polymer nanocomposites were prepared by solution casting method. Initially, the BT nanoparticles were dried at 90^0C for 6 h before mixing. The PMMA granules were dissolved in chloroform solvent using magnetic stirrer for 1 h then dried nanoparticles of BT in different wt%

(0.5, 1, 2, 4, 7 and 10 wt%) were added to this dissolved solution of PMMA. The resulting mixture was subjected to ultrasonic agitation at a frequency of about 24 kHz for 1 h to ensure uniform dispersion of BT nanoparticles in PMMA polymer. The homogeneous suspension of BT-PMMA was then poured in flat petri dish kept floating on mercury surface to obtain nanocomposite film of uniform thickness. The thin film of nanocomposite having thickness of 30 to 40 micron was peeled off from petri dish after complete evaporation of chloroform at room temperature with the help of doctor blade. These films were preserved in a desiccator for further characterizations.

2.4 Preparation of samples for dielectric study

The prepared nanocomposite films were cut in small sizes and their thicknesses were measured using thickness meter. These films were coated on both the sides with conducting colloidal silver for better electrical contact with the electrodes of sample holder. Samples were then kept in oven for 4 h at 40^0C for removal of solvent in silver paste. The sample was held between the specially fabricated sample holder having two circular tablets type of gold electroplated copper electrodes, which were connected to Impedance Analyzer. The sample holder was kept in a furnace which could be heated to different temperatures and maintained stable within $\pm 1^0$C. The maximum temperature for measurement was limited to 100^0C to avoid any deterioration of nanocomposites during the dielectric measurements.

2.5 Instrumentation

X-ray diffraction (XRD) patterns were obtained at room temperature at 2θ angles from 2^0 to 80^0 with a step size of 0.02, scanning speed 2^0/min using Cu Kα radiation on the x-ray diffractometer (Model Miniflex, Rigaku). The field effect Scanning Electron Microscopy (Fe-SEM) images were recorded using a SEM microscope (Model Ultra plus, Zeiss) for morphological and particle distribution study. The purity of the prepared PMMA-BT nanocomposites were analyzed on a Fourier Transform Infra Red (FTIR) spectroscope (Model spectrum 100, Perkin-Elmer). The dielectric properties were investigated on precision impedance analyzer (Model 6500B, Wayne Kerr) with component fixtures (Model 1011).

3. Results and discussion

3.1 X-Ray Diffraction studies

XRD pattern of pure BT, neat PMMA and nanocomposite films of concentration 4 wt%, 7 wt% and 10 wt% is shown in Fig. 1. XRD pattern of 0.5, 1 and 2 wt% composite films are not shown in the figure because of low visibility of the peak intensities. Careful analysis of the spectra reveal that neat PMMA polymer is amorphous in nature with broad band of peaks at around $2\theta = 17.85$, 31.10 and 42.96 (Tomar *et al.* 2011). The characteristic peaks of neat BT were found to be cubic in structure (JCPDS=31-174). By comparing the XRD pattern of nanocomposites with that of neat BT and neat PMMA, it was observed that the intensity of prominent peak of BT corresponding to $2\theta = 31.22$ increased with increasing concentration of BT in composites. The intensities of all the major peaks of BT are visible in nanocomposite with 10 wt% concentration of BT which suggests

