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Assessment of thermal and electrical conductivity enhancements in PC-PBT blends reinforced with hybrid MWCNT-GNP nanofillers

PC-PBT karışımlarının hibrit ÇDKNT-GNP nanodolgularla takviyesi sonucunda ısısal ve elektriksel iletkenlik iyileştirmelerinin değerlendirilmesi

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Assessment of Thermal and Electrical Conductivity Enhancements in PC-PBT Blends Reinforced with Hybrid MWCNT-GNP Nanofillers

Highlights

- *Polycarbonate-poly(butylene terephthalate) blends*
- *MWCNT-GNP nanofillers*
- *Analysis by Differential Scanning Calorimetry (DSC)*
- *Analysis by Thermogravimetric Analyses (TGA)*
- *Electrical Conductivity*

Graphical Abstract

The graphical abstract explains the variation of thermal stability and electrical conductivity in polycarbonate-poly(butylene terephthalate) (PC-PBT) blends with the addition of MWCNT-GNP nanofillers.

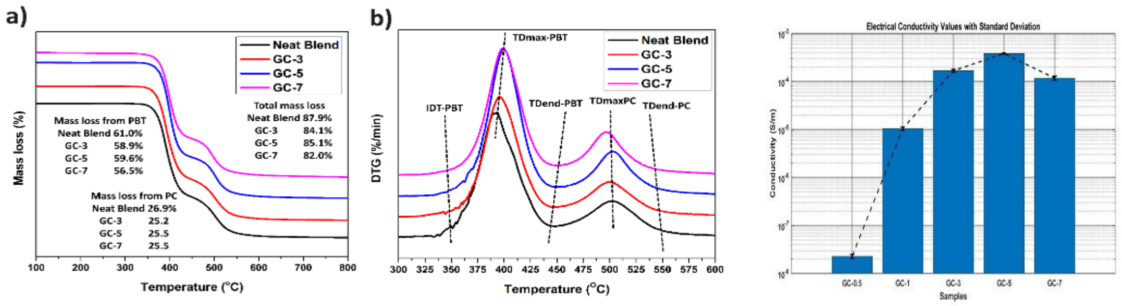

Figure. The prepared samples and analysis

Aim

It was aimed to examine the effect of MWCNT-GNP addition on thermal and electrical properties of polycarbonate-poly(butylene terephthalate) blends.

Design & Methodology

The study was carried out with the experimental studies based on DSC, TGA and Electrical conductivity.

Originality

The originality of this study lies in its detailed analysis of the effects of MWCNT-GNP addition on polycarbonate-poly(butylene terephthalate) (PC-PBT) blends.

Findings

The findings of the study suggest that incorporating MWCNT-GNP into polycarbonate-poly(butylene terephthalate) (PC-PBT) blends marginally enhances their thermal stability and substantially improves their electrical conductivity at elevated filler ratios.

Conclusion

The conclusion of the study indicates that adding MWCNT-GNP to polycarbonate-poly(butylene terephthalate) (PC-PBT) blends results in a modest increase in thermal stability and a considerable improvement in electrical conductivity at all filler ratios up to the optimum value of 5 wt. %. This suggests that hybrid MWCNT-GNP nanofillers can enhance the electrical and thermal properties of PC-PBT nanocomposites, making them more suitable for advanced engineering applications.

Declaration of Ethical Standards

The author(s) of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

Assessment of Thermal and Electrical Conductivity Enhancements in PC-PBT Blends Reinforced with Hybrid MWCNT-GNP Nanofillers

Araştırma Makalesi / Research Article

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ABSTRACT

This study aims to examine the improvement of thermal properties and electrical conductivity of PC-PBT blends through reinforcement with hybrid MWCNT-GNP nanofillers via melt-mixing. Differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) showed that the incorporation of nanofillers increased the crystallinity of the blends. On the other hand, a marginal decline in thermal stability was observed in the case of higher filler concentrations which was ascribed to the phase transitions within the polymer matrix. A conductive network was achieved with 5% wt. MWCNT-GNP weight fraction, and a notable reduction of 7 % was observed which was attributed to agglomeration effects. These findings reveal the importance of optimizing nanofiller concentration to achieve superior thermal and electrical performance in hybrid nanocomposites.

