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Morphology and mechanical properties of PAN nanofiber mat

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Abstract. Nanofibers have acquired greater interest due to their vast variety of possible uses. Nanofibers offer several options to change things physically and chemically during or after the manufacturing process to give them new properties. To exploit the full potential of nanofibers, it is necessary to comprehend the link between the mechanical characteristics, particularly tensile strength, of a nanofiber mat and its morphology. Electrospinning is a rapidly developing polymer processing technology because it provides a simple and effective method for manufacturing nano continuous fibres. This method permits the deposition of nanofibers on revolving collectors. Rotating collectors, such as the drum and electrodes with a gap between them, may readily form oriented fibres. Polyacrylonitrile is a common precursor material for carbon nanofibers (PAN). This research investigates the impact of collector drum's rotation speed on the morphology of the nanofiber mat and discusses the mechanical properties of Polyacrylonitrile (PAN) electro spun nanofiber mats with precisely aligned nanofibers. PAN nanofiber mats have more tensile strength (~37 %) than PA6 nanofiber mats and have (50 %) less elongation than PA6 nanofiber mats, according to a comparison with previous studies.

1. Introduction

Nanofibers play an exceptional role in manufacturing owing to their unique qualities, which are capable of altering the physical and chemical properties throughout synthesis [1 - 4]. Electrostatic forces and solution parameters affect the structure and shape of the final outcome, whether it fibres or yarns, according to the electrospinning processes literature [5]. Morphology has a significant impact on the ultimate physical and chemical characteristics of electro spun nanofibers, which are predominantly determined by electrospinning parameters [6]. Among these are the polymer solution's molecular weight, volume, solution flow rate, viscosity, conductivity, surface tension, syringe-to-collector distance, ambient temperature, and humidity [7]. Nanofibers' strength increases as their diameter decreases [8]. In addition, highly oriented, flexible nanofibers with exceptional mechanical properties are in extremely popular for several applications [9]. However, the relationship between mechanical property changes and the orientation/alignment of nanofiber mats practically unexplored. It should be noted that highly aligned fibres are extremely important, for example, in the production of implants in the healthcare sector. Highly aligned fibres have the potential to increase the load-bearing capacity of the meniscus in the health sector [10].

Electrospinning used to manufacture nanofiber mats from polymers in solution and melt, as well as non-polymeric nano particles such as ceramics and metals [11,12]. Electrospinning enables the synthesis of thin fibres ranging in diameter from a few tens to several hundred nanometres from a variety of polymers and polymer mixes. Electrospinning is currently generally acknowledged as one of the most effective industrial nanofiber production technologies [13]. Fibre-reinforced composites are used in a



variety of industries: e.g., medicine antibacterial characteristics [14, 15], reinforced polymer composites [16–18], civil engineering construction [19–22]. Electro spun nanofibers offer a broad variety of applications due to their multiple advantages, including oil/water separation, air filtration, tissue engineering scaffolding, drug delivery [23], sensors [24], composites for load bearing [25–27] and advance structures [28,29].

Carbon fibre precursors comprised of Polyacrylonitrile (PAN) fibres with different properties are becoming very valuable [30]. PAN has a substantially higher viscosity than other polymer solutions. Acrylic fibres, also known as polyacrylonitrile (PAN) fibres, may be manufactured using gel or wet spinning. These fibres may be spun for use in textiles or carbonised in an inert environment to create carbon fibres [31]. Flexible polymer Polyacrylonitrile is used to produce oxidised PAN fibres, textile fibres, and filtration hollow fibres [32], including ultrafiltration membranes and ultrafiltration membranes [33,34]. Several of these important properties of PAN fibres prompted our investigation.

The purpose of this study is to investigate the nanofibers deposited on a rotating drum during the electrospinning procedure, which produces extremely aligned fibres. The morphology of the nanofiber mat generated in response to the speed of the rotating drum determines the mechanical characteristics of Polyacrylonitrile (PAN) nanofibers. In addition, the morphology and mechanical characteristics of PAN nanofibers are compared to those of PA6 obtained in past studies [11].