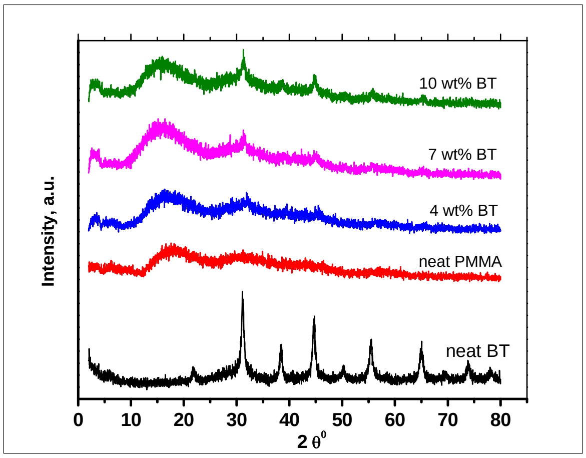
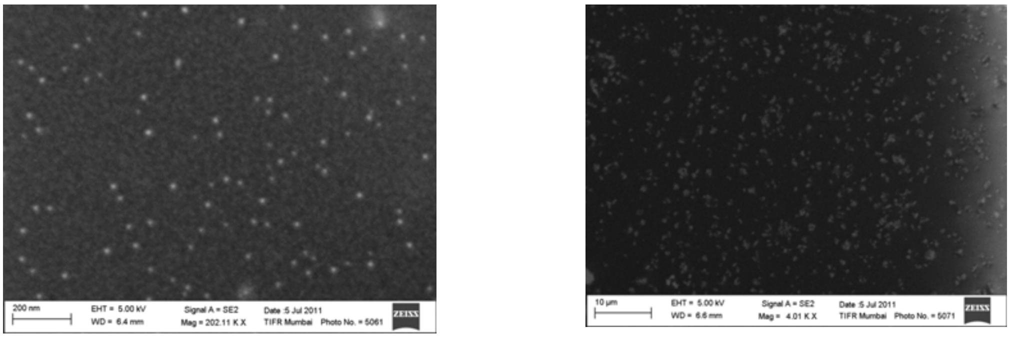
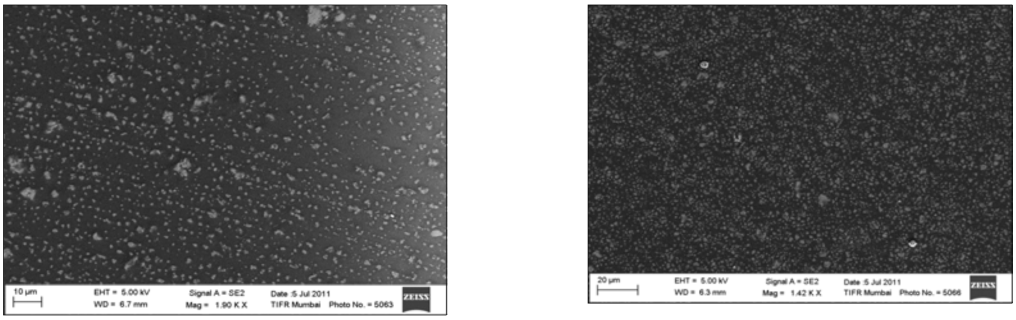


Fig. 1 XRD patterns of the BT-PMMA nanocomposite films with different weight fractions of BT



(a) 0.5 wt% BT-PMMA Nanocomposite (b) 4 wt% BT-PMMA Nanocomposite



(c) 7 wt% BT-PMMA Nanocomposite (d) 10 wt% BT-PMMA Nanocomposite

Fig. 2 SEM micrographs of BT-PMMA films with different weight fraction of BT filler

the presence of BT in nanocomposites. Furthermore, the crystal structure of BT was stable during and after preparation process of nanocomposite.

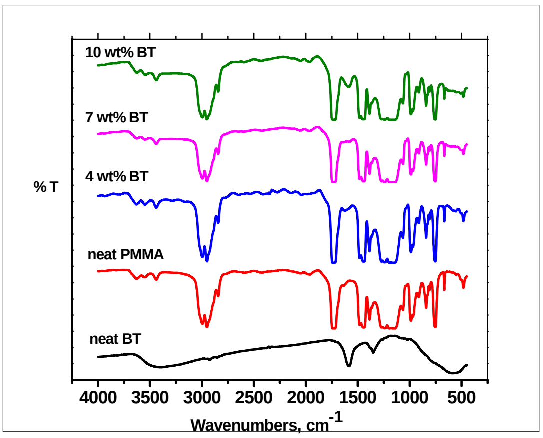


Fig. 3 FTIR transmittance spectra of pure BT nanoparticles and BT-PMMA nanocomposites with different wt% of BT

