Effect of Solution Concentration During Electrospinning Process on Morphological Features of Scalable Self-standing Polyvinyl Alcohol Nanofibrous Membranes

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ABSTRACT

Nanofibers and nanofibrous membranes composed of polymers, ceramics, carbons, and their hybrid materials, fabricated through electrospinning process, have garnered significant research interest due to their potential applications in diverse fields, including medical devices, environmental protection and remediation, and energy systems. Polyvinyl alcohol (PVA) has been extensively used as a functional component in nanofiber composites, a polymer binder for the fabrication of architectured carbon and ceramic nanofibers, and a scaffolding material for the preparation of freestanding ceramic nanofibrous membranes, via electrospinning. Consequently, investigating the effects of working parameters during electrospinning on the properties of the resulting products is essential. In this study, the influence of solution concentration on the morphological features and physical dimensions of self-standing electrospun PVA nanofibrous products collected on a rotational cylindrical drum, was examined using scanning electron microscopy (SEM), and the results were analyzed and discussed. The findings showed that increasing PVA solution concentration (6–14% w/v) led to larger fiber diameters, reduced membrane width, and increased membrane thickness. Based on the power law of polymer solutions, the diameter of the electrospun fibers increased as the PVA solution concentration was raised, following the relationship $D \sim \sqrt{C}\,\eta^{1/3}$. This research enhances the predictive capability regarding the morphology of the resulting PVA nanofiber products—particularly the fiber diameters—by identifying solution concentration as a key parameter governing the electrospinning process. This deeper understanding of how solution concentration affects the morphology of PVA-based nanofibers is expected to facilitate the efficient fabrication of nanofibrous mats utilizing PVA as a binder and/or structural component.

Keywords: Polyvinyl alcohol, Nanofibers, Electrospinning, Solution concentration, Morphology

1. INTRODUCTION

Electrospinning, a versatile method that uses electrostatic force to produce nanofibers and nanofibrous membranes of polymers, ceramics, carbons, and their hybrid materials, is a straightforward way to create ultra-fine fibers from polymer solutions or melts. It offers benefits like repeatability, control over fiber size, and low production cost. The process involves drawing a continuous polymer strand through a spinneret using a strong electric field, resulting in fibers that randomly collect on a grounded surface to form a non-woven mat. These fibers feature a high surface area relative to their mass or volume and high porosity, making them easily functionalized and suitable for various applications [1-5].

Among the diverse polymers utilized in electrospun nanofiber production, polyvinyl alcohol (PVA)—a water-soluble synthetic polymer—is commonly selected [6]. Its chemical structure is illustrated in figure 1. PVA is an atactic polymer that demonstrates a degree of crystallinity and possesses notable film-forming, emulsifying, and adhesive capabilities. Addition ally, it exhibits resistance to oils, greases, and various solvents. PVA is characterized by high tensile strength,

flexibility, biocompatibility, low tendency for protein adhesion, and low toxicity, and excellent barrier properties against oxygen and aromatic compounds [6-8]. These properties have supported its application such as medical devices, photographic films, and binder substances [9].

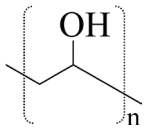

Figure 1. Chemical structure of PVA.

In the context of electrospinning, PVA is extensively used as a functional component in nanofiber composites [10-12], a raw material and binding polymer for the fabrication of architectured carbons [13-15] and ceramic nanofibers [16, 17], and a scaffolding material for the preparation of freestanding ceramic nanofibrous membranes [18]. Therefore, a systematic investigation into the influence of electrospinning parameters—comprising solution properties, processing conditions, and environmental factors—on the morphological characteristics, shape, and dimensions of the resulting nanofibers is crucial [19].

In recent years, numerous studies have explored the influence of various parameters on the morphology and diameter of electrospun PVA nanofibers [8], including solution viscosity [20], flow rate, degree of hydrolysis, applied voltage, collection distance, ionic salt addition [21], molecular weight [22], pH [23], surfactant addition [24], and collector type [25]. However, the findings have frequently been inconsistent or contradictory.

Among the electrospinning parameters, the concen tration of spinning solutions plays a critical role in determining the morphological and structural characteristics of the resulting nanofibers and nanofibrous membranes [8, 20, 26].

