EFFECT OF ALUMINA TRIHYDRATE WITH ACRYLONITRILE BUTADIENE STYRENE / GRAPHENE NANOPLATELETS AND BASALT FABRIC ON PROPERTY ENHANCEMENT

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During the last several decades, polymeric composites have attracted much attention from researchers and industrialists due to their excellent mechanical and thermal properties. Currently, these materials are gradually replacing metallic parts in bearings, home appliances, and electrical devices. This study investigated the physical and mechanical responses of thermoplastic composite laminates fabricated from acrylonitrile-butadiene-styrene polymer, graphene nanoplatelets, alumina trihydrate powder, and single-ply woven basalt fabric reinforced by a hot-pressing method. The main properties of these laminates were evaluated by applying appropriate testing standards. The obtained results indicated that the prepared thermoplastic composites possessed enhanced mechanical properties as compared with those of the polymer matrix without fillers.

1. Introduction

Prodigious efforts are made to meet societal concern to protect environment, allowing for 'green composites'. Generally, the properties of composite materials are influenced by various parameters like, filler concentration, orientation, aspect ratio, geometrical shape and degree of adhesion. Owing to the combined effect of filler material and matrix, an enhancement in properties of polymer composites is achieved.

Acrylonitrile-butadiene-styrene (ABS) terpolymer is being used in civil, mechanical, automobile, aerospace and structural applications due to its stability, toughness, impact resistant, light weight and smooth finish. The physical and mechanical properties are enhanced by incorporation of optimal ingredients to manufacture polymeric materials. An enhancement in mechanical properties of polymer is achieved by reinforcing fillers into it. A betterment in physical properties of acrylonitrile-butadiene-styrene has been widened its application in polymeric composites. The merits of hybridizing organic and inorganic particles with fibers have been reported by many researchers for decades. Numerous studies have been carried out in strengthening polymer composites by reinforcing carbon fibers, glass fibers, micro fillers, nanofillers, etc. Although synthetic fibers are a desirable reinforcing agent for polymers due to its easy accessibility and smooth nature; they do have their own demerits of greater manufacturing cost and environmental impacts such as discard and renewability.

The rising demand in environmental imbalance and global energy crisis lined an access for the natural fibers as a reinforcing agent for polymers in the current research area. The basalt fiber, one of the natural fiber reinforced polymeric composites have a great impact in research area due to its easy availability, environmental friendly nature, low cost, excellent thermal properties, high specific properties etc. Incorporation of fibers into acrylonitrile- butadiene-styrene matrix restrains the deformation of polymeric matrix. The enhancement in properties is attributed to interfacial interactions between the materials and matrix. Acrylonitrile-butadiene-styrene co-polymer

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improves the performance of natural fibers with appropriate surface coat on fibers [1]. Kenaf fibers bounded with acrylonitrile-butadiene-styrene matrix possess high airflow resistivity and could be used for acoustic purpose [2]. Development of interfacial adhesion increases tensile strength and modulus of composite specimen [3]. Short carbon fibers reinforced with acrylonitrile-butadiene-styrene reduces shrinkage and deformation of specimen whilst porosity increases [4]. Synergistic effect of nano particles with fibers improves the mechanical properties to a great extent. Nano particles disperse well with matrix thus increases the bond between additives like fiber and matrix. Nano sepiolite with glass fibers enhances stiffness of composite with high tensile modulus [5]. Tribological properties of composite laminates are improved by micron sized calcium carbonate (CaCO3) with acrylonitrile- butadiene-styrene.

