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Research Article

Influence of multi-walled carbon nanotubes on tensile and flexural properties of polyamide 66/short glass fiber composites

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Abstract

In this study, influence of multi-walled carbon nanotubes (MWCNTs) on tensile and flexural behaviour of 15% short glass fiber (SGF) reinforced Polyamide 66 (PA 66/15SGF) and 30% short glass fiber reinforced Polyamide 66 (PA 66/30SGF) is investigated. Test specimens composed of neat PA 66, PA 66/15SGF, PA 66/30SGF and PA 66/30SGF/MWCNTs are produced using plastic injection moulding machine; and their tensile and flexural properties are characterized. The effects of MWCNTs contents on the micro-structure and morphology of the composites were investigated by using a scanning electron microscope (SEM), fourier transform infrared spectroscopy analysis (FTIR) and optical microscopy (OM). Mechanical analyses reveal that neat PA 66 exhibits the lowest elastic modulus, 2.11 GPa, and tensile strength, 60.61 MPa, while the highest tensile modulus, 4.69 GPa, and strength, 87.05 MPa, are exhibited by PA 66/30SGF/MWCNT and PA 66/30SGF, respectively. In other words, with the addition of MWCNT, tensile strength of PA 66/30SGF decreases by 13.4 % whereas the elastic modulus increases by nearly 4.7 %. In addition, flexural test results shows that the integration of MWCNTs improves the flexural strength and flexural modulus of PA 66/30SGF by 1% and 12%, respectively.

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1. Introduction

Thermoplastics are commonly used in both commodity and industrial applications covering many industrial fields such as automobile, aeronautic and aviation, defence, sports industry and so forth [1, 2]. Some thermoplastics are considered to be suitable substitutes for metallic materials in industrial applications [3]. However, in spite of their advantages, thermoplastic materials may become deformed during the use or production [4]. In order to avoid these deformations and to increase their mechanical performance, thermoplastics can be filled with microscale or nanoscale reinforcing materials such as carbon fibers (CF), glass fibers (GF), carbon nanotubes (CNTs), nanoclays and so on [5, 6].

Being a thermoplastic, Polyamide 66 (PA 66) is one of the most outstanding materials used as engineering resin owing to its good mechanical, chemical and thermal performance [7, 8]. Apart from neat PA 66, there are various types of polyamides reinforced with materials such as glass fibers. The addition of glass fibers is known to increase the mechanical properties of polyamide-matrix composites [9-17]. The main factors determining the tensile properties of PA 66/GF composites are fiber fracture, diameter, length, orientation and interfacial strength [18-21]. In addition, production parameters such as mold temperature, injection pressure and speed may affect the mechanical properties of the polymer matrix composite materials [22, 23]. Recently, nanomaterials too have been used as reinforcement; and one of the most prominent of them are carbon nanotubes [24-26]. In terms of their forms, CNTs are categorized as single-walled carbon nanotubes (SWCNTs)

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[27] and multi-walled carbon nanotubes (MWCNTs) [28]. The use of MWCNTs as nanofillers is due to their superior mechanical properties such as very high tensile strength varying between 11-63 GPa [29] and Young's modulus, which is approximately 1 TPa [29, 30]. However, polymer matrices have drawn more attention in industrial applications owing to their light weight, easy machinability and production costs [31-34].

A number of theoretical and experimental studies have been carried out to date regarding CNTs/polymer composites [35-48]. Majority of these studies indicate that CNTs are able to improve the mechanical properties of polymer matrix composite systems. Coleman et al. [25], Miyagawa et al. [49] and F.S.A. Khan et al. [48] provided comprehensive reviews on the mechanical reinforcement of polymers by the use CNTs. It is suggested that well-dispersion of nano materials is an important parameter controlling the efficiency of load transfer and hence determining some of the mechanical properties [50]. Therefore, CNT-polymer matrix interaction and crack behaviour of CNTs/polymer composites have been the subjects of several studies [51-56]. Ajayan et al. [57] examined morphology of fractured epoxy/SWCNTs composites by SEM and observed SWCNTs stretching across a crack opening in the epoxy resin. Liu et al. [58] investigated the morphology and mechanical properties of MWCNTs-reinforced Polyamide 6(PA 6) composites. The authors reported a 26% reduction in tensile strength, which was explained by the brittleness of polymer matrix after the addition MWCNTs. Ferreira et al. [59] explored that addition of CNTs significantly improves the tensile strength and elastic modulus of PA 6/CNTs composites. Chopra et al. [60] studied PA 6/MWCNTs nanocomposites and reported that the presence of MWCNTs increases the tensile strength of Polyamide 6 by nearly 12%. Similarly, Kartel et al. [61] studied the tensile properties of PA 6/CNTs composites and reported that the tensile strength of the composite exhibits non-linear dependence behaviour by the addition of CNTs up to 0.5 wt.%. The mechanical tests performed by them showed that the PA 6 matrix composites incorporating 0.25 wt. % CNTs exhibit the highest tensile strength.

