

MODIFICATION OF CARBON FIBRE/EPOXY COMPOSITES BY POLYVINYL ALCOHOL (PVA) BASED ELECTROSPUN NANOFIBRES

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ABSTRACT

In this study, the effects of modifying interlaminar region of unidirectional carbon fibre/epoxy composites by the incorporation of electrospun polyvinyl alcohol (PVA) nanofibres were investigated. PVA nanofibres were directly deposited onto the carbon fabrics by electrospinning method to improve mechanical performance of those composites. The features of the electrospun nanofibres were characterized by microscopy techniques. The unidirectional carbon fibre/epoxy composite laminates with/without PVA nanofibre interlayers were manufactured by vacuum-infusion technique in a $[0]_4$ configuration. Tensile, three-point bending, compression, Charpy-impact and Mode-I fracture toughness tests (Double Cantilever Beam (DCB)) were carried out in accordance with ASTM standards to evaluate mechanical performance of the composites. Scanning electron microscopy (SEM) observations were made on the specimens to evaluate microstructural features. It was observed that the carbon fabrics were successfully coated with a thin layer of PVA nanofibres by electrospinning technique. The results showed that PVA nanofibres improve the mechanical properties of unidirectional carbon/epoxy composite laminates when subjected to in-plane loading. On the other hand, PVA nanofibres slightly reduced the mode-I fracture toughness values although they led to more stable crack propagation.

Keywords: Electrospinning, Nano-(Polyvinyl alcohol) fibres, interleaving, polymer composite

1. INTRODUCTION

Carbon fibre polymer composites are being extensively used in many structural applications due to their combination of high strength, stiffness and low density. However, they suffer from poor, matrix dominated properties such as interlaminar fracture toughness, transverse tension, compression and tension-compression fatigue. In order to have a high level of acceptance in broader applications, significant improvements in their matrix dominated properties are necessary. The potential improvement techniques for these composites can be classified into two approaches; (1) mechanical approach and (2) material approach [1, 2].

Mechanical approach includes transverse stitching and z-pinning. This approach has proven to be very effective in improving fracture toughness of fibre-reinforced composites. However, it has serious drawbacks considering the drop in the in-plane mechanical properties of composites. Steeves and Fleck [3] showed that the presence of z-pins decrease the tensile strength of the composite by 27% and the compressive strength of the composite by at least 30%. Reeder [4] found a 30% reduction in strength due to fibre misalignment caused by stitching operation. Material approach includes reinforcing matrix resins using additional nano-phases such as nanoclay, carbon nanotubes, nanowhiskers and vapor grown carbon nanofibres. Kumar et al. [5] systematically reviewed the studies focusing on the effects of these nanophases into matrix material. Phong et al. [6] examined if electrospun PVA nanofibres (nPVA) are applicable to epoxy matrix for the modification of carbon/epoxy composites. In their work, nPVAs were dispersed

into the epoxy matrix. The results were so encouraging that the initiation and propagation interlaminar fracture toughness values in Mode I were significantly improved by about 65 and 73%, respectively. There was also a slight increase of the tensile strength by about 5%. However, the incorporation of nanophases into epoxy matrix has some disadvantages such as the difficulty of dispersing nano-sized phases into the matrix and the enormous increase of resin viscosity. The latter especially is a serious problem for all infusion applications.

Dzenis and Reneker [7] proposed a novel approach (both mechanical and material) to solve the difficulties mentioned above. They used electrospun polymeric nanofibres as a second reinforcement in between the primary reinforcing fabrics prior to infusion. This cost-effective approach only requires addition of an extra-step to manufacturing flowchart, and there is no need for a radical change in the processing route. Although the idea is not new, there has been increasing attention devoted to this topic in recent years. Various types of polymeric nanofibres such as poly caprolactone (PCL) [8], polyacrylonitrile (PAN) [9], polyamide (PA) [10], polyamide 6/6 (PA 6/6) [11], polyvinylidene fluoride (PVDF) [12] and polysulfone PSF [13] have already been studied. Most of these studies mainly focused on the interlaminar fracture toughness of composite laminates. The experimental results proved that polymeric nanofibres are good candidates for toughening of structural composites. Unlike these studies, Bilge et al. [14] investigated the effects of polystyrene-c-glycidyl methacrylate (P(St-coGMA)) nanofibres on the carbon fi-

bre/epoxy composites subjected to in-plane loading. They concluded that P(St-coGMA) nanofibres are moderately efficient for improving in-plane properties of carbon fibre/epoxy composites. Palazzetti et al. [15] studied the self-reinforcing effect of PA 6/6 nanofibres on carbon/fibre epoxy laminates subjected to low velocity impact. They used prepreg materials on which the nanofibres were deposited. Although prepreg materials are often used in aerospace industry, resin infusion has gained great importance for the production of big composite parts such as wind turbine blades, yacht components etc. Van der Heijden et al. [16] improved the interlaminar fracture toughness of infusion molded laminates by using electrospun PCL nanofibres without compromising other mechanical properties. Herwan et al. [17] proved that load-bearing capacity of pin joined composite laminates can be improved by introducing PAN nanofibres between dry carbon fabrics.

