ORIGINAL ARTICLE

Graphene/poly(ethylene glycol) dispersions for fabrication of nanocomposites: effects of dispersion processing conditions

Vahid Shabafrooz[1] · Sudheer Bandla[2] · Jay C. Hanan[1,2]

Abstract

In the present research, ultrasonic exfoliation was employed to disperse graphene nanoplatelets (GNPs) in poly(ethylene glycol) (PEG) with two molecular weights. The GNP dispersions were added into poly(ethylene terephthalate) (PET) during injection molding to fabricate PET–GNP nanocomposites. The effectiveness of GNPs by weight (ε) in the nanocomposite was evaluated by the improvement in elastic modulus. The ε for the dispersion-dosed nanocomposite at 0.006 wt% was estimated to be 6.65 ± 4 and 20 ± 10 for PEG-600 and PEG-400, respectively. A review of sampled literature on PET nanocomposite elastic modulus improvement indicates that the effectiveness of GNPs obtained by dispersion dosing is significantly greater than other published results. Thus, the early promise of outstanding mechanical reinforcement by graphene in a polymer has been demonstrated.

Graphical abstract
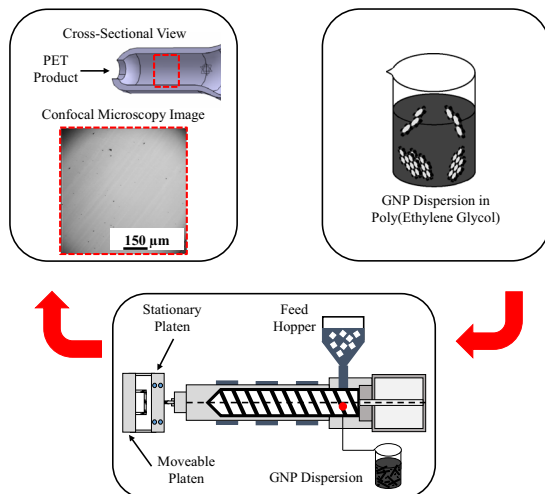
Keywords Poly(ethylene terephthalate) · Graphene nanoplatelets · Graphene dispersion · Ultrasonication · Nanocomposites

✉ Jay C. Hanan
  Jay.Hanan@gmail.com

1 School of Mechanical and Aerospace Engineering, Helmerich Advanced Technology Research Center, Oklahoma State University, Tulsa, OK 74106, USA

2 Research and Development, Niagara Bottling LLC, Ontario, CA 91761, USA

1 Introduction

Graphene is a planar, electrically conductive, elastic, crystalline allotrope of carbon that can be described as a one-atom-thick layer, arranged in a 2D, hexagonal pattern [1, 2]. Graphene can be produced in a form called graphene

nanoplatelets (GNPs). GNPs, with wide aspect ratio, represent a new class of graphene [3]. Graphene-based nanocomposites can consist of single-layer graphene sheets, few-layer graphene sheets, and multi-layer graphene sheets. Due to the differences in surface energies of the host polymer and graphene, graphene tends to agglomerate, which negatively impacts the properties of the nanocomposite [4]. Therefore, when producing graphene-based nanocomposites, the most significant obstacle is to obtain a uniform distribution of graphene sheets in the polymer matrix.

Research suggests that exfoliation and dispersion of graphene in various liquid media through ultrasonication is effective in improving distribution of graphene in polymers [5–10]. Graphene dispersions in an appropriate medium can be incorporated into polymer matrices through in situ polymerization. Depending on the chemical structure of the host polymer, different media can be selected to create a graphene dispersion. In an ideal situation, graphene would be uniformly incorporated into the polymer matrix. However, depending on the polymer molecule's chain structure, graphene may or may not affect the properties of the nanocomposite. For example, in the studies reported in polymerization of polymethylmethacrylate (PMMA)-based nanocomposites, Jang et al. [11] showed that graphene, due to its high surface area, can increase the glass transition temperature of the matrix. However, Liao et al. [12] did not observe an increase in the glass transition temperature. They discovered that this was due to the presence of surfactant molecules used to stabilize methyl methacrylate monomer (MMA) [12]. To explain this behavior, the interactions between polymer chains and incorporated graphene sheets need further investigation. One published method uses graphene dispersions prepared primarily using non-polar solvents. The non-polar solvents typically have high boiling points, leading to difficulties in both removal of the solvent during composite preparation and the formation of aggregations of graphene during solvent evaporation. Functionalization can improve the dispersibility of graphene. Functionalization can be divided into non-covalent (e.g., π–π interactions [13–15] and surfactants [16–18]) and covalent functionalization (e.g., graphene oxide (GO) [19] and reduced graphene oxide (rGO) [20, 21]). Using surfactants is the most promising route to improve the dispersion of graphene in a polar solvent [21]. However, surfactants can interfere with polymerization during the formation of polymer nanocomposites. The interaction between the surfactant molecules and the polymer matrix depends on the molecular structure of both components [22]. The presence of the surfactant molecules in a polymeric matrix can detrimentally alter the properties of the polymer. Removing surfactant molecules before incorporation into the polymer adds complexity to the

