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Effect of multi-walled CNTs polyurethane mats lamination with basalt fabrics reinforced-epoxy composites reviewed on tension and bending properties

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Abstract

Material technology continues to develop with various innovations and engineering to improve weaknesses in both mechanical and physical properties. In this study, electrospun fibres containing a multi-wall blend of CNT and Polyurethane (PU) with or without surfactant that laminated into a basalt fibre-reinforced composite were uniquely demonstrated. Multi-wall CNT 3wt% was added to the PU/MEK/DMF solution and produced using an electrospinning process. PU fibre mat containing 3wt% CNT was made without and with surfactants. Also, Basalt fibre reinforced epoxy composite as a control sample was produced. In addition, vacuum-assisted resin transfer printing has been used in the manufacture of composite panels containing both fibres. The aim of combining basalt fibre and PU CNT spun mats was to investigate their effect on the tensile and flexural mechanical properties. Tensile and flexural tests were carried out on a universal testing machine (UTM) in accordance to ASTM D 638 and ASTM D790 standards. FESEM and TEM on composite morphology test were done after testing. The results indicated that the basal matting fibre-reinforced epoxy composites stacked by PU mats with or without surfactants were affected by CNT inclusions. Nanofiber spun mats laminated in a basalt fibre composite lead to a considerable increase in both loads (i.e. tensile and flexural properties). The highest tensile and flexural load values occurred in the BF+PU-mat-2 sample with triton-x 100 surfactants compared to BFRP. The increase in tensile and flexural modulus values was at 13% and 17.3%, respectively. On the other hand, there was a decrease in shear failure due to tensile and bending loads due to the brittleness of the composite reinforcement. In conclusion, this CNF-mat lamination is highly suitable to be used to improve the strength properties of BFRP composites. It is highly recommended for automotive parts, marine compartments and storage insulation.

Keywords: Electrospinning; polyurethane; basalt fabric; stacking

1. Introduction

Engineering materials continue to be developed to achieve characteristics such as high stress, long deflection, lightweight, corrosion resistance, and environmentally friendly for future needs [1]. Fibre Reinforced Polymer (FRP) composite is non-metal material [2] that has received much attention and is designated as modern material. It is unique as it could be made from two different phases of material. Several advantages such as the behaviour of mechanical, chemical, physical, and economist, as well as environmental have been explored by FRP composites compared to metal [3–5]. However, it depends on the adhesion and cohesion force between them [6]. Thus, it becomes interesting to do more studies based upon the physical force against the FRP structures.

During the last two decades, the application of organic reinforcement has been powerful attention by the government in composite material products. This pressure is a part of the green products programme. To realize the target, a new class of reinforcement, i.e. basalt fibre (BF) was introduced [7–9].

BF is a unique reinforcement sourced from the volcano rock. As a fibre, it is produced by the melting process of the volcano rock [10] at a high temperature of around 1700ºC as shown in Figure 1 [11]. The important compound of BF is 64% of SiO_2, and 40% is shared with other compounds such as Fe_2O, Mg, and Al. Advantageously, BF is non-toxic, non-flammable and explosion-proof [9]. When being in contact with other chemicals, it does not produce ions, and chemical reactions that can damage health or the environment [12]. In addition, basalt has good hardness and thermal properties [13]. Several studies for behaviour of BF in composite structures have been conducted by [13–15]. The presence of cellulose, hemicellulose, pectates, pigments and waxes in natural fibres affects the binding capacity of the matrix; thereby, it reduces strength and increases delamination under tension and bending. Furthermore, the efforts that have been made so far are to increase the binding capacity of natural fibres, in this case, by combining BF [16] with other fibres [17]. Currently, the combination of Nano-sized materials has been widely used in

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an effort to increase the bond matrix and natural reinforcement [18]. This is done because the Nano-sized material is easily absorbed by the fibres. In addition, the carbon nanotubes [19] have an extraordinary influence on both the reinforcing and/or matrix, which is the potential to improve the mechanical, physics, electrical, and thermal properties of the composite materials [20–23]. Many scientists have done this method by mixing nanoparticles into the matrix. [24–27]. Generally, the application of CNTs is added into the polymer to improve the matrix ability in transfer load together with the fibres [20,21,25,28–34]. Based on the previous results of studies, the Nano-Carbon-Fibre (CNF) lamination method has not been commonly used to improve composite properties. This research has focused on the role of CNF-mat made through the electrospinning process.