In the present study, the effects of the PVA nanofibre modification of the interlaminar region on the mechanical performance of unidirectional carbon fibre/epoxy composites were investigated. PVA was chosen because it is a cheap, water-soluble, odorless and non-toxic polymer. There are only a few studies reported in the literature on the investigation of the effects of nano interleaving of the carbon fibre/epoxy composites manufactured by vacuum-infusion method [16, 18]. The objective of the present work is to investigate if the proposed approach is suitable for the structural composites made of dry carbon fabrics. Additionally, no reports have been published in the literature on the compressive properties of carbon fibre/epoxy composites interleaved by PVA nanofibres. This study was also intended to contribute to the literature and provide better understanding how these composites behave when they are interleaved by thermoplastic nanofibres.

2. EXPERIMENTAL PROCEDURE

2.1 Materials

PVA with an average molecular weight of 72,000 g/mol was used to produce nanofibres. The unidirectional carbon fabrics with a unit weight of 325 g/m² were supplied by Kordsa Global Inc. of Turkey. The epoxy resin Momentive L160 and its hardener H160 were used with the weight ratio of 80:20; respectively. This low-viscosity resin system is suitable for marine and aerospace applications.

2.2 Preparation of PVA nanofibres

PVA nanofibres were produced by electrospinning process. Fig. 1 shows the electrospinning set-up used in this study. It consists of 5 nozzles that are suited for the production of relatively larger area of uniform nanofibrous nonwovens, a cylindrical translating-rotating collector and a syringe pump. To produce PVA nanofibres, 15 wt. % of PVA solution was prepared by dissolving 15 g of PVA powder in 100 ml distilled water at 80°C for 6 hours by using a magnetic stirrer. A transparent and homogenous gel solution was obtained. The prepared solution was left to cool down

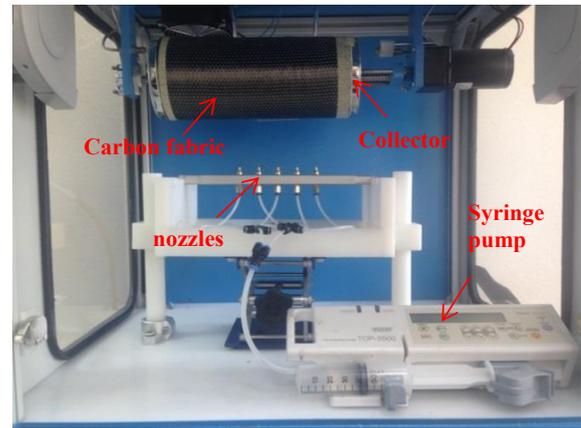


Fig. 1: Photograph of the current electrospinning system used in our laboratory.

to room temperature before electrospinning.

2.3 Deposition of PVA nanofibres on dry carbon fabrics

The prepared solution was placed into a 50-ml syringe which is attached to a syringe pump. The flow rate of the PVA solution was adjusted to 8 ml/h (1.6 ml/h for each nozzle), and the applied positive voltage was set in the range of 32–35 kV. The tip-to-collector distance was 12 cm. The nanofibres were collected onto the unidirectional carbon fabrics for the duration of 60 minutes. The color of the carbon fabrics changed from black to white gradually during the electrospinning process (Fig. 2). Fig. 3 shows SEM image of the carbon fabric coated with PVA nanofibrous veils. Once the fabrics were coated, they were heat

