CHARACTERIZATION OF ELECTROSPUN CARBON NANOFIBER MAT REINFORCED POLYMER COMPOSITES USING ULTRA-SONIC SCANNING METHOD

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More than ever before composites are being utilized in the wide applications such as aircraft, marine, chemical, automotive, biomedical etc. The characterization of these materials for various geometric configurations has become a primary concern to designers as well as manufacturers. Nano particle reinforcement and the nano fiber reinforcement in the resin matrix composite is one of the newer methods of composite material development. In this work, efforts have been made to fabricate three different concentrations of carbon nanofiber (CNF) /poly vinyl alcohol (PVA) electrospun mat has been produced by using electrospinning process and the mat was reinforced with polymer epoxy resin in three concentrations of CNF/PVA. Scanning electron microscope (SEM), X-ray diffraction (XRD) and ultrasonic testing (UT) was conducted on the specimens for analyzing the structural conditions of the coupons in turn for optimize the mechanical properties of the coupons using the three point flexural test.

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*Keywords:*Carbon nanofiber (CNF), Poly Vinyl Alcohol (PVA), SEM, ultrasonic Scanning and XRD.

1.Introduction

Electrospun fibers and mats have received great attention in recent decades and maintain to provide emerging revelations and opportunities, these materials remain within the conventions of nanotechnology research and applications [1-4]. Electrospinning has been regarded as the most significant approach to produce continuous nano fiber on a hugely immense scale and the fiber diameter can be adjusted from nanometers to micrometers [5]. The relatively sizable large concrete surface area and high porosity make electrospunnano materials magnetize consequential attentions in developing ultrasensitive sensors [6-8]. Carbon nanofibers (CNF) are the advanced material used for polymer reinforcement, because of their better tensile strength, excellent thermal and electric properties [9]. Another advantage is that it gives the better electromagnetic shielding. CNF's have been used as the reinforcement materials in polymers like polyethylene [10], nylon [11], and polycarbonate [12]. In advanced aerospace applications, structural composite are preferred due to their high temperature resistance. In military and aerospace field, fiber-reinforced composite are widely used due to their good thermal and mechanical properties. Organic matrix composites have been found applicable in jet engines. Conductive fillers in polymer-matrix composites have potential designates of achieving an attractive mixture of properties for aerospace applications [13,14]. The electrospun PAN-predicated carbon nanofiber can be utilized for making hierarchical nano structures [15,16], super-capacitors [17,18], filters [19], nano composites [20], catalyst supports for rechargeable batteries and/or fuel cells [21,22], and optoelectronics [23]. The tensile strength of the irradiated PAN/ lignin nanofiber mats gradually incremented to the non-irradiated PAN/lignin nanofiber mats. It increases the tensile strength could be attributed to the stabilization of PAN during the E-beam irradiation [24]. In the polymeric area, epoxy resins are

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well, predictable thermosetting matrices in the innovative composites, presenting a series of remarkable characteristics like excellent stiffness and specific strength, dimensional strength, chemical resistance and also strong bond to the reinforcement [25]. It was used as superior grade synthetic resins, used in the area such as electronics, rocketry, and astronautics industries. It's correlated to the good mechanical properties of epoxy resin matrix in the overview of CNF had been conducted [26]. To utilize the maximum properties of nanofibers, uniform dispersal and good dampening of the nanofibers into the resin matrix must be ensured [27]. It has been widely described that dry nanofibers frequently agglomerate, and thus it has impressively reduced their ability to bond with the epoxy resin matrix. All these limited interfacial things will disturb the macro-level material properties [28]. Such that, the distinct dispersal behavior of carbon nanofibers in polymers had an extreme effect on the physical properties of the nano composites studied [29]. In this work, we fabricated PVA/CNF using the electrospinning technique. The electrospun PVA/CNF mats were cured at the atmospheric temperature. Then the different percentage PVA/CNF mat has been produced by electrospinnig. Various proportions PVA/CNF mat has reinforced with the epoxy resin and it is cured at the room temperature, and the flexural testing carried out for all the coupons upto failure. The PVA/CNF mats were characterized by SEM, XRD, UT and UTM analysis, respectively.

