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Evaluating the effects of multi-walled carbon nanotubes on the mechanical properties of chopped strand mat/polyester composites

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ABSTRACT

In this research, the influence of adding multi-walled carbon nanotubes at various contents on the mechanical properties of chopped strand mat/polyester composites was investigated. Initially, the effect of the sonication time on the dispersion of carbon nanotube at the highest weight ratio (0.5 wt.%) was inspected. To achieve this goal, a new technique based on scanning electron microscopy, which utilizes the burn-off test, was introduced to visualize the dispersion state of carbon nanotubes. Subsequently, the effect of addition of multi-walled carbon nanotube on the tensile and flexural properties of the fiber reinforced composites was studied. The results of mechanical tests showed that adding only 0.05 wt.% carbon nanotube enhanced the flexural strength of the hybrid composite by 45% while the tensile strength was not changed significantly. Improvements in the tensile and flexural moduli were also observed. Moreover, theoretical relations between the tensile, flexural and compressive moduli based on the classical beam theory were employed to determine the effect of carbon nanotube on the compressive modulus of composites. The theoretical result showed 31% enhancement in the compressive modulus.

1. Introduction

Glass/polyester composite is a class of fiber reinforced polymer matrix composites (FRPs) with a wide range of usage in various fields of applications. Chopped strand mat (CSM)/polyester composite is a member of this family that due to low price and acceptable mechanical properties is used as turbine blades and composite super-structure such as bridges and decks [1,2].

Due to their excellent physical and mechanical properties [3], carbon nanotubes (CNTs) have played an important role in various fields of engineering material researches since their discovery by Iijima and Ichihashi [4]. In recent years, CNTs have been used as filler in order to enhance some physical, electrical and mechanical properties of polymers [5–9].

Various techniques such as ultrasonication, calendaring, high pressure homogenizing [10] and similar methods have been utilized to disperse CNTs in numerous media. Although these methods try to achieve full dispersion of CNTs, they have inevitable negative effect on the aspect ratio of CNTs. In other words, a well dispersion of CNTs leads to reduction in the aspect ratio as well as some possible damages to the atomic structure of CNTs [11]. Therefore, investigation the dispersion state of CNTs and their aspect ratio during the dispersion processes plays a significant role in controlling the properties of fabricated samples.

The techniques which are used to study the dispersion state of CNTs can be categorized in two methods of direct and indirect. By indirect methods, the CNT dispersion and aspect ratio are not visualized and are measured mostly via particle size analyzing methods [12]. Moreover, the effect of CNT dispersion on other physical properties such as viscosity [13], electrical conductivity [14], mechanical properties [8,15] and viscoelastic properties [16] are studied to indicate the dispersion status of particles. Changes in any of the studied properties can be attributed to the dispersion state of CNTs and their aspect ratios.

In direct methods, which are generally based on visualization, microscopy is utilized to image the CNTs. By such a method, the CNTs can be visualized while they are embedded in the matrices. Optical microscopy can be used only to image the state of agglomerations [17] although electron microscopes [18,19] and atomic force microscopes [20] can reveal both single and bundled CNTs. Usually a combination of both direct and indirect methods is used to better interpretation and verification of results.

Kovacs et al. [19] visualized more CNTs on surface of a polymeric matrix composite by optimizing the scanning electron microscope (SEM) parameters. However, their inspection was limited to approximately 50 nm of the sample depth and could not obtain more information from deeper regions. The critical issue concerning microscopy of CNTs in a polymer matrix is that very

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small and local areas can be seen at an acceptable scale, which requires more picturing of the specimen. As in transmission electron microscope (TEM) micrographs, a segment of nanotube can be imaged and total lengths of CNTs are usually hidden. In SEM and atomic force microscope (AFM) images, many CNTs are buried in the matrix and may not be imaged. It is also noteworthy that the whole length of CNTs which appears on the surface of specimen may not be seen as they might enter the bulk rather than lying on the surface. Thus, it causes difficulties for studying the aspect ratio of particles.

