



Morphological Study of Electrospun Polyvinylpyrrolidone Fibers at High Concentration Using Water and Ethanol Solvents

Doni Bowo Nugroho*, Nada Nadzira Ayasha Kamal, Jenni Bunga Enjelita Sidabalok, Rosita Wati, Nova Resfita, and Muhammad Wildan Gifari

Biomedical Engineering Study Program, Institut Teknologi Sumatera, Lampung, Indonesia 35365

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Abstract

In this study, polyvinylpyrrolidone (PVP) ($M_w \sim 40,000$ g/mol) fibers were fabricated using distilled water and ethanol via an electrospinning approach. Morphological evaluations were carried out with a Scanning Electron Microscopy (SEM). Fiber diameters were analyzed with ImageJ. Solution Concentration of PVP with solvents adjusted to 50% (m/v), respectively. Then, the solution was electrospun with voltages of 8 and 12 kV. PVP/distilled water solution produced a relatively regular fiber network with enlarged junctions at 8 kV. However, it was ribbon-like, bead-like, and film-like in voltages of 12 kV. This structure was attributed to high surface tension and slow solvent evaporation. Another solution, PVP/ethanol, produced a relatively regular fiber network at 8 kV. The structure was different at 12 kV voltages. Globular and particle-like morphologies appear in these conditions. Fiber diameter was analyzed in the layers formed under 8 kV electrospinning conditions. The fiber diameter in PVP/distilled water was 1.05 ± 0.50 μm . Meanwhile, the fiber diameter in PVP/ethanol was 2.46 ± 0.64 μm . Although the fiber diameter was larger in PVP/ethanol, the resulting fiber was more continuous and more stable.

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Abstrak

Dalam penelitian ini, serat polivinilpirolidon (PVP) ($M_w \sim 40,000$ g/mol) dibuat menggunakan air suling dan etanol melalui pendekatan elektrospinning. Evaluasi morfologi dilakukan dengan *Scanning Electron Microscopy* (SEM). Diameter serat dianalisis dengan ImageJ. Konsentrasi Larutan PVP dengan pelarut-pelarut disesuaikan masing-masing menjadi 50% (m/v). Kemudian, larutan tersebut dipintal secara elektro dengan tegangan 8 dan 12 kV. Larutan PVP/aquades menghasilkan jaringan serat yang relatif teratur dengan sambungan yang lebar pada 8 kV. Namun, lapisan terbentuk seperti pita, seperti titik-titik butiran, dan seperti film pada tegangan 12 kV. Struktur ini disebabkan oleh tegangan permukaan yang tinggi dan penguapan pelarut yang lambat. Larutan lainnya, PVP/etanol, menghasilkan jaringan serat yang relatif teratur pada 8 kV. Strukturnya berbeda pada tegangan 12 kV. Morfologi globular dan seperti partikel muncul dalam kondisi ini. Diameter serat dianalisis pada lapisan yang terbentuk pada tegangan elektrospinning 8 kV. Diameter serat PVP/aquades adalah $1,05 \pm 0,50$ μm . Sementara itu, diameter serat PVP/etanol adalah $2,46 \pm 0,64$ μm . Meskipun diameter serat lebih besar pada PVP/etanol, serat yang dihasilkan lebih kontinu dan lebih stabil.

1. Introduction

Polyvinylpyrrolidone (PVP) is a widely utilized synthetic polymer across biomedical, pharmaceutical, and electronic fields. This non-toxic polymer is biocompatible and highly soluble in various organic solvents, such as water and ethanol—qualities that are essential for various technological purposes (Kamli et al., 2023). PVP has the molecular formula $(C_6H_9NO)_n$ and contains carbonyl and amide groups, which enable it to interact chemically and physically with other materials through hydrogen bonding and van der Waals interactions (Husen et al., 2024). These

* Corresponding author.

E-mail address: doni.nugroho@bm.itera.ac.id

properties make PVP highly promising for a wide range of applications including coating manufacturing. One form of coating currently being widely developed is fiber. This coating offers many advantages, including its high surface area, porosity, tunable physical properties, and ability to mimic extracellular matrices in biomedical applications (Anjum et al., 2023; Ar et al., 2021; Rahanti & Kusumawati, 2022; Yuan et al., 2023).

