

Article

Optimization of Electrospinning Parameters for Bead-Free Morphology of Poly(vinyl alcohol) Nanofibers

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Abstract

Electrospinning is a relatively simple and promising method for producing polymeric, ceramic, and composite fibers, with diameters ranging from several nanometers to several micrometers. In this study, the influence of PVA solution concentration, tip-to-collector distance, and needle gauge at a constant applied voltage on the morphology and diameter of the electrospun fibers was investigated to optimize conditions for the production of bead-free nanofibers. PVA solutions with concentrations of 5, 7.5, and 10 wt.% were electrospun under varying processing conditions, and the resulting fibers were characterized by SEM. The results demonstrated a strong dependence of fiber morphology on polymer concentration. Low-concentration solutions (5 wt.%) produced fibers with significant bead formation, while increasing the concentration to 10 wt.% resulted in smooth and homogeneous fibers. A non-linear dependence of fiber diameter on tip-to-collector distance was observed, with 140 mm identified as the optimal distance for producing the thinnest fibers. Needle gauge also affected fiber morphology and process stability. The G22 needle produced finer fibers at the optimal collector distance of 140 mm, with the narrowest fiber diameter distribution, indicating the highest uniformity, whereas the G20 and G21 needles exhibited the broader distribution. Statistical analysis (ANOVA) confirmed the significance of the investigated parameters. These findings demonstrate that collector distance and needle gauge should not be evaluated independently and provide practical guidance for optimizing electrospinning conditions for the fabrication of uniform, bead-free PVA nanofibers.



Keywords: electrospinning; Poly(vinyl alcohol); nanofibers; ANOVA

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1. Introduction

In recent years, nanofibrous materials have gained considerable interest due to their unique physicochemical properties and versatile application potential. There are various methods for producing nanofibers, including drawing, phase separation, template synthesis, interfacial polymerization, template melt extrusion, melt blowing, self-assembly, force spinning, and electrospinning [1]. However, many of these methods are complex, not scalable, time-consuming, or only applicable to a limited range of polymers [2,3]. Among these techniques, electrospinning has attracted significant attention due to its simplicity

and versatility. The process involves the application of a high-voltage electric field, which drives a charged jet of polymer solution from a syringe toward a collector with opposite charge [4]. When the applied voltage exceeds the surface tension, a charged polymer jet is ejected and stretches as it moves toward the collector. Recent electrohydrodynamic models describe electrospinning as a multi-stage process involving Taylor cone formation, jet acceleration, bending instabilities, and solvent evaporation before fiber deposition on the collector [5]. During flight, the charged jet undergoes bending and whipping instabilities, which contribute to additional jet stretching and fiber thinning [6]. Simultaneously, solvent evaporation leads to polymer solidification and the formation of a network of fine fibers [7]. The reduction in polymer fiber diameter from microscale to nanoscale results in numerous advantageous properties. These include a high surface area-to-volume ratio, enhanced surface functionalization, and improved mechanical performance. As a result, nanoscale polymer fibers appear as promising materials for a variety of applications, including filtration, reinforcement materials, wound dressings, tissue engineering, and drug delivery [8]. The parameters affecting the electrospinning process can be divided into three main categories: solution parameters (concentration, molecular weight, viscosity, surface tension, and conductivity), process parameters (voltage, flow rate, the distance between tip and collector, and the size of the spinneret), and ambient parameters (humidity, temperature, pressure, and type of atmosphere). By optimizing these parameters, it is possible to produce electrospun fibers with the desired morphologies and diameters [9]. The relationship between polymer concentration and fiber morphology is important, as increased chain entanglement is known to promote the transition from beaded structures to smooth, continuous fibers [10,11]. Among the various polymers used for electrospinning, aqueous solutions of Poly(vinyl alcohol) (PVA) are particularly attractive due to their water solubility, biocompatibility, and ease of processing [8,11]. PVA is a non-toxic, odourless polymer that possesses effective oxygen and aroma barrier properties. Moreover, it exhibits good mechanical properties, including high tensile strength and flexibility, as well as excellent film-forming, emulsifying, and adhesive characteristics [12]. These characteristics make PVA a suitable material for various industrial applications, including biodegradable packaging, adhesives, textiles, paper, and coatings, as well as advanced medical uses such as drug delivery systems, contact lenses, and wound dressings [13]. Numerous studies have examined the impact of various parameters on the electrospinning of Poly(vinyl alcohol) (PVA) fibers, including the inherent characteristics of the polymer and the processing conditions [14,15]. For example, Koski et al. [10] demonstrated that the molecular weight of polyvinyl alcohol (PVA) significantly affects the structure of electrospun polymers. Lower-molecular-weight PVA tends to produce beaded fibers due to jet instability. In contrast, increasing the molecular weight results in the formation of uniform, defect-free nanofibers with larger diameters. Similarly, increasing solution concentration results in a transition from beaded to uniform fiber structures [10]. The degree of hydrolysis also influences fiber morphology and diameter. It was observed that, at a constant concentration of PVA solution, the average diameter of the electrospun PVA fibers increased as the degree of hydrolysis increased. Furthermore, the morphology of the electrospun PVA fibers changed from ribbon-like structures to uniform fibers, and eventually to beaded fibers [10,14,15]. Processing parameters such as applied voltage, tip-to-collector distance, and flow rate are also known to significantly affect jet stability and PVA fiber formation during electrospinning [14]. Recent multi-variable characterization studies continue to focus on optimizing these operational boundaries (such as flow rate and distance) specifically to achieve absolute uniformity in PVA-based matrices [16]. Despite numerous studies, there is still limited research on the combined effects of multiple processing parameters. While the individual effects of electrospinning parameters have been widely investigated [14], the interaction

