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Original Research

Morphological and Wettability Evaluation of Electrospun Polivinyl Alcohol (PVA) Nanofiber Membrane for Wound Dressing Application

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ABSTRACT

Many researches have focused on producing electrospun nanofiber membranes (ENMs) aiming to speed up physiological events of wound healing. However, it is still a challenge to fabricate functional ENMs using electrospinning technique, taken into account various factors that may affect the fibers diameter such as polymer solution properties, electrospinning parameters and ambient conditions. This study aimed to evaluate the combined electrospinning process parameters and characterize the morphology and wettability of the electrospun polyvinyl alcohol (PVA) nanofiber membranes produced. Process parameters that were optimized include applied voltage, flow rate, and distance to collector. The fabrication of PVA nanofiber membranes was done at 21 kV, 2 mL/h and 10 cm after optimization. Surface morphology and fiber diameter were analyzed using a scanning electron microscope (SEM). The water contact angle was used to analyze wettability. Electrospun PVA nanofiber membranes showed continuous random fiber, no bead defect, hydrophilic surface and average fiber diameters between 265 ± 86 nm and 281 ± 90 nm. Smaller fiber diameter between 200-700 nm can enhance cell migration. It is suggested from this study that the optimized parameters have successfully produced electrospun PVA nanofiber membranes with suitable fiber diameter for cell migration and can direct cell response, thus can be applied as a wound dressing material.

INTRODUCTION

Nanofibers were introduced as scaffolds for skin tissue engineering and as wound dressings for external skin repair due to their morphology and structure that mimic the native extracellular matrix (ECM) (Ottosson et al., 2017; Azimi et al., 2020). Electrospinning is preferred to produce nanofibers as this technique is simple, cost-effective, ease of use, and allow mixtures of polymers and other materials be used to form nano- and micro-sized fiber diameters (Parin et al., 2021; Rezk et al., 2019; Azimi et al., 2020; Yan et al., 2022). The electrospun nanofiber membranes (ENMs) have been utilized in many biomedical field such as implants, dressings, and medical devices due to the strong plasticity, flexible structure and large surface area-volume ratio, which are important for cell adhesion, proliferation and differentiation (Yan et al., 2022). Studies have shown that ENMs were able to guide fibroblast migration for wound healing (Ottosson et al., 2017) and can carry drugs for drug delivery (Li et al., 2018; Rezk et al., 2019).

Electrospinning process parameters (applied voltage, flow rate and distance to the collector), solution properties (polymer concentration, viscosity, conductivity, surface tension) and ambient conditions (room temperature and humidity) are the factors that influence the fibers morphology and diameter (Can-Herrera et al., 2021). It is noteworthy that applied voltage is one

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of the most important parameters that must be controlled to produce satisfactory quality of fibers (Nurfaizey and Abdul Munajat, 2020; Can-Herrera et al., 2021). At low applied voltages, droplets or bead formation will be produced instead of fibers due to insufficient Coulombic forces to overcome the superficial tension of the polymer solution (Can-Herrera et al., 2021). Earlier studies have demonstrated the effects of electrospinning process parameter alone/ in combination on the fiber diameter and morphology. Thin, crinkled polyacrylonitrile (PAN) fibers were obtained at applied voltage of 5 kV turned to thicker diameter fibers with uniform appearance at 15 kV and beaded fibers with reduced diameter were seen at 20 kV (Nurfaizey and Abdul Munajat, 2020). Higher applied voltage at 20 kV has resulted in a larger PAN fiber diameter and more prominent bead formation, most probably due to more fluid ejection and increased electrostatic repulsive forces on the liquid jet, respectively (Bakar et al., 2019).

Polyvinyl alcohol (PVA) is a synthetic, water-soluble polymer that has attained a remarkable attention for use in biomedical field due to its biocompatibility, hydrophilicity, and approval for use by Food and Drug Administration (FDA) (Asiri et al., 2021). In this study, electrospinning process parameters were optimized to produce electrospun PVA nanofiber membranes that meet the wound dressing application.

MATERIALS AND METHOD

Materials

Polyvinyl alcohol (PVA) (average Mw = 85-124 kDa, degree of hydrolysis = 87-89%) was purchased from Sigma-Aldrich.

