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POLYURETHANE ELASTOMER AS A MATRIX MATERIAL FOR SHORT CARBON FIBER REINFORCED THERMOPLASTIC COMPOSITES

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ABSTRACT

Short carbon fibers (CF) with different surface sized (epoxy (EP) and polyurethane (PU)) were used as reinforcing agent in thermoplastic polyurethane (TPU) based composites. Composites containing 5, 10, 15, and 20 weight % sized and desized CFs were prepared using melt-mixing method. The surface characteristics of CFs were examined by energy dispersive X-ray spectroscopy (EDX) and Fourier transform infrared spectroscopy (FTIR). Tensile testing, shore hardness test, dynamic mechanical analysis (DMA) and melt flow index (MFI) test were performed for determining final composite properties. The dispersion of CFs in TPU matrix was examined by scanning electron microscopy (SEM). Tensile strength, Youngs' modulus and Shore hardness of TPU were enhanced by the addition of sized CFs. About two-fold improvement for tensile strength and ten-fold improvement for Youngs' modulus were observed with the incorporation of 20 wt% EP-CF and PU-CF in TPU. The storage modulus of PU-CF containing composites was higher than those of TPU and other composites. No remarkable change was observed in MFI value of TPU after CF loadings. Processing conditions applied in this work was suitable for composite production. Sized CFs exhibited better dispersion with regard to desized CF due to the stronger adhesion of TPU matrix to fiber surface.

Keywords: Short carbon fiber, Mechanical properties, Thermoplastic polyurethane, Carbon fiber reinforced composites, Thermoplastics

1. INTRODUCTION

Carbon fibers (CF) have been used for advanced technologies thanks to their extraordinary properties such as low density, high tensile strength and Youngs' modulus. Applications of CF are dominantly compose of two main sectors including aerospace and transportation engineering [1]. The interest for carbon fiber reinforced thermoplastic composites has been increased in recent years. Recyclability of thermoplastics and opportunity to use in conventional techniques (extrusion, injection and compression molding) during their processing are the main advantages of thermoplastics over thermosets [2,3]. The growth rate of global thermoplastic composites market is expected to level up in next five years according to market research report [4]. Especially, the design and fabrication of ultra-low weight and mechanically strong automobile parts is becoming popular nowadays [5-9].

Thermoplastic polyurethane (TPU), a block copolymer, contains alternating hard and soft segments in its structure. Hard segment, which is composed of highly polarized groups such as low molecular weight glycols or diamine reacted with diisocyanate, provides strength, [10]. The soft segment is composed of polyester or polyether units. As a result of phase separation, hard segments are dispersed as microdomains in the structure and are hold together by interchain hydrogen bonding [11].

Polyurethane elastomers, which have desirable properties such as excellent mechanical strength, chemical resistance and easy processability, are used in many applications. Beside these specialties, TPU is a fully recyclable polymer that makes it cost effective. Major TPU applications are across a range of markets including footwear, automotive, sporting goods, medical devices, tubes, hoses, wires,

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cables and medical devices in the form of sheets, films, or profiles. TPUs have found effective use in the manufacturing of several automobile parts with their various forms [12-14]. TPUs can also be used in design and manufacture of various materials having tribological performance [15], shape memory actuation [16, 17], electromagnetic shielding [18-20] and seismic isolation [21,22] properties as they reinforced with CFs.

Short CF reinforced TPU composites were studied in very limited number of works a decade ago [23-25]. However, extrusion process was not preferred method for the production of these composites. The role of sizing layer of CF surface and the compatibility of CF with polymer matrix have not been well studied yet. In the current study, chopped CFs were incorporated in TPU by using melt mixing method. Two types of commercially sized CFs were used in four concentrations. The surface properties of CF samples were characterized by using FTIR and EDX spectroscopy techniques. The influence of loading level and the sizing types of CF samples on to mechanical and thermo-mechanical properties of TPU based composites were reported. Melt flow characteristics of TPU and relevant composites were also examined. Dispersion and alignment of CFs in polymer melt can be deduced from melt-flow index (MFI) measurements of composites. These observations are important for structural design and manufacture of required materials in order to control and establish of processing conditions [26,27]. The morphological characterizations of composites were also investigated by the help of SEM micrographs. The comparisons of sized CFs with desized CF varying concentrations were discussed.

