Reinforcing effect of polyurethane sizing on properties of acrylonitrile–butadiene–styrene composites involving short carbon fiber

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Abstract

In this study, we present the influence of sizing layer of short carbon fiber (CF) to the basic properties of CF reinforced acrylonitrile–butadiene–styrene (ABS) composites. Composite samples are prepared with four different loading ratio of 5, 10, 15 and 20% by weight using melt-compounding. Surface topography, elemental analysis and surface functionality of CF samples are confirmed by atomic force microscopy (AFM), energy-dispersive X-ray (EDX) and infrared (FTIR) spectroscopy techniques, respectively. Characterizations of composites are performed based on mechanical, thermo-mechanical, melt-flow (MFI) and morphological performances of composites. According to test results, mechanical properties of ABS are enhanced with CF additions. Polyurethane (PU) sized CF containing composites display higher tensile strength, modulus and hardness compared to desized ones. CF additions lead to increase in glass transition temperature of ABS copolymer. MFI values of composites are found to be in narrow range, hence CF additions cause no effect for processing conditions of ABS. According to SEM analysis, CF surfaces are covered by polymer matrix thanks to PU sizing layer of CF, whereas debondings are formed for desized CF-filled ABS matrix. Results indicate that PU-sizing is suitable for ABS/CF composite system.

Graphical Abstract

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1 Introduction

Carbon fiber reinforced polymer composites have great attention and have found various use in many application areas including transportation, construction, electronics, aerospace and defense industry. The main goal of its usage is the reduction of weight by replacing metal parts with carbon fiber containing polymeric structures [1, 2]. Research efforts and application fields based on carbon fiber and its composites are continued to grow steadily and they are considered to advanced materials in many topics [3–5].

Acrylonitrile–butadiene–styrene (ABS) terpolymer is a trendy candidate among all of the polymers thanks to expansion in its commercial use for additive manufacturing field recently. The structure of ABS copolymer is composing of polybutadiene groups dispersed in styrene and acrylonitrile parts. Polybutadiene is the elastomeric unit whereas styrene and acrylonitrile show thermoplastic behavior. These phases are responsible for mainly the toughness, dimensional stability and chemical resistance properties of ABS copolymer [6–8]. Research studies have been conducted to optimize and tune of basic properties of ABS copolymer in order to extent its effective use in numerous applications in a wide array of industrial fields including mostly additive manufacturing and automotive [9–12].

There have been several research studies that conducted on short CF containing thermoplastic composites in the literature. Anguita et al. developed dimensionally ultra-stable CF-reinforced polymeric structures for their specific applications for satellites in a recent study. They stated that near-zero in thermal expansion coefficient was reached for dimensionally stable CF-filled composite structures [13]. They observed that reinforcing capability of CF was mainly depending on adhesion between the fibers and polymer matrix. In another study, Dike produced short CF filled polyamide (PA) composites with different surface coated CF samples. He reported that composites involving isocyanate coated CF displayed the highest enhancement based on the mechanical properties of PA-based composites [14]. According to study reported by Aparna et al., tensile strength of polypropylene (PP)-PA blend exhibited remarkable increase after short CF inclusion [15]. Duchoslav et al. performed surface sizings for CF samples. They found that deterioration occur in sizing layer of CF surface during processing stage of composite manufacturing [16]. Rezaei et al. investigated that short CF addition caused improvement on strength and stiffness of

PP/CF composites [17]. In a recent study, Park et al. demonstrated that thermal stability and tensile behavior of PP/CF composite system were enhanced by applying irradiation [18]. Savas et al. studied the effect of CF addition to PP-based composites [19]. They observed that elastic modulus of PP was extended with the increase in CF content. Nie and his coworkers fabricated CF reinforced poly(vinylidene fluoride) (PVDF) composites in order to examine their wear resistance [20]. According to their findings, PVDF exhibited the highest tribological performance with 20% CF inclusion. Pinpathomrat et al. performed the production of CF-filled PA composites by fused deposition modeling and they found that CF donated UV resistance for PA matrix [21]. Jiang et al. performed CF containing polyurethane (PU)-based composites. They proved that surface coating of CF caused remarkable increase on tensile strength of composites [22].

