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Effects of Electrospinning Nozzle size and Voltage on Polyvinylpyrrolidone Fiber Structure Formation

In-Bo Shim · Taejoon Kouh*
Department of Physics, Kookmin University, Seoul 02707, Korea

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The formation of a polyvinylpyrrolidone fiber via electrospinning is investigated. Especially, the effects of the nozzle size and voltage during electrospinning are studied. For the experiment, three different nozzle sizes are used for fiber production under electrospinning at voltages of 5, 10, and 15 kV. A careful examination of the scanning electron micrographs reveals that both are important factors in determining the dimension and the uniformity of the electrospun fibers. A smaller nozzle size with a higher voltage is preferable for forming a fiber structure with a smaller diameter, down to sub-microns. Also, a high electrospinning voltage tends to lead to the uniform formation of the fiber structure, as indicated by the coefficient of variation in the fiber diameter distribution. The study presented here can help to guide and optimize the electrospinning process for the controlled production of fiber structure.

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I. INTRODUCTION

The electrospinning technique has regained the popularity due to its capability of producing very fine fiber structures based on the wide range of materials [1, 2]. Unlike other fabrication techniques, the electrospinning technique can be easily employed by continuous injection of desirable material to a nozzle — held at high voltage — and collecting the fiber on a separate substrate, grounded. The diameter of the fiber, pulled through a nozzle during the electrospinning process, can be below deep sub-micron, which can be highly attractive in various technological fields with high surface-to-volume ratio. Some of examples are the application in tissue engineering [3], development of composite materials [4], utilization in chemical process and environmental science [5] and fabrication of electronic devices [6].

Although the electrospinning can provide a simple means of manufacturing nanofibers, the physics involved in the electrospinning process is rather complex [7], and there are many processing factors governing the detailed formation of the resulting fiber structures such as the fiber diameter and the uniformity. The relevant factors can be the characteristics of the material and the solution to be electrospun, the injection rate into the nozzle, the size of the nozzle from which the fiber being pulled, and applied electric voltage between the nozzle and the collecting substrate as well as the distance between the nozzle tip and the substrate. Especially, the combination of these parameters during the electrospinning process makes the quantitative understanding and prediction of the resulting electrospun fiber structure rather challenging as pointed by other reports on the electrospinning technique [8–10].

In this manuscript, the formation of the electrospun fibers is investigated under different electrospinning parameters. Especially, the electrospinning nozzle size and the voltage applied to the nozzle are considered as two main factors and the electrospinning process is performed with different nozzle sizes and electrospinning voltages, while other electrospinning parameters are kept constant. The careful observation given in the manuscript

*E-mail: tkouh@kookmin.ac.kr

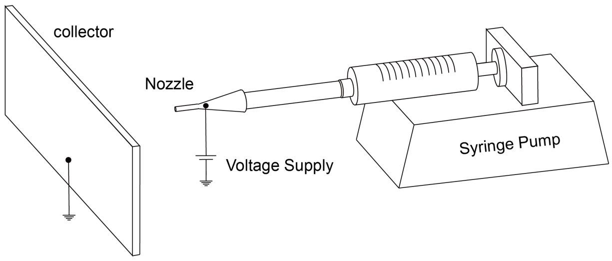

Fig. 1. Schematic of the electrospinning set-up. The flow of PVP solution is continuously delivered by the syringe pump to the nozzle at high voltage. The resulting electrospun fibers are deposited on the collector plate, held at ground.

can reveal how these two factors affect the size distribution of the resulting fiber structures and help to find the appropriate processing conditions for the productions of fiber structure with desired physical dimensions.

II. EXPERIMENTS

Figure 1 illustrates the electrospinning set-up used in the experiment. Prior to the electrospinning, the solution of polyvinylpyrrolidone (PVP) is prepared by adding PVP powder (molecular weight of 1,300,000) to ethanol. The mixture is continuously stirred for about 24 hours until PVP is completely dissolved. This solution is transferred to a syringe and slowly injected into the metallic nozzle, connected to a voltage supply, at the end of the syringe with a syringe pump. The fibers produced under high voltage are deposited on an aluminium-foil collector plate, held at ground. During the electrospinning process, the humidity is maintained below 25%. After the surface of the collector is coated with a thin layer of PVP fibers, the process is stopped and finalized by drying the electrospun fiber on the collector at 120 °C in air for 48 hours.

Since the effects of the nozzle size and the voltage are considered here, three different nozzles with inner diameters of 0.20, 0.40 and 0.84 mm are used under electrospinning voltages of 5, 10 and 15 kV. Besides the nozzle and the voltage, other parameters are kept fixed throughout the experiments: the concentration of PVP is 11 wt%, the flow rate controlled by the syringe pump is 1 ml/h, and the distance between the nozzle tip and the collector is 50 mm.

The resulting fiber structure is studied with scanning electron microscope. A number of scanning electron micrographs are taken at various positions on the fiber-coated collector surface for each experiment and the diameter of individual fiber is carefully measured. To avoid any modification of the fiber surface, the scanning electron measurements are taken without additional coating of the fiber with metallic film.

