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Effect of GNP Addition on Thermal and Electrical Properties of Polycarbonate-poly(butylene terephthalate) Blends

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GNP İlavesinin Polikarbonat-Poli(bütilen tereftalat) Karışımlarının Termal ve Elektriksel Özellikleri Üzerinde Etkisi

Mecit ÖGE*

Bartın Üniversitesi, Mühendislik Mimarlık ve Tasarım Fakültesi, Makine Mühendisliği Bölümü, Bartın, Türkiye

Abstract

PC-PBT/GNP nanocomposite samples were fabricated via melt-compounding technique. The agglomeration state of the produced samples was investigated via optical microscopy. The thermal properties of the samples were assessed with DSC and TGA techniques. Electrical conductivity tests were also performed to determine whether a conductive pathway is established due to GNP addition. The crystallinity calculations derived from DSC measurements showed that the crystallinity of samples was reduced with increasing GNP content. The increased degradation temperatures with increasing filler content showed that a slight improvement in the thermal stability of the PC-PBT blends is achieved by increasing the filler ratio. Electrical conductivity test results indicated establishment of a conductive pathway at higher filler ratios with 1.35 x 10^{-4} S/m and 6.89 x 10^{-4} S/m conductivity values for 5 % and 7 % filler weight fractions, respectively.

***Keywords:** PC/PBT Blend; Nanomaterials; Thermal Properties, Electrical Conductivity.*

Öz

PC-PBT/GNP nanokompozit numuneleri, eriyik karıştırma tekniği ile üretildi. Üretilen numunelerin topaklaşma durumu dijital mikroskopi ile incelendi. Numunelerin termal özellikleri DSC ve TGA teknikleri ile değerlendirildi. GNP ilavesi nedeniyle iletken bir yol oluşup oluşmadığını belirlemek için elektriksel iletkenlik testleri de yapıldı. DSC ölçümlerinden elde edilen kristallik hesaplamaları, GNP içeriği arttıkça numunelerin kristalliğinde bir miktar azalma olduğunu gösterdi. Dolgu maddesi içeriği arttıkça artan bozunma sıcaklıkları, PC-PBT karışımlarının termal kararlılığında dolgu oranının artmasıyla hafif bir iyileşme sağlandığını gösterdi. Elektriksel iletkenlik test sonuçları, % 5 ve % 7 ağırlık fraksiyonları için sırasıyla 1.35 x 10^{-4} S/m ve 6.89 x 10^{-4} S/m iletkenlik değerleri ile daha yüksek dolgu oranlarında iletken bir yol oluştuğunu gösterdi.

***Anahtar Kelimeler:** PC/PBT Karışımı; Nanomalzemeler; Termal Özellikler, Elektriksel İletkenlik*

1. Introduction

Polymer blends and their combination with reinforcing materials such as micro and nano-scale organic and inorganic fillers have become indispensable materials for substituting metal components with lighter polymer based-composites that have more enhanced electrical and thermal properties in a wide variety of industries. In the last couple of decades, Polycarbonate-poly(butylene terephthalate) (PC/PBT) polymer blends have found extended use particularly in automotive industry for interior and exterior automotive applications such as car bumpers, car consoles, spoilers, side panels, etc. In these applications, PBT provides thermal stability while PC provides impact resistance and dimensional stability (Ferreira et al. 2020). In polymer matrix composites, the polymers that form the matrix or base material group are organic compounds consisting of long molecular chains containing many bonds connected to carbon atoms and are divided into three groups: thermoplastic, thermoset and elastomer (Sastri 2014).

The properties of polymer matrix composites largely depend on the interface adhesion between the matrix and the filler. The shape, size and orientation of the filler material play an important role in the homogeneity and isotropy of the composite material (Guchait et al. 2022). The advantage of nano-sized fillers compared to micro-sized fillers lies in their extremely high surface/volume ratio, leading to increased surface energy due to Van der Waals bonds and surface tension, outcompeting other factors such as density and gravitational force, thus giving the material superior mechanical and thermal properties even at low additive rates (Ajitha et al. 2020, Alshammari et al. 2022, Meschi et al. 2015). Owing to the perfect interaction between the filler and the matrix materials in polymer matrix nanocomposites, superior properties can be obtained even at nano-filler ratios of less than 5 % by weight, compared to those obtained at micro-macro filler ratios up to 50 % (Liu and Cheng 2023).