We studied the effect of CNF-mat laminated with basalt fibres as the reinforcement of composites. Their mechanical properties would be observed under tensile and flexural loads. The CNF-mat was produced through an electrospinning process and by the mixtures of Polyurethane + DMF/MEK + 3wt% CNT with and without surfactant Triton-X-100 and epoxy resin as substrate. This research aims to observe the performance of CNF-mat against the mechanical properties of composites epoxy with basalt fibre reinforced under the tensile and flexural load. The CNT-mat in all variations was characterized in accordance to FESEM and TEM analysis.

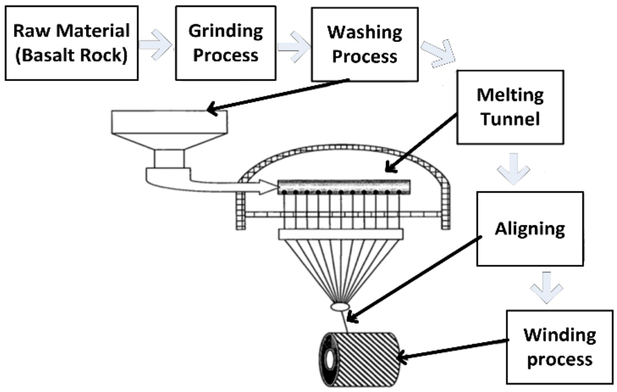

Fig. 1. Process manufacturing BF

2. Materials and Methods

2.1. Materials

CNF has prepared through electrospinning method as shown in Figure 2. Prior to electrospinning, we prepared the CNF solutions with a CNT concentration of 3 wt.% according to the weight of PU added to the mats PU solutions. Before mixing in the PU solutions, the CNTs in DMF/MEK were bath-sonicated (40Hz, Mujigae, Korea) for an hour. Then, mat PU (10 wt.%) solutions were mixed in the DMF/MEK in the ratio of 50:50 wt.% by overnight stirring. Next solution containing 3 wt.% CNT/DMF/MEK solution with a surfactant Triton-X in the ratio of 100:20 (CNT: Triton-X, wt./wt.%) was also produced to investigate the effect of surfactant on the CNT spread and its subsequent effect on the mechanical property enhancement of the composite.

2.2 Manufacture process

Figure 2 shows the electrospinning process carried out in the set-up. Electrospinning is a widely developed approach to producing polymer fibers with Nano-sized diameters [35–38] into ultra-thin layers. The morphology and diameter of the electrospinning fiber were significantly affected by temperature [35], and concentration [26,39,40]. In this work, a high voltage of 15 kV was employed to inject electrospinning solutions in a plastic syringe at a feed rate of 1 mL/h. The distance of the collector (flat Aluminum) was covered with basalt fabrics (250 mm x 250 mm) and the needle tip was kept constant at 150 mm. In addition, the humidity chamber was kept at 30% using the seal. In the last electrospinning process, the nanofiber laminates on basalt fiber were dried at 60ºC for two days inside an oven to remove any residual solvent.

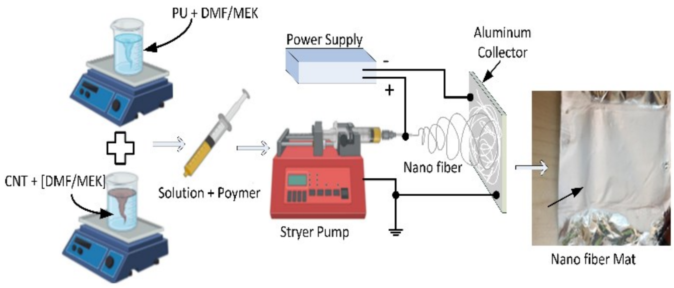

Fig. 2. Schematic representation electrospinning processes a. materials, and b. electrospinning setup