III. RESULTS AND DISCUSSION

Figure 2 shows the electron scanning micrographs of the electrospun fiber for the nozzle size of 0.20 mm under three different electrospinning voltages as well as the histograms of the fiber diameter distribution. The electron scanning micrographs show well-defined fiber structures with its size distribution changing with the applied voltage difference between the nozzle and the collector. At low voltage of 5 kV, the fibers have a mean diameter of 1 μm and its diameter gets smaller with increasing the electrospinning voltage. Also, with increasing voltage, it can be seen that the size distribution of electrospun fibers becomes narrower. These clearly suggest that these factors affect the formation of fibers through the electrospinning process.

To further demonstrate the effect of these on the electrospinning process, the diameter of the fibers, electrospun with different nozzle sizes and voltages, are measured as in Fig. 2 and the mean diameters at each electrospinning condition are compared in Fig. 3 (a). However, when the electrospinning is performed with the nozzle size of 0.84 mm at the applied voltage of 5 kV, the deposition of fibers on the collector surface is not observed, while the deposition of PVP fibers is clearly visible at other electrospinning conditions. This suggests that the bias voltage of 5 kV is not sufficient for the nozzle diameter of 0.84 mm to reach the critical point necessary for the formation of the electrified jet [11].

From the voltage dependence of the electrospun fiber diameter, one can notice that the fiber diameter gets smaller with higher voltage for all nozzle sizes as seen for the nozzle size of 0.20 mm. Between 5 and 10 kV, the mean diameter of the fiber decreases rapidly, while it varies slowly above 10 kV. Also, the nozzle size clearly

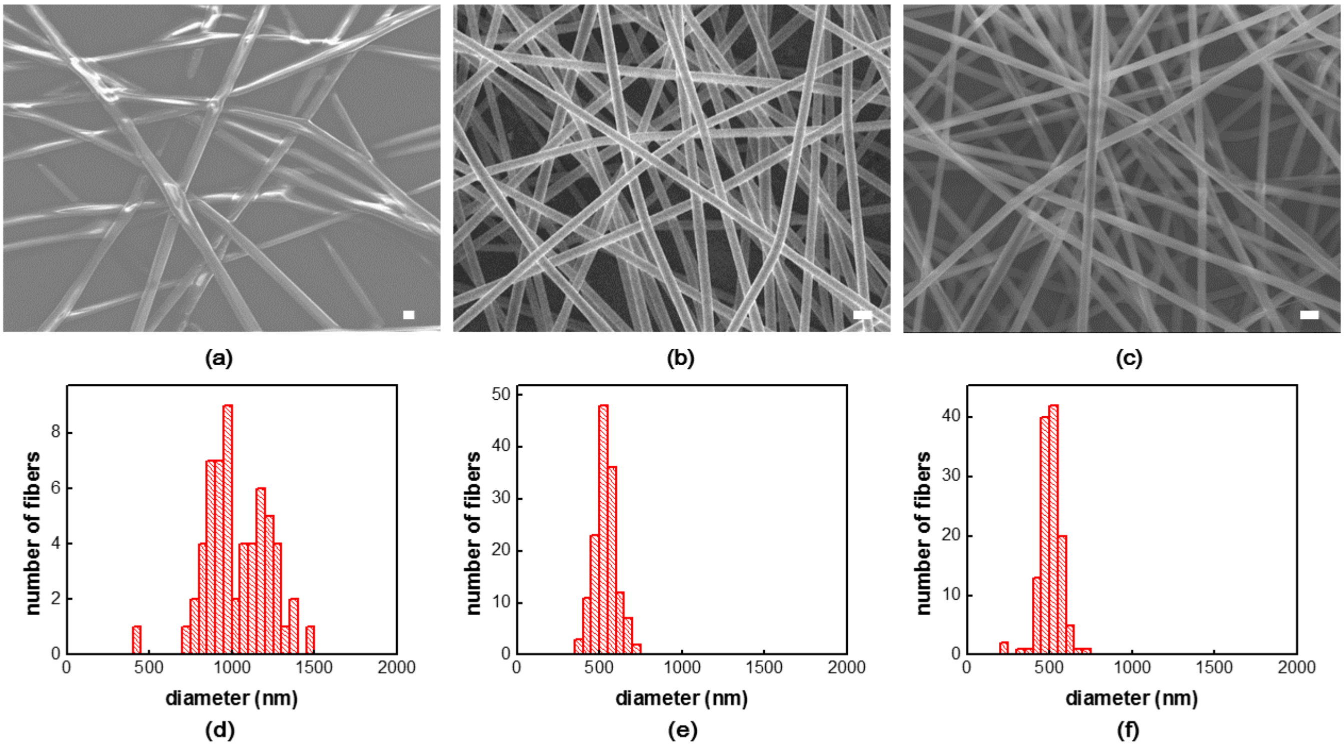

Fig. 2. (Color online) The electron scanning micrographs of electrospun fibers obtained with nozzle diameter of 0.20 mm at the electrospinning voltage of (a) 5, (b) 10 and (c) 15 kV. The corresponding histograms of the fiber diameter distribution are also shown for (d) 5, (e) 10 and (f) 15 kV.

affects the resulting fiber diameter. Increasing nozzle diameter generally results in larger fiber diameter. However, at low voltage of 5 kV, the nozzle sizes of 0.20 and 0.40 mm do not make much difference in mean diameter and the effect of the nozzle size on the mean diameter is rather evident at higher bias voltage.