between needle diameter and collector distance under fixed-voltage conditions remains insufficiently characterized for aqueous PVA systems. Therefore, this study systematically investigates the influence of solution concentration, tip-to-collector distance, and needle diameter (G20, G21, and G22) on the morphology and diameter of electrospun PVA fibers at a constant applied voltage. In addition, a two-way ANOVA was performed to quantitatively evaluate the significance and individual contributions of collector distance, needle diameter, and their interaction to fiber diameter variation.

2. Materials and Methods

Poly(vinyl alcohol) (PVA) with molecular weight 72,000 Da and 98% degree of hydrolysis was obtained by Valerus Ltd. Sofia, Bulgaria and used without further purification. Distilled water was used as the solvent.

PVA solutions with concentrations 5 wt.%, 7.5 wt.% and 10 wt.% were prepared by dissolving an appropriate amount of PVA in distilled water. The solutions were stirred for 4 h at 85 °C. The obtained polymer solutions were transferred into a 50 mL syringe prior to electrospinning.

The experimental setup consisted of a 50 mL syringe fitted with a stainless-steel needle of varying diameters (G20 (OD-0.91 and ID-0.6), G21 (OD-0.82 and ID-0.51), and G22 (OD-0.72 and ID-0.41), which were positioned vertically on a clamp, as illustrated in Figure 1. The syringe, filled with PVA solution at different concentrations (5, 7.5, and 10 wt.%), was placed on a syringe pump operating at a constant flow rate of 1.0 mL/h. A high-voltage DC power supply was used to generate an electric field of approximately 16 kV necessary for initiating fiber formation. The distance between the collector plate and the needle was adjusted from 110 to 160 mm. All experiments were conducted under controlled ambient conditions, maintained at 22 °C and 52% relative humidity.

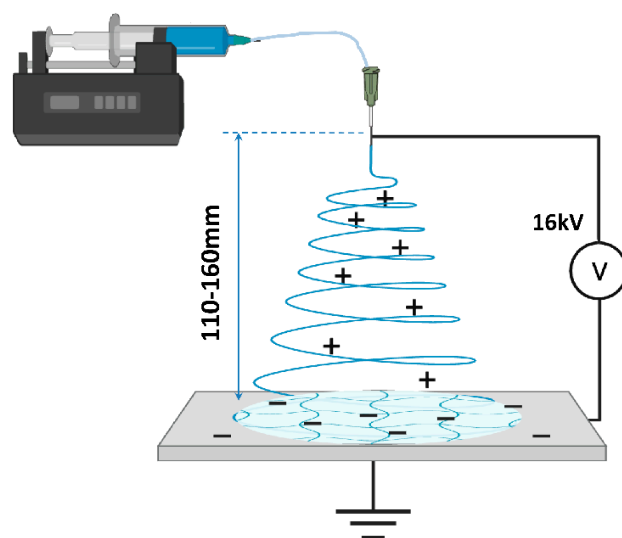


Figure 1. Schematic Diagram of the electrospinning process.

