Investigation of a New Needleless Electrospinning Method for the Production of Nanofibers

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ABSTRACT

This paper presents the possibility of nanofiber formation by a new multiple jet method. A novel needleless electrospinning apparatus was used to produce nanofibers. This employs a new design for supplying solution to a metal roller spinneret. The advantage of this setup is its ease of scaling-up for increased output. Using this new method it was possible to increase the nanofiber production rate because of the multiple jets. The productivity rate has been significantly enhanced and was 24-30 times higher than single needle electrospinning. It was also possible to produce thinner fibers than the single needle method. It was found that fibers produced by this novel needleless electrospinning had fewer beadings than fibers produced by the conventional electrospinning method. The effects of processing parameters including applied voltage and spinning electrospinning distance on eletrospun fiber diameter were also investigated. The study showed that the electrospun fiber diameter was strongly governed by the processing parameters. It was observed that there was a strong interaction between these parameters.

INTRODUCTION

Electrospinning is a process used to produce ultrafine polymeric fibers in the average diameter range of 50 nm to 500 nm [1]. The interest in nanofibers has increased drastically over the past several years. Due to their extremely high surface to weight ratio, nanofibers exhibit special properties that have opened up a wide range of potential applications. Nanofibers can be used in filtration, medical applications like drug delivery, tissue engineering, wound dressing [2-11], functional textiles [12], nano-electronics [13-16], nano-composites [17], and energy applications like solar cells and batteries [18]. The electrospinning process uses high voltage to create an electric field between a droplet of polymer solution at the tip of a needle and collector plate [19-21]. When the electrostatic force overcomes the surface tension of the drop, a charged jet of the polymer solution is ejected. As the solution moves away from the needle and towards the collector, the solvent evaporates and the jet rapidly thins and dries. On the surface of the collector a nonwoven mat of randomly oriented nanofibers is deposited. In recent years, the most important problem in electrospinning is its low production rate. Increased production rate is essential in order for nanofibers to be adopted for mass commercial purposes. Many research studies and patents were reported about an electrospinning method with a single capillary. However, it has been observed that it is very difficult to increase the production rate by the conventional method with a single capillary. A typical production rate from a single spinneret is 0.1-1g of fiber per hour, depending on the solution properties and operating parameters [22]. Operational and quality control issues such as nozzle clogging and special variation of the jets from the nozzle are often cited as problems encountered by these approaches. To solve these problems, several new methods have been developed in recent years. All of these methods share the feature that liquid jets are launched from a free liquid surface and are called free surface electrospinning or needless electrospinning. Such methods have several potential benefits, including simplicity of design, lower equipment and operating costs, higher production rate, higher fiber uniformity, higher web uniformity, and higher fiber packing density. A high productivity capillaryless method was developed by Jirsak [23]. In this method a rotating cylinder was used to obtain nanofibers and an increase in productivity was claimed. Kameoka and Craighead presented a method for the formation of oriented polymeric nanofibers using electrospinning deposition from an integrated microfluidic device. They studied the effect of the rotational speed of a counter electrode on the morphology of nanofibers [24]. Lee et al.

also patented a high speed apparatus for polymer web production by electrospinning [25]. Chu et al. developed a multiple jet system allowing high production rates for a commercial process [26]. He et al. applied vibration technology to electrospinning for the first time. Using this technology it was possible to produce much finer nanofibers compared to those manufactured by the conventional electrospinning method. Another new capillaryless approach was proposed by Yarin and Zussman [27] in 2004. This method comprised a two-layer system. The lower layer was a ferromagnetic suspension and the upper layer was a polymer solution. This system was subject to a magnetic field provided by a permanent magnet. Tomaszewski and Szadkowski constructed three types of multi-jet electrospinning heads (series, elliptical, and concentric) for the electrospinning method [28] .The head effectiveness was tested by using poly (vinyl alcohol)/water solution as the spinning liquid. In 2006, Dosunmu et al. presented a novel method for electrospinning multiple jets from a cylindrical porous tube. The solution was electrified and pushed by air pressure through the tube. This porous tube was made of polyethylene. They claimed that the consequent mass production rate is 250 times greater than by a conventional electrospinning method [29]. In 2009, Shan Tang et al introduced a new needleless electrospinning apparatus applying the method of splashing polymer solution onto the surface of a metal roller spinneret [30]. In this method the setup consisted of a metal roller spinneret as the positive electrode connected to a high voltage power supply. The polymer solution droplets were splashed onto the surface of the metal roller spinneret through the holes of the solution distributor, which was located above the spinneret. The production of nanofibers was enhanced by 24–45 times compared to a single-needle system. More recently, Niu et al [31] invented a spiral coil setup and proved that this method had higher fiber production rate and better control of fiber morphology compared to disc and cylinder electrospinning.

