Tensile Properties of Hybrid Biocomposite Reinforced Epoxy Modified with Carbon Nanotube (CNT)

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A tensile test was conducted to investigate the mechanical properties of hybrid bio-composites that have potential for application in helmet shells. Helmets can protect users from serious injuries, reducing traumas and deaths. Military helmets are made with 19 layers of Kevlar, and bicycle helmets are made of glass fibre reinforced plastic materials that are costly. Replacing or reducing these synthetic fibres with plant fibres would reduce costs and may allow for such materials to be recyclable, biodegradable, and more abundant, as the material has been ground or crunched. Flax woven fibre was used to fabricate one panel of composite (Flax only) and three panels of hybrid composite (FLXC, FLXG, and FLXK). In this project, the epoxy resin was modified by weight with 0 wt.%, 0.5 wt.%, 1 wt.%, 1.5%, and 2 wt.% multi-walled carbon nanotubes (MWCNTs). This study examined the effect of multi-walled carbon nanotube (MWCNT) concentration on the tensile properties of hybrid biocomposites. The experimental results suggested that the MWCNTs played an important role in improving the mechanical performance of hybrid biocomposites. It was found that optimum carbon nanotube (CNT) concentration improved the tensile performance of the materials by 2% to 5%. However, an excess CNT concentration led to the deformation of materials and reduced their mechanical performance.

Keywords: Carbon nanotube; Hybrid biocomposites; Tensile properties; Flax fibre; Glass fibre; Aramid fibre; Carbon fibre

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INTRODUCTION

Glass, carbon, and Kevlar fibre-reinforced polymer composites are the subject of increasing interest in many industries, such as aerospace, athletics, and various others. However, the use of synthetic fibres in the composite industry has created issues, such as the risk of inhalation during fabrication, low renewability, low biodegradability, and low recyclability. Moreover, the costs of synthetic fibres are higher than those of natural fibres. The use of biocomposite fibre to replace synthetic fibre is expanding. Research conducted on biocomposites has revealed the potential of several types of natural fibre, including flax (Jhala and Hall 2010; Yan and Chouw 2013; Dicker *et al*. 2014).

The numerous favourable properties of flax fibres have increased their demand in the composite industry. The great thermal insulation, improved vibration absorption, confirmed renewability, and comparable mechanical properties offered by flax fibres have widened their application in the development of new materials. Previous findings suggest that flax fibre is as stiff as conventional glass fibre, and it is known as the strongest natural fibre (Wambua *et al*. 2007; Yan and Chouw 2013; Pil *et al*. 2016). Moreover, flax composites displayed better energy consumption than jute and hemp composites (Wambua *et al*. 2007; Koronis *et al*. 2013).

In recent years, nanomaterials and their composites, such as carbon nanotube and nanoclay, have drawn a great deal of interest in a wide range of applications due to their excellent chemical, electrical, physical, mechanical, optical, and biological properties (Volder *et al*. 2013). Integrating a nanoconstituent into a polymer composite allows customization and optimisation. A carbon nanotube (CNT) is a nanomaterial that offers outstanding electrical, mechanical, and thermal properties. As a result, the inclusion of CNT in polymers and alumina composites has increased (Schadler *et al*. 1998; Liew *et al*. 2015).

Known to have remarkable physical and chemical properties, the incorporation of CNTs in science and technology has increased tremendously since their introduction in 1991. Carbon nanotubes have the potential to change and improve various areas in material science, and are a major contributor to nanotechnology. Two of the most easily obtainable types of CNTs that have high structural perfection are single-walled CNTs and multi-walled CNTs. Single-walled carbon nanotubes (SWCNT) consist of a single graphene sheet seamlessly wrapped into a cylindrical tube, and multi-walled carbon nanotubes (MWCNTs) comprise of an array of single-walled nanotubes that are concentrically nested, like the rings of a tree trunk.

Carbon nanotubes can be synthesized by a number of methods, such as laser-ablation, chemical vapour deposition (CVD), the substrate method, the sol-gel method, gas phase metal catalyst techniques, arc-discharge techniques, micro fabrication, electrospining, and others (Behabtu *et al.* 2013). The CVD method can control the growth direction on a substrate and synthesize a large quantity of carbon nanotube (Chang *et al.* 2005)

Thermosetting polymers, such as epoxy, vinyl ester, and polyester resins are frequently used as the matrix for hybrid bio-composites (Ku *et al.* 2011). Various research has shown that a sufficient distribution of nanotubes improves the physical performances of composites (Chang 2010). However, it is hard to disperse CNTs completely in resin, therefore, distinct dispersion techniques should be sought.

The introduction of hybrid composites from the combination of at least two types of conventional fibres has expanded the idea of producing hybrid bio-composites, in which one of the component fibres is a natural fibre. As the properties of hybrid composites have the advantages and disadvantages of both component fibres, the perfect combination of fibres can produce a new hybrid material with better properties at a lower cost (John and Thomas 2008). As such, a balance in cost and performance could be achieved through proper material design (The LHCb Collaboration 2003).

Similar to general laminate composites, the performances of hybrid composites can be controlled by several parameters, such as length of fibres, fibre to matrix interfacial bonding, fibre orientation, fibre stacking sequence, fibre to fibre ratio, and the total fibre content in the composites. The strength of the hybrid composite also depends on the failure strain of individual fibres. Optimal hybrid results are obtained when the fibres are highly strain compatible (Aaji *et al.* 2015). In this work, a hybrid biocomposite was selected due to the need to limit the use of non-renewable materials by mixing them with renewable materials. A hybrid biocomposite was also chosen to reduce the pollution and energy costs incurred in production and to create a material that has potential for re-use after its service life or demolition.