Enhanced electrical conductivity is another highly demanded feature for polymer materials used for exterior

*Sorumlu Yazar/Corresponding Author: Mecit ÖGE
e-posta/e-mail: mecitoge@bartin.edu.tr

automobile parts such as bumpers, that require electrostatic painting. It is thought that graphene-based nano-admixtures, which are used to reduce the electrical percolation threshold, which is an indicator of electrical conductivity in many other polymers and polymer mixtures (Krause et al. 2018, Rahaman et al. 2022), can also be used for this purpose in PC/PBT mixtures. Graphene-based nano-fillers have been often preferred over ceramic and metal-based fillers due to their superior structural and functional features and they have been widely used for reinforcement in nanocomposites. The features of the resulting nano-composite materials are closely related to the dispersion of the nano-filler within the matrix, the interaction between the matrix and the filler, as well as the direction of the fillers within the matrix, which is also related to the type of the subject graphene-based filler (Mittal et al. 2015). Due to their planar structure and high aspect ratio, enabling the stress transfer within the surrounding matrix phase, GNPs provide superior mechanical and other physical characteristics as compared to non-filled polymers (Mahmun et al. 2021).

Enhancement of polymer material and composite properties via addition of fillers have been the subject of numerous studies (Taşdemir and Karadirek, 2024; Uzay, 2023; Gümüş, 2021). Huang et al. (2018) introduced small quantities of carbon nanotubes (CNTs) into PC/PBT blends to observe the changes in their macroscopic properties. They reported that, CNTs show homogeneous dispersion and induce a certain level of phase separation in PC/PBT phase morphology. They also reported improvements in mechanical and electrical properties (Huang et al. 2018). In another attempt to improve PC/PBT blend properties, Bai, et al. (2022) achieved enhanced interfacial adhesion between PC and PBC via in-situ grafting during the melt blending of polycarbonate / polybutylene Terephthalate / ethylene-methyl acrylate - glycidyl methacrylate (PC/PBT/EMA-GMA) blend, leading to significant improvements in the impact toughness of PC-PBT (Bai et al. 2022). Wen and Zheng (2019) examined the impact of the selective distribution of graphite nano-platelets on the thermal and electrical conductivities of PC/PBT blends and concluded that the thermal and electrical conductivities of the un-filled blends could be substantially improved via segregated dispersion of graphite nano-platelets in one of the co-continuous components of the blend (Wen and Zheng 2019). In order to improve the properties of this blend, several other studies have been carried out on supporting it with different co-polymers, micro and nano fillers, examining various material properties with tests specific to the automotive industry, and developing different characterization methodologies. In these studies, it was generally observed that additive materials improved PC/PBT polymer matrix behavior. However, among these studies, no study has been encountered on assessment of the changes in the crystallinity, thermal stability and electrical conductivity of PC/PBT blends reinforced with graphene nano-platelets (GNPs). This study thus aims to investigate the impact of GNP addition on the thermal and electrical properties of PC-PBT blends. The experimental and theoretical results are discussed in relation to each other and in the context of existing literature.

2. Materials and Methods

2.1 Materials

Polycarbonate (PC-Lg Chem Lupoy, 1303EP-22, South Korea) and polybutylene terephthalate (PBT, Pimadure HS40N, Turkey) were purchased in granule form from Aydın Plastic Co. Ltd (Turkey) as the raw materials for polymer blends. Graphene nanoplatelets (GNPs) as the nanofiller were purchased in powder form from Nanografi Nano Technology (Turkey). The physical properties of the raw materials are shown in Tables 1 and 2.