Although a great number of studies on CNTs/polymer composites are available in literature, there are not sufficient amount of studies focusing on the composites reinforced with the combination of short fibers and nanofillers [62, 63]. Therefore, the full potential of nanofillers and the properties of their combinations with other reinforcement materials, such as glass fibers, have not fully become known yet. This paper aims to introduce the tensile and flexural properties of MWCNTs-integrated Polyamide 66/short glass fiber nanocomposites so that the results obtained from this study can be used in designing new thermoplastic composites with MWCNTs.

2. Experimental

2.1. Composite Constituents

Neat PA 66, 15 wt. % short fiber glass reinforced PA 66 (PA 66/15SGF) and 30 wt. % short fiber reinforced PA 66 (PA 66/30SGF) granules (Mat Polymer, Istanbul/Turkey) were used as polymer materials in composite. MWCNTs, which were obtained from the manufacturer, (Ege Nanotek Kimya Sanayi, Izmir/Turkey) were used as nano reinforcements in composites (Table 1).

2.2. Fabrication Method

Figure 1 (a) to (h) shows the preparation process of the PA 66/30SGF/MWCNTs composite. The plastic injection machine was used to produce the test specimens. The granules were fed to the machine via a hopper and then pushed towards the nozzle by a rotating screw in the hot resistances. The temperature in the resistances which was nearly 285°C and the rotary motion of the screw facilitated PA 66/SGF granules to melt and

adequately mix with MWCNTs. Once this melted mixture reached the nozzle, it was injected into the moulds being hold between two clamps and took its final shape.

Table 1. Properties of MWCNTs

Parameter	Value
Purity (%)	>95 % (CNT) >97 % (C)
Outer diameter (nm)	10-20
Interior diameter (nm)	5-10
Length (μm)	10-30
Surface area (m^2/g)	>200
Colour	Black
Ash	Mass < % 1.5
Electrical conductivity (S/cm)	>100
Density (tap) (g/cm^3)	0.22
Density (true) (g/cm^3)	2.1



Fig. 1 Preparation process of PA 66/30SGF/MWCNT composite specimens

2.3. Characterization

Four types of specimens in accordance with ISO 527-2 type-1A and ISO 178 standards were produced by plastic injection moulding machine. A JSM-7001 F machine was used to characterize scanning electron microscope (SEM) (Japan) properties. Tensile and flexural properties of the specimens were examined using Instron 5982 100 KN (USA) test machine at room temperature with a crosshead speed of 5mm/min. Mechanical tests and specimens are shown in Figure 2 (a) to (d).