The fiber morphology and diameter of the electrospun PVA fibers were determined using scanning electron microscopy (Model Zeiss, EVO 10, software SmartSem Version 7.05 with Service pack 3, Carl Zeiss Microscopy GmbH, Carl-Zeiss-Promenade 10, 07745 Jena, Germany). Fiber diameters were quantified from high-resolution SEM images using KERN VIS Lite microscope camera software (KERN & Sohn GmbH, Balingen-Frommern, Germany) after calibration against a certified stage micrometer. For each experimental condition, images were acquired from at least three non-overlapping regions of the fiber mat. Only fibers that appeared straight over a minimum length of 5 μm and remained fully in

focus were selected; fibers exhibiting beads, junctions, splits, or excessive curvature were excluded. For each selected fiber, three to five perpendicular diameter measurements were taken at random locations along its axis, and the mean value per fiber was recorded. All measurements were performed by a single trained operator following a written standard operating procedure. A total of 556 individual fibers were analyzed across all conditions. At the imaging magnification used ($\times 3000$), fiber diameters corresponded to approximately 8–9 pixels; in this size range, automated skeletonization-based tools are known to incur $>10\%$ error due to pixel discretization. Manual measurement with multiple sampling points per fiber was therefore adopted as the primary quantification method.

3. Results

3.1. Influence of PVA Concentration on the Nanofiber Formation

Among all the operational parameters, polymer concentration is one of the most important in the electrospinning process, as it significantly affects fiber production, as well as the morphology and diameter of the obtained fibers [17]. The effect of concentration is closely related to polymer chain entanglement, which is commonly described by the dimensionless Berry number ($B = [\eta]C$). For PVA with a molecular weight of $72,000 \text{ g mol}^{-1}$, the literature reports intrinsic viscosity values of 0.73 dL g^{-1} for similar aqueous PVA systems [18], allowing an estimation of the entanglement regime in the investigated concentration range. Based on literature estimations, the studied concentrations (5–10 wt.%) correspond to a transition from weakly entangled solutions, where bead formation is dominant, to sufficiently entangled regimes that support stable fiber formation. This transition is known to improve jet stability and suppress bead formation during electrospinning [10,18]. To evaluate this effect, electrospinning was carried out under fixed conditions, including a tip-to-collector distance of 140 mm, an applied voltage of 16 kV, and a G20 needle. At a concentration of 5 wt.%, SEM analysis revealed fibers with diameters below 200 nm together with numerous bead defects (Figure 2a). These observations are consistent with previous reports showing that insufficient polymer chain entanglement below a critical threshold leads to jet instability and capillary-driven breakup [6,10]. Increasing the PVA concentration to 7.5 wt.% improved fiber morphology and reduced the number of bead defects. SEM analysis revealed fibers with an average diameter of approximately 170 nm together with spindle-like beads measuring approximately 343 nm in width and 1800 nm in length (Figure 2b). Further increasing the concentration to 10 wt.% resulted in the formation of smooth, homogeneous, and bead-free nanofibers (Figure 2c). The average fiber diameter increased to approximately 200 nm. This increase is attributed to the higher solution concentration, which enhances the viscoelastic resistance of the jet and promotes more stable fiber formation while suppressing capillary-driven breakup [6,10].

3.2. Influence of Tip-to-Collector Distance and Needle Diameter on the Morphology of PVA Nanofiber

The influence of the tip-to-collector distance and needle diameter on the morphology and diameter of PVA nanofibers was further evaluated. The experiment was conducted using a 10 wt.% PVA solution, keeping all other parameters constant while varying the distance from 110 to 160 mm and the needle diameter from G20 (OD 0.9 mm), G21 (0.82 mm) to G22 (OD 0.72 mm). The results demonstrated a non-linear dependence of fiber diameter on the tip-to-collector distance at a constant voltage of 16 kV, with a characteristic minimum observed around 140 mm. As the distance increased from 110 to 120 mm, the fiber diameter generally increased, followed by a decrease at 140 mm, and a subsequent increase at larger distances (150–160 mm) (Figure 3).