Table 1. Laminate variation and concentrations CNT

specimens	Laminate configurations
BFRE	BF/Epoxy
PU mat-1	BF+Spun (3 wt% CNT)/Epoxy
PU mat-2	BF+Spun (3wt% CNT+Triton-X) + Epoxy

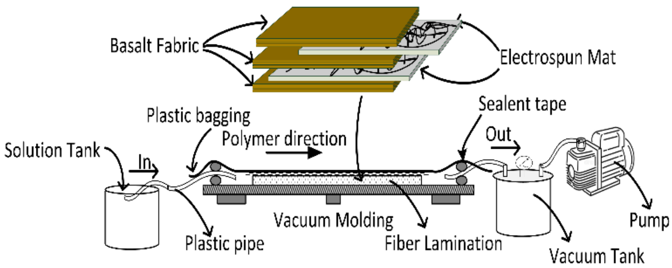

Fig. 3. Schematic of vacuum resin transfer injections moulding process

The BFRP laminated with CNF spun mats were fabricated through the VRTM as exhibited in Figure 3 [5]. Meanwhile, Table 1 shows the stacking sequence mode between BF and CNF PU mats. The mass ratio of the epoxy resin versus the hardener was conducted at 100:20 wt%. During the process, six layers of basalt fibers and four layers of CNF mats (with the size 250 x 250 cm, respectively) were employed and the vacuum of -0.8 kPa was maintained. Then, it was cured inside an oven with the temperature at 65ºC for 2 hours [41]. The BFRP containing six BF was also fabricated and evaluated as a control.

2.2. Measurement

The tensile test was used widely for testing the strength and strain of samples in a unidirectional manner. The ASTM D-638 standard was reflected for each sample's geometry. In this work, the testing of the samples was conducted on the Universal Testing machine (UTM) (Unitect-M brand R&B)

with a crosshead speed of 3 mm/min and load cells of 2 tons. In addition, an extensometer (Epsilon Tech. Corp. Model 3542) [42] was installed on the sample to measure the elongation values occurred during the tensile testing. In this test, to find consistency values, each variation of the samples was repeated 5 times.

A three-point bending is one flexural test manner of materials. This testing was conducted by placing the material on two supports on each end of the beam. The beam deflection was analysed statically on a central loaded. The BFRE-CNF sample geometry refers to standard ASTM D-790. This test used a similar machine and treatment the tensile testing where each sample was tested with five repetitions.

In the advanced study of material Nano size can usually be detected using the Nano Field Emission Scanning Electron Microscope (FESEM), Scanning Electron Microscopy (SEM), and Transmission Electron Microscopy (TEM). This is a non-destructive method for providing detailed information on the morphology, composition, and structure of materials [16]. It is utilized to find the good characteristics of materials regarding the influence of nanofiber laminated with microfiber on mechanical and physical properties. It has been conducted on a laminate cross-section of composite broken after tensile and flexural loads. The results FESEM and SEM analysis of each sample are shown in Figure 4 and 5, respectively.

3. Results and Discussion

3.1. Fiber characterization

Figure 4 illustrates Raman Spectroscopy (RS) of CNTs in PU nanofibres. There are two prominent peaks as a feature of CNT with a Raman shift of 1320 cm^{-1} and 1577 cm^{-1}. The D-band and the G-band are two peaks related to the defect and the plane vibration of the CNTs, respectively. As seen, the peaks of CNP-NF have neater than those of neat PU nanofibres. However, due to the interaction between PU and CNTs, new hydrogen has been generated, which causes a shift in peak.