Keywords: Polymer-matrix composites (PMCs), nanomaterials, thermal properties, DSC, TGA, electrical conductivity.

PC-PBT Karışımlarının Hibrit ÇDKNT-GNP Nanodolgularla Takviyesi Sonucunda Isısal ve Elektriksel İletkenlik İyileştirmelerinin Değerlendirilmesi

ÖZ

Bu çalışmada eriyik-karıştırma yöntemi kullanılarak ÇDKNT-GNP hibrit nanodolgu ile katkılanmış PC-PBT karışımlarının termal özellikleri ve elektriksel iletkenliklerindeki iyileşmelerin incelenmesi amaçlanmıştır. Diferansiyel tarama kalorimetresi (DSC) ve termogravimetrik analiz (TGA) sonuçları nano-katkı ilavesinin karışım kristalliklerini arttırdığını göstermiştir. Bunun yanında, yüksek katkı oranlarında numunelerin termal kararlılığında önemli bir düşüş gözlemlenmiş olup bu durum polimer matriks içindeki faz geçişlerine bağlanmıştır. Ağ. %5 katkı oranına sahip numunede iletken bir ağ elde edilmiş olup, %7'lik ağırlıkça katkı oranında elektriksel iletkenlikte düşüş gözlemlenmiş ve bu durum topaklaşma (aglomerasyon) etkisine bağlanmıştır. Elde edilen deneysel sonuçlar hibrit nanokompozitlerde üstün termal ve elektriksel performans elde edilmesi için nano-katkı kompozisyonunun optimizasyonunun önemini vurgulamaktadır.

Anahtar Kelimeler: Polimer-matrisli kompozitler (PMK), nanomalzemeler, termal özellikler, DSC, TGA, elektriksel iletkenlik.

1. INTRODUCTION

Improvement of thermal and electrical properties of polymer-matrix composites (PMCs) with nanofillers has attracted considerable attention [1, 2] owing to their potential applications across diverse industries. Polymer-matrix composite materials have become a widely utilized group of materials for achieving desired properties such as toughness [3], hardness [4], mechanical strength [5] and electrical/thermal conductivity [6, 7] in structural or functional components used in high-value industries such as aerospace, automotive, defense, biomedical, electronics, and energy [8]. Chemical or mechanical processes are employed in the production of polymer nanocomposites. The most widely employed fabrication methods include; i) intercalation method, where the filler size is gradually reduced to the nanoscale, ensuring the distribution of nano-platelet fillers within the polymer matrix, ii) in situ polymerization method, where the distribution of nanoparticles within a liquid monomer is achieved through the polymerization reaction, and iii) mechanical/melt method, where the intercalation of the polymer with nanoplatelets is achieved using the solution-mixing technique in an appropriate solvent. The polymer is dissolved in one solvent and the nanoplatelets in another. These solutions are then mixed, allowing the