3.2 Morphological study by SEM

Fig. 2(a) shows the Fe-SEM image of 0.5wt% BT nanoparticles embedded in PMMA. This indicates that the particles are well dispersed in the matrix of PMMA. The size of BT nanoparticles in composite appears to be 24-30 nm, which is same as reported (Upadhyay *et al*. 2011). A higher weight fraction of BT loading in PMMA resulted in the agglomeration in some localized regions. Therefore, SEM micrographs of higher concentrations were taken at lower magnification scale to show the uniform dispersion of nanoparticles over a larger area of the film as shown in Figs. 2(b)-2(d) for 4wt%, 7wt% and 10wt% respectively.

3.3 FTIR analysis of nanocomposites

The FTIR transmission spectra of neat BT, neat PMMA and some composites of different wt% of BT in PMMA are as shown in Fig. 3. The characteristic vibration bands with their probable assignments are tabulated in table No.1. The peak assignments of the FTIR spectra of PMMA were confirmed from the data reported (Haris *et al*. 2010, Tomar *et al*. 2011, Ahmad *et al*. 2007). It was observed that there is no significant difference in positions of the characteristic peaks in the FTIR spectra of BT- PMMA composites and neat PMMA. The characteristic peak at 583 cm^{-1} due to Ti-O stretching and the peak at 1351 cm^{-1} due to CO_3^{2-} of BT were disappeared in composites whereas peak at 1586 cm^{-1} due to OH$^-$ in BT spectra shifted at 1589 cm^{-1}in the spectra of the composites. FTIR bands at 1586 cm^{-1} and 1351 cm^{-1} are due to OH$^-$ and CO_3^{2-} vibrations respectively present as impurities in BT formed due to the adsorption of water and CO_2 from the atmosphere in BT.

The small shift and disappearance of peaks in the characteristic IR frequencies in PMMA- BT nanocomposite clearly suggests the formation of new nanocomposite due to substantial interaction between polymer chain and BT. The IR spectra of nanocomposites suggest that it possessed properties of both due to formation of structural chains between PMMA and BT. It is to be understood that the PMMA-BT nanocomposites had been prepared successfully which further corroborates the results of SEM and XRD.

Table 1 Data of characteristic vibration bands observed in PMMA and BT

PMMA peak Positions (cm^{-1})	Peak assignment of PMMA	BT peak Positions (cm^{-1})	Peak assignment of BT
2997 m	C-H asym. stretching in CH_3	1586 m	OH stretching
2952 vs		1351 m	CO_3^{2-} bending
2841 w	C-H asym. stretching in CH_3	583 m	Ti-O stretching
1729 vs	C=O stretching		
1484 m	O-CH_3 deformation		
1447 ms	CH_3 deformation		
1387 m			
1267 w	C-O stretching		
1239 m			
1191 m	CH_3 wagging		
1141 ms	CH_3 twisting		
1062 w			
991 m	C-C stretching		
967 w			
911 vw	CH_3 rocking		
841 w	CH_2 rocking		
810 vw	C=O inplane bending		
751 m	C=O out of plane bending		
667 w	C-O inplane bending		
482 w	C-C inplane bending		

3.4 Dielectric behaviour of nanocomposite

The dielectric studies of nanocomposites were carried out to see its response to an applied low ac voltage in the frequency range of 100 Hz to 10 MHz at excitation of 1 V and with temperature from 25^{0}C to 100^{0}C.