Table 1. Physical properties of blend constituents

Material	Melt Flow Index (MFI)	Density
PC	22 g/10 min (300°C /1.2 kg)	1.2 g/cm³
PBT	30-45 g/10 min (250°C /2.16 kg)	1.31 g/cm³

Table 2. Physical properties of GNPs

Purity	>99.9%
Thickness	3 nm
Specific Surface Area	800 m²/g
Diameter	1.5 µm

2.2 Sample Preparation

In the present work, PC and PBT in granule form and GNPs in nano-powder form were used as the raw materials for the preparation of the nanocomposite samples. For all samples, PC and PBT weight ratio was kept constant as 1:1 and GNPs were added with filler weight fractions of 0.5 %, 1 %, 3 %, 5 % and 7 %, the filled samples are named as G0.5, G1, G3, G5 and G7, respectively, throughout the article. PC-PBT granules and GNP nano-powders were subjected to melt compounding under high temperature and shear stress conditions using a lab-scale melt mixer (KÖKBİR, M40, Turkey) at 260 °C. To ensure homogenization of the mixture, the semi-finished products were further subjected to shredding, extrusion in a single screw extruder (KÖKBİR-Turkey) and granulation (KÖKBİR-Turkey), thus they took the final nanocomposite granule form. Afterwards, a lab-scale hydraulic press equipped with hot and cold sections was used to put the nanocomposite granules in plate forms to conduct the characterization studies. The work-flow diagram of the experimental studies is shown in Figure 1.

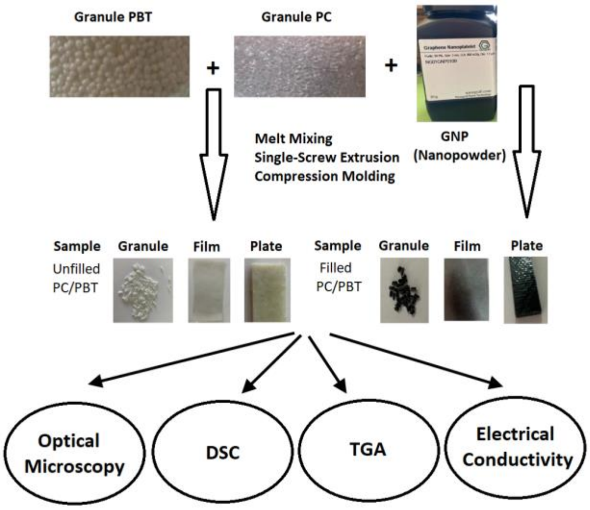

Figure 1. Work-flow diagram of experimental study

2.3. Optical Microscopy

An optical microscope (Nikon-ShuttlePix P400R-Japan) equipped with a motorized focusing stand controller (P-MFSC) and a touch panel display (P-TPM) was used to characterize the dispersion state of GNPs within the composite samples. Samples prepared in film form via compression molding with an approximate thickness of 1 mm were used to allow the transmission of the light through the samples.

2.4. Differential Scanning Calorimetry

Differential Scanning Calorimetry (DSC) analyses were performed to determine the melting and cold-crystallization behaviors of the samples using an AHP brand (Turkey) differential scanning calorimeter (DSC) with 3.6-4.0 µV/mW sensitivity. A heating rate of 10 °C/min was applied between 50 °C and 280 °C under nitrogen atmosphere. The crystallization behavior of PBT component of the produced nanocomposites were calculated using the following formula:

$$Xc = \frac{\Delta H_m - \Delta H_{cc}}{\Delta H_m^0 x \varphi} \text{ x } 100\ \% \qquad (1)$$

Where, ΔH_m is the fusion enthalpy and ΔH_{cc} is the cold crystallization enthalpy obtained during DSC, ΔH_m^0 is the fusion enthalpy for fully crystalline PBT (142 J/g), and φ is the weight ratio of the PBT component within overall sample weight (Huang et al., 2018).

2.5. Thermogravimetric Analyses (TGA)

TGA were performed to evaluate the thermal stabilities of the produced nanocomposites on a Seiko brand analyzer within the temperature range of 40 ℃ - 800 ℃ with a heating rate of 10 ℃/min under nitrogen atmosphere.

2.6. Electrical Conductivity

A Keysight brand insulation multimeter (U1461A) was used to evaluate the resistivity of the produced un-filled and nanocomposite samples. Two-point probe technique was applied, and the obtained resistivity values were converted and reported as conductivity values in S/m. Each test was repeated for five times and the average values were reported.