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polymer chains in the solution to intercalate between the nanoplatelet layers and replace the solvent [9–14]. The same process can also be carried out without using a solvent through melt compounding or melt intercalation methods. In this method, a high molecular weight polymer is melted at elevated temperatures, and the filler is then incorporated into the polymer matrix under high-temperature shear forces. Therefore, this method does not require the use of chemicals or solvents [15]. Thermodynamic and kinetic factors necessitate the modification of the filler material to exfoliate within the matrix under shear conditions in this method [16]. The melt intercalation (or melt compounding) method is a popular technique for producing polymer nanocomposites due to its ease of processing, cost efficiency, versatility, eco-friendly characteristics, and compatibility with traditional polymer processing technologies [17]. Poly(butylene terephthalate) (PBT) is a semi-crystalline polymer produced through the polycondensation of 1,4-butanediol and dimethyl terephthalate. In this structure, terephthalate provides strong intermolecular bonds, while butane ensures the molecular chain mobility and flexibility necessary for the crystalline structure [18]. PBT possesses a high melting temperature (Tm=225°C) due to the molecular rigidity provided by p-phenylene groups and exhibits excellent chemical resistance to aliphatic hydrocarbons, gasoline, detergents, oils, and alcohols. However, its primary disadvantage is low fracture toughness, resulting in poor impact resistance. To improve the low impact resistance of PBT, polycarbonate (PC) is used. PC is composed of long linear polyester chains of carbonic acid and phenol, featuring phenyl and methyl groups that contribute to molecular rigidity, thus providing superior thermal properties and excellent impact resistance. Its amorphous structure and high glass transition temperature (Tg=145°C) endow it with exceptional structural stability, making it highly suitable for molding processes [19]. The blends of these two widely used thermoplastic polymers, namely PC/PBT blends have found extensive application particularly in automotive industry for molded components providing superior chemical, thermal and impact resistance against harsh conditions [20].

Carbon-based nanofillers such as nano-sized fullerene, graphene nano-platelets, graphite, carbon nanotubes (CNTs) and their derivatives draw significant attention in the research area of nanocomposites as reinforcing materials [21]. CNTs are nano-sized reinforcing materials used in various types of composites. Their superior physical properties such as extremely high aspect ratio and resulting superior mechanical, electrical and thermal properties [22], have put these materials in the focus of composite materials research in recent decades.

CNTs offer a kind of nano-sized reinforcement that is lightweight, has a hollow core, and has immense aspect ratio. Both theoretical and experimental studies showed that CNTs have exceptionally high mechanical properties such as strength, stiffness and flexibility, as well as electrical and thermal conductivity [23–25].

CNTs are classified as single walled (SWCNT) and multiwalled carbon nanotubes (MWCNT) based on the number of the rolled-up graphene layers constituting the structure [26, 27]. MWCNTs are composed of multiple number of rolled up concentric graphene layers with 0.34 nm spacing between the layers with diameter range of 10 to 200 nm and length of up to hundreds of micrometers [26,28]. They have been fabricated mostly by two chemical vapor deposition (CVD) methods, namely, injection CVD and floating catalyst CVD methods [29, 30].

As another type of carbon-based nano-filler, graphene and its derivatives such as graphene nanoplatelets (GNPs) have also received huge interest [31] with similar properties with CNTs and due to their ease of fabrication and cost efficiency [32]. These materials are technically unfolded planar version of CNTs in the form of platelet-shaped sheets of graphene [33] ranging between 10-30 in number [34]. They also demonstrate enhanced electrical conductivity and mechanical properties due to their extremely high surface area [34], such that, its electrical conductivity is higher than that of copper and silver [35].

During the fabrication stage of polymer nanocomposites, using a combination of two or more of the mentioned nanofillers can further improve the various characteristics of the produced composites with a synergistic effect [36]. Li et al. (2019) proposed molecular engineered hybrid nanofillers for fabrication of thermoplastic polyurethane nanocomposites having superior mechanical properties by using one-dimensional CNT and two-dimensional graphene (G) as the constituents of the hybrid nanofiller. Reportedly, addition of only 1 wt % G-CNT hybrid filler provided 1.9 and 2.9-fold improvement in the tensile strength and toughness of the composite, respectively [37]. Yazik et al. (2023) studied the effect of hybrid MWCNT and montmorillonite (MMT) nanoclay content on shape memory epoxy nanocomposites' mechanical properties. They reported that, the hybrid filler consisting of 3 wt% MMT and 1 wt% MWCNT produced a synergistic effect in terms of tensile and flexural reinforcement [38]. In another report, Raimondo et al. (2022) concluded that, their hybrid MWCNT – graphene nanosheet (GNs) reinforced nanocomposites outcompeted their counterparts with single nanofiller in terms of electrical properties [39].

