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Enhancement of flexural modulus and strength of epoxy nanocomposites with the inclusion of functionalized GNPs using Tween 80

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Abstract

In this work, epoxy nanocomposite was prepared with the inclusion of unfunctionalized as-received GNPs (ARGNPs) and functionalized GNPs using surfactant Tween 80 (T80GNPs) in the epoxy resin using a mechanical stirrer. ARGNPs were used as it is, while T80GNPs were prepared through the adsorption of surfactant onto GNPs' surface using a sonication procedure in an ultrasonic bath. Characterization of nanoparticles using SEM shows that ARGNPs indicated a softer image representing a thinner layer of graphene stacks compared to T80GNP which has a tangible solid-looking image resulting from the sedimentation during the process of filtration. Elementally, both ARGNPs and T80GNPs were found to contain carbon, oxygen, and sulfur, as indicated by the EDX spectrum, with the C/O ratio for T80GNPs being 34.7% higher than that for ARGNPs, suggesting the adsorption of Tween 80 molecules on the GNPs after functionalization. FTIR spectroscopy confirms the attachment of Tween 80 molecules on GNPs surface with T80GNPs spectrum indicated higher peak intensity than ARGNPs. Flexural testing demonstrated that the addition of 0.9 wt.% ARGNPs and 0.9 wt.% T80GNPs to the epoxy increased the modulus of the nanocomposites to 72.1% and 82.6%, respectively, relative to neat epoxy. With the same amount of particle content, both nanocomposites showed increased strength, with ARGNPs and T80GNPs exhibiting strengths of 70.5% and 87.8%, respectively, relative to neat ероху.

Keywords: Graphene, Functionalization, Epoxy nanocomposites, Flexural modulus, Flexural strength

Introduction

Nanomaterials are tiny particles with dimensions in the nanoscale range on at least one side of their structure. They have attracted extensive research interest for their potential to enhance the properties of other materials when combined. Scientists have investigated several nanomaterials and documented their findings, which serve as references for further research in this field.



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Since its discovery in 2004, monolayer graphene has captured the attention of researchers due to its remarkable properties. These properties include being 200 times stronger than steel, modulus of ~ 1 TPa as well as being electrically and thermally conductive [1]. Additionally, it is lightweight and transparent. As a result of these advantages, researchers have expanded their studies to include similar particles with a similar structure. These particles include graphene oxide (GO), reduced graphene oxide (rGO), graphene nanosheets (GNs), and graphene nanoplatelets (GNPs). Because of their similar structure, these particles are typically grouped and referred to as materials within the graphene family [2].

Out of all the materials in the graphene family, graphene nanoplatelets (GNPs) have been extensively studied and reported on. They are easier and cheaper [3] to produce in large quantities compared to other particles. GNPs have been studied in various fields such as electrical and thermal conductivity [4] and mechanical properties [5, 6] and applied to many branches of the field including transportation [7] and construction [8].

Typically, in polymer nanocomposites, researchers often combine GNPs with epoxy resin to investigate the effects on the host polymer. The performance of nanocomposites is highly influenced by, firstly, the dispersion of nanoparticles within the matrix material. Nanoparticles based on carbon elements like GNPs and CNTs have strong van der Waals forces, causing each of these particles attracted from one to another [9, 10]. This condition is typically known as agglomeration. The formation of GNPs cluster and not evenly distributed in the matrix impart adverse effects to the final properties of the nanocomposites due to inefficient load-transfer of particles [11]. Secondly, nanocomposite performance is also greatly affected by the interaction between matrix-GNPs. It is also well known that carbon nanoparticles (e.g., GNPs) are hydrophobic which means they do not favor wet conditions [12]. This makes the intention to incorporate GNPs within matrix material more difficult due to the tendency to agglomerate and weak interaction with the host polymer. To tackle both of these problems, researchers are suggesting that the surface structure of GNPs be modified or functionalized prior the mixing with the matrix. This treatment involves adding a coupling agent or surfactant in a solvent which will repel particles from each other preventing agglomeration. At the same time, weak interaction with matrix material is improved by the existence of surfactant and chemical elements on the particles' surface structure through the reduction of surface tension [13, 14]. Studies have also examined mixing methods, with mechanical mixing being a common and user-friendly approach [15, 16].