Montazeri *et al*. (2010) found that the addition of MWCNTs to plain-weave glass/epoxy composites had a small impact on the tensile properties, which were influenced by the fibre properties. Nonetheless, as the MWCNTs strengthened the matrix-rich portion and the interface between the glass fibres, they also affected the inter-laminar fracture properties that were controlled by the matrix properties. They found that maximum tensile stiffness and strength were achieved with 0.4% MWCNT content.

In an article by Chandrasekaran *et al*. (2011), by making comparison with the 0.5% unfunctionalized MWCNT/epoxy/glass fibre composite, it was proven that the neat epoxy/fibre composite had a 41% higher inter-laminar shear strength. Bensadoun *et al*. (2016) stated in their article that by adding MWCNT to epoxy/fibre-reinforced polymer (FRP) laminate composites, the tensile strength and flexural strength increased. With increasing MWCNT content, the resulting composites demonstrated a minor increase in tensile strength.

Carbon nanotube addition has been found to enhance the inter-laminar fracture toughness of fibre reinforced plastics (Karapappas *et al*. 2009). However, there is a lack of research on the mechanism of the impact of the CNT on the mechanical properties. According to previous studies, an optimised CNT concentration with distinctive dispersion methods is needed to obtain optimal mechanical properties. Thus, a method of dispersion using acetone was standardised in this work and the percentage of CNT was varied in order to identify the optimum percentage of added CNT.

EXPERIMENTAL

Materials

Table 1 details the mechanical properties of four types of fibres; flax, glass, carbon, and aramid fibres, which were used to fabricate 300 mm × 300 mm panels. The woven carbon fibre, E-200 glass fibre, and flax fibre used were Flaxply BL1350 by Lineo NV (Saint Martin Du Tilleul, France) with a balanced weave. A 150 g flax/m^2 and aramid fibre was used with a plain-woven hexcel structure (Style 706 Kevlar KM-2, 600 denier, supplied by DuPont Kevlar, Ashtabula, OH, USA) with a real density of 180 g/m^2.

Table 1. Mechanical Properties of Flax, Glass, Carbon, and Kevlar Fibre (Salit *et al*. n.d.)

Fibre	Type	Strength; σ (GPa)	Failure Strain; $\mathcal{E}$ (%)	Modulus; E (GPa)
Flax	Natural	3.53	-	72
E-Glass	Synthetic	3.8	1.76	227
Carbon	Synthetic	3.5	4.7	74
Kevlar	Synthetic	3.4	3.55	82.6

The MWCNTs were produced by a state-of-the-art proprietary carbon vapour deposition (CVD) process, providing the highest quality MWCNT available. The diameters ranged from 12 nm to 15 nm, and they varied from 3 µm to 15 µm in length. Figure 1 shows the structure of a 12 to 15 nm diameter MWCNT under transmission electron microscopy (TEM), and Table 2 illustrates the specifications of MWCNTs for this work. The acetone solution used as a dispersing agent was dimethyl ketone (2-propanone), which was obtained from Friendemann Schmidt Chemical (Parkwood, Australia).

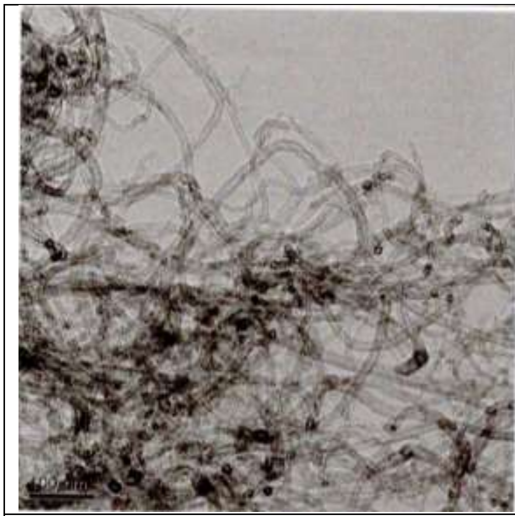

Fig. 1. TEM of 12 nm to 15 nm MWCNT

Available Diameters
12 nm to 15 nm
Available Lengths
3 µm to 15 µm

Table 2. Details of Multi-walled Carbon Nanotube

Properties	Specification	Test Method
Purity	> 97%	TEM
Amorphous Carbon	< 2%	TEM
Ash	< 0.2 wt.%	TEM
Specific Surface Area	230 m^2/g to 300 m^2/g	BET
pH Value	7 to 8	-
Layer	8 to 15	TEM

The matrix used was EpoxAmite 100 (Smooth-on, supplied by Mecha Solve Engineering, Selangor, Malaysia), which was cured with 102 Medium Hardener hardener (supplied by Mecha Solve Engineering, Selangor, Malaysia). The actual composition of the epoxy resin used was Diglycidyl Ether of Bisphenol A (DGEBA), while the chemical name of hardener used is polypropytriamine. The physical properties of the EpoxAmite 100 and 102 hardener combination provided by the manufacturer are shown in Table 3. Epoxy resin was used due to its lightweight properties, the minimal damage caused to the manufacturing equipment, and its better mechanical properties than other resins.

