

Proceeding Paper

Binder Molecular Weight, Concentration, and Flow Rate Optimization for ZnO Nanofiber Synthesis for Electronic Device Applications [†]

Harshada Mhetre ^{1,*}, Vikas Kaduskar ¹, Prashant Chougule ¹, Yogesh Chendake ², Nithesh Naik ³, Pavan Hiremath ³ and Ritesh Bhat ⁴

- ¹ Department of Electronics & Communication Engineering, Bharati Vidyapeeth, College of Engineering, Deemed to Be University, Pune 411043, Maharashtra, India; vpkaduskar@bvucoep.edu.in (V.K.); pachougule@bvucoep.edu.in (P.C.)
- ² Department of Chemical Engineering, Bharati Vidyapeeth, College of Engineering, Deemed to Be University, Pune 411043, Maharashtra, India; yjchendake@bvucoep.edu.in
- ³ Department of Mechanical and Industrial Engineering, Manipal Institute of Technology, Manipal Academy of Higher Education, Manipal 576104, Karnataka, India; nithesh.naik@manipal.edu (N.N.); pavan.hiremath@manipal.edu (P.H.)
- ⁴ Department of Mechatronics, Rajalakshmi Engineering College, Rajalakshmi Nagar Thandalam, Chennai 602105, Tamil Nadu, India; riteshbhat.rb@rajalakshmi.edu.in
- * Correspondence: hvmhetre@bvucoep.edu.in
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Abstract: This study explores the critical factors influencing the properties of zinc oxide nanofibers, which are crucial for their applications in optoelectronics, solar cells, and biomedicine. We specifically investigate the impact of binder molecular weight, binder concentration, and solution flow rate on fiber shape, spinning ability, and diameter. By electrospinning zinc acetate, polyvinylpyrrolidone, and dimethylformamide solutions, we demonstrate that the composition, rheological characteristics, and processing parameters significantly affect nanofiber characteristics. We achieved optimized fiber diameters ranging from 24 to 62 nanometers through meticulous parameter modification. Further analysis via FESEM, XRD, and EDS confirms the suitability of these nanofibers for electronic applications, highlighting their potential contributions to the mentioned fields.

Keywords: electrospinning; ZnO; PVP; nanofiber; molecular weight; flow rate



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1. Introduction

Nanofibers have diameters of up to 100 nm [1]. They can be made from polymers or other materials as initiators, with optimised properties and an applicability based on their compositional or formation parameters [2]. These nanofibers are one-dimensional (1D) nanoarchitectural materials with a high surface-area-to-volume ratio that enhances ion transport while maintaining a high mechanical strength [3]. This architecture supports solar cells and biomedical and other electronic applications [4,5]. Many studies have focused on one-dimensional nanostructures such as nanowires, nanorods, nanotubes, and nanofibers, which exhibit ultrafine grain properties and an enormous surface area to volume ratio, suggesting a wide range of new applications [6]. Various polymers have been reported for the formation of nanofibers. PVP is one such polymer, which can conveniently be stretched by electrospinning to produce fibers [7].

Electrospinning is a simple and convenient method to form organic, inorganic, or composite nanofibers with controlled lengths and consistent diameters [8,9]. It involves using electrical force to pull charged filaments of polymeric material (solutions or melts) in nanometer dimensions [10]. Electrospinning has advantages over both electrospray

and traditional solution dry fiber-spinning. It has gained attractiveness in laboratory-scale production, as few industries are dedicated to fiber production [11]. The process does not use coagulation chemistry or high temperatures, which is advantageous during the production of fibers with extensive and intricate joints. The electrospinning of molten precursors can also be used to avoid solvent contamination in the final product [12].

Polymer properties and their molecular weight influence the morphologies and other properties of electrospun fibers. In theory, molecular weight affects the polymer chain length, which, in turn, affects the solution's polymer chain entanglement and viscosity [13]. This would, in turn, affect the flow parameters and their microstructure. At constant solution concentration, a change in morphology from beading to microbanding with an increase in molecular weight has been reported [14]. Thus, the molecular weight can control the polymer morphology to a greater extent, even at low solution concentrations [15].

