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Fabrication of Polyvinylpyrrolidone Fibers by Means of Rotary Forcespinning Method

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Abstract. Fibers made from polymer materials have been widely developed as a carrier medium of active ingredients in drug delivery systems. In this research, PVP polymer was chosen because of its wide and safe use in the medical field. The purpose of this study was to produce PVP fibers that can later be applied as a carrier of active ingredients in drug delivery systems. The rotary forcespinning (RFS) method was chosen to shorten the time of production and to overcome the limitations of electrospinning method such as the use of high voltage and dielectric solutions. The PVP solution was varied in several concentrations (8 wt%, 10 wt%, 12 wt%, 14 wt%, 16 wt%, and 18 wt%) to achieve the best fibers morphology. The morphology and the diameter of fibers were analyzed using a digital microscope. From the microscope images, it can be shown that beaded fibers were formed when the concentration of polymer in the precursor solution was low. The number of beads decreased as the concentration of polymer increased. Beads-free fibers were fully formed at above certain polymer concentration.

Keywords. Fibers, polyvinylpyrrolidone and rotary forcespinning.

1. Introduction

Fibers, at a size ranging from micro-to-nanometer, have been developed intensively in engineering material for a wide range of applications. One of the broadest applications of micro/nanofibers is in the medical field. They can be used as a drug delivery system, body tissue scaffolding, membrane filter, transparent conductive fibers, biocatalysts and wound dressing [1]. The fibers at this size have many advantages such as it has a very high ratio of surface area to unit volume, low density, high porosity, and high flexibility [2–3]. These characteristics are very much needed if the fiber is used as a carrier for the active agent in drug delivery systems. By possessing these characteristics, the solubility, the dissolution rate, and the biological availability of the active agent can be increased significantly although it previously has low solubility when being swallowed directly in its original form.

Since the building block of fibers is a polymer, a precursor solution with a certain concentration of polymer must be first prepared before the fiber is being synthesized. Some commonly used polymers are poly(ethylene glycol) (PEG), polyvinylpyrrolidone (PVP), polyvinyl alcohol (PVA), polyacrylic

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acid (PAA), polyacrylamides, polylactic acid (PLA), cellulose acetate, and others [4]. In this study, PVP powder was used since it has been proven by the US Food and Drug Administration (FDA) for health applications, especially for food and medicine. Therefore, it can be used as a carrier of active agents in drug delivery systems [5]. Also, PVP can be dissolved very easily in ethanol which often being used as the solvent in medical application[6]. Additionally, there were reports that PVP powder can be successfully synthesized into nanofibers with the conventional electrospinning method [1,7]. Electrospinning is a technique to produce fibers by using a high electric field to form fibers [8]. Unfortunately, this method requires a very long time [1]. Therefore, a better method must be developed to reduce the duration needed.

Rotary forcespinning (RFS) is a method that can eliminate and/or minimize the limitations encountered in electrospinning. The high rotational speed of RFS ensures that the solution will run out rapidly compared to the time-consuming electrospinning [4,9]. Therefore, the use of RFS technique can improve the selection of material, increase the production rate, and lower the cost of fibers synthesis through environmentally friendly processes [10–11]. In our previous experiment, we used the RFS technique directly to encapsulate natural extract into PVP fibers [12]. In this research, we varied the concentration of PVP solution to observe the change of morphology of the fibers. To the best of our knowledge, there is no study that relates the concentration of PVP to the diameter of rotary forcespun fibers, although the similar study has been conducted using PEG [11].

2. Materials and methods

2.1. Synthesis of precursor solution

PVP powder K85-95 (1,300 kg·mol^{-1}) was purchased from Sigma Aldrich and ethanol, as the solvent, with 96 % purity was purchased from Sakura Pharmacy Store. The solution was synthesized at 40 °C, stirred by a magnetic stirrer at ± 400 rpm for ± 60 min until a clear homogeneous solution was achieved.The concentration of PVP in the solution was varied at 8 wt%, 10 wt%, 12 wt%, 14 wt%, 16 wt%, and 18 wt%.