Table 3. Physical Properties of Combination of EpoxAmite 100 and 102 Hardener

Physical Properties	PSI	Pa
Flexural strength; ASTM D790 (2002)	12,220	84.25×10^6
Flexural modulus; ASTM D790 (2002)	423,000	2.91×10^9
Ultimate tensile strength; ASTM D638 (2004)	8,180	56.4×10^6
Tensile modulus; ASTM D638 (2004)	450,000	3.1×10^9

Fabrication Methods

To well disperse MWCNTs in the epoxy, an enhanced method was well-established as a standard process throughout the study. First, MWCNTs were dissolved for 30 min in concentrated acetone with a mass a ratio of 1:50. Acetone is the best solvent for dispersing nanotubes into epoxy-based composites, as the use of DMF and ethanol would influence the mechanical performance of the composites during the pre-curing process (Lau *et al.* 2005). Subsequently, the mixture was homogenised for 30 min at 7,500 rpm. Ma *et al.* (2010) and Zhang *et al.* (2016) both stated that higher shear forces are needed, in the range of 5,000 rpm to 15,000 rpm, in order to achieve a fine dispersion of the polymer matrix and also to reduce agglomeration. A further 30 min of homogenisation was required after adding the resin to the dispersed solution. Finally, a mechanical stirrer was used to combine the mixture for 4 h at room temperature. The hardener- and MWCNT-laced resin solutions were then combined. The mixture was gradually stirred by hand before starting the lay-up process to reduce the formation of air bubbles in the mixture. A roller was used to reduce the presence of air bubbles in the composite laminate.

A pair of steel plates was used to fabricate sample panels of 300 mm × 300 mm. Each panel consisted of 6 layers of fibres. A total of 20 panels were fabricated in this work, including 5 panels of flax fibre, 5 panels of flax + glass hybrid, 5 panels of flax + carbon, and 5 panels of flax + aramid with alternating arrangements of the fibres (*i.e.*, flax/carbon/flax/carbon/flax/carbon as shown in Fig. 2). For each group of fibre/hybrid fibre composites, different concentrations of MWCNT were used in each panel. The laminate was cured under high pressure for 24 h at ambient temperature.

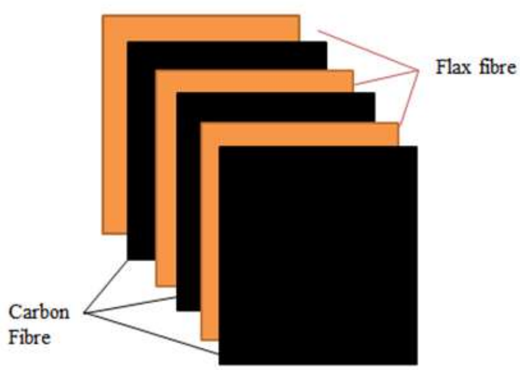

Fig. 2. Arrangement of fabricated hybrid biocomposite

Methods

The stress-strain relationship of the developed hybrid biocomposites was identified through tensile testing. The tests were conducted using an INSTRON 3366 device (Instron, Norwood, MA) with Bluehill software (Instron, Bluehill3, Norwood, MA). The test was conducted in accordance with ASTM D3039 (2014) with a plate size of 200 mm × 25 mm and 2.0 m to 3.0 m sample thickness for each composite. The samples were carefully cut from the laminate using a vertical band saw machine (Makita, Sri Kembangan, Selangor, Malaysia) and trimmed to the appropriate size. A grip pressure of 500 psi was applied to the samples. A standard head displacement at a speed of 1 mm/min was applied. For each sample, five specimens were tested and the average results were obtained.

Field emission scanning electron microscopy (FESEM, FEI Company, Model: Nova NanoSEM 30 Series) was carried out to identify the dispersion of multi-walled carbon nanotubes, the effects of the carbon nanotubes on the epoxy-fibre bonds, and the failure mechanism of the samples. In the specimen notation “#%/FLX”, #% represents the percentage of MWCNTs by weight modified epoxy resins, while FLX represent flax composites. FLXC represents the hybrid composite of flax and carbon, FLXG the hybrid composite of flax and glass, and FLXK the flax and Kevlar hybrid composite.

Table 4. Fibre Volume Fraction of Samples with Different CNT Contents

Sample code	CNT content (%)	Fibre volume fraction
0%/FLX	0	0.312
0.5%/FLX	0.5	0.317
1.0%/FLX	1.0	0.322
1.5%/FLX	1.5	0.323
2.0%/FLX	2.0	0.321
0%/FLXC	0	0.273
0.5%/FLXC	0.5	0.278
1.0%/FLXC	1.0	0.285
1.5%/FLXC	1.5	0.293
2.0%/FLXC	2.0	0.302
0%/FLXG	0	0.320
0.5%/FLXG	0.5	0.329
1.0%/FLXG	1.0	0.337
1.5%/FLXG	1.5	0.338
2.0%/FLXG	2.0	0.327
0%/FLXK	0	0.318
0.5%/FLXK	0.5	0.322
1.0%/FLXK	1.0	0.326
1.5%/FLXK	1.5	0.334
2.0%/FLXK	2.0	0.333

RESULTS AND DISCUSSION

Tensile Behaviour

The tensile properties are illustrated in Figs. 4a through 4g. Figures 4a through 4d show the typical tensile stress-strain curves of CNT-FLX, CNT-FLXG, CNT-FLXC, and CNT-FLXK hybrids, respectively. These curves indicate the ultimate tensile strengths at the maximum points and the tensile moduli of the curve gradients. Tensile modulus and strength represent the ability of a material to resist tensile deformation. The results for tensile strength and tensile modulus are clearly shown in Figs. 4e and 4f.