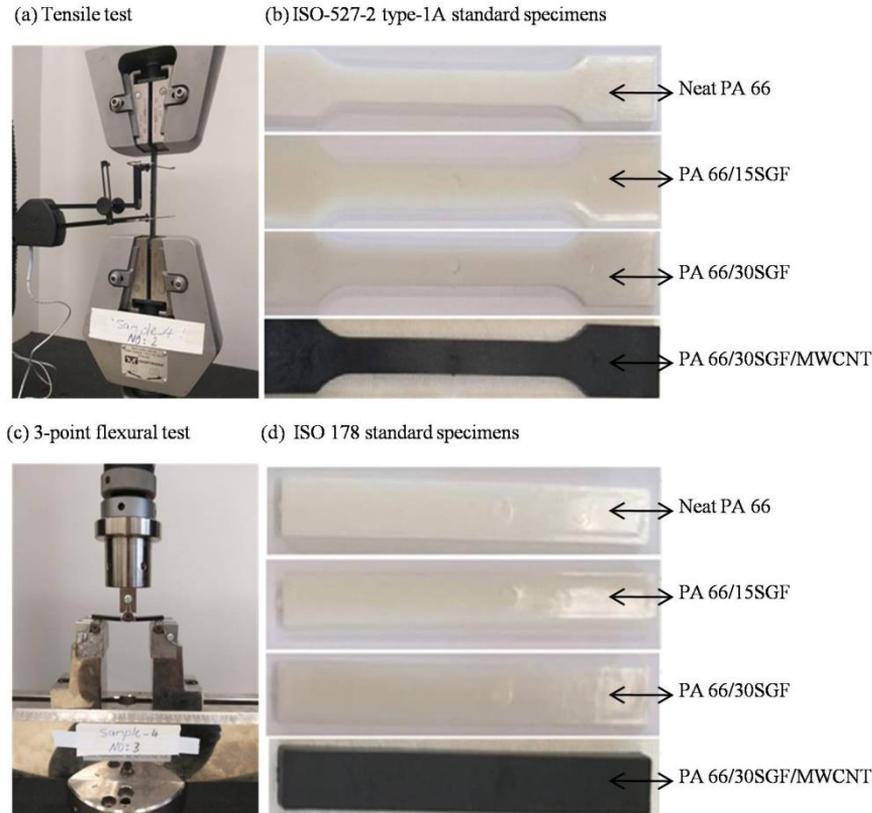


Fig. 2 Mechanical tests and specimens: (a) tensile test; (b) tensile test specimens in accordance with ISO 527-2 type-1A standard; (c) 3-point flexural test; (d) flexural test specimens in accordance with ISO 178 standard

3. Results and Discussion

3.1. Chemical Analysis

The aim of analyzing the molecular configuration of PA 66 by means of Fourier transform infrared (FTIR) spectroscopy is to correlate the structures to the performance properties of the final product. With sufficient knowledge about the chemical structure, polymerization reaction can be controlled and hence good performance properties can be achieved. Upon this purpose, FTIR spectra of neat PA66, PA 66/15SGF, PA 66/30SGF and PA 66/30SGF/MWCNT composites were measured and are shown in Figure 3. Due to very small weight fraction of MWCNTs in the composite and the affinity in chemical compositions of PA 66 and MWCNTs, the signature region did not exhibit a notable

difference. In neat PA 66, the absorption band at 3267 cm^{-1} is attributed to the stretching vibrations of N-H group. The absorption bands at 2912 cm^{-1} , 2843 cm^{-1} and 1192 cm^{-1} result from the symmetric and asymmetric C-H stretch vibrations and C-H twisting. The data obtained from FTIR analysis confirmed the chemical structure of PA 66 and PA 66/GF. Similar results were obtained by several researchers [64-66].

In this study, FTIR spectra of the composite specimens showed no significant change with regard to chemical composition of the constituent, which means that there are only physical interactions between the constituents. However, owing to the high-temperature (nearly $285\text{ }^\circ\text{C}$) and the rotary motion of the screw in the hot resistances of plastic injection machine, chemical interactions between the constituents might occur as well [67].

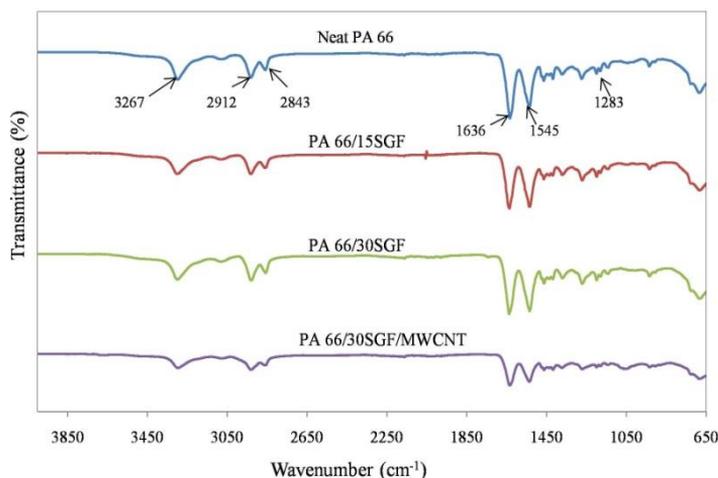


Fig. 3 FTIR spectra of the composite specimens

3.2 Tensile Test Results

Figure 4 (a) to (c) represent the load-displacement curves, stress-strain curves during tensile tests and the tensile test results of the composites, respectively. PA 66/30SGF exhibits the highest tensile strength (87.05 MPa) whereas PA 66/30SGF/MWCNT exhibits the highest elastic modulus (4.69 GPa). It can be inferred from the graph that the addition of glass fibers improves the tensile strength and elastic modulus of PA 66. This improvement could be explained by the good mechanical performance of glass fibers [10, 13, 68].