2.2. The production of fibers using rotary forcespinning method

In the RFS technique, the solution is placed inside a rotating container, in which the solution is then ejected out from the reservoir through a hole. The electric field used to attract fibers in the electrospinning process is replaced by the centrifugal force of motor rotation. As known from elementary physics, the centrifugal force (F) depends on mass (m), rotational speed (ω) and radius (R) and they are related according to F= $m\omega^2R$. The centrifugal force becomes the main factor in the production of fibers, and the stretch of this force causes the solution to turn into fibers. There are three main systems of the RFS technique: a motor system, a collector, and a heating system that controls the temperature and humidity around the motor [4,10]. Some steps of the RFS method in producing fibers are as follows (also described in Figure 1):

1. Jet initiation to stimulate the flow of the polymer solution through the hole,
2. Jet extension to increase the surface area of the polymer solution flow driven out from the reservoir through the hole (elongation process),
3. Solvent evaporation and solidification processes [13].

By using centrifugal force to stretch the fiber and by adjusting the parameters involved such as the inner diameter of the needle, the distance between the needle tip, and the collector, the concentration of the solution, the flow rate, and the surface tension, the RFS technique is a proficient method to produce fibers.

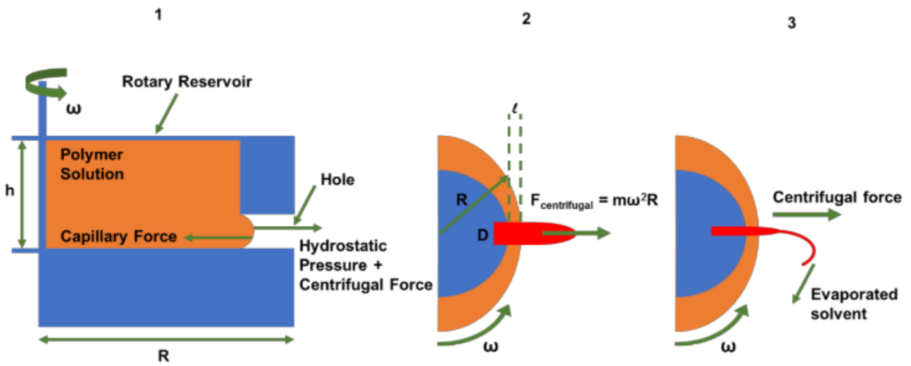

Figure 1. The mechanism of fibers formation using the RFS technique[13]

The homogeneous PVP solution was placed in a single nozzle syringe, which was connected to a silicon hose with an inner diameter of 0.45 cm. The collector was coated with an aluminum foil in which the fiber was attracted onto. The hole diameter was 0.6 cm, the distance between the hole and collector was 10 cm, the rotational speed was set to be 15,000 rpm. The flow rate of the solution in the syringe was 30 mL/h, and the relative humidity inside the chamber was maintained at 80 %. The spinning process was carried out at 23 °C.

2.3. Characterization of morphology and diameter of fibers

The characterization of the morphology of the fiber was performed using a digital microscope (National, DC3-163). The magnification of digital microscope used was varied from 100×, 200×, 400× to 1000×.To calculate the diameter of the composite fiber, we used ImageMIF software developed in the Laboratory of Electronics and Instrumentation in Department of Physics, Institut Teknologi Bandung.

3. Results and discussion

The synthesis of PVP fibers was initiated by preparing precursor solutions at six different concentrations, which were 8 wt%, 10 wt%, 12 wt%, 14 wt%, 16 wt%, and 18 wt%. The concentration of the solution was varied to observe the effect of concentration on the morphology of the fibers. Other than the concentration, all other parameters were kept constant. The resulting fibers were then characterized using the digital microscope (Figure 2).