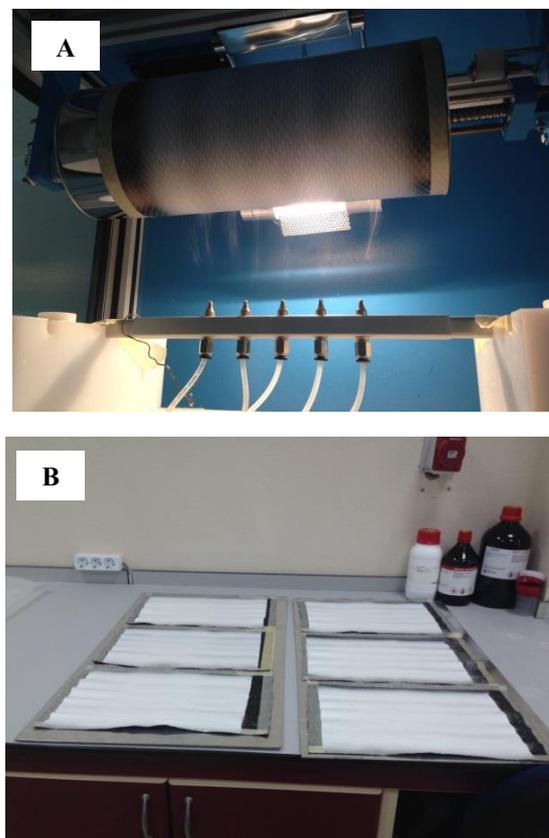


Fig. 2: (a) Deposition of PVA nanofibres (b) the unidirectional carbon fabrics with PVA nanoveils.

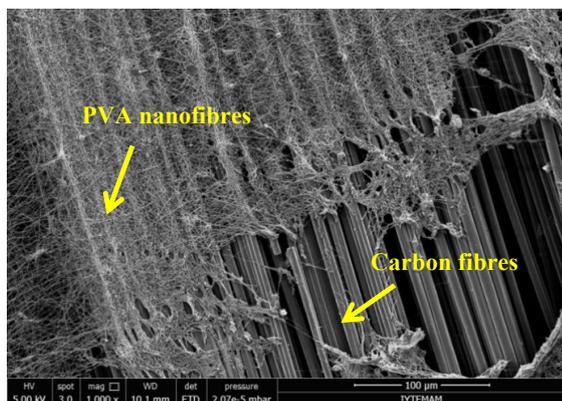


Fig. 3: SEM image of PVA nanofibres deposited on a carbon fabric.

treated (physically crosslinked) for 10 minutes at 150 °C to stabilize the PVA nanofibres and improve their mechanical strength. In order to determine nanofibre areal weight (g/m^2), the nanofibrous veil was cut into three small pieces after which it was carefully peeled off from the surface of the carbon fabric. They were weighted with an accuracy of 0.0001g. The average nanofibre areal weight was measured to be approximately $7.10 \pm 0.70 \text{ g}/\text{m}^2$.

2.4 Manufacturing of composite test specimens

The reference and PVA modified composites with four layers of unidirectional carbon fabrics were fabricated by vacuum-infusion technique. A Kapton® polyimide film (50 μm thick) was inserted between the second and third plies to form an initial crack along the interlaminar region of the DCB specimens. The vacuum infusion setup is shown in Fig. 4. Based on the visual observations during the vacuum-infusion process, no change of the resin flow due to the nanofibres on the resin flow was detected. Demolding of the manufactured composites was carried out after complete curing at room temperature, followed by a post curing in an oven at 80°C for 12 h. The fabricated composites were cut into desired dimensions using a water-cooled diamond saw. The cut edges of the specimens were lightly sanded with 280 grit sandpaper by hand. The thickness of the specimens was in the range of 1.5-1.6 mm. The effect of PVA nanofibres on the final thickness of the specimen was less than 1%.

3. MECHANICAL TESTING

All mechanical tests were performed using a universal tensile testing machine (Shimadzu model AG-IS) in accordance with relevant ASTM standards [19-22]. The tests were carried out at ambient conditions. Each test was repeated at least five times for both reference and nPVA-modified specimens.

The tensile tests were carried out at a constant crosshead speed of 2.0 mm/min up to failure (Fig. 5a). A digital video extensometer was used to measure the strain. The compressive strength and modulus of the specimens in the

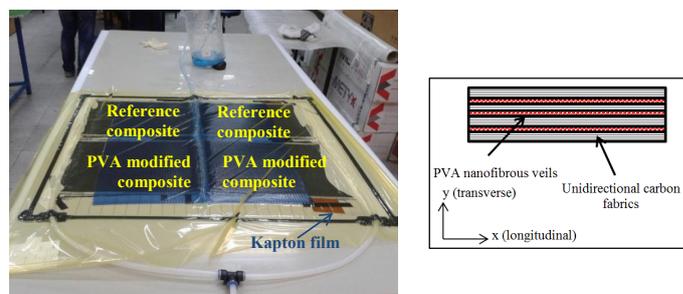


Fig. 4: Vacuum infusion setup and process.