Some researches have been conducted to investigate various effects of adding CNTs into FRP. Such type of composites containing reinforcements of different scales is called multi-scale composite. Generally, there are two major methods for fabricating such multi-scale composites. The first technique is based on growing CNTs on the surface of fibers and using these modified fibers for producing the composites. Mathur et al. [21] used this method and examined the effects of growing CNTs on carbon fibers on the enhancement in flexural and tensile strengths of phenolic based composites. Sager et al. [22] also investigated the interfacial shear strength of carbon fiber composites by using CNT grown fibers. In the second method, CNTs are dispersed in the polymeric matrix and subsequently utilized for fabricating the multi-scale composites. Godara et al. [23] used 0.5 wt.% of different types of CNTs to prepare multi-scale composites with unidirectional carbon/epoxy prepregs and reported the enhancement in the mechanical and thermal properties of the resulting nanocomposites. Kim et al. [24] used vacuum assisted resin transfer molding (VARTM) method to fabricate CNT/carbon/epoxy composites and studied their tensile and flexural behaviors.

Despite several researches on nano-reinforcing of FRPs, there is no specific investigation on reinforcing glass CSM/polyester composites by addition of CNTs in the literature. In the present study, the effects of adding multi-walled carbon nanotube (MWNT) at different weight ratios (0.05, 0.1, 0.3, and 0.5) on the mechanical properties of CSM/polyester composites were investigated. Mechanical stirring and sonication technique were employed to achieve a good dispersion state of nanotubes in polyester resin. To inspect the dispersion state of MWNTs, a novel technique was introduced. By applying this method on the nanocomposite contained 0.5 wt.% MWNT, the dispersion and aspect ratio of MWNTs which were laid on the glass fibers were studied by using SEM. Also, the suitable sonication time was obtained. Then, the tensile and flexural tests were performed in order to find out the effects of MWNT on the strengths and moduli in tension and bending states. The compressive strength and modulus were also calculated using theoretical relations.

2. Experiments

2.1. Materials

The chemical vapor deposition (CVD) grown MWNTs were supplied by Io-Li-Tech Co., (Germany) and had diameter between 10 to 20 nm and length in the range of 5–15 μm. Glass chopped strand mat with density of 450 kg/m^2 and unsaturated polyester resin (Boytec Co., Turkey) were employed to fabricate fiber reinforced composites. Curing process was performed by addition of cobalt (8%) and methyl ethyl ketone peroxide (MEKP) (Butanox M60) as recommended by the manufacturer.

2.2. Preparation method

After initial mechanical stirring (20 min at 2000 rpm) of MWNT and polymer, which was performed to achieve primarily distribution of MWNT agglomerates, sonication technique was employed to disperse nanotubes in the polyester.

To obtain suitable dispersion of MWNT in polyester, the highest filler ratio (0.5 wt.%) was chosen. Three different sonication durations (15, 45, and 135 min) were applied to the mixture of MWNT and polyester to investigate the effect of sonication time on the dispersion as well as the aspect ratio of MWNT. Thus, suitable sonication time could be obtained. The sonication within all duration was done at output power of 200 W (Hielscher UP400S, Germany) with a probe of 14 mm diameter and pulse rate of 0.5 which means that sonication wave was applied for 0.5 s and stopped for another 0.5 s. Also, all samples were ice-bath cooled to prevent any possible degradation due to overheat imparted by sonication. After sonication, the hardener constituents were added to the mixture and immediately the nanocomposites of MWNT/CSM/polyester were fabricated by hand lay-up method to undergo mechanical and burn-off tests.

After choosing the proper sonication time, MWNTs at 0.05, 0.1, 0.3, and 0.5 wt.% were added to the polyester resin and the same procedure as described formerly was applied. It is noteworthy that only sonication time was changed for each filler weight ratio [14]. After dispersion of the nanotubes in the polyester resin, the conventional hand lay-up method was employed to fabricate composite sheets. Subsequently, each sheet was cut into the pieces of proper dimensions for tensile and flexural tests according to the standards.

2.3. Characterization

2.3.1. Tensile and flexural properties

Tensile and flexural tests were performed in accordance with ASTM: D3039-08 and ASTM: D790-10, respectively to evaluate the effects of adding MWNTs into the polyester by using the universal testing machine, Santam STM-150. The effective parameters of tests such as minimum number of the required samples, speed, and dimensions. were chosen according to the standards.