3. Results and Discussions

3.1. Dispersion State of GNPs and Phase Morphologies of the Samples

In the present study, the dispersion state of the filler for the nanocomposite samples were characterized using an optical microscope and for this purpose the film samples were used to enable transmission of the light. Figure 2 shows the optical microscope images of the samples under 80x magnification (for the un-filled sample) and 400x magnification (for the filled specimens). Due to the operating principle of the microscope, the non-filled (those without filler) areas are represented with lighter tones and the filled areas (filler locations) are represented with darker tones. Also, in the figure, the black dots represent the agglomerates of GNPs and bigger dots or localized black regions are indicative of poorer dispersion of filler. As observed in the figure, localizations or agglomerations start to occur after the filler ratio of 3 wt. % (Figure 2d) and bigger dots and localizations can be observed in the cases of 5 and 7 wt. % (Figures 2e-f). The same effect induced by agglomeration of carbon-based nano-fillers that cannot be broken down by shearing and high temperature of melt mixing after certain filler ratios has been reported in other studies as well (Karteri et al. 2023). Nevertheless, the extent of agglomerations or localizations is not much visible even in the case of 7 wt. %. As also indicated by the optical microscope image of the film sample of binary un-filled PC/PBT blend shown in Figure 2a, there is no evidence of a multiphase structure at the macro scale likely to be induced by insufficient miscibility between the blend components.

3.2. *Thermal Behavior*

Figure 3 shows the DSC heating curves and the calculated degrees of crystallinity for the un-filled and nanocomposite samples, and Table 3 shows the corresponding melting characteristics used for calculation of the degrees of crystallinity. The heating curves are characterized by two distinct peaks which is consistent with the previous studies on PC/PBT blends.

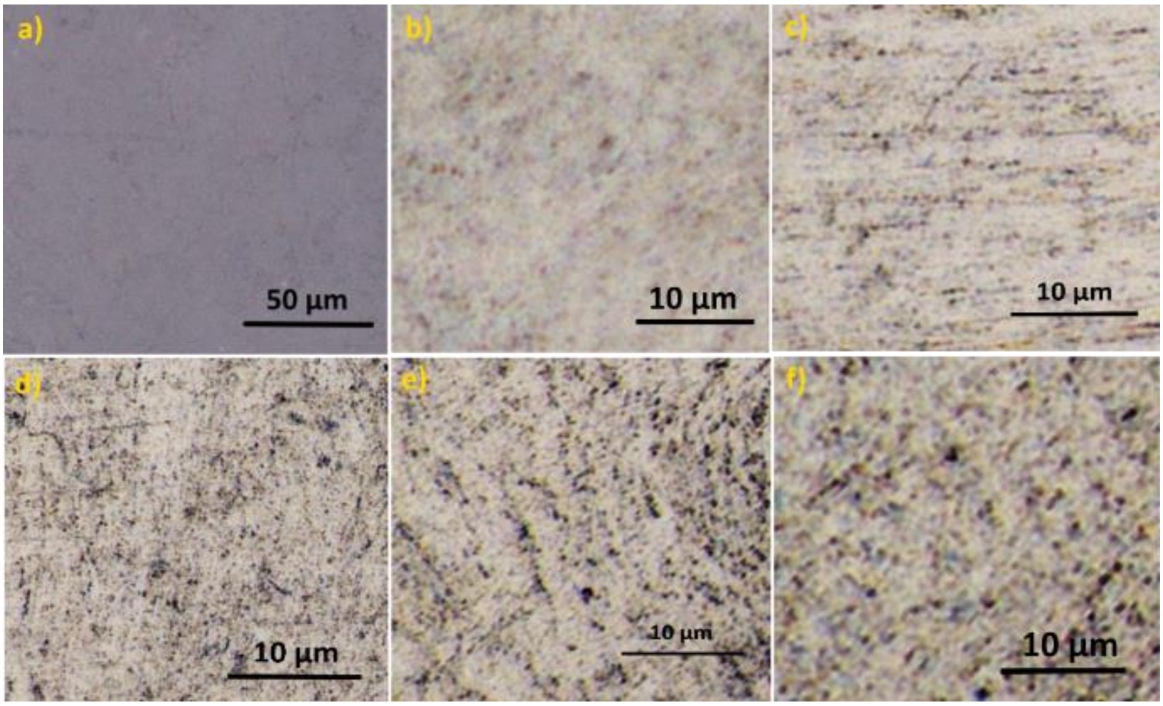

Figure 2. Optical microscope images of a) un-filled sample (80X), b) G0.5, c) G1, d) G3, e) G5 and f) G7 (400X)

The exothermic peaks between ~150 °C and ~200 °C arises from the cold crystallization of PBT constituent and the endothermic peaks are ascribed to the PBT crystallite fusion (Huang et al. 2018). As indicated by Figure 3, the highest degree of crystallinity is calculated for the un-filled PC-PBT sample. The crystallinity of G3 is close to that of the un-filled sample, then it is reduced by nearly 47% for G5 and increases again by ~29 % for G7. It was found from the measured values that, the change in the crystallinity of the intermediate filler ratios (G05 and G1) are negligible so they were excluded for ease of interpretation.