In the present study, we note that the addition of $0.4\text{ wt. } \%$ MWCNTs leads to a decline by 14% in tensile strength. This negative effect can be attributed to the poor dispersion and random orientation of the MWCNTs as well as their tendency to form agglomerates in the matrix. Moreover, it is obvious that the presence of MWCNTs increases the elastic modulus of PA 66/30SGF by 4.7% . Therefore, it could be suggested that MWCNTs contributes to the mechanical performance of PA 66/GF by sharing the external stress as well as bridging along the cracks. Moreover, they strengthen the composite system by improving the surface of glass fibers. As a result of even load distribution along the matrix, mechanical properties of the specimens increases. The obtained data also show that MWCNTs are compatible with glass fibers, which is very promising for the development of hybrid-filler composite systems.

Similar to this study, Jin et al. [69] noted a slight increase in the elastic modulus of PA 66/GF composite with the incorporation of CNTs and MWCNTs, which was attributed to the

interconnecting effect between the glass fibers and the PA 66 as a result of MWCNT coating. Qiu et al. [65] reported that the addition of 1.0 wt.% MWCNTs improves the elastic modulus of PA 66 by 3.14%. Furthermore, the authors observed that the SCF reinforced Polyamide 6/MWCNT composites incorporating low MWCNT content behave like polymer composites containing two different types of fillers whereas those incorporating high MWCNT content behave like short fiber reinforced nanocomposites.

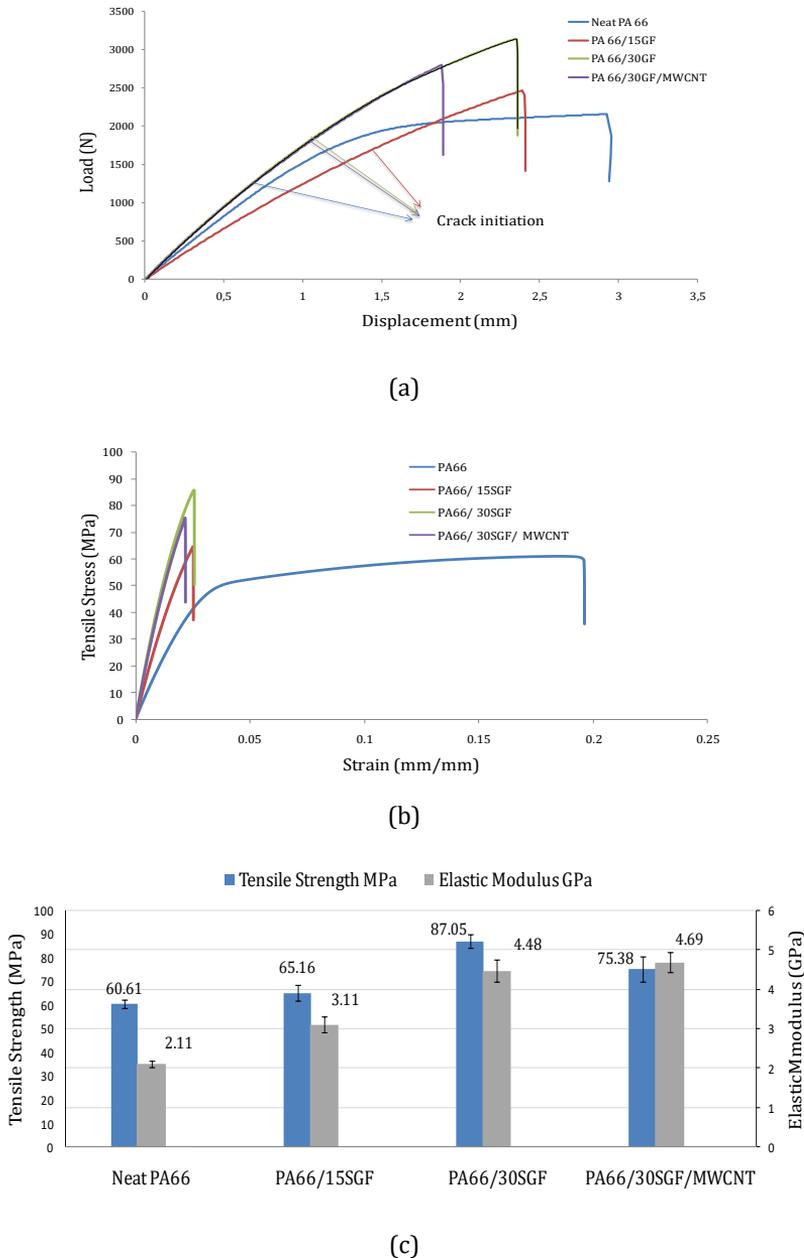
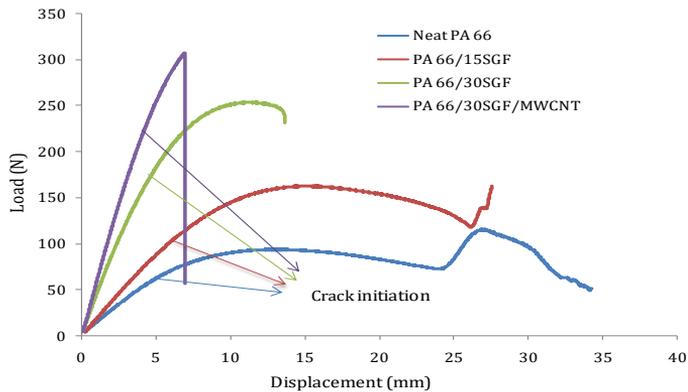


