International Journal of Future Generation Communication and Networking
Vol. 13, No. 2, 2020 pp.608-616

COMPARATIVE ANALYSIS OF THERMAL PROPERTIES OF POLYMER NANOCOMPOSITES

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Abstract

Nanocomposites based on poly (ethyl methacrylate) (PEMA) and polyvinyl chloride (PVC) with different types of nano-materials [Barium zirconate ($BaZrO_3$), silicon dioxide (SiO_2), and MMT etc] has been prepared. Their molecular interactions are available inside the polymeric matrix and various thermal properties primarily based on these substances are analyzed by the way of TSDC (Thermally Stimulated Discharge Current). The peaks available in the TSDC spectra are the evidence to confirm the appropriate interaction between the polymer and nanofiller. Their dielectric traits have been strongly prompted because of the uniform spread of the nanofillers throughout the matrix. These efficient polymeric matrix based nanocomposites could be applied as efficient dielectric materials in the various electronic applications.

Keywords: *Nanocomposites, TSDC, MMT, $BaZrO_3$, SiO_2.*

INTRODUCTION

Polymers have extraordinary electrical insulation properties along with excessive resistivity and high dielectric strength, due to which these are extensively implemented as dielectric cover for electrical wires. The dielectric effects of polymers are relatively more and turn out to be even better because of the use of nanofillers required to improve their softness that reduces the electrical resistivity as well as improves dielectric constant. The principal aspects of massive attention in the dimension of relaxation characteristics are due to the potential outcomes of polymeric composites as cited in the literature [1]. Those improvements are exhilarating equally in conditions of industrial use and for the commercial perception received through analyzing the mechanisms controlling the improved dielectric characteristics [2]. These innovative materials are recommended for storage capacitors and electric insulation of small losses and the unnecessary hurdle of resistance [3].

The nanocomposites of the polymeric matrix have peculiar structural and practical properties [4] which can differ from the properties of polymeric substances and nanofillers. The nanocomposites with favoured characteristics can be evolved based on their length, form and availability of particles and level of filling of fillers. As a result, explorations of the characteristics of nanocomposites are vital for present electronics. The nanocomposites of desired properties have infinite purposes in industries of electronic gadgets [5, 6].

TSDC method helps in investigation of charge movements in noticeably resistive resources and composites in several uses to recognize the characteristics of the composites [7]. The conduction pathway in insulator may be defined on the idea of the carrier movement; however, because of the contribution of excessive impedance, the mobility analysis helps in the resolution of various problems. Because of present motive, transient currents in insulators offer precious results approximately the charge provider generation, delivery and recombination [8–11]. The notable dielectric and electric properties of nanofillers, primarily based on polymeric substances are substantially applied in RAM and electronics components.

Inorganic fillers have been applied in the diffusion of polymer to locate some higher electronic and physical characteristics [12]. The excessive thermal stability and massive floor area of the nanofillers makes them tremendous substances for numerous industrial usages [13-16]. Consequently, various price-effective fillers are applied within the diverse polymeric matrix to have a look at their impact at their structural and dielectric characteristics. In this analysis, numerous nanofillers like Barium zirconate ($BaZrO_3$), Silicon Dioxide (SiO_2) and MMT have been used in the polymeric matrix (PEMA and PVC) to find the DC parameters and relaxation process.

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THEORETICAL ANALYSIS

TSDC is one of the sensitive techniques to investigate the molecular structure, transport processes and charge storage in highly insulating polymers and their nanocomposites. The mechanism of charge generation, storage and transportation in polymeric material is not completely understood. Application of polymers in the electronic and electrical industry is increasing day by day. The study of electrical properties through thermal relaxation effect is making a bridge for current-voltage temperature measurements [17].

