

DECOUPLING FIBRE DIAMETER FROM FIBRE ORIENTATION: COLLECTOR ROTATION SPEED AS AN INDEPENDENT CONTROL VARIABLE IN ELECTROSPINNING, VALIDATED AGAINST KRENCHEL'S ORIENTATION MODEL

Shohruxmirzo Mahsudov a,*

Sherzod Korabayev a

Fayzulla Bekchanov b

Bexruzбек Xamidullayev a

Orif Alimov c

^a Namangan State Technical University, Namangan, Uzbekistan

^b Kimyo International University in Tashkent, Tashkent, Uzbekistan

^c Jizzakh Polytechnic Institute, Jizzakh, Uzbekistan

* Corresponding author. E-mail: shohruxmahsudov@gmail.com

Abstract

Collector rotation speed is widely used to align electrospun fibres, but whether it also alters fibre diameter is usually judged from mean values without testing the difference statistically, so that the two effects remain conflated. This work separates them. Webs were produced on a laboratory system at collector speeds of 200, 1100 and 2000 min⁻¹, corresponding to peripheral velocities of 1.05, 5.76 and 10.47 m s⁻¹ on a 100 mm drum, with all other parameters held constant. Fibre diameters were measured on 100 fibres per specimen and inclination angles on 250–494 fibres per specimen. The mean diameters are 180.11, 175.72 and 182.23 nm; their 95% confidence intervals overlap completely and all pairwise comparisons are non-significant ($p = 0.33–0.78$), establishing that collector speed does not affect fibre diameter. Over the same range the mean inclination angle falls from 46.3° to 23.7° with high significance, the angular distribution changes from uniform to approximately exponential, and the fraction of fibres deviating by less than 20° from the machine direction rises from 22% to 65%. The breaking load increases from 51.6 to 138.4 cN, a factor of 2.68. Krenchel's orientation efficiency factor, evaluated by integrating the fourth moment of the direction cosine over the measured angular distributions rather than assuming a limiting case, rises from 0.375 to 0.686 and predicts a strength ratio of only 1.83; the ratio of observed to predicted strengthening is 1.46 at 1100 min⁻¹ and 1.47 at 2000 min⁻¹. The constancy of this excess factor identifies a second, multiplicative strengthening mechanism, attributed to mechanical post-drawing of the fibre by the fast-moving collector, which accounts for the remaining third of the effect. The relative elongation at break falls from 27.00% to 16.31%. Collector speed is therefore established as an independent control variable that governs fibre placement without affecting fibre geometry, and the strength gain is attributed specifically to orientation efficiency rather than to any change in the fibres themselves.

Keywords: Electrospinning; collector rotation speed; peripheral velocity; fibre orientation; Krenchel factor; breaking load; statistical significance; nanofibrous web.



Introduction

An important feature of the structure of electrospun materials is the nature of the fibre arrangement, that is, their orientation along one or several directions. In a material intended for biomedical or cosmetic use, fibre orientation governs the rate and character of biodegradation, the nature of interaction with the wound surface or tissue, the rate at which drug substances penetrate the surface layers of the material, and the ease with which the material can be removed from the substrate without damage [1].

In tissue engineering a random fibre arrangement is often appropriate, since it closely imitates the structure of the extracellular matrix. At the same time the human body contains structures whose cells are arranged in an ordered manner: in natural blood vessels, for example, smooth muscle cells are arranged helically within the wall [2, 3]. To produce cell scaffolds of this type, fibres with controlled orientation are required, that is, fibres aligned in the desired direction.

Although the electrospinning method is simple in physical terms, many parameters influence the formation of the fibres and their deposition on the substrate. Phenomena such as the whipping instability and the bending instability of the jet lead to unpredictable deposition behaviour, which is a limiting factor in the production of straight and oriented fibres [4, 5]. Analysis of studies carried out worldwide shows that the simplest way of obtaining parallel nanofibres is to increase the rotational speed of the collecting electrode [6].

Two shortcomings are common in the existing treatments. First, the effect of collector speed on fibre diameter is usually assessed by comparing mean values, without testing whether the differences exceed the measurement scatter; as a result it is not established whether the collector affects diameter at all. Second, the increase in strength associated with alignment is reported as an empirical fact without comparison against the models of composite mechanics, so it is not known whether the gain is attributable to orientation alone or to accompanying changes in the fibres.