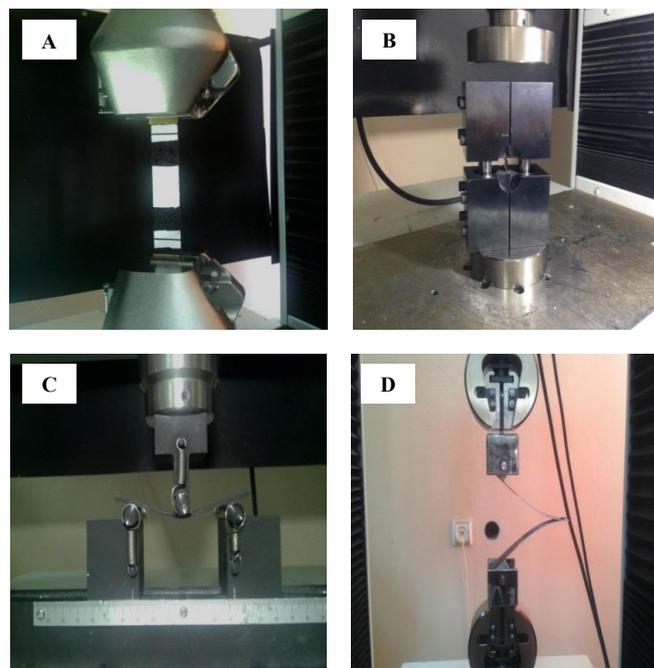


Fig. 5: Photographs of the test specimens under (a) tensile, (b) compressive, (c) flexural and (d) Mode- I loading.

longitudinal and transverse direction were determined by performing compressive tests. The dimensions of the compression test specimens were 140 mm in length and 12.7 mm in width. The specimens were placed in an anti-buckling fixture and loaded until failure at a constant crosshead speed of 2.0 mm/min (Fig. 5b). The flexural properties of the reference and PVA modified specimens were obtained from three point bending tests (Fig. 5c). Charpy impact tests were carried out according to ISO-179 standard [23] on 10 mm x 80 mm rectangular notched specimens, using the CEAST® Resil Impactor having maximum hammer energy of 15 J and hammer tangential speed of 3.46 m/s.

The Mode-I interlaminar fracture toughness (G_{IC}) of the composites was determined by DCB experiments. The dimensions of the DCB test specimens were 150 mm in length and 25 mm in width. Aluminum tabs were bonded to outer surfaces of the DCB specimens to transfer the opening forces (Fig. 5d). Load, opening displacement and crack length were recorded for the energy release rate (G_I) calculation during the tests. G_I was calculated from Modified Beam Theory (MBT) method given as follows [22]:

$$G_I = \frac{3P\delta}{2B(a+|\Delta|)} \frac{F}{N} \quad (1)$$

where P , δ , B and a are the load, displacement, specimen width and crack length, respectively. F , N and Δ are the correction factors for large displacements, the stiffening caused by the metal blocks and crack tip rotation and deflection, respectively. Δ was determined from a linear regression analysis of $(C)^{1/3}$ versus a data, where C is the compliance (δ/P).

4. SCANNING ELECTRON MICROSCOPY AND DYNAMIC MECHANICAL ANALYSIS

Scanning electron microscopy (SEM) observations were made to determine nanofibre morphology and to better understand the failure modes. The specimens were sputter-coated with gold for 90 seconds and examined under a Philips XL 30S FEG scanning electron microscope. Dynamic mechanical analysis (DMA) was carried out on the specimens by using a DMA Q800 (TA Instruments, USA) equipment. The dimensions of the specimens were 65 mm in length and 10 mm in width. At least three specimens were tested for each laminate. The heating rate was 5°C/min from room temperature to 150°C and the frequency was 1 Hz. The glass transition temperature (T_g) of the reference and modified specimens were determined from the peak of the $\tan\delta$ spectrum.

5. RESULTS AND DISCUSSION

5.1 Nanofibre morphology

Fig. 6 shows the SEM images of electrospun PVA nanofibres before and after heat treatment. It was observed that PVA nanofibres were successfully produced using electrospinning technique. Continuous and relatively uniform fibres were obtained. Thermal sealing of PVA nanofibres after heat treatment in the intersections can be seen in Fig. 6b. Nanofibre diameter distribution was determined by measuring 50 fibres per sample with Image J software. The average nanofibre diameter was measured to be 257 ± 58 nm before heat treatment. After heat treatment, the average diameter of nanofibres was increased to 329 ± 58 nm. Some partially melted nanofibres were observed. The formation of crosslinks within the nanofibres and also partially joining of the individual fibres due to partial melting stabilize the PVA nanofibres and hence improve their strength.

