Electrospinning and characterization nanofibers and nano particle of Polyvinylpyrrolidone

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Abstract. Electrospinning method used to fabricate fibers both micrometers and nanometers scale. Nanofibers of Polyvinylpyrrolidone (PVP) have been developed using an electrospinning method. It is PVP acting as matrix and ethanol analyst as a solvent. We used the same concentrate of PVP I namely 10% (w/v) for three difference molecule-weight materials namely PVP 1.300.000 kg/mol (PVP-1), PVP 360.000 kg/mol (PVP-2) and PVP 40.000 kg/mol (PVP-3). The spinning process of the three molecule-weight PVP could be produced with various parameters, 0.3 flowrate, 10 kV voltage, and 12 cm needle length. Observation using a microscope shows the morphology of each solution (PVP-1), (PVP-2), (PVP-3) will generate large fibers, medium fiber, and nanofibers. The diameter of each fiber is 2620, 1760 and 1380. From the diameter analysis, we conclude that a smaller molecule-weight of PVP will obtain smaller fibers. The FTIR results give an overview of the interactions between PVP molecules that are shown against the PVP peaks. Nanofibers and nanoparticles can be applied in medicine, high-performance air filters, military and medical protective textiles, sensors, advanced composites, photo vault cells, and separation membranes. This study will show the difference form of PVP with the same concentrate for three difference molecule-weight.

1. Introduction
Electrospinning is a simple and versatile process by which nanoscale fibers can be produced using an electrostatically driven jet of a polymer solution or polymer melt [1–3]. It has austerity and prompt procedure allow production of polymeric fibers with diameter ranging from nanometer to micrometer scale [4,5]. This technique involves (1) Coulomb forces resulted from the applied electrical charge and (2) expansion of the polymer solution. Upon exposure to a very high electrical potential or voltage difference, the charged polymer is attracted to the collector and forms fiber strands [4]. The morphology, size, and surface of the fibers can be customized by adjusting. processing parameters such as molecular weight, polymer concentration, electric voltage, needle-collector spacing, needle shape, polymer solution flow rate, and collector geometry; or by adjusting solution parameters such as conductivity, viscosity, and surface tension [6,7]. The structure of the nanofiber can be tailored to fulfill the desired characteristics, depending on the application. Electrospun nanofibers show great promise for developing many types of novel drug delivery systems (DDSs) owing to their high surface area, high porosity, and
ability to encapsulate high drug loadings [8]. It also has function in air filtration, tissue engineering [9] and wound healing purposes [10,11].

Polyvinylpyrrolidone (PVP) along with polyvinyl alcohol (PVA) and polyvinyl acetate (PVAc) are vinyl polymers that are extensively used for the development of oral controlled-release products [12]. From the crystalline state point of view, the vinyl polymers are amorphous in nature. Then, based on the water solubility, PVP is a water soluble polymer. Among them, PVP is often used for solid dispersions containing amorphous drugs in which amorphous-state drugs show improved dissolution rates compared to their crystalline counterparts [13,14].

In this study, Polyvinylpyrrolidone (PVP) nanofibers were successfully prepared by the electrospinning and characterized in three different concentrations. The morphology and structure of the PVP-1, PVP-2, and PVP-3 nanofiber mats were analyze scanning electron microscope (SEM), Fourier transforms of the infrared spectrophotometer (FT-IR). The effect of increased PVP molecule weight in the PVP-1, PVP-2, and PVP-3 nanofibers on morphology.

2. Experimental

The materials used to produce PVP nanofiber and nano particle were Polyvinylpyrrolidone (PVP) which PVP-1 (MW 1,300,000 kg mol⁻¹), PVP-2 (MW 360.000 kg mol⁻¹) and PVP-3 (MW 40.000 kg mol⁻¹) obtained from Sigma Aldrich. Ethanol (analytical grade, Merck) was used as the solvent.

The fabrication of PVP fiber started with dissolving PVP at a concentration 10% into Ethanol. A series of polymer molecule weight was prepared: 1.300.000 kg mol⁻¹, 360.000 kg mol⁻¹ and 40.000 kg mol⁻¹ which then labeled as PVP-1, PVP-2, and PVP-3, severally. Each precursor was stirred by using magnetic stirrer for ±3 hours at temperature 40°C to produce homogeneous solution.