Table 3. Melting characteristics of un-filled and nanocomposite samples

Sample	Tcc (°C)	ΔHcc (J g-1)	Tm (°C)	ΔHm (J g^{-1})
PC-PBT	198.1	5.49	233.9	0.07
G3	188.8	5.46	232.1	0.29
G5	178.2	3.17	232.8	0.32
G7	138.6	3.9	232.7	0.2

The values in Table 3 show that, there is also negligible variation in the melting temperatures (Tm) of the samples whereas cold crystallization temperatures (Tcc) are significantly reduced by increasing the filler ratio. It is also noteworthy that Tcc of the samples has a sharp drop in the case of G7 to 138.6°C from 198.1°C for the un-filled sample. The crystallization kinetics in polymer nanocomposites are greatly influenced by the two opposing effects of fillers. One is their acting as heterogeneous nucleation sites which favors crystallinity, and the other is hindrance of molecular chain mobility by nanofillers resulting in reduced crystallinity rates (Cyras et al., 2018). The adverse effect of increasing filler ratio on the crystallization kinetics is also attributable to the increased agglomeration hindering the growth of crystallites (Zare 2016) as well as the reduced distance between graphene nanoplatelets which reduces the probability of incorporation of additional nucleation sites into the growing polymer chain (Tarani et al., 2023). The transesterification reaction between PC and PBT is also accountable for the limited crystallization of PBT (Devaux et al., 1982). It can be postulated based on the above considerations that, the limiting effects of GNPs on the overall crystallization kinetics of the blend composites outcompeted the favoring effects in the particular case of 5 wt. % filler addition which led to a ~47 % drop in the crystallinity rate. This drop is attributed to the increased agglomeration of nanofillers during the melt compounding process, and the subsequent increase (from 23.69 % at G5 to 30.57 % at G7) can be ascribed to the reduced extent of transesterification.

As also indicated by the crystallinity calculation results shown in Figure 3., the crystallinity of samples is reduced with increasing filler ratio. The reduction in the crystallinity of PBT constituent induced by GNPs is likely to induce impairment of the mechanical properties of the samples. However, mechanical properties such as tensile, flexural strength and impact strength are dependent on various mechanisms other than crystallinity, such as the extent of phase separation and transesterification that are likely to vary with varying filler content.

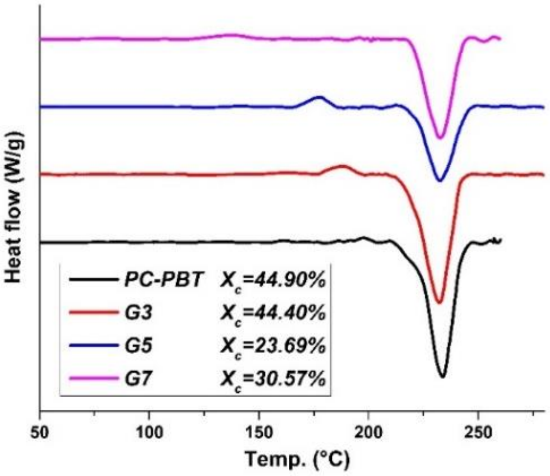

Figure 3. DSC heating curves and calculated degrees of crystallinity (Xc) of PC/PBT/GNP samples with varying GNP content.