With the above information, one can see that there have been various research and patents on the development of new electrospinning methods. However, a significant part of our information on the relationship between processing parameters and electrospun nanofiber morphology comes from literature reported on the conventional method [22, 32-35]. The aim of this study was to explore another new splashing needleless electrospinning setup to produce nanofibers and investigate the effects of processing parameters on the morphology of the produced nanofibers. In this process, a new design for supplying solution to a metal roller spinneret was used. The advantage of this setup is its ease of scaling-up.

MATERIALS

Polyoxyethylene (PEO), with an average molecular weight (Mw) of 1,000,000, was obtained from shanghai Liansheng Chemical (China), and used as received. 9wt% solution concentration was prepared by dissolving the PEO in distilled water. The dynamic viscosity was equal to 4600 mPa.s and the electrical conductivity was equal to 71.5μs/cm.

SPLASHING ELECTROSPINNING METHOD

In this method, instead of a capillary, a splashing needleless electrospinning method is used to form nanofibers. The setup consisted of a metal roller spinneret as the positive electrode connected to a high voltage power supply for generating electric field, a stationary collector, and a solution supplying system. *Figure 1* illustrates the schematic setup of the method. During electrospinning, the polymer solution droplets are splashed onto the surface of the metal roller spinneret through the holes of the solution distributor which is located above the spinneret. (*Figure 2*) shows how PEO solution from the solution distributor is feed to the surface of the metal roller spinneret. There is a height difference of 45 cm between the solution distributor and the metal roller spinneret so that a gravitational field is added to the polymer solution droplets when they are emitted from the distributor. The polymer solution droplets are pre-tensioned by their self-weight stresses. The shapes of the droplets change to oval when they hit the surface of the spinneret. This dramatically reduces the critical voltage value needed to form spinning cones. It is worth mentioning that, in previous electrospinning designs, the charged polymer solutions lead to some problems in operation, such as interferences between adjacent charged solution jets caused by the repulsion between the jets, and this may lead to reduced fiber production rate and poor fiber quality, which is the main obstacle to practical application; whereas in our setup, the splashing solution droplets are not charged before contacting the surface of the spinneret. When the voltage is applied, many polymeric jets are ejected from solution droplets adhering on the surface of the metal roller spinneret. These multi-jets undergo strong stretching, bending instability, and solvent evaporating in the electric field. The solidified nanofibers are deposited on the collector. The advantage of this setup is its potential scale-up for increased output of electrospun nanofibers by increasing the solution distributor length. Also, gravity is used to assist in conveying the solution to the electrospinning sites which simplifies the electrospinning setup.

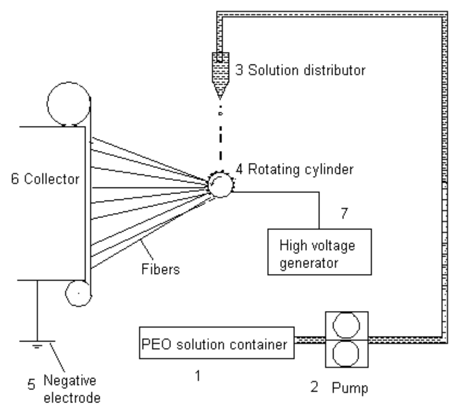

FIGURE 1. Splashing electrospinning setup.

FIGURE 2. Image of solution distributor.

The spinneret used in the present study was 30 cm long with a 5 cm diameter. All experiments were carried out at normal conditions (T=22±1ºC, RH=65%) and under normal atmospheric pressure. Spinning distance (D), which is the distance between the roller spinneret surface, and collector counter and applied voltage (V) were selected as the processing parameters. Different applied voltage values (45, 50, 60 kV) and various spinning distance values (20, 25, 30, 35, 40 cm) were used. The rotational speed of the cylinder was 4 rpm and was held constant during the electrospinning process. The flow rate was held at 0.38-0.45 milliliters per minute.