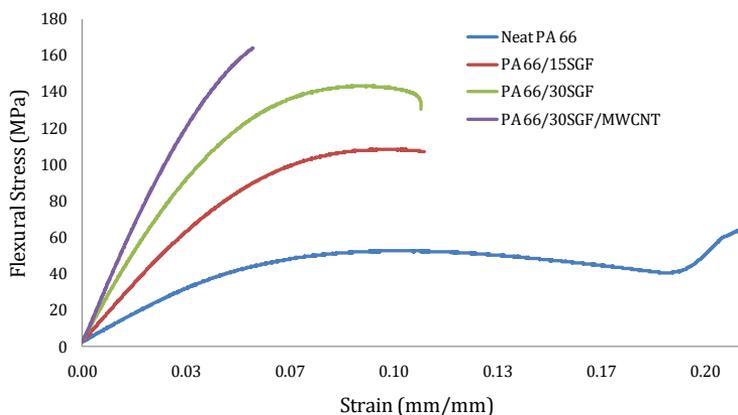
Fig.4 (a) Load-displacement curves of the specimens after the tensile tests; (b) Stress-strain curves of the specimens; (c) Tensile properties of the specimens

3.3 Flexural Test Results

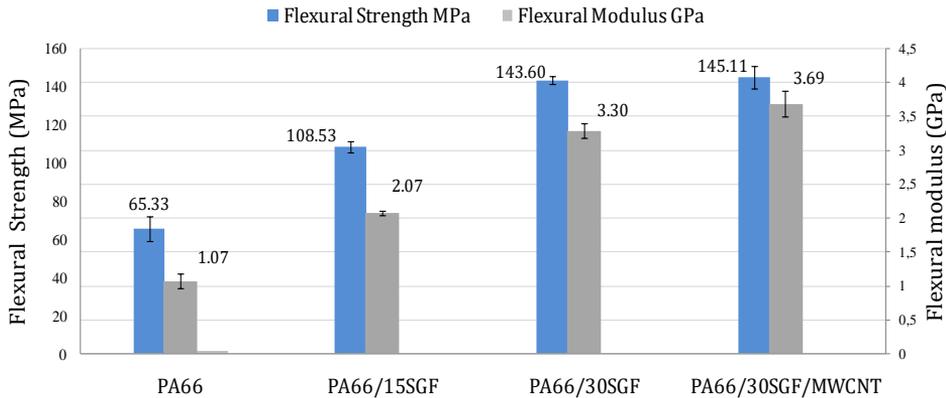
Fig 5 (a) to (c) demonstrate the load-displacement curves and stress-strain curves of the specimens during 3-point flexural tests and the flexural test results of the specimens, respectively PA 66/30SGF/MWCNTs composite exhibits the highest flexural strength (145.11 MPa) and flexural modulus (3.69 GPa) while neat PA 66 exhibits the lowest strength (65.33 MPa) and elastic modulus (1.07 GPa). Flexural strength and flexural modulus of the specimens significantly increase with increasing SGF content, which can be explained by the good mechanical properties of glass fibers as well as their well dispersion and homogeneous distribution in the matrix. Besides, an increase in flexural strength (by 1%) and flexural modulus (by 12%) after MWCNTs integration was observed, which can be attributed to the good surface interaction between the nanotubes and the PA 66 matrix as well as the surface improvement of glass fibers as a result of the MWCNTs coating. These results are compatible with a number of studies in literature. Autay et al. [9] studied the flexural properties of SGF reinforced PA 66 and reported that reinforcement resulted in an enhancement in the maximum flexural stress by nearly 36.3% for PA 66/10GF and 47.2% for PA 66/30GF in comparison to neat PA 66. Koilraj et al. [70] investigated the flexural properties of injection moulded PA 66/MWCNT and reported that the incorporation of 0.5 wt.% CNT content increases the flexural modulus by 5.8%.