Supaphol and Chuangchote [8] studied the effect of the concentration of PVA solutions (which is directly related to the viscosity of the polymer solutions) on the morphology of electrospun PVA nanofibers fabricated using the electrospinning process with a stationary collector. It was found that the fiber diameters increased with increasing solution concentration and that the deposition area of the fibers on the collector decreased accordingly.

Meanwhile, Mohammad Ali Zadeh *et al.* [20] employed PVA solutions as binders for the sol–gel of cationic ions in the preparation of electrospun mullite nanofibers. Their study focused on the effect of the viscosity of PVA solutions on the morphology of the resulting nanofiber products. They observed that increasing the viscosity of the PVA solution significantly affected the morphology and led to a noticeable increase in the fiber diameters.

However, despite the conceptual explanations of how the concentration and viscosity of polymer solutions affect the morphology of the nanofiber products, no work has yet described a mathematical relationship between these parameters (e.g., polymer solution concentration or viscosity) and the morphology of the resulting electrospun PVA nanofiber products (such as fiber diameters).

Until 2025, when Munawar *et al.* [26] investigated the relationship between solution viscosity, solution concentration, and the diameter of electrospun fibers, employing a power-law relationship (to describe the dependence between the viscosity and concentration of polymer solutions) and drawing upon the findings of Schubert to explain this relationship. This research contributes to predicting the morphology of resulting nanofiber products (e.g., fiber diameters) by defining the parameters during the electrospinning process. In their work, polycaprolactone (PCL) was used as the polymer model, and a rotational collector wheel was employed to collect the fibers.

Here, in order to fabricate self-standing PVA nanofibrous membranes via the electrospinning process using a rotational cylindrical collector, the effect of the concentrations of the spinning solutions on the morphological characteristics, shape, and dimensions of the resulting nanofibrous membranes was primarily investigated. This study represents the first instance of exploring the relationship between the concentrations of the spinning solutions and the diameters of the electrospun fibers in the preparation of self-standing PVA nanofibrous membranes via the electrospinning, employing mathematical equations to describe this relationship.

2. MATERIALS AND METHODS

The solutions for electrospinning were prepared by dissolving PVA powder (Sigma-Aldrich, M_W ~ 160,000 g

mol^{-1}, degree of hydrolysis (DH) of ≥ 85.0%) with a predetermined amount into distilled water in a closed glass bottle under gentle magnetic stirring at 50 °C for 12 h, before naturally cooled to a room temperature. Afterwards, transparent spinning solutions at different concentrations (6%, 10%, and 14% w/v) were achieved. All chemicals in this work were used as received.

For the electrospinning process, the spinning solution was loaded into a medical plastic syringe with a No.21-guage-size stainless needle. To investigate the effect of solution concentration on the dimensions (width and thickness) of the obtained nanofibrous membranes, the supplied voltage was fixed at 15 kV, spinning distance (needle-tip-to-collector distance at 8 cm, rotational collecting speed of 100 rpm, and a spinning duration of 1 h. Meanwhile, the solution feeding rate was controlled at 1.0 mL h^{-1}. This value represents the flow rate at which the polymer solution was consistently delivered to the spinneret tip, ensuring adequate supply to form a stable Taylor cone and achieve uniform fiber formation, without the accumulation of solution droplets at the needle tip during electrospinning. The electrospinning was conducted under ambient conditions, with a relative humidity of 25% and at room temperature (25 °C). The scheme of electrospinning setup employed in this work is shown in figure 2.

The morphology of as-electrospun nanofibrous membranes was observed using scanning electron microscope (SEM; JEOL, JSM-IT500 INTOUCHSCOPE™) and fibers' diameters were measured using Image J. Thirty fibers were selected to evaluate the average fiber diameter ($\bar{x}$) and the corresponding standard deviation (σ) calculated using equation (1) and (2), respectively.

$$\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n} \quad (1)$$

$$\sigma = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n-1}} \quad (2)$$

where x_i and n are the fiber diameters in the data distribution and total number of fibers, respectively.