TSDC shows excellent characteristics properties of electret formation, charge storage properties, relaxation properties and others. The depolarization current of very low value i.e. ($\approx 10^{-17}$A) can be measured by this technique. Discharging current is related to dipolar re-orientation of molecules, space charges and free charges freezing because of application of an electrostatic field. This study provides valuable information about the role of interface between filler particles and polymers on polymer blend and polymer nanocomposites samples [18].

The effects of several phenomenons such as morphology**,** chemistry of surface structure, surface of highly disperse permeable elastic solid and hydration of materials on the relaxation process have been clearly observed. TSDC results are also comparable with dielectric loss measurement, the low equivalent frequency $(\approx 10^{-3}\ \text{Hz})$ and high sensitivity (ability to detect dipole concentration of 10^{15} dipoles cm^{-3}). This makes the usefulness of TSDC for the study of amorphous relaxations in polymers. Thermally stimulated discharging currents of an electret can mainly be investigated by one of the following methods (i) short-circuit thermally stimulated discharging current (i.e. current TSD with shorted electrodes) (ii) open-circuit thermally stimulated discharging current (i.e. current TSD with an air-gap). The several processes take place during thermally stimulated discharging which are similar to those occurring during charging. The mechanisms are responsible for thermo-electrification of polymer samples including interfacial polarization, space charge polarization because of migration of charges at microscopic distances, electronic or ionic diploes polarization and dipolar polarization [19].

The disorientation of dipoles exhibits a pair of positive and negative charges. It requires a definite amount of energy which is equal to less than one electron volt. So, that dipole can be made to rotate over a potential barrier by the energy of thermal motion at a higher temperature, but at lower temperature dipoles can hardly rotate due to the presence of some structural hindrance. In the case of TSD, these dipoles will go on rotating until they are randomly oriented.

TSDC thermogram consists of several peaks due to disorientation of diploes at low temperature with low activation energy, while dipoles have disorienting at the higher temperature with high activation energy. If the differences of activation energies corresponding to TSDC peaks are very small, it means that energy is distributed continuously. The shape of the TSDC peak is either sharp or broad. The broad peak associated with low-temperature disorientation of dipoles or molecules that is called β-peak. The alpha (α) peak obtained approximately near to the glass transition temperature (T_g) due to the movement in main chain segment of polymer matrix. Maxwell-Wagner interfacial trapping of charges is possible in case of multiphase polymers. In this case, some charges are produced at the interface known as space charges. This is due to the different permittivity and conductivity of two or more phases. Maxwell-Wagner effect is possible in polymers having crystalline and amorphous phases, where the conductivity of the amorphous part is higher than the crystalline part. The electrode prepared in polymer thin film has played a vital role in the neutralization process [20].

The discharging of polarization charges is given by the following relation [20]:

$$p(t) = P_0 \exp\left(-1/\tau\right)^{\alpha} \qquad \text{where } 1 \geq \alpha > 0 \tag{1}$$

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It is known as exponential law, where P_0 = initial polarization, τ = time constant. If heat is given to sample linearly at the rate β=dT/dt, then

$$P(T) = P_0 \exp\{-1/\beta \int_{T_0}^{T} dT'/\tau(T')\}^{\alpha} \quad (2)$$

where T_0 = initial temperature, it is logical to suppose that τ obeys the Arrhenius law for its temperature dependence:

$$\tau(T) = \tau_0 \exp\left(\frac{E_a}{kT}\right) \quad (3)$$

where E_a = activation energy, k = Boltzmann's constant; τ_0 = characteristic relaxation time. The relation for TSD current density is explored from equation (1) and (2) as:

$$i(T) = -\alpha \frac{P_0}{\tau_0} \exp\left(\frac{-E_a}{kT}\right)[s(T)]^{\alpha-1} \exp[-(s(T))^{\alpha}] \quad (4)$$

The total charge released during TSD is given by

$$Q(T) = \frac{1}{\beta\tau_0} \int_{T_0}^{T} \exp\left(\frac{-E_a}{kT'}\right) dT' \quad (5)$$