Notably, most research has demonstrated improved properties in epoxy/GNP nano-composites compared to neat epoxy. For instance, Eayal et al. [17] added GNPs to epoxy resin and evaluated their mechanical properties. The results showed that microhardness increased by 8% in nanocomposites containing 4.5 wt.% of GNPs, compared to neat epoxy. However, other mechanical tests such as tensile strength and fracture strain decreased by 22% and 45%, respectively, in the nanocomposite containing 4.5 wt.% GNPs, likely due to the formation of aggregates and agglomeration of GNPs. This caused poor interaction and stress concentration within the epoxy matrix, leading to reduced tensile strength and fracture strain.

In a recent study, King et al. [18] added varying amounts of GNPs (ranging from 1 to 6 wt.%) to epoxy and tested their mechanical properties. They found that the addition

of GNPs resulted in an improvement in modulus, with the greatest enhancement (19%) seen in epoxy containing 6 wt.% GNPs, relative to neat epoxy. However, the study did not explain why the inclusion of GNPs improved tensile modulus. Despite the improvement in modulus, the tensile strength and strain decreased with the inclusion of GNPs. With the addition of 6 wt.% GNPs, the tensile strength decreased by 53.1%, and the tensile strain decreased from 8.0 to 1.4%, indicating brittle behavior. The researchers suggested that the reduction in strength and strain was due to structural disturbance caused by the presence of GNPs, which disrupted the molecular structure of the epoxy-hardener interaction compared to epoxy-hardener without GNPs.

In a study conducted by Prolongo et al. [19], it was found that the tensile modulus increased when 1 and 5 weight percent (wt.%) of GNPs were added to epoxy. The value jumped by 8.8% and 21.9% respectively, relative to neat epoxy. Additionally, the tensile strength increased by 2.8% but decreased by 39.1% for 5 wt.% GNPs, relative to neat epoxy. However, the study also showed that adding GNPs beyond 1 wt.% caused a decrease in tensile strain and toughness. The researchers concluded that adding GNPs to epoxy at 1 wt.% improved the properties of the matrix but increasing the GNP content up to 5 wt.% had a negative effect. The inferior properties were attributed to the excessive amount of GNPs, which caused wrinkling and debonding from the epoxy. This was evidenced by SEM images of the fractured surface. GNPs were also found to not act as a barrier for toughening, as they have weak interfaces and cause embrittlement of the whole system, as observed in SEM images with debonded indications.

This study involved creating a structural composite by combining GNPs with epoxy. Before mixing, the GNPs were functionalized using a surfactant known as Tween 80. The resulting nanocomposite was then compared to as-received GNPs/epoxy nanocomposites as well as neat epoxy to determine the impact of the functionalization on the overall system's properties. The novelty of this work lies in the utilization of Tween 80 as a surfactant for the functionalization of GNPs. Several significant effects after functionalization and performances when combined with epoxy could give some ideas on the GNPs dispersion and compatibility as well as epoxy/GNPs interfacial adhesion.

Methods

Materials

Graphene nanoplatelets (GNPs) and Tween 80 ($C_{64}H_{124}O_{26}$) were purchased from Sigma Aldrich through Permula Chemical and Kumpulan Saintifik Pt. Ltd., respectively. GNPs that have been used in this work were 5 µm of particle size (average), 15 nm thickness (average), surface area of 50–80 m²/g, bulk density of 0.03–0.1 g/cm³, and purity with oxygen content < 1% and residual acid content < 0.5 wt.%. Epoxy resin (Araldite LY556) that has been used is a diglycidyl ether of bisphenol A (DGEBA) and was bought from Huntsman along with a hardener triethylenetetramine (HY951) which was also purchased from Permula Chemical. Figure 1a shows the molecular structure of DGEBA with 2 epoxide groups at both ends, and Fig. 1b shows the molecular structure of triethylenetetramine curing agent, HY951.