5.2 Mechanical test results

Fig. 7(a) represents the load-displacement curves of tensile test specimens. The curves showed the main features of unidirectional composites under tensile loading such that the load was almost linear initially, and then reached to the highest value followed by a sharp drop. The reference tensile specimens rapidly failed catastrophically accompanied by a burst sound. The failure of PVA-modified specimens was not catastrophic; they remained intact indicating the improved matrix properties. The presence of PVA nanofibres on the interlaminar region increased the tensile strength of unidirectional carbon composite laminates by 12%. This is due to improved matrix cracking resistance at the free edges. No significant change in elas-

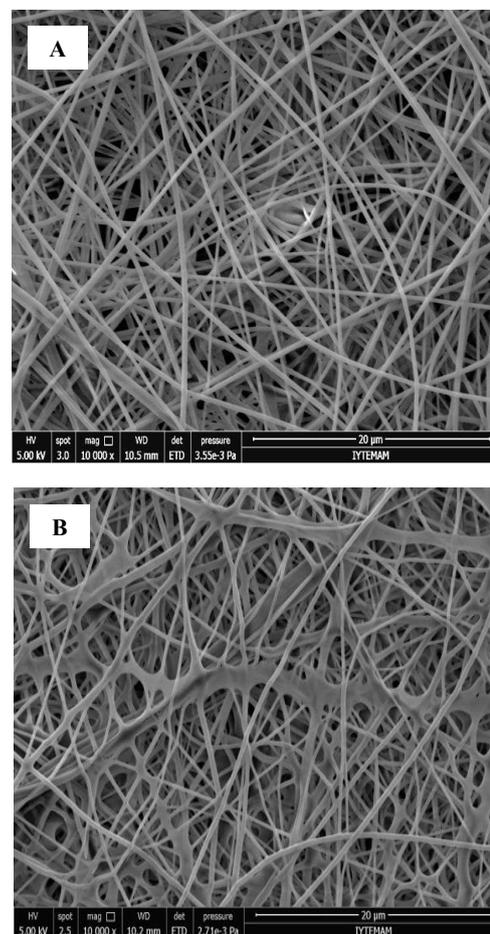


Fig. 6: SEM images of PVA nanofibres (a) as-spun and (b) after heat treatment.

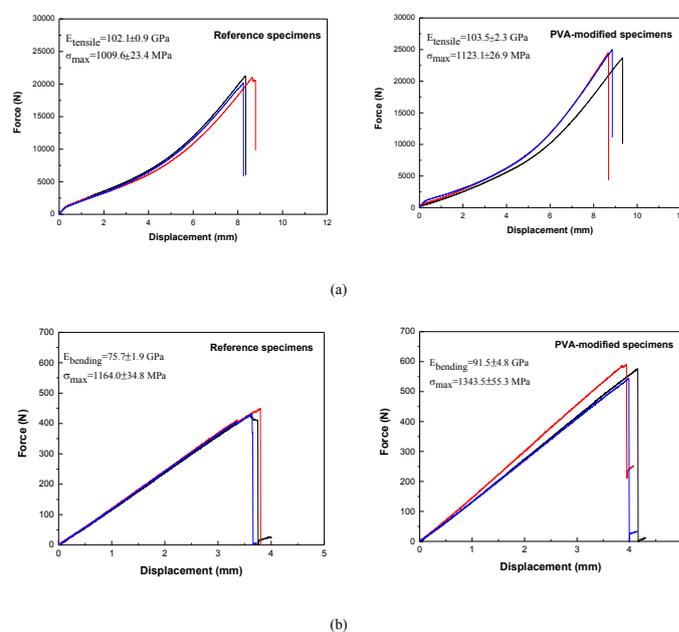


Fig. 7: Load-displacement curves of the specimens under (a) tensile and (b) flexural loading.

tic modulus of unidirectional composite laminates was observed, as expected. This is because the elastic modulus in the longitudinal direction is a fibre-dominated property. The plastically deformed and necked PVA nanofibres and excessive deformation of epoxy matrix can be seen in Fig. 8. These results also confirm previous study by Bilge et al. [14], demonstrating PVA nanofibre interleaving can improve the tensile strength of carbon/epoxy composites. Fig. 7(b) shows the load-displacement curves of the reference and PVA-modified test specimens under flexural loading. The flexural modulus was improved by 21% with PVA nanofibre interlayers. The flexural strength of PVA-modified specimens was 16% higher than those of reference ones. The improved matrix properties increased in-plane bending performance. These results are consistent with those obtained in previous studies by Herwan et al. [17] and Palazzetti et al [24].