Investigating the binder molecular weight, concentration, and flow rate is crucial in nanofiber synthesis for the following reasons:

Control over Fiber Properties: Binder molecular weight can impact the viscosity and rheological properties of the spinning solution. Understanding its influence helps researchers to control the fibre's diameter, morphology, and mechanical properties. Different applications may require nanofibers with specific characteristics, and adjusting the binder's molecular weight allows for the fine-tuning of these properties.

Optimizing Solution Compatibility: The concentration of the binder affects the overall solution's properties, including its viscosity and conductivity. Finding the right binder concentration is essential to ensuring that the electrospinning process operates smoothly and consistently. Too low a concentration can lead to instability during spinning, while too high a concentration can result in clogging or non-uniform fibres.

Production Efficiency: Flow rate plays a critical role in the electrospinning process. This determines the rate at which nanofibers are produced. Researchers can balance a high production efficiency with the control of fiber characteristics by optimising the flow rate. This is particularly important when scaling up nanofiber production for commercial applications.

Application-Specific Tailoring: Different applications, such as optoelectronics or tissue engineering, require nanofibers with specific properties. Investigating these parameters allows researchers to tailor nanofibers to meet the exact requirements of their intended application, ensuring that the resulting materials perform optimally in their intended role.

Investigating binder molecular weight, concentration, and flow rate in nanofiber synthesis is essential for achieving precise control of the nanofiber's properties, optimizing the electrospinning process, and tailoring the nanofibers to suit various applications. This research contributes to the development of advanced materials with enhanced performance characteristics for multiple industries and technologies.

The current work aims to investigate and optimize the binder (polymer) molecular weight, binder concentration, and the flow rate of prepared solution in the synthesis of ZnO nanofibers via electrospinning.

2. Experimental Section

The materials used were Polyvinyl pyrrolidone((C₆H₉NO)_n) (PVP, mol. Wt. = 10 k; 40 k; 1300 k). Zinc acetate ((CH₃CO₂)₂Zn) of molecular weight 183.48 and 99% purity. N, N-dimethylformamide (HCON(CH₃)₂) (DMF) [16]. All were purchased from Sigma-Aldrich, Bangalore, India and used without any further purification.

2.1. Optimization of Molecular Weight

In a typical synthesis, 21.5 wt% of PVP (with molecular weight = 10 k) and 1.8 g zinc acetate were mixed with 15 mL DMF using a magnetic stirrer. After a clear solution formed, mixing was stopped, and the mixture was left to sit at room temperature for approximately 18 h. The same procedure was carried out for the other two PVPs with molecular weights of 40 k and 1300 k. All three prepared solutions were used for electrospinning. A 2 mL syringe was filled with this precursor solution using a stainless-steel needle. The needle was

connected to the anode of a high-voltage power generator (0 to 50 kV), and an aluminium collector was grounded. A 20 kV DC input voltage was placed between the collector and needle, with the distance between them being maintained at 15 cm. A precise (2 mL/h) needle pump was used to maintain a liquid flow rate of 1.5 mL/h. The generated ZnO nanofibers were then gathered on an aluminium collector and kept at room temperature for about a day [17]. The experimental setup shown in Figure 1.