The novelty of the present work is that the influence of collector rotation speed on fibre diameter and on fibre orientation is separated by statistical testing, and that the increase in strength accompanying alignment is compared quantitatively with the prediction of Krenchel's orientation efficiency factor.

The aim of the work is to determine quantitatively the influence of collector rotation frequency on the nature of fibre arrangement in a nanofibrous material, and to establish theoretically the effect of fibre orientation on the deformation properties of the material.

2. Materials and methods

The experiments were carried out on a Fluidnatek LE-50 laboratory electrospinning system (Bionicia, Spain). Nanofibre specimens were obtained from aqueous solutions of poly(vinyl alcohol), Arkofil PPL grade, containing 14% of the fibre-forming polymer. Materials were produced on a cylindrical anodised aluminium drum collector of 100 mm diameter at three rotational speeds — 200, 1100 and 2000 min⁻¹ — with all other process parameters held constant for all specimens (Table 1).

Table 1. Process parameters held constant during specimen production.

Parameter	Value
Specimen production time	30 min
Emitter-to-collector distance	10 cm
Emitter voltage	29 kV
Collector voltage	9 kV
Mean field strength	3.8 kV cm ⁻¹
Spinning-solution feed rate	1.3 mL h ⁻¹
Collector drum diameter	100 mm
Substrate	paper coated with a layer of black printer ink

To reduce the collector rotation frequency to a kinematic indicator comparable between installations, the peripheral (linear) velocity of the drum surface was computed as

$$v = \pi \cdot D \cdot n / 60, \quad (1)$$

where D is the drum diameter (m) and n the rotational frequency (min⁻¹). For $D = 0.1$ m the speeds of 200, 1100 and 2000 min⁻¹ correspond to $v = 1.05$, 5.76 and 10.47 m s⁻¹ respectively. The structure of the fibrous material was visualised and the fibre diameters determined by scanning electron microscopy using a LEO 1420 microscope (Carl Zeiss, Germany). When the micrographs were taken, the specimens were oriented relative to the direction of the collector peripheral velocity. Fibre diameters were determined for each specimen from 100 measurements, while inclination angles were measured for all fibres contained in the micrographs. Because the number of fibres visible in a micrograph depends strongly on the collector rotation frequency, the number of micrographs used differed between specimens: four micrographs (494 fibres) at 200 min⁻¹, and five micrographs each at 1100 and 2000 min⁻¹ (250 and 268 fibres respectively).

The standard error and 95% confidence interval of the mean diameters were computed from the coefficient of variation:

$$S = \bar{d} \cdot V / 100; \quad SE = S / \sqrt{n}; \quad \Delta = 1.96 \cdot SE. \quad (2)$$

The significance of differences between specimens was assessed by the pairwise comparison statistic

$$t = (\bar{d}_2 - \bar{d}_1) / \sqrt{(SE_1^2 + SE_2^2)}. \quad (3)$$

Breaking load and elongation at break were determined on a Time WDW-20E tensile testing machine. Specimens were tested at a deformation rate of 50 mm min⁻¹ with a gauge length of 10 cm; the test specimens were 5 cm wide.

3. Results

3.1. Structure of the webs

Scanning electron micrographs of the specimens produced at the extremes of the speed range are shown in Figs. 1 and 2; the arrow indicates the direction of the peripheral velocity of the collecting drum.

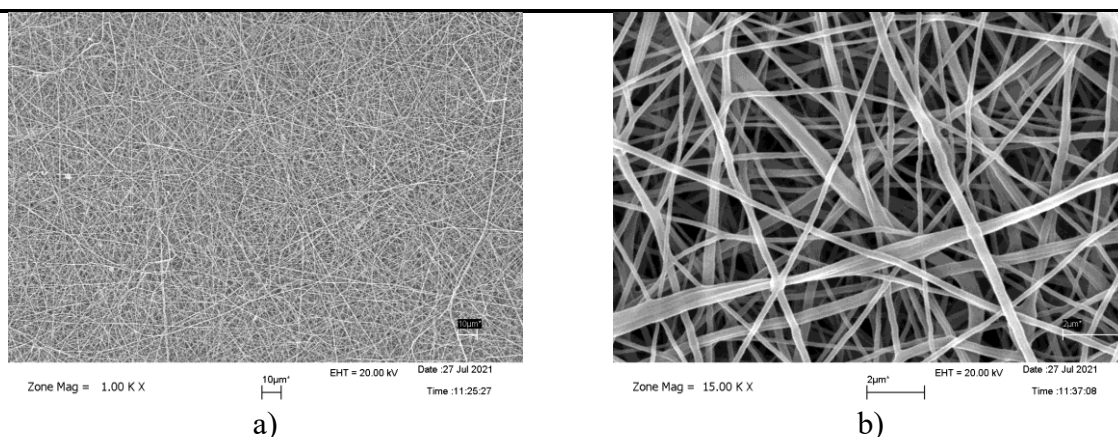


Fig. 1. Structure of the nanofibrous material produced at a collector speed of 200 min^{-1} ($v = 1.05 \text{ m s}^{-1}$): a) $1000\times$; b) $15000\times$.