Among various fabrication methods, the electrospinning technique is a versatile method for producing fibers from a wide variety of polymers (Zahra et al., 2024). It offers several adjustable parameters, such as applied voltage (kV), flow rate (mL/h), the distance between the needle tip and the collector (cm), and needle gauge (G) (Şenol & Akkoyun, 2020). These parameters significantly influence the morphology of the resulting fibers. In addition, polymer concentration and solvent characteristics are critical factors that determine the quality of the fiber morphology (Nasouri et al., 2015; Vongsetskul et al., 2015).

Previous studies have reported that PVP with a molecular weight of 1,300,000 g/mol has been widely used to produce uniform and continuous fibers with nanoscale (nanofibers) in the concentration range of 5–12 wt% using solvents such as ethanol, acetic acid, or water (Srilistari et al., 2021; Virginia et al., 2020). The choice of solvent also affects the resulting nanofiber morphology. In contrast, low-molecular weight PVP (10,000 g/mol and 55,000 g/mol) requires higher concentrations—above 50%—to obtain well-formed nanofibers (Kamli et al., 2023; Zahra et al., 2024). Furthermore, optimizing the electrospinning parameters is crucial for achieving uniform fibers when using low-molecular weight PVP (Ataei et al., 2024). However, studies focusing on high-concentration PVP solutions remain limited, even though such systems could provide valuable insights into the relationship between processing parameters and fiber structure.

This study focuses on investigating the morphology of fibers produced from low-molecular weight PVP (~40,000 g/mol). The polymer solutions were prepared at a concentration of 50% m/v, and electrospinning was conducted at two applied voltages: 8 kV and 12 kV. The resulting fiber morphology was analyzed using scanning electron microscopy (SEM). The findings were then evaluated and compared with previous studies. This work is expected to contribute to a better understanding of the limitations and potential of electrospinning PVP at high concentrations for future biomedical applications (Hou et al., 2018; Yuan et al., 2023).

2. Research Methods

2.1 Materials

Polyvinylpyrrolidone (PVP) (K30, Mw ~40,000 g/mol) was used as the polymer to produce fibers. Distilled water and ethanol (analytical grade, Merck) were employed as solvents. All chemicals were used without further modification.

2.2 Preparation of PVP Solutions

Two types of PVP solutions were prepared at a concentration of 50% (m/v), namely PVP dissolved in distilled water (PVP/distilled water) and PVP dissolved in ethanol (PVP/ethanol). Each solution was stirred at room temperature (~25 °C) for 1 hour until a homogeneous viscous solution was obtained. The prepared solutions were then transferred into a 10 mL plastic syringe equipped with a stainless-steel needle. The overall process of preparing the PVP solution is illustrated in **Figure 1a**.