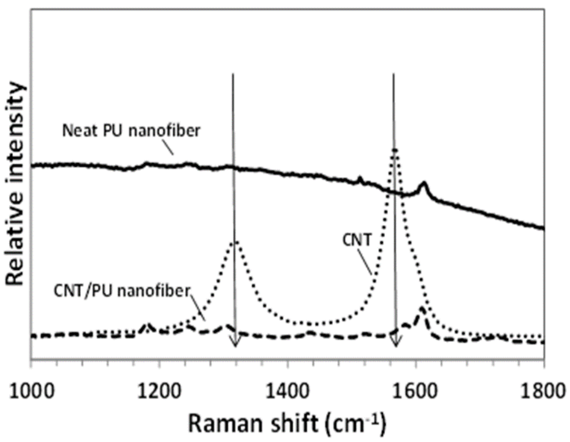

Fig. 4. Raman spectroscopy of neat PU nanofibre, CNTs, and CNP nanofibre

Furthermore, in the microscopic studies by FESEM and TEM observations, it can be seen that the CNTs of 11 Nm diameter was smaller than the diameter of the electrospinning CNP fibre. Thus, CNTs fibre was very well used as a filler in the structure of composites as it could bind well fibres. This increased the characteristics of the composite more than the previous characteristic because CNTs could distribute the loads to the polymer well and evenly. The results showed that the addition of triton-X surfactant (CNTs+Triton-X) could help to distribute CNTs more evenly, compared to those without surfactant, which could form agglomeration [43]. Due to the agglomeration phenomenon in the dispersions of CNTs, it is possible to degrade the mechanical properties of polymeric composites without surfactant [44]. Thereby, nanoparticle dispersion in the polymer matrix is functionally very important to carry out.

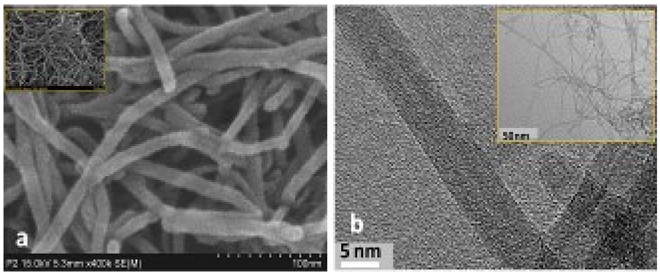

Fig. 5. Characteristics of MWCNT images analysed through; (a) FE-SEM, (b) TEM

Figure 5(a) and 5(b) show the CNF morphology containing 3wt% CNT with and without surfactant. The CNF fibre weave has a white colour with a smooth surface. As observed, the CNF weave was white with a smooth surface. However, the pores appeared (red arrows) in the structure with solid cylindrical fibre shapes. Also, Figure 6 (a-c) show agglomeration occurred with increasing CNT concentration (yellow arrow), if without surfactant, and they formed nanofibre knots bonded together (blue arrow). Previous studies [21,23,45,46] explained that the fewer beads formed would affect the mechanical properties of CNF. However, the thermal conductivity of CNF as well as the electrical properties of nanofibre increased [47].

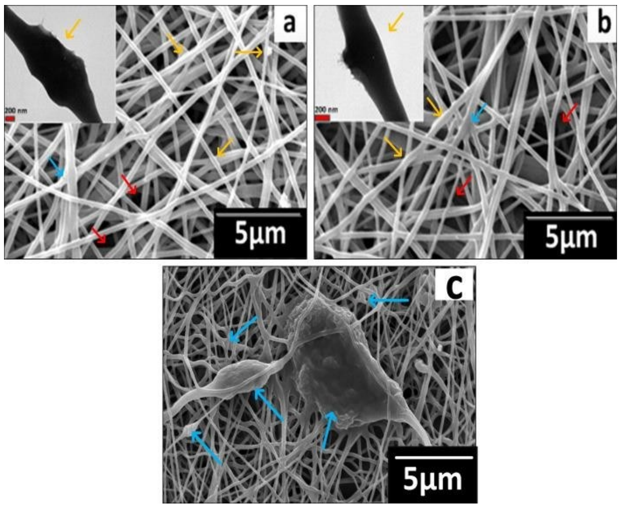

Fig. 6. FESEM and TEM of electrospun mat nanofiber containing a) 3wt % CNT, b) 3wt % CNT+Triton-X

3.2. Mechanical properties

The mechanical properties of CNF-mats were carried out according to variations planned in the present study. Figure 7 (a-d) show the tensile and flexural test of the sample containing 3wt% CNF mats with/without surfactant. Figure 7(a) shows the load-displacement curves of tensile load and load–displacement curve of flexural shown in Figure 7(b). Then, Figure 7(c) illustrates the strength average value comparison between the tensile and flexural loads. For the modulus of elasticity, average values between tensile and flexural loads are shown in Figure 7(d).