Solution Preparation and Evaluation of Combined Electrospinning Process Parameters

For the optimization purposes, two combinations of applied voltage, flow rate and distance tip to collector were assessed. Two 10% wt of PVA solutions were prepared in distilled water (Parin et al., 2021). The nanofiber membrane samples, NF1 and NF2 were prepared by dissolving 0.5 g PVA in 5 mL distilled water and stirred for 1 h at 90 °C until clear solutions were obtained and left cooled before electrospinning. 3 mL of PVA solution was supplied to the syringe pump that was attached to the 23G electrospinning needle (NE-300, New Era Pump Systems Inc., USA). Table 1 shows the combination of electrospinning process parameters that were performed for samples NF1 and NF2. The electrospinning was carried out at room temperature for 10-15 minutes and the produced nanofiber membrane samples were collected on the flat plate collector covered with aluminium sheet. These nanofiber membrane samples were further dried at room temperature for 24 h.

Surface Morphology of Electrospun PVA Nanofiber Membranes

Surface morphology of both NF1 and NF2 samples was observed without coating using a Hitachi Table Top Scanning Electron Microscope (TM3000, USA). Each sample was cut into a small size. The fiber diameter distribution was analyzed using ImageJ software (NIH, USA) and 20 measurements were taken for each sample (n = 20).

Solution Preparation and Fabrication of Optimized Electrospun PVA Nanofiber Membranes

After SEM analysis were conducted on NF1 and NF2 samples, three replicates of PVA solution were prepared as mentioned earlier. 3 mL of PVA solution was supplied to the syringe pump that was attached to the 23G electrospinning needle (NE-300, New Era Pump Systems Inc., USA). The applied voltage was fixed at 21 kV with flow rate of 2 mL/h and the nozzle-collector distance was fixed at 10 cm. Three optimized electrospun PVA nanofiber membranes were produced and labeled as PVA1, PVA2 and PVA3.

Characterization of Optimized Electrospun PVA Nanofiber Membranes

The PVA1, PVA2 and PVA3 were analyzed for their surface morphology and wettability. Surface morphology was carried out using a scanning electron microscope (JSM-IT300LV, JEOL, USA). The samples were gold sputter-coated before analysis. Nanofibers diameter were measured using ImageJ software (NIH, USA) and 50 measurements were taken randomly for each sample. Wettability was determined using a contact angle analyzer (AST VCA Optima, USA). Water droplet size was set at 2.0 µL and five images from different areas of each nanofiber membrane sample were taken to get the contact angle values (mean ± standard deviation).

RESULT AND DISCUSSION

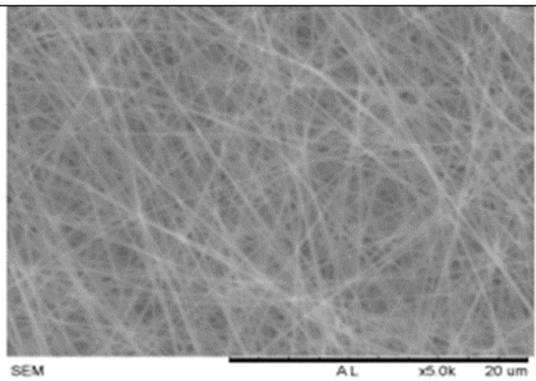
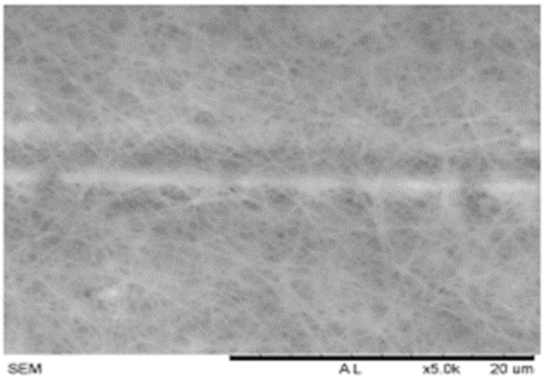
Solution Preparation and Surface Morphology of Electrospun PVA Nanofiber Membranes

In this study, 10% wt PVA solution was the chosen concentration for the fabrication of PVA electrospun nanofiber membrane. In their study, Bakar et al. (2019) revealed that increase in concentration of PAN solution resulted in a larger fiber diameter (8% wt<10% wt<12% wt) with no bead formation observed for 10% wt and 12% wt. The reason for this is most likely due to the higher viscosity resistance of more concentrated polymer solution that produces larger fibers and regulates the flow, controlling the bead formation (Bakar et al., 2019).

