

DEVELOPMENT AND CHARACTERISATION OF BIAXIAL PAN NANOFIBER MATS

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This study addresses the challenge of enhancing the strength of polyacrylonitrile (PAN) nanofiber mats in the transverse direction, which is a critical aspect for filtration applications targeting micro-sized viruses and bacteria. Traditional methods of producing orientated nanofibers often result in mats with limited transverse strength. To overcome this, our research focused on developing biaxial nanofibers, which exhibit enhanced interbonding in both axial and transverse directions. Using a single-syringe electrospinning setup with a rotating drum collector, we successfully fabricated biaxially orientated PAN nanofiber mats. Scanning electron microscopy (SEM) analysis was employed to visualise and confirm the biaxial arrangement and porous structure of the mats. Mechanical testing further demonstrated that these biaxially arranged nanofibers possessed comparable strength in both orientations, signifying a significant advancement in the structural integrity of PAN nanofiber mats for filtration applications.

Keywords: *Aligned nanofibers, biaxial nanofibers, polyacrylonitrile (PAN), porosity.*

1. INTRODUCTION

Electrospinning emerges as a cost-effective technique for the manufacture of polymer fibres, characterised by diameters ranging from ten to several hundred nanometres [1]. This method has engendered a wide array of applications, notably in the realms of optical materials [2], sensor fab-

rication [3], nanocomposites [4]–[7], tissue engineering scaffolds [8], wound care, drug delivery systems [9], filtration technologies [10], protective clothing [11], and antibacterial textiles [5], [12], [13].

Polyacrylonitrile (PAN) nanofibers, produced through electrospinning, are fre-

quently employed as precursors for continuous carbon nanofibers via processes such as stabilisation and carbonisation. Additionally, these fibres are extensively used in filtration and catalysis. PAN nanofibers are particularly effective in various filtering scenarios, encompassing air and water purification, biofluid processing, and the extraction of toxic substances and deleterious contaminants from contaminated aqueous environments [14]. Because of their expansive surface area and electrostatic properties, PAN nanofibers are proficient at trapping airborne particles, such as dust and microbes, in air filtration systems. Similarly, in water filtration applications, they efficiently eliminate pollutants, including toxic agents, chemical compounds, and bacteria [15].

The biocompatibility and environmental safety of PAN nanofibers render them particularly suitable for medical and biological applications. For example, they can be used as filtering materials on dialysis machines, in wound dressings [16], or as scaffolds for tissue engineering [17]. PAN nanofibers have numerous advantages over traditional filtration media, establishing their utility in a wide spectrum of filtering applications [18].

2. MATERIALS AND METHODOLOGY

In the fabrication of biaxial nanofibers, polyacrylonitrile (PAN) with an average molecular weight of 150,000 (typical) and a CAS number of 25014-41-9 was used. The solvent used for this process was N, N-Dimethylformamide (DMF), which has an average molecular weight of 73.10 g/mol, designated as ACS Reagent (solvent) with a purity of $\geq 99.8\%$, and a CAS number of 68-12-2. This solvent was obtained from Sigma Aldrich Chemicals, Germany.

However, a notable challenge with conventionally aligned nanofiber mats lies in their limited strength in the transverse direction. These mats predominantly bear loads along the direction of the nanofibers, resulting in increased susceptibility to particle penetration. This limitation significantly undermines their effectiveness in filtration applications [19].

To address this issue, we introduce a novel method for producing biaxially arranged nanofiber mats at a laboratory scale. The approach involves the preparation of orientated nanofiber mats which are subsequently sectioned into smaller pieces. These segments then undergo a secondary electrospinning process to create an additional aligned layer, culminating in a biaxial arrangement. The fabricated nanofiber mats were subjected to a rigorous evaluation, including mechanical testing, analysis of fibre orientation, and scanning electron microscopy (SEM) analysis. This comprehensive investigation aims to determine the efficacy of the biaxial nanofiber configuration in enhancing the mechanical properties and, as a consequence, for the subsequent enhancement of the filtration efficiency of PAN nanofiber mats.

The electrospinning process was carried out as previously described by the authors [19]–[21], with a notable modification. Following the production of a 40 cm x 5 cm nanofiber mat, it was segmented into 5 cm x 5 cm squares. These sections were then rotated 90 degrees and reattached to the rotating drum to facilitate the formation of a secondary layer of nanofibers, thus achieving a biaxial configuration. The entire procedure of biaxial nanofiber fabrication is illustrated in Fig. 1.