As seen in Figure 7(a), a linear increase occurred in tensile load for each sample. In this case, PU contained CNT 0.3 wt.% with surfactant Trinitron – X that had the highest value in strength. However, they had the lower displacement rates. It showed that the material became more brittle.. This result was also declared in experiments by several material scientists [48,49]. Hereinafter, a similar characteristic is also shown for flexural loads (see Figure 7(b)). This phenomenon further described that the proper mixing of CNTs and the presence of surfactants could help to spread the CNTs evenly, thereby improving the characteristics of the composite material [50]. These results were are also determined by the characteristics of the CNF-PU nanofiber mats as shown in Figure 4, 5, and 6.

Figure 7(c) shows the characteristics in which, due to tensile and flexural loading, there was a linear increase. As we observed, the addition of nanofiber with the characteristics shown in Figure (4-6), had a positive effect on the tensile and flexural loads. It can be seen that the tensile stress had a higher value than the flexural strength for each of sample types. The highest tensile strength value was shown by PU-Mat-2 (BF + 3wt% CNF + surfactant) compared to PU-Mat-1 and BFRP. The average values of tensile at the break of the samples were 415.30 MPa, PU-mat-1; 435.74 MPa, and PU-mat-2; 467.30 MPa, respectively. The difference number of tensile strengths PU Mat-2 to other configurations was 11.35% approximately. The overall variation of CNF-PU laminates increased for tensile strength was 4% and 12%, respectively to BFRE. On the other hand, triton-X surfactant was effectively caused by improving tensile strength. It had some arguments similar to the previous studies by [16] and [51]. Furthermore, similar phenomena for the flexural testing result of each sample type shown in Figure 7(c) are PU-mat-1 (378.1 MPa), and PU mat-2 (444.09 MPa) compared to BFRP (378.7 MPa). Also, the average values the flexural of samples were 15.87 GPa, PU-Mat-1 17.94, and PU Mat-2; 19.20GPa, respectively. According to the testing results, the flexural strength linearly increased for every concentration of CNT-PU mat laminated with BF of 10.6 % and 17.3 % to the BFRE composites, respectively. Also, the flexural strength linearly increased for every concentration of CNT-PU mat laminated with BF of 10.6 % and 17.3 % to the BFRE composites, respectively.

Figure 7(d) shows the modulus of elasticity comparison of BFRP, PU-Mat-1 and PU-Mat-2 under tensile and flexural loads. The modulus of elasticity average value of tensile loads for BFRP, PU-Mat-1 and PU-Mat-2 were 17.11 GPa, 20.19 GPa, and 19.35 GPa, respectively. Furthermore, the modulus of elasticity average values of flexural loads for the BFRP, PU-Mat-1 and PU-Mat-2 were 15.87 GPa, 17.94 GPa, and 9.20 GPa, respectively. Based on the modulus of elasticity of tensile and bending loads, it can be explained that the increase occurred was linear for each sample variation in terms of BFRP, namely an average of 12% and 15%.The increase in stress due to tensile and bending loading in the composite with basalt fibre reinforcement was strongly determined by the layer of CNF-PU mat. The best result was shown by the lamination of basalt fibre with CNF-PU mat with triton -X surfactant. In this case, triton-X surfactant helped in dispersing CNT so that the resulting solution between PU and CNT was more uniform [52]. In addition, due to the uniformity of the solution, it avoided agglomeration (see also performances in Figures 4, 5 and 6). As a result, it can improve the interfacial tension and load transfer in accepting the tensile and flexural load. These results were also widely reported in several previous publications. [27,48,53,54].

