Study of Viscoelastic Behavior and Mechanical Characteristics of Graphene-Filled ABS Composites

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ABSTRACT

Acrylonitrile-Butadiene-Styrene (ABS) thermoplastic composites were reinforced with graphene fillers for three different weight ratios of 0.3%, 0.6%, and 0.9%. The compounding was prepared using a twin-screw extruder, and the specimens were made using an injection molding machine. The thermal gravimetric analysis (TGA)study revealed that the thermal stability of ABS composites was improved (~7%) by adding graphene fillers in pure ABS.The tensile and flexural strength of the ABS/grapheme composites was maximumwith the addition of 0.6 wt% graphene fillers. But the impact strength was reduced (~42%) for the addition of graphene fillers of more than 0.6 wt%. The stiffness of pure ABS was enhanced by more than ~27% with the addition of graphene fillers. It was found from a Dynamic Mechanical Analysis(DMA) study that the storage modulus and Tan Delta values of the composites were improved compared to pure ABS. The storage modulus was increased by more than ~70% over a wide range of temperatures. The addition of graphene fillers in the ABS matrix improved the glass transition temperature (increased from 110 ˚C to 118 ˚C). Scanning Electron Microscope (SEM) images confirmed the homogeneous mixing of the ABS matrix and the graphene fillers, and better dispersion was noticed with 0.6 wt% of graphene fillers.

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Introduction

For the last few decades, metallic components have been replaced by lightweight plastic and composite materials in the automobile and manufacturing industries. Acrylonitrile Butadiene Styrene (ABS) is one of the very commonly used engineering plastic materials in automobile industries because of its lightweight and ease of processing characteristics. The glass transition temperature of ABS material is 105 ℃, and it is used for various applications within a temperature range of – 20 ℃ to 80 ℃ because of its low melting temperature. Extensive research is available to enhance polymer matrix's mechanical, thermal, and viscoelastic characteristics by adding fillers. The purpose of adding fillers in thermoplastic polymers is to reduce cost and improve the physical, mechanical, and thermal characteristics. Shrinkage and warpage are the problems encountered while producing injection-molded plastic parts. These issues can be addressed by adding a certain amount of fillers to the plastic materials. Mechanical and tribological properties characterized the incorporation of ZnO as filler in a pure ABS polymer matrix. It was reported that the tensile and flexural strengths increasedup to 10 wt% of the filler content before declining. Still, the stiffness of the composites gradually increased with the increment in the wt % of the ZnO in the ABS matrix of the injection-molded composites [1].

Thermoplastic composites were also prepared with Polytetrafluoroethylene (PTFE) and nano zirconia as filler in the ABS matrix via compression molding. It is reported that the thermal stability, storage modulus, tensile strength, and flexural strength are improved. However, the damping properties are reduced due to the addition of nano zirconia [2]. Investigation results revealed that the inclusion of graphite in ABS material lowers the mechanical properties. In contrast, the tribological properties are improved as the wt% of graphite (Gr) increases, and it was concluded that 7.5 wt % of filler shows the best coefficient of friction and weight loss [3]. The ABS's thermal, mechanical, and electrical properties were enhanced by adding carbon nanotubes (CNT). The stiffening effects of ABS are improved for the (1, 2, 4, 6, and 8 wt%) of CNT addition, and the 6 wt% of CNT shows good electrical conductivity compared to pure ABS [4]. Incorporating nano alumina in ABS increases thermal stability but reduces the stiffness of pure ABS. The alumina nanocomposites and carbon nanotubes with ABS improve hardness, Young'smodulus, tensile strength, and flexural modulus [5]-[6]. The rheological, thermal, and mechanical properties were studied, and it reported that 20wt% of Gr in ABS decreases melt flow index (MFI) and ABS+20wt% of Gr composites exhibited the highest heat capacity (0.84 J/g) [7]-[8].

