LATVIAN JOURNAL OF PHYSICS
AND TECHNICAL SCIENCES
2024, N 5

DOI: 10.2478/lpts-2024-0038

OPTIMISATION OF ELECTROSPINNING PARAMETERS FOR HIGH-STRENGTH ORIENTED PAN NANOFIBRE MATS

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This study explores the optimisation of electrospinning parameters to enhance the strength and orientation of polyacrylonitrile (PAN) nanofibre mats, which are pivotal in filtration applications and other areas requiring high-strength, uniformly distributed nanofibres. The focus is on the electrospinning process, particularly on a rotating drum collector, which is instrumental in determining the morphology of nanofibres. Key process variables, including the distance between the collector and the syringe, feed rate, applied voltage, and polymer concentration, have been meticulously examined to understand their influence on the physical properties of PAN nanofibres. Our findings indicate that parameters such as voltage, collector-to-syringe distance, and drum rotation speed (RPM) significantly impact the mechanical performance of the nanofibre mats. The optimised electrospinning conditions produced PAN nanofibre mats with tensile strengths ranging from 1.61 MPa to 21.78 MPa, demonstrating a notable improvement in strength due to controlled fluid dynamics and expulsion of polymer solution during the process.

Keywords: *Aligned nanofibres, optimisation, PAN nanofibre mat, response surface methodology.*

1. INTRODUCTION

Electrospinning emerges as a cost-effective methodology for the production of nanofibres [1]. This technique involves the collection of nanofibres in various ways; for instance, when using a flat plate collector, the structure of the nanofibres tends to be randomly orientated. In contrast, in drum-rotating collectors, nanofibres are typically characterised by higher strength and are orientated in a singular direction [2].

Polyacrylonitrile (PAN) nanofibres have garnered considerable attention due to their wide range of applications in filtration [3], biomedical engineering, drug delivery [4], scaffolding, nanocomposites [5], [6], antibacterial textiles [7]–[9], and protective clothing [10]. In such applications, the strength of the nanofibres is of paramount importance [11].

The collection of nanofibres on a flat plate collector presents fewer variables to control, as the fibres are deposited randomly, necessitating primarily the prevention of bead formation during electrospinning. However, the scenario changes significantly when nanofibres are electrospun onto a rotating drum. A critical variable in this context is the speed of the rotating drum, along with other parameters that become consequential in relation to the drum speed. The electrospun nanofibres may be collected in a semisolid or solid state, necessitating the avoidance of bead formation.

Thus, it becomes imperative to optimise the electrospinning process parameters to achieve high-strength nanofibre mats when collected on a rotating drum.

Table 1 encapsulates a summary of the literature concerning various methods of collecting nanofibres and the parameters used there. A notable variation in the choice of parameters is evident from this summary.

Table 1. Summary of Electrospun Process Parameters in the Fabrication of Nanofibre Mats

Collector type	Voltage (kV)	Distance (cm)	Speed of RD RPM	Applications	Reference
drum	15	10	400	Wastewater filtration	[12]
drum	18	15	600	Textile composites reinforced	[13]
drum	16	18	800	Gas toluene removal	[14]
drum	16	15	500	Air gap membrane distillation	[15]
roller	12	20	500	Super-capacitors	[16]
roller	15	18	800	Hybrid nanofibre filtration	[17]
drum	20	10	1000	Future high-power target	[18]
drum	17	15	1200	Wound healing	[19]
roller	16	10	1000	Super-capacitor	[20]
drum	15	20	400	Dust removal	[21]
roller	20	20	400	Non-crimping laminated	[22]
drum	18	18	1200	Textile composites reinforced	[23]
drum	20	20	1200	Annealed PAN nanofibres	[11]
drum	16	15	600	Battery	[24]
drum	20	10,15,20	500	Carbonized nanofibres	[25]
roller	14	20	800	Creating novel metal	[26]
drum	17.5	10	400	Orientated ferromagnetic Ni1-xFex nanofibres	[27]
roller	10	15	800	Enantioselective material	[28]
drum	11	10	600	Junction-free electrospun nanofibre network	[29]
drum	20	10	1000	Anti-adhesion barrier	[30]
drum	18	10, 20	1200	Food packaging	[31]

The literature reveals a broad spectrum of process parameters that are used for the production of nanofibres. In this context, six process parameters have been identified as critical in the electrospinning process. Among these, three are considered primary due to their significant impact on the quality of the nanofibres: voltage, collector drum distance, and drum speed. Secondary parameters – flow rate, needle gauge, and polymer viscosity (wt / wt % of PAN polymer) – are considered interrelated. Alterations in the needle gauge or polymer viscosity are likely to proportionally impact the flow rate, thus warranting a holistic approach to optimising these parameters. Therefore, in this research, these secondary parameters are kept constant to maintain consistency and focus on the primary variables.