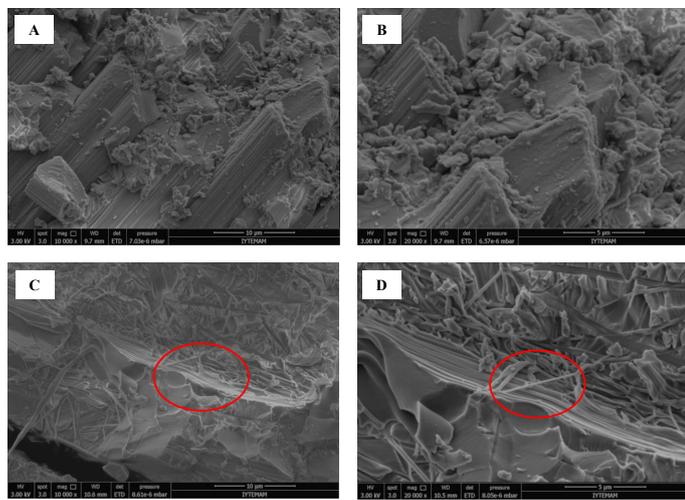
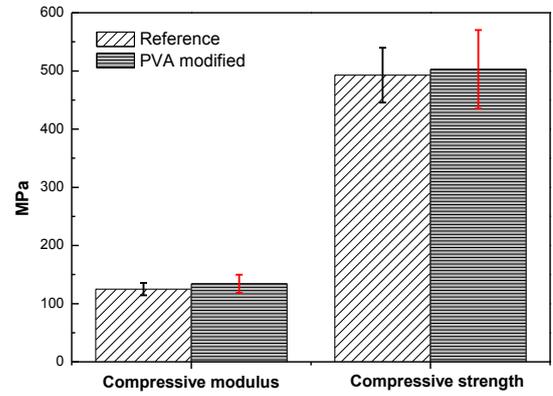


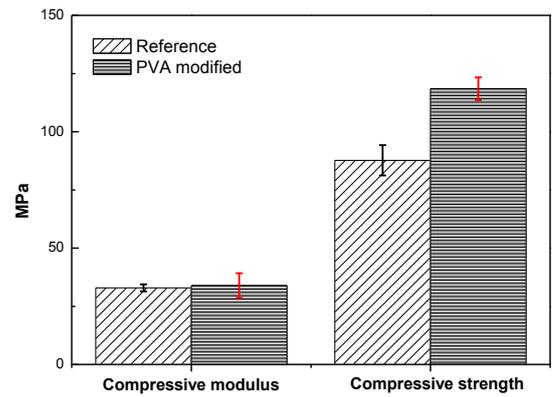
Fig. 8: SEM images of the (a-b) non-modified, (c-d) modified test specimens after tensile test.

Fig. 9 shows compression test results in longitudinal and transverse directions. It is seen that there is no effect of PVA nanofibres on the compressive modulus and strength values in the longitudinal direction. However, it is worth nothing that PVA nanofibres can significantly increase the compressive strength of the prepared composites by 35% in the transverse direction. Compressive strength, especially in the transverse direction, is a fibre/matrix interface and matrix dominated property. Therefore, it is associated with that PVA nanofibres provides stronger fibre/matrix interface in comparison to reference specimens. These results are consistent with previous study by Phong et al. [6]. It is well-known that improved interfacial properties increase the mechanical performance of the composites.

The impact fracture energy of the reference specimens was measured to be 90.1 ± 4.0 kJ/m². The impact fracture energy increased about 11% (100.2 ± 5.6 kJ/m²) by introducing PVA nanofibres on the interface area of composite laminates. The improved impact energy can be explained by analyzing the SEM pictures of the fracture surfaces of reference and PVA-modified specimens. As seen in Fig. 10



(a)



(b)

Fig. 9: Compression test results for (a) longitudinal direction and (b) transverse direction.