2. EXPERIMENTAL

2.1. Materials

The commercial polyester-based thermoplastic polyurethane was purchased from Pasific (Covina, USA) under the trade name of Texalan® 485A. It has a density and hardness of 1.20 g/cm^3 and 85 (Shore A), respectively. Chopped carbon fibers (3 mm), which were commercially epoxy (AC 1101) and polyurethane (AC0101) sized, were obtained from Dowaksa (Yalova, Turkey). They both have density of 1.76 kJ/m^2 determined according to ISO 10119 and their mechanical properties determined according to ISO 10618 as tensile strength of 4200 MPa, modulus of 240 GPa and elongation of 1.8 % cited by producer. Commercially polyurethane sized CF was annealed at 450 ˚C for 4 hours for the removal of sizing layer. Samples were subjected to dichloro ethane to further the cleaning of CF surfaces. Desized CF samples were dried at 100°C overnight for solvent removal and coded as DS-CF. Epoxy and polyurethane sized CFs were coded as EP-CF and PU-CF, respectively. Sized CFs were used as received.

Reagent grade dichloro ethane ($C_2H_2Cl_2$) was supplied by Sigma Aldrich.

2.2. Preparation of Composites

TPU was dried at 100˚C for 2 hours prior to compounding. TPU based composites were prepared with the melt mixing in counter rotating twin screw microextruder (15 ml microcompounder, DSM Xplore, Netherlands) at a screw speed of 100 rpm at 210˚C for 8 minutes. CF was incorporated at four different compositions of 5, 10, 15 and 20 weight % in TPU matrix. Test samples were prepared by injection molding instrument (Microinjector, Daca Instruments) at a barrel and mold temperature of 215˚C and 40˚C, respectively.

2.3. ATR-FTIR Analysis

FTIR measurements in attenuated total reflectance (ATR) mode were performed by using IR-spectrometer (Bruker VERTEX 70) at a resolution of 2 cm^{-1} with 32 scans from 600 to 3800 cm^{-1} wavenumbers.

2.4. Scanning Electron Microscopy (SEM)/Energy Dispersive X-ray Spectroscopy (EDX)

The surfaces of CFs and cyro-fractured of composites were examined by Field Emission Scanning Electron Microscope (FEI Quanta 400F). The SEM photographs were taken at different magnifications varied from x500 to x10,000. Elemental analyses were studied at the indicated spots in SEM micrographs of CF sample surfaces by using EDX technique.

2.5. Tensile Test

The measurements of the tensile properties were carried out by using Lloyd LR 30 K universal tensile testing machine with load cell of 5 kN at crosshead speed of 5 cm/min. Tension tests were conducted on dog-bone shaped samples according to the standard of ASTM D-638. Tensile strength, percentage elongation at break and tensile modulus values were recorded. All the results represent an average value of five samples with standard deviations.

2.6. Hardness Test

Shore A hardness values of composites were determined by electrical Shore hardness tester (Zwick digital hardness tester) according to ISO 7619-1 standard.

2.7. Dynamic Mechanical Analysis (DMA)

Dynamic mechanical analyses were carried out using Perkin Elmer DMA 8000 in dual cantilever bending mode at a frequency of 1 Hz. The test was carried out in the temperature sweep mode from -60 to 150 ^{o}C at a heating rate of 5 ^{o}C/min.

2.8. Melt Flow Index Test (MFI)

Melt flow property measurements were studied by Coesfield Material Test, Meltfixer LT. The test was carried out at process temperature (200˚C) under specified load of 5 kg.

3. RESULTS AND DISCUSSION

3.1. ATR-FTIR Analysis of Fiber Surfaces

ATR-FTIR analysis was performed in order to indicate the differences between sized and desized CF samples by means of functional groups on their surfaces. The ATR-FTIR spectra of desized and sized CF samples are shown in Figure 1. Absorption peaks at 2850 cm^{-1} and 2930 cm^{-1} are due to –CH hydrocarbon groups of desized CF [28]. The absorption band seen at 1720 cm^{-1}, which corresponds to C=O streching vibrations of carbonyl and carboxyl groups, appears for EP-CF and PU-CF [29]. C–O vibrations of sized CF samples can be seen at 1050 cm^{-1} [30]. The characteristic hydroxyl group peaks seen at 700 cm^{-1} (O–H stretching) and around 3500 cm^{-1} (O–H bending) present for only sized CF samples [31].