Figure 4e shows the impact of CNT concentration on the ultimate tensile strength (σ_t^{max}). For flax, 0%/FLX exhibited the highest σ_t^{max}, followed by 1%/FLX, 0.5%/FLX, 1.5%/FLX, and 2%/FLX. Next, for the flax and glass hybrid, 0%/FLXG exhibited the highest σ_t^{max}, followed by 1%/FLXG, 1.5%/FLXG, 2%/FLXG, and 0.5%/FLXG. For the flax and carbon hybrid, the highest σ_t^{max} was obtained from 1%/FLXC with 340.13 MPa, followed by 0%/FLXC, 0.5%/FLXC, 1.5%/FLXC, and 2%FLXC. For the flax and aramid hybrid, 1%/FLXK exhibited the highest σ_t^{max}, followed by 0%/FLXK, 1.5%/FLXK, 2%/FLXK, and 0.5%/FLXK.

As depicted in Fig. 3, the scattering of CNTs through the epoxy proves that the CNT fully dispersed in the epoxy resin, and the uneven fracture surface of FLXC with CNT proves that the CNT prevented crack propagation and bypasses the crack. Besides, the gap shown in Fig.4 shows that inclusion of CNT improved the interfacial bond between the epoxy resin and the fibre as the gap was reduced at 1%/FLXC compared with 0%/FLXC. However few voids start to exist at 1% of CNT concentration, and CNT agglomeration was obviously seen on the 2%/FLXC fractured surface. Agglomeration of CNTs will weaken the interfacial properties between the resin and the fibre and manifest as deformities in the composite. Moreover, the viscosity of the epoxy increased with increasing CNT content, resulting in poor wetting behaviour of the epoxy during the lay-up. Increased viscosity also increases the number of entrapped air voids (Zhang *et al.* 2014) in the composite, as shown in Fig. 3.

Figure 4f shows the effect of CNT concentration on tensile modulus (E_t) of all the tested composites. All composites showed a smooth decreasing trend as the CNT concentration increased. However, for the FLXG and FLXK hybrids, there were slight increases in value at 1%/FLXG, 2%/FLXG, and 1.5%/FLXK.

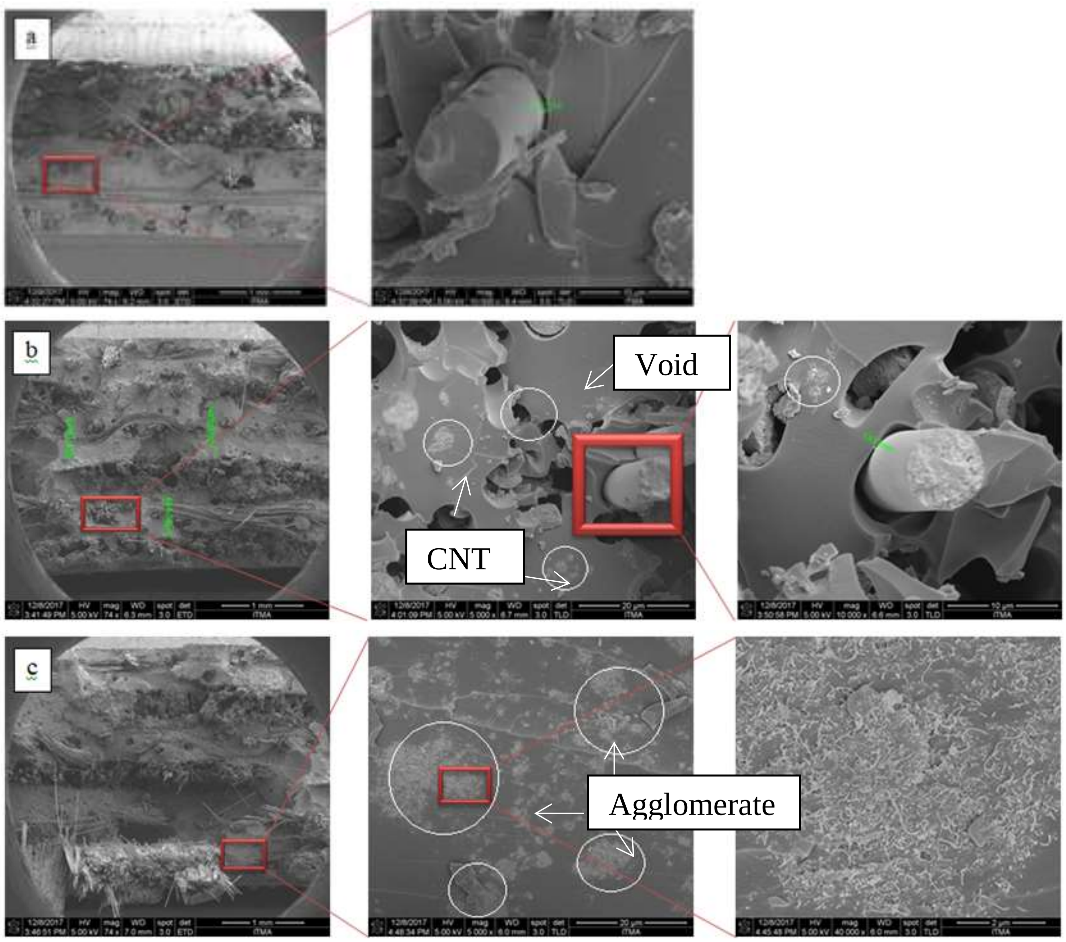

Fig. 3. FESEM images of the fracture area of (a) 0%/FLXC, (b) 1%/FLXC, and (c) 2%/FLXC

Figure 4g shows that the CNT-modified hybrid biocomposite exhibited high failure strain on FLX and the FLXK hybrid, but low failure strain for the FLXG and FLXC hybrids. The graphs of failure strain percentage for FLX and the FLXK hybrid showed the same increasing trend at a certain percentage of CNT, then started to decrease. For the FLXG and FLXC hybrid composites, lower percentages of failure strain were obtained at

0.5% CNT concentration than 0% CNT concentration. There were slight increases at 1% CNT concentration, and decreases at 1.5% and 2% CNT concentration.