2.3.2. Compressive properties

In order to determine the effect of MWNTs on the compressive properties of MWNT/CSM/polyester composites, the theoretical approach developed by Mujika et al. [25] was used. They found a relation between tensile, flexural and compressive moduli in fiber/polymer composites based on the classical beam theory. When a composite beam is subjected to bending condition, one portion of cross section area is under tension while another experiences the compression state. According to Mujika et al., λ is defined as the ratio of tensile modulus to compressive modulus:

$$\lambda = \frac{E_T}{E_C} \tag{1}$$

By using moment equilibrium equation and defining parameter $\beta = \frac{4}{(1+\sqrt{\lambda})^2}$ then:

$$E_B = \beta E_T \tag{2}$$

The compressive modulus of the samples then can be determined using Eqs. (1) and (2) in term of the tensile and flexural moduli that were obtained from experimental results.

2.3.3. Characterization of dispersion

After fabrication of the nanocomposites, they were cut into the pieces and were put into a furnace with the environment atmosphere and heated up to over 470 °C for 1 h. At this temperature, the polyester resin is burned off and glass strand mat and MWNT remain intact. It is worthwhile to mention that high purified MWNT can be obtained at this temperature [26]. It means that amorphous carbon is also burned off. Therefore, almost MWNTs remain on the glass fibers. The burned off specimens were then collected on SEM sample holders and coated with Au–Pd to prepare them for SEM. Tescan Vega II electron microscope was used to investigate the fracture surface and dispersion state of MWNTs.

3. Results and discussion

3.1. Effect of sonication energy

Fig. 1 shows the initial size and aspect ratio of added MWNTs. It is obvious that the total length of a particle cannot be followed and measured easily in the micrograph as the particles are highly entangled. However, it is clear that CNTs had very long length near to the manufacturer datasheet. After dispersion of MWNTs in the polymeric matrix, the introduced method was applied to investigate the dispersion state and aspect ratio of them, qualitatively.

Figs. 2–4 show the burned off specimens, which were fabricated with sonication durations of 15, 45 and 135 min, respectively. In these figures, glass fibers of 10 μm diameter can be observed which acted as the substrate and MWNTs laid on them after burn off the polymer. As can be seen apparently in Fig. 2, an agglomeration containing a large number of MWNTs was laid on a glass fiber. Also, dispersed single MWNTs are available on the glass fibers. In contrast with Fig. 1, MWNTs lengths and diameters can be more easily measured. However, the lengths of MWNTs in the agglomerate are not traceable. Fig. 2 depicts a partial dispersion of MWNTs.

It is noticeable that utilizing glass fibers as substrate enables deploying the resin between them and prevents heavily stacking of MWNTs on each other after burning off the polymer. It is in fact the key role of using glass chopped strand mat. In case of burning off CNTs/polymer composites, what remains is just heavily stacked CNTs.

In comparison with Fig. 2, Fig. 3 presents the dispersed MWNTs which were sonicated with the intermediate time. The MWNTs are well separated, dispersed and no agglomeration can be seen in the micrograph. The MWNTs are not entangled unlike what is appeared in Fig. 2. The state of MWNTs in Fig. 3 proves that particles had been dispersed well in the matrix before the burn off practice as they remained evenly on the surface of glass fibers.

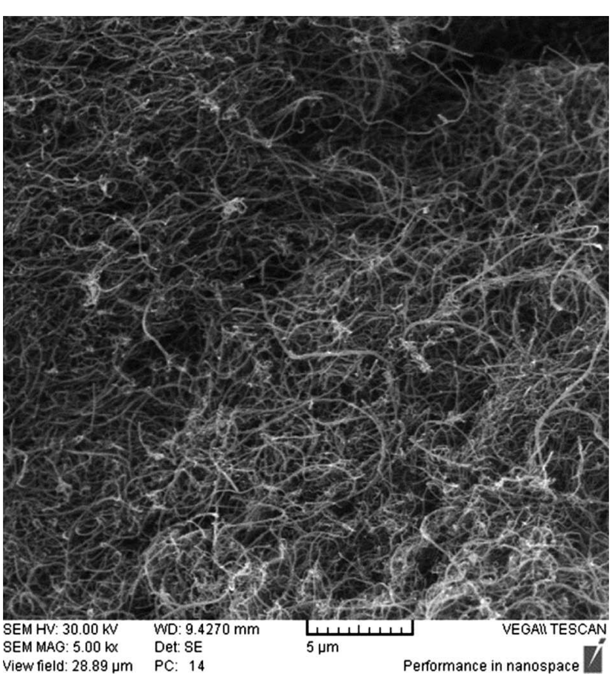

Fig. 1. SEM micrograph of supplied MWNT.