2. MATERIALS AND METHODOLOGY

The present study focusses on elucidating the effects of the electrospinning process parameters on the ultimate tensile strength of orientated PAN nanofibres. The PAN nanofibres were synthesised using PAN powder and N,N-dimethylformamide (DMF). Polyacrylonitrile (average molecular weight: 150,000; CAS number: 25014-41-9) and N,N-dimethylformamide (DMF; ACS reagent (solvent), purity ≥ 99.8%; CAS number: 68-12-2) were obtained from Sigma-Aldrich Chemicals (Merck KGaA, Darmstadt, Germany).

The electrospinning procedure was conducted according to the methodology previously established by the authors [11], [23], [32]. Figure 1 delineates the schematic of the electrospinning setup used in the Mechanics and Biotextile Research Laboratory at Riga Technical University.

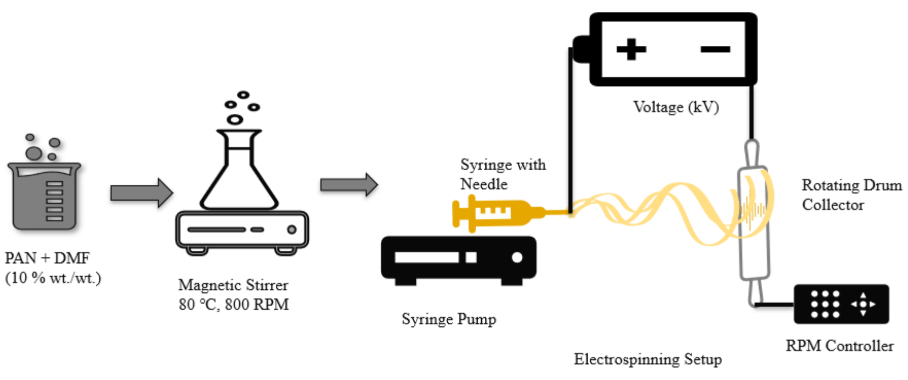

Fig. 1. Schematic diagram of the electrospinning setup.

For the optimisation of the process parameters, the range was chosen based on a comprehensive review of the literature as detailed in Table 1.

Table 1. Range of Process Parameters for Optimisation

Process Parameters	Parameters Used
Solution	PAN + N, N- Dimethylformamide
Polymer, wt/wt % (constant)	10
Needle size ml/hr (constant)	18
Flow rate, ml/hr (constant)	1
Tip to collector distance, cm	5, 10 and 15
Applied voltage, kV	15, 17.5, 20
Speed of rotating drum, RPM	400, 800, 1200

Response surface method (RSM) was employed as a statistical technique to optimise the electrospinning process, which involves analysing the relationship between the electrospinning settings and the ultimate tensile strength. Response surface methodology was implemented using the Design Expert software, and experiments were designed using the Box-Behnken. Seventeen experiments were designed as summarised in Table 2.

Table 2. Design of Experiments

No.	PAN + DMF (10 % wt./wt.)	Needle Size (Ga)	Flow rate of the solution (ml/hr)	Voltage (kV)	Needle to drum distance (cm)	Speed of rotating drum (RPM)
1.	10	18	1	15	15	400
2.	10	18	1	20	15	1200
3.	10	18	1	17.5	10	1200
4.	10	18	1	17.5	15	800
5.	10	18	1	15	10	800
6.	10	18	1	17.5	15	800
7.	10	18	1	17.5	15	800
8.	10	18	1	17.5	5	1200
9.	10	18	1	20	5	800
10.	10	18	1	17.5	15	800
11.	10	18	1	20	10	800
12.	10	18	1	20	15	400
13.	10	18	1	15	15	1200
14.	10	18	1	17.5	15	800
15.	10	18	1	17.5	10	400
16.	10	18	1	17.5	5	400
17.	10	18	1	15	5	800