3.4.1 Frequency dependence of dielectric properties

The impedance analyzer measures the capacitance (C) and dissipation factor (D, also known as loss tangent tan δ) of the sample directly. The dielectric constant (ε'_r, also known as relative permittivity) is calculated using the equation

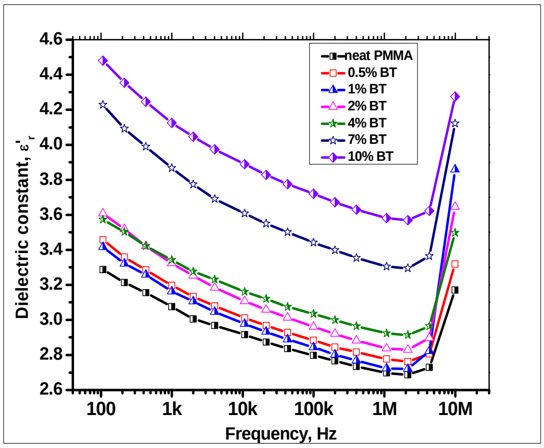


Fig.4 (a) ε'$_r$ as a function of frequency at 25 ^{0}C

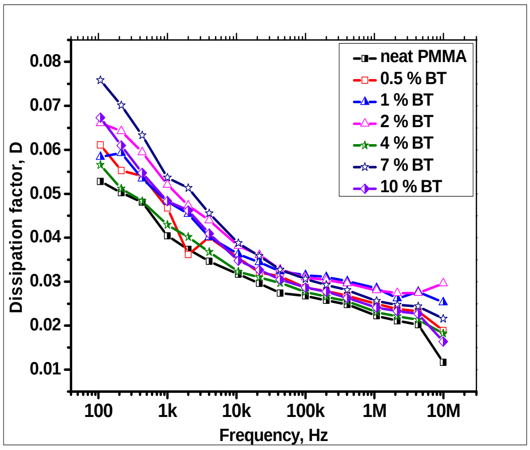


Fig.4 (b) D as a function of frequency at 25 ^{0}C

$$\varepsilon'_r = \frac{t.C}{\varepsilon_0.\pi\left(\frac{d}{2}\right)^2}$$

where, t = thickness of nanocomposite film

C =equivalent parallel capacitance in farad

ε_0 = permittivity of vacuum = 8.854 x 10^{-12} Fm^{-1}

d = diameter of sample holder electrode

ε'_r and D values of composites plotted as a function of frequency for different wt% of BT as filler in a pure PMMA at 25^{0}C are shown in Figs.4(a) and 4(b) respectively. It was observed that ε'_r and D decreased with increasing frequency from 100 Hz to 2 MHz gradually in neat PMMA and nanocomposites. There is 18.24% fall in the value of ε'_r with 60.38% fall in D value in neat PMMA, whereas 20.31% decrease in ε'_r with 65.67% decrease in D was noted in 10 wt% nanocomposite from 100 Hz to 2 MHz. Lowest value of ε'_r = 3.58 and D = 0.024 were noted at 1MHz for 10 wt% nanocomposite at 25^{0}C. The highest value of D is 0.067 (at 100 Hz) and lowest is 0.016 (at 10 MHz) at 25^{0}C for 10 wt% BT-PMMA nanocomposite, which is quite acceptable for a low loss nanocomposite material. PMMA is a polar plastic in which dipoles are created by an imbalance in the distribution of electrons in the presence of an electric field. The ε'_r of the nanocomposites decreased exhibiting frequency dependence which may be attributed to reduction in the polarizations with increasing frequency caused by dipolar groups. Bigger dipolar groups have difficulty to orient with the same pace as the alternating field at higher frequency. Besides, interfacial polarization caused by space charge which is dominant in heterogeneous materials due to large volume fraction of interfaces between BT nanoparticle and PMMA is responsible for higher dielectric constant at lower frequencies of applied field as reported by (Kobayashi *et al.* 2009). Since permittivity of both BT nanoparticles and pure PMMA decreases with increasing frequency. The combined permittivity of BT-PMMA nanocomposites shows decrease in ε'_r with increasing frequency.