This study aims to examine the effect of MWCNT-GNP nanofiller addition on thermal and electrical conductivity properties of PC-PBT blends. The blend ratio of 1:1 wt. for blend constituents, and hybrid filler ratio of 1:1 wt. for MWCNT/GNP nanofillers were determined based on related literature works and preliminary examinations. Afterwards PC-PBT/MWCNT-GNP nanocomposites were produced with hybrid nanofiller weigh fractions of 0, 0.5, 1, 3, 5 and 7 %. The crystallinity and thermal stability of the nanocomposites were assessed using differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). Additionally, the effect of different nanofiller concentrations on electrical conductivity was evaluated. The findings of this study will offer insights into the optimal nanofiller concentration necessary to achieve superior performance in hybrid nanocomposite systems, thereby enhancing their application in high-performance materials.

2. MATERIAL AND METHOD

Polycarbonate (LG-Chem grade) and poly(butylene terephthalate) (Pimadure grade: HS40N) were purchased from Aydin Plastic Co. Ltd. in granule form. GNPs (size: 3 nm, Ø: 1.5 µm, purity > 99.9%, specific surface area: 800 m2/g) and MWCNTs (purity > 96% and outside diameter < 8 nm) were procured from Nanografi Nanotechnology (Turkey). Neat PC-PBT blend and hybrid PC-PBT/GNP-MWCNT nanocomposite samples were fabricated via melt-compounding with a small-scale melt mixer (KÖKBİR, Turkey) (melt temperature: 260°C, crew speed: 30 rpm). PC / PBT and GNP/ MWCNT weight ratios were kept as 1:1. Hybrid nanofillers were added into the blends with the following weight ratios: 0.5, 1, 3, 5 and 7 wt.%. The non-filled and filled samples were labelled as PC-PBT (for the neat blend); and GC-05, GC-1, GC-3, GC5 and GC-7 for reinforcement ratios of 0.5, 1, 3, 5 and 7 wt.%, respectively. After melt mixing, the semi-finished product was processed via shredding, single screw-extrusion, granulation and the final products were obtained in film form (with ~1 mm thickness) via compression molding (250℃ melt temperature and 5 MPa pressure). Digital microscopy (Nikon-ShuttlePix P400R-Japan) was carried out to observe the dispersion state of the nanofillers via transmission of the light through the produced samples.

Differential scanning calorimetry (DSC) measurements were conducted to assess the cold-crystallization and melting characteristics of the samples produced in granular form, using an AHP differential scanning calorimeter with a sensitivity of 3.6 to 4.0 µV/mW. The heating cycle ranged from 50°C to 280°C at a rate of 10°C/min. Prior to the DSC measurements, tin calibration standard was used for temperature calibration of the DSC apparatus. During the calibration the temperature went up to 240 °C initially with 10 °C/min until 210 °C and then automatically switched to 1 °C/min to capture the accurate melting point of Tin. The result is corrected in the software in accordance with the reference melting point of Tin (231.9 °C). Thermal stabilities were evaluated with a thermogravimetric analyzer (TGA-Seiko), heating the samples from 40 °C to 800 °C at a rate of 10°C/min. Prior to the experiments, TG weight calibration was performed to ensure acquisition of precise weight change data and two reference pans within the calibration weight kit provided by the supplier with known differential weight were used for this purpose. During the weight calibration, verification criterion was set to ± 0.10 %. The crystallinity of the PBT constituent of the nanocomposite samples was evaluated using Equation 1.

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

where, X_C is % crystallinity, ΔH_{cc} is the cold crystallinity enthalpy obtained during differential scanning calorimetry measurement, ΔH_m is the fusion enthalpy, $\Delta H_m^0$is fully crystalline PBT's fusion enthalpy (142 J/g), and φ_{PC} and φ_{filler} are relative weight fractions of the PC constituent and the filler, respectively, calculated for the complete sample weight [40, 41].

The conductivity values in S/m were calculated based on the measured resistance values. A Keysight brand insulation meter, as shown in Fig. 1, was utilized for the measurements. The 2-probe method was employed during the measurements. The average of five measurements is recorded for each sample.