Melt compounded graphene nanoplatelets with acrylonitrile-butadiene-styrene matrix enhances the tensile modulus of composites [6]. Few layer graphene (FLG) with multiwalled carbon nanotubes (MWCNT) reinforced with acrylonitrile- butadiene-styrene matrix enhances the tensile strength and modulus of composites [7]. Reinforcement of graphene nanoplatelets in acrylonitrile-butadiene-styrene matrix improves the conductivity and elasticity of composite with reduction in impact strength. Multiwalled carbon nanotubes enhances the interfacial bond strength of short polyimide Fibers coated with polydopamine (PDA) reinforced with acrylonitrile-butadiene-styrene [8]. Carbon Fibers when added to acrylonitrile-butadiene-styrene deals with an increment in mechanical properties of composites [9]. Blends of additives with acrylonitrile-butadiene-styrene tend to possess a brittle nature with micro voids [10]. A tremendous enhancement in engineering properties is found with addition of clay with polymer. Research area has a remarkable consequence with clay as additive with polymer. Though there are limitations in the field of engineering properties of clay with polymers, the mechanism of property enhancement is yet in the process of study.

Engineering polymers have been engrossed towards the extensive dispersion of organic clay resulting improved engineering properties. Nevertheless, enhancement in engineering properties of clay reinforced with polymers is in the field of research. Fire retardant property of polymer composite is improved with synergistic effect of nano fillers with acrylonitrile-butadiene-styrene [11]. Clay when dispersed with acrylonitrile-butadiene-styrene/Polyamide 6 significantly improves the flame retardant property [12]. Organic montmorillonite clay into acrylonitrile-butadiene-styrene matrix enhances thermo-mechanical property of composite irrespective of method of manufacture. It also reduces the linear shrinkage of composite [13]. Butadiene rubber in acrylonitrile-butadiene-styrene improves morphological characterization of acrylonitrile-butadiene- styrene foams and considerable reduction in density of mechanical behavior of structure. Fused deposition modelling, one of the most commonly used technique to fabricate composites with deposition of extruded thermoplastic polymer above glass transition temperature in layers provides better results. Acrylonitrile-butadiene-styrene possesses good interlayer bond strength resulting better tensile strength [14].

The previous studies were focused on the properties of glass fiber/acrylonitrile-butadiene-styrene or acrylonitrile-butadiene-styrene/graphene nanocomposites. The hybrid combination of acrylonitrile-butadiene-styrene/basalt fabric/graphene/alumina trihydrate composites have not been investigated yet to the knowledge of the author. However, from the literatures reviewed, it is well known that very limited number of research work has been carried out in the mechanical and physical properties of nano graphene platelets reinforced acrylonitrile-butadiene-styrene/alumina trihydrate powder sandwiched with woven basalt fabric. In this study, the main objective was focused to achieve better mechanical and physical properties of fiber reinforced thermoplastic polymer.

2. Materials processing and sample preparation

2.1. Materials

Acrylonitrile-butadiene-styrene (ABS) terpolymer, Absolac – 920 with 1.04 g/cm^3 as density and 21 g/10 min as melt flow index was used in the present study. Woven type basalt

fabric with surface density of 400 gsm was used to reinforce the composite laminate. The average diameter of fiber in the woven fabric was 20 μm. Graphene nano platelets of 15 microns as diameter and alumina trihydrate (Al_2O_3) of 100 microns were used.

2.2. Sample preparation

A constant quantity of graphene nano platelets (5 wt. %) and varying (5, 10, 15 and 20 wt. %) alumina trihydrate was mixed mechanically with acrylonitrile-butadiene-styrene. The mixture was then hot pressed to prepare samples of 2 ± 0.5 mm thick specimen. The obtained samples were again hot pressed by placing a single ply woven basalt fabric at a temperature of 220 °C under a pressure 1500 psi and a cooling rate of 20 °C/min to 3 ± 0.5 mm thick as adopted test dimensions.

2.3. Tests carried

Mechanical characteristics of thermoplastic composite specimen were studied through various test standards. Tensile properties of test samples were calculated according to ASTM D3039, international standard using Universal Testing Machine. The test was carried out with a load capacity of 50 kN with a cross head speed at 5 mm/min. Average of three test samples were considered for maximum stress. The flexure strength of samples was tested with a cross head speed of 2 mm/min. Flexure samples were prepared for ASTM D790 standard and subjected to three-point bending load. The impact strength of fabricated composite specimens was determined by Charpy impact testing machine according to ASTM D256.