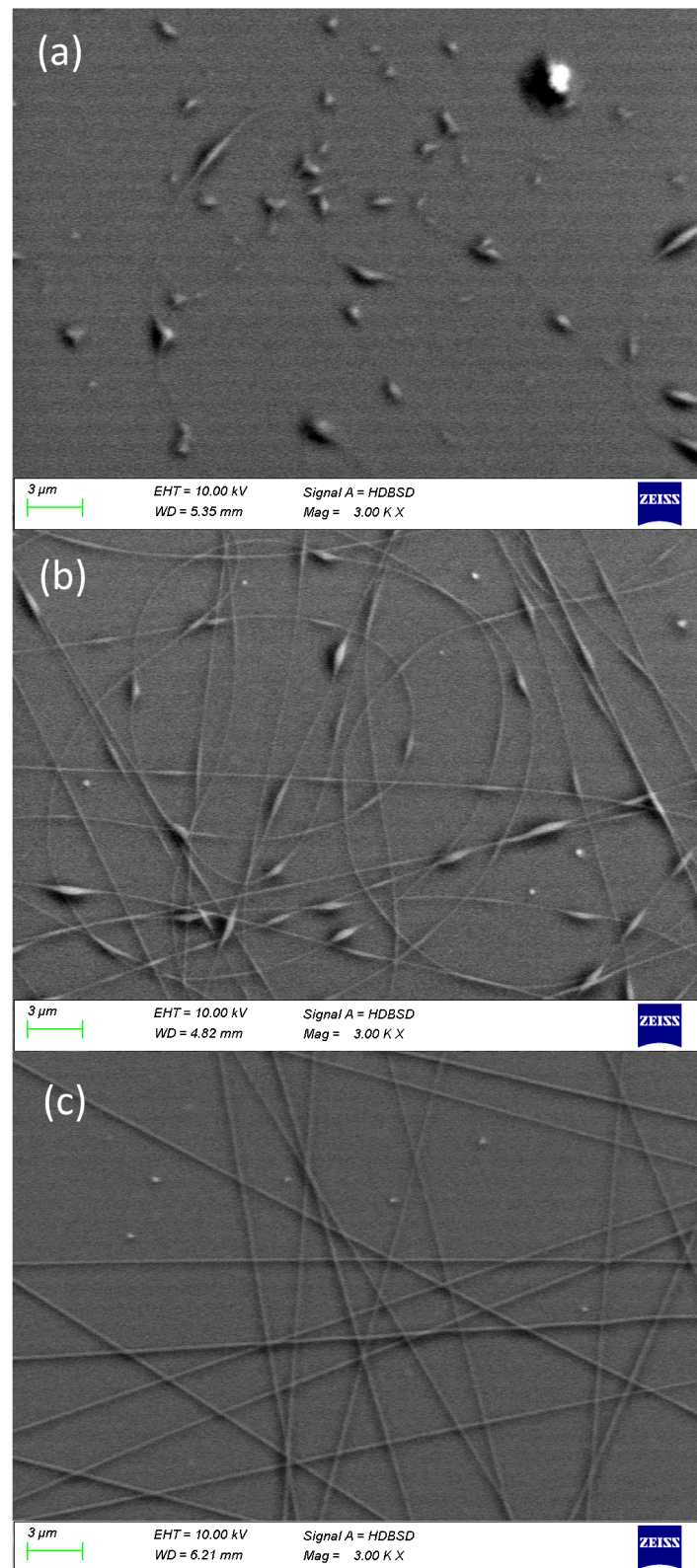


Figure 2. SEM images of PVA nanofibers at (a) 5 wt.%, (b) 7.5 wt.% and (c) 10 wt.% (needle G20, flow rate 1 mL/h and applied voltage 16 kV).

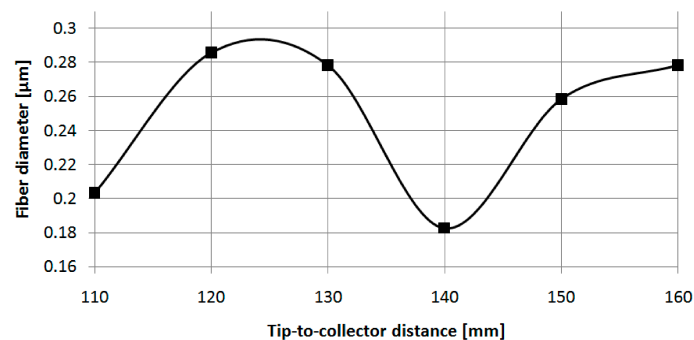


Figure 3. Effect of applied tip-to-collector distance on fiber diameter (10 wt.% PVA solution, needle G22, flow rate 1 mL/h and applied voltage 16 kV).