Short CF reinforced ABS-based composites were also studied in the literature. Investigations of these composites mostly dealt with the mechanical performance. Love et al. fabricated short CF loaded ABS composites and they displayed that tensile strength and modulus of ABS raised with the addition of CF [23, 24]. Yang et al. stated that 10% filling ratio of CF caused remarkable increases for flexural and tensile strength values of ABS [25]. Huang et al. investigated that CF addition to ABS results in improvement for strength and modulus values of neat ABS [26]. Hull et al. showed that processing conditions of ABS filaments are significantly affected by CF content in the case of 3D printing applications [27]. Quan et al. demonstrated that short CF loaded ABS filaments can be optimized for the manufacturing of composite parts using fused deposition modelling [28]. According to research works regarding polycarbonate-ABS blends conducted by Lee et al. and Baek et al., CF incorporations led to increase in tensile strength and elastic modulus values of blends on the contrary reduction in impact resistance [29]. Ning et al. fabricated CF reinforced ABS composites using additive manufacturing process. They postulated that porosity of 3D printed parts yields reductions on mechanical properties of composites [30].

The objective of this study is to investigate the effect of surface sizing and amount of CF to properties of ABS-based composites. The presented results reflect the potential use of ABS in wide range of areas including additive manufacturing, transportation, construction and aerospace applications. In the light of related research highlights, the role of polyurethane layer on CF to the properties of ABS/CF composite system was

Table 1 Process parameters of composites

Parameter	Specification	Unit
Processing temperature	230	°C
Mixing time	5	min
Screw speed	100	rpm
Barrel temperature	240	°C
Mold temperature	50	°C
Injection pressure	8	bar
Holding time	3	min

postulated first time with this study. For this purpose, desized and PU-sized chopped CFs were compounded with ABS copolymer using melt-blending process. Melt-mixing technique was used for preparation of composites since this processing method is widely applied in industrial fields. This method is also providing similar processing methodology with additive manufacturing technology. Surface covering of CF samples was visualized using atomic force microscopy (AFM), energy-dispersive X-ray spectroscopy (EDX) and infrared spectroscopy (FTIR) test techniques. Characterizations of composite samples were performed based on the effect of sizing layer on fiber and concentration of CF. Basic properties of composites including mechanical, thermo-mechanical, melt flow and morphological behaviors were reported in order to provide information for material researchers to produce ABS-based composites with required parameters. The article is organized by the explanation of materials, composite fabrication and characterization methods followed by the brief discussion based on the test data and conclusion remarks are indicated according to findings in the final section.

2 Experiment set-up

2.1 Materials

In this study, ABS was used as polymer matrix and short CF samples were used as reinforcing agent. ABS copolymer was purchased as chip form by Lanxess, Cologne, Germany. It has the trade name of Lustran ABS M203FC. CF samples were supplied from Dowaksa, Yalova, Turkey under the commercial name of Aksaca AC 0101. It has chopped form with 3 cm length. This CF grade has density of 1.76 kJ/m^2 , diameter of $7 \mu\text{m}$ and surface covering of polyurethane with the sizing ratio of 1.5–2.0%. Mechanical properties of CF sample are cited by producer as tensile strength of 4.2 GPa, percent elongation of 1.8% and modulus of 240 GPa. Commercially sized CF was annealed at 450°C for 4 h that

it would be possible to the removal of sizing layer and this desized CF sample was named as DS-CF.