The molecular weight was measured with a high melt flow rate analogous to low molecular weight. Melt flow index (MFI) of materials used was determined conferring to ASTM D1238. Stress relaxation of specimen was studied according to ASTM D2991. Creep behavior of the samples was considered under tensile method by ASTM D2990.

Scanning electron microscope (SEM) analysis was observed to study the failure patterns under tensile load. Compatibility between polymer and fillers was also observed through fractographic analyzed images.

3. Results and discussion

3.1. Tensile strength

The mechanical properties of the prepared laminates such as the ultimate tensile strength and Young's modulus were determined from the force-displacement curves obtained by a universal testing machine.

The ultimate tensile strength of the thermoplastic laminates increased after its reinforcement with woven basalt fabric and nano and micro-fillers, Fig. 1. A linear increase in the tensile strength of the thermoplastic composite laminate was observed. The tensile strength of the sample containing 20 wt.% alumina trihydrate powder is 27% greater than that of the control sample (containing 0 wt.% alumina trihydrate powder), owing to the stronger interfacial interactions between the fibres and the matrix that stiffened polymer chains [15]. In contrast, a 24% drop in the tensile strength with respect to that of the control sample was observed for the specimen containing 5 wt.% alumina trihydrate powder, which might result from the low number of nanoparticles that was insufficient for interlocking polymer molecular chains. Moreover, the low filler content in the acrylonitrile-butadiene-styrene matrix likely increased the interfacial stress concentration. The further addition of alumina trihydrate powder gradually increased the tensile strength of the produced material. Therefore, incorporation of filler stiffened the molecular links of polymer chains, which improved the composite tensile properties. A 17% decrease in tensile strength was also observed after increasing the alumina trihydrate powder content from 15 to 20 wt. %, which was caused by either the presence of excess filler particles in the polymeric matrix or insufficient space for intermolecular interactions.
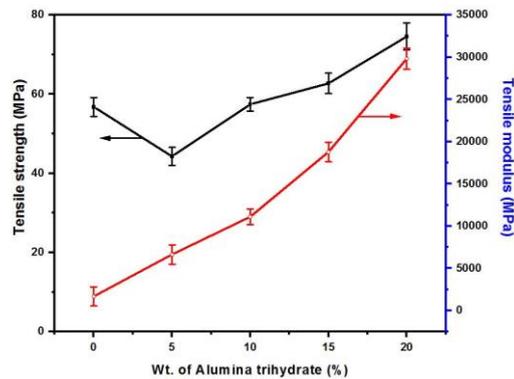
Fig. 1. Tensile strength and modulus of tested specimen

At the same time, the strain at break measured for the control sample decreased after the addition of 5 wt. % alumina trihydrate powder, which resulted from the poor filler adhesion to the acrylonitrile-butadiene-styrene matrix. The latter phenomenon could also be caused by the larger local stress concentrations at the locations of particulate fillers and woven basalt Fibres, orientations of polymer molecules, or high cooling rate of the mould.

Despite the disparity observed for the laminate specimen containing 15 wt. % alumina trihydrate powder, the overall trend appears to be linear. The results of tensile testing indicate that the sample resistance to tensile load was low due to the relatively weak matrix-fibre bonds. Moreover, the addition of basalt fibres to the acrylonitrile-butadiene-styrene matrix containing graphene nano-platelets and alumina trihydrate powder significantly increased specimen's ductility because the adequate interlocking of these fibres and the matrix improved their interfacial bonding.

The observed increase in the tensile strength of the composite laminate represents the enhancement of its stiffness due to the better adhesion between the fibres and the matrix. It is noteworthy that increasing the weight fraction of alumina trihydrate powder in the composite up to 15 wt. % produced a substantial effect on its tensile properties.

The Young's (tensile) modulus of the 20 wt. % alumina trihydrate powder laminate was higher than the values obtained for the other specimens. The agglomeration of particles or their low degree of interaction likely resulted in the discrepancies observed for their tensile strengths and Young's moduli. Overall, the increase in the content of alumina trihydrate powder in the thermoplastic composite matrix increased the elastic modulus of the produced material, Fig. 1.