2. Experimental Method

2.1. Materials and Methodology

The Polyacrylonitrile (PAN) (C_3H_3N)_n average M_w 150,000 (Typical) granuels; CAS number 25014-41-9 (Sigma Aldrich chemical, Merch KGaA, Darmstadt, Germany) solution was prepared in N, N-Dimethylformamide (HCON (CH₃)₂) solvent, ACS reagent, >=99.8%; CAS number 68-12-2 (Sigma Aldrich chemical, Merch KGaA, Darmstadt, Germany). To prepare solution 15 wt/wt% PAN is mixed with solvent and mixed for 10 hours on Magnetic stirrer (Thermo Scientific™ Cimarec +™ Stirring Hotplates Series, USA) under + 80 °C (where room temperature was 22 ± 1 °C; moisture content, 60%). In the electrospinning setup (Fisherbrand™ Single Syringe Pump, a needle-based electrospinning machine, Danbury, (CT06811), USA), with a spinning-chamber temperature of 22 ± 1 °C and 36% relative humidity, using a 20 Ga needle, a rotating drum collector (Shenzhen Tongli Tech Co Ltd, (D-608) Shenzhen, China; Rotating Collector RC-5000, D140, L50) was used to electro spun the nanofiber mat at several rotating speed. Two hours of electrospinning were performed, the parameters are listed in Table 1.

Table 1. Process Parameters for solution preparation for electrospinning and boundary conditions for collector to run at various speeds.

Process parameters	Parameters used
Solution	PAN + N, N-Dimethylformamide
Polymer (wt/wt %)	15
Flow rate (ml/hr)	0.60
Tip to collector distance (cm)	25
Collector type	Rotating drum
Time of electrospinning (hours)	2
Speed of rotating drum (RPM)	300, 900 and 1800

Electrospinning is conducted for the same period at the rotating drum speeds of 300, 900, and 1800 revolutions per minute. After electrospinning specimens from the nanofiber mat were prepared for mechanical testing.

2.2. Tensile test of specimen

The 40 mm x 40 mm square piece of paper used to contain the nanofiber mat specimen. Figure 1(a) depicts the arrangement of test specimens and their standard test dimensions. The dimensions of the PAN nanofiber mat are 10 mm x 40 mm, and the specimen's edges are aligned with paper to ensure the specimen's safety and to provide optimal grip for the equipment used to hold the specimen.

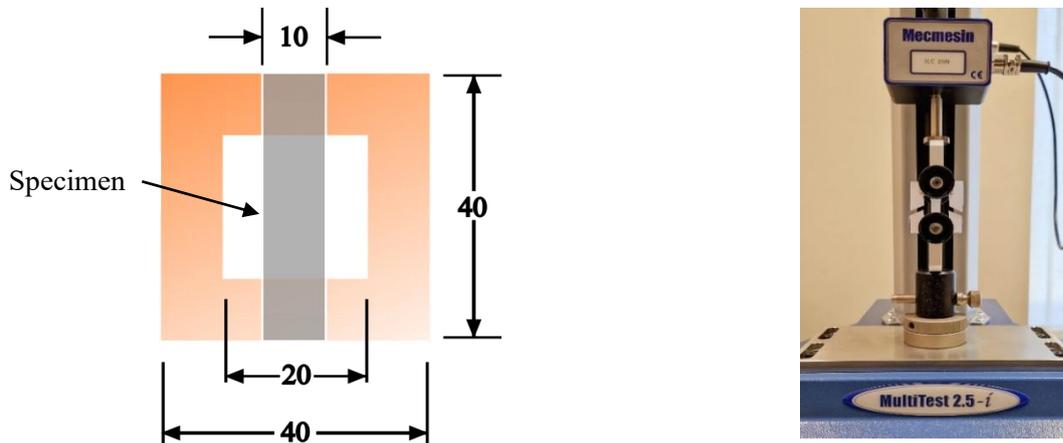


Figure 1. (a) Preparation of specimens for testing (b) Specimen placed on the Mecmesin's Multi-Test 2.5-i

After placing the paper containing the specimen onto the machine, the paper is cut using scissors and the machine is permitted to analyse the sample. The tensile testing equipment figure 1(b) demonstrates mechanical testing using Mecmesin's Multi-Test 2.5-i (The x-axis velocity was 1 mm/min).

3. Results

3.1. Results obtained from SEM

At a magnification of 1500, a vacuum of 10⁻² Torr, an ion coater operating at a current of 6 mA, a gold cover, and a coating thickness of 10 nm, images were captured using a Hitachi TM300 Tabletop Microscope SEM. Figures 2 shows the orientation of PAN nanofibers collected in the revolving drum at three independent speeds.