(a)



(b)



(c)

Fig. 5 (a) Load-displacement curves of the specimens after three-point flexural tests; (b) Stress-strain curves of the specimens; (c) Flexural properties of the specimens

Figure 6 (a) and (b) represents the optical micrographs of the tensile fractured specimens after 3-point flexural tests. Failure modes of the composite specimens are dominantly matrix cracks along the direction of loading. Compared to the composite specimens with MWCNTs, longer cracks are observed in fractured PA 66/SGF.

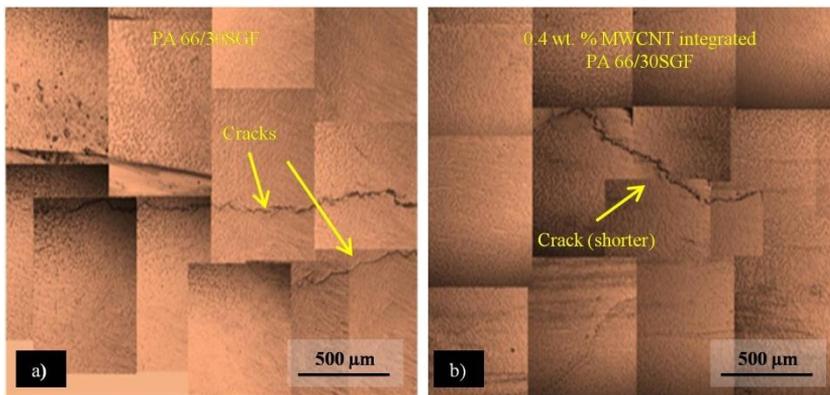


Fig. 6 Optical micrographs of fractured (a) PA 66/30SGF and (b) 0.4 wt.% MWCNT integrated PA 66/30SGF specimens monitored after 3-point flexural tests

3.4. Fracture Aspects of the Composite Specimens

SEM images of fracture surface morphologies of PA 66/30SGF/MWCNT composite are shown in Figure 7 (a) to (c). Figure 7 (a) indicates the SEM image of an individual glass fiber coated with MWCNTs. Higher magnification SEM images of the individual MWCNT coated glass fiber are shown in Figure 7 (b) and (c).

Uniform distribution of the fillers and their facial interaction with the matrix are key parameters for an effective reinforcement mechanism. To obtain good mechanical properties, fillers should evenly share the external stress applied to the matrix material. Regarding the CNT-polymer composites, CNTs are expected to bridge across the cracks formed inside the matrix during the fracture. This behaviour of CNTs prevents the crack

opening and propagation and hence improves the mechanical performance of the composite. Furthermore, if incorporated together with fibers, CNTs can function as effective interface modifiers improving the surface area of fibers and facilitate the adhesion between fiber and matrix.