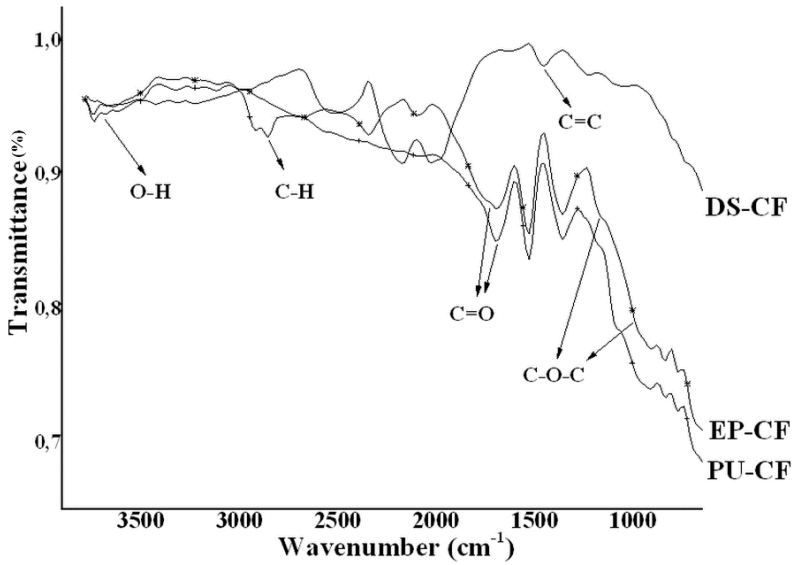

Figure 1. FTIR spectra of CF samples

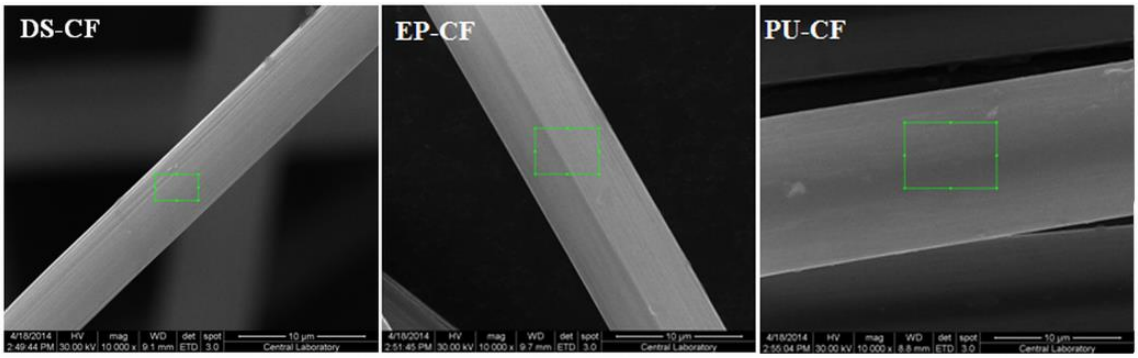

Figure 2. SEM micrographs of CF surfaces

3.2. SEM/EDX Analysis

SEM/EDX analysis was conducted to CF samples in order to obtain the sizing layer on CF surface as both physically and chemically. SEM images of desized and sized CF samples with x10,000 magnification are shown in Figure 2. Narrow grooves can be seen on the surface of desized CF sample. These grooves seen as almost disappear for EP-CF sample. For PU-CF, absence of grooves can be seen clearly. Surfaces of sized CF samples are seen as smoother relative to desized CF surface. According to the elemental analysis data (Table 1 and Figure 3) obtained green regions on SEM micrographs, it is clearly observed that the oxygen content of desized CF is raised by a factor of about 3 fold for PU sized and about 4 fold for EP sized CF samples. The highest oxygen content in EP-CF indicates the existence of epoxy unit on the fiber surface. Remarkable nitrogen percentage resulting from nitrile group on urethane containing surface is observed for PU-CF.