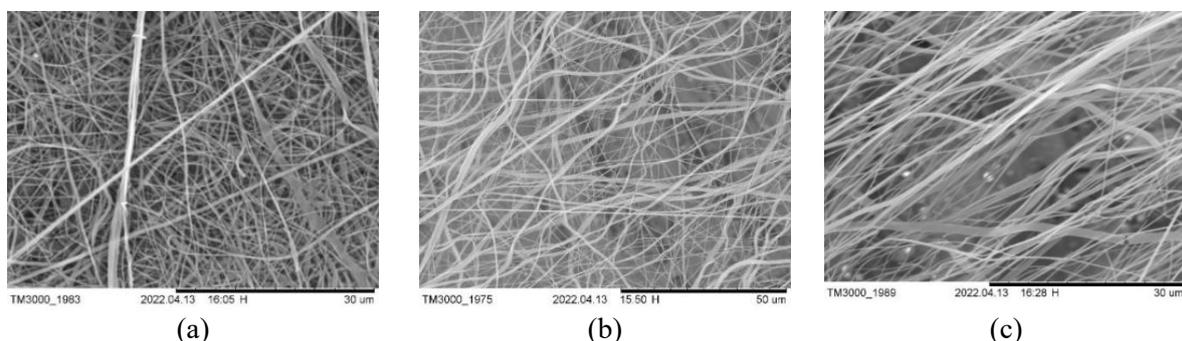


Figure 2. Morphology of nanofibers collected in rotating drum (a) speed 300 rpm; (b) speed 900 rpm; (c) speed 1800 rpm

As seen in the pictures above, the shape and alignment of the PAN Nano fibre mat clearly improve as the rotating drum speed increases. In the following section, the alignment statistics of nanofibers are discussed.

3.2. Statistics of alignment and diameter of nanofibers

The fibre alignment was quantified by applying the fast Fourier transform (FFT) to SEM images using ImageJ analysis software [35]. The statistical alignment of PAN Nano fibre mats at 3 different speeds are 300 rpm, 900 rpm and 1800 rpm are shown in figure 3, 4 and 5 respectively.

As seen in figure 3, the fibres are evenly scattered in all directions up to a maximum concentration of 0.0175. By increasing the drum's rotational speed to 900 RPM, the orientation of the nanofiber changed from 0 to 90 degrees. At 1800 RPM, nanofiber morphology reveals that all nanofibers are aligned between 15 and 20 degrees with the maximum amount of 0.076.