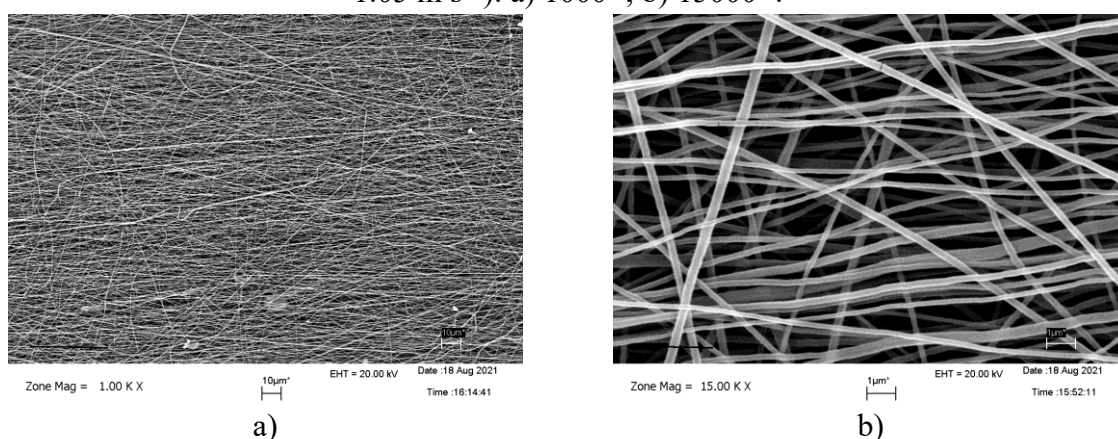


Fig. 2. Structure of the nanofibrous material produced at a collector speed of 2000 min^{-1} ($v = 10.47 \text{ m s}^{-1}$): a) $1000\times$; b) $15000\times$.

3.2. Fibre diameter

The measurement results, together with the statistical indicators computed from Eq. (2), are summarised in Table 2.

Table 2. Effect of collector rotation frequency on the structural parameters of the electrospun material.

n, min^{-1}	$v, \text{m s}^{-1}$	$\bar{d}, \text{nm}$	$V, \%$	S, nm	SE, nm	95% CI, nm	$\bar{\theta}, \text{degrees}$
200	1.05	180.11	30.6	55.11	5.51	169.3–190.9	46.3
1100	5.76	175.72	23.1	40.59	4.06	167.8–183.7	39.4
2000	10.47	182.23	28.7	52.30	5.23	172.0–192.5	23.7

The 95% confidence intervals of the three mean diameters overlap completely. The pairwise comparisons performed with Eq. (3) confirm that the differences are not statistically significant: between 200 and 2000 min^{-1} the difference is 2.12 nm ($t = 0.28$; $p = 0.78$); between 200 and 1100 min^{-1} it is -4.39 nm ($t = -0.64$; $p = 0.52$); and between 1100 and 2000 min^{-1} it is 6.51 nm ($t = 0.98$; $p = 0.33$). The collector rotation speed therefore affects neither the fibre diameter nor

its uniformity: the observed differences do not exceed 7 nm and lie entirely within the limits of statistical error.

3.3. Fibre orientation

The picture for fibre orientation is entirely different. As the rotation speed was increased from 200 to 2000 min⁻¹ the mean fibre inclination angle decreased from 46.3° to 23.7°, that is, by almost a factor of two. Using standard deviations estimated from the shapes of the distributions (uniform distribution at 200 min⁻¹, $S = 90/\sqrt{12} = 25.98^\circ$; approximately exponential distribution at 2000 min⁻¹, $S \approx \bar{\theta} = 23.7^\circ$), the significance of this difference is $t \approx 12.1$, that is $p < 0.001$.