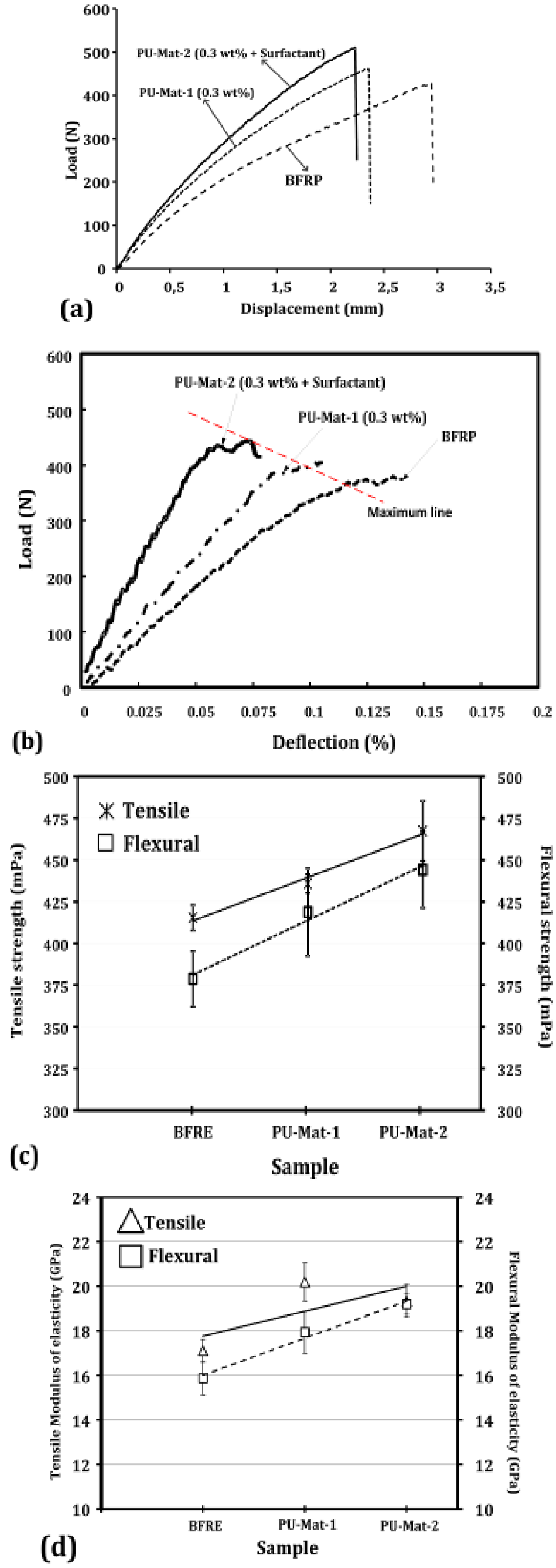

Fig. 7. (a) Load-displacement curve of tensile, (b) load-displacement of flexural, (c) stress-strain Tensile and Flexural, (d) modulus elasticity tensile and flexural

3.3. Failure properties

A failure is a characteristic of each of the materials as the behaviour of its inability to receive or absorb energy either statically or dynamically. In this study, the reported fracture behaviour was only focused on the incorporation of filler with nanoparticles with properties as shown in Figure 4 to improve load transfer on the matrix. few studies have reported on strengthening Nano fillers for epoxy matrices in FRP lamination [6,34,55–57].