Table 1. SEM/EDX results 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
EP-CF	88.26 /90.92	11.74 /9.08	0 /0
PU-CF	92.59 /94.33	7.41 /5.67	2.53 /2.22

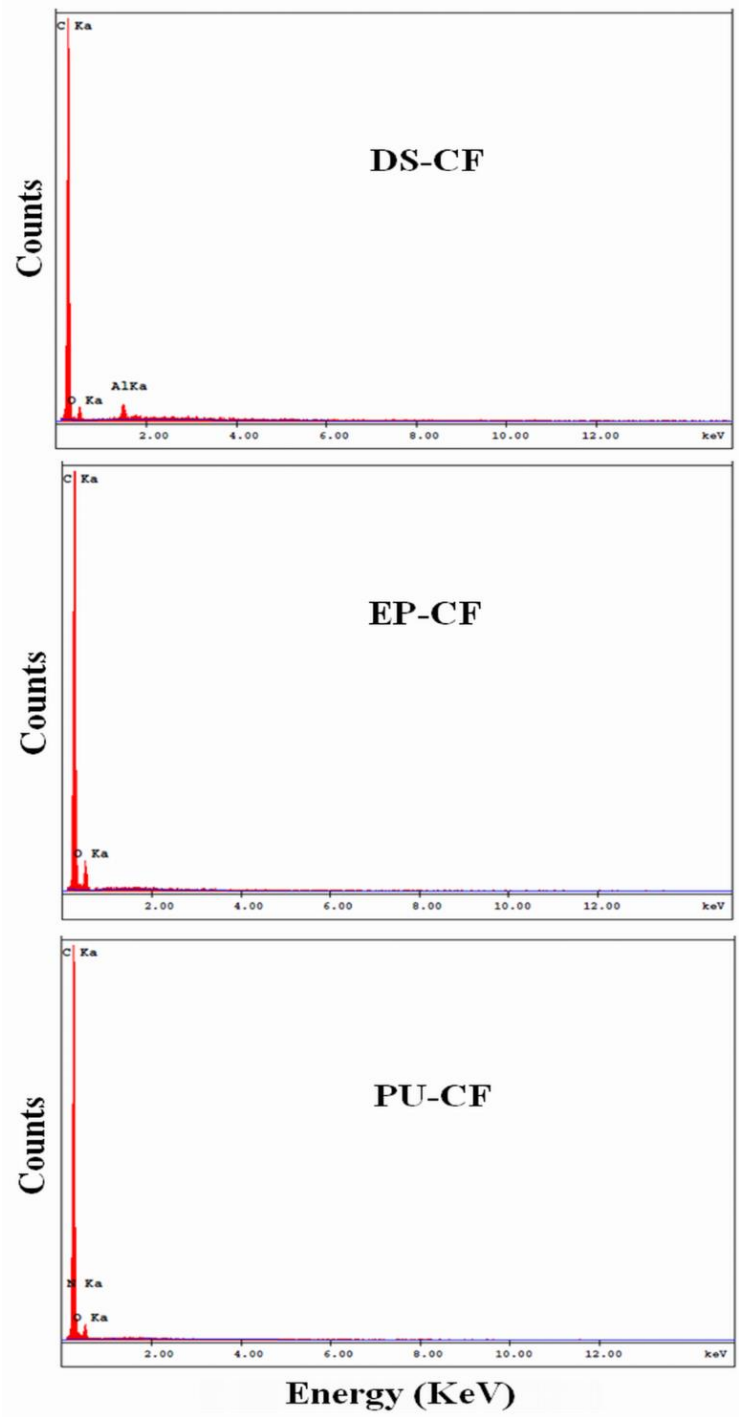

Figure 3. EDX graphs of CF samples

Representative SEM micrographs of cyro-fractured surfaces of composites at magnifications of ×500 (left side) and ×3,500 (right side) are shown in Figure 4. The observations from the SEM micrographs that sized CFs exhibit more homogeneous dispersion with respect to desized CF. It is observed from the micrographs of TPU/DS-CF composites that fibers are tend to form bundles. Debonding and large gaps are seen between desized CF and TPU. On the other hand, PU-CF and EP-CF are covered by TPU matrix because of the improved interfacial adhesion. It is clearly seen that sized CFs are dispersed homogeneously in polymer matrix.