Figure 1. Conductivity Measurement Apparatus

3. RESULTS AND DISCUSSION

3.1. DSC and TGA Analysis Results

Fig. 2 shows the DSC heating curves of the neat and the filled blends, Table 1 shows the corresponding cold crystallization and melting points of the blends arising from the PBT constituent; as well as the degrees of crystallinity calculated for each sample as per Eq.1. In Fig. 2, the exothermic peaks which are more distinctive for the filled samples arise from cold crystallization of the PBT component, and the endothermic peaks relate to the crystallite fusion of PBT [40]. As indicated in Fig. 2 and Table 1, the neat blend exhibits the lowest crystallinity rate with 4.78% and the rate of crystallinity increases with increasing filler fraction. In terms of the crystallinity rate of the samples, ~50% increase is observed by 3% filler addition compared to the neat blend; ~13% increase is observed by GC-3 compared to GC-5; and ~12% increase is observed by GC-7 compared to GC-5. Thus, it is evident that, the presence of hybrid GNP-MWCNT nanofillers led to an increase in the crystallinity.

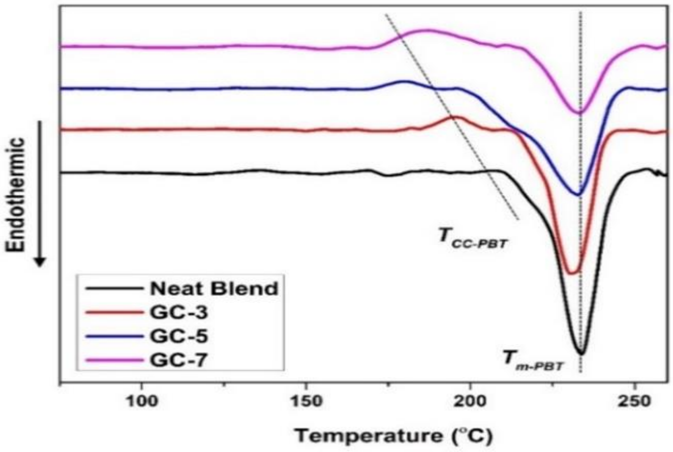

Figure 2. DSC heating curves of PC-PBT/MWCNT-GNP samples with varying filler content

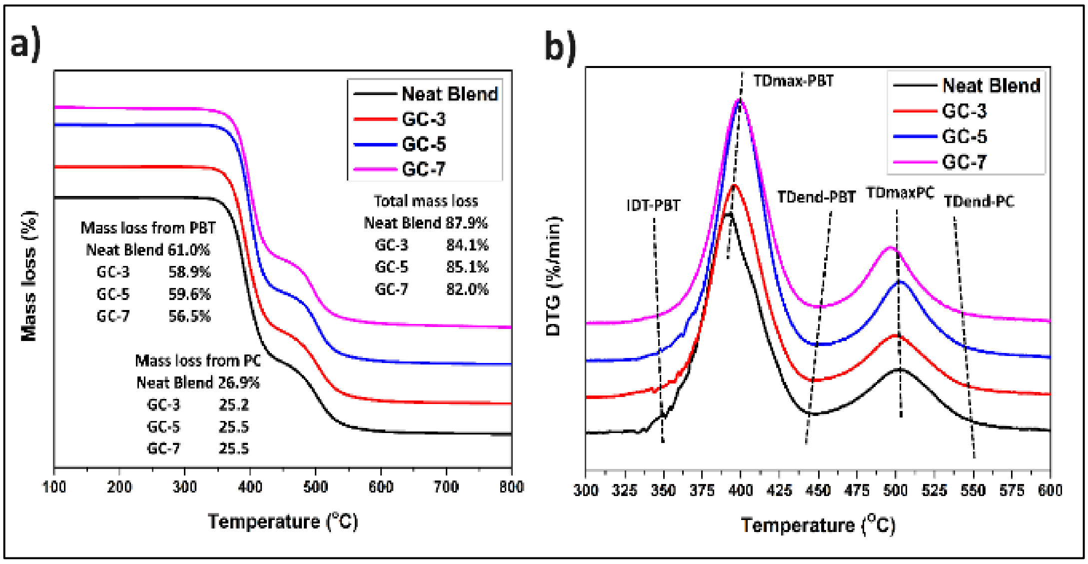

Figure 3. a) TG and b) DTG curves of the neat blend and the nanocomposites

Table 1. Cold crystallization and melting points; and degrees of crystallinity of samples