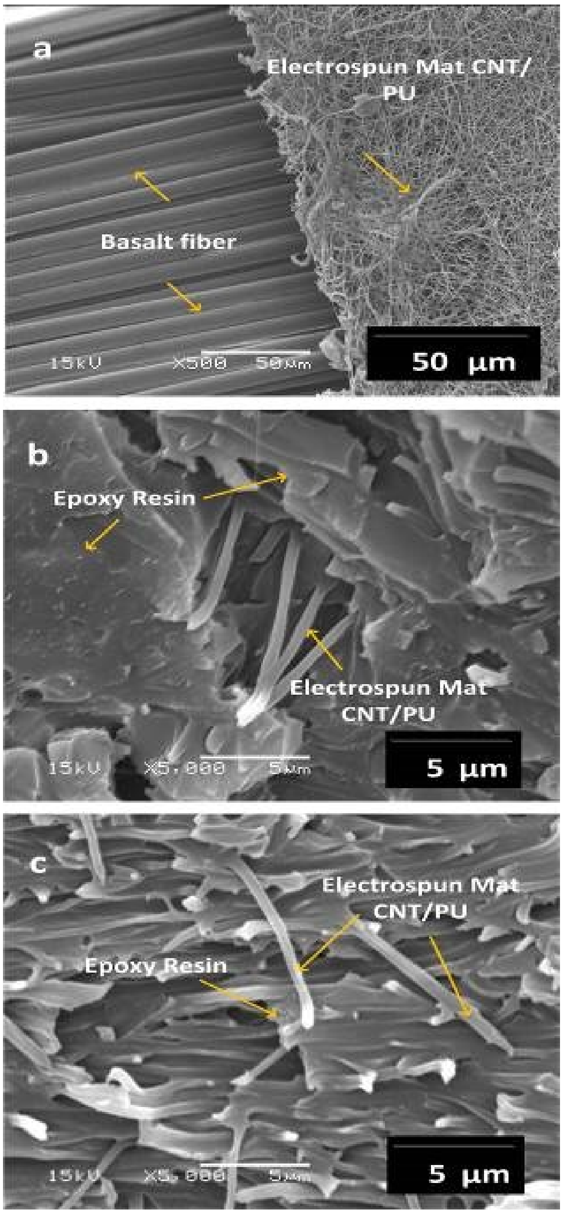

Fig. 8. (a) interleaving among BF-CNF-mat (b) Cross-section fractured surfaces of BFRE laminated electrospun mat CNF nanofibers; (c) Cross-section fractured surfaces of BFRE laminated electrospun mats CNF + Triton-X

Figures 8 (a-c) show the failure of composites epoxy with reinforcement basalt fibre and CNF-mat. The laminate position between basalt fibres and CNF-mat is shown in Fig. 8a. In fact, between the basalt fibres and nanofiber, there were a number of different striking fibre sizes. Also, basalt fibre had a rougher surface compared to nanofiber. This has made the nanofiber very thin and had a wide surface area. Regarding the above matter, we can brief that an increase in the interface of the fibre was due to a greater, and more even absorption of the matrix to bind the fibre [6,58]. This is evidenced by the trend of increasing the tensile and flexural strength of the BF-CNF composites with variation number of CNT, as indicated by the cross-sectional shape of the fracture obtained through SEM analysis. In addition, Figure 8 (b and c) shows a cross-section of spun mat containing 3wt % CNF with and without surfactant (i.e. triton-X) after loading, respectively. As seen in Fig. 8 it can be observed that the relatively solid fracture manner occurred. In other words, they had fewer voids number on the surfaces. However, there were some observed slight delamination and cracks only on the matrix occurred. As previously reported [59], nanofibers have a surface area with several orders of magnitude wider than basalt fibres. In these cases, the increase in the surface area of the nanofiber is caused by the emergence of CNTs on the nanofiber. Despite it can improve the interfacial bond of the epoxy. Then, the uneven repair of CNT spread can be carried out by added surfactants [43]. Wherein, the surfactants have a function to dissolve the CNTs and provide CNTs more dispersebility into the PU nanofibers. Furthermore, it can be argued that CNTs spread has a significant influence on the properties of nanofibers, which means CNT-PU nanofibers can improve a matrix capability in transfer load as shown in Figure 8(c).

3.4. Discussions

Fibre-reinforced composite materials have been extensively designed, developed and applied to engineering products due to their properties and advantages over metallic materials. Several advantages of FRP include lightness, corrosion resistance, and affordability. In addition, FRP can be formed from materials available in the environment and can be combined between organic and inorganic materials. In their sustainability, composite materials have also been developed by applying reinforcing materials based on nanofibers [45]. Many studies have been conducted to determine the performance of nanofibers applied as a part of the FRP structures [6,26,40,60,61]. The main application of nanofibers is to improve performance and mechanical and/or physical properties [62]. Furthermore, apart from the availability of nanofiber materials, techniques for how these materials are formed are also developed, such as by using the lamination method.