Trends in Fig. 4e, which shows the tensile strength of FLX and FLXG hybrid, display increases from 0.5% CNT concentration to 1% CNT concentration, and decreases from 1.5% CNT concentration to 2% CNT concentration. However, the value of tensile strength for FLX and the FLXG hybrid at 1% was slightly lower than that of the 0% CNT concentration. According to (Zhang *et al.* 2016), inclusion of CNT in composites does not enhance the tensile strength, but it improves the fracture work and failure strain of glass fibre, which showed a similar trend to that of the tensile test result. However, in this work a different result was observed, in which the optimum results were obtained at 1% CNT concentration, where the value of tensile strength for the FLXC and FLXK hybrids increased. This shows that a suitable CNT concentration may improve the tensile performance of certain materials. This was because a higher quantity of CNT increased the viscosity of the matrix, which made it harder for the matrix to be absorbed into the fibre.

The failure strain slightly decreased with an increasing amount of CNT for all specimens. This was due to the increasing concentration of CNT contributing to CNT agglomerations and void content. As the CNT concentration increased, it led to an excessively viscous mixture that did not wet and impregnate the fibres. Hence, if the composite was subjected to an in-plane displacement, stresses would not be effectively transferred to the fibres and the overall structural performance of the composite would be compromised (Tehrani *et al.* 2013). According to (Gojny *et al.* 2004), low CNT content increases the failure strain of composites and high CNT content decreases failure strain. This was one of the main reasons why the manual lay-up fabrication method was chosen instead of the vacuum diffusion method.

In general, the addition of CNT influenced the tensile properties of hybrid biocomposites, and the optimum CNT concentration enhanced the interfacial properties between the epoxy resin and the fibre. The FLXC and FLXK hybrids showed positive results in tensile strength and fracture, which are the fundamental properties of material selection for application in helmet shells.

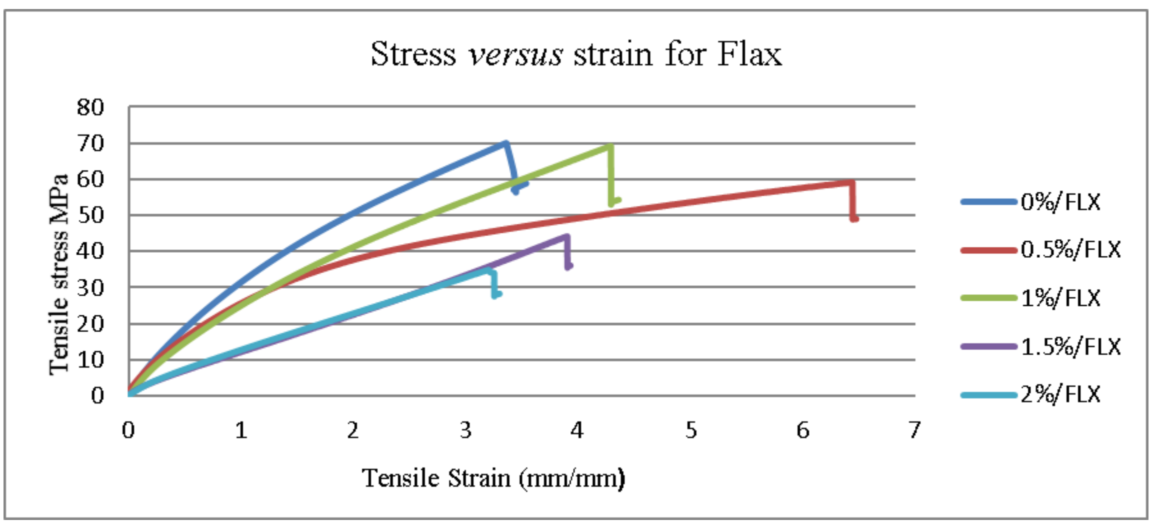

Fig. 4a. Stress-strain curves for flax with all five respective percentages of CNT