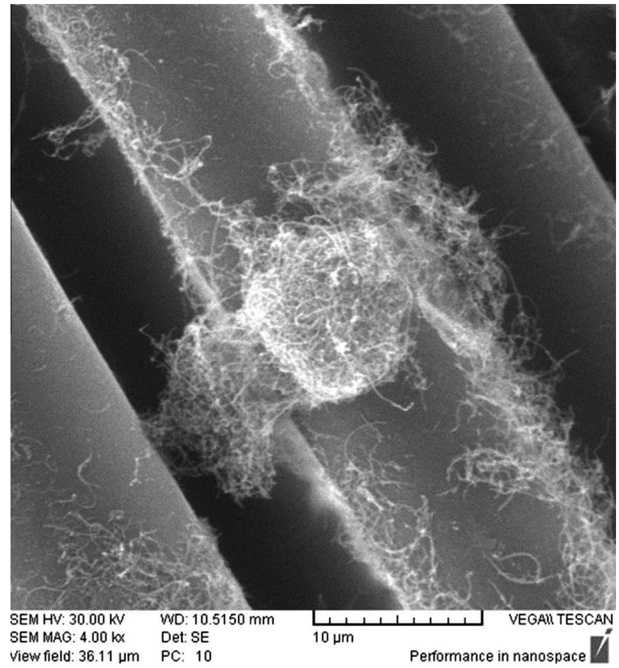

Fig. 2. SEM micrograph of specimen sonicated for short time.

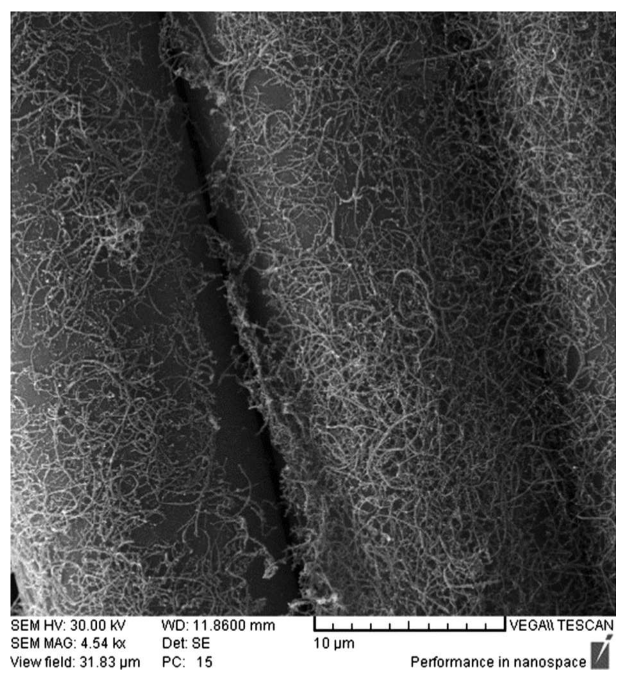

Fig. 3. SEM micrograph of specimen sonicated for medium time.

Fig. 4 shows a completely different state of MWNTs. It can be seen neither agglomeration nor many particles with the original sizes left on the glass fibers. The remains were MWNTs of very small aspect ratios compared with what exists in Figs. 2 and 3. It indicates that higher sonication time shortened the MWNTs [27].

The amount of energy given to a suspension by sonication is proportional to the sonication time; i.e. the more sonication time, the higher energy used to disperse particles in a suspension. Figs. 2–4 present how increasing sonication time and thus energy altered the dispersion and aspect ratio of MWNTs. To quantify the qualitative results obtained by the SEM, the flexural strength of fibrous nanocomposites were measured. Flexural test was