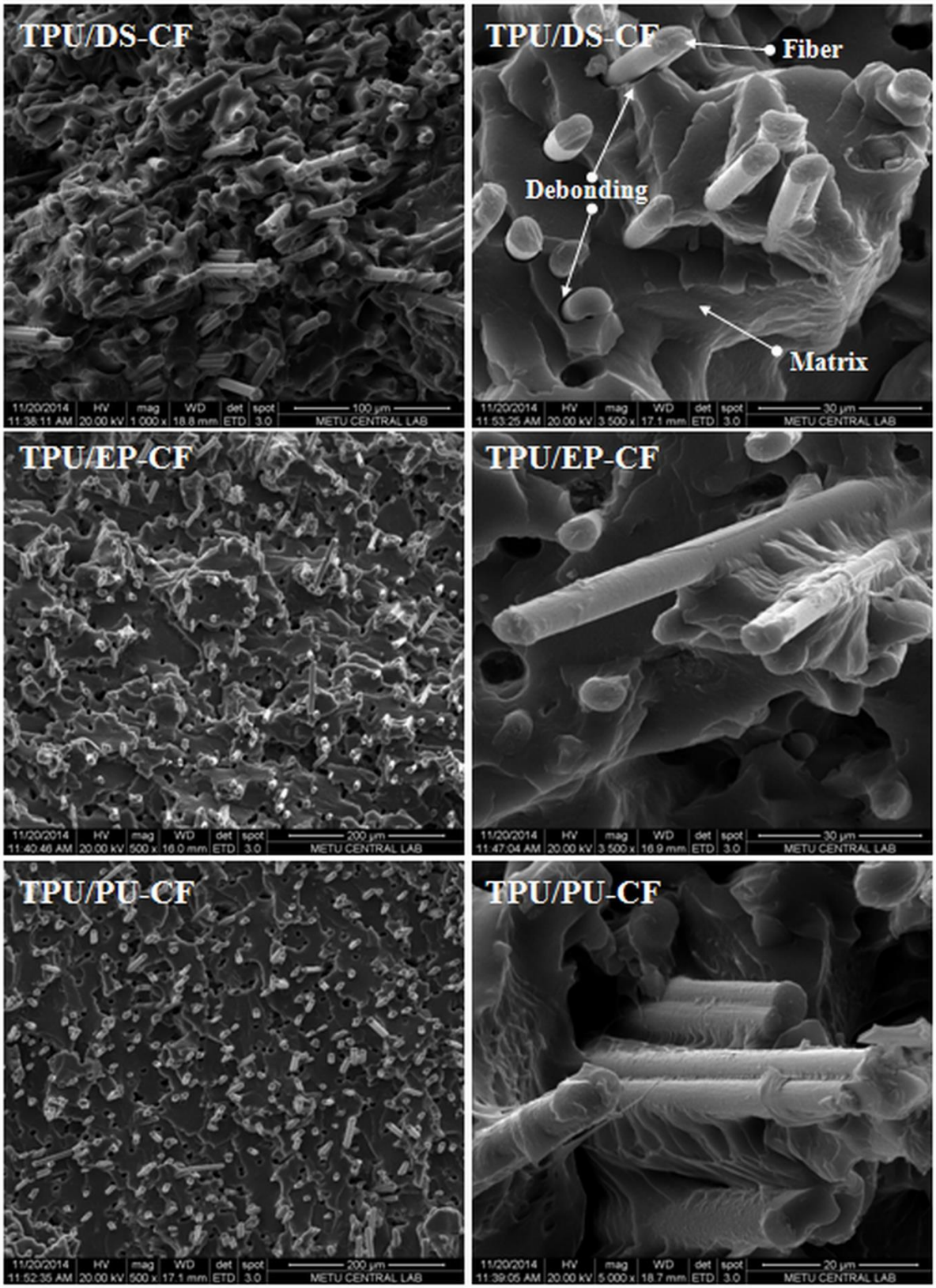

Figure 4. SEM micrographs of composites

Table 2. Tensile test results of TPU and its composites

SAMPLES	Tensile Strength (MPa)	Elongation at break (%)	Youngs' modulus (MPa)
TPU	24.8±1.7	331.8±4.3	33.4±3.0
TPU/DS-CF 5%	21.0±1.2	284.6±6.7	123.5±4.2
TPU/DS-CF 10%	23.7±2.1	151.5±4.2	124.4±5.2
TPU/DS-CF 15%	27.3±1.5	68.8±5.3	174.4±4.8
TPU/DS-CF 20%	29.4±1.8	36.1±4.4	240.2±7.3
TPU/EP-CF 5%	23.6±1.5	82.1±3.8	133.3±4.9
TPU/EP-CF 10%	33.1±1.9	64.8±2.8	186.1±5.5
TPU/EP-CF 15%	35.9±1.7	50.8±2.3	244.2±5.4
TPU/EP-CF 20%	50.5±2.6	32.7±3.2	317.4±6.8
TPU/PU-CF 5%	24.8±2.2	125.6±5.2	137.4±4.0
TPU/PU-CF 10%	36.0±2.4	58.6±4.7	191.2±4.6
TPU/PU-CF 15%	40.6±2.0	39.6±3.3	254.4±6.1
TPU/PU-CF 20%	51.8±3.2	38.4±2.7	329.3±7.2