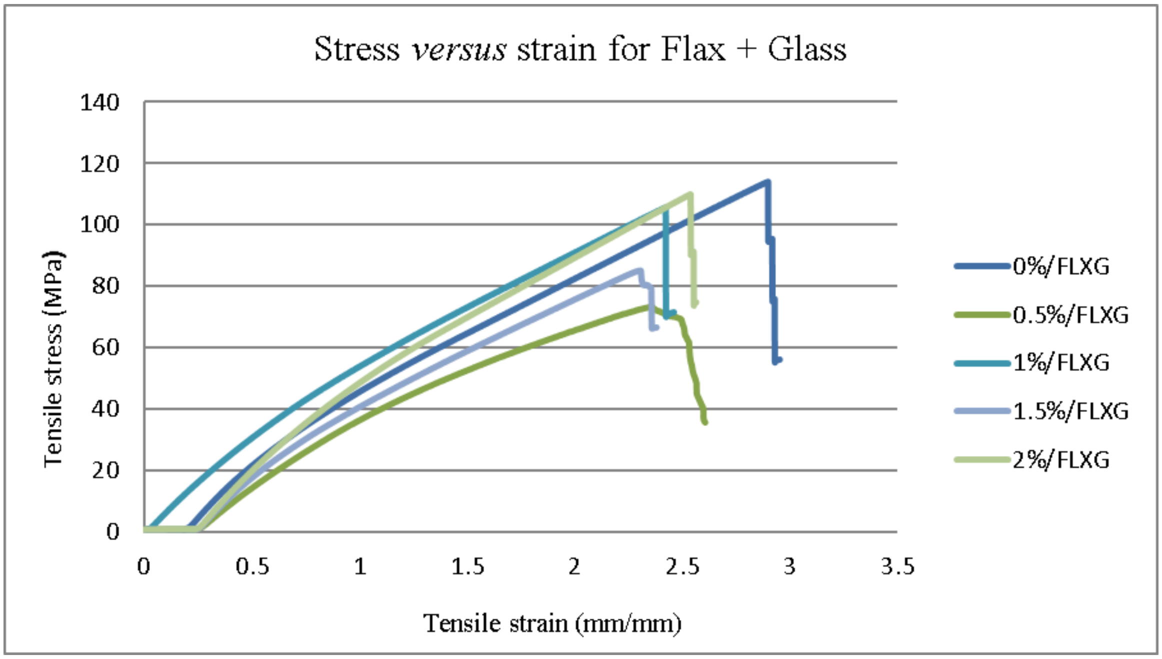

Fig. 4b. Stress-strain curves for flax + glass hybrid with all five respective percentages of CNT

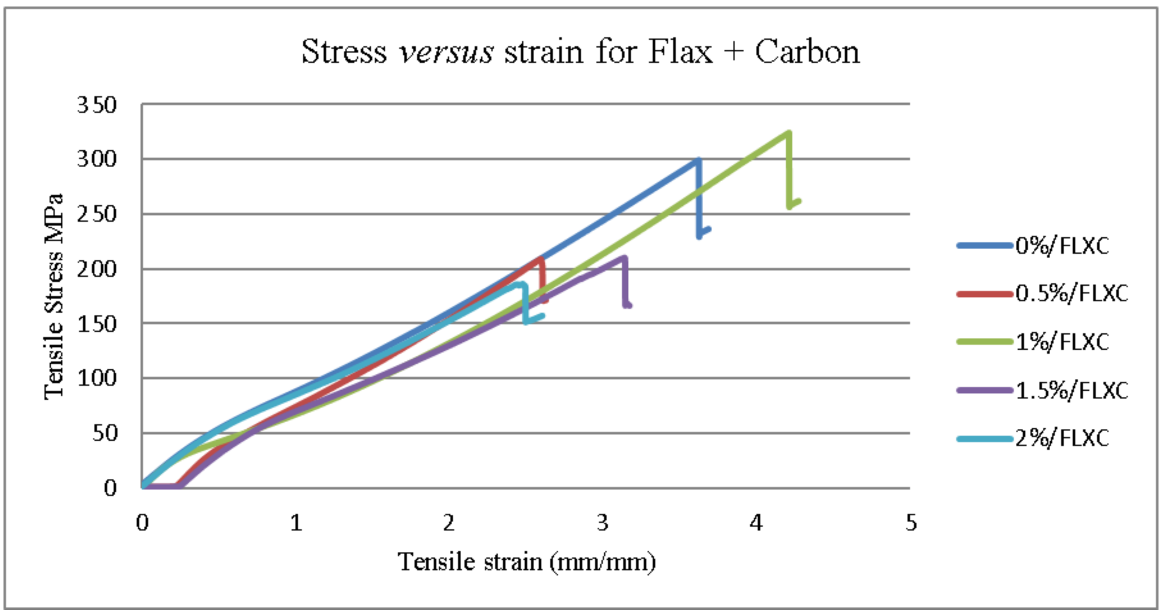

Fig. 4c. Stress-strain curves for flax + carbon hybrid with all respective percentages of CNT