process and increases the production cost of nanocomposites [23, 24]. Based on the aforementioned findings, further investigation of dispersing graphene in a polar medium, without the aid of surfactants, is needed to incorporate them into a compatible polymer matrix and create nanocomposites with higher efficiency.

According to Zhang et al. [25], ethylene glycol (EG), a polar medium, can be used to disperse graphene. EG, due to its low viscosity, may not be a suitable medium for applications involving injection molding as low viscosity of the flow can cause screw slippage during the injection. For those applications, a medium with higher viscosity is needed to fabricate a uniformly distributed nanocomposite [9]. With the objective of improving the distribution of GNPs into the matrix, ultrasonication was used in this research to disperse and exfoliate GNPs in poly(ethylene glycol) (PEG), a compatible dispersion medium to poly(ethylene terephthalate) (PET). PEG with low and high viscosities, PEG-400 (M_w = 400 g/mol) and PEG-600 (M_w = 600 g/mol), was used to study the effects of the dispersion medium's viscosity on the stability of the GNP dispersions. PET–GNP nanocomposites were fabricated through injection molding via a dispersion dosing method. With an objective of understanding the effects of the addition of GNP dispersions and the dispersion medium on PET's properties, several process parameters (e.g., sonication time, amplitude, and centrifugal force) were undertaken to prepare the GNP dispersions. Experiments were designed to evaluate enhancement of nanocomposites, aiming to gain insight into how dispersion processing changed the properties of nanocomposites.

2 Experimental details

2.1 Materials

A commercially available grade of PET (M_w = 84,100 g/mol) was obtained from Leading Synthetics, Australia (Oz-PET TM GG-2180). N006 grade GNPs with surface area of 15 m²/g (GNPs-15) were obtained from Angstron Materials. PEG-400, obtained from Sigma-Aldrich, and PEG-600, obtained from the DOW Chemicals, were used to prepare the GNP dispersions.

2.2 Preparation of GNP dispersions

The GNPs-15 were dispersed in PEG-400 and PEG-600 dispersion media using an experimental procedure reported by Shabafrooz et al. [26]. Figure 1 represents the ultrasonication method used in this research to prepare the dispersions.
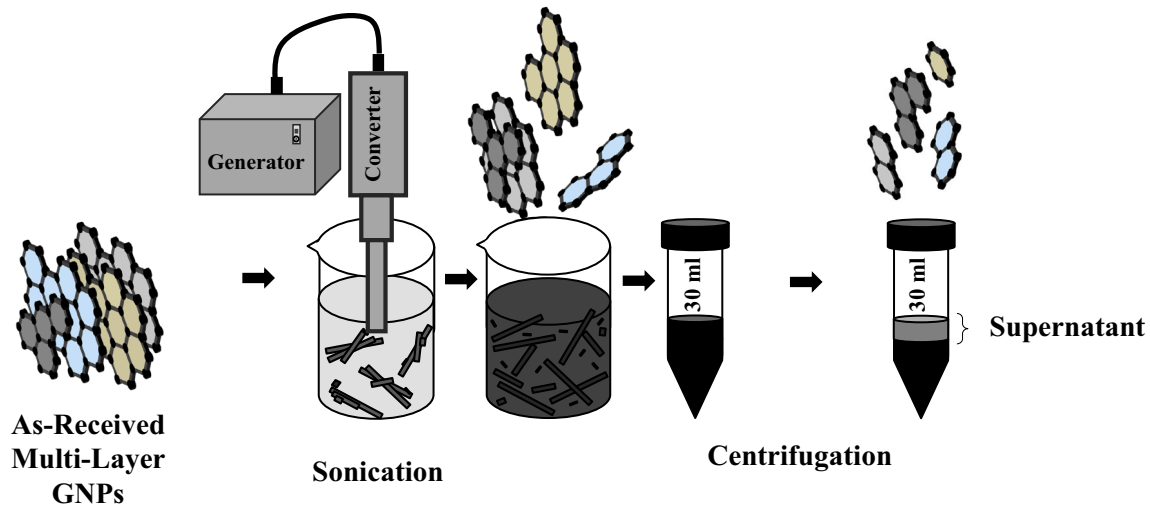
Fig. 1 Schematic representation of the ultrasonication method to prepare GNP dispersions