The area under TSDC peak is used to calculate the value of the total charge released. The trait of thermal current produced by the different delocalized traps is similar to that which is produced by the dipolar polarization. The tail at low-temperature of equation (4) is given by:

$$Ln\ i(T) = const\left(\frac{-E_a}{kT}\right) \quad (6)$$

Thus, the slope of $Ln\, i(T)$ versus 1/T provides information about activation energy from the TSDC peak that is familiar as the initial rise method of Garlick and Gibson [21]. The peak temperature (T_m) at which the highest current produced is obtained by differentiating equation (4) w.r.t. temperature and equating to zero (i.e. di/dT=0), T_m is given by

$$\tau_0 = kT_m^2 / \beta E_a \exp(E_a / kT_m) \quad (7)$$

The relaxation time, τ at T_m and at some other different temperature can be obtained easily by putting the value of τ_0 in equation (7) and it can be represented by the equation (8) in a simple way:

$$\tau = \tau_0 \exp(-E/kT) \quad (8)$$

A lot of research has reported for pristine polymers, polymer blends as well as polymer nanocomposites. The investigation of the TSDC depolarization behaviour has been reported in our previous work for PEMA/ $BaZrO_3$ and PVC/ SiO_2/ MMT nanocomposites with different weight ratios as a function of polarizing field, and polarizing temperature separately [22-23]. In this paper, the comparison of all these nanocomposites which are made of three different types of nanofiller and two different types of the polymer matrix is presented and diversified and some interesting conclusions have been observed.

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EXPERIMENTAL

MATERIALS

Poly(ethyl methacrylate) (PEMA) and Polyvinyl Chloride (PVC) powder have been procured from Redox. Solvent (DMF) has been bought from Merck. $BaZrO_3$ (length much less than 100 nm), SiO_2 nanoparticles (length 5 nm) and MMT have been acquired from Sigma Aldrich. All of the chemicals and nanofillers are used as supplied.

SAMPLE PREPARATION

The nanocomposite of PEMA matrix

The nanoparticles of PEMA material with $BaZrO_3$ is used as filler developed by spin-coating technique [22]. In this process, several weight percentage (%) is mixed in dimethyl formamide solvent (40 ^{0}C) for 1 h. Similarly, a mixture of various weight percentages (%) (2, 4 and 6) of $BaZrO_3$ with DMF is arranged by stirring. The $BaZrO_3$ solution has been added gradually in PEMA solution with mixing for 30 min. After blending of $BaZrO_3$ in the polymeric mixture, the resultant solution has been sonicated for 5 min. At the time of stirring, the binder silane (5 ml) and surfactant (5 ml) have been added in nanocomposites to develop polymer-nanoparticle interactions. The ultimate blended material employed for the fabrication of thin films on fused silica.

The nanocomposite of the PVC matrix

For the preparation of nanocomposites [23], 5mg of PVC is added in 50ml of DMF. The inorganic fillers are incorporated in DMF for 15 minutes using an ultra sonicator. PVC has been blend and put on a stirrer for 2h for inclusive mixing of polymer material. Thin films are developed on a glass substrate and dried in a vacuum oven for 12 hrs at 40 ^{0}C. The numerous benefits of composites with less than 10% inorganic fillers have been investigated in polymers. Hence, PVC composite films of various amounts of inorganic fillers (2, 4, 6 wt.%) have been developed. The thin layer of 4 wt. % filler has superior dispersion and consistent deviation of dielectric properties.