Graphene consists of many carbon atoms, which are arranged in hexagonal positions. Graphene and graphene oxide has better mechanical (hardness, elasticity) and thermal properties (thermal conductivity), and the

scientific community has used this material to improve the properties of other plastic materials. The strength of graphene is many times more than steel, and it finds applications in electronics, solar panels, and automotive electric vehicles. Graphene is incorporated into various polymers to enhance the properties of the polymer, and the results showed that it affects the mechanical and thermal characteristics of the matrix. It was also reported that graphene oxide could be used as a compatibilizer in ABS composites to improve the thermal and mechanical properties and enhance the adhesion between the matrix and filler. The tensile strength, flexural strength, and flexural modulus increased by 30, 15, and 90%, respectively [9]. Researchers used graphene as filler in thermoplastic materials like Polyamide (PA), High-density Poly Ethylene (HDPE), Poly Propylene (PP), and Polyethylene (PE) to improve the toughness, mechanical, thermal, and thermal conductivity [10]–[16].

It was reported that adding only 0.6wt% graphenes enhances the mechanical properties [17]. The optical properties of Poly-Methyl—Met-Acrylylate and Polyvinyl alcohol (PMMA-PVA)/graphene oxide composites improve the optical properties (absorption – 400%, absorption coefficient – 337%, reflective index – 51%), and these types of composites find applications in radiation shield and optoelectronic applications. The incorporation of graphene and graphite in polyester material reported improved viscoelastic and thermal properties. They characterized the properties using Dynamic Mechanical Analysis (DMA), Thermo gravimetric Analysis (TGA), and Scanning Electronic Microscope (SEM)and made the analytical inference of storage modulus and damping factor by Einstien and Neilson method, respectively [18]. It was noticed that the dispersion of graphene oxide made efficient chain grafting and improved the bonding between the matrix and filler [19]-[20].

The dynamic mechanical analysis (DMA) technique characterizes the viscoelastic properties of injection-molded plastic composites. It is one of the most commonly employed techniques to measure storage modulus, loss modulus, and the Tan Delta of thermoplastic composite materials. The plastic parts used for automobile applications are subjected to vibration with various frequencies and temperature changes.

In the dynamic mechanical analysis method, the storage modulus gives information concerning time and temperature. The stiffness of any material is directly proportional to the storage modulus, and the loss modulus demonstrates how the plastic material dissipates energy for the heat generated by molecular movement. When the temperature increases in any material, the molecules move faster, and the internal frictional resistance also increases. As a result, the heat is converted from one form to another. The damping factor Tan Delta is the ratio between the storage and loss modulus. The peak value of Tan Delta in dynamic mechanical analysis indicates the glass transition temperature of plastic composite. Tan Delta value indicates

the compatibility and interfacial adhesion between two phases of composite [23]-[27]. The heights of Tan Delta peaks are also an indication of bonding quality.

Research has been done to investigate the mechanical, thermo-mechanical, thermal, and dynamic mechanical characteristics of neat ABS by adding ZnO, nano zirconia, alumina, copper, and carbon nanotubes. Still, tothe author's knowledge, no such work has been reported on ABS/graphene composites prepared by injection molding [1], [3], [5]. This research aims to investigate the viscoelastic, thermal, and mechanical properties ofcomposites prepared by adding 0.3, 0.6, and 0.9 wt% of graphene via the injection molding process and compare the results with the neat ABS polymer. The SEM images revealed the uniform dispersion of graphene powder in the pure ABS. DMA, TGA, and Universal Testing Machine (UTM) instruments investigated the dynamic mechanical, thermal, and mechanical characteristics. The results showed that the presence of graphene filler improved pure ABS's storage modulus, mechanical properties, and thermal stability.

Methodology

Materials

Acrylonitrile Butadiene Styrene (ABS) of 1.06 g/cm^3 and melt flow index (MFI) of 28 gm/10 minutes were procured from Formulated Polymers, Chennai, in the granular form. The graphene powder of particle size 60 microns was purchased from Chem – O – Chem, a Chemical supplier in Chennai, India. Figure 1 shows macroscopic images of the procured raw materials ABS and Graphene.
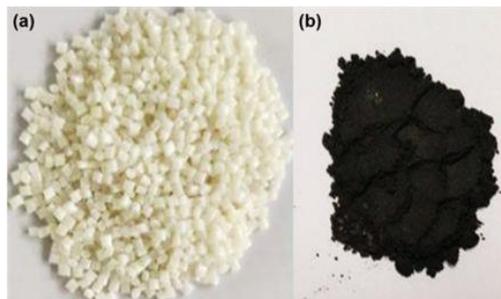