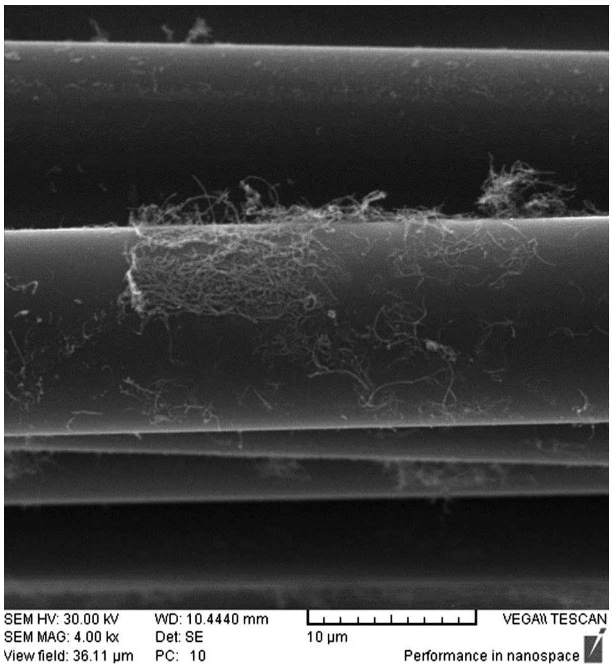

Fig. 4. SEM micrograph of specimen sonicated for long time.

performed on samples contained 0.5 wt.% MWNT. The flexural strengths of samples are shown in Fig. 5. As shown in this figure, the low and high energy levels caused degradation in the flexural strength of MWNT/CSM/polyester composites. These degradations occurred due to the weak dispersion and damage of nanotubes, respectively. In addition, the flexural strength of low sonicated sample was less than that of the samples without nanotubes. Presence of large agglomeration of MWNTs acted as stress concentration and as a result the strength of composites was decreased. On the other hand, applying long sonication time to the mixture of MWNT/polyester caused reduction of aspect ratio which led to deterioration of the enhancing effects of MWNT. The obtained suitable sonication time agreed with previous works [14,28]. Therefore, to eliminate the effect of over-sonication on the samples which contained lower amounts of filler, proper sonication times were chosen [14].

3.2. Tensile properties

Figs. 6 and 7 present the experimental results of tensile strength and modulus of MWNT/CSM/polyester composites, respectively. According to the results, the tensile strength did not show any significant improvement by adding MWNTs. Although the tensile strength of neat resins such as polyester and epoxy resins have been enhanced by adding CNTs [29,30] but in the present research, the tensile strength of CSM/polyester composite were not affected by addition of CNTs. It is because of high volume fraction of glass fibers (ca. 40 wt.%) in the composites, which played the major role in the tensile strength of MWNT/CSM/polyester composites. However, the presence of MWNT affected the modulus of composites.

As shown in Fig. 7, increasing the amount of nanotubes up to 0.3 wt.% caused increase in the value of the tensile modulus. By more increase in the amount of filler, the modulus was decreased. This reduction might be due to presence of more agglomeration at that weight ratio. The highest increase in the tensile modulus was 18% which occurred at 0.3 wt.% MWNT.

3.3. Flexural properties

Adding MWNT to the CSM/polyester composites had more significant effect on the flexural properties compared with the tensile properties. The results of flexural tests show that by adding very low weight fraction of MWNTs, the flexural strength of the resulting composites had a large enhancement. According to Fig. 8, at only 0.05 wt.% MWNT, the flexural strength was increased by 45%. At larger weight ratios, this great enhancement was vanished although the results were close to that of the 0.05 wt.%. Similar to the tensile modulus, the flexural modulus was also enhanced with increasing the weight ratio of MWNTs till 0.3 wt.%. At 0.3 wt.%, over 20% growth of flexural modulus was observed (Fig. 9). Higher flexural strength compared with the tensile one is due to the nature of flexural test in which area above the neutral axis is under tension and the area below the neutral axis is subjected to the compression (or vice versa related to direction of bending moment). Similar trend in the flexural strength of nanoreinforced composites was reported by Hossain et al. [31]. They investigated the flexural properties of carbon nanofiber (CNF) embedded in the glass/polyester composites and reported 31% enhancement in the flexural strength at 0.1 wt.% CNFs. Degradation in the mechanical properties was occurred at 0.4 wt.% of CNFs due to agglomerations. Sanchez et al. [32] also obtained the highest flexural strength of carbon fiber/epoxy composites by adding 0.1 wt.% of CNTs. At higher weight fractions of CNTs including 0.2 and 0.3 wt.%, the flexural strength of the resulting composite decreased. It is noteworthy that in neither of the mentioned researches [31,32], CNT and CNF were not added at lower weight fraction of 0.1 wt.%.