At shorter distances (110–120 mm), the relatively high electric field strength promotes strong initial jet stretching; however, at 110 mm this effect dominates, resulting in thinner fibers compared to 120 mm. At 120 mm, the reduced field strength combined with insufficient development of bending and whipping instabilities leads to comparatively thicker fibers. As the distance increases toward 130–140 mm, a balance between electric field strength, jet stretching, and solvent evaporation is achieved, resulting in enhanced elongation and a minimum fiber diameter at approximately 140 mm. Beyond this optimal distance (150–160 mm), the continued reduction in electric field strength weakens electrostatic stretching forces and reduces jet stability, while capillary-driven instabilities become more pronounced, leading to an increase in fiber diameter. This non-linear behavior is consistent with electrohydrodynamic models of polymer jets, which suggest the existence of an optimal flight distance where electric-field-driven elongation, solvent evaporation, and bending instabilities act synergistically to minimize fiber diameter. Similar behavior has been reported in previous electrospinning studies and theoretical analyses of jet thinning and whipping instabilities [5,6,19]. Further, the influence of needle diameter on the morphology and diameter of electrospun PVA fibers was investigated. In all cases, a clear dependence on the average fiber diameter, tip-to-collector distance, and needle diameter was observed. For the G20 needle, the average fiber diameter initially increased from 0.268 μm at a distance of 110 mm to 0.292 μm at 120 mm. This was followed by a decrease to 0.211 μm at 130 mm, reaching a minimum of 0.203 μm at 140 mm. After 140 mm, a slight increase was observed at 160 mm, with a diameter of 0.227 μm (Figure 4).

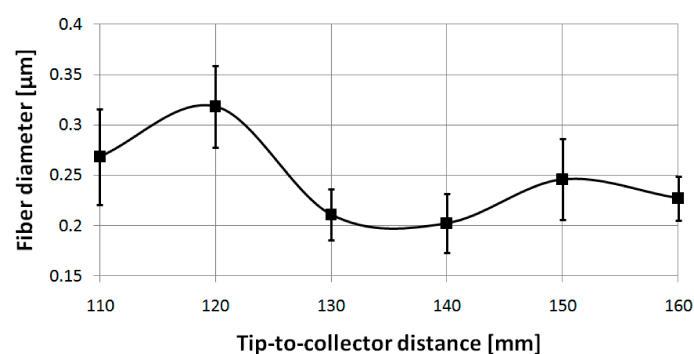


Figure 4. Effect of needle G20 and tip-to-collector distance on fiber diameter of 10 wt.% PVA solution with standard deviation error bars.

A similar but more pronounced trend was observed for the G21 needle. The fiber diameter initially increased from 0.194 μm at 110 mm to 0.249 μm at 130 mm, followed by a gradual decrease with increasing distance, reaching a minimum of 0.197 μm at 140 mm. After this, an increase to 0.250 μm at 160 mm was observed (Figure 5).

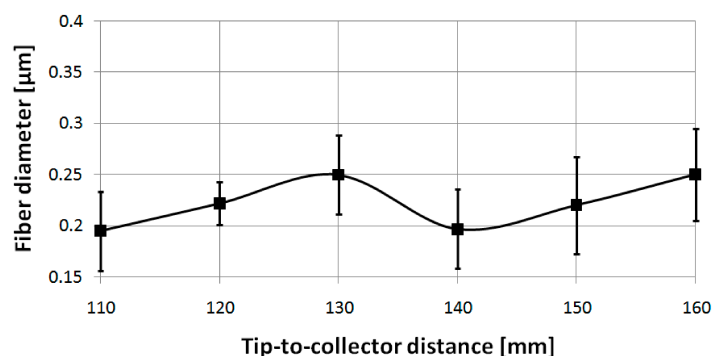


Figure 5. Effect of needle G21 and tip-to-collector distance on fiber diameter of 10 wt.% PVA solution with standard deviation error bars.

For the G22 needle, the average fiber diameter increased as the collector distance was increased from 110 mm to 120–130 mm, followed by a sharp decrease to a minimum value of approximately 0.180 μm at 140 mm. Further increasing the distance resulted in a gradual increase in fiber diameter, reaching about 0.280 μm at 160 mm (Figure 6).

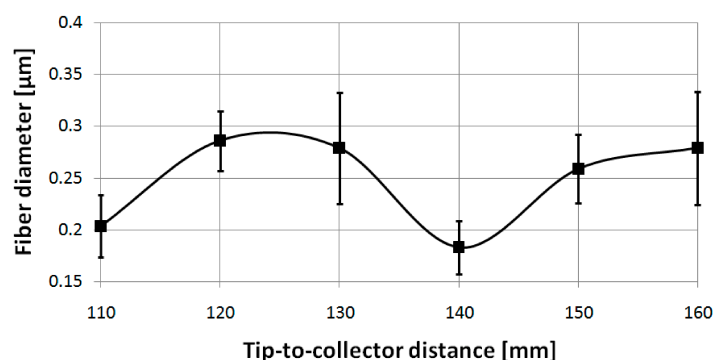


Figure 6. Effect of needle G22 and tip-to-collector distance on fiber diameter of 10 wt.% PVA solution with standard deviation error bars.