Meanwhile, the thickness of the nanofibrous membranes obtained was measured using a digital thickness gauge (Mitutoyo 547-561S, 1 μm resolution). The thickness measurement was performed at five equally spaced positions across the membrane width including the center (C), the edges (E; both left and right), and the midpoints between the center and the edges (MEC; both left and right).

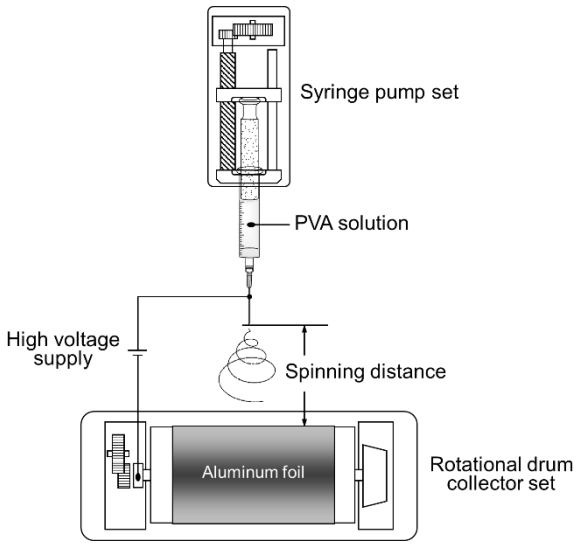

Figure 2. Electrospinning setup used in this work (Top-view).

The viscosity of PVA solutions was determined using a rotational rheometer (Anton Paar MCR302), with the temperature maintained at 25°C through a temperature control unit. Rheological measurements were conducted using a parallel plate configuration with a diameter of 50 mm and a gap of 0.6 mm. To minimize solvent evaporation during testing, a suitable solvent trap was employed. Each measurement was preceded by a pre-shear at 0.1 s^{-1} for 1 min, followed by a 1-min rest period (0 s^{-1} shear rate) to eliminate any artifacts arising from sample handling and loading.

3. RESULTS AND DISCUSSION

A comprehensive understanding of the electrospinning process requires consideration of six fundamental forces: (1) the gravitational force acting on the polymer solution; (2) the electrostatic force that drives the charged jet from the needle toward the collector within an electric field; (3) the Coulombic stretching force resulting from repulsion between like charges within the jet, which promotes jet elongation and thinning; (4) the viscoelastic force of the polymer resisting deformation; (5) the surface tension opposing the jet elongation; and (6) the drag force arising from air resistance during the jet's trajectory [8, 27, 28].

To investigate the effect of spinning solution concentration—the principal solution parameter—on the morphology, shape, and dimensions of the resulting nanofibrous membranes during the electrospinning process, all other parameters, including process parameters (applied voltage, spinning distance, collector rotational speed, and spinning duration) and ambient parameters (temperature, humidity, and pressure), were kept constant.

It is well established in electrospinning that achieving uniform ejection of the charged jet requires a spinning solution with an appropriate concentration or viscosity. At low concentrations, insufficient molecular entanglement leads to jet instability and the formation of droplets—a phenomenon known as electrospraying—rather than a continuous jet [8, 29, 30]. Conversely, excessively high concentrations hinder the continuous flow of the polymer solution from the nozzle. Therefore, a critical concentration threshold must be surpassed to ensure stable jet formation, and electrospinnability is confined to a specific concentration or viscosity range, beyond which droplet formation becomes predominant [8, 31].