3.3. Tensile Properties

Tensile test data of pristine TPU and relevant composites are represented in Figure 5 and relevant data are listed in Table 2. The general trend is observed with the addition of CF is the same regardless of CF type. Tensile strength and Youngs' modulus increase and elongation at break decreases with the increasing amount of CF in TPU matrix. The inclusion of CF even at the lowest concentration (5 wt%) enhances Youngs' is modulus by a factor of about 3.5 times with respect to neat TPU. It is well known fact that the filler with higher stiffness than the matrix can increase the Youngs' modulus of the composites. The highest modulus values are observed for PU sized CF loaded composites owing to better adhesion between PU-CF and TPU matrix. Similar result was obtained in the literature [32] by the means of PU sized CF and this result is also in agreement with theoretical predictions in which the increase in Youngs' modulus occurs nonlinearly with increase in short fiber amount in polymer composites [33]. On the other hand, the additions of sized and desized CF in TPU cause remarkable in elongation at break due to the restriction of TPU chain mobility. The increase in glass transition temperature also supports this comment (see DMA part). The reduction in elongation at break is more significant for sized CF loaded composites as compared to those of DS-CF to stronger interfacial bond formations. Although 15 wt % DS-CF loading is needed for exceeding the tensile strength of pristine TPU, 10 wt% CF loading is enough for sized CFs. PU-CF containing composites have higher tensile strength and Youngs' modulus than those of EP-CF containing ones at all concentrations. This trend arises from the polyurethane coating on the CF surface which is likely to be more compatible with TPU matrix.