All PAN nanofibre mats were produced as detailed in Table 2. 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). The samples were conditioned and stored at room temperature according to ISO 139:1973 "Standard Environments for Conditioning and Testing", specifying a temperature of 21 ± 1 °C, a relative humidity of 60 %, and an atmospheric pressure of 760 mm Hg. The sample dimensions were in accordance with ASTM D882-18, with a size of 50 mm × 10 mm (length × width). Five measurements were made to determine the tensile properties. The thickness of the nanofibre mats was gauged using a digital micrometre (range: 0–25 mm; Digimatic micrometre, MDC-25PX, code No. 293-240-30, serial No. 71912410, Mitutoyo, Japan). A 50 mm × 40 mm paper template with an inside cut of 30 mm × 20 mm was used. Both ends of the sample were adhered to the paper template using double-sided thin scotch tape (3M Scotch Magic Tape (Matte Finish) 3/4" × 36-yard Desk Dispenser refills). After attaching the paper and sample to the tensile testing apparatus, the sides of the paper template were excised with scissors, as defined in the methodology [5], [11].

3. RESULTS AND DISCUSSION

The experimental results of 17 distinct runs were meticulously analysed. The findings pertaining to the ultimate tensile strength (UTS) of the electrospun PAN nanofibres are summarised in Table 3.

Table 3. Summary of the Ultimate Tensile Test Results Obtained from the 17 Experiments

	Factor 1	Factor 2	Factor 3	Response 1
	A: Voltage	B: Distance	C: Rotation	UTS
Run	kV	cm	RPM	MPa
1	15	15	400	1.74
2	20	15	1200	2.5
3	17.5	10	1200	5.11
4	17.5	15	800	2.55
5	15	10	800	4.64
6	17.5	15	800	2.45
7	17.5	15	800	2.64
8	17.5	5	1200	17.45
9	20	5	800	10.36
10	17.5	10	800	2.56
11	20	10	800	8.63
12	20	15	400	3.04
13	15	15	1200	1.61
14	17.5	15	800	2.53
15	17.5	10	400	16.08
16	17.5	5	400	21.78
17	15	5	800	15.92

The evaluation of the primary responses reveals significant insights. For example, when the voltage was set at 15 kV with a 15 cm distance between the collector and spinneret and a drum rotation speed of 400 RPM, the UTS of the PAN nanofibre mat was 1.74 MPa. On the contrary, increasing the voltage to 20 kV and the drum rotation speed to 1200 RPM, while maintaining a constant distance of 15 cm, resulted in an increase in UTS to 2.50 MPa. This suggests that adjustments in voltage and drum rotation, with a fixed distance, can significantly enhance the tensile strength of PAN nanofibre mats.

Further analysis shows that when the voltage was maintained at 17.50 kV but the distance and rotation were adjusted, there was a noticeable impact on the tensile strength. For example, a 50 % reduction in tensile strength was observed when the distance was increased to 16 cm with a reduction in the drum rotation speed to 400 RPM. This indicates that the rotational speed of the drum has a more pronounced impact on the tensile strength of the PAN nanofibre mats compared to the distance parameter.

In the eighth experimental run, the tensile strength was maximised to 17.45 MPa with a decrease in distance and an increase in voltage, alongside an optimised drum rotation speed. Furthermore, in the 15th, 16th and 17th runs, as the applied voltage was increased and the distance and drum rotation speed were decreased, the tensile strength improved markedly, reaching 21.78 MPa. These findings underscore the significant influence of voltage and rotation speed on the tensile strength of PAN nanofibre mats.

The statistical analysis of the model is presented in Table 4, which provides information on the sequential p-value, the lack of fit p-value, adjusted R^2, and predicted R^2.

Table 4. Statistical Summary of the Model

Source	Sequential p-value	Lack of fit p-value	Adjusted R^2	Predicted R^2	
Linear	**<0.0001**	**<0.0001**	**0.7868**	**0.9602**	**Suggested**
2FI	0.5831	<0.0001	0.7709	0.2798	
Quadratic	0.1067	<0.0001	0.8559	-2.0389	
Cubic	<0.0001		0.9999		**Aliased**

Figure 2 represents a model that integrates the coded factors of the electrospinning process and the corresponding results obtained from the experimental runs. This figure is crucial in visualising the relationships and interactions between the various parameters studied and their impact on the ultimate tensile strength of the PAN nanofibres. It likely includes graphical representations, such as response surface plots or contour plots that depict how changes in the electrospinning parameters influence the tensile strength. This visual aid is instrumental in providing a clear and concise understanding of the experimental data and facilitating the interpretation of complex relationships between the coded factors and the resultant properties of the nanofibre mats.