Fig. 1 Molecular structure of **a** DGEBA araldite LY556, **b** triethylenetetramine curing agent, HY951, and **c** Tween 80

Modification of graphene nanoplatelets

Three types of samples were produced in this work which are neat epoxy, epoxy/ARGNPs (nanocomposite with As-received GNPs), and epoxy/T80GNPs (nanocomposite with functionalized GNPs using Tween 80, T80GNPs). Functionalization of GNPs using Tween 80 was prepared as follows. Firstly, in an empty cleaned glass bottle, 500 ml distilled water was prepared, and then by using a pipette, 10 ml of Tween 80 was mixed. This solution was then placed in an ultrasonic bath and sonicated for 30 min until the mixture became homogenous. In a separate container, 5 g of ARGNPs were weighed and then mixed with the solution containing distilled water and Tween 80. The resulting suspension was then sonicated in an ultrasonic bath for 4 h to ensure the adsorption of Tween 80 onto the GNP surface. After 4 h, the suspension was filtered, rinsed several times using distilled water until pH = 7, and then dried in an oven at 50 °C for 72 h. The produced GNP is termed as T80GNPs. The same procedure was repeated to get a sufficient amount of T80GNPs. Figure 2 illustrates the functionalization procedure of GNPs using Tween 80 in this work.



Fig. 2 Functionalization procedure of GNPs

Fabrication of epoxy/graphene nanocomposites

Preparation of nanocomposites was carried out by firstly weighing the desired amount of epoxy LY556 followed by curing agent HY951 with a ratio of 10:1. The desired amount of particles (either ARGNPs or T80GNPs) was determined by weight percentage (wt.%) relative to the weight of epoxy and curing agent. In this work, 0.3, 0.6, and 0.9 wt.% of either ARGNPs or T80GNPs were added into the system and mixed using a mechanical stirrer at 2000 rpm for 30 min. This weight fraction was used following previous work in the literature [20–22]. After that, the suspension was placed into the flexible mold made by polytetrafluoroethylene (PTFE) sheet and placed in a vacuum oven at 60 °C for 30 min to remove air bubbles. The final curing of suspension was carried out in a drying oven at 150 °C for 4 h. Sample for neat epoxy was prepared using the same procedure but without adding any GNPs (Fig. 3).

Characterization

Two types of characterization have been performed in this work. First is the characterization of GNP particles which focuses on the T80GNPs that have been functionalized using Tween 80. Secondly is the characterization of the performance of the cured nanocomposites when subjected to load (mechanical testing).

Characterization of GNPs

Scanning electron microscope (SEM) FEI Quanta 450 was used to examine the physical appearance of ARGNPs and T80GNPs. The image was captured at 8 kV using 2000 magnification. EDX was also performed to obtain the amount of elements in both particles so that the effects of functionalization could be observed. Fourier transform infrared spectroscopy (FTIR) was also performed to confirm the adsorption of Tween 80 on GNPs. The spectrum was recorded using Nicolet iS50 Thermo Fisher FTIR Spectrometer over the range of $4000-400~\rm cm^{-1}$.

Characterization of cured nanocomposites

Flexural testing was performed to determine the bending properties of the cured composites. It was carried out following ASTM D790 standard with each sample repeated 3 times (n=3) at a speed of 2 mm/min. Samples were prepared by cutting using a diamond cutter followed by polishing using 600 grit size sandpaper and finishing using 5000 grit size to eliminate sharp edges avoiding stress concentration during testing. Each

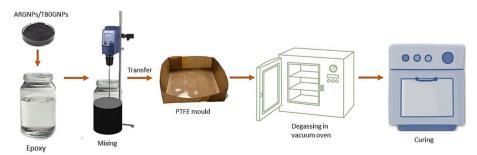


Fig. 3 Procedure to fabricate epoxy/GNPs nanocomposite

specimen was cut into 125 mm length, 12.7 mm width, and 3.2 mm thickness with a span distance between two support points of 52 mm. The fractured surface of the specimen was observed through SEM to evaluate the mechanism of the failure.