The velocity regime of the process also affects the distribution law of the fibre inclination angles (Fig. 3). At a rotation speed of 200 min⁻¹ the distribution of inclination angles corresponds to a uniform law; as the speed increases the distribution approaches an exponential law.

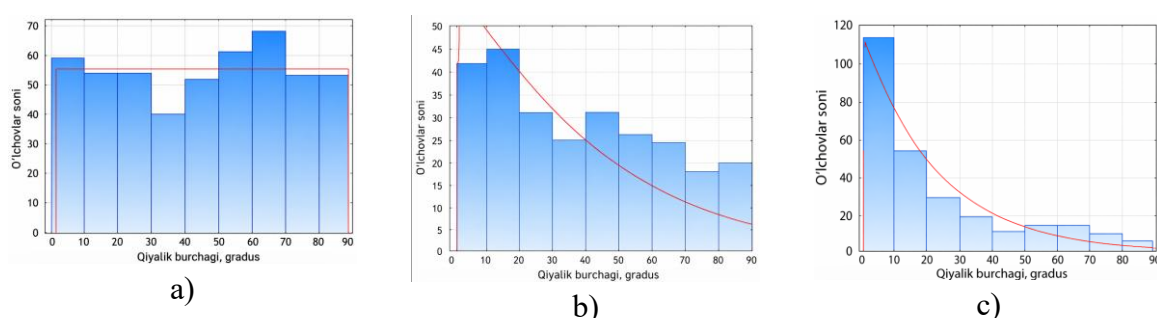


Fig. 3. Distribution of nanofibre inclination angles in specimens produced at different collector speeds: a) 200 min⁻¹; b) 1100 min⁻¹; c) 2000 min⁻¹.

At a collector rotation frequency of 1100 min⁻¹ only 33% of the fibres deviate from the direction of the peripheral velocity by not more than 20°; at 2000 min⁻¹ the proportion of such fibres reaches 65%. With random arrangement (200 min⁻¹) the corresponding fraction is approximately 22%. The proportion of fibres lying in the transverse direction (70°–90°) also decreases appreciably: 15% at 1100 min⁻¹ and 6% at 2000 min⁻¹. The specimens also differed visually: those obtained at the lowest collector speed had a matt surface, whereas as the speed increased the specimens acquired a gloss, which is likewise explained by the increasing degree of fibre orientation in their structure.

3.4. Tensile behaviour

The stress–strain curves obtained during the tensile tests are shown in Fig. 4, and the resulting physical and mechanical properties are given in Table 3.

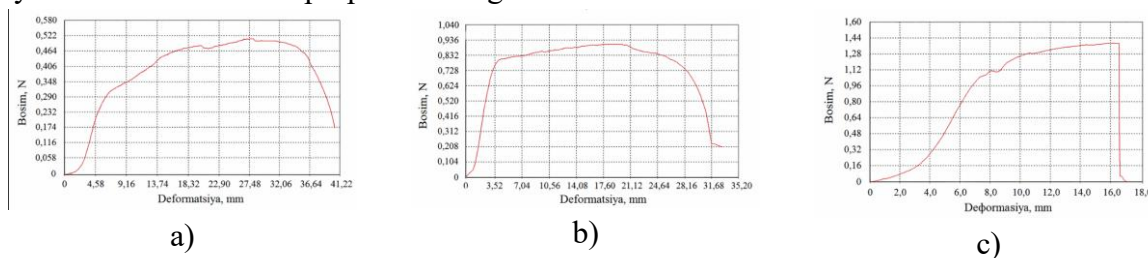


Fig. 4. Stress–strain curves of the nanofibrous materials produced at different collector speeds: a) 200 min⁻¹; b) 1100 min⁻¹; c) 2000 min⁻¹.

Table 3. Effect of fibre orientation on the properties of the electrospun material.

n, min^{-1}	$\bar{\theta}, \text{degrees}$	Fraction within $20^\circ, \%$	Breaking load P, cN	Elongation at break $\epsilon, \%$
200	46.3	22	51.6	27.00
1100	39.4	33	91.2	18.40
2000	23.7	65	138.4	16.31

The relationship between breaking load and mean inclination angle is, in a linear approximation,

$$P = 228.6 - 3.70 \cdot \bar{\theta}, \quad r = -0.986. \quad (4)$$

Although the absolute value of the correlation coefficient is high, with only three experimental points it does not reach formal significance at the 0.05 level ($p = 0.11$); Eq. (4) should therefore be regarded as a quantitative estimate of the trend rather than as an established relationship.

