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Abstract. Nanofibers have obtained considerable interest for use in various applications. Polyvinyl alcohol (PVA) has been used to achieve many benefits for diverse pharmaceutical and biomedical applications. We investigated in this study the effects of applied voltage, needle diameter, and flow rate on morphologies of PVA nanofibers. A constant volume of the feeding solutions delivered to the needle at a flow rate of 1and 2 mL/h with high potentials of voltage was applied as they exit the needle. After that, the electrospun fibers collected on the ground connected aluminum foil. The electrical conductivity measurements of feeding solutions performed at room temperature. Characterization of the PVA nanofibers conducted using scanning electron microscopy (SEM) and attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR). The obtained nanofibers SEM images show beads when using the flow rate at 1 mL/h, whereas increasing the voltage and the flow rate improved the morphology of the nanofibers to uniform without beads. The FTIR results show that O-H and C-O bands are the main attributing to the chemical functionality of PVA nanofibers. As a conclusion, the high voltage and flow rate considered as the most critical parameters that impacted on PVA nanofibers morphology.

1. Introduction

In recent years, focus on nanofibers research has increased and nanofibers have obtained a considerable interest in several applications such as energy storage, healthcare, biotechnology, environmental engineering, defence and security [1-2-3]. Nanofibers could be synthesized from natural polymers (e.g., chitosan, fibronectin, gelatin, collagen, silk, ethyl cellulose) in addition to synthetic polymers (e.g., polylactic acid (PLA), polyglycolic acid (PGA), polylactic-*co*-glycolic acid (PLGA), tyrosine-derived polycarbonates, poly ε-caprolactone (PCL), polyurethane (PU), polyvinyl pyrrolidone (PVP), polyvinyl alcohol (PVA), and their various combinations) [2].

Polyvinyl alcohol is categorized as a polyhydroxy polymer which has been studied intensively due to its excellent film forming and physical properties, with high processability, biocompatibility, hydrophilicity and excellent chemical resistance [4-5-6]. This polymer has been utilized in several areas, such as food chemistry, pharmaceuticals, medicine, and biotechnology [7]. Particularly PVA

has high usefulness in diverse pharmaceutical and biomedical applications [8] where it has the Food and Drug Administration (FDA) approval [7-8]. Polyvinyl alcohol has also been used as PVA hydrogels to fabricate contact lenses, wound dressings, coatings for sutures and catheters. It is also prominent to be modified as a water-soluble and biodegradable carrier that beneficial in drug delivery systems [7-9].

Electrospinning is an economical and convenient way to industrialize nanofibers [10]. In electrospinning, a high electrical potential is applied to a polymer solution [10] where fibers are derived by usually charging the fluid versus the ground at a short distance, resulting in charging the injection into the liquid from an electrode [11] The infected charge mark depends on the polarity of electrode; that is, the negative pole produces a negatively charged fluid [11]. The charged liquid is referred to the ground pole from an opposing polarity, forming a so-called Taylor cone at the opening of nozzle where a polymer solution is extracted by an electric field to form a liquid jet [11]. When a polymer jet solvent evaporates, the jet hardens, and polymer fibers are formed [11].

Electrospun membranes are highly potent in tissue regeneration since they can closely mimic the structure of extracellular matrix [12-13]. Therefore, in this study, PVA nanofibers were electrospun at high voltage with two numerals of flow rate and two sizes of needle diameter. The changes in chemical composition and morphology were observed on different parameters of PVA nanofibers through an attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR) and scanning electron microscopy (SEM) analyses.

2. Materials and Methods

2.1. Sample Preparation

A PVA solution was prepared by dissolving PVA (fully hydrolyzed, MW 30000, Japan) in deionized water at a concentration of 25%. The solution stirred at 80°C for 2 h to obtain a homogeneous solution. Then, the PVA solution electrospun using an electrospinning instrument (NaBond Technologies Co., Limited, Shenzhen, China) which consisted of the syringe needle, ground electrode, aluminum sheet and a high voltage supply.