2. Experimental Work

2.1. Material

Polyvinyl alcohol (cold) molecular weight 85,000-1, 24000 was obtained from central drug house (p) Ltd and used without further purification. Nanofiber polymer matrix (Araldite standard epoxy resin CY 230-1, HUNTSMAN) and hardener (Aradur HY 951) in weight ratio 10:1. Epoxy resin and hardener were purchased from (Leo Enterprises Ltd). The resin was of low processing viscosity and enhances mechanical properties. Carbon nanofiber used for the fabrication was purchased from (Sigma Aldrich). They are 100 nm in diameter, 20-200 μm long and >98% carbon basis.

2.2. Electrospinning

Electrospinning is a process for making ultra-fine fibers, first Patented in 1934. Electrospinning method presently produces micro-scale fibers at a commercially possible level using electrostatic forces to pull fibers from a capillary of polymer solution. According to Deitzel et al., The method can be considered a variation of the electro spray process. The polymer solution consists of a prearranged mixture of polymers in the suspended solvent. A droplet of polymer solution forms at the needle's tip due to gravity and is held in place by surface tension. Formation of the nanofiber begins when the electrostatic force is greater than the surface tension of the droplet. The fiber is formed as the ejected jet stream is narrowed by whipping itself as a result of an increase in surface charge density due to the evaporation of the solvent [30].

2.3.Electrospinning process

The electrospinning method contains electrostatic forces in the formation of nanofibers. This method is totally different from conventional fiber spinning technique. It depends on the mechanical force to make fibers by extruding the polymer solution through a spinneret and after drawing the subsequent filaments as they harden and set. Electrospinning has the application of an electric field between a needle tip and a grounded collector through a high-voltage source. A pending droplet of polymer solution at the needle's tip is changed to a semicircular shape and then into a conical shape it is called as a Taylor cone to the electric field. When the strength of the electric field causes a higher effect than the surface tension of the polymer solution, the solution is ejected to the grounded aluminum collector. If the viscosity of the solution is low, the polymer solution jet will break up into droplets. However, when the viscosity of the polymer solution is high enough, a continuous jet is formed. A sequence of electrically induced bending uncertainties in the air results in stretching and elongation of the jet in a cone-shaped volume. Rapid evaporation of the solvent during the elongation process reduces the fiber diameter. The non-woven mat

collected on the grounded surface contains continuous fibers from the micro-scale to the nano-scale. Figure 1 shows the schematic diagram of an electrospinning setup.

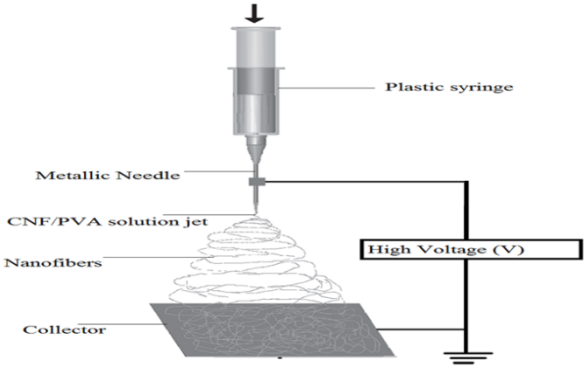

Fig. 1 shows the schematic diagram of an electrospinning setup

2.4. Preparation of PVA/CNF mat

The weight ratio of the CNF in PVA is 0.005%, 0.008% and 0.015% by weight. Homogenous solution was obtained by heating at 60˚ C for 4 hours with constant stirring and 1 hour sonication use of the ultra-sonicator for obtaining homogenous solutions. During this process, a huge voltage was supplied to a droplet of the CNF/PVA polymer solution at the needle's tip. The CNF/PVA polymer solution was then supplied through a 5 ml syringe, using a feed pump at a feed rate of 1 ml/HR. While a constant voltage of 10 Kv for the complete process. Thus the PVA/CNF mat has been collected on the ground collector and the different proportion mat has been shown in figure 2.

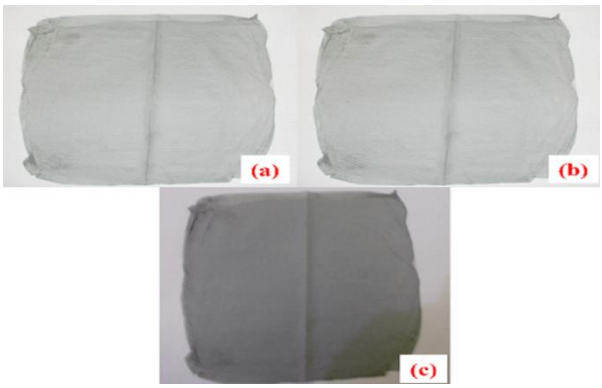

Fig. 2. CNF/ PVA mat made at (a) 0.005 wt.%, (b) 0.008 wt.% and (c) 0.015 wt.%.