The monitored SEM images in Figure 7 (a) to (c) demonstrate that some individual MWCNTs function as bridges between the surface of glass fiber and the PA 66 matrix. This bridging phenomenon contributes to toughness improvement by allowing the release of stress and absorbing the fracture energy. Similar observations were made in a number of studies. Qian et al. [71] observed nanotubes bridging across the cracks in polystyrene matrix by means of a TEM and noted that the elastic modulus of the composite increases by nearly 25% with the inclusion of 1 wt.% CNT. Punch et al. [72] examined the fractured surface morphology of PA 6/SCF/MWCNT composites by SEM and obtained clear images of nanotubes interconnecting lumps of the PA 6-matrix. Jin et al. [69] too obtained clear SEM images of CNTs and MWCNTs bridging across the cracks formed inside the PA 66/GF composites. The authors also revealed that MWCNTs coating glass fibers can significantly improve the interaction between the glass fiber and the matrix.

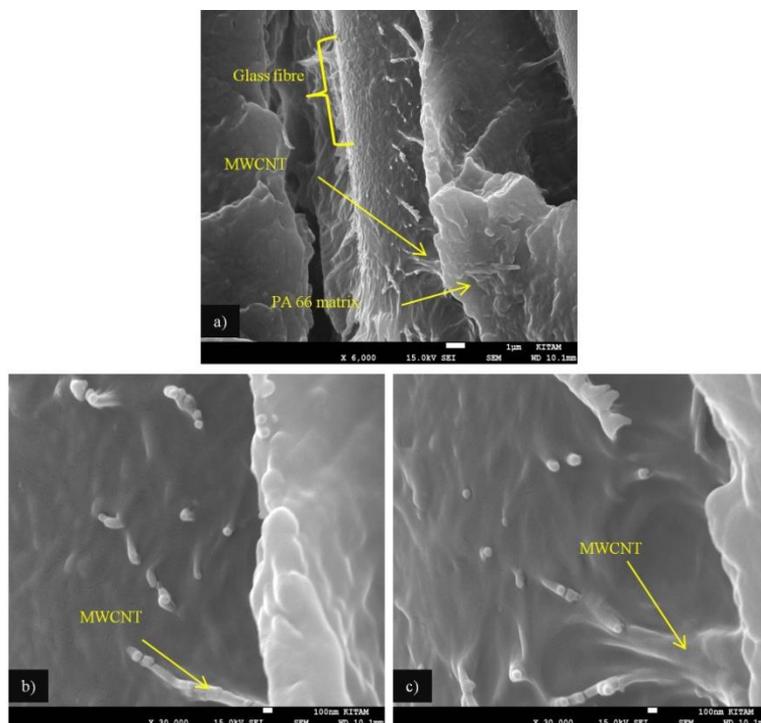


Fig. 7 SEM images of fractured MWCNTs-integrated PA 66/30SGF specimen

Figure 8 (a) and (b) show MWCNT pull-out, which probably occurred due to the fracture and poor interfacial interaction. While a crack is opening, nanotube is stretched absorbing the fracture energy transferred from the matrix. When the fracture ends, the crack somewhat closes and nanotube loosens getting a curved form as shown in Figure 8 (a) and (b).

Figure 9 (a) and (b) show the MWCNTs embedded within polymer matrix, which indicates good interfacial interaction. However, entangled MWCNT agglomerates were also observed as shown in Figure 9 (c), which restricts the dispersion and adversely affects the

mechanical properties. MWCNTs are prone to agglomerate. Earlier, agglomeration tendency of CNTs was attributed to the Van der Waals forces alone; however, the long length and high polarizability of the CNTs could also be determining factors that enhance the energy required to disperse a nanotube within the matrix [73].

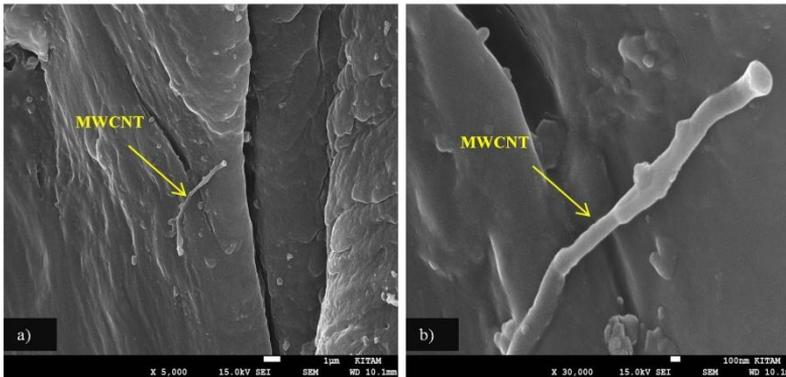


Fig. 8 SEM images of an individual MWCNT pull-out in a fractured PA 66/30SGF matrix