Figure 1: (a) Pure ABS, and (b) graphene

Compounding and sample preparation

The moisture of granular ABS material was removed by keeping it in an oven for 6 hours at a temperature of 80 ˚Cand then mixed manually with graphene powder of wt% 0.3, 0.6, and 0.9. The amount of ABS material was 2 kg for each composition, and the paraffin oil used was 10 mL to mix the ABS and graphene. A counter-rotating twin-screw extruder was used for the uniform dispersion of the graphene filler in the ABS matrix. Before compounding, both materials were kept for 4 hours at a temperature of 80 ˚C in an oven. The temperature ranges of the three zones in the twin-screw extruder barrel were maintained in the range of 220-260 ℃ and the screw speed was maintained at 80rpm. Water cooling was given at the exit of the extruder to remove the heat caused due to the extrusion process. Then it was passed through a pelletizer, and uniform sizes of granules were obtained.

The samples were prepared by injection molding technique in a 300 x 300 mm two-plate mold. The injection pressure for injecting the material was set at a pressure of 85 bar, a holding pressure of 50 bar, and back pressure was 15 bar. The overall cycle time per cycle was 68 seconds.

Characterizations

Thermo Gravimetric Analysis (TGA)

This method is commonly used to assess the thermal stability of polymeric materials. The loss of mass of the samples was measured using TGA Q50 V20.13 Build 39 instrument, and the temperature was increased from 30 ℃ to 800 ℃ with a heating ramp of 10 ℃/min.

Dynamic Mechanical Analysis (DMA)

The DMA tests were conducted on DMA (DMS 6100, SII Nano Technology, Japan) instrument (Polymer Nano Technology Center, BSACIST). The samples of dimensions 45 x 10 x 2 mm were heated at a rate of 3 ℃/min. The composites' storage modulus, loss modulus, and Tan Deltawere tested at1 Hz. The results were compared with the neat ABS.

Analysis of mechanical properties

The dog bone shape specimenwas obtained from injection molding dimensions 160 x 10 and 3 mm. The tensile test was conducted per international ASTM D638 using the universal testing machine of load cell capacity 5 kN at a crosshead speed of 50 mm/min. Test results were recorded, and the average value was calculated. The flexural strength of the composites was measured as per ASTM standard D790, and the specimen size was 100 x 10 x 3 mm.

Hardness is another important property of any material, and it was measured for the samples prepared using a digital-type hardness tester. The average values of a minimum of 5 samples were taken.

Scanning Electron Microscopy (SEM)

The morphological analysis of the composites was conducted using a scanning electron microscope (SEM Carl Zeiss, USA, Bruker,German). The dispersion of filler in the matrix and the voids created during the compounding of the matrix and filler were analyzed using SEM images with different magnifications.

Result and Discussion

Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) is a material characterization technique used to investigate the thermal stability of polymeric materials. In TGA, the mass loss with the temperature increase at a constant rate is measured. Particularly the plastic products, injection molded, and the polymers used for adhesives and food industries are analyzed the thermal stability as plastic materials lose their properties as the temperature changes [2], [6], [26].

Derivative thermogravimetry (DTG) is a derivative curve. It is possible to find the temperature at which the thermal degradation starts, the maximum degradation rate, the temperature at the end of thermal degradation, and the number of steps involved in the thermal degradation of the composite materials. The addition of graphene particles in the neat ABS matrix restricts the movement of the polymer chain, and as a result, the thermal property of ABS material improves.