CHARACTERIZATION OF NANOFIBERS

Fiber formation and morphology of the electrospun PEO fibers were determined using a scanning electron microscope (SEM). Fiber diameters were measured with Image J digital image processing software [36] (National Institutes of Health, USA). This program measures the number of pixels and scales the length according to the calibration provided by the user. Fiber diameters were measured from the SEM images using Image J's line-drawing feature as reported in previous studies [37-39]. First the scale was set. Then, pixels between two edges of a fiber perpendicular to the fiber axis were counted. Each fiber diameter was measured at the location where the fiber was identified as a single fiber. There may be up to half a pixel error in both directions which should yield 1-pixel error in measuring fiber diameter. The number of the pixels was then converted to nanometers (nm) using the scale and the resulting diameters were recorded. The mean fiber diameter and standard deviation were also reported for analysis. Nine SEM electrospun fibers mats were analyzed. For each SEM image, 30-45 fibers were randomly chosen and measurements of their diameters were recorded.

RESULTS AND DISCUSSION

Productivity

In this work, we explored a novel needleless electrospinning apparatus to produce the nanofibers. Productivity is defined as the total mass of fibers produced per unit time and is readily measured as the rate of mass accumulated on the collector during experiment [27, 29, 39]. In this study it was observed that it was possible to increase the production rate by using this new approach, where the nanofibers are electrospun from multiple jets. For instance, for the following parameters: applied voltage value of 60 kV and spinning distance value of 30 cm; applied voltage value of 45 kV, spinning distance of 25 cm; and applied voltage value of 50 kV and spinning distance value of 20 cm, it was possible to obtain a large amount of fibers as one can see in *Figures 4(c), 2(a)* and *2(b).*

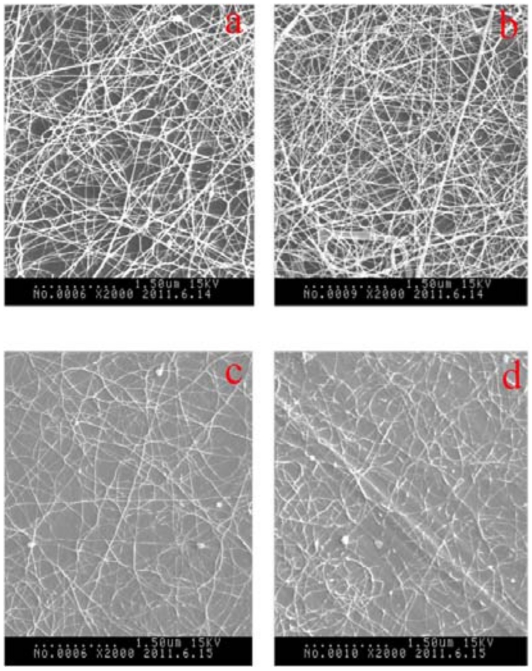

FIGURE 3. SEM (X2000) images by splashing needleless method (a): V=45kV；d=25cm; (b): V=50kV; d=20cm; (c) V=50kV; d=40cm; (d) V=50kV; d=30cm.

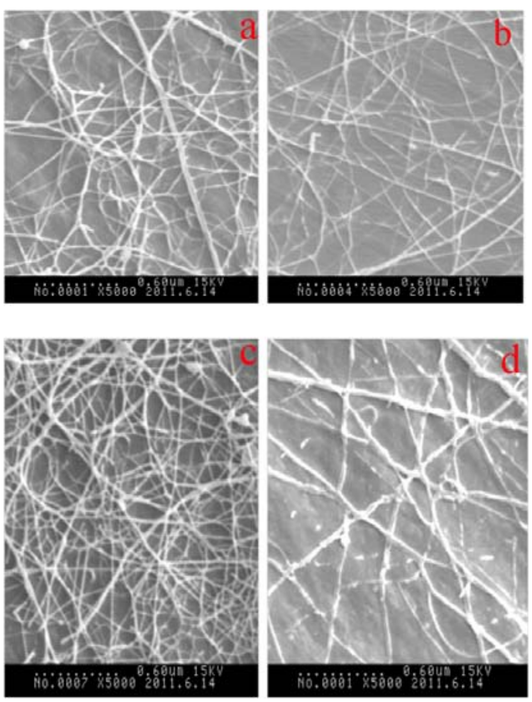

FIGURE 4. SEM (X5000) images by splashing needleless method (a): V=60kV; d=35cm; (b): V=60kV; d=20cm; (c): V=60k; d=30cm; (d): V=45kV; d=20cm.

To determine the average production rate of this approach, the mass of produced fibers was measured as the average value from all fibers produced in the experiment. The results show that this approach can produce 9–12 g/h of PEO fibers for a 30 cm long roller spinneret. In many researches, it was reported that, from a single capillary, the productivity rate range is from 0.1to1.0 g/h of the polymer solution. But sometimes it is more or less, for example 0.3-0.5 g/h in our experiment. The productivity rate of splashing needleless electrospinning has been significantly enhanced and was 24-30 times higher than from single needle electrospinning. It was also observed that the highest productivity occurred at high applied voltages.