ELECTRET FORMATION

Polymer samples have to be prepared in a circular shape like discs of the size of the diameter of 5 cm. Silica gel filled desiccator is used to prevent the sample from moisture. All polymeric samples like pristine PEMA, pristine PVC and their nanocomposites made by the addition of nanofiller like SiO_2, $BaZrO_3$ and MMT have been vacuum-aluminized on both sides. A sample holder for preparing the electret and measurement of electrical current has been placed in a double-walled oven which is thermally insulated and digitally controlled. The temperature range extending from 25^{0}C to 450^{0}C has been used. The Interlink Electronics Ltd. made variable transformer has been used to control the uniform heating rate. An electrometer is used to short-circuit the sample and obtain TSDC measurements. A linear heating rate of 3^{0}C min^{-1} has been used to heat up the sample from room temperature. The depolarization currents of polymeric samples have been measured with respect to temperature at a linear heating rate of 3^{0}C min^{-1} by a Keithley electrometer (Model No. 6514). This model of electrometer has been designed to measure the very small amount of direct current (DC) ranging from 10^{-7} to 10^{-12} A. The depolarization current has been recorded in the temperature range from 30 ^{0}C to 200 ^{0}C with a heating rate of 3 ^{0}C.

During the experiment, different values of temperature are used for charging and discharging of samples at different times. The DC electric field has however remained constant of the order of 100kv/cm in all the cases. TSDC thermograms have been obtained at the polarizing temperature of 50 ^{0}C with 100kv/cm polarizing field. Each sample has been polarized thermally for the time of 2.5 hour afterwards it is discharged at a different temperature during time t_d.

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RESULTS AND DISCUSSION

TSDC analysis of polymer, as well as their composites, have been analyzed and mentioned in the following figure-1. Pure polymer, as well as a composite with 4 wt% $BaZrO_3$ is having two relaxation peaks, while the remaining sample has a mono-relaxation peak. A primary relaxation peak of nanocomposite of PEMA has been available at 80-85ºC and a subsequent peak near 160–165ºC. However, other samples have a sole peak at ~ 110-120^{0}C while dual peaks are observed for composite (4 wt%) of $BaZrO_3$ are present in the section of 110-120 and 165-170ºC, correspondingly. Charge carriers generated in the composite material develop the polarization which formed the dipolar orientation at the polymeric interface that significantly helps via interfacial or Maxwell–Wagner kind of polarization [24]. The big surface areas of the inorganic fillers are accountable for the development of an interfacial polymeric level (shell) involved in the constituent part of core [25].

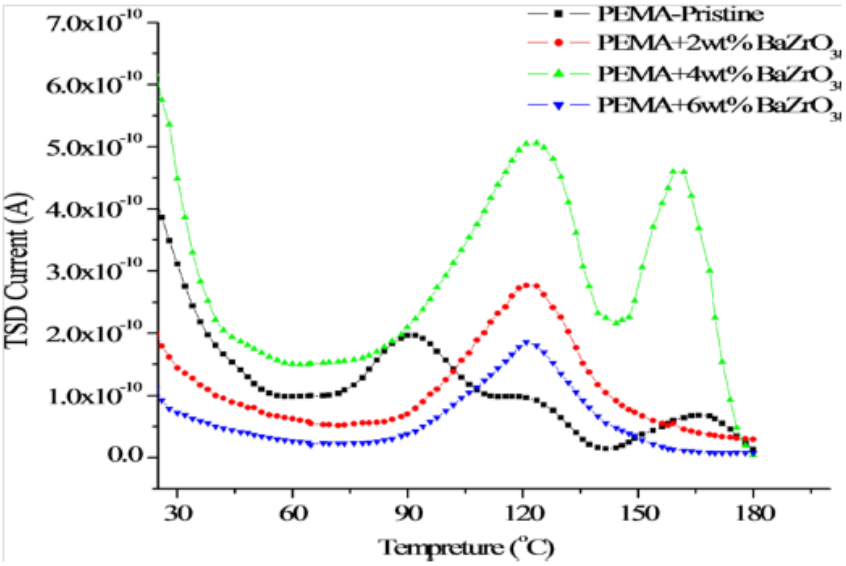


Figure-1: TSDC thermograms of different wt% $BaZrO_3$ in PEMA based composites at a poling field of 50 kVcm-1

The TSDC spectra of PVC disclose relaxation peaks at 83ºC, composite of SiO_2 (4wt.%) at 89/112ºC, composite of MMT (4wt.%) at 130ºC, composite of SiO_2 and MMT (each 4wt.%) at 84 /131ºC. The TSDC spectra of PVC and composites are mentioned in the following figure-2. A decline of existing peaks of TSDC with a composite of SiO2 and composite of SiO_2 and MMT fillers are obtained because of the interfacial effect between polymeric matrix and fillers.