The experimental observation can be understood as the overall effect of these factors on the jet coming out of the nozzle [10,12]. We expect that the reduction in the fiber diameter associated with high voltage is due to the increasing repulsive force within the electrified jet at the end of the nozzle, leading to stretching of the jet into thinner fibers. Also, since the amount of the jet from the end of the nozzle will increase with the nozzle size, associated with the formation of large Taylor cone, this will tend to form fibers with large diameters. However, it does not follow simple linear relationship. For instance, almost doubling the size of the nozzle from 0.40 to 0.84 mm does not result in increase in the mean fiber diameter in same ratio — for the nozzle diameters of 0.40 and 0.84 mm at 15 kV, the mean diameters are 783 and 831 nm, respectively. Such suggests the interplay between these factors during the electrospinning process.

The uniformity of the fiber structure is also considered. Since the mean fiber diameter varies with the nozzle size, the coefficient of variation, determined from the histogram of the fiber diameter distribution, is considered and presented in Fig. 3 (b) to compare the uniformity of the obtained fiber structure at various conditions. Figure 3 (b) shows that the uniformity of the fiber is sensitive to the nozzle size, rather than to the applied voltage. For the nozzle diameter of both 0.20 and 0.40 mm, the coefficient of variation lies in the range of 10 to 20% regardless of the bias voltage, but for 0.84 mm the value of coefficient is above 30%, slightly decreasing with voltage.

IV. CONCLUSIONS

In summary, the formation of electrospun PVP fiber structure is examined at various electrospinning conditions. The experimental observation within the electrospinning conditions considered here shows that small

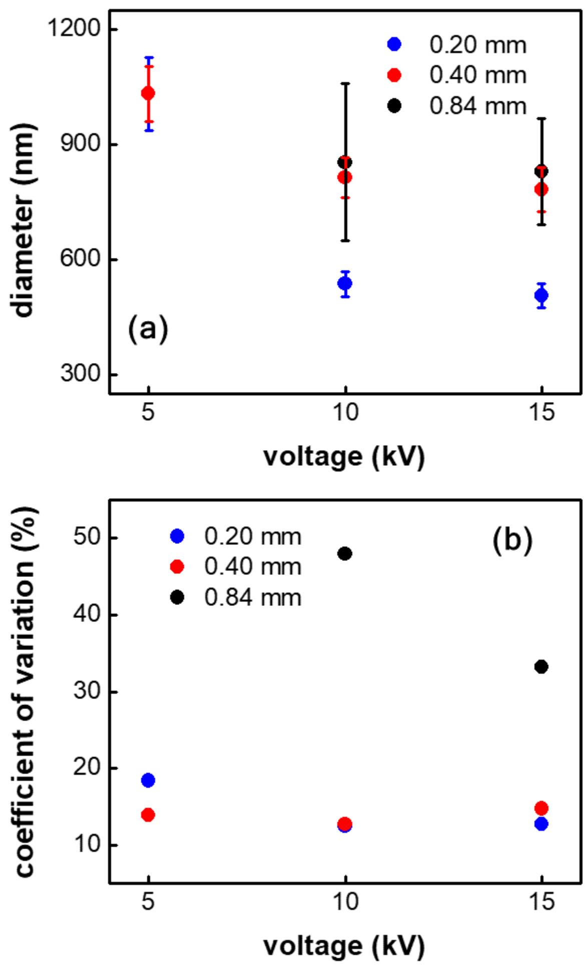

Fig. 3. (Color online) (a) Mean diameter of the electrospun fiber as a function of the applied voltage for nozzle sizes of 0.20, 0.40 and 0.84 mm. (b) Coefficient of variation of the electrospun fiber diameter as a function of the applied voltage for nozzle sizes of 0.20, 0.40 and 0.84 mm.

nozzle size and high voltage is preferable to obtain thin fiber structure down to 500 nm. Also, fine tuning of the fiber structure over wide range of the diameter is viable when the smallest nozzle diameter is used. For example, the mean diameter of the fiber within the range from 1 μm to 500 nm is possible for the nozzle size of 0.20 mm with the bias voltage between 5 and 15 kV. One of the important aspects of the fiber production is the uniformity. The coefficient of variation indicates that the uniformity highly depends on the nozzle diameter with tendency of leading to less variation in fiber dimension with smaller nozzle diameter. Considering the recent interests in the electrospinning technique and nanofiber production, the study presented here can be beneficial to understand and tailor the necessary electrospinning process parameters for precise control of structural characteristics.

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