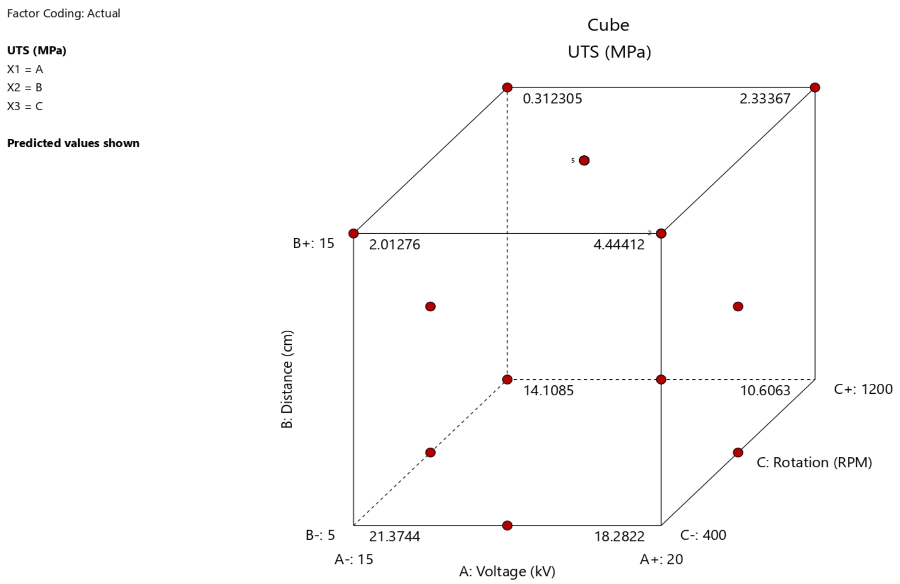

Fig. 2. Model representation with coded factors and obtained results.

Visual analysis (Fig. 3) suggests that optimising the distance between the collector and spinneret, coupled with a reduction in applied voltage, leads to an increase in the surface area of the PAN nanofibre mats. The optimal values were identified at a voltage of 15 kV and 5 cm distance, signifying that the optimisation of distance and voltage enhances the tensile strength of the PAN nanofibre mats.

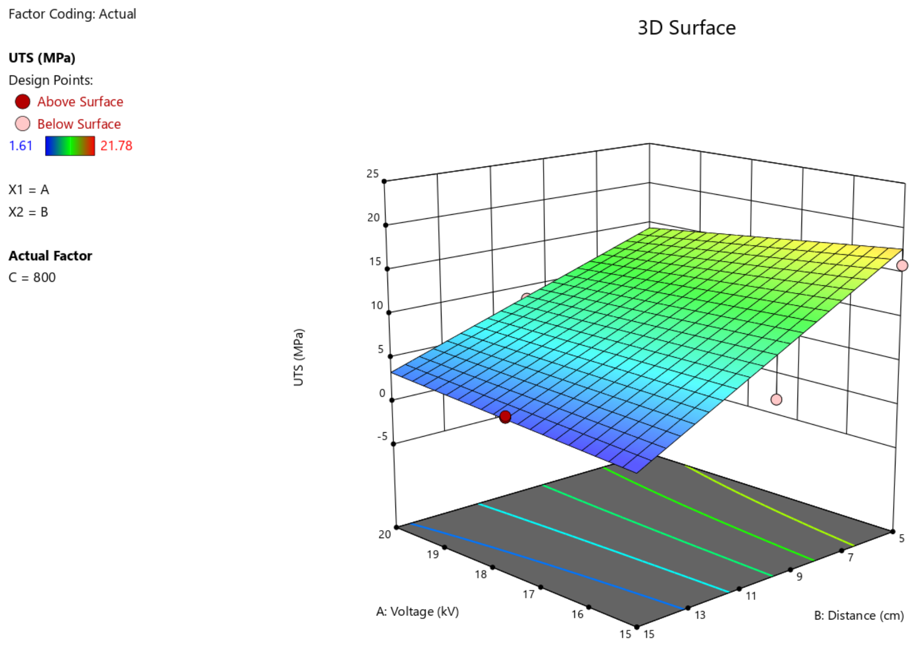

Fig. 3. 3-D visual of the influence of changes in electrospinning parameters on the surface area and ultimate tensile strength of PAN nanofibre mats.

Figure 4 illustrates the intricate relationship between the applied voltage, the distance of the rotating drum collector, and their collective impact on both the desirability function and the ultimate tensile strength (UTS) of the PAN nanofibres. This figure is pivotal for understanding how variations in these key electrospinning parameters influence the final quality and mechanical properties of nanofibre mats.