To calculate the power density, the power delivered to the convertor with the probe in air (not immersed in the sample) can be recorded. Without changing the amplitude, the probe should be immersed into the sample and the amount of delivered power can be again recorded. The difference between power readings is divided by the area of the probe to calculate the power that is being delivered to the sample (power density) using the following equation:

$$P = \frac{P_2 - P_1}{\pi r^2} \tag{1}$$

where P, power density (W/cm^2); P_2, power when probe is immersed (W); P_1 power when probe is in air (W); r radius of probe tip (cm).

Using the power density, the total input energy delivered during sonication can be calculated using the following equation:

$$E = P \times t \tag{2}$$

where E, energy density released to the sample during sonication (J/cm^2); P, power density (W/cm^2); t, time (s).

In a study conducted by Shabafrooz et al. [9], it was found that PET–GNP nanocomposites fabricated through PEG-GNP dispersion dosing outperformed those that were prepared using a masterbatch route. To further understand the effects of dispersion dosing, the nanocomposites were produced by dosing GNP dispersions, prepared by varied process parameters. Individual studies were conducted on the effects of centrifugation, concentration of dispersions, and different sonication times and amplitudes employed during dispersion preparation on the final properties of PET–GNP nanocomposites. Using a 19-mm probe, a P_1 value of 5 W and P_2 values of 70 W and 140 W were recorded for the amplitude of 50% and 100%, respectively. Power density and energy density were evaluated using

Eqs. 1 and 2 and were used to study the influence of sonication energy. The concentration of GNPs in PEG after centrifugation was estimated using an experimental procedure reported by Khan et al. [27]. The final concentration of GNPs in PET was evaluated based on the GNP dispersions used for dosing and the process parameters involved in injection molding. Tables 1, 2, and 3 summarize the process parameters that were involved in each study.

2.3 Preparation of PET nanocomposites

PET nanocomposites were fabricated through injection molding using PEG-GNP dispersions. A custom mold, based on the ASTM D 638 standard, was used for mechanical testing of cylindrical samples [28, 29]. The PET nanocomposites were prepared based on the process reported earlier [9]. Pristine PEG was dosed into PET during nanocomposite processing to fabricate PEG-dosed PET samples (labeled as PEG) as a baseline to isolate the effects of the addition of GNPs on the properties of the nanocomposites. Dosing equipment (U2011 dosing system—REPI, LLC) was utilized to add the GNP dispersions in the PET melt at a 1.5% dosing rate.

2.4 Characterization of PET nanocomposites

Mechanical testing was performed using an Instron Universal Testing Machine (INSTRON 5967), equipped with a 30-kN load cell. The effectiveness of GNPs by weight (ε) in the nanocomposites was evaluated based on the following equation:

$$\varepsilon = \left(\frac{E_2 - E_1}{\text{wt\%}}\right) \times \frac{1}{150} \tag{3}$$

Table 1 The process parameters involved in centrifugation study

Concentration of GNPs in medium (mg/mL)	Sonication time (min)	Sonication amplitude (%)	Centrifugation speed (RCF)	Concentration of GNPs in PET (wt%)
1	180	50	n/a	1×10^{-3}
1	180	50	260	9×10^{-4}
1	180	50	2350	4.5×10^{-4}

Table 2 The process parameters involved in concentration study

Concentration of GNPs in medium (mg/mL)	Sonication time (min)	Sonication amplitude (%)	Centrifugation speed (RCF)	Concentration of GNPs in PET (wt%)
5	180	50	n/a	0.006
10	180	50	n/a	0.01
15	180	50	n/a	0.02

Table 3 The process parameters involved in sonication energy study

Concentration of GNPs in medium (mg/mL)	Sonication time (min)	Sonication amplitude (%)	Centrifugation speed (RCF)	Concentration of GNPs in PET (wt%)
10	30–180	50 and 100	n/a	0.01

where ε, value representing the effectiveness of GNPs by weight; E_2, elastic modulus of the PET–GNP nanocomposite; E_1, elastic modulus of PET; wt%, weight fraction of GNPs used to prepare the nanocomposites; 150, theoretical value for elastic modulus of the fully crystalline PET phase (GPa) [30].