Sample	$T_{cc\text{-}PBT}$ (°C)	$T_{m\text{-}PBT}$ (°C)	X_c (%)
Neat Blend	203.18	233.73	4.78
GC-3	195.27	230.65	7.19
GC-5	180.186	232.7	8.14
GC-7	186.807	233.07	9.12

Fig. 3a shows the thermogravimetric and Fig. 3b shows the derivative thermogravimetric curves of the neat samples and of those produced with 3, 5 and 7% wt. filler fraction. Since the thermal stability of the nanocomposite constituents can be deemed as PC>PBT> Carbon-based fillers, a reduction in the mass loss of PC and PBT constituents may impede a clear interpretation of thermal stability, as a reduction in the mass loss is expected with increasing filler ratio regardless of the other factors. On the contrary, an increase in the mass loss of a constituent or the overall sample mass with increasing filler ratio relates to a reduction in the thermal stability. Thus, it can be concluded from the mass loss values in Fig.3a that, GC-3 (with 84.1% mass loss) has a higher thermal stability than GC-5 (with 85.1% mass loss) when the total mass loss is considered. A similar behavior is expected in the case of initial decomposition temperatures (IDT), the temperatures of maximum rate of mass loss (TD_{max}), and the temperatures of end of degradation (TD_{end}) shown in the derivative thermogravimetric (DTA) curves in Fig.3b and the corresponding values in Table 2, such that, these temperatures are expected to be slightly offset rightward (or increased) with increasing filler ratio when other factors (such as a positive or negative effect of hybrid filler on the thermal stability) are ignored. However, as shown in Table 2, increasing the filler ratio from 5% wt. to 7% wt. resulted in slight reductions in IDT-PBT (from 372.8°C to 371.0°C), TD_{max}-PBT (from 399.0 °C to 398.6 °C), TD_{end}-PBT (from 430.0 °C to

429.5 °C), TD_{max}-PC (from 502.9 °C to 497.3 °C) and TD_{end}-PC (from 539.2°C to 528.2°C) temperatures. When combined with the mass loss results derived from Fig.3a, it can be concluded that there is a slight reduction in the thermal stability of the samples as the filler weight fraction is increased gradually from 3% wt. to 5% wt. and from 5 wt.% to 7% wt., which can be also interpolated for the intermediate filler ratios of 0.5% wt. and 1% wt. This finding agrees well with the DSC findings indicating a transition from a PC-continuous phase to a less thermally stable PBT continuous phase. Although it cannot be postulated for the lower filler fractions (0.5% wt. and 1% wt.) to have improved thermal properties compared to the neat blend based on the present findings, it can be nevertheless concluded that, increasing the filler fraction do not impose serious impairment on the thermal stability particularly for the lower filler fractions.