3. Characterization

3.1. Scanning electron microscope

Fig. 3 a, b, c shows the SEM image of the PVA/CNF fiber orientation. It is a process for clear resolution imaging the coupon surface, where the electron beams were scanned across the surface of the coupon and these signals collected by deflector form images displayed on a cathode array tube. SEM is one of the most widely used methods for nano material characterization. The diameter of the CNF mat is measured using the Image J software and found in the range of 206 nm to 1010 nm for three concentrations (0.005%, 0.008% and 0.015%). Moreover, the cut section view of the PVA / CNF reinforced composites bonded with resin is depicted in Figure 3 d, e, f.

The top view and bottom view of the CNF coupon shows the presence of cavities and a smooth surface as shown in Figure 3 g and h respectively.

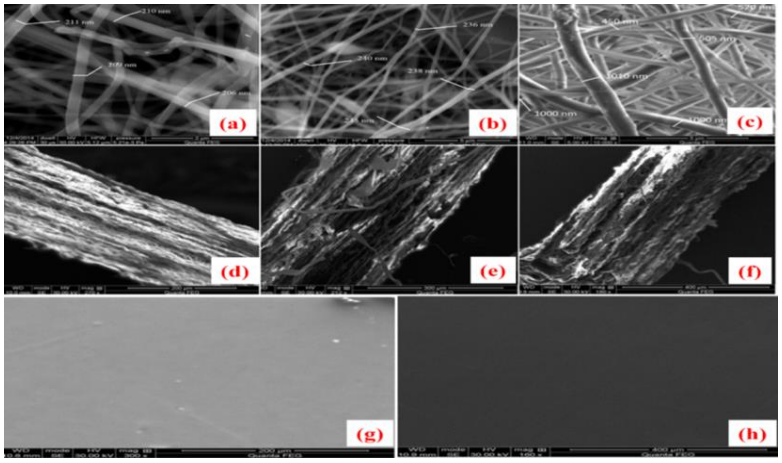

Fig. 3. (a), (b) and (c) shows CNF diameter at 0.005 wt.%, 0.008 wt.% and 0.015 wt.%. (d), (e) and (f) depicts the sectional view of CNF/PVA mat reinforced coupon at 0.005 wt.%, 0.008 wt.% and 0.015 wt.%. (g) Rough surface (h) Smooth surface

3.2. X-ray Diffraction (XRD)

XRD was used to determine the structural properties of the PVA/Carbon nanofiber mats. The PVA/Carbon nanofiber mat has the diffraction peak at 2Θ of 16.8$^{\theta}$ and 28.8$^{\theta}$ were assigned to (100) and (110) crystallographic plane. These results indicate that the PVA nanofiber mats were converted into carbon nanofiber mat as shown in figure 4.

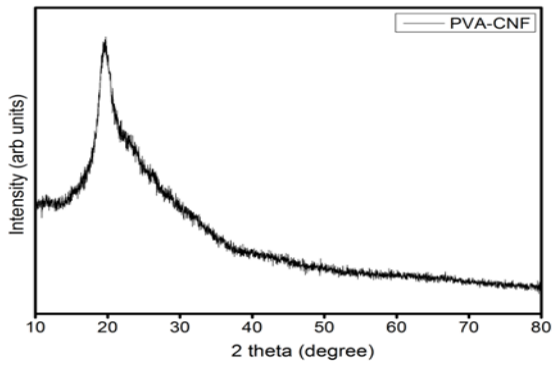

Fig. 4. XRD shows the PVA Nanofiber mats were converted to carbon nanofiber mat.

3.3. Pulse-echo ultrasonic testing

Ultrasonic testing is a versatile inspection method to identify the internal and external flaws in the material. In ultrasonic testing a transducer sends out a pulse of energy and the same transducer receive the energy back. Reflection occurs due to the presence of discontinuities and the surface of the test sample. The amount of reflected sound energy is displayed with the change of time, which provides the inspected information about the size and the location of the flaws as shown in figure 5.