Differential scanning calorimetry (DSC—Q2000) was carried out to understand the effects of GNPs on thermal properties of PET. An experimental procedure, reported by Shabafrooz et al. [31], was used to perform DSC testing and to evaluate the percent crystallinity using the following equation:

$$X_c = \left[\frac{\Delta H_f - \Delta H_{cc}}{\Delta H_c^\circ} \right] \times 100 \tag{4}$$

where X_c is percent crystallinity; ΔH_f is the heat of fusion (J/g); ΔH_{cc} is the heat of cold crystallization (J/g); ΔH_c° is the heat of fusion for 100% crystalline polymer, 140.1 J/g for PET [32].

To understand the thermal properties, the heat of fusion and the heat of cold crystallization were collected from the first heating cycle to determine the crystallinity. Luminosity of the PET nanocomposites was measured using a VIS-IR spectrophotometer (UltraScan PRO—HunterLab).

3 Results

3.1 Study I: effects of centrifugation

3.1.1 Mechanical properties

Figure 2 represents measurements of elastic modulus and tensile strength of PET, PEG, and PET nanocomposites prepared in the centrifugation study. Initially, GNP dispersions were prepared at a concentration of 0.001 wt%. Centrifugation at 260 RCF and 235 RCF was employed, resulting in final concentration of GNPs in PET estimated at 0.0009 wt% and 0.00045 wt% (Table 1). As shown, there seems to be an increasing trend in modulus of both PEG samples compared to pristine PET. While there are no significant improvements in modulus of the PEG samples after dosing dispersions before and after centrifugation, the tensile strength of PEG-600 samples, however, increased by 11% after dosing GNP dispersion at 0.001 wt%.

3.1.2 Thermal properties

Figure 3 represents crystallinity measurements of the PET, PEG, and PET nanocomposites prepared in the centrifugation study (Table 1). As shown, the addition of PEG-400 leads to a higher improvement in crystallinity of PET
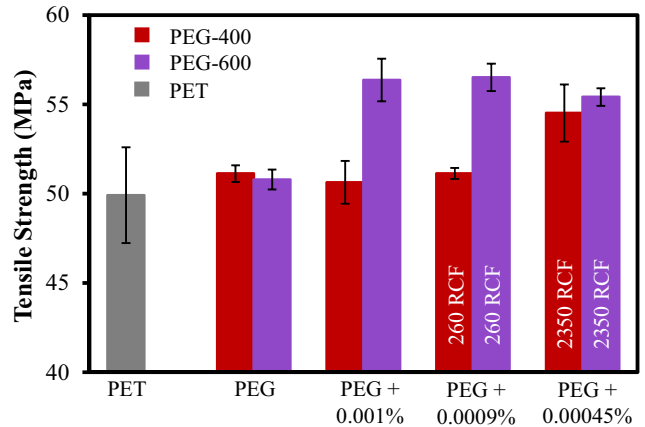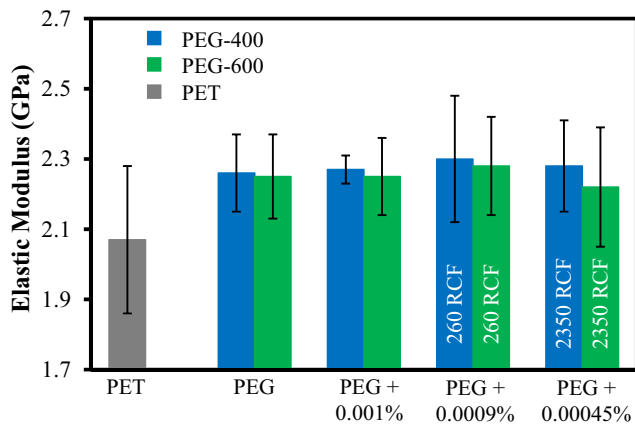
Fig. 2 Measurements of the elastic modulus and tensile strength of PET, PEG, and PET nanocomposites prepared by PEG-GNP dispersions dosing through centrifugation study
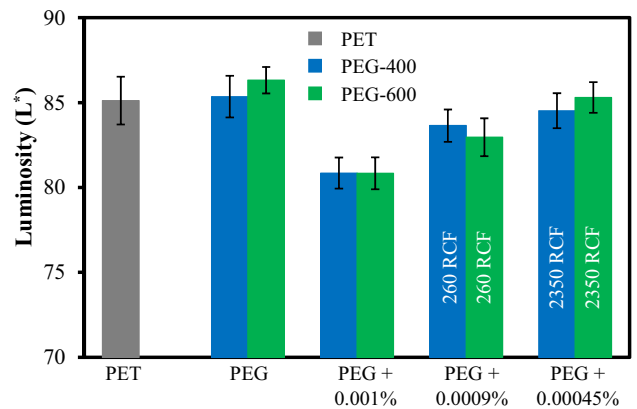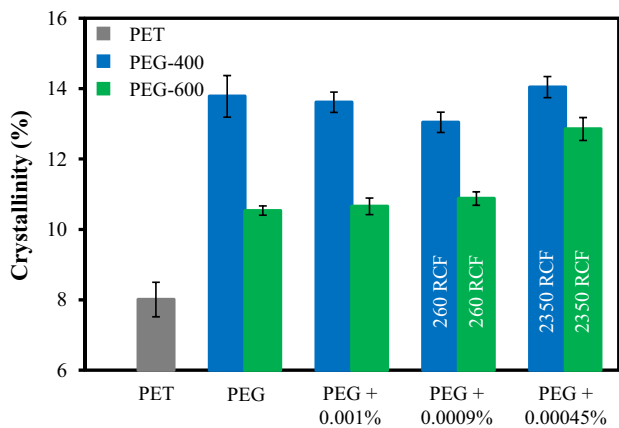