2.2 Composite preparation

ABS copolymer and CF samples were dried at 100°C for a period of 2 h to avoid possible moisture before compounding. Composites were fabricated by laboratory scale twin screw extruder (MC 15 HT, Xplore Instruments BV, Netherlands). CF samples were incorporated at four different compositions of 5, 10, 15 and 20% by weight in ABS matrix. ABS and composite samples obtained from extrusion process were shaped using laboratory scale injection molding instrument (Micro-injector, Daga Instruments, USA). Test specimens were shaped as standard dog-bone sample during injection molding process in accordance with ASTM D-638 standard. Test samples have dimensions of $7.6 \times 2.0 \times 80 \text{ mm}^3$. Gauge length of dog-bone shaped test specimen is 50 mm. Processing parameters applied during production of composites are listed in Table 1.

2.3 Characterization techniques

Fourier transformed infrared spectroscopy (FTIR) analysis was applied for short CF samples in attenuated total reflectance (ATR) mode using IR-spectrometer of Bruker VERTEX 70 (Massachusetts, USA). The analysis was

Fig. 1 FTIR spectra of CF samples**Table 2** EDX data of CF samples

Samples	C Wt%/At%	O Wt%/At%	N Wt%/At%
DS-CF	96.93/97.68	3.07/2.32	0 /0
CF	92.59/94.33	7.41/5.67	2.53/2.22

Fig. 2 SEM and AFM images and EDX data of CF samples

carried out in the wavenumber range between 600 and 3800 cm^{-1} at a resolution of 2 cm^{-1} with 32 scans. JSM-6400 (JEOL Ltd, Tokyo, Japan) scanning electron microscope (SEM) was utilized to visualize the morphological properties of composites. The surfaces of fractured test samples obtained from the impact test were coated with the thin layer of gold for the aim of creating conductive

surfaces. Chemical compositions of CF surfaces were evaluated using energy dispersive X-ray spectroscopy (EDX) mode of SEM equipment. Topographical characterization of CF surface was examined by Nanosurf AFM device (Liestal, Switzerland). Tensile test measurements of ABS and composites conducted using Instron 5565A (Buckinghamshire, England) tensile testing machine with the accordance of ASTM D-638 standard. Crosshead speed of 5 mm/min and 5 kN load cell were applied.

Fig. 3 SEM micrographs of composites

Tensile strength, elongation at break and tensile modulus parameters were recorded as an average of minimum five samples. The un-notched Izot impact test was carried out with Coesfeld Material impact tester (Dortmund, Germany). Impact energy measurements were done according to ASTM D256 standard using a 4 J pendulum. Recorded values represent an average value of at least five samples with standard deviations. DMA studies were performed with DMA 8000 (Perkin Elmer, Massachusetts, USA) dynamic mechanical thermal analyser. Test specimens obtained from injection molding with dimensions of $7.6 \times 2.0 \times 50 \text{ mm}^3$ were subjected to DMA

Fig. 4 The variations of tensile parameters with respect to CF amount

test in dual cantilever bending mode at the temperature range from -120 to 140°C with 1 Hz constant frequency and heating rate of $5^\circ\text{C}/\text{min}$. Melt-flow index (MFI) measurements were conducted using melt flow indexer (Coesfield Meltfixer LT, Dortmund, Germany) equipment. Tests were done under a standard load of 2.16 kg at the processing temperature of ABS and composites (230°C). MFI values of each sample were calculated as an average value of at least eight samples with standard deviations.

3 Results and discussion

3.1 Surface characteristics of CF samples

Figure 1 represents the IR spectra of CF and DS-CF samples. Absorption peaks around 3000 cm^{-1} are attributed to C–H vibrations of hydrocarbon groups. The characteristic peaks of hydroxyl group can be seen at 3600 and 800 cm^{-1} which are stem from the vibrations of O–H bending and O–H stretching, respectively [31, 32]. These oxygen-related peaks are present for sized CF sample whereas they cannot be seen for DS-CF sample. The two peaks located near to 1000 cm^{-1} are stem from the C–O vibrations. Additionally, absorption bands can be seen at nearly 1300 and 1700 cm^{-1} which correspond to C–N and C=O stretching vibrations [33, 34]. These characteristic peaks are owing to nitrile and carboxyl groups of isocyanate unit regarding PU cover on CF surface which are absent for DS-CF sample. The difference between the surfaces of two CF samples are confirmed by these findings.