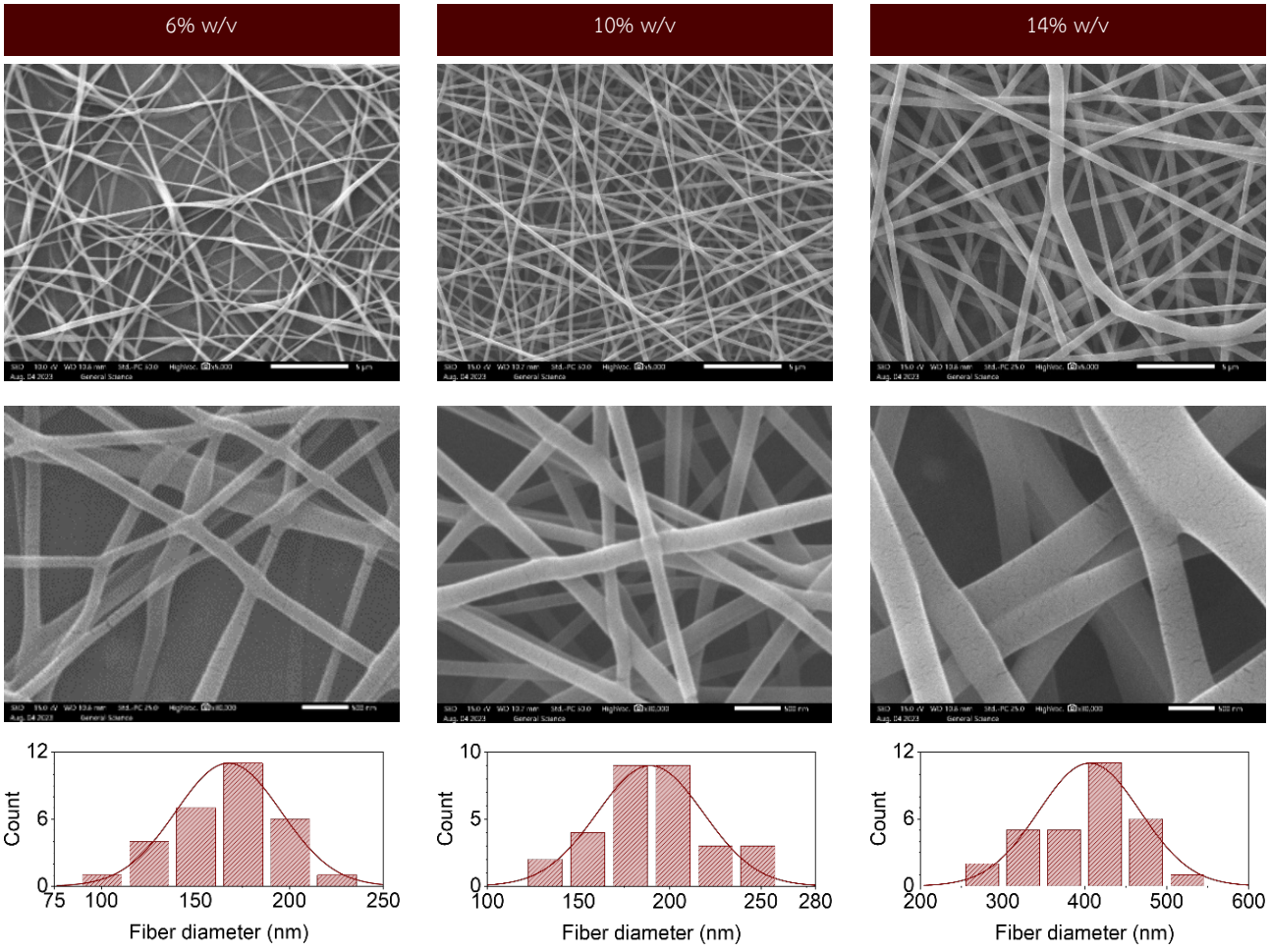

Figure 3. SEM images at low (upper raw) and high (lower raw) magnification of the PVA nanofibers fabricated from various solution concentrations. The scale bars represent 5 μm and 500 nm for the low and high magnification, respectively.

Figure 3 presents SEM images depicting the morphology of the electrospun fibers and their corresponding fiber size distributions. The findings revealed that the fibers electrospun using different concentrations were obtained with the formation of uniform fibers and without any beads along the fiber length. This outcome is attributed to the sufficiently high solution viscosity, which enhances the viscoelastic forces to a level that fully suppresses jet breakup, thereby enabling more uniform elongation of the charged jet under Coulombic stress [8]. Electrospinning of the 6% w/v PVA solution resulted in fibers with a diameter