Table 1 Electrospinning process parameters.

Sample	PVA (g)	Distilled water (mL)	Applied voltage (kV)	Flow rate (mL/h)	Distance to collector (cm)
NF1	0.5	5	21	1.5	15
NF2	0.5	5	25	2	10

Table 2 Fiber diameter and surface appearance (without coating) of PVA electrospun nanofiber membranes prepared at two combinations of electrospinning process parameters.

Sample	Process parameters	Fiber diameter (nm) mean ± SD	SEM image
NF1	21 kV 1.5 mL/h 15 cm	173 ± 9	
NF2	25 kV 2 mL/h 10 cm	171 ± 7	

The NF1 and NF2 nanofiber membranes were fabricated by varying three combined electrospinning process parameters; i.e. applied voltage, flow rate, and distance to the collector to identify which combination gives better nanofiber structural formation and fiber diameter. Only a slight difference in fiber diameter was observed between the two nanofiber membrane samples (Table 2), indicating that both combined parameters was suitable to produce PVA nanofiber membrane with small fiber diameter of less than 200 nm. It is important to note that smaller fiber diameter (<140 nm) may inhibit cell infiltration and therefore limits cell proliferation (Can-Herrera et al., 2021).

It can be seen from the SEM images that both NF1 and NF2 nanofiber membranes have homogenous appearance and produced bead-free nanofibers (Table 2). From these results, the combined parameters for NF2 nanofiber formation were chosen as the optimized process parameters; 25kV applied voltage, 2 mL/h flow rate and 10 cm distance (Table 2). However, sparks were observed at 25kV, thus the applied voltage was set to 21 kV and its effect on morphology and wettability of nanofibers was evaluated.

Surface Morphology of Optimized Electrospun PVA Nanofiber Membranes

Figure 1 presents the SEM micrographs of three replicates of electrospun PVA nanofiber membranes prepared according to optimized process parameters. It can be observed that the optimized process parameters were able to produce continuous, random, smooth electrospun PVA nanofibers with increased average fiber diameter (Table 3). This finding was in accordance to the study by Abd El-aziz et al. (2017) who reported that at constant flow rate and distance to collector, the applied voltages of 15 kV and 30 kV resulted in average diameter of PVA nanofibers of 231.5 ± 80 nm and 172.5 ± 45 nm, respectively. In this study, the applied voltage of 21 kV has resulted in larger fiber diameter above 250 nm (Table 3) as compared to 25 kV which produced small fiber diameter of less than 200 nm (Table 2). However, other studies revealed that higher applied voltages generally produce fibers with larger diameter, as observed for electrospun PAN (Bakar et al., 2019; Nurfaizey and Abdul Munajat, 2020) and PCL nanofibers (Can-Herrera et al., 2021). This could indicate that polymer type, molecular weight, concentration, surface tension and viscosity influence the diameter of the fibers produced (Islam et al., 2019).

Higher voltages at 20 kV and 25 kV were reported to produce points of contact between polycaprolactone (PCL) fibers and branched structures when elecrospun at flow rate of 1.5 mL/h and 12 cm distance of collector (Can-Herrera et al., 2021). Nonetheless, these features were not observed for the optimized electrospun PVA nanofiber membranes produced in this study, indicating that the combined applied voltage, flow

Table 3 Fiber diameter and wettability of electrospun PVA nanofiber membranes.

Sample	Fiber diameter (nm) mean ± SD (n = 50)	Contact angle (°) mean ± SD (n = 5)
PVA1	265 ± 86	39 ± 9
PVA2	281 ± 90	37 ± 4
PVA3	274 ± 88	42 ± 7