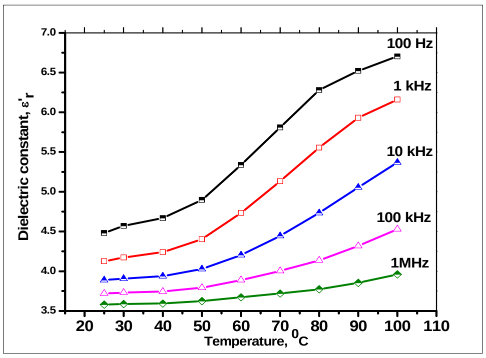


Fig. 5 (a) ε'_r versus temperature at different frequency for 10 wt% BT nanocomposite

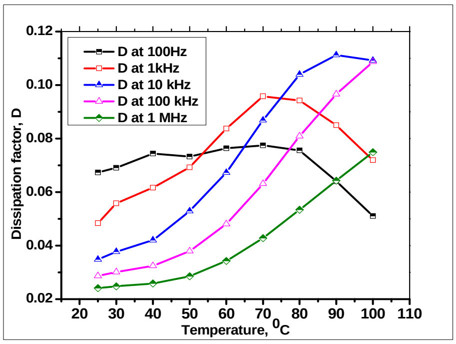


Fig. 5 (b) D versus temperature at different frequency for 10 wt% BT nanocomposite

The decreased in D values with increasing frequency may be due to the space charge effect and decrease in electrical conductivity. This may be due to the inability of the charge carriers to traverse the thickness of the material at the higher frequencies. D became stable within a narrow range of 0.02 to 0.03 at a high frequency of 1 MHz and above. Such low dielectric loss was obtained by the presence of a dense matrix of polymer around nano BT particles due to the formation of insulating boundary outside the dielectric cores. This results in restricting the charge transfer between the dielectric nanoparticles as reported (Singha and Thomas 2008a). ε'_r values increased for all nanocomposites after 4 MHz indicating the beginning of dielectric relaxation process for nanocomposites.

3.4.2 Temperature dependence of dielectric properties

Figs. 5(a) and 5(b). shows plots of ε'_r and D as a function of temperature studied in the frequency range 100Hz to 1MHz. It is evident from these graphs that ε'_r and D increased with increasing temperature. It may be attributed to segmental mobility of polymer at elevated temperature. Increase in ε'_r is pronounced at lower frequencies such as 1 kHz and less at higher frequencies above 100 kHz.

3.4.3 Filler concentration dependence of dielectric properties

Variation of ε'_r with nano BT filler concentration is as shown in Fig. 6(a). It was observed that ε'_r value of the nanocomposites increased with increase in weight fraction of BT in nanocomposite. Similar observations were obtained by Sebastian for polymer ceramic composites (Sebastian and Jantunen 2010). There was an increase of 32.71 % in the value of ε'_r in 10 wt% BT nanocomposite at 1 MHz at 25^0C as compared to neat PMMA. A small decrease in ε'_r is observed for 0.5 wt% and 1 wt% of BT at higher temperatures which may be due to thermal expansion of polymer and ceramic disrupting the chains of contacting particles as reported earlier (Singha and Thomas 2008b). The Variation of D with nano BT filler concentration is as shown in Fig. 6(b). D was seen to be decreasing nominally at 0.5 wt % and 1 wt % BT filler and retaining marginally same values for all other filler wt%. An increase in D value with increasing