Results and discussion

Microstructural image by SEM

Figure 4 shows both images of ARGNPs and T80GNPs that were captured using SEM. From the figure, it can be observed that ARGNPs indicated a softer image of particles compared to T80GNPs. ARGNPs in Fig. 4a showed more characteristics for being a sheet-like structure compared to T80GNPs, the characteristics to which researchers refer to GNPs typically when they are describing GNPs shape. On the other hand, T80GNPs showed a more obvious solid image. The comparison between these two can be explained as follows. The preparation of ARGNPs by the manufacturer was carried out by a thorough drying process followed by a shaking procedure using a vibro-sieve that caused the separation of GNPs [23]. In contrast, T80GNPs which have been functionalized in this work appear to be solid flakes, representing agglomeration between particles since the T80GNPs layer sticks to each other during the process of filtration due to gravity. This was expected before the SEM image was taken. During the process of collecting those functionalized GNPs from the filter paper (after filtration), it can be observed that the T80GNPs are more tangible when touched using a spatula in terms of their state and form compared to ARGNPs. This indicates the agglomeration occurred due to gravity during the filtration process. However, despite being in an agglomeration state, it is believed that the T80GNPs will not affect dispersion within epoxy resin since the functionalized GNP has undergone further exfoliation during sonication and was encapsulated with Tween 80 molecules. Therefore, separation from agglomeration and uniform dispersion of particles within epoxy can be achieved at a high rate of spindle speed during shear mixing using the mechanical stirrer.

Energy dispersive x-ray spectroscopy

Figure 5 shows the EDX spectrum for both ARGNPs and T80GNPs. From the figure, it can be seen that 4 peaks appear for both samples with the first peak known as the noise peak or zero energy strobe resulting from the electronic noise [24, 25]. Another 3 peaks represent carbon, oxygen, and sulfur where carbon is known to be the main component

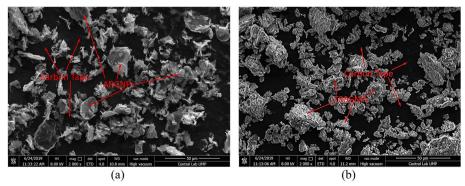


Fig. 4 SEM images of a ARGNPs and b T80GNPs

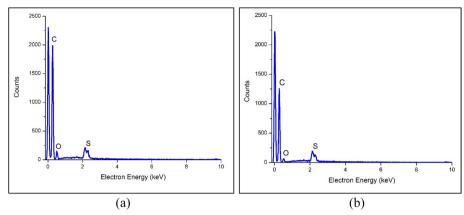


Fig. 5 Energy dispersive x-ray (EDX) spectra for a ARGNPs and b T80GNPs

of the hexagonal graphene structure. Oxygen and sulfur are probably introduced during the acidification process of graphite to convert into GNPs using acid sulfuric (H₂SO₄) [26]. In addition, it is well known that hydrogen is not indicated in the peak of Fig. 5 due to undetectable by EDX [27]. Moreover, no additional elements were introduced to the surface structure of GNPs after being functionalized with Tween 80. This is in agreement with the molecular structure of Tween 80 $(C_{64}H_{124}O_{26})$ which contains only carbon, hydrogen, and oxygen (Fig. 1c). The effects that can be observed from the EDX spectrum for both samples could be in the ratio of C/O where T80GNP indicated a higher ratio of C/O (Table 1). The higher amount of carbon on T80GNPs must have come from the surfactant where Tween 80 has attached and adsorbed to GNPs surface during 4 h of sonication process. This indicated that the functionalization process was effective and successful. In a previous study by Moral et al. [28], a similar method was used to assess the efficacy of oxidation treatment on carbon nanotubes (CNTs). From the EDX spectroscopy, they found that the oxygen content had increased while the calculated ratio of C/O had decreased after the oxidation functionalization. They reasoned that the result was attributed to the high amount of oxygen element in the treatment used. To confirm the adsorption of Tween 80 molecular structure onto GNPs as indicated by EDX, FTIR was also performed and discussed in the following subsection.