In this research, we produced nanofibers mat formed from a mixture of polyurethane (PU) and CNT as a solution in a mixture of MEK and DMF. This solution was created through an electrospinning process [29,58,60] at a high voltage of 15 kV [23]. It has been informed that the electrospinning process for producing nanofibers provides many advantages either from the geometry of the resulting fibre, low cost, or from the ease of performance as also confirmed by other researchers [63]. The applications of MEK and DMF as a solvent for the chemical reactions of CNT and PU have been conducted. Here, DMF has hydrophilic behaviour with a density of 0.95 g/cm^3 at a temperature of 20ºC, which is very effective for separating and suspending CNTs. In addition, MEK is butanone ($CH_3CCH_2CH_3$) with a function for solvent and/or diluent similar function with DMF. It has a number of physical characteristics such as fluids, brightness, and odour that resembles acetone. Then, multi-wall nanotubes (MWCNT) were employed as mixtures as it is known to be capable of providing a change and an increase in the mechanical strength of the composite. Besides, it has been informed that using nanofibers formed woven through an electrospinning process can reduce the density of the composite. The positive roles of CNTs in the mechanical properties of polymer composites have been confirmed by several studies. For example, the effect of MWCNTs on the mechanical properties of composites in average values could increase up to 21.3% [50,64].

In contrast to expectations that the use of CNTs can improve the mechanical properties of polymer composites, there have been several unique phenomena in the process i.e. agglomeration (see Figure 8), which was caused by the non-uniformity of the CNT mixtures in the solution. However, it could be managed by adding the surfactant that could help to provide greater disperse of CNTs evenly distribution [43] on the CNF-mats (this effect significantly improved the evenness of the fibers as shown in Figure 5 and 6). Additionally, we have agreed that the distribution of CNTs has a significant effect on the properties of nanofibers, indicating that CNTs-PU nanofibers could help to increase the ability of the matrix to transfer loads. In other results, CNT caused an increase in the composites material properties including stiffness, strength and strain [65]. From the results obtained from this study, where nanofibers were combined with basalt fibres in a laminate, there has been an increase in the mechanical properties of the FRP composite [62]. Also, in future it can bring make an effective contribution to the quality and characteristics of FRP composites in engineering structures [66]. The results of the study showed that the lamination of nanofibers on basalt fibres changed the mechanical properties of single-fibre composites, thus, according to the target to be achieved, this composite can be applied as a compartment of the electric vehicle due to the material that has light weight, high strength, and high modulus of elasticity. In addition, the laminate mode between the basalt fibres and nanofiber containing the CNT improve their heat transfer so better to use for thermal isolation.

The microscopic studies by FESEM and SEM (see Figure 4, 5 and 6) showed that the presence of GNPs up to 0.3 wt % led to the stability of the transfer/lubricating film by enhancing the adhesion of the basalt fibres to the epoxy resin. The study on using polyurethane (PU) mat spun as part of the composite structures (basalt fibre reinforced epoxy matrix) is interesting in which the mats effectively improve the strength and modulus of elasticity. The PU mat also showed a controlled distribution load of the matrix before breakoff though it had a brittle manner (according to the properties of CNT-PU nanofiber as shown in Figure 4). Thus, they are possibly used as automotive and boat parts for having lightweight, excellent mechanical properties, and being eco-friendly. In addition, they may be applied for energy harvesting and storage due to their higher energy conversion and storage efficiency compared to other materials.

4. Conclusion

In this research, composite reinforcement engineering was carried out by laminating single reinforcement (basalt fibres) with CNF-mat. This research aims to improve the mechanical properties of single-reinforced composites. Currently, fibres containing CNTs with or without Triton-X-100 surfactant have been prepared by electrospinning. The fibres (CNF-mat) were laminated with basalt fibre as reinforcement to the epoxy composite fabricated via vacuum-assisted resin transfer moulding.

The mechanical properties of the composite materials were tested by tensile and bending loading. Also, FESEM and TEM analyses were carried out to characterize CNF-mat. The test results showed that the lamination of CNF-mat with basalt fibre increased the tensile strength and flexural strength effectively by 13% and 17.3% due to the BFRE, respectively. CNF-mat containing 0.3wt% CNT + surfactant triton -X showed an even dispersion of CNTs; thus, it had the highest value on tensile and flexural loads compared to other variation samples. On the other hand, there was a decrease in shear failure due to tensile and bending loads due to the brittleness of the composite reinforcement.

It can be concluded that the increase of tensile and flexural properties was significantly determined by the lamination of CNF-mat. However, their displacement became shorter. The change was caused by the spread of CNTs on the solution. Additionally, this method has given innovation in the enhancement properties of composite material in the future.

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