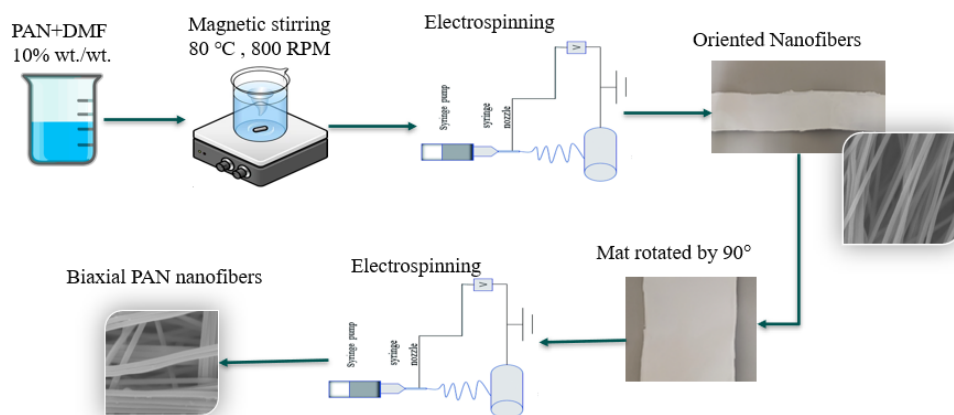


Fig. 1. Fabrication process of orientated and biaxial nanofibers.

For the acquisition of scanning electron microscopy (SEM) images, a Hitachi TM300 tabletop SEM was used. This instrument was set to a magnification of 1500, a vacuum of 10^{-2} Torr, and an ion coating current of 6 mA. The nanofibers were coated with a layer of gold (Au) with a thickness of 150 Å to enhance image quality. The orientation and porosity of the fibres were analysed using the OrientationJ plug-in for ImageJ software (ImageJ, National Institutes of Health, Bethesda, MD, USA). Contrast enhancement was applied to the SEM images to facilitate clearer observation. The mean diameter and standard deviation of the nanofibers were determined by measuring the diameter of 100 randomly selected nanofibers from three different SEM images.

Tensile properties were measured using

a Mecmesin Multi-Test 2.5-i tensile testing machine equipped with a 25-N sensor (PPT Group UK Ltd., t/a Mecmesin, Newton House, Spring Copse Business Park, Slinfold, UK). Samples were conditioned at room temperature according to ISO 139:1973, “Standard Environments for Conditioning and Testing”. This standard specifies a temperature of 21 ± 1 °C, a relative air humidity of 60 %, and an atmospheric pressure of 760 mm Hg. The dimensions of the test samples were 50 mm \times 10 mm (length \times width), according to ASTM D882-18. A total of five measurements were made to ascertain the tensile properties of the nanofiber mats. The thickness of these mats was measured using a digital micrometre (range: 0–25 mm; Digimatic micrometre, MDC-25PX, code No. 293-240-30, serial No. 71912410, Mitutoyo, Japan) [19].

2. RESULTS AND DISCUSSION

Scanning electron microscopy (SEM) images presented in Figs. 2a and 2b depict the microstructure of polyacrylonitrile (PAN) nanofibers, both in their orientated and biaxial configurations. These images

unequivocally demonstrate that by rotating the initial nanofiber mat and subsequently performing a secondary electrospinning process, it is feasible to fabricate biaxial nanofibers.

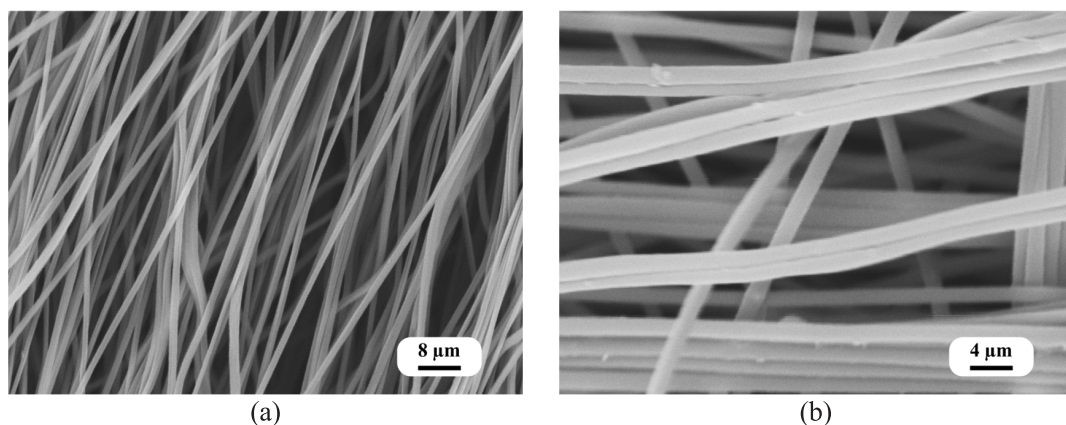


Fig. 2. SEM images of (a) oriented and (b) biaxial PAN nanofibers.