Fig. 3 Crystallinity measurements of PET, PEG, and PET nanocomposites prepared by PEG-GNP dispersions dosing through centrifugation study

Fig. 4 Luminosity measurements of PET, PEG, and PET nanocomposites prepared by PEG-GNP dispersions dosing through centrifugation study

matrix compared to PEG-600 dispersions. It was found that the addition of PEG-400 and PEG-600 improved the crystallinity of the PET matrix by 72% and 32%, respectively. A maximum of 22% improvement in crystallinity was observed for the sample prepared through the addition of GNP dispersions in PEG-600 at 2350 RCF.

3.1.3 Luminosity analysis

Figure 4 represents the luminosity measurements of PET, PEG, and PET nanocomposites prepared by PEG-GNP dispersions dosing method. While the addition of PEG did not affect the luminosity of PET samples, increasing the centrifugation speed of the PEG-GNP dispersions increased the luminosity, making them lighter in color. Based on the previous sections, it was found that increasing

the centrifugation force from 260 RCF to 2350 RCF had a positive impact on mechanical and thermal properties of the PEG samples. Although the dispersions contained a significantly lower concentration of GNPs, the data showed improvements in tensile strength (Fig. 2) and crystallinity (Fig. 3) of the PEG samples.

3.2 Study II: effects of concentration

3.2.1 Mechanical properties

Measurements of elastic modulus and tensile strength of PET, PEG, and PET nanocomposites prepared in concentration study (Table 2) are presented in Fig. 5. As shown, increasing the concentration of the GNPs in PEG can be detrimental to the mechanical properties of the matrix. The data showed a dispersion of PEG-400 and PEG-600 at
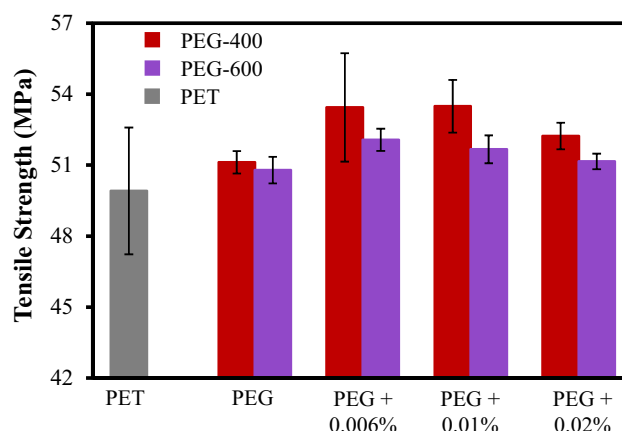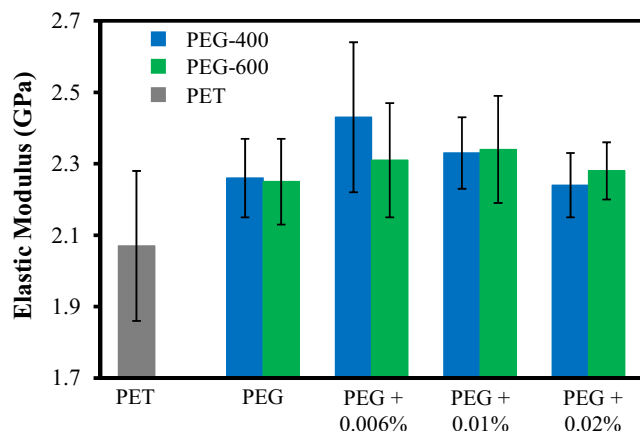
Fig. 5 Measurements of the elastic modulus and tensile strength of PET, PEG, and PET nanocomposites prepared by PEG-GNP dispersions dosing through concentration study

0.006 wt% GNP concentration improved the modulus of PEG samples at 8% and 3%, respectively.