In general, the strength and ductility of a tested thermoplastic specimen increased with the incorporation of alumina trihydrate powder into the polymer matrix, which also increased its toughness due to the more homogeneous particle distribution.

3.2. Flexure strength

Flexural testing was performed to determine the flexural properties of the studied samples (including their abilities to resist bending forces) according to the ASTM D790 standard. In this study, flexural strengths and flexural moduli were calculated from the corresponding force-displacement curves. It was found that the flexural strengths of the thermoplastic laminates increased with an increase in the concentration of alumina trihydrate powder in the nano-filled polymer matrix. In particular, the flexural strength of the thermoplastic composite laminate increased by 41.83% after increasing the alumina trihydrate powder content from 5 to 10 wt.% Fig. 2.

However, a 23.52% reduction in flexural strength as compared with that of the control specimen was observed for the sample containing 5 wt. % alumina trihydrate powder. Afterwards, the composite flexural strength linearly increased in the concentration range from 5 to 20 wt. % because the considerable increase in the surface area of particulate filler promoted interfacial interactions and enhanced the ability of the tested specimen to resist larger loads. However, the

flexural strain of the sample containing 5 wt. % alumina trihydrate powder was slightly lower than that of the control sample, which likely resulted from the low degree of particulate interfacial interactions.
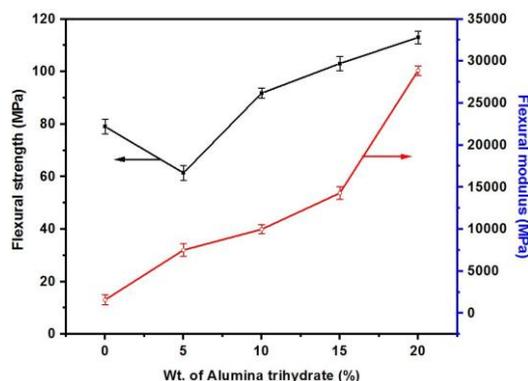
Fig. 2. Flexural strength and modulus of thermoplastic samples

The flexural modulus of the composite laminate also increased with an increase in the content of alumina trihydrate powder in the graphenated acrylonitrile-butadiene-styrene matrix reinforced with basalt fabric, as shown in Fig. 2. Hence, the fibre-matrix interactions along with the addition of alumina trihydrate powder play an important role in the improvement of the composite flexural properties.

No delamination of the fabricated thermoplastic composite laminates was observed for any of the tested specimens, and matrix deformation mostly occurred due to the presence of basalt fabric. Owing to the embedment of fibres with a smooth and clean surface into the polymeric matrix, the flexural properties of the tested specimens were significantly enhanced. The observed linear increases in the flexural characteristics were likely caused by the more uniform distributions of nano- and micro fillers in the acrylonitrile-butadiene-styrene matrix and its stronger interactions with the fabric [19]. The anticipated stress concentration zones at the fibre-matrix interface exhibited strong interlocking properties, and no buckling was detected for the tested laminate specimens. High stiffness and small specimen elongation were achieved by restricting the molecular movement in the acrylonitrile-butadiene-styrene matrix. Overall, the strong adhesion between the smooth matrix surface and woven basalt fabric significantly increased their bonding strength.

3.3. Impact strength

The results of Charpy impact tests revealed that the incorporation of 20 wt. % alumina trihydrate powder into the thermoplastic polymer matrix increased its impact strength (19.56 J) from that of the control sample (9.78 J), as shown in Fig. 3. Thus, the addition of nano and micro fillers into acrylonitrile-butadiene-styrene gradually increased the impact strength of tested specimen, which might be attributed to the good interlocking properties of the high molecular weight chains of fillers toward the acrylonitrile-butadiene-styrene matrix reinforced with basalt fabric.
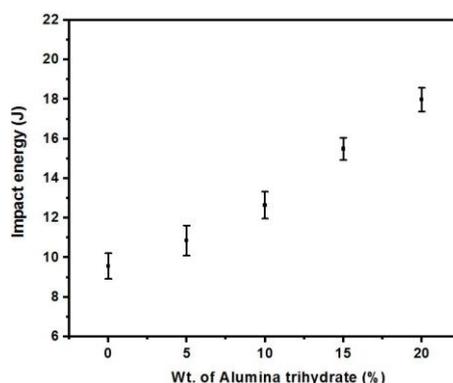
Fig. 3. Charpy impact energy of specimen