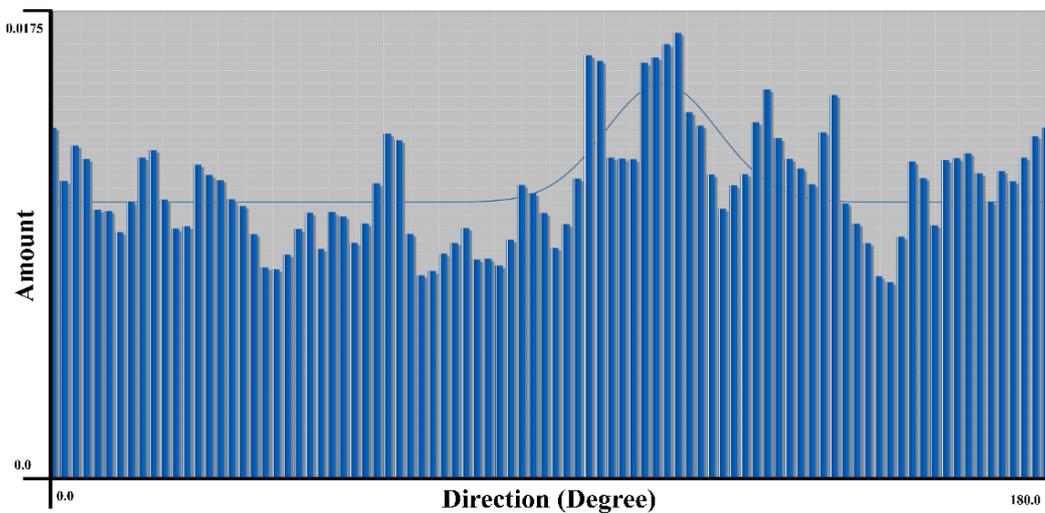


Figure 3. Alignment of PAN nanofibers at 300 RPM

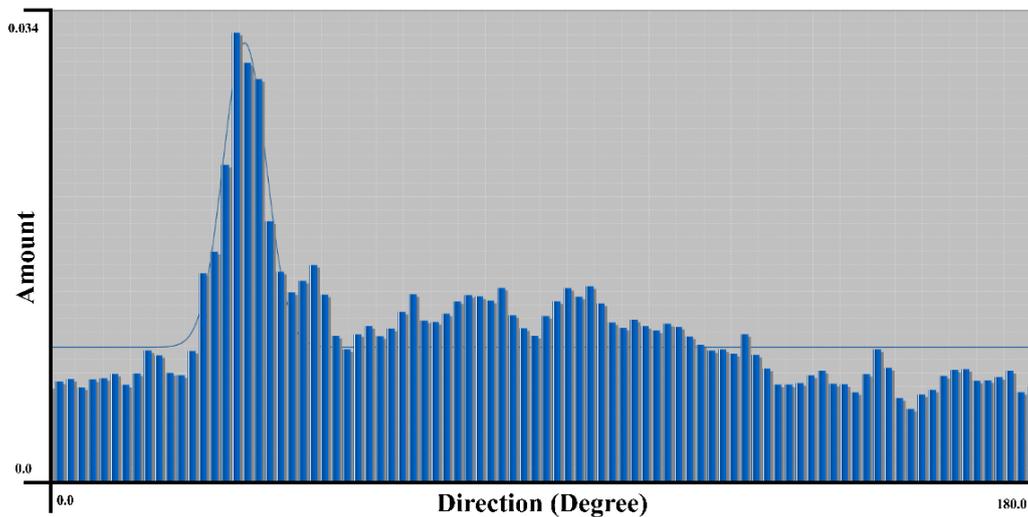


Figure 4. Alignment of PAN nanofibers at 900 RPM

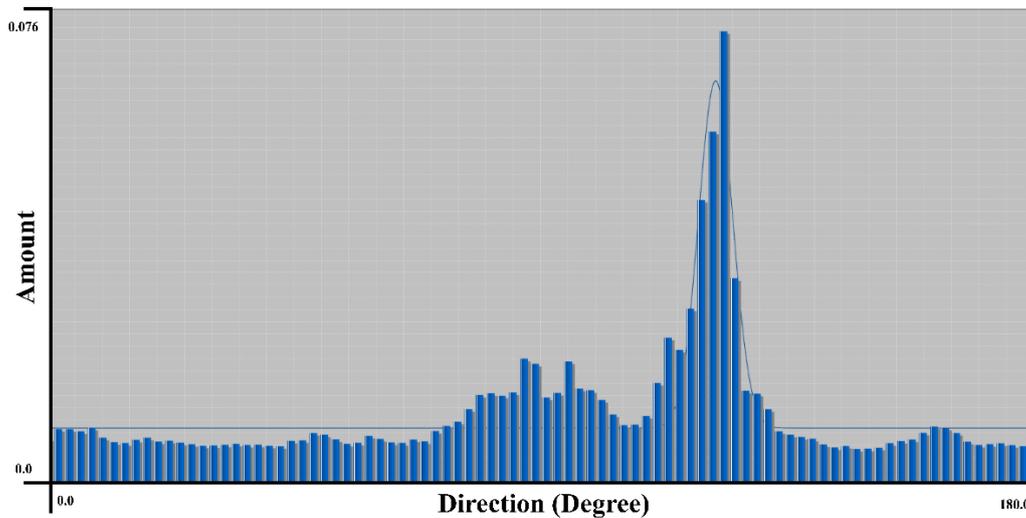


Figure 5. Alignment of PAN nanofibers at 1800 RPM

The average diameter of nanofiber was found by taking the diameter of 100 samples and measuring them with ImageJ. Figure 6 demonstrates the statistical distribution of nanofiber diameter at various speeds.