4. Discussion

4.1. Why diameter is unaffected

That the collector rotation speed does not affect the fibre diameter is the expected result from the physics of the process. The fibre diameter is established in the Taylor cone and in the jet-splitting and stretching zone, that is, before the fibre reaches the collector; the drum merely draws in and orients a fibre that has already solidified. The statistical analysis confirms this qualitative argument quantitatively: across the three regimes the differences are non-significant at $p = 0.33$ – 0.78 .

The practical value of this finding is that collector speed is an independent control variable. Diameter is set by the formulation, orientation by the collector kinematics, and the two can be specified separately — which is not generally true of the other process variables, since voltage, distance and feed rate all influence diameter and morphology simultaneously.

4.2. Transition of the angular distribution

The transition of the angular distribution from a uniform to an exponential law reflects the ordering mechanism. At low speed the peripheral velocity of the collector (1.05 m s^{-1}) is small compared with the deposition velocity of the fibre, so no direction is preferred and angles are distributed with equal probability between 0 and 90° . As the velocity increases to 10.47 m s^{-1} a drawing force appears and the probability of small angles rises sharply, which produces the exponential distribution.

Reporting the peripheral velocity rather than the rotational speed is important here. The same rotational speed on drums of different diameter produces different degrees of alignment, so a study that reports only the speed cannot be reproduced on other equipment; Eq. (1) should be applied before the value is published.



4.3. Quantitative test against Krenchel's orientation model

The increase in breaking load can be tested against composite mechanics rather than merely compared with it. The efficiency of a fibrous system along the loading direction is characterised by Krenchel's orientation efficiency factor, which for a planar distribution of fibre orientations is the fourth moment of the direction cosine [7, 8]:

$$\eta_0 = \langle \cos^4\theta \rangle = \int p(\theta) \cdot \cos^4\theta \, d\theta / \int p(\theta) \, d\theta, \quad (5)$$

where $p(\theta)$ is the probability density of the inclination angle. For a uniform distribution on $[0^\circ, 90^\circ]$, Eq. (5) gives $\eta_0 = 3/8 = 0.375$, the classical two-dimensional random value; for perfect alignment $\eta_0 = 1$.

Rather than assuming either limiting case, η_0 was evaluated from the measured distributions. At 200 min^{-1} the angular distribution is uniform (Section 3.3), so $\eta_0 = 0.375$ exactly. At 1100 and 2000 min^{-1} the distribution approaches an exponential law truncated to $[0^\circ, 90^\circ]$; the decay constant of each was fixed by requiring the distribution mean to reproduce the measured mean angle (39.4° and 23.7° respectively), and Eq. (5) was then integrated numerically. The results are given in Table 4.

Table 4. Orientation efficiency computed from the measured angular distributions, and the resulting predicted and observed strength ratios.

n, min ⁻¹	$\bar{\theta}$, degrees	Distribution	η_0 from Eq. (5)	Predicted P/P ₂₀₀	Observed P/P ₂₀₀	Excess factor
200	46.3	uniform	0.375	1.00	1.00	—
1100	39.4	exponential	0.452	1.21	1.77	1.46
2000	23.7	exponential	0.686	1.83	2.68	1.47

Two results follow, and the second is the more informative. First, orientation efficiency does account for a substantial part of the strengthening: raising the collector speed from 200 to 2000 min^{-1} increases η_0 from 0.375 to 0.686 , which alone predicts a strength ratio of 1.83 , that is 68% of the observed 2.68 . Second, and unexpectedly, the ratio between the observed and predicted strengthening — the excess factor — is 1.46 at 1100 min^{-1} and 1.47 at 2000 min^{-1} . Its constancy to within 1% at two independent speeds indicates that the residual strengthening enters multiplicatively alongside the geometrical term rather than as an additive offset or as random scatter.

It is worth stating explicitly why the geometrical term cannot account for the whole effect. Inverting the argument, the orientation efficiency that would be required to explain the observed strength at 2000 min^{-1} purely geometrically is $\eta_0 = 0.375 \times 2.68 = 1.006$, that is the perfectly aligned limit. This is incompatible with the measured distribution, in which only 65% of fibres lie within 20° of the machine direction and 6% remain in the transverse 70° – 90° band. The same inversion at 1100 min^{-1} requires $\eta_0 = 0.663$, which would correspond to a mean angle near 25° against the 39.4° actually measured. A purely geometrical explanation is therefore excluded by the data at both speeds.