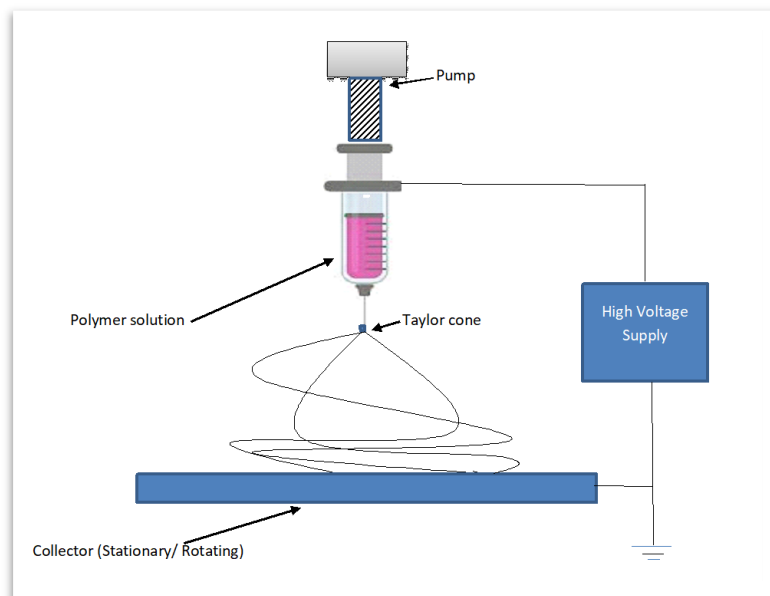


Figure 1. Electrospinning setup.

2.2. Optimization of the Binder Concentration

After optimizing the molecular weight to control the morphology of nanofibers, the next step was to optimize the binder concentration to control the fiber diameter. A solution containing PVP (with molecular weight = 1300 k) with concentrations 20 wt%, 17 wt%, and 14 wt%, respectively, in 15 mL DMF, is prepared using a magnetic stirrer. After dissolving PVP in a solvent, 1.8 g zinc acetate was mixed and further stirred. The mixture was allowed to sit at room temperature for approximately 18 h. All three prepared solutions were used for electrospinning.

A 2 mL syringe was filled with this precursor solution using a stainless-steel needle. The electrospinning parameters were the same as in previous experiments (applied voltage at 20 kV, flow rate at 1.5 mL/h, and distance between needle and collector at 15 cm).

2.3. Optimization of Flow Rate

After optimizing the molecular weight and concentration of the binder, flow rates were optimized to synthesize thin and continuous fibers. For this, using a magnetic stirrer, PVP (with molecular weight = 1300 k) was dissolved in 15 mL DMF with a concentration of 14 wt%. After dissolving PVP in a solvent, 1.8 g zinc acetate was mixed and stirring continued. The mixture was allowed to stir at room temperature for approximately 18 h. Prepared solutions were used for electrospinning.

A 2 mL polycarbonate syringe was filled with this precursor solution using a stainless-steel needle. Electrospinning was carried out at different flow rates: 1 mL/h, 0.8 mL/h and 0.5 mL/h. The applied voltage remained constant throughout these trials at 20 kV. The distance between the needle and collector was kept constant at 15 cm.

2.4. Characterization of Nanofibers

Later, the characterization of the morphological and electro-structural features of all three different samples was carried out. Scanning electron microscopes (FESEM-FEI Nova

Nano SEM 450, Hillsboro, OR, USA), energy-dispersive X-ray spectroscopy (EDS) (Bruker Flash 6I30, Billerica, MA, USA), and X-ray diffraction (XRD-Rigaku Miniflex, Tokyo, Japan) analysis was carried out for ZnO nanofibers, produced with different molecular weights of PVP.

3. Result and Discussion

ZnO is a promising material in optoelectronics applications. It has a low band gap energy of 3.2 eV, which results in the easy excitation of an electron to the conduction band [18]. Such excitation would trigger electron motion in a closed circuit. This resulted in its application in solar cells [19]. Such electron mobilization is critical during optoelectronics and sensor applications. Hence, it was thought to optimize the properties of ZnO. Their nanoparticles showed enhanced activity and an increased outcome due to the enhanced surface properties and their containing more free electrons than macro-material. Thus, defining a suitable method for the formation of nanoparticles and their property-tuning is essential. These properties seem to be dependent upon electrospinning parameters and solution composition [19]. Out of these components, the binder's molecular weight and concentration, would affect the flow properties and forces acting on the precursor solution. The flow parameters would also significantly affect the forces acting on the precursor solution in a constant electrostatic field [19]. These forces would affect the solution, nanofiber deposition, and formation of nanofibers.