Figure 3 illustrates the diameter distribution of the nanofibers. Analysis of the SEM images of uniaxial and biaxial nanofiber samples revealed that the average diameter of the nanofibers was 512 nm, with a

standard deviation in a normal distribution of ± 95 nm. In certain instances, the nanofibers exhibited a minimum diameter of 325.045 nm and a maximum diameter of 805.713 nm.

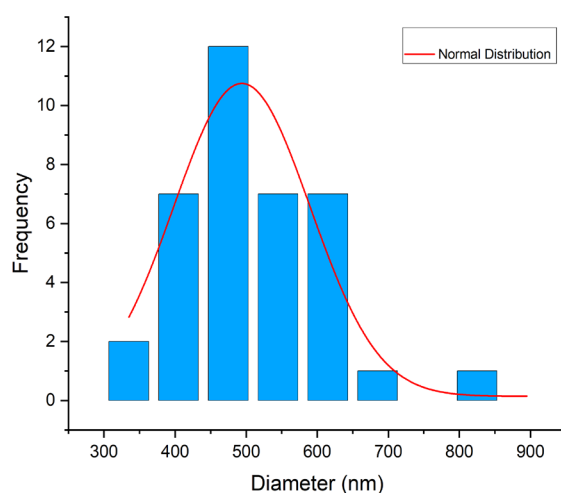


Fig. 3. Distribution of the diameter of nanofibers.

Porosity, indicative of the spaces within the nanofiber structure, is quantified as the ratio of the volume of voids to the total volume of the sample, typically expressed as a percentage. Porosity analysis revealed that uniaxial nanofibers exhibited a higher porosity of 52.7 %, in contrast to the 38.3 % observed in biaxial nanofibers. This differ-

ence is attributable to the single-axis orientation of the uniaxial fibres, leading to larger voids, while the biaxial fibres, aligned along the X and Y axes, exhibit reduced porosity.

Figures 4a and 4b present the orientation of nanofibers in uniaxial and biaxial arrangements. The orientation analysis indicated that the uniaxial fibres predomi-

nantly displayed angles within a maximum variation of 10 degrees, while the biaxial fibres exhibited orientation within a range

of -1 to +1 degrees, perpendicular to the orientated fibres.

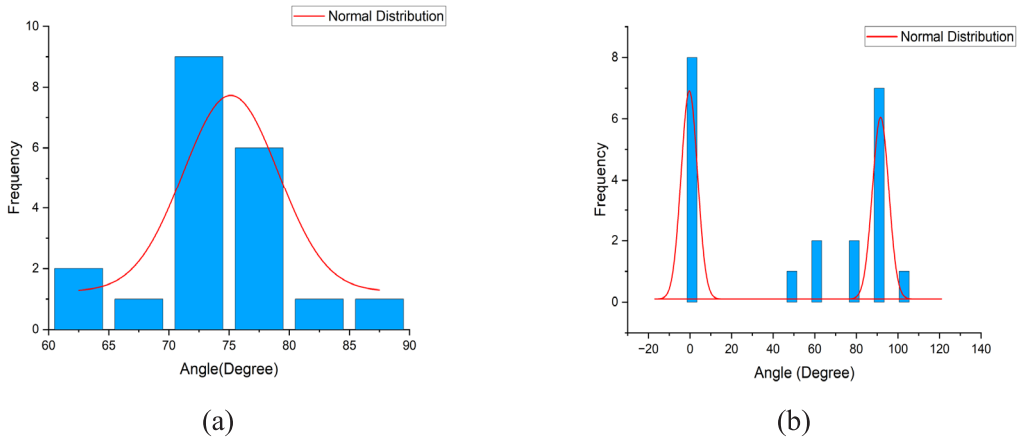


Fig. 4. Orientation of (a) aligned fibres and (b) biaxially arranged fibres.

Figure 5 shows the representative stress-strain curves for oriented and biaxially arranged nanofibers. The elastic modulus of the uniaxially oriented nanofibers was determined to be 524 ± 15 MPa, while for the biaxial nanofiber mats, it was 343 ± 12 MPa. The elongation at break was recorded at

12 ± 1 % for oriented fibres and 8 ± 1 % for biaxial fibres. The maximum tensile strength for uniaxial and biaxial fibres was found to be 11 ± 1 MPa and 8 ± 1 MPa, respectively. The summary of the mechanical properties of orientated and biaxial nanofibers is shown in Table 1.