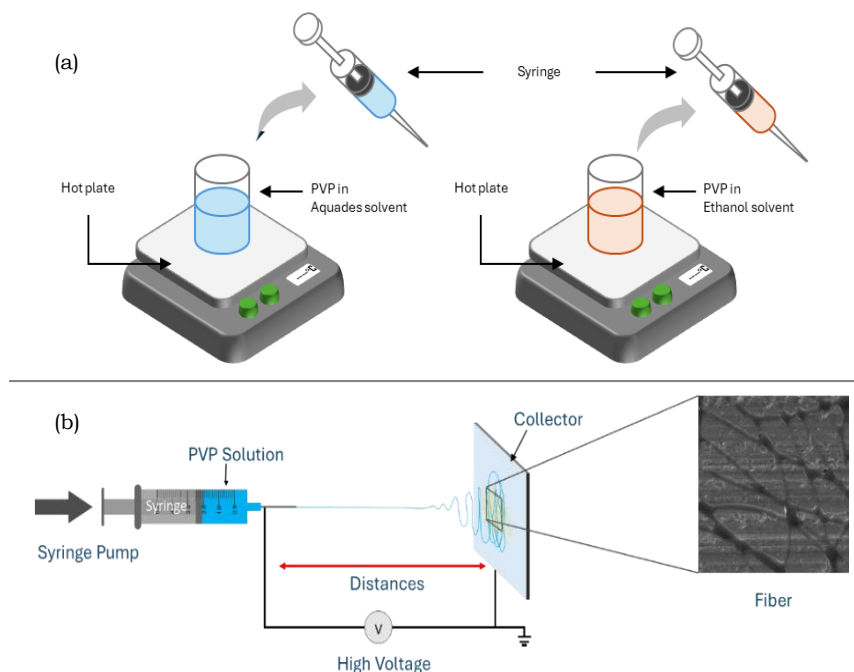


Figure 1. (a) Preparation process of PVP solution using distilled water and ethanol as solvents. (b) Schematic diagram of the electrospinning setup.

2.3 Electrospinning Process

Electrospinning was performed using a laboratory-scale electrospinning unit (ILMI-N101, Indonesia). The process parameters were set as follows: applied voltages: 8 kV and 12 kV; tip-to-collector distance: 15 cm; solution flow rate: 0.005 mL/min (0.3 mL/h), controlled using a syringe pump; collector: aluminum foil placed on a collector plate; deposition duration time: 10 minutes. During the electrospinning process, ambient temperature and humidity were used. The overall setup of the electrospinning process is illustrated in the schematic diagram shown in **Figure 1b**. After the electrospinning process, the sample was cut into small pieces for further morphological testing.

2.4 Characterization of Fibers

The surface morphology of electrospun PVP fibers was characterized using Scanning Electron Microscopy (SEM, JSM-6510LA, JEOL, Japan) at an accelerating voltage of 10 kV. Before SEM analysis, each sample was coated with a thin layer of metal to enhance surface conductivity and prevent charging during observation. The fiber diameter distribution was analyzed from SEM images using ImageJ software, with at least 30 measurements taken per sample to obtain the average diameter distribution. After SEM characterization, the samples cannot be reused because the metal coating process to enhance surface conductivity can alter the original properties of the material, especially in non-conductive polymers such as PVP.

3. Results and Discussions

The electrospinning process is influenced by various parameters, particularly the characteristics of the PVP solution (such as solvent type, concentration, surface tension, conductivity, and viscosity) and operational conditions (applied voltage, nozzle-collector distance, flow rate, and humidity) (Utkarsh et al., 2020). In this study, two different solvents—distilled water and ethanol—were used at two electrospinning voltages, 8 kV and 12 kV, to investigate the combined effect of these parameters on the surface morphology of PVP fibers.

3.1 PVP Fibers with Distilled Water Solvent

Error! Reference source not found. **a–d** shows the SEM morphology of PVP fibers electrospun using distilled water as the solvent. The images were taken at 10,000 $\times$ magnification to observe the fiber structures in detail. At an electrospinning voltage of 8 kV, the deposition morphology appears to form continuous fibers (see **Figure 2a–b**). However, there is some fiber enlargement at the intersections between fibers. This phenomenon likely occurs due to the low viscosity of the solution. The low viscosity is due to the solution still having a low concentration (Nasouri et al., 2015). Furthermore, the solution becomes hydrophilic due to the water solvent, resulting in a slow evaporation process. The slow evaporation rate does not maximize drying of the polymer when deposited onto the collector. This is consistent with previous research that found this condition will produce morphologies such as flattened ribbons and beads (Mikheev et al., 2013).