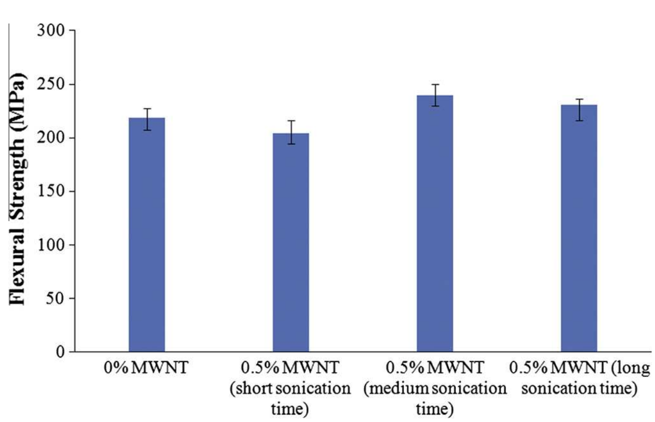

Fig. 5. Effect of sonication time on the flexural strength of MWNT/CSM/polyester composites.

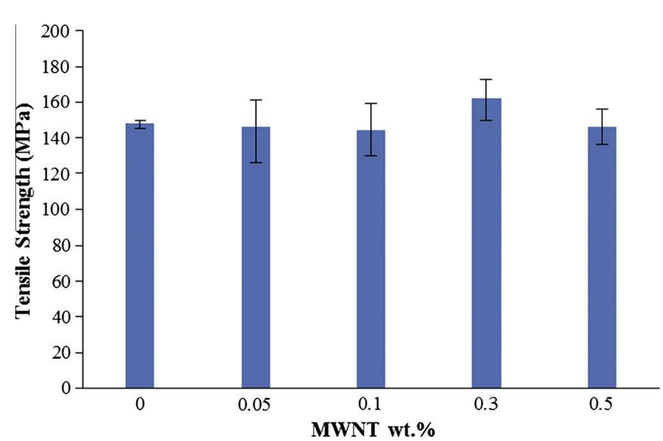

Fig. 6. Tensile strength of MWNT/CSM/polyester at different nanotubes contents.

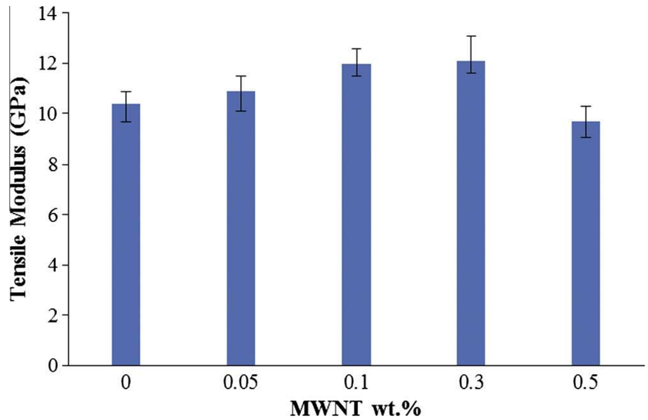

Fig. 7. Tensile modulus of MWNT/CSM/polyester at different nanotubes contents.

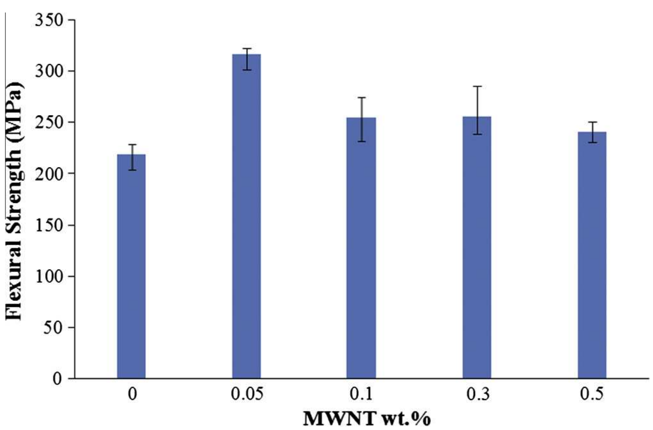

Fig. 8. Flexural strength of MWNT/CSM/polyester at different nanotubes contents.