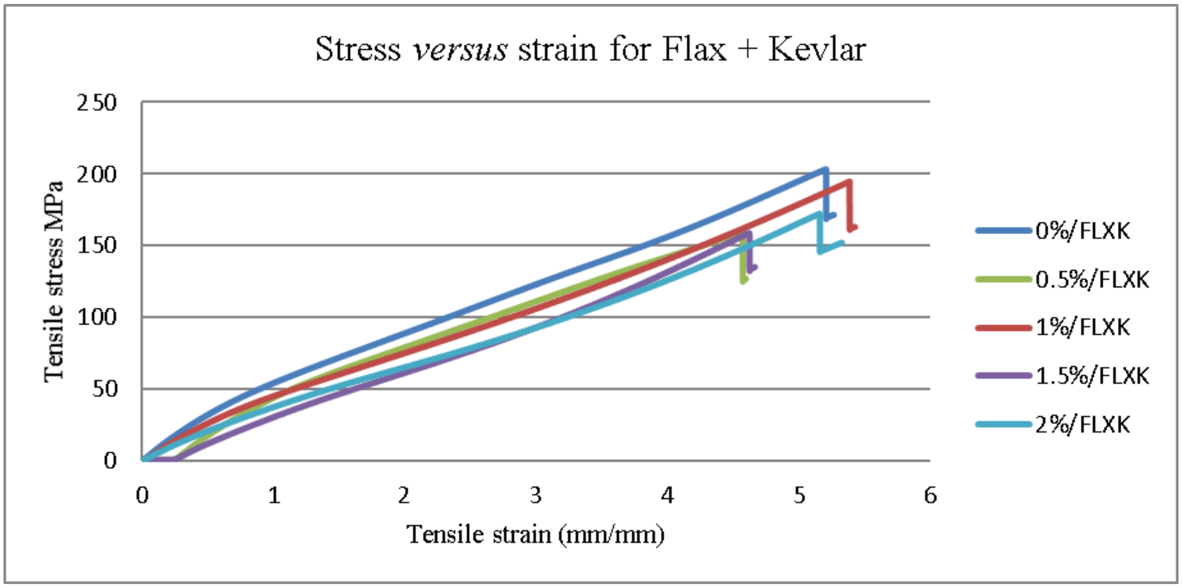

Fig. 4d. Stress-strain curves for flax + aramid hybrid with all five respective percentages of CNT