The needle connected to the high voltage supply, which generates a positive DC voltage and current, up to 35 kV. The electrospinning process then conducted at room temperature with a fixed needle-electrode distance at 12 cm. The positive voltage applied to the polymer solution varied at 15 and 16 kV while the volumetric flow rate varied at 1 and 2 mL/h. The effect of needle diameter was only investigated for the sizes of 18G and 21G.

2.2. Morphology Analysis

The morphology of the PVA nanofibers was investigated by SEM (JEOL, iT300LV, Japan) at an accelerating voltage of 10 kV. Before the SEM observation, the nanostructured membranes of PVA were dried under vacuum (0.1 mbar) and coated with a gold thin film using a gold sputter coater (Q150R, Quorum Technologies Ltd, England). The SEM images were captured at 3000x magnification to visualize the formation of mat nanofibers, possible beads and electrospray fibers.

2.3. Chemical Functionality Analysis

The chemical functionalities of the PVA nanostructured membranes analyzed by ATR-FTIR (L1600301, PerkinElmer, UK) between wave numbers of 500 and 4000 cm^{-1}. The scanning performed at 4 cm^{-1} resolution. The acquired ATR-FTIR spectra were then annotated and analyzed using Originlab software (Origin Pro 8, OriginLab Corporation, USA).

3. Results and Discussion

Electrospinning has been proven to be a potential technique for fabricating polymeric fibers with diameters ranging between micrometers and nanometers due to the ease of use, cost-effectiveness and adaptability [10-14]. In this study, similar parameters of PVA nanofibers were used according to Uyar *et al.* [15] which reported that the PVA (25%, w/w) could be electrospun into finest uniform nanofibers.

Two flow rates (1 mL/h and 2 mL/h) with two sizes of needle diameter (18G and 21G) and two different voltages (15 kV and 16 kV) were applied to determine the best morphology (Figure 1 and Figure 2). Increasing the flow rate to 2 mL/h has displayed smooth surface nanofibers without bead formation compared with the electrospun at 1 mL/h. The results were similar for both sizes of needle diameter and voltage due to the fact that difference both needle diameter and voltage might and might not affected on fibers diameter [11].

The voltage then tailored at 15kV and 16 kV, independently, to remove the tip needle droplets and to acquire homogenous mat nanofibers [11]. Increasing the voltage with the flow rate of 2 mL/h has provided uniform nanofibers without beads formation while some beads appeared on the nanofibers which have been electrospun at 1 mL/h flow rate. The high voltage affects the continuous electric current that was a quasi-stable point where the jet flow rate was equal to feed rate according to Fallahi *et al.* [16].

The nanofibers were homogenously electrospun without beads by using the 21G needle diameter compared to 18G needle diameter, specifically observed on the SEM images at 1mL/h flow rate while the SEM images of 2 mL/h did not show a significant difference when various sizes of needle diameter used at different voltages.

First, the applied voltage was chosen 15 kV and 16 kV with two sizes of needle diameter (18G,21G), and used two flow rates (1mL/h, and 2mL/h) to determine the best parameter of PVA nanofiber. Figures 1, 2 (b) and (d) the results display that the nanofiber of PVA obtained smooth surface compare with Figure 1, 2 (a) and (c) which PVA nanofibers include few beads. Thus, it may be concluded that increasing the flow rate to 2mL/h obtained smooth nanofibers with both size of needle diameter and different voltage due to high voltage could effect on a continuous electric current that was the quasi-stable point where the jet flow rate was equal to the feed rate according to [16].