Elemental compositions of CF surface supplied by EDX analysis data are listed in Table 2 and EDX spectra are shown in Fig. 2. The oxygen content of PU sized CF was found to be higher than that of DS-CF by the factor of 2.4. This sample also gained remarkable nitrogen composition thanks to the sizing layer containing urethane segment. Narrow grooves can be seen on the surface of desized CF sample. The smooth surface of desized CF and relatively rougher surface of CF can be clearly observed from SEM micrographs and AFM images of CF samples in Fig. 2.

3.2 Morphological study

SEM micrographs of composites are displayed in Fig. 3. It can be confirmed by SEM micrographs that sized CFs dispersed uniformly into ABS phase. This may due

to the improvement of compatibility of CF with ABS matrix by the help of PU-sized surface of CF. Sized CF surfaces were covered by polymer phase and almost no debonding formation was observed for CF filled ABS composites. On the other hand, large gaps were formed between DS-CF and ABS matrix which may stem from the lack of adhesion regarding the inertness DS-CF surface to polar ABS matrix. In the case of CF amount, it can be observed that fibers were oriented well in one direction for 5% and 10% CF loading levels thanks to injection molding process. Further addition of CF caused some disorientations of fibers into polymer matrix. These findings from the SEM analysis provide strong evidence for the effect of sizing layer of CF to ABS matrix.

3.3 Mechanical performance

The variations of tensile parameters including tensile strength, percentage strain and tensile modulus with respect to CF amount are displayed in Fig. 4. Tensile strength and tensile modulus of unfilled ABS were improved gradually with increase in CF concentration. Composites containing PU-sized CF exhibited approximately 10% higher tensile strength values compared to that of desized CF. Similarly, ABS/CF composites gave higher tensile modulus than ABS/DS-CF for all of the filling ratios. These results would mean that polyurethane cover at CF surface caused enhancement on adhesion between fiber and ABS phases by reason of interfacial interaction. R^2 values located in tensile strength and tensile modulus graphs were found to be linear (nearly 1) which indicates that increase in these parameters are proportional with the concentration of CF. This also implies that strength and modulus values are predominantly depend on fiber concentration as claimed by Kelly and Tyson equation [6, 35]. Fiber shortening might be carried out with negligible amount during processing thus, it caused no influence for tensile parameters of composites. On the other hand, percent elongation of neat ABS showed sharp reduction after CF inclusions regardless of sizing. This awaited finding is related with the fact that high aspect ratio fiber additions cause drops down in strain of polymeric materials.

Impact strength values are represented as the function of CF content in the Fig. 5. This figure implies that impact strength of neat ABS extended up to higher levels with increase in CF content. DS-CF loaded composites displayed nearly 10% lower impact strength values with respect to ABS/CF. This may due to better adhesion of sized CF with ABS matrix relative to DS-CF. This behavior can be explained by the formation of stress failures of CF surface with its surrounding ABS matrix. Surface sizing of CF promotes to resistance against deformation thanks to

Fig. 5 Impact test data of composites

Table 3 Shore D hardness parameters of composites

CF Content (%)	ABS/DS-CF	ABS/CF
0	77.4 ± 0.1	77.4 ± 0.1
5	78.1 ± 0.1	78.8 ± 0.1
10	79.0 ± 0.1	80.2 ± 0.2
15	80.1 ± 0.2	81.4 ± 0.1
20	81.3 ± 0.1	82.7 ± 0.1

improvement for interfacial adhesion in CF/ABS composite system [36–38].

Shore D hardness values of ABS and its composites are listed in Table 3. CF inclusions raised Shore hardness of unfilled ABS to higher values. Hardness of composites increased steadily with the loading level of CF. Composites involving sized CF displayed higher values compared to desized ones for all concentrations due to have higher load bearing capacity.