Fourier transform infrared spectroscopy (FTIR)

In FTIR spectroscopy, infrared radiation that hits the sample will cause the energy from the radiation to be absorbed by the molecule within the sample, producing vibrational and rotational movement of the molecules. The infrared radiation that was not

Table 1 Wt.% of each element for ARGNPs and T80GNPs

Element	ARGNPs (wt.%)	T80GNPs (wt. %)
Carbon	80.440	84.268
Oxygen	18.348	14.284
Sulfur	1.212	1.448
Ratio C/O	4.38	5.90

absorbed will pass through or be transmitted through the sample and recorded in the form of wavenumber resulting in the peak formation in the spectrum. Figure 6 shows the FTIR spectrum for both ARGNPs and T80GNPs that look identical but with tangible differences in the intensity of those peaks. T80GNPs exhibited higher peak intensity compared to ARGNPs due to functionalization using Tween 80 ($C_{64}H_{124}O_{26}$). The higher intensity in FTIR peak for T80GNPs occurred because the attachment of Tween 80 on GNPs caused higher interaction and higher absorption of infrared radiation compared to ARGNPs. Previously, a similar result was reported by Zhang and Chen [29] and Wang et al. [30] where identical FTIR spectrums were observed but with different peak intensities due to the effect of the surface modification process. In addition, from Fig. 6, both particles indicated several peaks at 1391 cm⁻¹, 1634 cm⁻¹, 2362 cm⁻¹, 2921 cm⁻¹, and 3434 cm⁻¹ which corresponds to the stretching bond of C-O, C = C, C = O, C =

Flexural properties

There are 2 analyses and comparisons that could be made from the flexural testing. Firstly, the effect of both nanoparticles on the properties of epoxy nanocomposite. Secondly is the effect of functionalized GNPs using Tween 80 (T80GNPs) on the flexural properties of epoxy nanocomposite compared to nanocomposite made by GNPs without any modification (ARGNPs).

Figure 7 shows the stress-strain curve from the flexural testing. In general, it can be observed that the modulus is higher for nanocomposites with either ARGNPs or T80GNPs compared to neat epoxy. As an example, by adding 0.9 wt.% T80GNPs and 0.9 wt.% ARGNPs into epoxy, the modulus of nanocomposite was increased by 82.6% and 72.1% respectively, relative to neat epoxy. This could be due to several reasons. Firstly, GNPs with high carbon content are known to have high modulus. Therefore, when

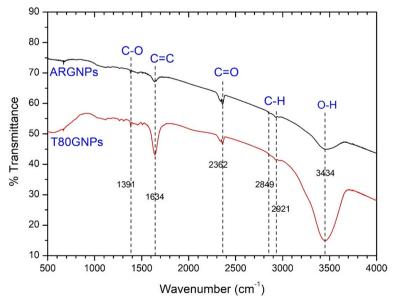


Fig. 6 FTIR spectrum of ARGNPs and T80GNPs

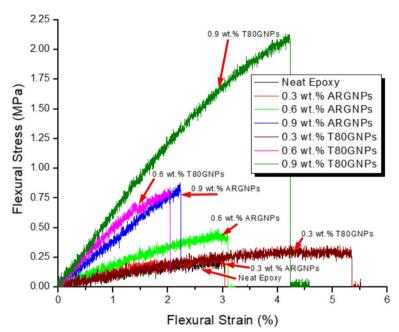


Fig. 7 Flexural stress-strain curve for neat epoxy, epoxy/ARGNPs, and epoxy/T80GNPs

combined with epoxy, the modulus of GNPs helped to boost the modulus of epoxy; hence, the improved modulus was recorded for epoxy/GNPs nanocomposite compared to neat epoxy [32]. This finding agrees with the rules of mixture for composite, which dictate that the elastic modulus of a composite material should be between the elastic moduli of its constituents [33, 34].

Secondly, in addition to modulus, the value of flexural strength is also improved for all nanocomposites regardless of particle content in the system compared to neat epoxy. For example, at 0.9 wt.% T80GNPs and 0.9 wt.% ARGNPs, the strength increased by 87.8% and 70.5% respectively, relative to neat epoxy. Higher strength was indicated by both nanocomposites compared to neat epoxy probably due to, those particles which were embedded within the host material, acting as barriers, attempting to block the crack propagation to get through to fail the structure. However, since the external force is too high for the sample to endure, the samples are finally broken but with a higher strength limit for epoxy/GNPs nanocomposite compared to neat epoxy. Several other groups [35–37] have reported a similar trend of modulus and strength when adding graphene nanoparticles into epoxy relative to neat epoxy.