Effect of binder polymer molecular weight:

The nanofibers were prepared using different molecular weights of PVP of 1300 k, 40 k, and 10 k Daltons. Instead of nanofibers, a porous structure was formed, as shown in Figure 2. Figure 2A,B for molecular weights 10 k and 40 k. Electrospinning results in particles of many different shapes and sizes, such as spherical, cylindrical, and irregular particles. The nanoparticles were developed and combined in cluster form to obtain a porous structure. Electrospun particles are a favourable option for scenarios that require a large surface area or where the particles must be readily dispersed in a liquid medium. At low concentrations and molecular weights, the cross-section of the fibers was round. A polymer's molecular weight affects its rheological properties, electrical conductivity, dielectric properties, and surface tension when dissolved in a solution. This would affect the transition of solution from the needle to collector. These rheological properties affect the flow, transition properties and forces applied to the polymer solution. This would affect the formation and properties of formed nanoparticles. PVP fibers experience a transition from a rounded surface to a flat surface as their molecular weight increases. This causes both the diameter of the fibers and the interfiber spacing to increase [20].

The electrospinning fiber's shape and diameter depend on the polymer solution's intrinsic properties, including its polymeric material, molecular weight or polymer chain congruence, as well as its concentration or viscosity, and stability. The surface tension and viscosity of the polymer solution influence the diameter and shape of the fibers. The viscoelastic properties of polymer solutions are influenced by their surface tension and viscosity. A polymer solution's viscosity and stretching behaviour can be influenced by its constituent molecules' molecular weight and chain congruence. Such an increased viscosity and reduced stretchability in high-molecular-weight polymers results in the production of thicker fibers. The spinnability and stability of the electrospinning process are contingent upon the viscosity or concentration of the polymer solution. Elevated concentrations and viscosities lead to the formation of thicker fibers. The stretchability of polymer solutions is influenced by various factors, such as polymer concentration, molecular weight, and viscosity. Stretchy solutions yield thinner fibers. The electrospinning process is influenced by the electrical properties of the polymer solution, which, in turn, affect the formation and elongation of the polymer jet. The electrical charge density of polymers is influenced by the conductivity of the solution, impacting the stretching and deformation of the polymer jet. The wettability of electrospun fibers is contingent upon the polarity and surface tension of the solvent. A greater surface tension leads to the formation of thicker fibers, whereas

a lower surface tension leads to the formation of thinner fibers. The morphology and dimensions of electrospun fibers are contingent upon the inherent characteristics of the polymer solution [21].