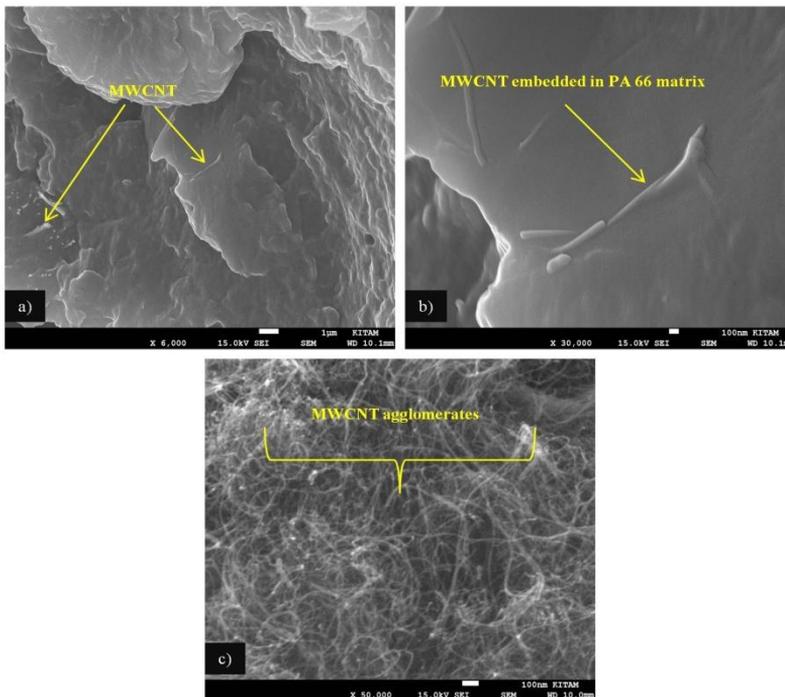


Fig. 9 SEM images of (a) MWCNTs embedded in PA 66/30SGF; (b) (higher-magnification) MWCNT embedded in PA 66/30SGF; (c) agglomerated MWCNTs

4. Conclusions

In this study mechanical properties of neat PA 66, PA 66/15SGF, PA 66/30SGF and 0.4 wt.% MWCNT integrated PA 66/30SGF were investigated. Furthermore, the effects of MWCNTs contents on the micro-structure and morphology of the composites were

investigated by using a scanning electron microscope (SEM), fourier transform infrared spectroscopy analysis (FTIR) and optical microscopy (OM). The conclusions based on the findings are summarized as follow:

- FTIR spectra revealed no chemical interaction between PA 66, SGF and MWCNTs, which means that there are only physical interactions between the constituents.
- The mechanical tests shows that PA 66/15GF and PA 66/30GF composites exhibit improved tensile and flexural properties compared with neat PA 66, which is due to the good mechanical properties of the glass fibers and the sufficient distribution of the external stress throughout the matrix.
- Regarding PA 66/30SGF/MWCNT composites, the presence of MWCNTs results in improvement in elastic modulus by 4.7%, flexural strength by 1% and flexural modulus by 12%.
- The improvement in the mechanical properties with the addition of MWCNTs is explained by i) high mechanical properties of MWCNTs, ii) the bridging phenomenon of MWCNTs, which prevents crack opening and propagation during the fracture of the composite
- Despite the improvement in elastic modulus and flexural properties, a decrease in the tensile strength was observed. This failure is due to the presence of MWCNT agglomerates acting as defects or stress concentration sites in PA 66/30SGF composite system.
- From the SEM images of MWCNT coated glass fibers, we can deduce that MWCNTs modify the surface area of fibers and some individual MWCNTs function as bridges between the surface of glass fiber and the PA 66 matrix.
- Considering the improvement in elastic modulus, flexural modulus and flexural strength, it can be concluded that even a small mass fraction of MWCNT is capable of enhancing the mechanical performance of glass fiber filled PA 66. This achievement proves that MWCNTs are quite promising for designing new thermoplastic composites.

Our future study will be investigating the tensile, bending and Charpy impact properties of hemp fiber reinforced thermoplastic composite materials.

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