From a mechanistic perspective, needle diameter determines the shear conditions acting on the polymer solution as it flows through the spinneret. A reduction in needle diameter increases shear rate, which in shear-thinning PVA systems leads to more efficient jet formation and enhanced stretching under the electric field [20]. Accordingly, smaller needles (G22) enhance jet stretching and lead to finer fibers. At the optimal collector distance of 140 mm, the G22 needle also produced the most uniform fiber diameter distribution (Figure 7c). However, considering the full experimental range, the fiber diameter obtained with G22 showed a stronger dependence on collector distance, suggesting greater sensitivity to processing conditions. In contrast, larger needles (G20–G21) produced slightly thicker fibers at the optimal collector distance of 140 mm (Figure 7a,b) but exhibited less pronounced diameter changes across the investigated collector distances.

Table 1 summarizes the fiber diameter statistics obtained at the optimal collector distance of 140 mm using 10 wt.% PVA solution. The mean fiber diameters were comparable among the three needle gauges, ranging from $0.196 \pm 0.020 \mu\text{m}$ for the G22 needle to $0.203 \pm 0.021 \mu\text{m}$ for the G20 needle. The G22 needle produced fibers with the narrowest diameter distribution (standard deviation = 0.020 μm ; interquartile range = 0.0240 μm), indicating higher uniformity at this distance, whereas the G21 needle showed the broadest distribution (standard deviation = 0.039 μm ; interquartile range = 0.059 μm).

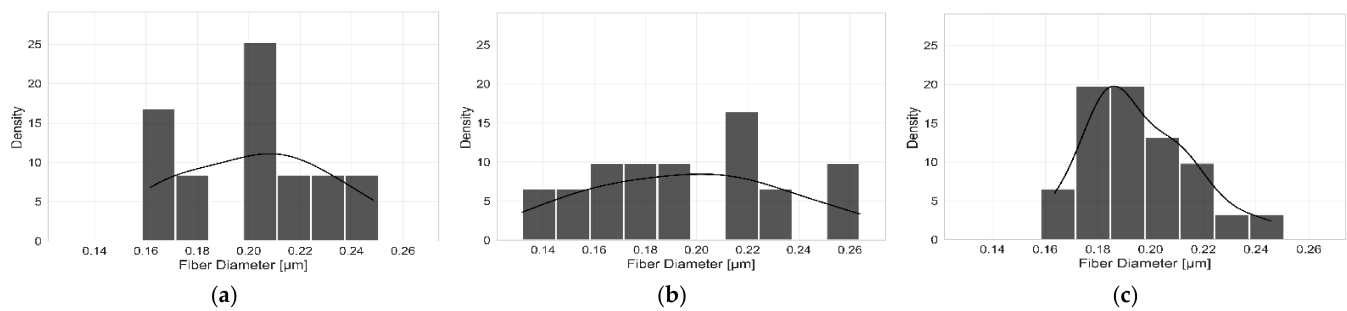


Figure 7. Fiber diameter distribution histograms for different needle diameters at a tip-to-collector distance of 140 mm: (a) G20, (b) G21, (c) G22.

Table 1. Fiber diameter statistics at collector distance of 140 mm using 10 wt.% PVA solution.

Needle Gauge	n	Mean $\pm$ SD [μm]	Median [μm]	Interquartile Range
G20	49	0.203 $\pm$ 0.021	0.205	0.0411
G21	53	0.197 $\pm$ 0.039	0.197	0.0589
G22	51	0.196 $\pm$ 0.020	0.187	0.0240

These findings indicate that, at a collector distance of 140 mm, the G22 needle produced not only the smallest mean fiber diameter but also the most uniform fiber distribution. Therefore, the combination of the G22 needle and a collector distance of 140 mm represents the optimal processing condition for achieving both reduced fiber diameter and high diameter uniformity.

3.3. Statistical Analysis

To statistically evaluate the significance of these observations, a two-way analysis of variance (ANOVA) was performed on log-transformed fiber diameters to assess the effects of needle diameter (G20, G21, and G22) and collector distance (110–160 mm) and their interaction.