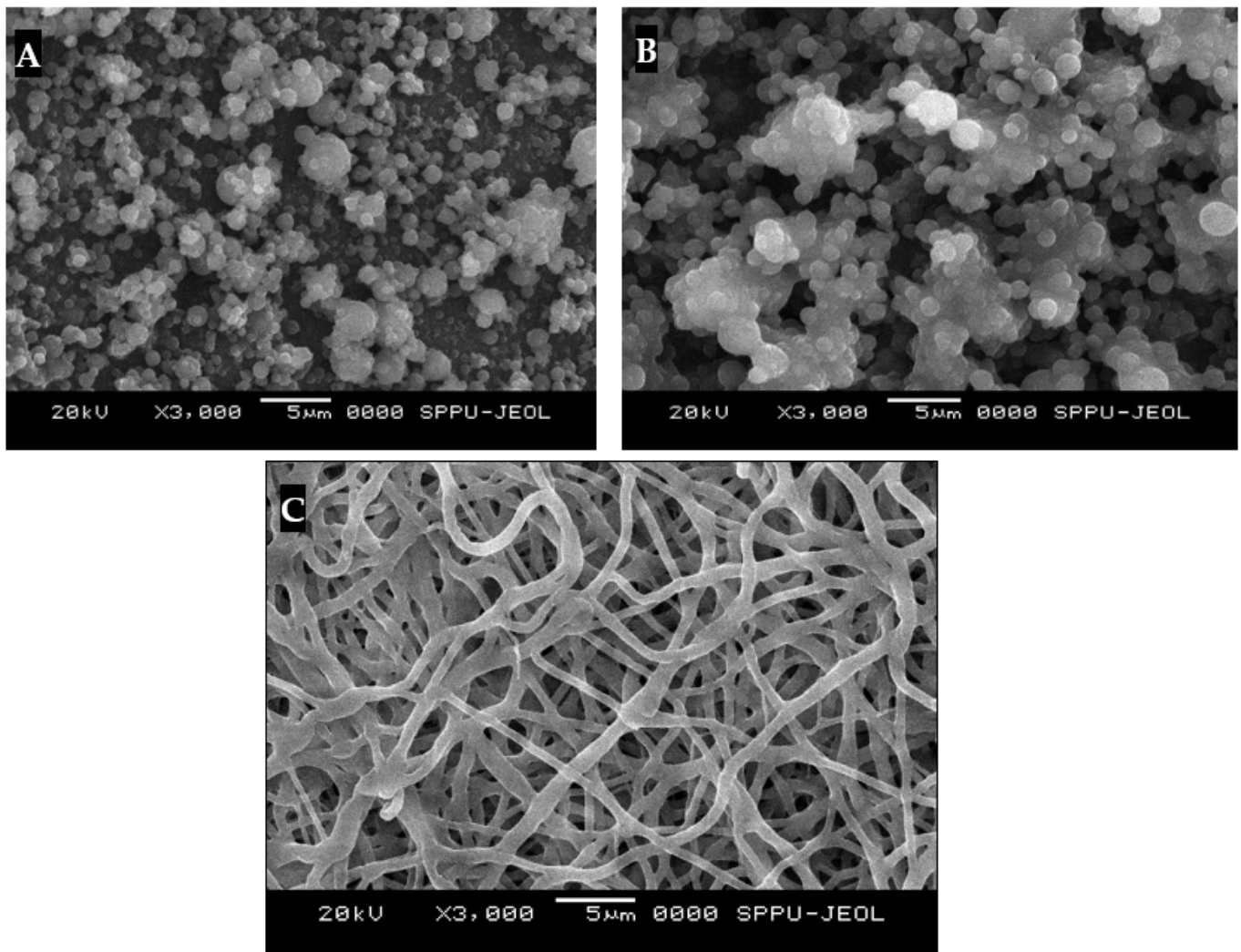


Figure 2. FESEM image of PVP + ZnO nanofiber with PVP MW of 10 k (A), 40 k (B), and 1300 k (C).

The characteristics of nanofibers are influenced by the flow rate of the polymer solution during electrospinning. Increasing the flow rate typically results in the augmentation of fiber thickness and width. The excessive flow rate during electrospinning can result in instability, forming bead-on-string structures characterized by polymer solution droplets connected by thin fibers. The quality and consistency of the fiber may be compromised. The insufficient flow rate during the electrospinning process can result in inconsistent fiber production and instability caused by material flow. The electrospinning flow rate is contingent upon several factors, including the viscosity and concentration of the polymer solution, the distance between the spinneret and collector, and the electrical properties of the system. To produce nanofibers of superior quality for a particular application, it is necessary to optimize the flow velocity [22].

3.1. Field Emission Scanning Electron Microscopy (FESEM)

FESEM is a method used for seeing microscopic topographical features on a surface or a complete fractional item [23]. FEI Nova Nano SEM 450 (FESEM) images of ZnO nanofibers are shown in Figures 2–4. The obtained fibers were continuous and free from beads or

breaks, as shown in Figure 4, while beads were generated when lower-molecular-weight PVP was used, as seen in Figures 2 and 3. Under the present conditions, the minimum diameter is 24 nm [24]. According to the literature, a high molecular weight stimulates the formation of larger diameter fibers, while a lower molecular weight causes the fibers to shrink in size [21].