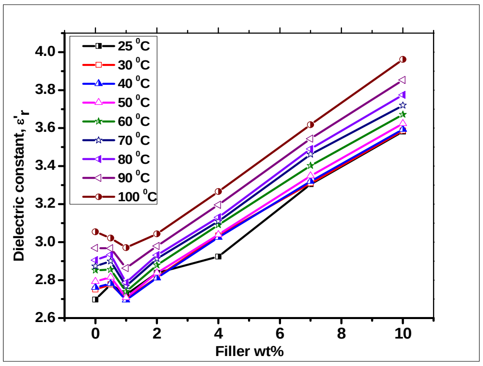


Fig. 6 (a) ε'_r versus filler wt% at different temperature at 1 MHz

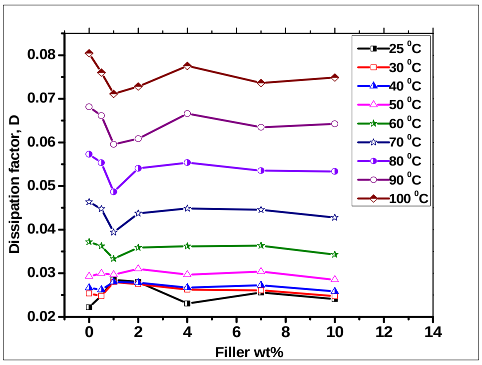


Fig. 6 (b) D versus filler wt% at different temperature at 1 MHz

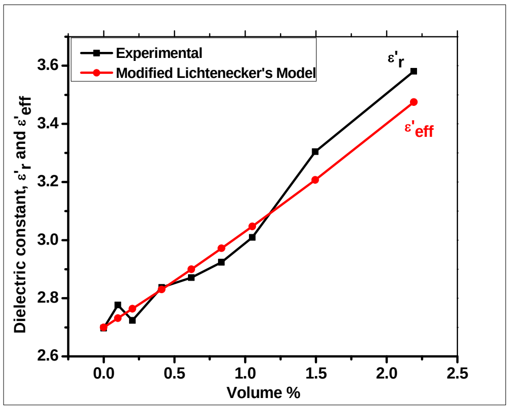


Fig. 7 Dielectric constant versus filler volume concentration using experimental and modified Lichtenecker’s mixing model

temperature has been noted, indicating increase in loss at higher temperatures due to reasons stated above.

The dielectric properties of the composites are influenced not only by the relative permittivity of the components but also by other factors such as the morphology, dispersion, and the interactions between the two phases. Therefore, it is important for electronics application to predict effective ε'_r of nanocomposite from the relative permittivity of polymer matrix and volume fraction of the filler. Though there are many equations to calculate ε'_r of a nanocomposite for low filler content, the Modified Lichtnecker’s logarithmic equation showed good agreement with experimental data as reported by Sebastian et at in their review (Sebastian and Jantunen 2010).

This rule considers the composites as a random mixture of nearly spherical inclusions. Fig. 7. shows calculated and experimental values of ε'_r against filler volume% using Modified Lichtnecker’s logarithmic equation

$$\log \varepsilon_{eff} = log\, \varepsilon_m + v_f(1-n)log\left(\frac{\varepsilon_i}{\varepsilon_m}\right)$$

Where, ε_{eff}, ε_i, ε_m are the dielectric constants of the composites, filler and matrix respectively, v_f is the volume fraction of the ceramic, and n is the fitting parameter or the morphology factor. The volume fractions were calculated from weight fractions of nanocomposites using known density values of polymer and ceramic.

Modified Lichtenecker's model (ε_{eff}) fit the experimental data (ε'_r) at smaller fraction very closely and deviated at higher volume fraction of BT nanoparticles as shown in Fig.7. The graph shows that dispersion of nano-filler in polymer matrix is quite good and values of ε'_r were found to be near predicted values.

4. Conclusions

Nanocomposite films of BT nanoparticles and PMMA polymer were prepared with practically homogeneous distribution. The dielectric constant of nanocomposites increased with filler concentration. Dielectric constant and dissipation factor decreased with increasing frequency, whereas they increased with increasing temperature. ε'_r closely fit the calculated value by Modified Lichteneker's logarithemic model. It has quite low dielectric constant and loss hence can be a possible candidate in the field of nanodielectrics.

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