The fiber diameter data were log-transformed prior to statistical analysis because the raw measurements exhibited significant deviations from normality (Shapiro–Wilk test on residuals, $p < 0.0001$) and homogeneity of variances (Levene’s test, $p < 0.0001$). After log transformation, the assumptions of the two-way ANOVA were approximately satisfied, although mild deviations remained due to the large number of observations ($n = 556$). The normality of the residuals from the two-way ANOVA performed on log-transformed fiber diameters was visually examined using a normal Q-Q plot (Figure 8). The majority of the data points closely followed the theoretical reference line, indicating that the residuals were approximately normally distributed. Only mild deviations were observed in the lower and upper tails, which is typical for fiber-diameter measurements even after logarithmic transformation. Although the Shapiro–Wilk test on the residuals yielded a statistically significant result, this is expected with a large sample size and does not compromise the validity of the ANOVA results, which are known to be robust to such minor departures from normality in electrospinning studies.

The analysis revealed statistically significant main effects of needle diameter ($F_{2,538} = 8.70$, $p = 0.0002$) and collector distance ($F_{5,538} = 6.62$, $p < 0.0001$), as well as a highly significant needle diameter $\times$ collector distance interaction ($F_{10,538} = 5.21$, $p < 0.0001$) (Table 2). These factors together accounted for 16.02% of the total variation in fiber diameter, while the residual (unexplained) variation was 83.98% of the total variance, reflecting the complex and stochastic

nature of electrospinning as well as additional sources of variability like fiber diameter and measurement uncertainty.

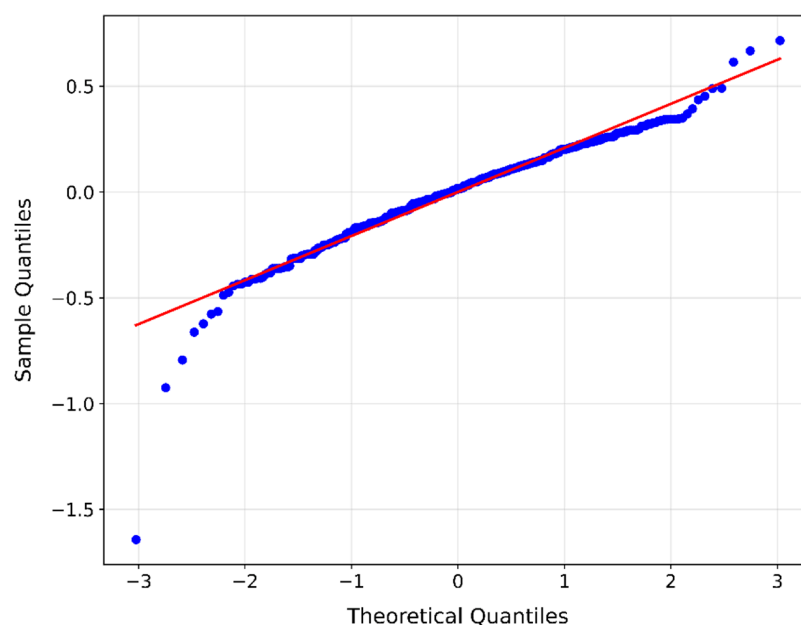


Figure 8. Q-Q Plot of residuals from two-way ANOVA on log(diameter) scale.

Table 2. Two-way ANOVA on log-transformed fiber diameters.

Factor	Sum of Squares	df	F	<i>p</i> -Value	% of Total Variation
Needle diameter	0.8223	2	8.70	0.0002	2.71%
Collector distance	1.5642	5	6.62	<0.0001	5.16%
Needle $\times$ Distance (interaction)	2.4642	10	5.21	<0.0001	8.14%
Residual	25.4376	538	-	-	83.98%

Main effects plots (Figure 9a,b) showed a mild decrease in mean log fiber diameter as needle gauge increased from G20 to G21, followed by a slight increase at G22 (Figure 9a), and generally smaller diameters at intermediate collector distances (140 mm) compared with both shorter (110 mm) and longer (150–160 mm) distances (Figure 9b).