The TGA graphs of ABS/Graphene composites prepared with 0.3, 0.6, and 0.9 wt% of graphene are represented in Figure 2. The degradation of ABS/graphene composites is observed in two steps. The degradation of the composite starts slowly in the first step at a temperature of 190 ℃. The loss of mass from 100% to 95% takes place over a temperature range from 190 ℃ to 390 ℃ due to degradation. During the second step of degradation, there is an abrupt drop in tweight lossat a temperature of 390 ℃ for all three ABS/graphene composites [28]. This degradation is related to the evolution of the volatile products derived from polybutadiene. It is observed that the thermal stability of ABS material is increased due to the addition of graphene filler. The ash content in ABS/graphene is larger than thatof pure ABS, and it may be one of the reasons to improve thermal stability.
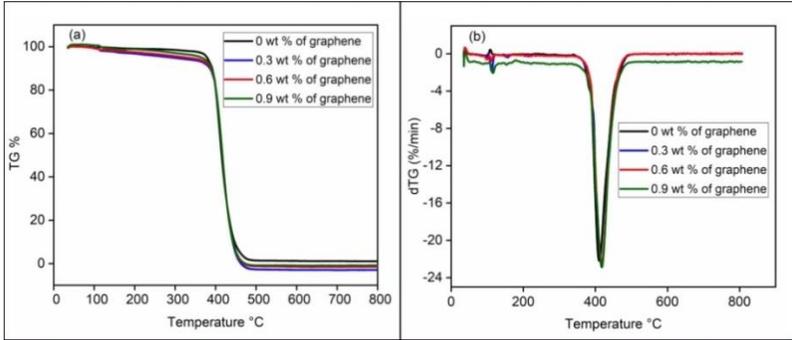
Figure 2: TGA Thermograms (a) temperature vs. TG% (b) temperature vs DTG

Dynamic Mechanical Analysis (DMA)

The DMA technique is used to understand the viscoelastic characteristics of ABS/graphene composites. The material properties may vary based on temperature, time, frequency, and stress. So, to understand these effects on the materials DMA test is important. In the DMA test, the storage modulus refers to the amount of energy stored in the polymer. The loss modulus is an indicator of the amount of energy the polymer dissipates. How the viscoelastic material absorbs and releases,the Tan Delta quantifies energy. The peak value of Tan Delta indicates the glass transition temperature.
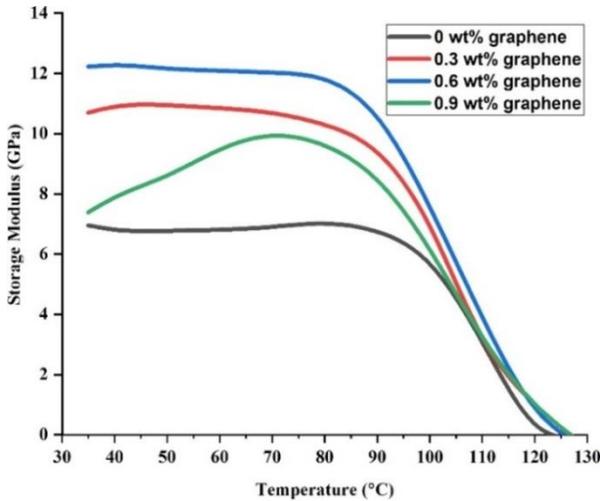

Figure 3: Effect of graphene content in storage modulus in ABS/graphene composite
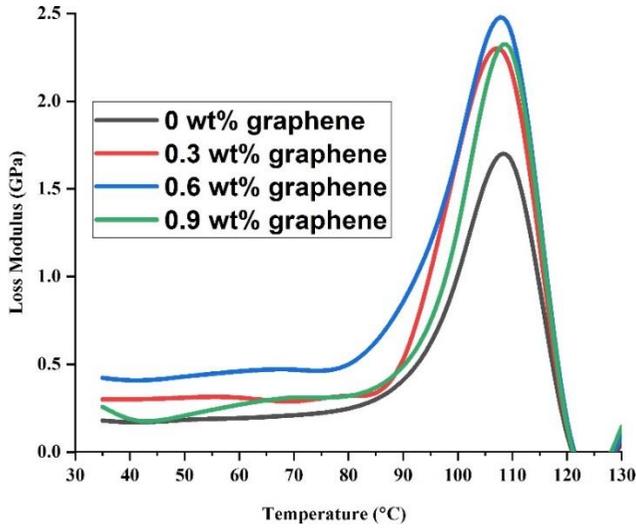
Figure 4: Effect of graphene content in loss modulus of ABS/graphene composite
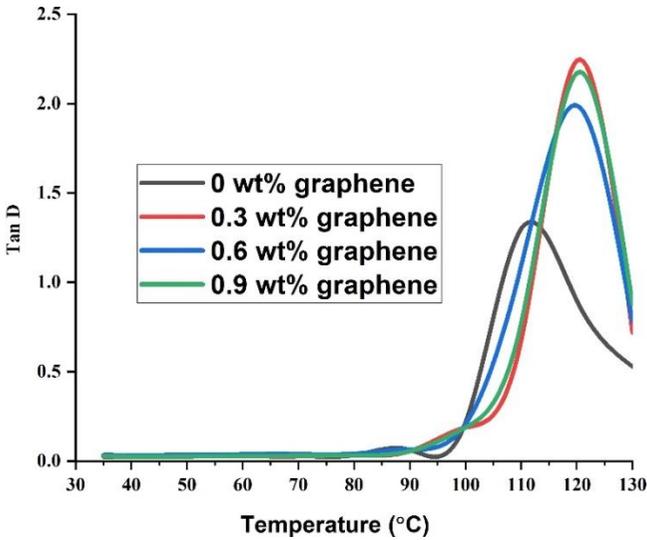