distribution of 136.56 ± 27.94 nm. Meanwhile, fibers obtained from 10% and 14% w/v PVA solutions exhibited average diameters of 242.35 ± 29.20 nm and 440.79 ± 39.48 nm, respectively. At a constant electrospinning condition, increasing the solution concentration led to a progressive increase in fiber diameter, primarily due to increased solution viscosity. These observations are generally consistent with previously reported results [8, 21, 22, 32]. The viscosity of aqueous PVA solutions, as characterized using the rheometer, was determined to be 60, 359, and 1,427 mPa s, at concentrations of 6%, 10%, and 14% w/v, respectively. The relation between viscosity (η) and concentration (C) for the PVA solution is shown in figure 4. It seems the concentration dependence of the viscosity follows a power law (see equation (3)) [26, 33]. This phenomenon generally occurs over a high concentration range when the polymer concentration exceeds the reciprocal of the intrinsic viscosity ($C > [\eta]^{-1}$, where $[\eta]$ represents the intrinsic viscosity of the polymer solution) [33]. In this work the exponent α was found to have a value of approximately 4. A previous report discussed that exponent α values ranging from 3 to 5.4 are associated with the facilitation of complete fiber formation during electrospinning [34].

$$\eta \sim C^{\alpha} \quad (3)$$

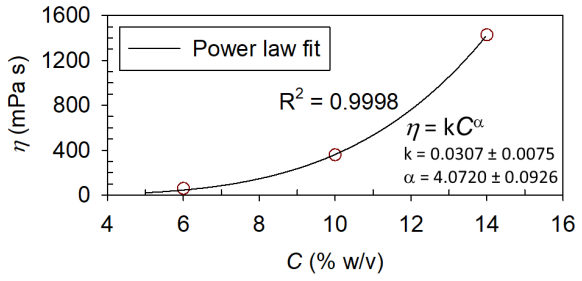

Figure 4. The relationship between viscosity (η) and concentration (C) in PVA aqueous solutions exhibits a power-law behavior.

On the other hand, in electrospinning, three characteristic regimes are typically observed, each corresponding to a specific range of polymer concentrations [34, 35]. The first regime arises in the semi-dilute region, where the polymer concentration exceeds the critical overlap concentration (C^*) but remains below the critical entanglement concentration (C_e). In this range, polymer chains overlap without sufficient entanglement, leading to the formation of beads or particles rather than continuous fibers. As the concentration increases beyond C_e, chain entanglements begin to develop, resulting in a beads-on-string morphology characterized by beads connected by short fiber segments [36]. When the concentration reaches translation point (approximately 2 times C_e for PVA aqueous system with PVA M_w of 145–180 kg mol^{-1} and DH level of 88%) [35], the level of entanglement becomes sufficient to suppress instabilities in the electrospinning jet, enabling the formation of smooth, uniform (bead-free) fibers—this state is referred to as the heavily entangled or concentrated regime [34, 35, 37, 38]. These regimes are broadly applicable across various polymers, though the specific concentration thresholds vary depending on the polymer system [35].

The C_e is the concentration at which polymer chains begin to overlap and form a transient network due to entanglements, significantly increasing the solution's viscosity and elasticity, which is primarily determined by the molecular weight of the polymer and the strength of intermolecular interactions, particularly hydrogen bonding. According to a detailed rheological study, the C_e was calculated using equation (4) [36, 39].

$$C_e = \frac{\rho M_e}{M_w} \quad (4)$$

where ρ is the density of polymers. M_e is the entanglement molecular weight and M_w is the weight-average molecular weight of polymers.

In this work, for PVA aqueous solutions, with a polymer M_w of 160,000 g mol^{-1}, a polymer density of 1.3 g cm^{-3}, and M_e of 3,750 g mol^{-1} [39, 40], the C_e is approximately 2.98% w/v. The PVA aqueous solutions were investigated within a concentration range of 6–14% w/v, which exceeds the entanglement concentration about 2 times C_e. This indicates that the solutions fall within the entangled to concentrated (heavily entangled) regime of polymer concentration. In this regime, sufficient molecular entanglement promotes the formation of smooth, continuous, uniform (bead-free) fibers, as confirmed by the SEM images of the electrospun fibers at each concentration. This is also consistent with the previous report by Ewaldz E, *et al.* [35]

The identification of these regimes can be achieved through shear rheological analysis in conjunction with microscopic characterization of the electrospun fibers, particularly through variations in the slope of the relative viscosity or specific viscosity (η_{sp}) versus concentration curve [35]. In this study, shear viscosity (i.e., η_{sp}) was evaluated across a range of concentrations, as presented in figure 5. The specific viscosity (η_{sp}) is determined using equation (5) [37].

$$\eta_{sp} = \frac{\eta - \eta_0}{\eta_0} \quad (5)$$

where η_0 is solvent viscosity. In this work, water was used as the solvent; as a result, the η_0 is 1 mPa s.