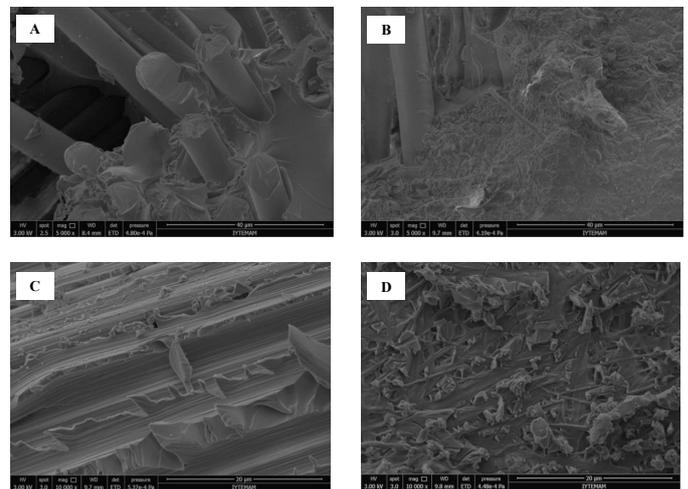


Fig. 10: SEM images of fractured surfaces of (a) reference and (b-d) PVA-modified specimens after impact.

(a), reference specimens had a glassy and smooth fractured surface and showed no sign of deformation. These are main characteristics of poor interfacial bonding strength and impact energy. However, as seen in Fig. 10(b-d), PVA-modified specimens exhibited more complex fractured surface which indicates higher deformation and impact energy.

Fig. 11 shows load-displacement curves for DCB specimens. It was observed that the reference specimens exhib-

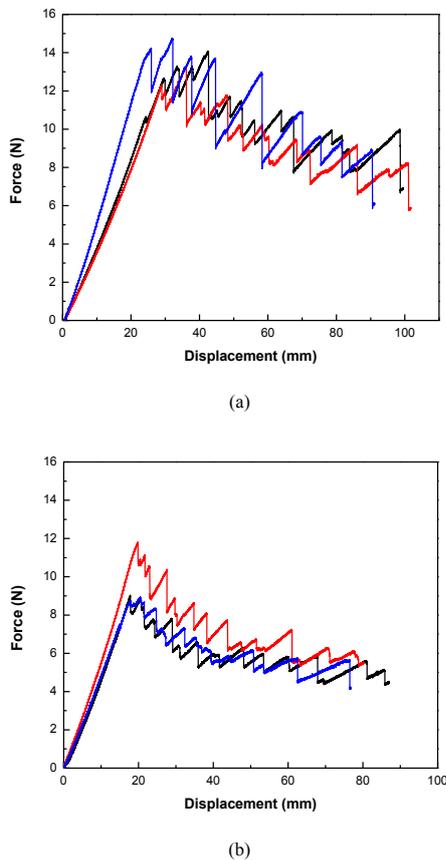


Fig. 11: Force-displacement curves of the DCB test specimens (a) reference and (b) PVA-modified specimens.

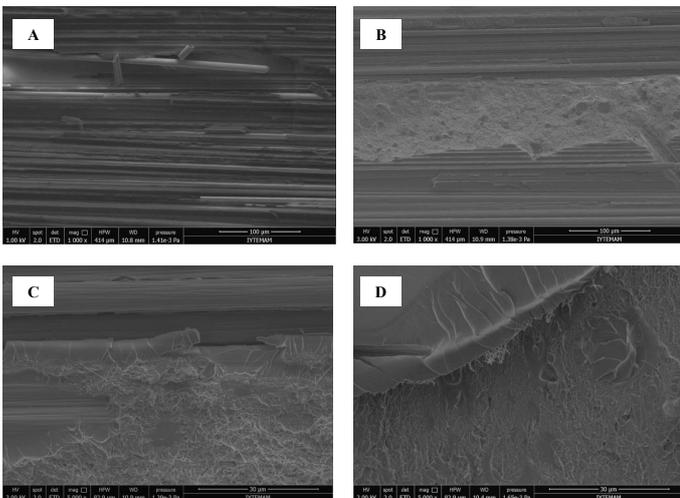


Fig. 12: Scanning electron micrographs of DCB test specimen fracture surfaces; (a) reference and (b-d) PVA-modified specimens. (Crack propagates from left to right).