Figure 5: Effect of graphene content in Tan D of ABS/graphite composite

The mechanical properties correspond to changes in temperature examined by thermomechanical characterization. The storage modulus, loss modulus, and Tan Delta curves as a function of temperature are shown in

Figure 3, Figure 4, and Figure 5. From the DMA curves of the prepared composites, it was observed that the gradual increase of graphene filler in the ABS matrix has a substantial impact on improving the storage modulus at all temperature ranges, and similar results are reported by researchers [21]-[22], [25]-[27]. One of the main reasons for to increase in the storage modulus and glass transition temperature (T_g) may be the large difference in mechanical properties of the matrix and filler [27].

Table 1: Storage Modulus corresponds to different temperatures

Temperature in ℃	Storage modulus of Neat ABS in GPa	Storage modulus of ABS with 0.3 wt% of graphene in GPa	Storage modulus ABS with 0.6 wt% of graphene in GPa	Storage modulus ABS with 0.9 wt% of graphene in GPa
40	6.87	11	12.3	7.63
70	6.89	10.7	11.8	10.2
100	4.66	7.22	8.23	6.75

Table 1 shows that at all the temperatures, the storage modulus maximum corresponds to 0.6 wt% of graphene in the ABS matrix compared to the other composites. The storage modulus of 0.6 wt% graphenes in ABS at a temperature of 40 ℃ is 79% higher than that of pure ABS. Its modulus is much higher than the other composites, even at higher temperatures. The addition of graphene increases the storage modulus compared to neat ABS, but the optimum value corresponds to 0.6 wt% of graphene.

As the storage modulus decreases beyond 0.6 wt% of graphene, the probable reason may be the deterioration of chemical bonding between the ABS matrix and the graphene filler. The weak bonding is due to agglomeration of filler content beyond 0.6 wt% of graphene,which is evident from the SEM images (Figure 9) of composites. It is noticed that the storage modulus becomes very negligible beyond the glass transition temperature, and the curve is a rubbery plateau [25]-[26]. The increased stiffness is due to the homogeneous dispersion of graphene in the ABS material, which restricts the movement of a polymer chain, which is difficult to move.

From the storage modulus vs. temperature, it was observed that the storage modulus of the composites slowly decreases from 35 to 80 ℃ and its value falls abruptly from 80 ℃ to 120 ℃. At a temperature of 84 ℃, the storage modulus of 0.3, 0.6, and 0.9 wt% graphenes with ABS composites are 10.4 GPa, 11.2 GPa, and 9.6 GPa, respectively.

Tan Delta is the representation of the damping of the composite materials, and it also represents the glass transition temperature of polymer materials. From the Tan Delta vs. temperature curves, it is noticed that the

glass transition temperature of 0.3, 0.6, and 0.9 wt% graphene composites are 120 °C, 116 °C, and 115 °C, respectively.It was found that the addition of bentonite and alumina fillers in ABS improves the thermal stability and increases the glass transition temperature (T_g) of the ABS matrix [2], [5]. Compared to bentonite and alumina, the improvement in the glass transition temperature of ABS was found slightly more in graphene filler.