Fiber Morphology

With approximately 30-45 fibers per SEM image, a total of 400 fiber diameters were measured. Mean fiber diameter (MFD) along with standard deviation of fiber diameter (SDFD) values were then calculated and recorded for analysis. *Table I* shows the experimental conditions and statistics of fiber diameters for each run, 196.29 to 357.68 nm, and the range of standard deviation, 33.46-105.37nm. Thinner fibers (lower MFD) were formed at applied voltage of 50kV and spinning distance of 35 cm. Uniform fibers (lower SDFD) were produced at applied voltage of 50kV, and spinning distance of fibers produced by the new method depended upon the combination of applied voltage and spinning distance. Applied voltage values of 50 kV and 60 kV combined with appropriate spinning distance values should produce thinner fibers (lower MFD), as well as uniform fibers (lower SDFD), as can be seen from *Table I* and *Figure 5*. But fiber diameters were prone to increase for applied voltages approaching 60kV. At lower applied voltages, much thicker (higher MFD values) fibers were produced. Another observation was that fibers produced by splashing needleless method presented fewer beadings, while more beadings were often observed with fibers produced by the conventional electrospinning method.

TABLE I. Experimental conditions and Statistics of nanofiber diameters (in nanometer).

Sample#	V	D	Min	Max	MFD	SDFD
1.	45	20	240	678.82	357.68	102.37
2.	45	25	178.89	486.42	303.25	75.65
3.	45	30	186.31	368.55	250.27	54.01
4.	50	20	132.58	732.20	282.39	126.68
5.	50	25	147.39	319.28	224.34	53.17
6.	50	30	129.78	316.32	196.29	41.85
7.	50	35	149.54	223.36	184.71	21.96
8.	50	40	125.90	283.28	201.23	33.46
9.	60	20	133.54	320.99	202.04	40.03
10.	60	25	220.90	369.19	280.74	49.85
11.	60	30	163.49	339.00	235.70	42.05
12.	60	35	157.38	368.41	223.58	45.37

Min: Minimum; **Max**: Maximum;

Electrospinning Processing Parameters

Using the splashing needleless set up, long spinning distances (10-40 cm) were possible, while 10-20 cm was considered as the effective range for spinning distances in the conventional method. By contrast, high applied voltages (>=45 kV) were necessary for splashing needleless method, while for the conventional method, only values between 15-25 kV are enough, reported in the literature. At applied voltage value of 60 kV and at spinning distance of 40 cm, no fibers were produced. There was a lack of solidification of jets because the liquid jets were separated into small droplets. Similar phenomenon was observed at applied voltage of 50 kV and spinning distance of 45 cm. A good balance should be maintained between the applied voltage and the spinning distance for successful electrospinning. And this balance could be dependent on the solution property, ambient conditions, and the geometry of spinneret. This indicates that for a type of polymer and a given solution concentration, there is a range of processing parameters for which the solution is spinnable as observed in electrospinning methods [32-35].

Effects of Processing Parameters on Fiber Morphology

To clearly understand the impact of processing parameters on the electrospun nanofiber morphology produced by the splashing needleless method, the processing parameters were investigated.

The effect of the applied voltage (V) on MFD is illustrated in *Figures 5(a)* .MFD decreased as well as increased with the increase of applied voltage while spinning distance was held constant (D=30 cm).

The effect of the applied voltage (V) on SDFD is illustrated in *Figures 5(c)*. SDFD decreased as well as increased with the increase of applied voltage while spinning distance was held constant (D=30 cm). When applied voltage is low, the electric field strength (E) is low resulting in less stretching of the jets before they are deposited on the collector. With higher voltage, more solutions will be drawn from the solution droplets. Therefore, the initial jets become thick favoring the thicker fibers formation. Applied voltage affects both the polymer mass and jet elongation. In fact, with higher voltages, more solutions will be drawn from the solution droplets, and the polymer mass is the dominant factor. So the mass flow rate from the surface to the collector will increase and the solution will be drawn quickly from the spinneret favoring the formation of thicker fibers. But when the applied voltage exceeds a certain limit, jet elongation becomes the dominant factor. In this case the electric field strength (E) will be high enough (E=V/D) resulting in strong stretching of the jets. By increasing the spinning distance, the electric field decreases and the final resulting fiber morphology will be determined by the combination effects of mass flow and electric field.