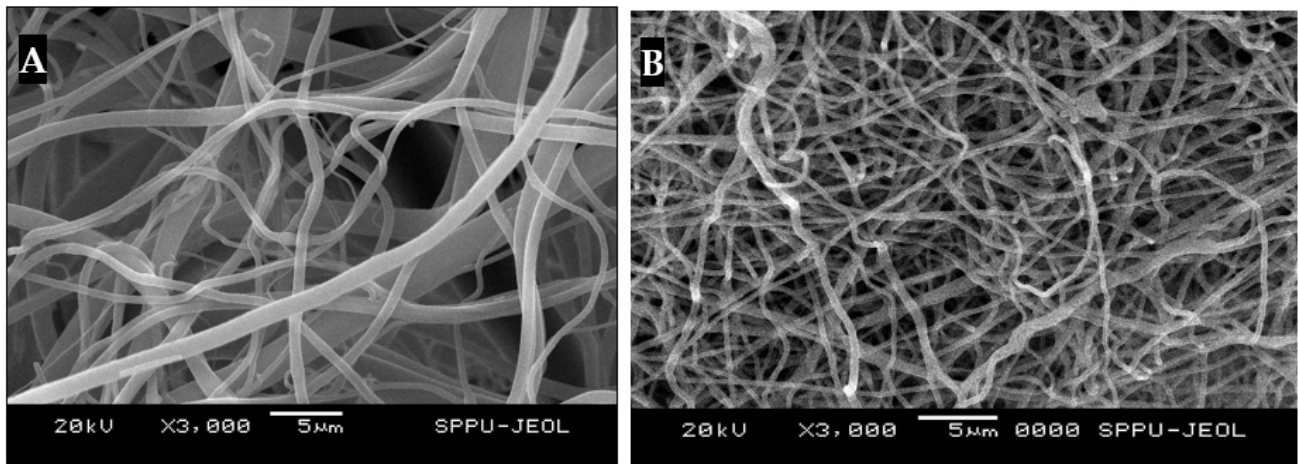


Figure 3. FESEM image of PVP + ZnO nanofiber with PVP MW of 1300 k, the concentration of binder at (A) 17 wt%, and (B) 14 wt%, with flow rate at 1.5 mL/h.

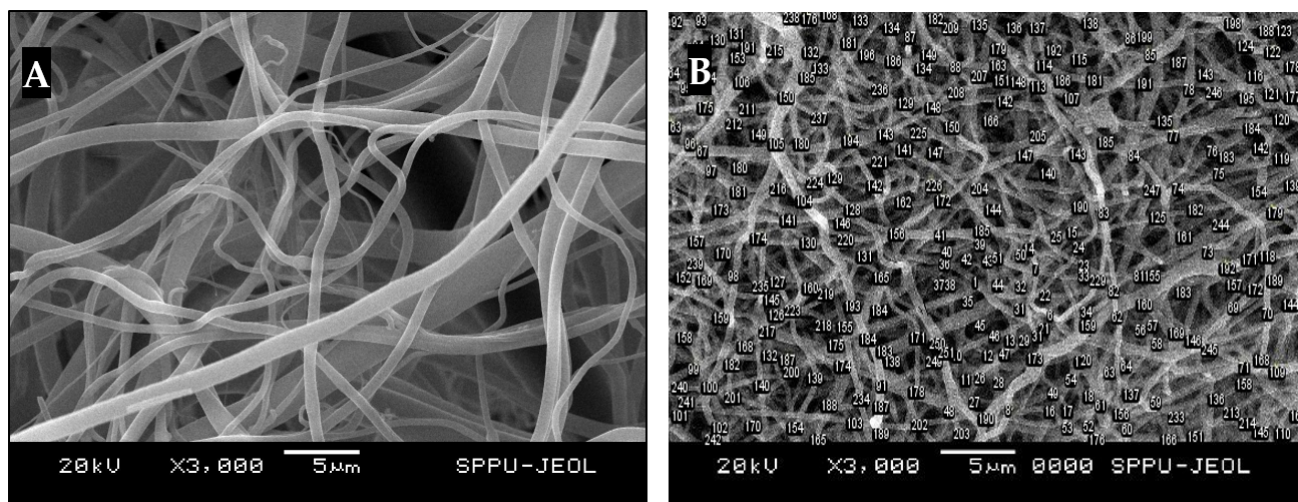


Figure 4. FESEM image of PVP + ZnO nanofiber with PVP MW of 1300 k concentration of binder at 14 wt%, and flow rate at (A) 0.8 mL/h and (B) 0.5 mL/h.

The smaller fiber diameter that the SEM saw coincided with a lower polymer content that resulted in the solution having a lower viscosity (Table 1, Figure 3). Beads-free continuous fibers were seen in FESEM pictures of the fibers electrospun from a solution containing 14 wt%, which is a typical outcome of electrospinning from a dilute solution.