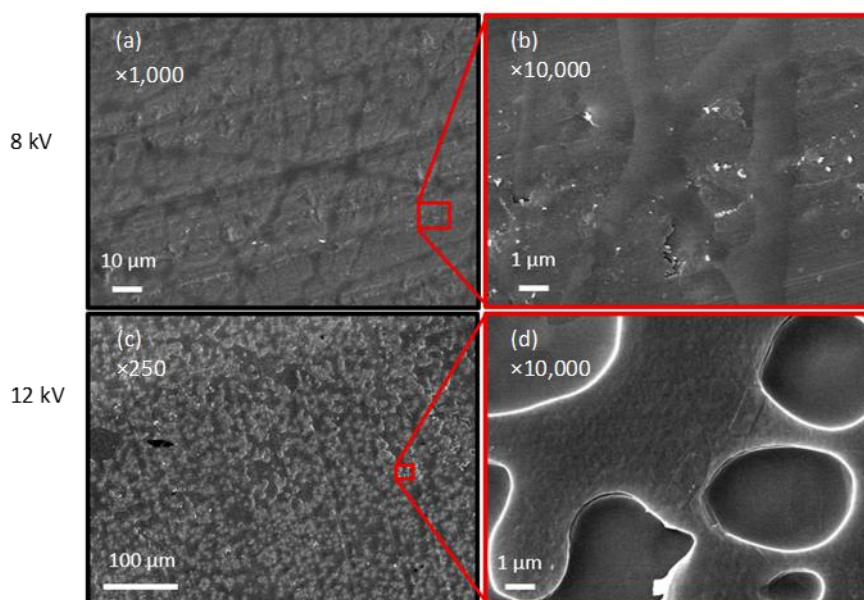


Figure 2. Surface morphology of PVP layers prepared with distilled water as solvents at electrospinning voltages of (a–b) 8 kV and (c–d) 12 kV.

The electrospinning voltage was increased to 12 kV, resulting in a layer with the morphology shown in **Figure 2b–c**. The morphology differs significantly in this situation. The fibers that should have formed did not appear at all. SEM images show a layer with non-uniform pores. The pores appeared circular or spherical in shape. This phenomenon indicates phase separation during the drying process, or bead-on-membrane formation (Cao et al., 2022). It is commonly reported when polar solvents with low volatility are used under high voltage conditions (Nguyen et al., 2023). Furthermore, the strong electrostatic force at 12 kV induced rapid phase separation, leading to solvent-

rich regions that evaporated slowly and formed large pores (Xue et al., 2019). This indicates that the electrospinning process was not optimal and even failed to produce fibers. Higher voltages are required for solutions with lower viscosities. A PVP solution with a 50% concentration at 12 kV electrospinning voltage was not able to produce good fibers.

From these results, it can be concluded that an applied voltage of 8 kV is more effective for producing fiber structures from water-based PVP solutions, although the fiber diameter distribution remained heterogeneous. Further variations in parameters, such as polymer concentration and applied voltage, are required to confirm and optimize these findings (Nasouri et al., 2015; Vongsetskul et al., 2015).

3.2 PVP Fibers with Ethanol Solvent

Figure 3a-d are SEM images of the PVP coating produced by the electrospinning process. At an electrospinning voltage of 8 kV, the coating morphology exhibits the formation of fibers. The resulting PVP/ethanol fibers differ in size at the junctions between the fibers, even forming beads (Korycka et al., 2018). This can occur when the fibers formed on the collector are not yet dry and are already overlapped by the next fiber. Another influencing factor is the use of ethanol as a solvent. Ethanol has a lower surface tension than water, making the electrospinning process easier. However, high evaporation rates or volatility can cause problems with the needle during the electrospinning process (Ewaldz et al., 2022). This can lead to uneven fiber yield and the appearance of beads.

At an electrospinning voltage of 12 kV, the resulting PVP/ethanol coating morphology is shown in **Figure 3c-d**. The coating forms fibers, but they are not continuous, and their diameters appear very inconsistent. The fibers appear broken despite having visible fiber paths. The high voltage creates a strong electric field, and the low viscosity of the solution causes the fibers to break apart into small droplets with paths (Ahmadi Bonakdar & Rodrigue, 2024). High voltage, rapid evaporation, and low ethanol viscosity result in an unstable jet stream, resulting in poor fiber formation (Shabani et al., 2025; Shim & Kouh, 2020).

Comparing electrospinning results at two different voltages, 8 kV and 12 kV, provides new insights. For low-molecular-weight PVP, further optimization of the voltage setting is necessary, even with increased concentration. At 8 kV, the fiber yield is more visible and offers potential for further optimization compared to fiber yield at 12 kV. Previous studies reported for low-molecular-weight PVP indicated that increasing the solution concentration is necessary to obtain optimal fiber yield (Pinarbaşı & Cengiz Çallıoğlu, 2022; Zahra et al., 2024).