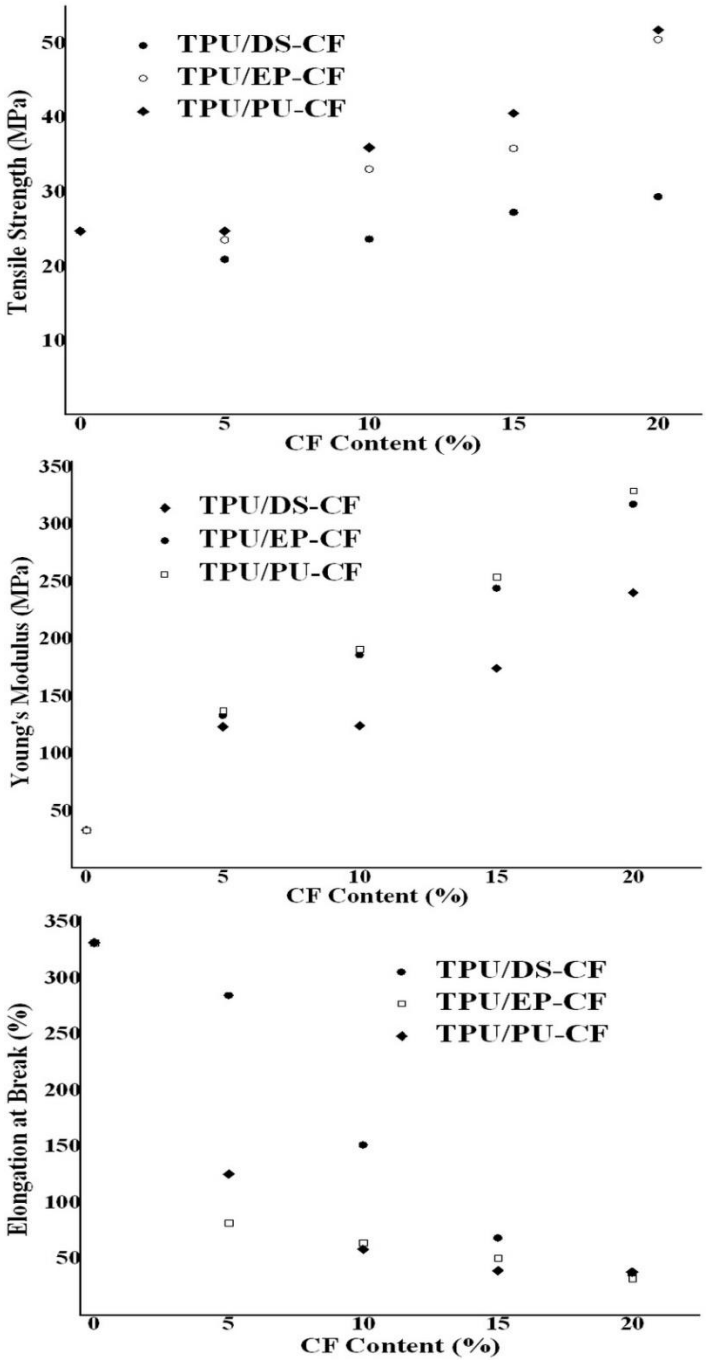

Figure 5. Tensile test results

3.4. Shore Hardness Test

Shore hardness is the characteristic parameter for elastomers and their composites. The hardness values of TPU and related composites are listed in Table 3. It can be seen from these values Shore A that the hardness of TPU increases with the increasing amount of either desized CF or sized CFs. Among the samples, PU-CF containing composites exhibit higher hardness values at all concentrations with respect to the composites containing the same amount of CF. The addition of 20 wt % PU-CF leads to

approximately 11 units increase with respect to neat TPU. Sized CF loaded composites give higher hardness results because of load transfer occur more effectively compared to desized ones [34].

Table 3. Shore A hardness of TPU/CF composites

CF Content (%)	TPU/DS-CF	TPU/EP-CF	TPU/PU-CF
0	84.8±0.2	84.8±0.2	84.8±0.2
5	86.7±0.1	90.2±0.1	92.7±0.1
10	88.8±0.2	91.4±0.2	93.1±0.1
15	89.6±0.2	92.9±0.1	94.0±0.2
20	90.0±0.1	93.3±0.1	95.6±0.2

3.5. Dynamic Mechanical Analysis

The dynamic storage modulus versus temperature graphs of TPU and 20 wt % CF containing composites are shown in Figure 6. It can be seen from these curves that the storage modulus of all the samples shows the sharp reduction at around -40 ^{0}C which corresponds to glass transition temperature of TPU. All CF composites show higher storage modulus than neat TPU at whole temperature range. The difference in the storage modulus of CF containing composites becomes negligible over 60 ^{0}C. The highest storage modulus is observed for PU-CF loaded composite at whole temperature range. It is concluded that TPU/PU-CF has the highest stiffness with respect to all composites. This may be due to the better compatibility of PU-CF with TPU matrix.

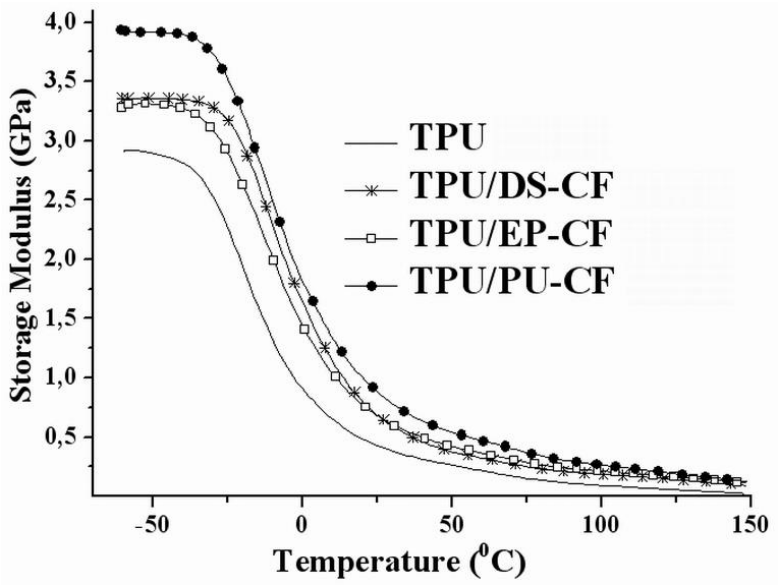

Figure 6. Storage modulus vs. temperature curves

The damping properties of composites are analyzed with their tan δ versus temperature curves which are shown in Figure 7. The peak of tan δ curve of TPU shifts to higher temperatures by the addition of both desized and sized CFs. This means that T_g of TPU increases with the addition of CF. The inclusion of PU-CF to TPU causes the reduction of tan δ. This observation arises from the hindrance of TPU chain motions due to the improvement of interfacial interactions [35]. The peak value of tan δ curve of TPU is remained unchanged after EP-CF addition.