The complete dispersion of nano- and micro-fillers across the polymer matrix likely enhanced its impact characteristics and immobilised the molecular chains [16]. The incorporation of basalt fabric also produced a positive effect on the impact strength of the composite specimen. Thus, the addition of 5 wt% graphene platelets to the acrylonitrile-butadiene-styrene matrix resulted in impact strength of 9.78 J, which was further increased by the addition of alumina trihydrate powder.

3.4. Melt flow index

The molecular weight of the composite structure has a strong effect on the heat resistance of the acrylonitrile-butadiene-styrene matrix. The melt flow index of this matrix (characterising its flow ability) filled with 5 wt.% graphene nano-platelets and various concentrations of alumina trihydrate powder (5, 10, 15, and 20 wt.%) increased with an increase in the molecular weight of the continuous phase at a temperature of 280 °C, thus enhancing the material tolerance to heat, Fig. 4.
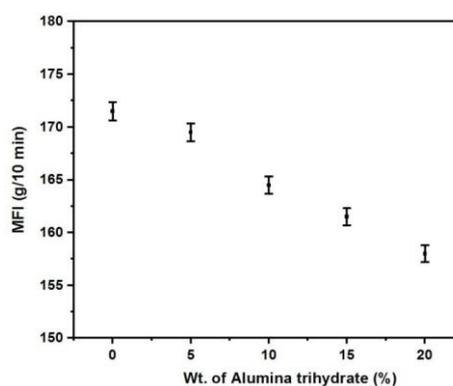
Fig. 4. Melt flow index of various wt. % alumina trihydrate powder

In other words, the combination of graphene nano-platelets (representing short stacks of graphene sheets) with alumina trihydrate powder increased the thermal resistance of the acrylonitrile-butadiene-styrene system. Generally, the higher is the internal frictional resistance of polymer molecular chains, the greater is the melt flow rate. The slower motion of polymer chains increases the heat resistance and distortion temperature of the polymer. Hence, the melt flow index of a particulate-filled polymer matrix decreases with increasing filler content. Activation energy (E_{act}) for mobility of polymer chain links was evaluated from Arrhenius equation expressed as in equation (1).

$$Log\ (\text{MFI}) = Log\ (C_o) - \frac{E_{act}}{2.30\ R} \times \frac{1}{T} \tag{1}$$

Where,

MFI - Melt Flow Index

C_o - pre-exponential factor

E_{act} - activation energy

R - Universal gas constant (8.314 J/mol.K)

T - Chosen temperature for Melt Flow Index test

The measured activation energy of the control sample was 93 kJ/mol, and that of the sample containing 20 wt. % alumina trihydrate powder was 119 kJ/mol. Therefore, the activation energy positively correlated with the interaction strength between the acrylonitrile-butadiene-styrene matrix and fillers in the molten state.

3.5. Stress relaxation modulus

Stress relaxation testing was performed according to the ASTM D2991 standard. Samples were conditioned at 23±2 and 50±2 °C for 48 h prior to the testing procedure. Time dependence of the specimen tensile relaxation was investigated at strain rates of 0.25, 0.5, 0.75, 1, 2, 3, 4, and 5%. Note that the slow molecular relaxation of copolymers affects the long-term stress relaxation of the material, while the interactions of polymer chains change above the glass transition temperature (Tg) [17]. The duration of the experiment was varied between 10 and 1800 s after the crosshead activation. The majority of polymeric materials produce linear stress-strain curves at strains below 0.6 [18]. In addition, the nominal stress of a material plotted against the logarithm of time is also represented by a straight line. A strain reduced modulus was developed by Landel and Stedney expressed as in equation (2).