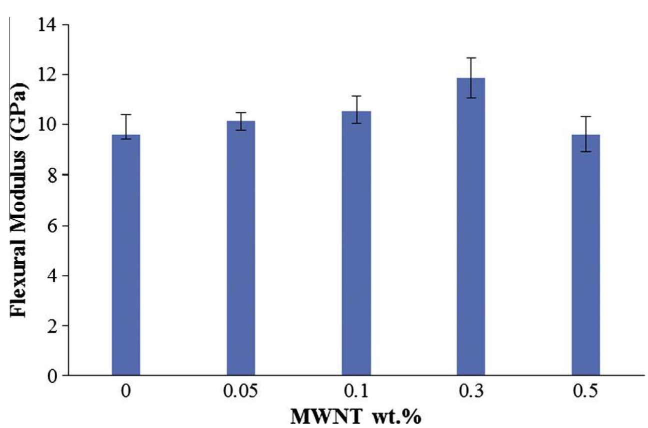

Fig. 9. Flexural modulus of MWNT/CSM/polyester at different nanotubes contents.

3.4. Compressive properties

Similar to the tensile properties, the compressive properties of polymeric resins have been enhanced by adding CNTs. The enhancement can be occurred in both compressive strength and modulus [33]. In this section the compressive moduli of MWNT/CSM/polyester composites were calculated analytically according to Section **2.3.2** using Eqs. (1) and (2). First, the parameter β was calculated for samples and then λ was determined according to β. The compressive modulus then can be found using λ and the tensile modulus. For verification of the results, the compressive modulus of samples containing 0 wt.% MWNTs was compared with

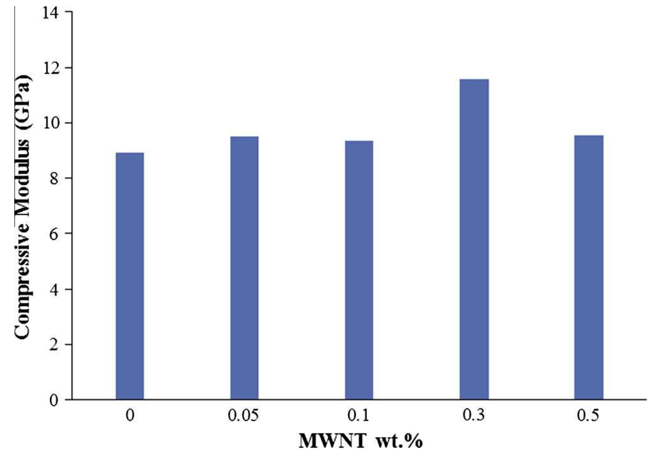

Fig. 10. Compressive modulus of MWNT/CSM/polyester at different nanotubes contents.

the experimental data in [34]. For 40 vol.% glass fiber, the ratio of tensile to compressive modulus, λ, is 1.13. For the experimental data that were obtained in this investigation, λ was 1.18 which is close to the calculated result. The compressive modulus for various weight fractions of CNTs was calculated and is presented in Fig. 10. As can be seen in Fig. 10, enhancement of 31% at 0.3 wt.% MWNT was observed. This enhancement is due to improvement in the mechanical properties of nano-reinforced resin. Similar to the tensile and flexural moduli, degradation in the compressive modulus was remarked at 0.5 wt.% MWNTs, as can be seen in Fig. 10. The reduction of compressive modulus is due to degradation in the tensile and flexural properties of polyester resins imposed by MWNT agglomeration.

4. Conclusions

The aim of this work was to evaluate the effect of addition of MWNTs on the mechanical properties of conventional CSM/polyester composites and also determination the influence of sonication time on the dispersion state of nanotubes in such composites. The results showed that there was an optimum sonication time with regards to restraining other sonication parameters such as output power and probe diameter. Low sonication time did not have the ability to disperse MWNTs fully in the polyester resin. On the other hand, applying too much sonication time could damage the nanotubes and as a result, less efficiency was reported. Also, mechanical properties of the resulting multi-scale composites at various weight ratios of nano-reinforcements were studied. According to the results, the flexural strength of MWNT/CSM/polyester composites was enhanced by 45% at only 0.05 wt.% MWNT. Moreover, the tensile, flexural, and compressive moduli of composites were improved by increasing the amount of MWNTs, reaching a peak at 0.3 wt.%. At higher weight ratios, the dispersion limitations as well as fabrication difficulties due to the increase in the mixture viscosity were major reasons for degradation of mechanical properties. The SEM micrographs of burned off specimens confirmed the dispersion state and aspect ratio of MWNTs affected by sonication.

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