3.2.2 Thermal properties

Crystallinity measurements of PET, PEG, and PET nanocomposites are presented in Fig. 6. While the addition of PEG-400 dispersions at concentrations ranging from 0.006 to 0.02 wt% did not impact the crystallinity of PEG-400 samples, the PEG-600 dispersion at 0.006 wt% concentration did increase the crystallinity by 11%.

3.2.3 Luminosity analysis

Figure 7 represents luminosity measurements. Increasing the GNP concentration to 0.02 wt% affects the luminosity of the samples, making them much darker compared to the control sample.
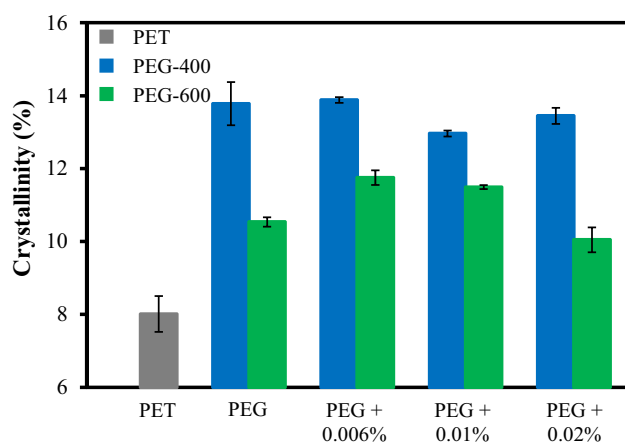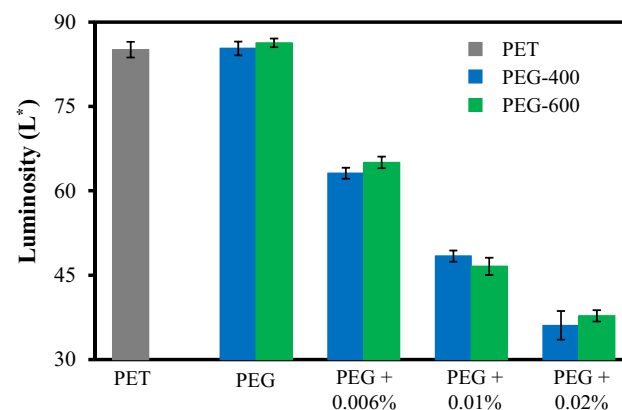

Fig. 7 Luminosity measurements of PET, PEG, and PET nanocomposites prepared by PEG-GNP dispersions dosing through concentration study

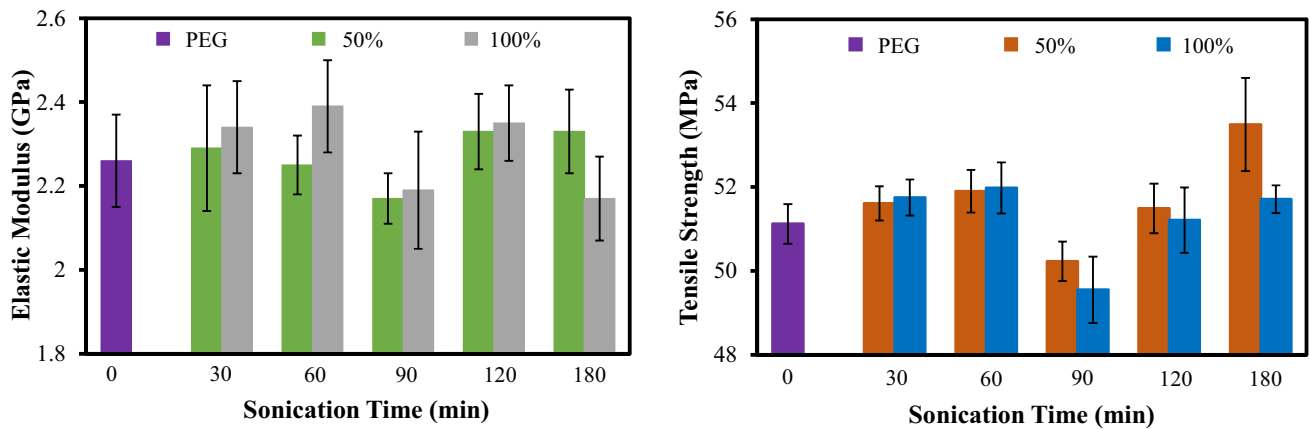## 3.3 Study III: effects of sonication energy

With an objective of understanding the effects of sonication energy on the properties of the nanocomposites, two different amplitude settings were used to prepare the GNP dispersions in PEG-400. The energy densities were estimated using Eqs. 1 and 2 in Sect. 2.2. Table 4 summarizes the energy densities calculated for different sonication times.