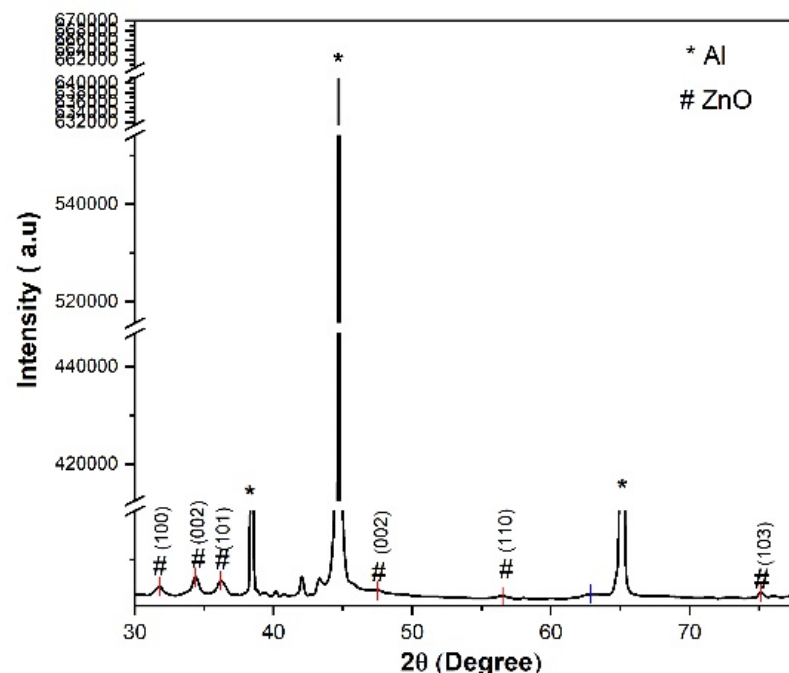
Figure 4 shows the influence of the solution flow rate. At a modest flow rate (0.5 mL/h), controlled fiber diameters with a relatively narrow dispersion of fiber width were found.

Table 1. Optimization of process parameters.

Exp. No	The Molecular Weight of the Binder	The Concentration of Binder (wt%)	Flow Rate (mL/h)	Applied High Voltage (kV)	Distance between Needle Tip to a Collector (cm)
1	10 k	21.5	1.5	20	15
2	40 k	21.5	1.5	20	15
3	1300 k	21.5	1.5	20	15
4	1300 k	20	1.5	20	15
5	1300 k	17	1.5	20	15
6	1300 k	14	1.5	20	15
7	1300 k	14	1	20	15
8	1300 k	14	0.8	20	15
9	1300 k	14	0.5	20	15

3.2. X-ray Diffraction (XRD)

Nanofiber structure was studied via X-ray diffraction analysis. The as-prepared samples were calcined at 450 °C for two hours in-atmosphere, and their XRD patterns are displayed in Figure 5 XRD measurements on an aluminium substrate with calcination were used to characterize the structural properties of the deposited layer [25]. The nanofibers generated by electrospinning were collected on aluminium foil, whereby aluminium peaks at around are also observed (Figure 6) [26,27]. Five diffraction peaks correspond to the wurtzite crystal structures (100), (002), (101), (110), and (103), respectively, at 31.76°, 34.34°, 36.20°, 56.50°, and 62.84°. Five diffraction peaks exist at 31.76°, 34.34°, 36.20°, 56.50°, and 62.84°, corresponding to the wurtzite crystal structures (100), (002), (101), (110), and (103), respectively. All apparent diffraction peaks, which were authorized when compared to the Joint Committee on Powder Diffraction Standards (JCPDS) card of ZnO (36–1451), confirmed that the pure hexagonal wurtzite structure of the ZnO nanofibers was created with great crystallinity [28,29]. Additionally, significant diffraction peaks in the XRD data were produced by the aluminum foil substrate utilized to gather fibers [28].