The thermogravimetric (TG) and derivative thermogravimetric (DTG) curves of the un-filled and nanocomposite samples are shown in Figure 4a-b. On each curve in Figure 4b, the peak on the left represents the thermal degradation behavior of PBT and the one on the right represents that of PC component of the samples. The mass loss values acquired from the TGA software are included on the TG graph. Up to the filler ratio of 7 wt. %, total mass loss seems to be slightly reduced with increasing filler addition. Mass loss data (Figure 4a) may not provide precise information as to the thermal stability of the samples since filler mass is involved in the calculation (Tarrío-Saavedra et al., 2008). Nevertheless, after incorporating 7 wt. % filler, a slight increase is observed which seemingly arises from the decomposition of a higher amount of PC. This can be ascribed to the reduced miscibility between PC and PBT, due to the relatively lower extent of transesterification induced by high filler content (7 wt. %). The initial decomposition temperatures (IDT), the temperatures of maximum rate of mass loss (TDmax), and temperatures of end of degradation (TDend) are shown on the DTG graph (Figure 4b) for the un-filled and nanocomposite samples. As shown in the figure, the onset (IDT) temperatures for the PBT phase increases with 3 wt. % filler addition (from 355.3°C to 374.1°C, and slightly reduced with 5 wt. % and 7 wt. % filler ratios (369.7°C and 369.5°C, respectively). TDmax for PBT likewise exhibits an overall increase with addition of filler (from 393.8°C for the un-filled sample to 401.8°C for G7). TDend for PBT is increased from 428.2°C for the un-filled sample to 430.0°C for G7. TDmax for PC likewise increased from 502.3°C for the un-filled sample to 508.4°C for G7. TDend for PC is however reduced from 552.8°C for the un-filled sample to 541.5°C for G7.The overall increase in degradation temperatures indicate a slight improvement in the thermal stability of the polymer blend by addition of filler. It can be stated based on the results that; the highest thermal stability as compared to the un-filled sample was achieved with 3 wt. % GNP addition (considering the onset degradation temperature for PBT). Similar results have been obtained in previous works. Wijerathne et al. (2023) reported that, GNP had insignificant effect on the thermal stability of virgin or recycled PC matrix (Wijerathne et al., 2023). In another work on the thermal stability of GNP reinforced polypropylene samples, Liang et al. (2018) attributed the improvement in the thermal stability to the mass-transport barrier effect induced by GNPs due to their high aspect ratio hindering the transport of decomposition products to material surface, i.e. a “thin sheet barrier effect”, the interactions between the nanofiller and the macromolecular polymer chains, and absorption of GNPs on the decomposition products’ free radicals. They also reported that, increasing the filler ratio also induces a heat transfer effect by forming a heat transfer network of GNPs, which competes with the thin sheet barrier effect to reduce thermal stability (Liang et al., 2018). In the present work, the initial increase and subsequent decrease in the degradation temperatures can also be ascribed to the contributive effect of a similar barrier effect and the reduction at higher filler ratios can be ascribed to the agglomeration of GNPs.

3.3. *Electrical Conductivity Results*

The electrical conductivities of the un-filled PC/PBT samples and PC/PBT-GNP nanocomposites with varying filler ratios are shown in Table 4. The electrical conductivity test result of the un-filled PC/PBT sample could not be received as it remained below the measuring sensitivity of the test device.

Table 4. Linear current conductivity test results

Samples	Conductivity (S/m)
G0.5	(2.59 ± 0.12) x10^{-8}
G1	(3.22 ± 0.16) x10^{-8}
G3	(8.46 ± 0.42) x10^{-6}
G5	(1.35 ± 0.06) x10^{-4}
G7	(6.89 ± 0.34) x10^{-4}

It can be accordingly inferred from the fact that test results could be received after G0.5, the electrical conductivity of all nanocomposites are higher than the un-filled sample. The conductivity of G1 is slightly higher than that of G0.5 and it increases with two orders of magnitude for G3. This can be attributed to the fact that, carbon-based nano-fillers with high width-thickness ratios, as in the case of GNPs, are reported to tend to form conductive pathways at low percolation threshold values (Chen et al., 2019) after a certain weight ratio, which turns out to be 5 wt. % in the present case.