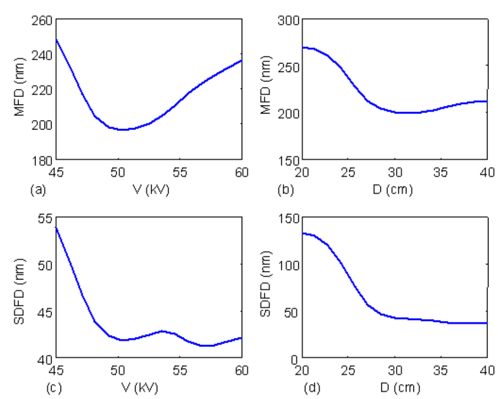

FIGURE 5. (a): Impact of applied voltage on MFD while spinning distance is held constant (D= 30 cm); (b): Impact of spinning distance on MFD while applied voltage is held constant (V=50 kV); (c) Impact of applied voltage on SDFD while spinning distance is held constant (D=30 cm); and (d): Impact of spinning distance on SDFD while applied voltage is held constant at (V=50 kV).

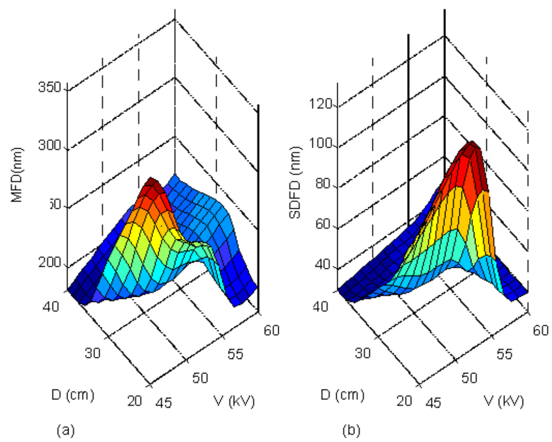

FIGURE 6. Combined effects of electrospinning distance and applied voltage on (a): MFD; (b) SDFD.

The effect of spinning distance (D) on electrospun fiber morphology is depicted in *Figures 5(b)* and *5(d).* This analysis shows that both MFD and SDFD decreased with the increase of spinning distance, while applied voltage was held constant (V=50 kV). At short spinning distances, the solvent will not have enough time to evaporate before jets are deposited on the collector resulting in thicker fibers formation. Varying the spinning distance affects both the jet flight time and the electric field strength. On one hand, longer spinning distance will provide more time for the jets to stretch in the electric field before they are deposited on the collector, and solvent will have enough time to evaporate. Therefore the fiber diameter will be prone to decrease. On the other hand, by increasing the spinning distance, the electric field strength will decrease (E=V/D) resulting in less stretching forces which leads to thicker fibers formation. The final fiber diameter will be determined by the balance between the two effects.

Figure 6 shows the combined effects of simultaneous variations in applied voltage and spinning distance on both MFD *(Figure 6(a))* and SDFD *Figure 6(b)).* The surfaces representing in a compact way all the information in the parameters controller were plotted. The nonlinear relationship existing between processing parameters and fiber diameter are illustrated. This nonlinearity, sometimes called the “control surface,” is affected by the entire main controller parameters. Two combined parameter based investigations can help to provide insight into how to control and improve the design of the electrospinning process. The curvatures of the response surfaces also reveal that there were interactions between the two parameters. It was observed that the fiber diameter and its distribution depend on the combination of processing parameters instead of each parameter separately.

CONCLUSION

In this study, needleless splashing electrospinning method was used to produce nanofiber webs. The processing ability of this method was investigated and compared to that of the conventional method. It was observed that it is possible to increase the production rate of nanofibers by this novel electrospinning method, where the nanofibers are electrospun from multiple jets. The productivity rate of splashing needleless electrospinning has been enhanced by 24-30 times higher than that of single needle electrospinning. Also the fibers produced by splashing needleless method present fewer beadings than the conventional method. The effects of two processing parameters, applied voltage and electrospinning spinning distance, on nanofiber morphology were investigated. Electrospinning distance and applied voltage influence the nanofiber diameters, as reported by some previous studies. It was also observed that for a type of polymer and at a given solution concentration, there is a range of processing parameters for which the solution is spinnable, as has been observed other electrospinning methods. It was found that there is a huge interaction between the two parameters. Extensive experiments have to be done and the results have to be compared to that of other electrospinning methods, in order to assess the acceptable relationship between electrospun fiber morphology and processing parameters.

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