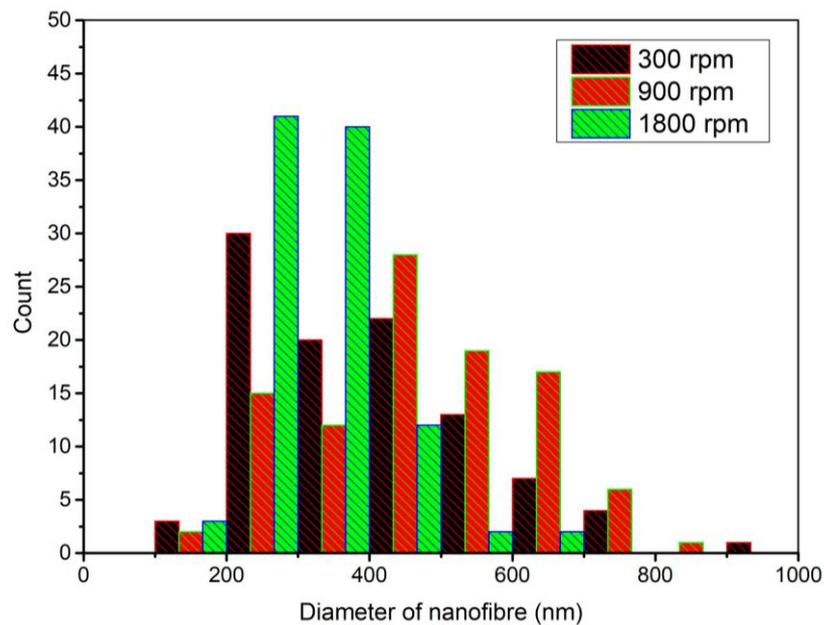


Figure 6. PAN Nanofiber diameter with varying drum rotation speeds

At a lower rotational speed of 300 rpm, the diameter of nanofibers ranges from 200 to 800 and is evenly distributed over this range. Increasing the speed of the revolving disc to 900 revolutions per minute produces a similar range of diameters, with the majority being around 400 nanometers. The highest speed of the rotating drum is 1800 rpm, the graph shows that there is a clear difference at higher speeds. Most of the nanofibers have a diameter between 200 and 400 nm, and very few are found below 200 nm (~3 %) or above 400 nm (~15 %). Increasing the speed of the spinning drum stretches nanofibers and enhances their diameter uniformity.

3.3. Result of tensile testing:

Figure 7 shows the findings of the force versus displacement examination of five samples (manufactured under 1800 rpm, which is a perspective membrane). The elastic modulus was calculated using a linear graph with a 0 to 0.1 displacement. The average young's modulus measured is $\sim 2.1 \pm 0.1$ GPa.

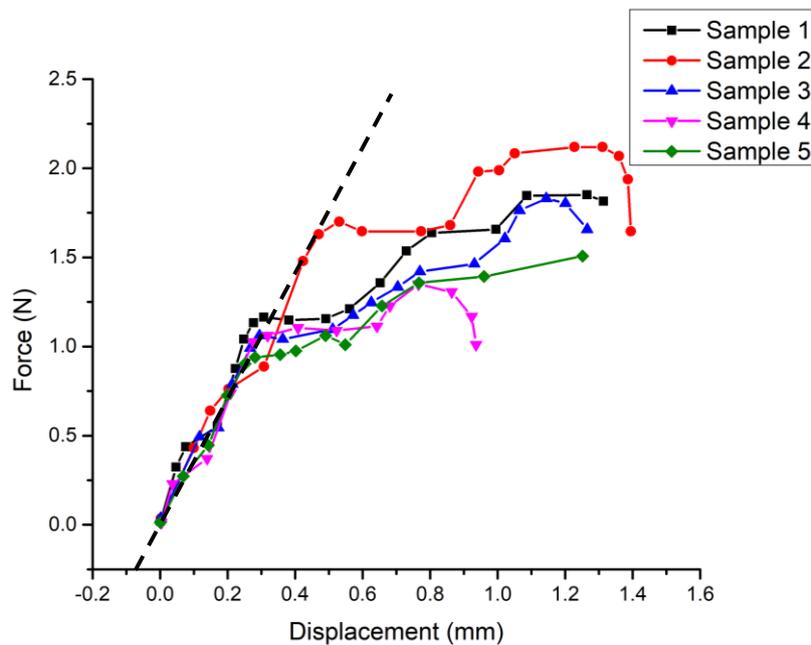


Figure 7. Force – Displacement curve for PAN nanofiber specimen

The average mean tensile strength of PAN nanofiber mat is 18.1 ± 2.9 MPa, and the average elongation is 2.6 ± 0.16 percent. PAN nanofiber mat offers 37.3% greater tensile strength and 50% less elongation than PA6 nanofiber mat [11]. Accordingly, the rigidity of PAN nanofiber mats is greater than that of PA6 nanofiber mats, giving PAN nanofiber mats enormous potential for usage in heavy composite structures and smart materials. Adding nanoparticles to a polymeric solution may have additional benefits depending on the nanoparticles added [12].

4. Conclusion

- Thus, was statistically evaluated the effect of rotating drum speed on nanofiber orientation; and strength of the perspective PAN membrane was measured.
- The ultramicroscopic image reveals that the rotational speed of the drum controls the nanofibers' alignment and shape. The fast rotation of the drum stretches the fibres, causing alignment and a decrease in diameter. This suggests that by modifying the collector speed, the alignment and mechanical characteristics of nanofibers might be improved.
- Comparing the mechanical characteristics and morphology of PAN nanofiber mat to those of PA6 nanofiber mat reveals that PAN nanofiber mat has 37.3 % more tensile strength, while elongation is decreased by 50 %.
- The findings of this research may be used to improve the electrospinning process. The examination and comparison of the unique mechanical behaviour of nanofibers presented in this paper will be valuable for a variety of applications and may assist in the development of the next generation of structural nanocomposites.

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