Mechanical characteristics

The mechanical properties of the composites are presented in Figure 6, Figure 7, and Figure 8. The shore hardness with the varying wt% of graphene is shown in Figure 6a, and with the addition of 0.6 wt% of graphene in the ABS, the matrix increases the shore hardness up to 93 and reduces when 0.9 wt% of graphene was incorporated in ABS [2], [6], [25], [28]. The impact strength vs. the wt% of graphene is represented in Figure 6b. It shows that the impact strength of ABS material continuously decreases with the increases in wt% of graphene content. It has been reduced from 187 J/m to 109 J/m, a reduction of 41.71% compared to the pure ABS material [2].
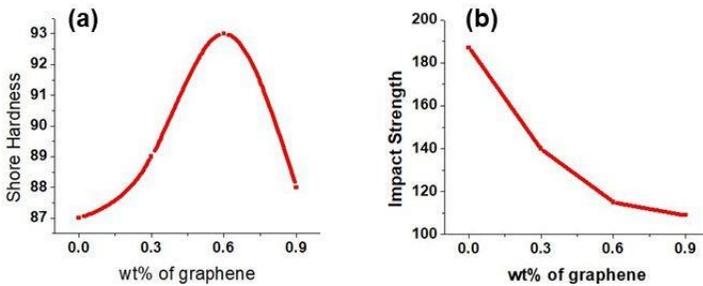

Figure 6: Effect of wt% of graphene in ABS matrix; (a) shore hardness, and (b) impact strength
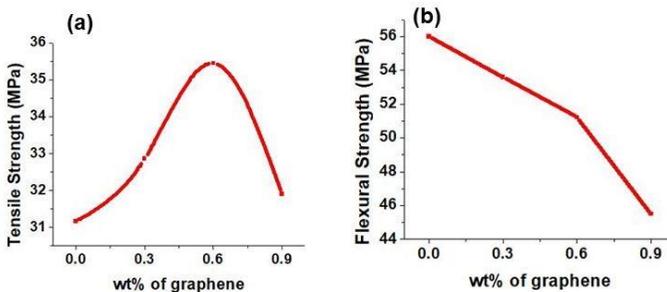

Figure 7: Effect of wt% of graphene in ABS matrix; (a) Tensile strength, and (b) flexural strength

The tensile and flexural strengths of the composites with the different wt% of graphene are shown in Figures 6a and 6b, respectively. The tensile strength increased with the increase of graphene content up to 0.6 wt% and was then found to decrease, but the flexural strength of the composites continuously decreased with an increase in the filler content.We can attribute this strength weakening to the result of poor dispersion of graphite content (0.9 wt% of graphene) in the ABS. With a smaller amount of graphene powder, the bonding between the matrix and filler was strong, and the tensile strength increased [5]. But at a higher graphene concentration, graphene dispersion was difficult, and agglomeration and voids reduced the tensile strength. The tensile strength is increased from 31.17 MPa of neat ABS to 35.45 MPa, corresponding to 0.6 wt% in the matrix [29]-[30], which has an increment of 13.73%, and the flexural strength is reduced from 56 MPa to 45.5 MPa (reduction of 18.75% compared to neat ABS). It was reported that the mechanical properties increased by 1.8 times with the inclusion of reduced graphene oxide in ABS [25]. It was also reported that the hardness and modulus improved by 49% and 61%, respectively, by adding MWCNT in ABS [6].
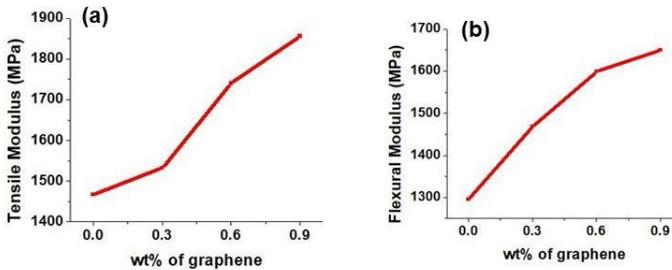