$$E_r \times (t) = E_r\ (t)\ f(\alpha) \tag{2}$$

Where,

$f(\alpha)$ - function of extension ratio α

E_r - Relaxation modulus

t - Time

In this study, strain was applied to a tested specimen at a constant rate for a desired elongation. It was assumed that $f(\alpha)$ was independent of time and temperature, which were equal to approximately 100 min and 39 °C, respectively. No notable changes in the relaxation parameters were observed at strains (yields) below 3%. The obtained stress relaxation data were explained in the framework of the Maxwell model developed for completely viscous and elastic materials. According to Maxwell, stress relaxation modulus is expressed as in equation (3).

$$E_r(\text{t}) = \frac{\sigma(t)}{\varepsilon} \tag{3}$$

Where,

$E_r(\text{t})$ - Relaxation modulus

σ (t) - Stress with respect to time

ε - Strain rate

Relaxation time characterises the stress relaxation behaviour of a viscoelastic material and is defined as the time required for the completion of the stress relaxation process. In this work, the sample containing 20 wt. % alumina trihydrate powder exhibits a larger drop in stress (0.25%) while the least drop in stress (5%) in 30 min, Fig. 5. Furthermore, a greater decrease in stress was

observed at a higher applied force. These results indicate that the stress relaxation time of basalt fibres was relatively small.
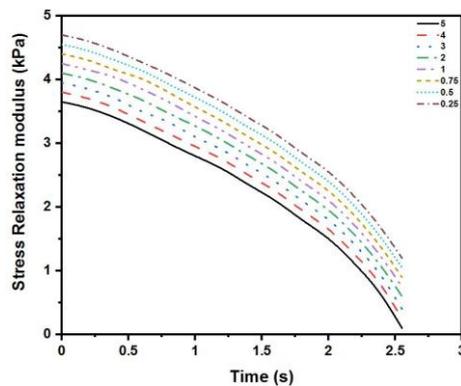
Fig. 5. Stress relaxation of tested specimen

In summary, the time dependence of the relaxation modulus of a tested specimen should be taken into account when considering its practical applications related to viscoelastic properties.

3.6. Creep

Creep deformations of the prepared composites were examined at varying stresses starting from 4.7 MPa (which corresponded to approximately 10% yield strength of neat acrylonitrile-butadiene-styrene) and temperature of 30 °C for up to 3600 s. It is well known that a time-dependent creep strain curve consists of a transient creep region, a steady-state region, and an accelerating creep region [19]. As expected, the creep stability of the composite laminate increased with pressure, Fig. 6.
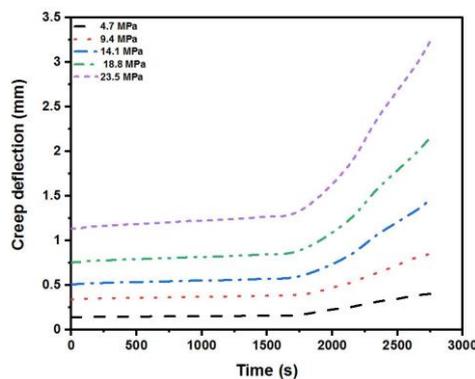
Fig. 6. Creep deflection of specimen.

Polymers behave like common elastic materials below Tg. The bond angles between different polymer chain segments affect the parameters of elastic deformation. When the strain is constant, the stress induced in the system relaxes [20]. Conversely, at a constant stress, the retardation of strain relaxation occurs, and the material starts to creep. In this work, the creep behaviour of the composite material was evaluated at a constant temperature while varying the pressure and frequency. The obtained creep rate increased with increasing pressure.