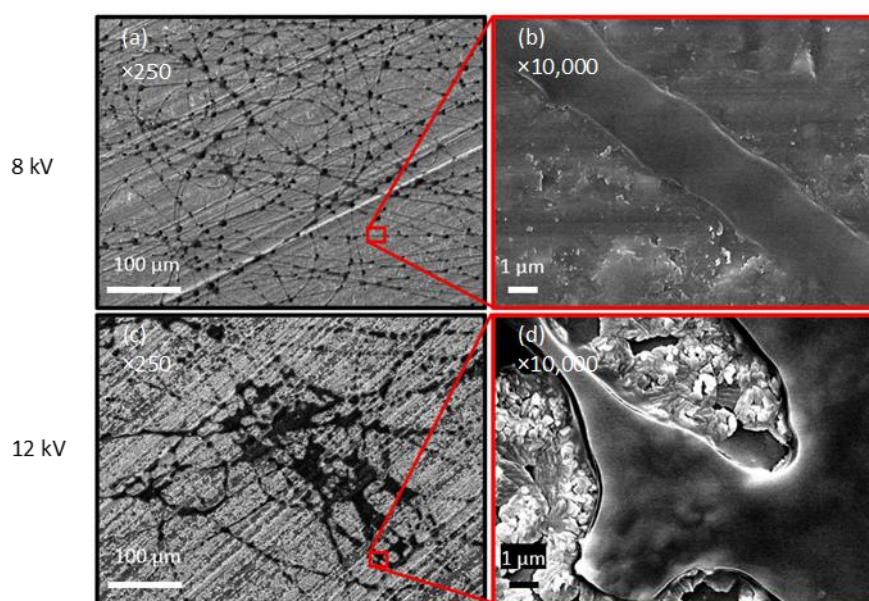


Figure 3. Surface morphology of PVP layers prepared with ethanol as solvents at electrospinning voltages of (a–b) 8 kV and (c–d) 12 kV.

3.3 Fiber Diameter Distribution

Figure 4 shows the diameter distribution of PVP fibers with water and ethanol solvents. The two images analyzed are the results of PVP deposition at an electrospinning voltage of 8 kV. These two layers were chosen because they produced relatively successful fiber formation and showed continuous fibers at 8 kV. Quantitative diameter analysis was not conducted for samples electrospun at 12 kV because the resulting structures were bead-rich and film-like, indicating incomplete fiber formation. The PVP fibers with water solvent showed a diameter distribution between 0.31 and 1.83 µm, with a peak diameter of ~0.69 µm. The average fibers diameter was 1.05 ± 0.50 µm (see **Figure 4b**). This broad distribution indicates non-uniform fiber formation, with the presence of beads and fragmented particle-like structures (see **Figure 4a**). Such instability is consistent with previous studies reporting that water-based PVP electrospinning often results in unstable structures due to the high surface tension and slow evaporation rate of water (Virginia et al., 2020).

Figure 4d shows the diameter distribution of PVP/ethanol fibers. Fiber deposition conditions at an electrospinning voltage of 8 kV. Fiber diameters ranged from 1.29 µm to 3.54 µm with a dominant diameter range of

1.85–2.41 μm . The average fiber diameter was $2.46 \pm 0.64 \mu\text{m}$. These results indicate that although the fiber diameter with ethanol solvent was on average larger than the fiber diameter with water solvent, the diameter distribution was narrower and more uniform (see **Figure 4c**). This condition shows the stability of the jet flow. These results are similar to previous research reports that solvents with high volatility such as ethanol can improve the stability of jet flows and continuous fibers, even though the resulting diameter is larger (Cengiz Çallioğlu & Kesici Güler, 2019; Kamli et al., 2023; Srilistari et al., 2021; Sukowati et al., 2023; Utkarsh et al., 2020).