To evaluate the effect of functionalization, a comparison could be observed between nanocomposites made by T80GNPs and ARGNPs. As mentioned earlier and as can be seen in Fig. 7, nanocomposites made by T80GNPs exhibited higher modulus and higher strength compared to nanocomposites made by ARGNPs. Firstly, this could be attributed to the improved interface interaction between T80GNPs-epoxy compared to ARGNPs-epoxy [38]. It is well known that materials rich with carbon elements can be considered fairly hydrophobic and a little bit difficult to get wet due to high surface tension. Functionalization which was carried out using surfactants like Tween 80 probably has lowered the surface tension of the carbon surface on the GNP structure. This happened when one end of Tween 80 molecules (typically known as the head of

the surfactant) was adsorbed onto GNPs while another end (typically known as the tail) interacted with epoxy. This creates a phenomenon which typically known as improved interface interaction. Due to this, T80GNPs are probably mixed better within epoxy with higher wettability compared to ARGNPs. Therefore, higher modulus and higher strength were exhibited by nanocomposite fabricated using T80GNPs compared to ARGNPs. Secondly, improved dispersion of T80GNPs in epoxy compared to ARGNPs could have been the reason. Earlier, it was stated that the SEM image of T80GNP nanoparticles on the carbon tape had a solid appearance, unlike ARGNPs. However, this was due to the gravity during the filtration procedure. With functionalization, the T80GNPs have been encapsulated with Tween 80 molecules on the outer surface of their structure. Therefore, when mixed with epoxy using a high-speed stirring rate of the mechanical propeller, the agglomerated GNPs (but encapsulated with Tween 80) will be able to separate and overcome the van der Waals forces between them. Hence, better dispersion was achieved which finally improved mechanical properties. Several other groups [39-41] have reported similar trends of modulus and strength when they added functionalized nanoparticles into epoxy and compared them with the behavior of unfunctionalized nanoparticles in the epoxy. However, it can be seen that the obtained flexural properties in this work are lower compared to the expected value. This is probably due to entrapped air within the sample as a result of imperfect processing conditions. Figure 8 and Table 2 show the summary of the flexural modulus and flexural strength of all samples.

The fractured surface of the sample from the flexural testing was evaluated using SEM to support the discussed results. This procedure is important because the fractured

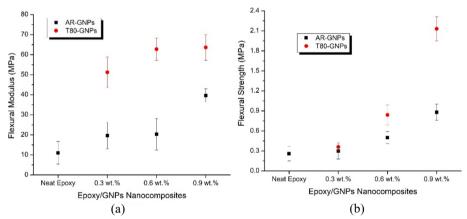


Fig. 8 Flexural modulus a and flexural strength b of epoxy-containing ARGNP and T80GNPs

Table 2 Flexural modulus and flexural strength

Particle content (wt.%)	Flexural modulus (MPa)		Flexural strength (MPa)	
	Epoxy/ARGNPs	Epoxy/T80GNPs	Epoxy/ARGNPs	Epoxy/T80GNPs
0	11.07 ± 5.6		0.26 ± 0.11	
0.3	19.6 ± 6.6	51.2 ± 7.6	0.3 ± 0.12	0.36 ± 0.06
0.6	20.29 ± 7.8	62.73 ± 5.6	0.5 ± 0.09	0.84 ± 0.15
0.9	39.65 ± 3.3	63.61 ± 6.4	0.88 ± 0.12	2.13 ± 0.18

image contains information about how the specimen failed. Figure 9 shows the images from SEM, representing each sample. From the image, it can be seen that the fracture surface of the pure epoxy (Fig. 9a) is smooth with hardly any contours, showing almost no obvious peaks and valleys. This indicates the failure with less or no obstacle and the specimen of neat epoxy resisted the flexural force with less barrier. In contrast, the image representing epoxy containing ARGNPs and T80GNPs as in Fig. 9b and c indicated a rougher surface due to the dispersion and distribution of nanoparticles, and hence more entanglement is shown within the nanocomposite structure. The dispersion of GNPs within epoxy has become a barrier for cracks to propagate. To break the specimen, the crack has to find other paths to bypass the barrier (GNP particles). As a result, higher strength and modulus were observed for nanocomposites containing ARGNPs and T80GNPs compared to neat epoxy.