3.7. Fractographic analysis

The micromorphological characteristics of the failure patterns obtained for the fabricated composite specimens under tensile loads were studied by scanning electron microscopy. The coagulation of acrylonitrile-butadiene-styrene polymer species promoted the dispersion of

graphene platelet nanoparticles and alumina trihydrate powder and toughening of craters and holes in the composite structure [21]. The relatively smooth polymer matrix ensured its strong interfacial interactions with the added basalt fibres, Fig. 7.
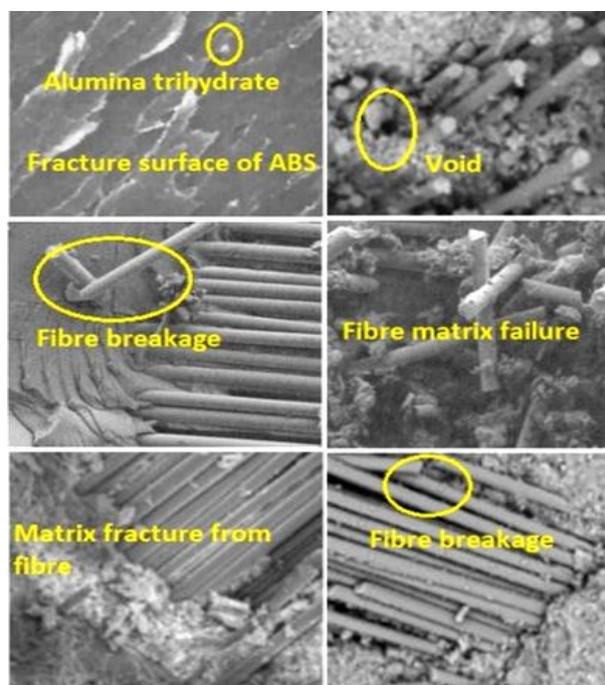
Fig. 7. Scanning electron microscope images of fractured tensile specimen.

Meanwhile, the composite specimen containing 20 wt. % alumina trihydrate powders exhibited high tensile strength. Despite the strong interfacial interaction, the density of the matrix near the fibre surface decreased as compared with that of the bulk polymer, owing to the applied tensile load. As a result, fibre breakage occurred.

4. Conclusions

In this work, the physical and mechanical properties of the acrylonitrile-butadiene-styrene polymer reinforced with graphene platelets, alumina trihydrate powder, and woven basalt fabric were investigated. The studied parameters of the produced composites included tensile strength, flexural strength, impact strength, shrinkage, melt flow index, stress relaxation modulus, and creep rate.

The ultimate tensile strength of the composite specimen containing 20 wt. % alumina trihydrate powder samples was 27% higher than that of the control sample. The reinforcement of the thermoplastic composite matrix with woven basalt fabric converted it from a brittle material to a ductile one. Moreover, the stacking arrangement of graphene nanoplatelets and alumina trihydrate powder increased the stiffness of the basalt fabric-reinforced composite laminate.

The flexural strength of the specimen containing 20 wt. % alumina trihydrate powder exceeded the value obtained for the control sample by 35%, owing to the strong adhesion of basalt fabric to the thermoplastic composite matrix. No composite delamination was observed for the tested specimens due to the high bonding strength between these components.

The impact strength of the composite specimen increased by 67% after the addition of 20 wt. % alumina trihydrate powder because of the stronger interaction of the matrix with basalt fabric, which increased the material toughness.

The flowability of the thermoplastic composite matrix was significantly improved by the addition of alumina trihydrate powder. Its melt flow index decreased with increasing powder content.

The thermoplastic composite laminates reinforced with basalt fabric exhibited excellent stress relaxation properties at a constant strain rate. A relatively large drop in stress was observed at a force value higher than the applied force for the time-dependent tensile relaxation.

The creep stabilities of the composite specimens were examined at high applied pressures. The creep rates of the thermoplastic composite laminates measured at low stress levels remained stable due to the high resistance of basalt fabric to creep rupture.

Very good compatibility between the polymer matrix and basalt fabric was confirmed by the results of fractographic analysis. The strong adhesion of the fabric to the laminate prevented composite delamination. However, the matrix near the fibres softened leading to fibre pull-out during tensile testing.

The results of this work indicate that the fabricated composite laminates can be potentially used in manufacturing automobile parts (including brakes) as well as in aerospace, civil engineering, architectural, and marine applications due to the unique combination of their physical and mechanical characteristics.

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