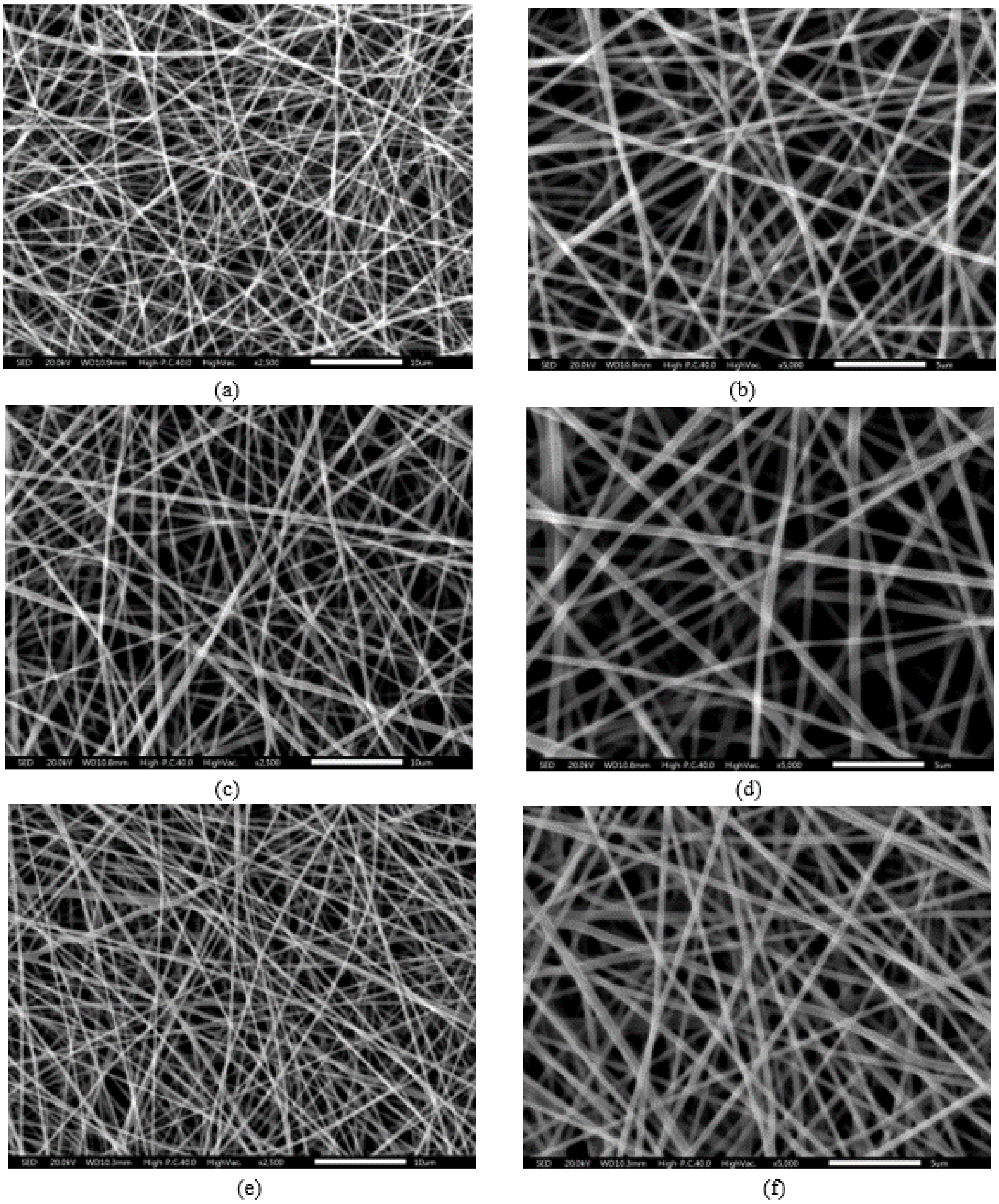


Fig 1 SEM micrographs of electrospun PVA nanofiber membranes at 2,500x and 5,000x magnifications for samples (a,b) PVA1, (c,d) PVA2 and (e,f) PVA3.

rate and distance was suitable to produce good quality of electrospun PVA nanofiber membranes. The flow rate and distance must also be controlled to produce bead-free uniform fibers. Although it is recommended to have slow flow rate to give enough time for the polymer solution to polarize (Islam et al., 2019), it can be seen from this study that flow rate of 2 mL/h was able to produce uniform, homogenous fibers with no beads present (Figure 1). Beaded fibers could be produced when the distance between the needle tip and collector are short (Islam et al., 2019).

The average fiber diameter of optimized electrospun PVA nanofiber membranes (265-281 nm) was suitable for migration of certain types of cells. The nanofibers with diameter between 200-700 nm allow some cells to migrate faster than microfibers of diameter between 1.1-5.7 μm (Jenkins and Little, 2019). Kaniuk et al. (2020) demonstrated that fibroblasts spread better on fibers as compared to film, taking into account the larger surface area and more contact points for cells provided by the fibrous scaffolds. However, the cell migration analysis was not conducted in this study and thus contributes as the limitation of this study.