The graph presents a combination of response surfaces or line graphs, detailing how adjustments in voltage and drum distance affect desirability – a metric often used to gauge the overall effectiveness and efficiency of process parameters – and the UTS, a critical measure of the strength of the nanofibre mat. Such a visual representation is invaluable for identifying optimal parameter settings and understanding the trade-offs involved in the electrospinning process. It helps clarify the point at which the parameters converge to maximise both the desirability and the mechanical strength of the nanofibres, thus providing a comprehensive overview of the parameter optimisation process.

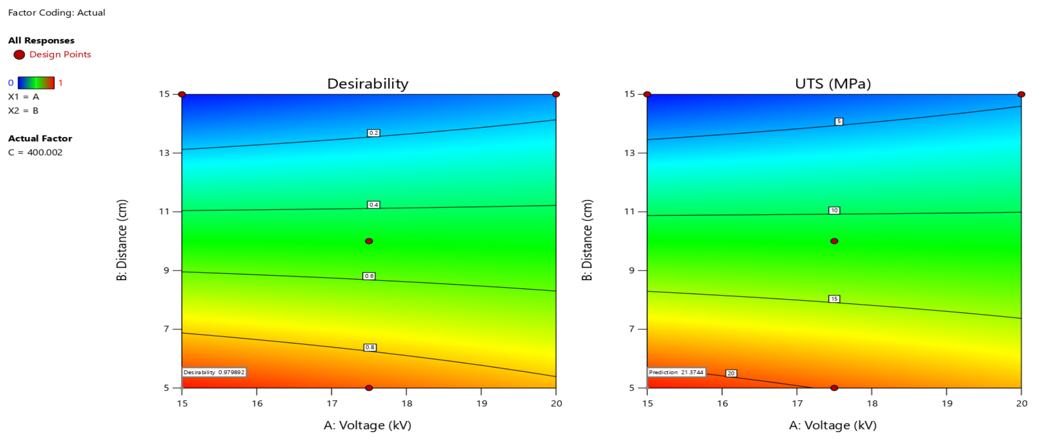

Fig. 4. Graphical representation of desirability and UTS in relation to voltage and rotating drum collector distance.

Equation (1) integrates the coded factors such as voltage (coded A), distance between the spinneret and the collector (coded as B), and the speed of the rotating drum (coded as C). It could be a polynomial or another form of regression equation that best fits the experimental data. The coefficients in the equation represent the influence of each factor and their interactions on the UTS of the nanofibre mats.

$$UTS = +9.18 - 0.2677\,A - 6.91\,B - 2.34\,C + 1.38\,AB - 0.1025\,AC + 1.39\,BC. \qquad (1)$$

Equation (1) represents the final predictive model in terms of coded factors. This equation represents the quantified relationship between the coded electrospinning parameters and their collective impact on the ultimate tensile strength of the PAN nanofibres, serving as a crucial predictive tool for determining optimal process conditions.

CONCLUSIONS

The comprehensive study undertaken to optimise the electrospinning parameters for the production of high-strength-orientated polyacrylonitrile nanofibre mats has yielded pivotal insights, underpinned by the extensive analysis of experimental data. The meticulous exploration of key parameters – applied voltage, distance between collector and spinneret, and the speed of the rotating drum – has culminated in a nuanced understanding of their collective impact on the ultimate tensile strength (UTS) of the nanofibres.

Figures 3 and 4 serve as crucial visual aids, illustrating the intricate relationship between these electrospinning parameters and the mechanical properties of the PAN nanofibre mats. Figure 3 effectively maps the interaction of the coded factors with the UTS, providing a clear graphical representation of the complex interplay at work. Figure 4 further delves into the correlation between the parameters and both desirability and UTS, offering a comprehensive view of how varying the electrospinning parameters can optimise the quality and strength.

Equation (1), the final predictive model derived from the study, encapsulates the essence of these findings. It quantitatively represents the relationship between the coded electrospinning parameters and the UTS of the PAN nanofibres. This equation stands as a testament to the robust statistical methodology employed, offering a predictive tool that can significantly streamline the process of determining optimal electrospinning conditions.

In conclusion, this research not only enhances our understanding of the electrospinning process, but also significantly contributes to the field of nanotechnology, particularly in the production of high-strength nanofibre mats. The optimisation of electrospinning parameters, as delineated in this study, has crucial implications for various industrial applications where the strength and quality of nanofibre mats are paramount. The insights gained from this research hold a substantial value for future academic and industrial pursuits, paving the way for advancements in nanofibre fabrication technology and its myriad applications.

ACKNOWLEDGEMENTS

The research has been supported by the Doctoral Grant programme of Riga Technical University.

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