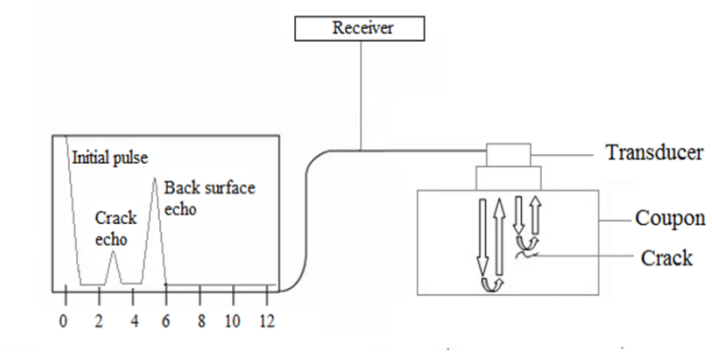

Fig. 5. Working principle of ultrasonic testing

Ultrasound scanning is conducted on the coupons to identify the defect. The cavity is identified on the surface of the coupons as shown in table 1

4. Experimental work

4.1. Preparation of the composite

CNF/PVA nanofiber mat reinforced composites were fabricated by hand lay-up, and the matrix was cured at the room temperature. The hand lay-up method is the simplest method for composite molding. Eight layers CNF mat was used for the flexural coupon.

4.2. Fabrication of epoxy resin coupon and CNF/PVA nanofiber mat composites

The CNF/PAV nanofiber mat composite laminate was fabricated using a hand-lay-up method. The thickness of the plate is 2 mm with each layer of a thickness of 0.09 mm. The laminate was fabricated using CY 230-1 epoxy with hardener HY951. Coupon with the dimension 80, 25, and 2 mm, length, width and thickness respectively were fabricated without any edge damage. The ASTM D790 coupons are shown in figures 6 (a) (b) and (c).

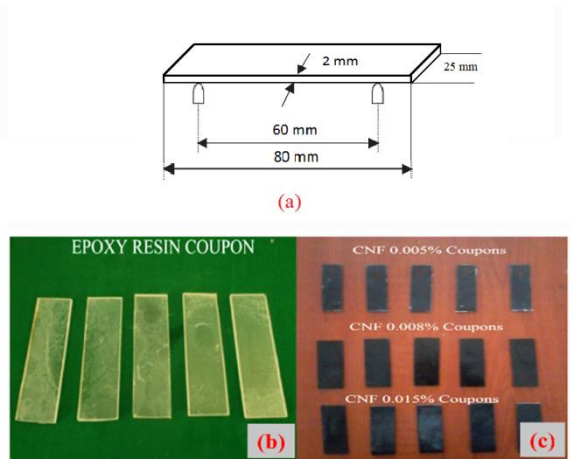

Fig. 6. (a) 3-Dimensions of the coupon, (b) Pure epoxy resin coupon and (c) CNF mat reinforced coupon

4.3. Flexural testing:

The flexural coupons obtained from the CNF mat laminates are subjected to the Point load was applied using a DAK 10-ton universal testing machine (UTM). Twenty coupons were tested. The spans were 60 mm and the support length is 10 mm each side. The feed rate was 5 mm/min

maintained throughout the testing. The test specimens were loaded with a three point bending test as per ASTM D790. The load was gradually applied in the middle of the coupon. The load was applied until the coupon failure and was repeated for all coupons as shown in figure 7.

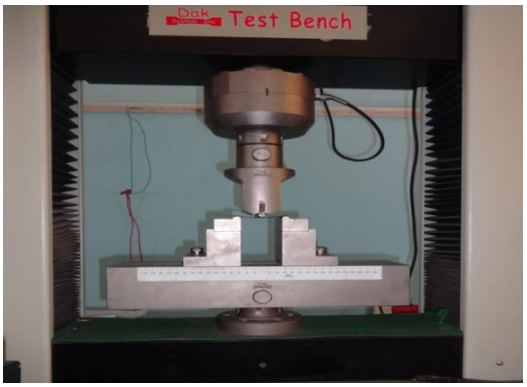

Fig. 7 Shows the flexural test setup

5. Discussion

Ultrasonic scanning was conducted on the coupons to identify the defects. In the twenty coupons fifteen is defect free in pure resin four coupons and in the CNF mat reinforced eleven coupons are defect free. The cavities and crack were identified on the surface of the pure resin and the CNF reinforced coupons as shown in table 1.