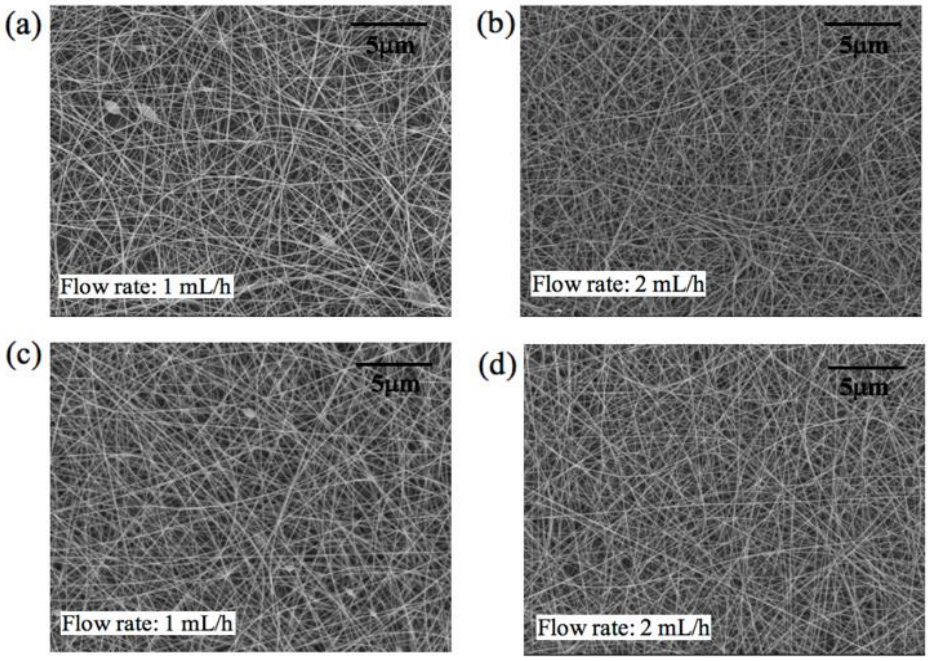

Figure 1. SEM Morphologies of electrospun PVA nanofibers with different flow rates (1 and 2 mL/h), 15 kV and different sizes of needle diameter: (a) 18G, (b) 18G, (c) 21G, (d) 21G.

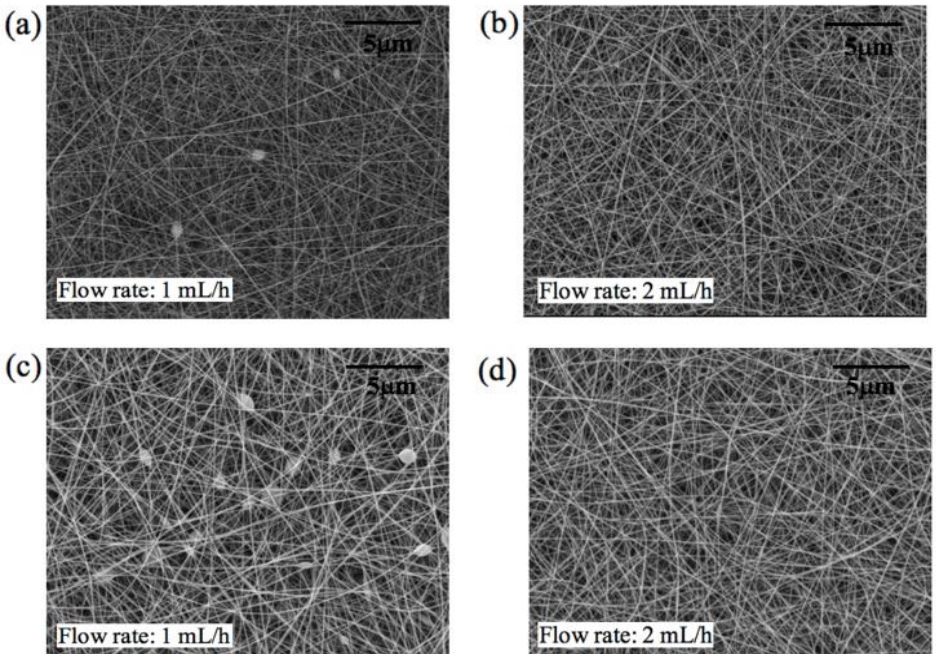

Figure 2. SEM Morphologies of electrospun PVA nanofibers with different flow rates (1 and 2 mL/h), 16 kV and different sizes of needle diameter: (a) 18G, (b) 18G, (c) 21G, (d) 21G.