As illustrated in Figure 2, the variation of the polymer concentration has a significant effect on the formation of beads and the diameter of the fibers formed. The fiber synthesized from precursor solution with the lowest concentration (8 wt%) produced beaded fibers with a large number of beads. As the concentration of the solution increased, the number of beads decreased. The fibers made from a precursor solution with a concentration of 16 wt% were beads-free. However, when the concentration of the polymer solution was increased further to 18 wt%, beaded fibers were re-formed although at a smaller number compared to the beads at made from the precursor solution with a concentration lower than 16 wt%. Therefore, it can be concluded that most uniform morphology of fibers was achieved when the concentration of polymer was 16 wt%

To explain the formation of fiber beads, one must refer to the surface tension and viscosity of the precursor solution. In this experiment, the concentration of 14 wt% can be called as the critical concentration since it produced the very small amount of beads. The term critical concentration is a specific concentration in which the morphology of the fibers starts to be continuous and also free of beads. When the concentration is less than the critical concentration, the viscosity of the solution is low. At low viscosity, the polymer chain has low binding energy, which is then not strong enough to form fibers. On the other hand, high surface tension causes the jet to become unstable, resulting in the decrease of surface area per unit of mass and the jet tends to form spherical shapes. Therefore, spherical droplets will appear on the fibers and the viscoelastic force in the polymer solution will withstand sudden changes of shape (spheres) which will then produce fibers with beads. As the concentration of polymer becomes higher, the viscosity will be larger as well. This will cause the polymer chain to have a stronger bond as the concentration goes near to the critical concentration. At this situation, beads-free fibers are then formed. Even if there is still some small amount of beads, the slight increase in polymer concentration will reduce the number of beads although the diameter of beads will increase [11].

However, as shown in Figure 2, there were beads of fibers made from the precursor solution at a concentration of 18 wt%, which should not exist based on the argument in the previous paragraph. This shows that there are other factors that also trigger the formation of beads other than the viscosity and surface tension. The factors that may trigger the formation of beads in the fiber are the rotational speed and the centrifugal force generated by the ac motor. Just as there is critical concentration, there also exists critical rotational speed, which is the critical rotational speed for each concentration of polymer in the precursor solution to form fibers [14]. The greater the concentration of the solution, the greater the rotational speed is required.

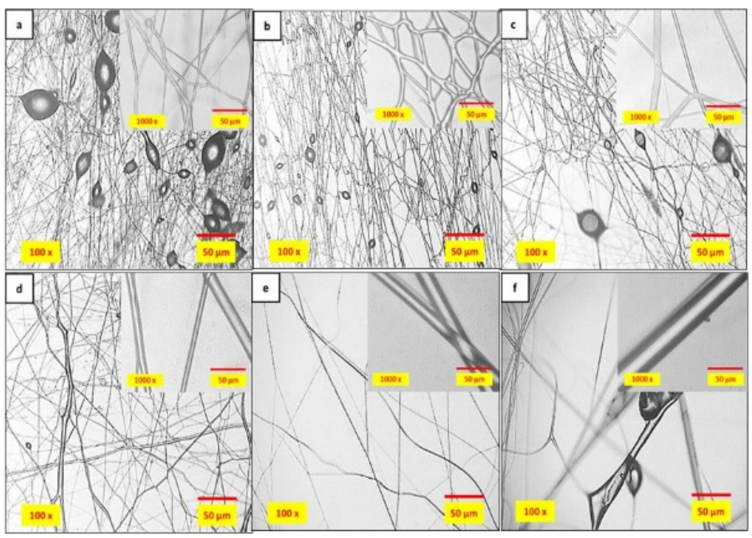

Figure 2. The morphology of PVP fibers under a digital microscope with a magnification of 100× for PVP concentrations of (a) 8 wt%, (b) 10 wt%, (c) 12 wt%, (d) 14 wt%, (e) 16 wt% and (f) 18 wt%

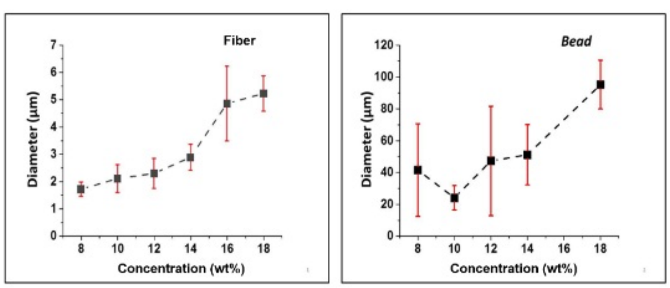

Figure 3. The effect of increasing the concentration to the average diameter of fibers and beads.