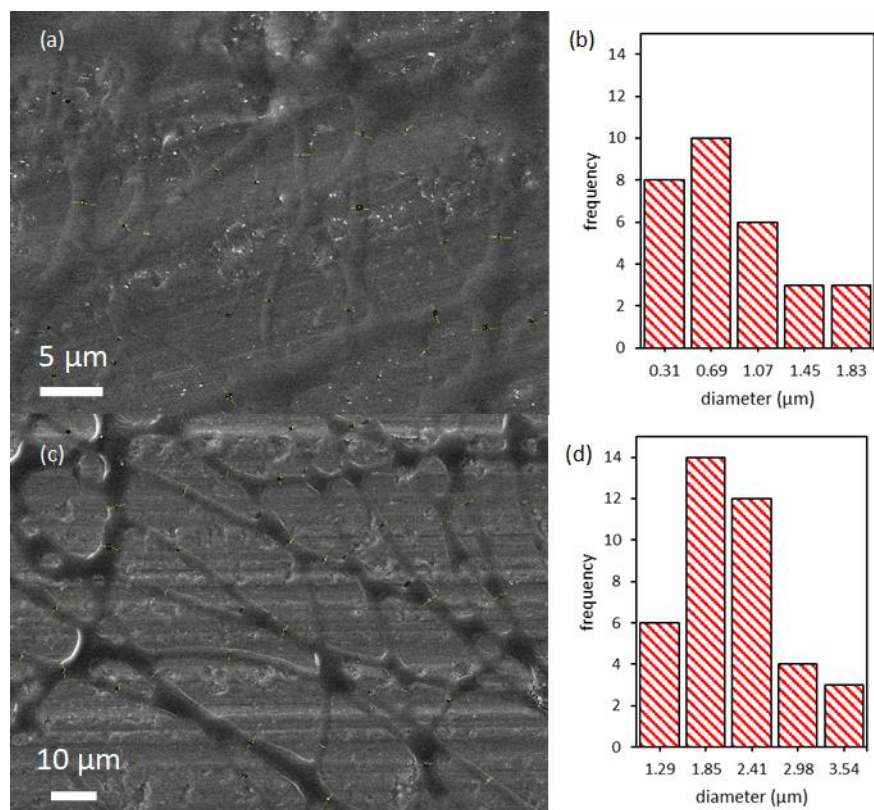


Figure 4. Morphology and fiber diameter distribution of electrospun PVP layers prepared with (a–b) distilled water and (c–d) ethanol at an applied voltage of 8 kV.

3.4 Relevance to Previous Studies

Table 1 presents a summary of previous studies on the fabrication of PVP fibers in different molecular weights, solvents, and electrospinning parameters. Polyvinylpyrrolidone (PVP) with a molecular weight of about 1,300,000 g/mol has been widely employed in electrospinning approach. Aria et al. (2016) reported that PVP solutions with concentrations ranging from 5 to 12 wt% could form nanofibers with diameters between 0.4 and 2.8 μm when electrospun at an applied voltage of 19 kV. In another study, Utkarsh et al. (2020) employed PVP of the same molecular weight using ethanol as the solvent and successfully obtained nanofibers within a similar concentration range of 8–12 wt%. The resulting fibers were also continuous with a diameter range of 0.53 to 2.54 μm .

PVP with a molecular weight of 360.00 g/mol has also been investigated for its ability to produce nanofibers by Sukowati et al. (2023). Nanofibers can be produced with PVP at concentrations of 2–8 wt% using ethanol as a solvent. The obtained diameters ranged from 0.06 to 0.33 μm at a voltage setting of 14.5 kV. These studies have demonstrated that low concentrations and relatively high voltages can produce continuous fibers with stable jets.

In contrast, studies using aqueous solvents (e.g., ultrapure water, rose water, lavender water) generally produce bead-like morphologies. Cengiz Çallioğlu & Kesici Güler (2019) reported that 12 wt% PVP (Mw 360,000 g/mol) in ultrapure water produced nanofibers with beads $\sim 0.16 \mu\text{m}$ in diameter, highlighting the difficulty of stabilizing water-based electrospinning due to high surface tension and slow evaporation. Similar bead-like structures were reported using rose water and lavender water as solvents.