Table 1 Cavities and cracks present on pure epoxy and CNF reinforced coupons.

Coupon No.	Pure resin/ Observed Defect	CNF 0.005% Observed Defect	CNF 0.008% Observed Defect	CNF 0.015% Observed Defect
1	Defect free	Defect free	Cavity (2 mm)	Defect free
2	Defect free	Defect free	Crack (3 mm)	Defect free
3	Defect free	Defect free	Defect free	Defect free
4	Defect free	Cavity (4 mm)	Defect free	Defect free
5	Cavity (4 mm)	Defect free	Defect free	Cavity (2 mm)

5.1. Flexural testing

The flexural strength of the carbon nano fiber reinforced composite coupon was increased while increasing the carbon nano fiber contents a drastic improvement in the flexural strength. Since the excess reinforcement beyond the optimum level was increasing the porosity of the matrix, the percentage of the CNF is optimized as 0.015% as shown in the table 2 and figure 8.

Table:2 Strength of CNF reinforced composites at different ratios.

Coupon	Pure resin, Load in Kg	0.005% CNF, Load in Kg	0.008% CNF, Load in Kg	0.015% CNF, Load in Kg
1	6.4	7.8	6.3	12.1
2	7.4	8.4	7.8	11.2
3	6.3	8.0	9.1	12.5
4	5.9	6.9	8.9	14.0
5	5.2	8.4	9.4	9.0

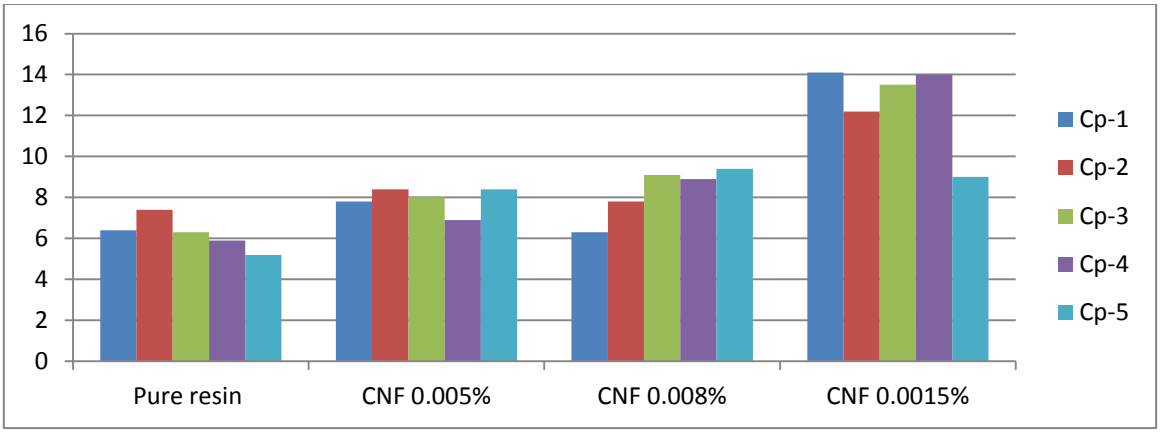

Figure 8. Variation in strength due to defect as well as the percentage of CNF

6. Conclusion

Based on the experimental observations, the result clearly indicates that the increasing CNF/PVA improve the flexural strength of the epoxy resin. In 0.015% of CNF concentration the fiber formations are randomly present. It has smooth edges nanofiber of diameter ranging from 450 – 1010 nm. The dispersion of CNF is visible with the contrast variation on the SEM image. The ultrasonic scanning method shows the cavity and crack present in pure resin and CNF reinforced coupons as shown in table 1, it reduces the mechanical strength. XRD shows that the PVA/Carbon nanofiber mats were converted to carbon nanofiber mat. This can suggest that the 0.015% CNF/PVA mat can give better enhancement strength of the regular epoxy resin. The more optimized mechanical strength, i.e., Flexural strength has been obtained in 0.015% of carbon nano fiber mat reinforcement. The outcome of this research work may provide the best platform for the nano composite manufacturers for determining the standards.

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