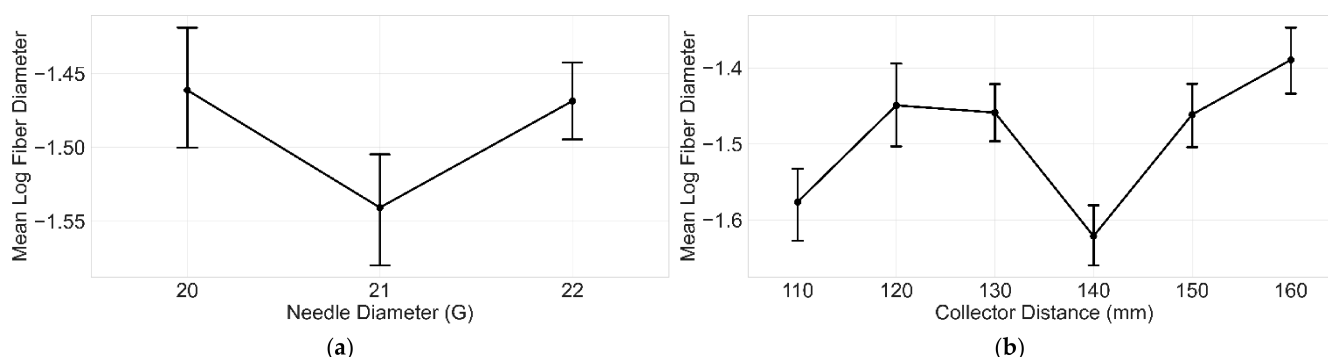


Figure 9. Main effects plots showing the influence of (a) needle diameter and (b) collector distance on the log-transformed mean fiber diameter, averaged across levels of the other factor.

The main effects plots further illustrate the trends identified by ANOVA: a mild decrease in mean log fiber diameter from G20 to G21 needles, followed by a slight increase

at G22, while the smallest diameters were generally achieved at intermediate collector distances of 130–140 mm.

The high residual variation (83.98%) observed in the two-way ANOVA model merits explicit discussion. In electrospinning, the charged polymer jet undergoes complex, inherently chaotic bending and whipping instabilities whose precise dynamics are sensitive to minute local variations in electric field, solution properties, and ambient conditions. These intrinsic process fluctuations, together with the stochastic nature of solvent evaporation and fiber deposition, contribute substantially to the observed scatter in fiber diameters even under nominally identical experimental settings. A secondary contribution arises from measurement uncertainty inherent to manual diameter quantification on SEM images, despite the use of calibrated software (Microscope VIS 2.0 Lite) and multiple measurements per fiber. Quantifying the exact partition of residual variance between process variability and measurement error would require a dedicated Gage Repeatability and Reproducibility study, which lies beyond the scope of the present optimization work. Critically, the highly significant *p*-values obtained for needle diameter, collector distance, and their interaction—despite the large residual—demonstrate that the investigated factors exert effects that are robustly detectable above the background noise. The physical consistency of the results, most notably the reproducible non-linear dependence of fiber diameter on collector distance with a clear, physically interpretable minimum at 140 mm across all three needle gauges, further corroborates the authenticity and reliability of the experimental findings. These trends align with electrohydrodynamic models of jet thinning and with previously reported behavior in PVA and other polymer systems.

Collectively, the statistical evidence, the visual confirmation provided by SEM imaging and diameter-distribution histograms, and the agreement with theoretical expectations establish that the identified optimal parameter combination—10 wt.% PVA solution, a collector distance of approximately 140 mm, and an appropriately selected needle gauge—provides a reliable and reproducible route to high-quality, bead-free PVA nanofibers. The strong interaction between needle diameter and collector distance underscores that process optimization must consider these factors jointly rather than in isolation.

4. Conclusions

In this study, the influence of solution concentration, tip-to-collector distance, and needle diameter on the morphology and diameter of electrospun Poly(vinyl alcohol) (PVA) nanofibers was systematically investigated. Increasing the PVA concentration from 5 wt.% to 10 wt.% promoted the transition from beaded structures to homogeneous, bead-free nanofibers, highlighting the importance of sufficient polymer chain entanglement for stable fiber formation. A non-linear relationship between fiber diameter and tip-to-collector distance was observed, with an optimal spinning distance of approximately 140 mm yielding the thinnest fibers. Needle diameter also affected fiber formation. While the G22 needle produced the finest fibers, at the optimal collector distance of 140 mm it also provided the narrowest diameter distribution, indicating the highest uniformity among the three needle gauges. Two-way ANOVA confirmed that needle diameter, collector distance, and their interaction significantly influenced fiber diameter. Overall, the combination of 10 wt.% PVA, a collector distance of approximately 140 mm, and an appropriately selected needle diameter provided the most favorable conditions for producing uniform, bead-free nanofibers. These findings provide practical guidance for optimizing electrospinning parameters in applications requiring controlled fiber morphology and diameter.

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R.B. and K.G.S.; visualization, D.S.G. All authors have read and agreed to the published version of the manuscript.

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Abbreviations

The following abbreviations are used in this manuscript:

PVA Poly(vinyl alcohol)

SEM Scanning electron microscopy

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