3.2. Electrical Conductivity

The peak conductivity is achieved at a 5% concentration, registering at 3.82 x 10^{-4} S/m. However, at a 7 wt. % concentration, there is a significant reduction in conductivity, measured at 1.18 × 10^{-4} S/m. The observed reduction in conductivity at a 7% concentration may be attributed to the saturation of nanofillers or potential agglomeration, where an excess of nanofillers clusters together, thereby diminishing the effective conductive network. With the filler ratio of 1 wt. %, the nanoparticles within the blend seem to have achieved sufficient contact to form a conductive network as indicated by the sharp increase in the electrical conductivity value (Table 3). Although there are reports on a negative correlation between the percolation threshold and matrix crystallinity for polymer blends [42], the extent of such correlation cannot be precisely defined based on the findings of the current research. Thus, the increase in the electrical conductivity up to the filler ratio of 5 wt. % is rather ascribed to the homogeneous dispersion up to this filler ratio, which finding is also consistent with the optical images in Fig. 4 indicating increased level of agglomerations of nano particles. Apparent agglomerations and localizations of black dots are observed for the filler ratio of 7 wt.% in Fig. 4f, whereas in the other cases (Figs. 4b-e) a more homogeneous dispersion of nanoparticles is evident. Several reports are available on the positive impact of homogeneous nano-filler dispersion on the electrical properties of polymer nanocomposites [43,44]. In Fig.4a the microstructure of the neat PC/PBT blend void of black localizations confirms that the black regions in the other micrographs are representative of the GC nanofiller.

Table 2. Onset, maximum and end points of DTG peaks in Fig.3b.

Sample	IDT-PBT (°C)	TD_{max}-PBT (°C)	TD_{end}-PBT (°C)	TD_{max}-PC (°C)	TD_{end}-PC (°C)
Neat Blend	353.4	392.7	427.3	501.2	551.9
GC-3	362.9	395.5	430.9	499.8	545.0
GC-5	372.8	399.0	430.0	502.9	539.2
GC-7	371.0	398.6	429.5	497.3	528.2

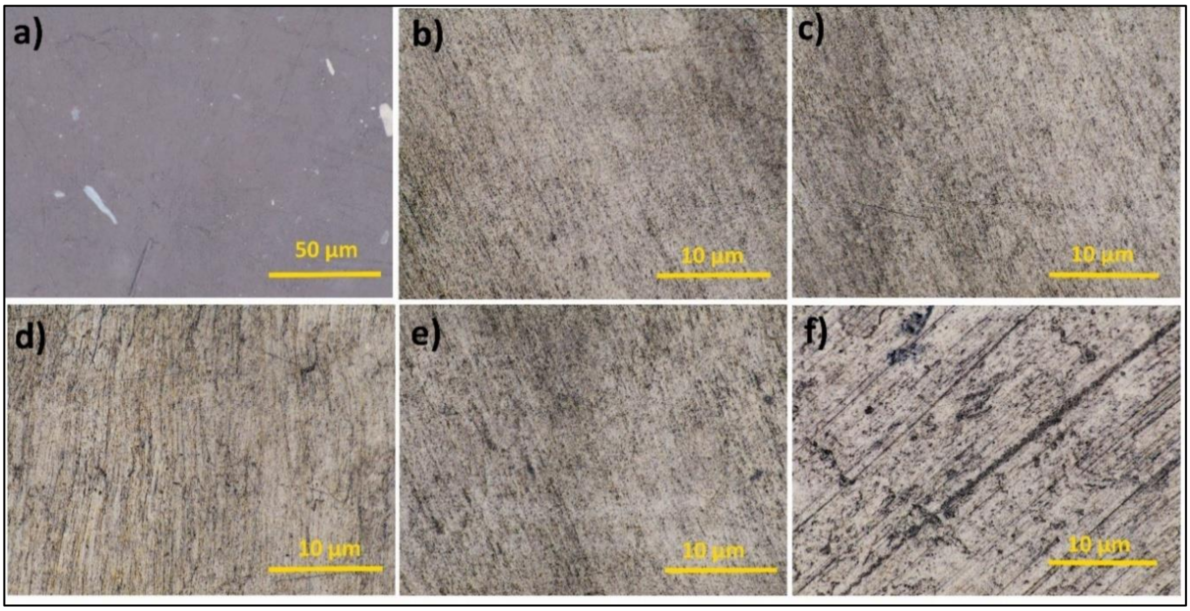

Figure 4. Digital microscope image of a) neat PC/PBT sample; b) GC-0.5, c) GC-1, d) GC-3, e) GC-5, f) GC-7 prepared in film form.