Wettability of Optimized Electrospun PVA Nanofiber Membranes

Figure 2 presents the water contact angle images of a single replicate of electrospun PVA nanofiber membranes. The results shown in Table 3 depicts that PVA1, PVA2 and PVA3 samples had low contact angle values which represent hydrophilic surface, good wettability and good adhesiveness. This result is supported by the earlier findings that mentioned water contact angle of PVA nanofibers are less than 90° due to the hydrophilic

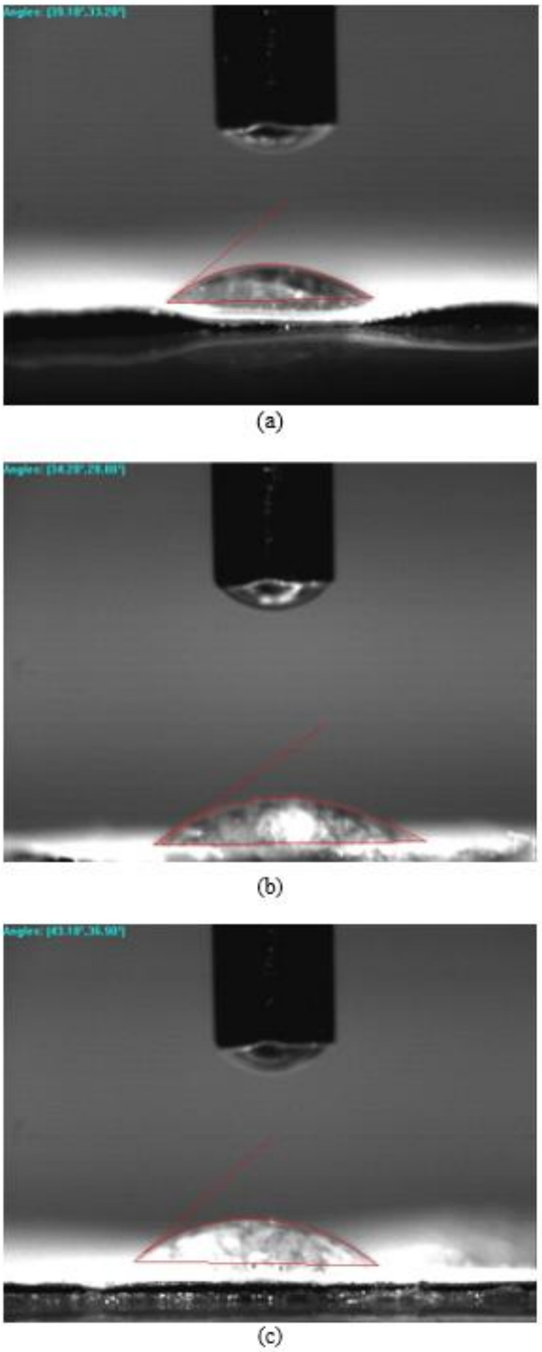


Fig 2 Water contact angles of electrospun PVA nanofiber membranes: (a) PVA1, (b) PVA2 and (c) PVA3.

property of the PVA (Hu et al., 2021). Wettability property is an important indication of the potential cell attachment, migration and proliferation on the material surfaces (Asiri et al., 2021). Therefore, the optimized electrospun PVA nanofiber membranes are suitable to be used as a wound dressing material as fibroblast cells generally spread well on hydrophilic surface (Kaniuk et al., 2020). In comparison to an early study, PVA nanofibers prepared at a concentration of 8% wt, applied voltage of 20kV, flow rate of 0.5 mL/h and 15 cm distance had thinner fiber diameter of 230 ± 59 nm and contact angle value of 83 ± 0.9°, close to hydrophobic property (Asiri et al., 2021). It is suggested that hydrophilic/hydrophobic characters of fibers can be influenced by the polymer concentration and electrospinning process parameters.

CONCLUSION

It can be concluded from this study that electrospun PVA nanofiber membranes morphology and wettability are influenced by the combined electrospinning parameters and the change in a single parameter such as applied voltage affects the fiber diameter. The electrospun PVA nanofiber membranes were suitable for use as wound dressing material due to their hydrophilic surface and desirable fiber diameter that may support cell migration. It is recommended that the future work covers the in vitro wound healing assay to assess the cell migration involving these electrospun PVA nanofiber membranes.

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