3.4 Thermo-mechanical response

Storage modulus, loss modulus and $\tan \delta$ curves as a function of temperature can be seen in Fig. 6. The peak values of loss modulus and $\tan \delta$ curves correspond to glass transition temperature (T_g) of polymeric material. The T_g values indicated in Fig. 6 signify to T_g of styrene-acrylonitrile (SAN) portion in ABS structure [39]. Composite samples containing DS-CF yielded higher storage modulus values compared to sized CF-filled composites. Relatively higher stiffness of DS-CF with respect to CF may be the reason of this finding since DS-CF inclusions led to compensation of polymer matrix [20]. The highest loss modulus was observed for 15% CF containing composite. ABS/20 CF composite gave nearly identical loss modulus with ABS/15 CF composite. It is clearly seen from the curves that sized CF additions led to remarkable increase in loss modulus values and T_g more than DS-CF samples since strong adhesion between two phases promotes restriction of segmental mobility of polymer chains [40, 41].

$\tan \delta$ curves in Fig. 6 implies that all of the CF additions caused shifting for T_g of neat ABS to the higher temperatures. Improvement of nearly 6 units on T_g value of ABS was observed for the highest amount of CF (20%). DS-CF containing composites exhibited almost identical T_g values each other. The shifting of T_g values are mainly related with the vibration-damping property of the composites as they subjected to constant deformation during the DMA measurements [42, 43].

Fig. 6 DMA curves with respect to temperature

3.5 MFI measurements

MFI test provides information regarding viscosity and processability of polymers. MFI values of unfilled ABS and composites are given in Fig. 7. As shown in Fig. 7, DS-CF containing composites exhibited slightly lower MFI values with respect to sized CF containing ones. CF inclusions at lower

Fig. 7 MFI values of ABS and composites

loading ratios caused slight decrease in MFI parameters of ABS copolymer. Melt flow behavior of polymer phase was extended with higher CF loadings since high aspect ratio fibers well-dispersed in ABS matrix. Composites displayed narrow MFI range compared to unfilled ABS. This result indicates that production of CF reinforced ABS composites can be operated without obvious processing problems in the case of additive manufacturing applications [44, 45].

4 Conclusion

In this current study, desized and PU-coated short CF were compounded with ABS matrix. Conventional production techniques of polymeric composite materials, melt compounding and injection molding process, were performed due to their cost-effective and practical characteristics. Mechanical, thermo-mechanical, melt-flow and morphological properties of produced composites were postulated. The presence of polyurethane layer was confirmed by surface analysis of CF samples. According to tensile test results, tensile strength and tensile modulus of neat ABS increased with the incorporation of the CF at higher filling ratios in which 10% and 15% concentrations displayed higher values. On the other hand, percent elongation of ABS reduced drastically with CF loadings. Impact test revealed that CF additions yielded remarkable improvement for impact strength values of ABS. Sized CF filled composites exhibited relatively higher impact strength than desized CF. Shore hardness of ABS enhanced by incorporation of CF regardless of sizing. DMA analysis showed that loss modulus and glass transition temperature of neat ABS were extended with the addition of CF at higher amounts. Loading levels of 15% and 20% CF gave higher storage modulus values among all of the composites. Sized CF inclusions resulted in higher storage modulus and glass transition temperature values with respect to desized ones. CF additions caused no meaningful difference in MFI value of ABS which means that compounding CF with ABS can be taken place with no obvious problems in the case

of processing conditions of composites. SEM analysis visualized that sized CF surfaces were covered suitably by polymer matrix which arises from the compatible interfaces between CF and ABS copolymer. Debonding formations were clearly observed between DS-CF and polymer phase due to the lack of adhesion. As an overall conclusion, sized CF samples display better results with respect to desized CF samples. PU-sized CF reinforced composites showed remarkably higher mechanical and thermo-mechanical properties compared to desized CF filled composites. These finding indicated that PU sizing is suitable for ABS/CF composite system in the future applications of additive manufacturing.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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