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In this experiment, the rotational speed was kept constant for all concentrations of the precursor solution. At the concentration of 18 wt%, the rotational speed of 15000 rpm was not sufficient to produce a centrifugal force capable of attracting fibers. Thus, the fibers formed were not perfectly cylinder, and some spherical droplets appeared. Due to its large concentration, the resulting fibers had a large diameter and large beads. The exact relationship between the concentration and diameter is illustrated in Figure 3. It can be seen that for a higher concentration, the average diameter of fibers is greater as well.The fibers made from the solution concentration of 18 wt% had the largest average diameter, which was 5.22μm [4,11,15]. Lastly, Figure 4 depicts the distributions of the resulting fiber diameter. To decide whether a group of fibers is homogenous, a ratio called as the coefficient of variation (CV) is used, which is simply a ratio between the standard deviation of fibers diameter over the average fiber diameter. For CV larger than 0.3, the fibers are non-uniform, otherwise, the fibers are uniform.

As shown in Table 1, all CVs have a value less than 0.3. However, it should be taken into note that the beaded fibers were fully achieved when the precursor solution was PVP 14 and 16 wt%. Since both sets of fibers have CVs below 0.3, the resulted fibers can be categorized as homogenous [15].

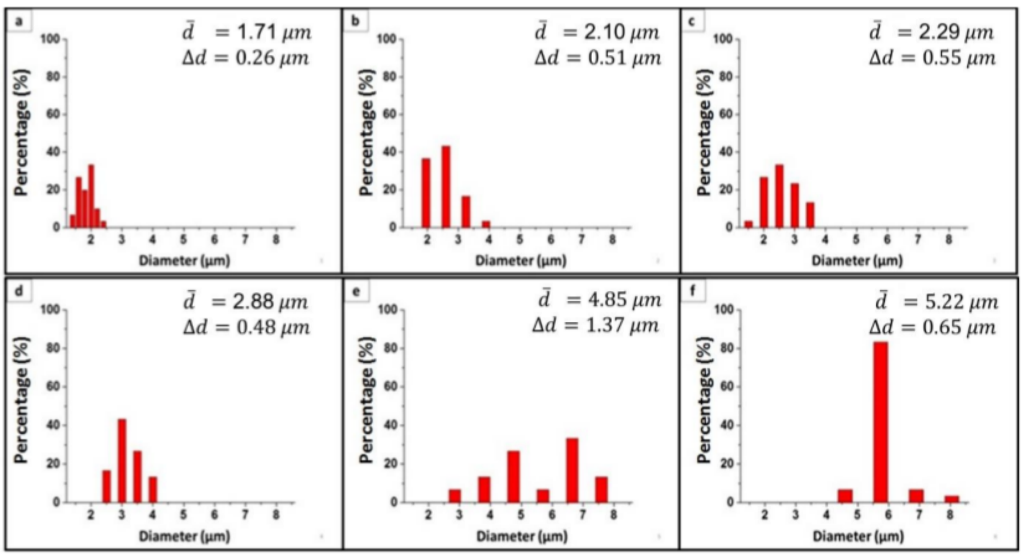

Figure 4. The distribution of PVP fibers diameter produced from the precursor solution concentrations of (a) 8 wt%, (b) 10 wt%, (c) 12 wt%, (d) 14 wt%, (e) 16 wt%, and (f) 18 wt%

Table 1. The physical characteristics of rotary forcespun fibers

Solution concentration (wt%)	Average diameter of fiber [μm]	Coefficient of variation
8	1.71±0.26	0.15
10	2.10 ± 0.51	0.24
12	2.29 ± 0.55	0.24
14	2.88 ± 0.48	0.17
16	4.85 ± 1.37	0.28
18	5.22 ± 0.65	0.12

4. Conclusions

The fibers built up from PVP have been successfully synthesized using rotary forcespinning method although the fibers contained some beads. The morphology and diameter of fibers are affected by several parameters, such as solution concentration, viscosity, surface tension, and rotational speed. The greater the concentration of the solution, the viscosity will be greater thus resulting in the greater average diameter of fibers, and vice versa. The value of viscosity also indicates the binding strength of between polymer chain, which is a very important to determine whether fibers will be formed. The large surface tension leads to the formation of beads on the fibers. A solution with low viscosity indicates that the bonds in the polymer chain are not strong enough to be attracted and to form fibers.

Acknowledgments

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