The PEMA with 2 wt%, 6 wt% $BaZrO_3$, neat PVC and PVC with 4 wt% MMT are having sharp mono relaxation peaks and presence of a mono peak possibly because of the very small contribution of dipolar polarization or it may be shrouded by the interfacial type of polarization. The charges accumulated in this type of polarization, at the interfaces, are defused by the ohmic conduction current which flows in the reverse order. In polymer nanocomposites, due to increase in the volume of charge carriers and the number of trapping sites, peak temperature has been increased. The plasticization effect due to the doped nanofiller in the existence of surfactant and silane is also accountable for the mono peak in TSDC spectra. The neat PEMA, PEMA with 4 wt% $BaZrO_3$, PVC+4 wt% SiO_2 and PVC+4 wt% SiO_2 + 4wt% MMT are having two different relaxation peak that occurred because of clustering of nanofiller in the matrix of polymer.

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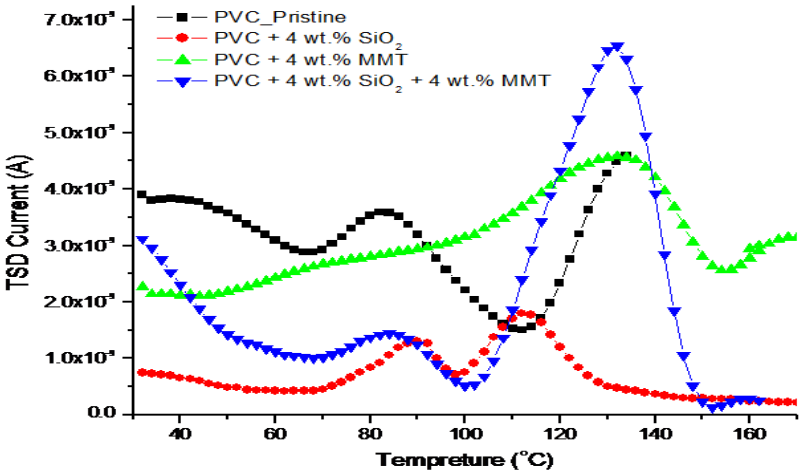


Figure-2: TSDC thermograms of PVC based nanocomposites at the poling field (Ep) of 100 kV/cm-1.

The activation energy and relaxation time have been obtained by equation (6) and (8). The deviation from the TSDC peak has been calculated by Simpson's rule. In current exploration, charge released and peak current deviation has been observed with filler. The peak temperature changes with filler in their matrix. The polarization charges, maybe because of inoculation from the electrodes and inorganic fillers inside the matrix of polymer and charge carriers incorporated from the delocalized traps inside the polymer matrix. The nanoparticle used as a filler should be attached to the polymer matrix in a chemical and physical manner. The interaction among the nanofiller and polymer represented the reinforcement effect strength.

PVC and PEMA polymers are highly polar in nature, so there should be a great attraction between nanofiller and polymer at the interface. Hence the polymer material can easily roll over the nanoparticle and make better interface. Decline or amplify of current in TSDC spectra as a contrast to pure PEMA and PVC represented either particle are agglomerate or dispersed consistently with the interface of minute and huge thickness. Consequently, TSDC spectra established continuation of the interface among the fillers and polymeric matrix. The TSDC parameters including activation energy, charge released, peak current and peak temperature for different nanocomposites are shown in the following table 1.