The most plausible origin of the multiplicative excess is mechanical drawing of the fibre by the collector. At 2000 min^{-1} the peripheral velocity is 10.47 m s^{-1} , which is of the same order as the terminal velocity of the electrospun jet; a fibre arriving at a surface moving at that speed is subjected to additional axial tension while its polymer chains still retain mobility. Such post-drawing increases molecular orientation and crystallinity within the fibre and therefore raises its intrinsic tensile strength, an effect well established for conventionally drawn fibres and reported for electrospun fibres collected at high take-up velocities [9, 12]. Because this mechanism scales the strength of every fibre irrespective of its inclination, it multiplies the geometrical factor instead of adding to it — which is precisely the behaviour observed.

Two alternative explanations are less consistent with the data. An increase in the packing density of the web would raise the load-bearing cross-section, but it would also be expected to vary between the two speeds rather than to produce an identical factor. Increased inter-fibre bonding would raise strength but would be expected to reduce elongation disproportionately, whereas the measured elongation falls by a factor of only 1.66 while strength rises by 2.68.

This analysis also revises a simpler comparison that the data invite. The observed ratio 2.68 is numerically close to $8/3 = 2.67$, the ratio of the perfectly aligned to the two-dimensional random limit, and it is tempting to read this as exact agreement with Krenchel's model. The agreement is fortuitous: it requires the 2000 min^{-1} material to be fully aligned, which it is not. When the model is applied to the distribution actually measured, it accounts for two-thirds of the effect and identifies the remaining third as a distinct physical mechanism — a more informative result than apparent perfect agreement.

4.4. Elongation at break

The decrease in relative elongation from 27.00% to 16.31% fits the same picture. In a random network, deformation occurs first through reorientation of the fibres and only then through their own extension; in an ordered structure the first mechanism is largely absent, so the material extends less and fails sooner. The trade-off between strength and extensibility is therefore intrinsic to orientation control and cannot be avoided by adjusting the collector alone.

4.5. Selection of the velocity regime and limitations

Recommendations on the choice of the velocity regime should follow from the requirements placed on the material. If the objective is a more compliant and extensible material with a disordered network structure — a wound dressing or a cosmetic mask — a low collector rotation frequency should be set. Conversely, where high strength is required, as in vascular prostheses and tendon or ligament scaffolds, a high rotation frequency should be used.

The study has three limitations. First, the mechanical tests were performed on a single specimen for each regime, so the relationship of Eq. (4) did not attain statistical significance; increasing the number of replicates to at least five is required. Second, the standard deviations of the angles were not measured directly but estimated from the distribution laws; these estimates suffice to establish the order of the t statistic but not to construct exact confidence intervals. Third, because the areal density and thickness of the specimens were not measured, the breaking load



could not be converted to a specific strength in MPa; this quantity is necessary for comparison with literature data and is a direction for further work.

5. Conclusions

It was established statistically that the collector rotation frequency does not affect the fibre diameter: the 95% confidence intervals of the mean diameters for the three regimes overlap completely and the pairwise differences are non-significant ($p = 0.33\text{--}0.78$).

As the collector rotation frequency is increased from 200 to 2000 min^{-1} (peripheral velocity from 1.05 to 10.47 m s^{-1}), the mean fibre inclination angle decreases from 46.3° to 23.7° with high statistical significance ($t \approx 12.1$; $p < 0.001$).

The distribution of fibre inclination angles corresponds to a uniform law at 200 min^{-1} and approaches an exponential law as the speed increases; the fraction of fibres deviating by less than 20° from the machine direction rises from 22% to 65%.

The breaking load increases from 51.6 cN to 138.4 cN, a factor of 2.68. Krenchel's orientation efficiency factor, evaluated from the measured angular distributions, rises from 0.375 to 0.686 and accounts for a factor of 1.83, that is two-thirds of the observed strengthening; the residual excess factor is 1.46–1.47 at both elevated speeds and is attributed to mechanical post-drawing of the fibres by the fast-moving collector, which raises their intrinsic strength independently of their orientation.

The relative elongation at break decreases from 27.00% to 16.31%, so that strength and extensibility trade off against one another; the velocity regime of electrospinning should therefore be selected according to the intended application of the material. Collector rotation speed is established as an independent control variable that governs fibre placement without affecting fibre geometry.

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Data availability. The data supporting the findings of this study are available from the corresponding author upon reasonable request.

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