**Figure 5.** XRD pattern of calcined ZnO fibers with aluminium foil as substrate.

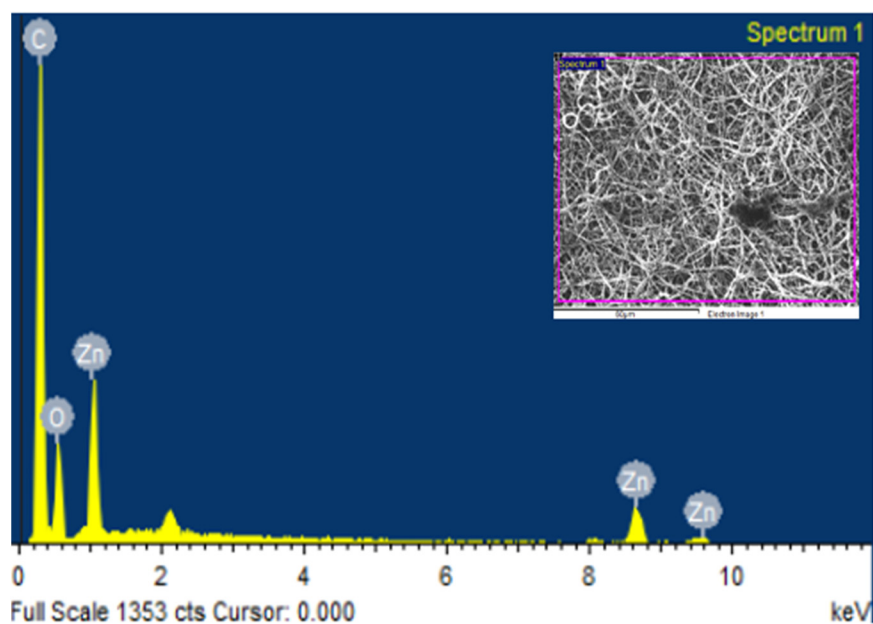


Figure 6. EDS spectra of calcinated ZnO fibers.

3.3. Energy-Dispersive X-ray Spectroscopy (EDS)

Energy-Dispersive X-ray Spectroscopy (EDS) (Bruker X-Flash 6130) is a method that analytically identifies the elements (elemental composition) present in each material. Figure 6 shows that strong peaks corresponding to Zn and O were found in the spectrum, which confirms the formation of the ZnO thin film. Carbon is formed during handling and electrospinning. These components are outcomes of combinations of polymer binders, where the presence of Zn was found in three different crystal lattices. These are distributed throughout the electrospun matrix. The concentration and distribution are higher at the nodes of fibers. These nodes are prone to providing nanoparticles rather than fibers during calcination. As discussed earlier, a similar particle formation and segregation are observed in SEM.

4. Conclusions

In conclusion, our research findings have profound implications for the enhancement of electronic device applications by fine-tuning nanofiber synthesis parameters. The self-bundling nature of a single droplet, resulting in the formation of multiple fibers with precise size and morphology control, offers an exciting avenue for property optimization in the context of electronic devices.

Of particular significance is our discovery that employing PVP with a molar mass of 13,000 k enables the production of nanofibers with an average size of as small as 271.30 nm at the appropriate PVP concentration. Notably, we achieved the smallest fiber diameter of 24 nm. These dimensions hold great promise for electronic device applications, where their miniaturization and control over material properties are essential for their superior performance.

Furthermore, our investigation into the molecular weight of PVP shed light on its role in influencing fiber morphologies. Lower-molecular-weight PVP led to the oxidation and combustion of PVP filaments during electrospinning, forming smaller particles. In contrast, higher-molecular-weight PVP preserved the thread's integrity and facilitated the creation of smooth nanofibers. Understanding the molecular weight's impact on nanofiber structure is invaluable for tailoring materials to meet the specific demands of electronic device applications.

Future research endeavours in this area should focus on leveraging these optimized parameters to design nanofiber-based materials explicitly engineered for electronic devices.

This could entail exploring novel combinations of binders, solvents, and processing conditions to achieve even finer control over nanofiber properties. Additionally, efforts should be directed towards integrating these nanofibers into electronic components and assessing their real-world performance.

Building upon these findings, we anticipate substantial advancements in developing electronic devices with an enhanced performance, miniaturization, and adaptability to various applications.

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Conflicts of Interest: The authors declare, there are no conflicts of interest.

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