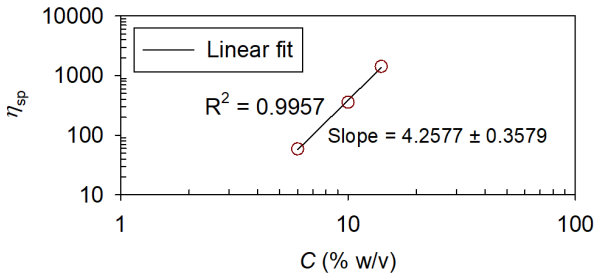

Figure 5. Relative viscosity of PVA aqueous solution at various concentration.

In this study, when the concentration of PVA aqueous solutions exceeded approximately 2 times the critical entanglement concentration, placing the system within the concentrated regime, the slope of the specific viscosity (η_{sp}) versus concentration plot was found to be approximately 4.26. This result is consistent with a previous report, which observed a slope of 4.3 in the concentration range associated with complete fiber formation via electrospinning [35]. The slightly different slope value is reasonably attributed to the smally different molecular weight of the PVA used herein.

Empirical scaling laws have shown fiber diameter scales with viscosity, as a power law with exponents around 1/3 to 1, linking concentration and viscosity directly to fiber size [26, 38, 41]. However, the relationship between fiber diameter and viscosity is frequently well described by a power-law dependence with an exponent of approximately 1/3 [26, 41].

Figure 6 illustrates the correlation between fiber diameter (*D*), viscosity (η), and concentration (*C*). As recommended by Schubert and supported by previous studies [33, 42], analyzing fiber diameter solely as a function of viscosity is insufficient without explicitly accounting for concentration. When electrospinning occurs without jet splitting, the interdependence of *D*, η, and *C* can be described by equation (6) [26]. This relationship was experimentally validated by plotting $\frac{D}{\sqrt{C}}$ against $\eta^{1/3}$. The resulting linear fit fell within experimental error and confirmed that viscosity is proportional to the nanofiber diameter raised to the power of 1/3.

$$D \sim \sqrt{C}\,\eta^{1/3} \quad (6)$$

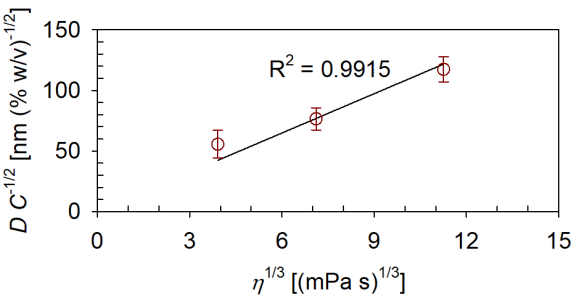

Figure 6. Ratios of fiber diameter to the square root of concentrations as a function of $\eta^{1/3}$ for PVA fibers electrospun at different concentrations (6%, 10%, and 14% w/v).

The results clearly demonstrate that polymer solution concentration is a critical parameter influencing fiber diameter. Increased concentrations lead to higher viscosity, which hinders the elongation of the polymer jet under the electric field, thereby producing thicker fibers. This observation aligns with both theoretical predictions and the experimental findings of the present study, which collectively indicate a positive correlation between fiber diameter and viscosity. In contrast, lower concentrations decrease viscosity, facilitating greater jet elongation and resulting in finer fibers [26, 43]. This study utilized equation (6), originally formulated and empirically validated in Schubert's work [33, 42], as a foundational model for analyzing the electrospinning process. The equation serves as a crucial framework for validating and refining experimental results under varying conditions. By incorporating this established model, the current research not only confirms its relevance in new experimental contexts but also aims to deepen the

understanding of the electrospinning mechanism for the preparation of PVA nanofibers with controllable morphologies. This integration enhances theoretical comprehension, provides novel empirical evidence, and contributes to advancing the precision and capabilities of electrospinning techniques.