Moreover, the image of epoxy-containing T80GNPs (Fig. 9c) is evenly and consistently rougher in all areas than epoxy-containing ARGNPs (Fig. 9b), indicating T80GNPs were dispersed better within epoxy compared to ARGNPs. The better dispersion of T80GNPs could have been achieved due to the sonication process and the addition of Tween 80 in the suspension during functionalization. As mentioned previously, the surfactant improved the surface tension of high surface tension of carbon nanoparticles like GNPs and encapsulated the GNPs to prevent agglomeration, reducing their hydrophobicity characteristic, to achieve better dispersion. Furthermore, during functionalization, GNP was probably able to exfoliate further, peeling off and creating more surface area to interact with epoxy resin when they were mixed. As a result, the barrier effects from T80GNPs were intensified, resulting in higher resistances from failing and finally contributing to a higher value in flexural strength and modulus. This can be evidenced by

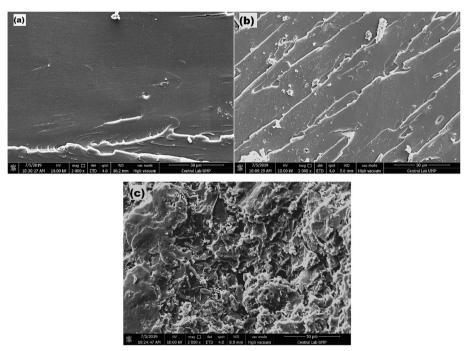


Fig. 9 SEM images of a neat epoxy, b epoxy/ARGNPs 0.6 wt.%, and c epoxy/T80GNPs 0.6 wt.%

a rougher image observed for epoxy/T80GNPs fractured surface compared to epoxy/ARGNPs as in Fig. 9b and c.

Conclusions

It can be concluded that the presence of both ARGNPs and T80GNPs in the epoxy resin increased the flexural properties of the nanocomposites relative to neat epoxy. At 0.9 wt.%, the modulus of nanocomposites containing both particles increased by 39.65% (epoxy/ARGNPs) and 82.6% (epoxy/T80GNPs) relative to neat epoxy. This is attributed to the fact that GNPs with high carbon content, possess high modulus. Hence, the resulting nanocomposites showed improvement compared to neat epoxy. In comparison between two nanocomposites containing T80GNPs and ARGNPs, higher modulus was recorded for epoxy/T80GNPs probably due to improved interface interaction between particle and epoxy. Flexural strength indicated a similar pattern of behavior where it was increased by 70.5% (epoxy/ARGNPs) and 87.8% (epoxy/T80GNPs) relative to neat epoxy. The dispersed particles within epoxy probably act as a barrier preventing the crack from passing through, hence higher strength was recorded. Epoxy/T80GNPs gave higher strength compared to epoxy/ARGNPs probably due to the same reason as in modulus with improved dispersion for T80GNPs compared to ARGNPs.

Abbreviations

GNPs Graphene nanoplatelets
ARGNPs As-received GNPs

T80 Tween 80

T80GNPs GNPs functionalized with Tween 80 SEM Scanning electron microscope

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Authors' contributions

WZAWA collected and analyzed the data. R Junid was the main supervisor of this research and contributed to writing the manuscript. N. Sazali contributed to funding the research through material acquisition, spectroscopy services, and resources. JP Siregar and JA Razak contributed to the conceptualization of the study and investigation. DF Fitriyana and T Cionita reviewed and edited the manuscript. J Jaafar helped in formal analysis and original draft preparation. All authors have read and approved the final manuscript.

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Availability of data and materials

The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Competing interests

The authors declare that they have no competing interests.

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