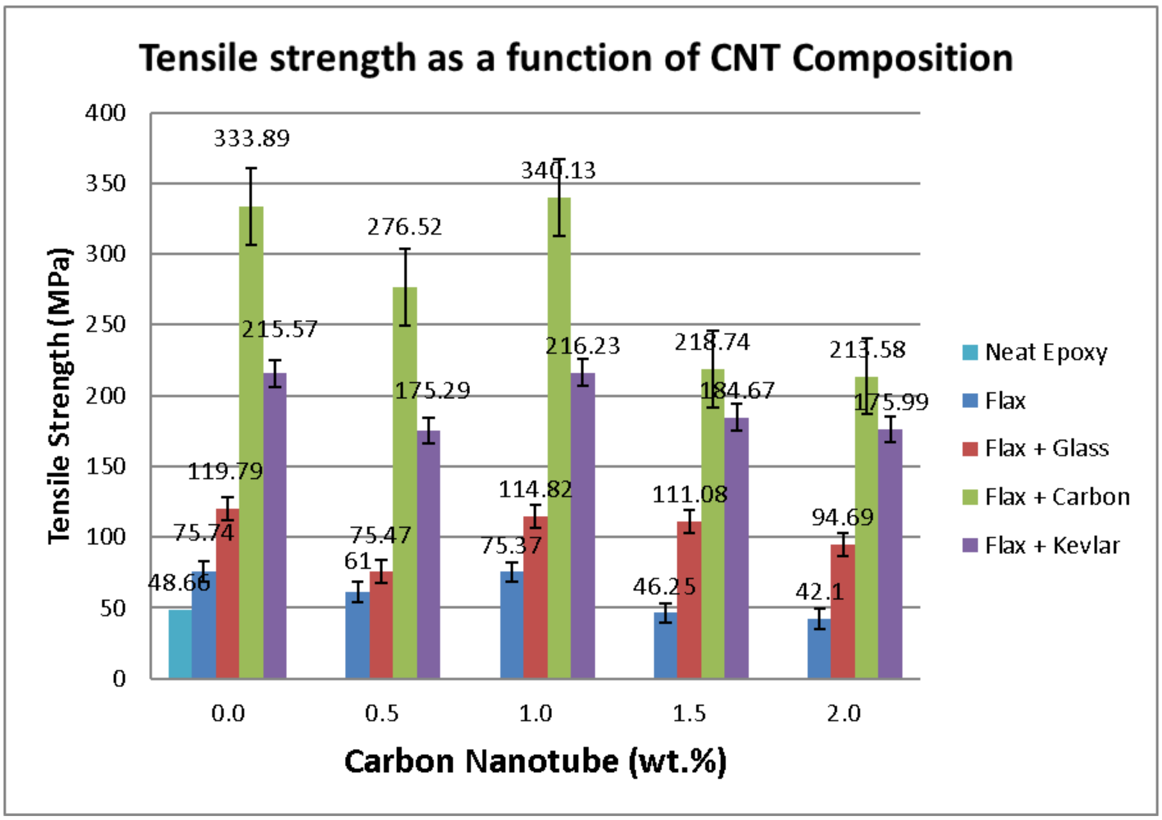

Fig. 4e. Ultimate tensile strength

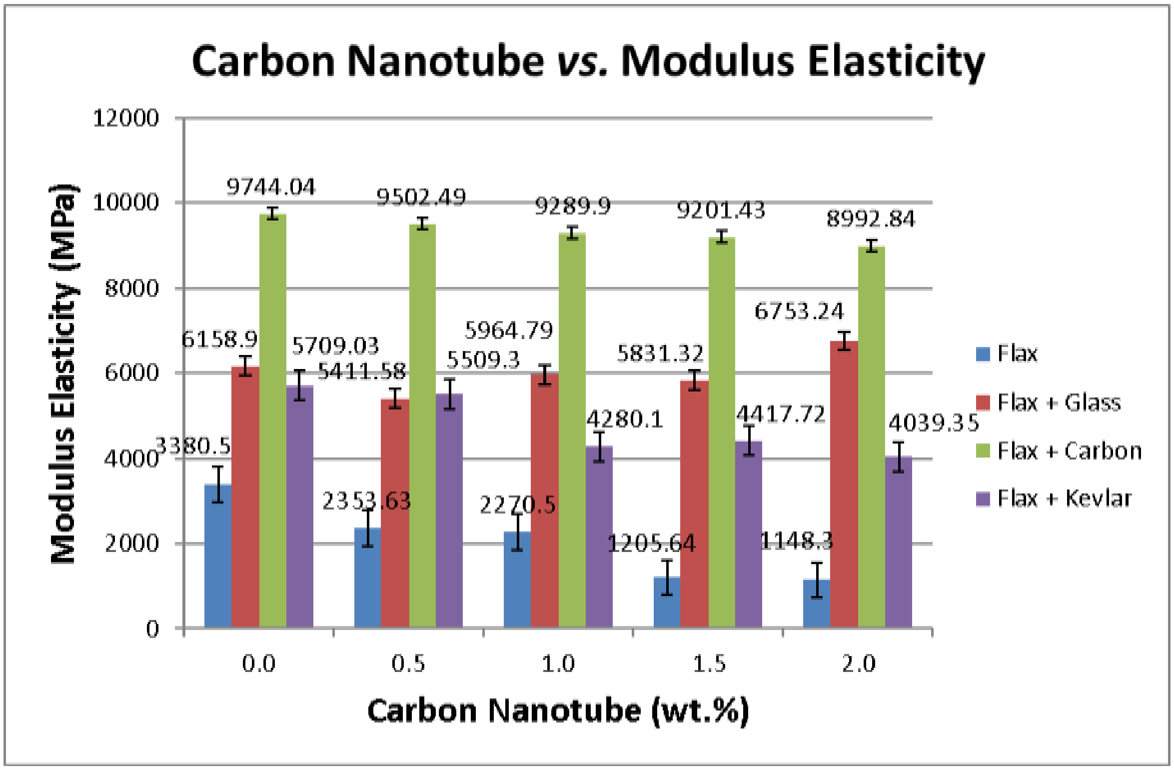

Fig. 4f. Tensile modulus of elasticity

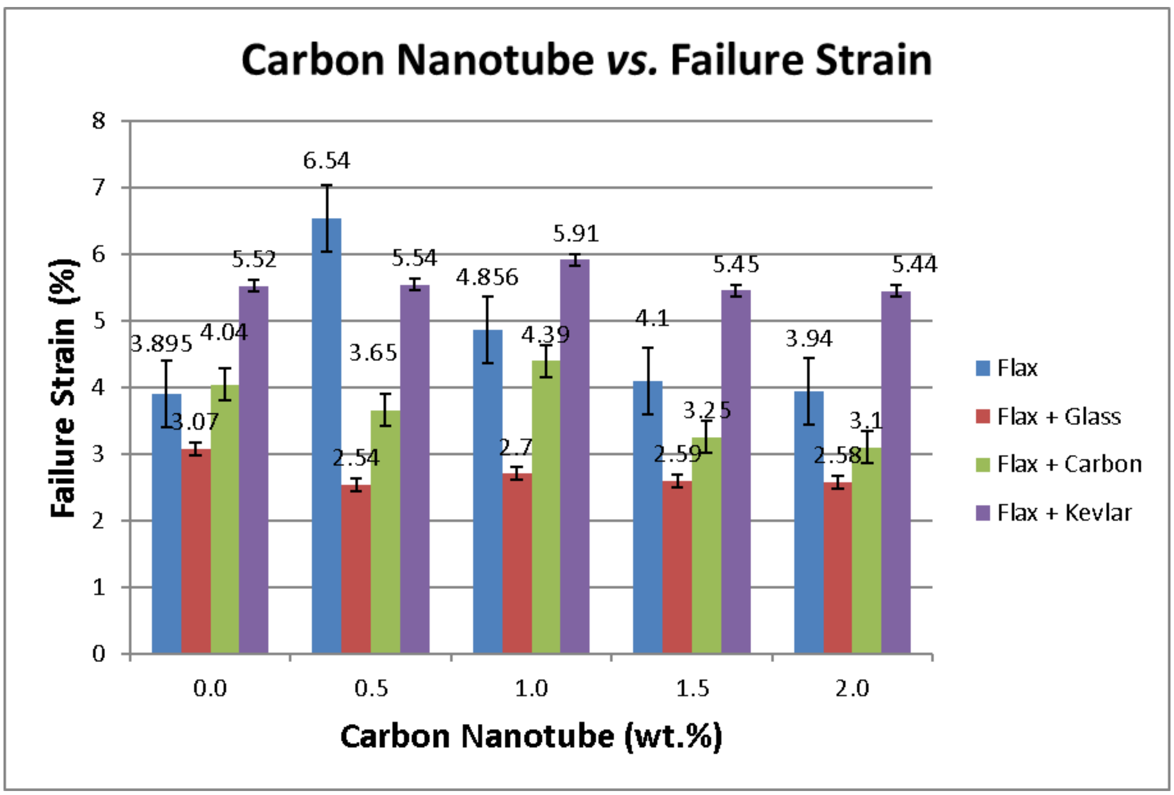

Fig. 4g. Failure strain

CONCLUSIONS

1. This study investigated the effects of multi-walled carbon nanotubes (MWCNT) concentration on the tensile properties of fabricated hybrid biocomposites.
2. The results suggested that the tensile properties improved drastically at 1% concentration of the carbon nanotubes.
3. From this work, it can be concluded that the hybrid composites of flax and carbon (FLXC) and that from flax and Kevlar (FLXK) had favorable characteristics. However, the FLXC hybrid was the best combination of materials in terms of tensile properties, and was the most suitable to be used as a new material for environmentally friendly helmet shells.

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