ited higher force values with relatively larger load drops as compared to those for modified specimens. These larger load drops indicates unstable crack-propagation within the delamination region. On the contrary, modified specimens experienced a more gradual decrease in load. As stated by Moroni et al. [25], the bridging effect of the nanofibres inserted into the interlaminar region is negligible at the initial stage of the experiments. As the crack propagates, this mechanism led to more stable crack propagation. The average mode-I fracture toughness of the reference specimens and modified specimens were found to be 0.33 and 0.24 kJ/m², respectively. The lower fracture toughness val-

ues of the modified composites is associated with the lower fracture energy of the nanofibre modified matrix region near to the fibre/matrix interface and the reduced amount of matrix ahead of the crack tip in the interlayer. Additionally, fibre nesting (fibre bridging) observed on the reference specimen edges during the test caused an increase in fracture toughness values. The SEM micrographs of the DCB test specimens fracture surfaces are shown in Fig. 12. The micrographs were taken from the middle of the specimens (away from the Kapton film) for interpreting fracture mechanisms. Fig. 12 (a) shows the fracture surfaces of the reference specimens without nanofibres. They were smooth and featureless (indicating low matrix ductility) which are the main characteristics of unidirectional composites under Mode-I loading. As stated by Greenhalgh et al. [26], Mode-I fracture toughness is controlled by processes such as cohesive fracture of the matrix and fibre bridging. The broken fibres pulled out from the resin are visible in Fig. 12 (a) as an evidence of the fibre bridging. The fracture surfaces of nanofibre modified specimens exhibited a combination of brittle and ductile regions (plastic deformation of the matrix and PVA nanofibres) as can be seen in Fig. 12 (b-c). Fig. 12 (d) clearly shows deformed PVA nanofibres in the matrix. This supports the hypothesis of more stable crack propagation in the nanofibre modified specimens. When a crack slowly propagates, the matrix has time for plastic deformation [27]. Although the Mode-I fracture toughness values seemed to be negatively affected, the authors believed that these nanofibres had a potential to improve the Mode-II fracture toughness. Under Mode-II loading, in-plane shear stresses exist between plies therefore the advantages of these nanofibres would be more pronounced. For instance, in Moroni's study [25], it was shown that the presence of Nylon 6,6 electrospun nanofibres caused a decrease in the Mode-I fracture toughness about 17% while these nanofibres improved the Mode-II fracture toughness about 12%. Another way to increase nanofibre interleaving effect on the mechanical performance is to increase electrospinning time. As the amount of nanofibres increases, the possibility of nanofibre bridging increases. On the other hand, this would make the laminate thicker which would lead to reduced strength in the composite interface as also highlighted by Palazzetti et al. [28]. It is critical that the electrospinning time should be optimized considering the manufacturing feasibility and performance of the composite laminates. DMA results show that the reference carbon/epoxy composites exhibited a glass transition temperature (T_g) of about 100 °C. The T_g was found to slightly increase from 100 °C to 105.3 °C with the presence of PVA nanofibres on the interlaminar region of the composites. This is due to the higher glass transition temperature of PVA nanofibres as compared to those for epoxy matrix.

Table 1 shows the comparison of PVA nanofibre interleaving with other recent results available in the literature. It can be seen from Table 1 that PVA nanofibre interleav-

Table 1: Comparison of the effect of PVA nanofibre interleaving obtained within the present study and reported by other groups.

Reference	Nanofibre	Property	% increase
Present study	PVA	Tensile strength	+12
		Flexural strength	+16
		Compression strength*	+35
		Impact energy	+11
		Mode-I fracture toughness	-27
[14]	P (St-co-GMA)	Tensile strength	+12, +18**
[17]	PAN	Flexural strength	+21
[24]	Nylon 6,6	Flexural strength	+40
[25]	Nylon 6,6	Mode-I fracture toughness	-17

* in the transverse direction

**depending on the lay-up orientation

ing showed good performance for improving in-plane mechanical properties compared to other nanofibres. From the manufacturing point of view, the solvent and its vapor should be non-toxic to the machine operators. The polymers such as Nylon 6,6 and PAN require harmful solvents while PVA can be dissolved in water with little heating. Additionally, PVA nanofibres can be reinforced with the addition of different nanoparticles which may lead to better performance.

6. CONCLUDING REMARKS

In this study, the effects of electrospun PVA nanofibre interlayers on the mechanical performance of unidirectional carbon fibre/epoxy composites were investigated. For this aim, carbon/epoxy laminates with/without PVA nanofibre interlayers were manufactured by vacuum infusion process. A series of mechanical tests were conducted on the reference and PVA-modified specimens. The following conclusions were made based on the experimental results; The tensile strength, compressive strength (in the transverse direction), flexural modulus and flexural strength of the laminates with PVA nanofibre interlayers were improved by 12, 35, 21% and 16%, respectively, as compared to those for reference specimens. Charpy impact tests revealed that PVA nanofibre interleaving enhanced impact strength by about 11% as compared to those for reference specimens. Although PVA nanofibres had a negative effect on Mode-I fracture toughness values, these nanofibres led to more stable crack propagation.

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