The polymer solution was electrospun using Electrospinning machine (Nachriebe 601) as illustrated in schematic diagram in Figure 1. The apparatus consists of a high voltage power supply, syringe with needle diameter of 0.8 mm, syringe pump, and aluminium-coated rotating collector. The apparatus was placed on a temperature and humidit-controlled chamber (25±0.5°C, RH 50%). Initially, the polymer solution was loaded into the syringe. The polymer was then dispensed by the syringe pump at a constant flowrate of 0.3 ml/hr. The needle tip was positively charged at 10 kV to attract the polymer solution out of the needle. The grounded drum collector was allowed to collect the resulted fiber at rotation speed of 200 rpm and tip collector distance of 12 cm. The Taylor Cone formation on the needle tip was monitored using a charge-coupled device (CCD) camera.

![Figure 1. The schematic of nachriebe 601 electrospinning apparatus.](image)

The morphology of PVP-1, PVP-2, and PVP-3 nanofibers were determined using a scanning electron microscope (SEM, JSM-6510; JEOL, Tokyo, Japan). The size distribution of the PVP-1, PVP-2, and PVP-3 fibers were determined using Origin ver.8 software (OriginLab Corporation, USA). The functional groups and changes occurring in PVP-1, PVP-2, and PVP-3 nanofibers were identified using
a Fourier Transform Infrared Spectroscopy (FTIR) spectrum (Alpha; Bruker, Germany) at 500-4000 cm⁻¹ spectral range. All samples were trimmed into 3 × 20 mm rectangle with similar thickness and clamped on both sides of the apparatus and then stretched at elongation rate of 20 mm/min. The functional group in each sample were determined by looking at the number of peaks in the form of transmittations. Moreover, the information about compound structure and the purity of PVP could be assessed.

3. Results and discussion

3.1. PVP nanofibers image

The physical appearance of PVP-1, PVP-2, and PVP-3 produced from the electrospinning process can be seen in Figure 2. Macroscopically. PVP which has a difference in molecular weight but dissolved by same solution concentration will obtain heterogeneous fiber morphology. The nanofiber form of PVP-1 (MW 1,300,000 kg/mol⁻¹) are shown in Figure 2a. which is shaped like fine white layer. The form of PVP-2 (MW 360,000 kg/mol⁻¹) Nanofibers are displayed in Figure 2b. whose shape is similar to PVP-1 but it’s thinner and more brittle than PVP-1 layers. Afterwards, PVP-3 (MW 40,000 kg/mol⁻¹) were accumulated to form a pile of white particle which are shown in Figure 2c. The successful formation of the fibers was a result of three contributing factors. The first factor is the Coulomb force (Fc), where high electrical voltage applied on the needle tip induces charge on polymer and consequently the polymer has a tendency to move towards the grounded collector. The second factor is the loading force (Fd) from the syringe pump, which provides polymer drop available to be spun. The third factor is the surface tension force (Fγ) at the polymer-air interface, which retains the polymer droplet on the needle tip. The equilibrium of these three forces results (Eq. 1) in a cone jet formation known as Taylor's cone [15,16].

\[
F_c + F_d - F_\gamma = 0
\]  

(1)

When Fc is introduced to the polymer, the polymer chains elongates while the solvent evaporates, resulting in solidified fiber formation on the collector surface [6,17]. When the voltage was set at 10kV percursors droplets would form Taylor’s cone, the equations can be written by:

\[
V_c = \sqrt{\frac{\gamma d^2}{\varepsilon R}}
\]  

(2)

In which \(V_c\) is a critical voltage that caused precursors being attracted and forming a cone, \(d\) the distance of the needle tip to the collector, \(\gamma\) is a solution surface tension, and \(R\) is the main curvature of precursor. As shown in the figure, the electrical voltage plays an important role in the formation of nanofiber as described previous research [18]. The increased of nanofiber diameters might occur with high voltage due to the acceleration of the mass flow of the polymer solution by excessive electrostatic force [19].

![Figure 2](image-url)  

**Figure 2.** Electrospun nanofibers and nano particle Polyvinilpyrrolidone with 10% concentration.
3.2. Morphology and diameter of PVP nanofiber

The morphology of PVP-1, PVP-2 and PVP-3 fiber is shown in Figure 3. The level of solution viscosity was affected by solution concentration and dissolved molecular weight. It is polyvinylpyrrolidone with a different molecular weight that influence the precusors’ viscosity. High polymer molecular weight (1,300,000 kg/mol) produced unbeaded fibers (figure), which was attributed to the viscosity of the solution.