It is observed that large numbers of external factors affect the TSDC characteristic. By increasing polarizing temperature (T_p), the peak current has been also increasing and shift towards higher temperature side. This TSDC behavior is similar to that reported in several polymers [26-29]. The decrease in T_p has significantly affected the TSDC peak due to accumulation of space charges. The small space charge peak attributed the charges are not trapped in different trapping level. The TSDC spectra have been significantly affected by polarization time also as like polarization temperature. The depolarization current is the function of the intensity of the applied field. The intensity of TSDC peak increases with the increase of the polarizing field; it confirmed the presence of dipolar polarization mechanism. On the other hand, nonlinear variation in peak current with the polarizing field is the origin of space charge polarization. The material of the electrode is affecting the TSDC characteristics because of the variation of conductivity and different work function. The Charge storage in electrets depends on the thickness of polymer samples; hetro-charge released by TSDC has a linear relationship with the thickness of samples. The TSDC magnitude is also a function of heating rate. The magnitude of TSDC peak increases with the increase in heating rate.

The polymer placed in the shell has different physical properties as compared to the bulk polymer because of immobilization. The polymer chains mobility is decreased if attractive forces lay between the interfacial layer and the filler resulting in glass transition temperature increases. If repulsive forces are there, the polymer chain mobility is enhanced resulting in a plasticizing effect occurs in

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the range of glass transition temperature. In this TSDC experiment, peak moved towards the upper side close to the glass transition temperature has been observed in the case of nanocomposites; it is an evidence of attractive interaction between nanoparticles and polymer interface. The nonlinear behaviour of peak current with wt% of $BaZrO_3$, MMT, SiO_2 indicates that besides dipolar polarization some other types of polarization are also responsible for TSDC peak. By increasing the concentration of $BaZrO_3$ nanoparticles, the activation energy increases which show the hardening of the polymer chain.

Table 1: Showing various parameters of TSDC spectra of PEMA and PVC based nanocomposites poling field at 50 ($kVcm^{-1}$)

Samples	Relaxation	Peak Position (^oC)	Peak current (A)	Activation energy (eV)	Charge release 10^{-8} (C)
PEMA Pristine	α	91.00	19.82	0.3433	8.72
PEMA + 2 wt% $BaZrO_3$	α	121.00	27.71	1.1806	18.55
PEMA + 4 wt % $BaZrO_3$	α	122.00	51.05	00.56	29.70
	β	160.00	46.09	46.09	15.09
PEMA + 6 wt % $BaZrO_3$	α	121.00	18.60	18.60	11.44
PVC Pristine	α	83.13	03.59	0.181	01.03
PVC + 4wt% SiO_2	α	90.72	01.32	0.761	03.19
	β	112.54	01.81	0.654	05.46
PVC + 4 wt % MMT	α	132.18	04.58	0.297	02.68
PVC + 4wt% SiO_2 + 4 wt % MMT	α	84.36	01.43	0.795	03.46
	β	131.70	06.57	0.736	03.21

CONCLUSIONS

In the present investigation, the peak current and charge released showed variations with nanofiller. The peak temperature also shifted towards the higher side in the presence of nanofiller in nanocomposites. The α-relaxation close to the glass transition region and MWS relaxation at some higher temperature has been observed in the TSDC thermogram. The observed value of charge released and activation energy also confirmed the presence of these two types of the relaxation process. For pristine polymer PEMA and PVC, the single peak has been observed at 91^0C and 83^0C which are near to corresponding glass-transition temperature. When different types of nanofiller doped into the polymer matrix, this alpha-peak shifted towards the higher side that confirmed the proper interaction between nanofiller and polymer matrix. The value of peak temperature and peak current depends on the type of filler and concentration ratio. The 2nd peak is also observed with 4% concentration of nanofiller that confirmed the MWX type of polarization occurred dominantly in that nanocomposites. These polymeric matrix based nanocomposites can be applied as a suitable dielectric for the manufacturing of capacitor and electronic inactive filter.

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