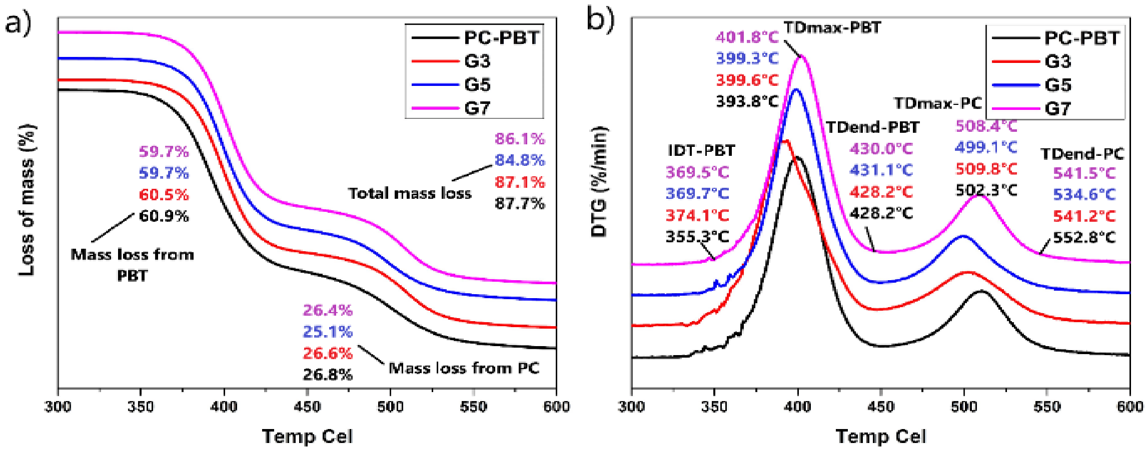

Figure 4. a) TG and b) DTG curves of un-filled and nanocomposite samples.

The formation of a conductive pathway at this filler ratio is more evident from the graph showing the conductivity (in S/m) versus filler ratio (Figure 5).

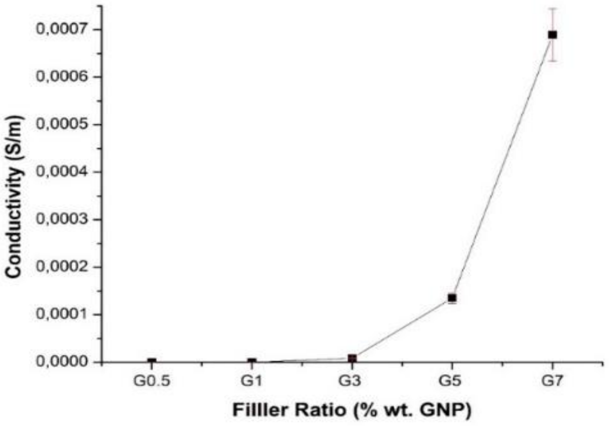

Figure 5. Variation of electrical conductivity with increasing filler ratio (each data point represents the average of 5 test results).

4. Conclusions

Polymer nanocomposites consisting of PC, PBT as the polymer blend phases and GNP as the filler constituent were prepared via melt compounding method using a melt mixer. After the microstructural analyses, DSC, TGA and electrical conductivity measurements performed on the produced specimens, the following conclusions are drawn:

- Increasing the GNP filler weight fraction resulted in reduced degrees of crystallinity up to the filler fraction of 5 wt. % GNP after which a slight increase is observed with sample G7. The reduction is attributed to increased agglomeration and the subsequent increase is attributed to reduced transesterification by the effect of increased filler content.

- According to DTA-TGA results, the highest thermal stability is achieved with 3 wt. % GNP addition due to a mass-transport barrier effect induced by GNPs.

- According to the results of electrical conductivity tests, a conductive pathway was established with the filler ratio of 5 wt. % (1.35 x10^{-4} S/m) which is higher than that of G0.5 with four orders of magnitude. Electrical conductivity was further improved by ~410 % with G7, meaning that achievement of higher values is possible with higher weight fractions of GNP.

The results of the present work are believed to contribute to the efforts in improving the properties of PC/PBT blends which have found wide application in automotive industry. The promising results presented in this work warrant further research on enhancement of PC/PBT properties using carbon nano-fillers.

Declaration of Ethical Standards

The authors declare that they comply with all ethical standards.

Credit Authorship Contribution Statement

Author: Conceptualization, Supervision, Methodology, Investigation, Data curation, Writing – original draft, Writing – review & editing, all authors read and contributed to the manuscript.

Declaration of Competing Interest

The author declares no conflict of interest.

Data Availability

All data generated or analyzed during this study are included in this published article.

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