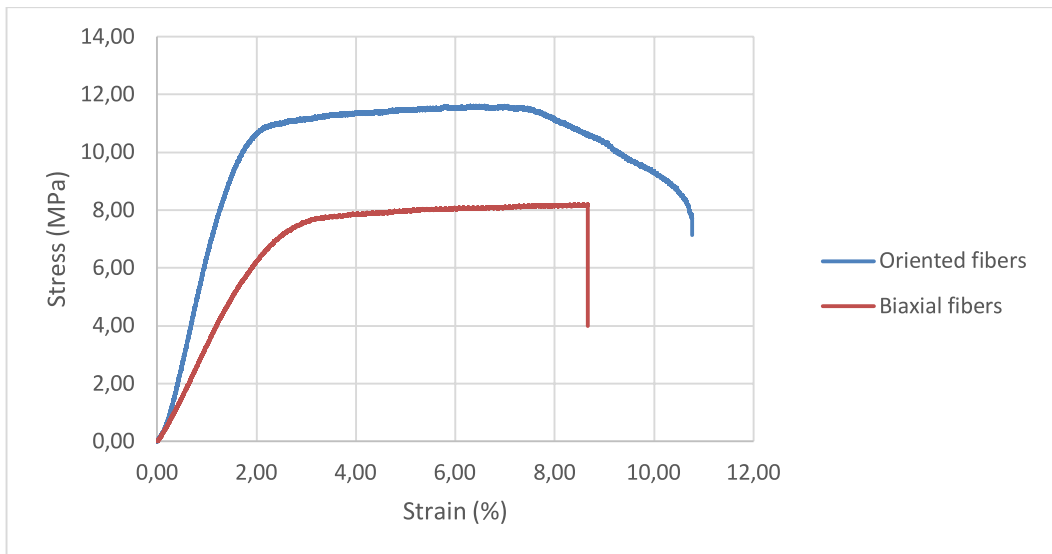


Fig. 5. Representative stress-strain curve of orientated and biaxial nanofibers.

Table 1. Summary of the Mechanical Properties of Orientated and Biaxial Nanofibers

	Thickness (μm)	Elastic modulus (MPa)	Ultimate tensile strength (MPa)	Elongation at break (%)
Nanofiber mat with oriented nanofibers	112 \pm 8	524 \pm 15	12 \pm 1	11 \pm 1
Nanofiber mat with biaxial nanofibers	225 \pm 10	343 \pm 12	8 \pm 1	8 \pm 1

Theoretically, the mechanical properties of biaxially arranged nanofibers would be approximately half of those observed in uniaxially aligned fibres. However, the findings of this study indicate a slight deviation from this expectation, with the properties being marginally above half. This discrepancy could be attributed to the phase in which the nanofibers are collected during electrospinning. The SEM analysis

confirms that the fibres are not collected in a liquid phase. The observed mechanical properties suggest that some fibres are collected in a semi-solid state, leading to their fusion with the pre-existing-oriented nanofibers. This interaction could potentially contribute to the enhanced mechanical properties observed in biaxially arranged nanofiber mats.

3. CONCLUSIONS

This study has successfully demonstrated the feasibility of fabricating biaxially arranged polyacrylonitrile (PAN) nanofiber mats using an innovative electrospinning technique. This method involved the rotation and subsequent layering of electrospun nanofiber mats, resulting in a biaxial fibre arrangement. The comprehensive analysis conducted through scanning electron microscopy (SEM), mechanical testing, and porosity evaluation has yielded insightful findings relevant to the structural and functional attributes of these nanofiber mats.

SEM analysis provided a clear visual representation of the nanofibers in both their uniaxial and biaxial orientations. This image was pivotal in confirming the successful creation of biaxial nanofibers and their distinct morphology. The measured diameters of the nanofibers indicated reasonable uniformity with a moderate range of variance, thereby ensuring consistent fibre quality.

Porosity analysis revealed that the biaxial nanofibers exhibited a lower porosity percentage compared to their uniaxial counterparts. This result can be attributed to the dual-axis alignment in biaxial fibres, which effectively reduces void spaces, making them denser and potentially more suitable for applications requiring finer filtration capabilities.

The mechanical properties of the nanofiber mats, as assessed through tensile testing, indicated that the biaxially arranged nanofibers possess a mechanical strength slightly higher than theoretically predicted. Although the uniaxial nanofibers showed higher values in terms of elastic modulus, elongation at break, and ultimate tensile strength, the biaxial fibres demonstrated respectable performance, considering their structural complexity. This unexpected enhancement in mechanical properties can be partly attributed to the phase in which the nanofibers were collected during electrospinning, with some fibres potentially

being in a semi-solid state, leading to fusion with the preexisting-orientated nanofibers.

In conclusion, the development and characterisation of biaxial PAN nanofiber mats has opened new avenues in the field of nanofiber technology. The unique structure of these biaxially arranged nanofibers holds significant promise for a variety of applications, particularly in filtration and

other fields where enhanced mechanical strength and reduced porosity are desirable. Future studies may focus on optimising the fabrication process to further improve the mechanical properties and functional performance of these biaxial nanofiber mats, potentially expanding their applicability in both industrial and medical domains.

ACKNOWLEDGEMENTS

This research/publication has been supported by the Doctoral Grant programme of

Riga Technical University.

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