The ATR-FTIR analysis of nanofibers electrospun from blended solutions was carried out to discover any peak change that referenced to chemical interactions [17]. The ATR-FTIR spectra in Figure 3 shows the functional groups of PVA nanofibers which have been electrospun at 2mL/h flow rate with different voltages and needle diameters. All nanofibers were composed of similar functional groups of PVA. However, the 21G needle has produced greater peak intensity for both 15 and 16 kV compared to the 18G due to better homogeneity of nanofibers distribution without bead disturbance (Figure 2). The 21G needle has projected greater full coverage of mat nanofibers to be detected by the ATR-FTIR analysis. In all nanofibers, the functional groups of PVA were presented at 1000-1320 cm^{-1} caused by the bond of C=O stretching [18]. While the hydroxyl group (O-H bond) of PVA were recorded at 3250-3400 cm^{-1} [19] and 910-950 cm^{-1}. Besides, peaks of C-H-C-H and C=O stretch were observed at 610-700 cm^{-1} and 1665-1710 cm^{-1} [20], respectively. Another variable peaks of C=H bond were also noticed at (2850-3000 cm^{-1}) [21] and 1450-1470 cm^{-1}[20].

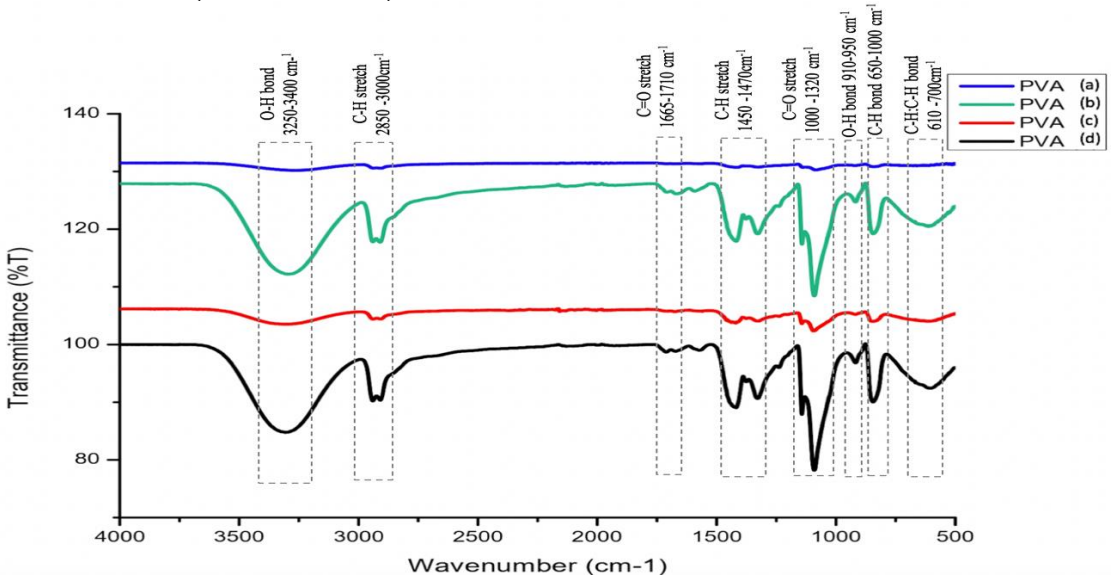

Figure 3. ATR-FTIR spectra of PVA nanofibers at flow rate of 2mL/h, voltage and needle diameter of (a) 15 kV and 18G, (b) 15 kV and 21G, (c) 16 kV and 18G, (d) 16 kV and 21G.

4. Conclusion

Electrospinning process and the factors affecting the properties of electrospun nanofibers have been widely studied for different fields in recent time. An applied voltage, flow rate and needle diameter are some of the affecting parameters during electrospinning. In this study, the formation of nanofibers was improved with the increasing of electrospun voltage and flow rate. However, there was no clear effect of needle diameter on the morphology of PVA nanofibers, in spite of its chemical functionalities. Therefore, tailoring the parameters of electrospinning in necessary to achieve desirable properties of nanofibers for a specific application.

Acknowledgments

The author acknowledges the support from ministry of higher education (MOHE) KPT grant transdisciplinary research grant (TRGS) R. J130000.7845.4L843, UTM university research grant Tier 1 Q. J130000.2545.16H98, the members of Biomedical Materials research group (B-MEMS), the Saudi Arabian Cultural Mission (SACM) and Advanced Diagnostic and Progressive Human Care Research Group.

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