3.3.1 Mechanical properties

Figure 8 represents the measurements of elastic modulus and tensile strength of samples prepared by pristine PEG (with zero sonication time) and PET nanocomposites from the sonication energy study (Table 3). The results shown in Fig. 8 reflect an inconclusive effect of sonication energy on the elastic modulus. However, we see a 5% increase in tensile strength of the PET nanocomposites when the GNP

Fig. 6 Crystallinity measurements of PET, PEG, and PET nanocomposites prepared by PEG-GNP dispersions dosing

Table 4 Calculation of energy density for PEG dispersions prepared at 50% and 100% amplitudes

Sonication time (min)	50% amplitude		100% amplitude	
	Energy density (kJ/cm^2)	Energy density (10^{23} eV/cm^2)	Energy density (kJ/cm^2)	Energy density (10^{23} eV/cm^2)
30	41.4	2.58	86.4	5.39
60	82.8	5.16	172.8	10.78
90	124.2	7.75	259.2	16.17
120	165.6	10.39	345.6	21.57
180	248.4	15.50	518.4	32.35

Fig. 8 Measurements of the elastic modulus and tensile strength of PEG and PET nanocomposites fabricated by dosing PEG-GNP dispersions which were prepared at 50% and 100% amplitudes

dispersion, prepared at 180 min and 50% amplitude, was used. The energy densities of these data points are summarized in Table 4. The data here suggest that approximately the same amount of energy delivered over twice the duration (248.4 kJ/cm^2 over 180 min vs. 259.2 kJ/cm^2 over 90 min) is more effective to improve the tensile strength. Additionally, the delivered energy at 180 min and 100% amplitude (518.4 kJ/cm^2) results in no significant improvement in tensile strength. This suggests a certain energy threshold, used to produce the GNP dispersion, beyond which the dispersion does not improve. Further investigation is needed to establish a relationship between other energy deliveries (kJ/cm^2 per min) and the properties of the PET nanocomposites.

3.3.2 Thermal properties

Figure 9 summarizes the crystallinity measurements of PEG and the PET nanocomposites prepared by PEG-GNP dispersions dosing. With an exception of 30-min sonication time, it was found that the crystallinity of the nanocomposites is lower than that of PEG samples.
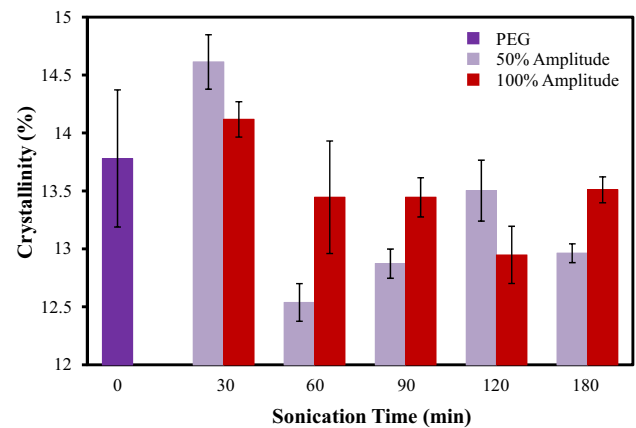
Fig. 9 Crystallinity measurements of PEG and the PET nanocomposites prepared by PEG-GNP dispersions dosing through energy study

3.3.3 Luminosity analysis

The luminosity measurements of PET nanocomposites prepared by PEG-GNP dispersions are shown in Fig. 10. As shown, higher-amplitude sonication leads to a lower value of luminosity, which makes the samples darker.
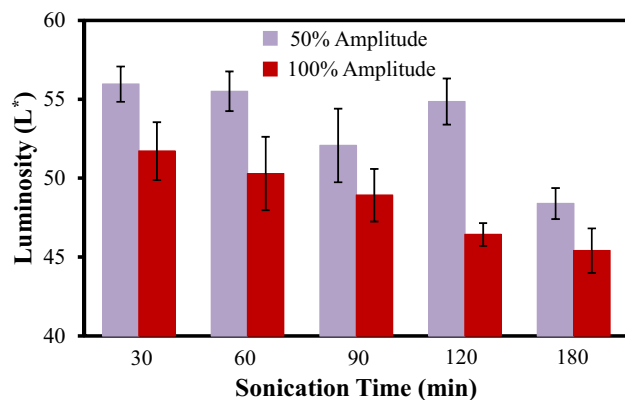
Fig. 10 Luminosity measurements of PET nanocomposites prepared by PEG-GNP dispersions dosing through energy study

Additionally, it was found that an increase to sonication time decreased the luminosity of the nanocomposites.