Figure 7(Top) and (Middle) show selected optical images illustrating the deposition area of the electrospun fiber mats and their widths from PVA solutions at various concentrations under an applied electrical potential of 15 kV, spinning distance of 8 cm, solution feed rate of 1 mL h^{-1} and rotational collecting speed of 100 rpm, for 1 h, which was performed in air relative humidity of 22% and atmospheric pressure, and at room temperature (25 °C). Under the fixed electrospinning conditions, the widths of the spun fiber mats consistently decreased with increasing solution concentration. This may be attributed to the enhanced viscoelastic force at higher concentrations, which reduced the likelihood of jet thinning and caused the onset of bending instability to occur nearer to the collector [44, 45]. This result is consistent with the increased membrane thickness observed at higher solution concentrations (see figure 7(Bottom).

4. CONCLUSIONS

This study investigated the effect of spinning solution concentration on the morphology and dimensions (width and thickness) of PVA nanofibrous membranes. The results demonstrated that the average diameter of the electrospun PVA nanofibers increased with rising solution concentrations (6–14% w/v), while other parameters were held constant. Additionally, higher solution concentrations led to a reduction in membrane width and a consistent increase in membrane thickness. This research strengthens the predictive understanding of the morphology of the resulting PVA nanofiber products, particularly the fiber diameters, by elucidating the critical role of solution concentration as a key parameter in the electrospinning process. These findings provide a deeper understanding of how solution parameters influence the structural features of PVA-based nanofibers and nanofibrous membranes, thereby supporting the effective development of nanofibrous materials using PVA as a binder and/or structural component. The results obtained in this study provide valuable insights for the fabrication of hybrid ceramic nanofibrous membranes for environmental applications, particularly flue gas purification and water treatment, which is currently under investigation in our laboratory.

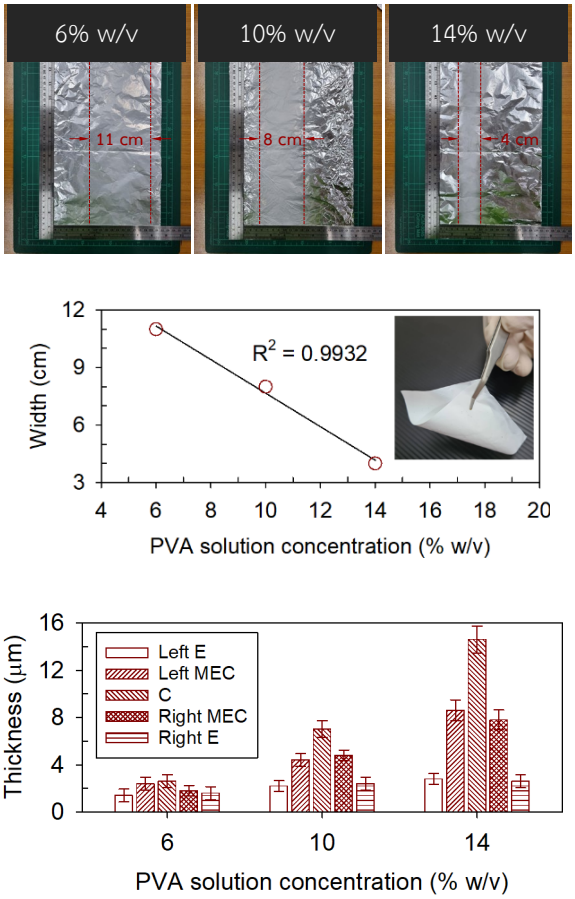

Figure 7. Digital images (Top) of the PVA nanofibrous membranes and their widths (Middle) and thickness (Bottom) electrospun from various PVA concentrations. The inset in the middle image displays the self-standing PVA nanofibrous membranes electrospun from a spinning solution at a concentration of 10% w/v, after being peeled off from the aluminum foil.

5. ACKNOWLEDGMENTS

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