Zahra et al. (2024) examined a different aspect, focusing on the molecular weight of PVP (10,000 and 55,000 g/mol). In their work, two concentrations (80 wt% and 50 wt%) were tested both dissolved in ethanol. Continuous fibers were obtained, with diameters of approximately 0.25 and 0.38 μm , respectively. Based on these results, the ethanol solvent can support continuous fiber formation. Variations in concentration are necessary to determine the best fiber yield. Low molecular weight PVP requires higher concentrations than high molecular weight PVP.

The results of this study differ significantly from previous studies because the concentration used was very high. Both PVP/distilled water and PVP/ethanol at 50% (m/v) produced bead-rich, irregular, or film-like morphologies, regardless of the applied voltage (8 or 12 kV). The excessive viscosity at these high concentrations inhibited jet elongation, resulting in unstable structures, unlike the continuous fibers obtained at lower concentrations. This shows that although previous studies focused on optimizing electrospinning in the moderate concentration range, this study reveals morphological limitations that arise at very high PVP concentrations.

Table 1. Comparative Evaluation of Electrospun PVP Fibers Reported in Previous Studies.

Materials	Mw (g/mol)	Solvent	Concent.	Voltage (kV)	Flowrate (mL/h)	Distance (cm)	Fiber results	Diameter (μm)	Year	Ref.
PVP	1,300,000	Ethanol	5 wt% 6 wt% 8 wt% 12 wt%	19	0.1	10	nanofiber	0.40-2.80	2016	(Aria et al., 2016)
PVP	360,000	ultra-pure water	12%	26.4	0.8	19	nanofiber with beads	0.16	2019	(Cengiz Çallioğlu & Kesici Güler, 2019)
		Rose water	12%	26.4	0.8	19	nanofiber with beads	0.16		
		Lavender water	12%	26.4	0.8	19	nanofiber with beads	0.12		
		Acetic acid	12%	26.4	0.8	19	nanofiber	1.00		
		Ethanol	12%	26.4	0.8	19	nanofiber	0.72		
PVP	1,300,000	Ethanol	8 wt% 10 wt% 12 wt%	10, 13, 15	0.6, 0.8, 1	10,13,15	nanofiber	0.54-2.54	2020	(Utkarsh et al., 2020)
PVP	360,000	Ethanol	2,4,8 wt%	14.5	0.6	15	nanofiber	0.06-0.33	2023	(Sukowati et al., 2023)
PVP	10,000	Ethanol	80 wt%	20	0.5	14	nanofiber	0.24	2024	(Zahra et al., 2024)
	55,000	Ethanol	50 wt%	20	1.0	14	nanofiber	0.38		
PVP	~40,000	Ethanol	50% (m/v)	8, 12	0.3	15	fiber with beads	1.05	2025	This work
		Distilled water	50% (m/v)	8, 12	0.3	15	fiber with beads	2.46	2025	This work

4. Conclusion

Morphology was studied for electrospun Polyvinylpyrrolidone (PVP) fibers at a concentration of 50% (m/v) using distilled water and ethanol as solvents. PVP had a molecular weight of 40,000 g/mol and electrospinning was done at 8 and 12 kV. SEM characterization of the fibers and ImageJ analysis were performed. The formation of fibers was influenced by the solvents and electrospinning voltages. High surface tension and slow evaporation of distilled water as a solvent resulted in electrospinning producing irregular structures with beads and film-like areas. Ethanol as a solvent led to smoother, more uniform fibers formation at 8 kV, but jet instability at 12 kV created fibrous structures with globular and particle-like morphologies. PVP/distilled water fibers yielded an average diameter of 1.05 ± 0.50 μm and PVP/ethanol fibers 2.46 ± 0.64 μm at 8 kV. Overall, these results indicate that solvent properties and applied voltage strongly affect fiber uniformity and diameter. Ethanol facilitates better fiber formation due to its lower surface tension and faster evaporation rate compared to water. Therefore, optimizing solvent selection and electrospinning parameters is crucial to achieve defect-free PVP fibers with controlled morphology.

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