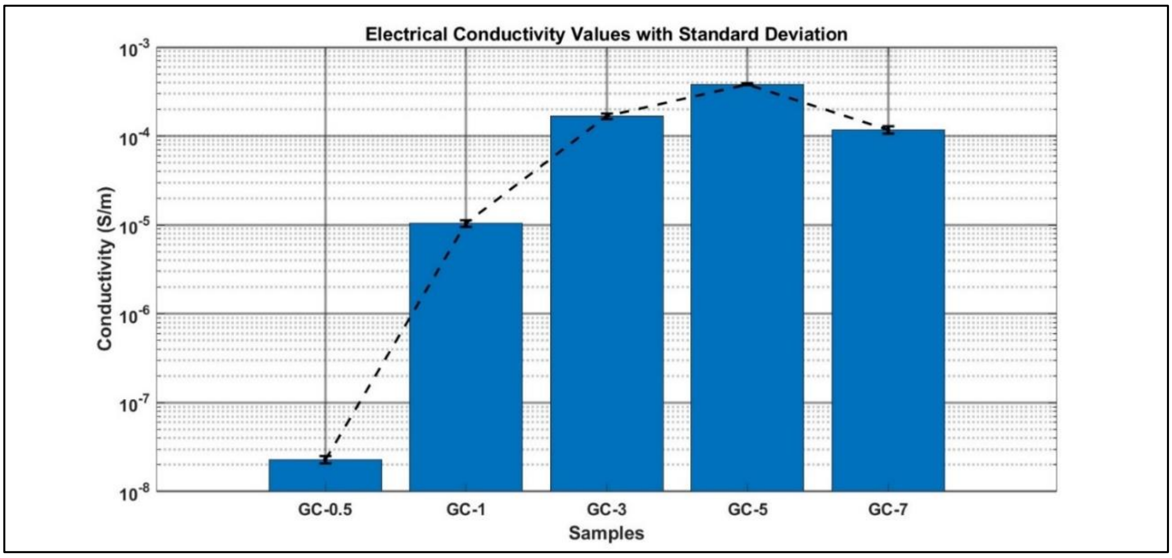

Figure 5. Variation of electrical conductivity values of samples

Fig. 5 shows the variation of electrical conductivity values for varying filler content and it can be clearly observed that a conductive network is achieved with GC-1 sample by an increase in the conductivity with ~3 orders of magnitude compared to GC-05 and the maximum value is reached with GC-5 sample.

Table 3. Electrical conductivity values (RSD: Relative standard deviation)

Samples	Conductivity (S/m)	RSD
GC- 0.5	2.29 x10^{-8}	9.78 %
GC- 1	1.04 x10^{-5}	8.85 %
GC- 3	1.67 x10^{-4}	6.38 %
GC- 5	3.82 x10^{-4}	2.18 %
GC- 7	1.18 x10^{-4}	9.26 %

4. CONCLUSIONS

This study investigates the improvement of thermal and electrical conductivity properties in PC-PBT blends through reinforcement with hybrid MWCNT-GNP nanofillers. The main findings of the study are as follows:

- The rates of crystallinity derived from DSC analyses increased with increasing filler weight fraction which is likely to have a positive influence on the mechanical and tribological properties of the blend.
- According to the DTA and TGA analysis results, thermal stability increased with increasing filler fractions which is attributable to phase transitions within the polymer matrix.
- A conductive network was achieved with 1 wt.% nano-filler ratio and the electrical conductivity peaked at 5 wt. % nano-filler concentration, beyond which a decline was observed due to the agglomeration of GC nanoparticles.
- These results underscore the necessity of optimizing filler concentration to achieve superior thermal and electrical performance in hybrid nanocomposite systems.

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DECLARATION OF ETHICAL STANDARDS

The author of this article declares that the materials and methods used in their studies do not require ethical committee approval or any legal-specific permission.

AUTHORS' CONTRIBUTIONS

Tuba ÖZDEMİR ÖGE: Conceptualization, supervision, methodology, investigation, data curation, writing-original draft, and writing-review and editing.

CONFLICT OF INTEREST

There is no conflict of interest in this study.

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