Figure 8: Effect of graphene content in ABS matrix (a) tensile modulus (b) flexural modulus

Figures 8a and 8b represent the tensile modulus and the flexural modulus of neat ABS and the ABS/graphene composites with varying graphene content ratios. The tensile and flexural modulus of the composites increased with the increase in the wt% of graphene in the matrix. The tensile modulus shows an improvement of 4.43, 18.6, and 26.51% with the addition of 0.3, 0.6, and 0.9 wt% of graphene in the thermoplastic ABS material. Similar results were reported by adding ZnO and nano zirconia in ABS [1]-[2]. The flexural modulus increases gradually from 1296 MPa to 1650 MPa, an increase of 27.31% compared to the neat ABS material [10]-[16]. The addition of graphene content in ABS improves the tensile and flexural modulus, which may be attributed to an increase in stiffness due to the

uniform dispersion of graphene filler, which restricts the chain mobility, making it difficult for the polymer chains to move.

SEM analysis

Figure 9 represents the SEM images of ABS/graphene composites varying wt% of graphene (0.3, 0.6, and 0.9). The dispersion of graphene filler in the ABS matrix is visible in the SEM images. The dispersion of graphene corresponding to 0.6 wt% is much better compared to 0.3 and 0.9 wt% of graphene in ABS. The homogeneity in mixing reflected in the dynamic mechanical and the mechanical properties, as it was represented the 0.6wt% of graphene gave better properties than the composites prepared with 0.3 and 0.9 wt% of graphene. The size of the voids in the ABS with 0.9 wt% graphenes is much larger than the other composites.The chemical bonding between the ABS matrix and the graphene filler was strong when a smaller amount of graphene was added to ABS. So, the properties were gradually increasedup to 0.6 wt% of graphene in ABS. But when a higher amount of graphene was added in ABS, the graphene powder hadhigh surface energy, and it was easy to agglomerate and also created voids. It made the composite weaker, and the mechanical properties started decreasing. It is mainly attributed to the weak interface interaction and weak chemical bonding between the matrix and filler.
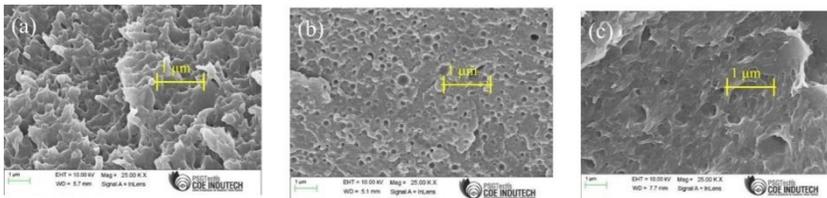

Figure 9: SEM Images; (a) ABS and 0.3 wt% graphene, (b) ABS and 0.6wt% graphene, and (c) ABS and 0.9 wt% graphene

Conclusions

The compounding of ABS and graphene was carried out by extrusion, and the specimen was prepared by injection molding. TGA, DMA, and UTM characterized the thermal, dynamic mechanical, and mechanical properties. As per the results obtained from the various characterization, ABS/graphene composites are promising candidates and potential future materials for automotive and 3-D printing applications. The DMA studies confirmed that the storage modulus of the pure ABS matrix improved with the addition of graphene content over a wide range of temperatures. The storage modulus is

enhanced by 70% over a wide range of temperatures. The tensile strength, flexural strength, and shore hardness of the ABS/graphene composite increased up to 0.6 wt% of graphene, and beyond 0.6 wt% of graphene in ABS, the properties decreased. The tensile and flexural modulus of the composites gradually increased with the increase in the graphene particulates. Still, the flexural strength of the pure ABS decreased with an increase in filler content. The stiffness of ABS/graphene composite corresponds to the 0.9wt% of graphene content in the ABS matrix, increased by 27% compared to the neat ABS. The TGA results revealed that the thermal stability of the pure ABS material improved due to the inclusion of graphene. The better dispersion of filler and the adhesion between the filler and matrix enhanced the viscoelastic, mechanical, and thermal properties of pure ABS material.

Contributions of Authors

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Conflict of Interests

All authors declare that they have no conflicts of interest.

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