4 Discussion

As shown in Sect. 3.1, centrifugation, employed during dispersion processing, has a positive impact on luminosity of PET nanocomposites. Centrifugation separates out the unexfoliated GNPs from exfoliated ones. Increasing the centrifugation speed leads to a lower fraction of more light-absorbing unexfoliated GNPs, resulting in an increased luminosity of resulting PET nanocomposites. Based on the data collected in Sect. 3.2, it was found that the luminosity of nanocomposites was a function of the concentration of

GNPs, specifically the number of platelets per unit volume. An increase in the sonication time, and therefore energy density, during dispersion processing, decreases the luminosity of the nanocomposites by also increasing the number of platelets per unit volume by fracturing the GNPs during sonication (Sect. 3.3). GNP dispersions in PEG-400 at 0.006 wt. % improved the elastic modulus and tensile strength of PEG samples by 8% and 4%, respectively. This was higher compared to the effects of PEG-600 at 0.006 wt%, 3% improvements for both elastic modulus and tensile strength. No significant improvement was observed for the elastic modulus and the tensile strength of PEG samples when GNP dispersions of > 5 mg/mL were dosed. This is because at such high concentrations, the dispersions may never process well during sonication, resulting in a dispersion of agglomerated and poorly distributed multi-layer GNPs [9, 26]. The effectiveness of the abovementioned PET nanocomposites was evaluated using the equation proposed in Sect. 2.4. A review of a subset of the published data on PET nanocomposite elastic modulus improvements, summarized in Table 5, allows for a comparison of nanofiller effectiveness. This indicates that dispersion dosing is significantly more effective than previously published results.

Although PEG dispersions had positive impacts on the properties of the resulting PET–GNP nanocomposites, further research is required to characterize the samples to understand the effects of morphology of the GNPs under different conditions on physical properties. Raman spectroscopy has been previously utilized to characterize the

Table 5 List of a subset of the published studies on PET nanocomposites

Preparation method	E_1, PET (GPa)	Nanofiller	E_2, nanocomposite (GPa)	Concentration (wt%)	Effectiveness (ε)	Refs.
Polymerization	2.31	SWNTs[a]	2.08	0.0005	-3	[33]
Injection molding	0.90	MMT[b]	0.87	0.03	-0.006	[34]
Melt blending	1.80	PACP[c]	1.93	0.05	0.02	[35]
Melt extrusion	1.58	ZrPP[d]	2.22	0.05	0.08	[36]
Polymerization	2.21	MWNTs[e]	2.89	0.01	0.45	[37]
Melt blending	0.80	Graphene	1.0	0.0008	1.60	[38]
Polymerization	2.40	MWNTs	7.30	0.02	1.63	[39]
Polymerization	1.20	POSS[f]	4.40	0.01	2.13	[40]
Dispersion dosing	2.25	GNPs	2.31	0.00006	6.65 ± 4	PEG-600
Dispersion dosing	2.26	GNPs	2.43	0.00006	20 ± 10	PEG-400

[a]Single-walled carbon nanotubes

[b]Montmorillonite

[c]Hexakis (para-allyloxyphenoxy) cyclotriphosphazene

[d]Organic–inorganic hybrid-layered zirconium phenylphosphonate

[e]Multi-walled carbon nanotubes

[f]Polyhedral oligomeric silsesquioxane

GNP dispersions. Based on the results published by Shabafrooz et al. [26], GNP dispersions did not exhibit defects to the microstructure of the GNPs across the explored processing conditions. Therefore, further investigation using other techniques such as AFM and XPS can be considered to explore the PEG dispersions.

5 Conclusions

PEG shows promise to be used as a dispersion medium for GNPs in production of PET–GNP nanocomposites. Improvements in crystallinity, modulus, and strength of PET were observed in samples prepared by dosing. A significant improvement of over elastic modulus was observed in tensile strength with an initial concentration of 1 mg/mL used for dosing. Similar behavior was observed for the same dispersions after centrifugation, resulting in final GNP dispersions with concentrations of < 1 mg/mL. The higher viscosity of the medium leads to a more uniform dispersion of the exfoliated GNPs before and after centrifugation, resulting in an improved distribution of GNPs in the PET matrix. Further investigation is needed to better understand the influence of the dispersion medium on the properties of the PET matrix.

Compliance with ethical standards

Conflict of interest The authors declare that they have no competing interests.

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