At low polymer viscosity (MW 40,000 kg/mol) has fewer inter-chain bonds, causing the polymer solution to be unable to maintain continuous elongation during the stretching of the jet [9]. This condition will result in beaded fiber. It has been established that the formation of beads is associated with viscoelasticity of the solution [11]. In electrospinning process, viscoelastic force permits continuous conversion of the jets into fibers instead of beads. In low viscosity solutions, the viscoelasticity of polymer droplet on the needle tip is unable to overcome Rayleigh instability, an axisymmetric rotation that causes the stretched jet to break up and form beads [20]. As the polymer molecular weight decreases (MW 1,300,000 kg/mol and 40,000 kg/mol) beaded fibers were produced (Figure 3c).

The effect of PVP concentration on fiber diameter is displayed in Fig. 3a. It was found that higher PVP molecular weight produced fibers of greater diameter. In fact, a PVP viscosity are influenced by the weight of molecular solute. Fiber diameter is strictly controlled by the viscosity of polymer, where the degree of chain entanglement is increased with higher viscosity, leading to the formation of a larger fiber diameter [21].

In this study, the fiber diameter of polyvinylpyrrolidone 10% (w/v), PVP-1 (MW 1,300,000 kg/mol), PVP-2 (MW 360,000 kg/mol), and PVP-3 (MW 40,000 kg/mol) are 2.620 nm for PVP-1 shown at figure 3a, 1.760 nm for PVP-2 shown at figure 3b, and 1,380nm for PVP-3 shown at figure 3c. It is
PVP-3 which has lowest both molecular weight and viscosity among other dissolved PVP. Therefore, from these data above, we can see that smaller polymer viscosity will obtain smaller diameter of fiber and it will contain a lot of beads.

3.3. Fourier-Transform Infrared (FTIR)

FTIR analysis was performed to study the influence of the process on the chemical structure of the polyvinylpyrrolidone (PVP) 10% (w/v) nanofiber. It is also conducted to identify characteristic functional groups in PVP-1, PVP-2, and PVP-3 nanofibers. The FTIR spectrum of PVP is shown in Figure 5a. FTIR spectrum at 3000-3500cm⁻¹ shows a broad peak direction at wave number 3391cm⁻¹ (PVP-1), 3402cm⁻¹ (PVP-2), and 3419cm⁻¹ (PVP-3) which are hydroxyl groups including stretching O-H bonds [20,22]. if it is seen from the peak, hydrogen bonds from PVP-1, PVP-2 and PVP-3 peak added (up). Another peak appears at wave number 2932cm⁻¹, 2953cm⁻¹, 2954cm⁻¹, all three bonds are identified as a stretch of C-H bond. A stretch of C = O (carbonyl group) bonds regulated for PVP-1, PVP-2 and PVP-3 samples can be seen from the peaks shown at wave numbers 1643cm⁻¹, 1644cm⁻¹ and 1645cm⁻¹. At peak 1288cm⁻¹, 1287cm⁻¹ and 1286cm⁻¹, there is a bending of C-N bonds. While at peaks 1438cm⁻¹, 1422cm⁻¹ and 1421cm⁻¹, it occurs a bending of the C-H bond. This can be seen from the peak between wave numbers 1000 - 1500cm⁻¹. For PVP 1 samples, PVP 2 and PVP 3 wave numbers move to higher values, and peaks between 500 - 1000cm⁻¹ happen a bending of the C-C bond. The higher ethanol content causes the hydroxyl peak to be sharper and more visible from 3000cm⁻¹ to 3500cm⁻¹, consequently the hydroxyl and carbonyl (CO) peaks move to the lower wave number direction. The stretching peak of this carbonyl appeared in 1643cm⁻¹ in PVP-1, 1644cm⁻¹ in PVP 2 and 1645cm⁻¹ in PVP 3, the movement peak of the carbonyl stretch and other peaks was considered as a result of the comparison of PVP molecules caused by the electrospinning process.

4. Conclusion

A smooth and uniform nanofiber with diameter 2.860nm (PVP-1), 1.760nm (PVP-2), and 1.3800nm (PVP-3), was manufactured by electrospinning method using Polyvinylpyrrolidone with concentration 10% (w/v) in three different molecular weight PVP-1 (MW 1,300,000 kg/mol⁻¹) PVP-2 (MW 360,000 kg/mol⁻¹), and PVP-3 (MW 40,000 kg/mol⁻¹). the difference of molecular weight influenced the solution viscosity. it is also affected nanofiber structure. The PVP-3 has smallest diameter and producing lots of
beads. Moreover, the polymer remained amorphous in PVP-3. This study provides an insight into enhancement of physical characteristics of hydrophilic by the right choice of polymer blends.

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