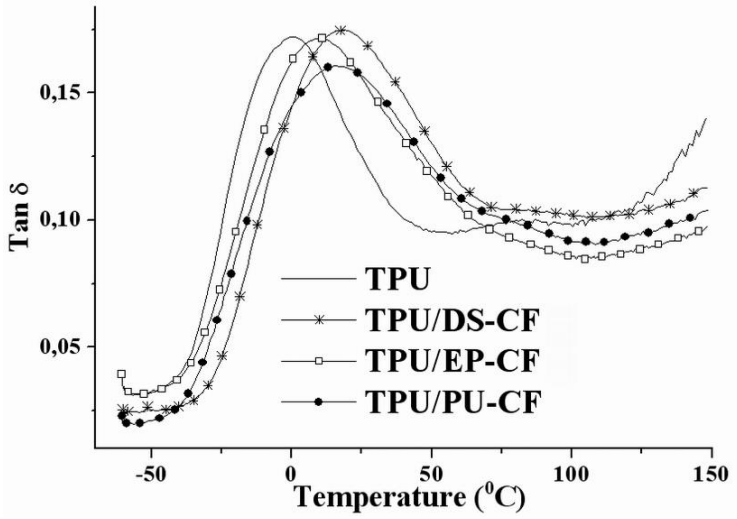

Figure 7. Tan δ vs. temperature curves

3.6. Melt Flow Properties

MFI values of unfilled TPU and related composites are given in Figure 8. MFI values of DS-CF containing composites are lower than that of neat TPU and they exhibit decreasing trend with increasing amount of CF (the exception of 10 wt% concentration). The DS-CF tends to form bundles due to the poor dispersion which is arising from the incompatibility between CF and TPU. The formation of bundles causes the reduction in MFI values because of fiber-to-fiber separation creating a complicated flow in thermoplastic matrix [36, 37]. On the other hand, the MFI values of sized CF containing composites are slightly lower than neat TPU at the lowest loading level (5 wt%). The further addition of EP-CF and PU-CF causes slight increase in MFI values. There is a correlation between the mechanical test results and MFI values. The alignment of fibers in the loading direction leads to increase in melt flow rate, strength and the modulus of composites. MFI values of sized CF containing composites are slightly higher as compared with TPU/DS-CF for all concentrations. As an overall investigation, it can be said that the incorporations of both desized and sized CFs do not cause remarkable change in MFI value of TPU at its process temperature.

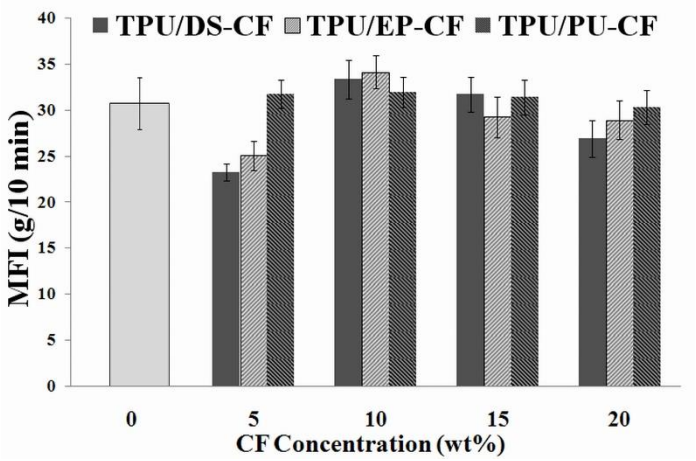

Figure 8. MFI values of TPU and its composites

4. CONCLUSION

This current study deals with the effect of CF amount and sizing on the mechanical, thermo-mechanical, and morphological properties of TPU based composites. According to tensile test results, the tensile strength of composites exhibits increasing trend with increase in CF content. The greatest tensile strength value is observed at the highest loading level (20 wt %) in all cases. The highest improvement of tensile properties is achieved for PU sized CF loaded composites. PU and EP sized CF inclusions cause remarkable increase in Shore hardness of TPU. DMA analysis reveals that the improvement in storage modulus of PU sized CF containing TPU composites is higher than those of EP sized CF and desized CF loaded composites. The glass transition temperature of TPU is shifted to higher values by the addition of CF regardless of sizing type. According to MFI values, no significant differences are obtained among composites. This means that CF addition causes no obvious problems the processability of TPU when the production of CF reinforced TPU composite parts will be considered by traditional production methods, extrusion and injection molding. These results show that TPU is a suitable matrix for chopped CF reinforced thermoplastic composites.

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