EXPERIMENTAL INVESTIGATION OF SALT EFFECT ON ELECTROSPINNING PARAMETERS AND NANOFIBER MORPHOLOGY

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Abstract

Dependent parameters such as throughput, throughput/jet, current, current/jet, number of jets, spinning area on the roller and distance between jets were studied for selected polymer solution forming high quality nanofibers via needleless electrospinning. The density of fiber-forming jets is controlled by conductivity of polymer solution. Conductivity affects the fiber morphology and diameter. The objective of research was to obtain finer and non-fibrous fiber morphologies with appropriate productivity, via controllable solution property for further applications.

Keywords: Needleless electrospinning, PEO, throughput, current

1. INTRODUCTION

Nanofibers gain big interest last dedications due to their unique and specific properties. The aimed application area of electrospun fibers include tissue engineering scaffolds, wound dressing, blood vessels, drug delivery and release control, filters, catalyst and enzyme carriers, sensors, etc. Electrospinning is one of the most popular methods to produce nanofibers for forming fibers with submicron-scale diameters through the action of electrostatic forces. Interest in the electrospinning process has increased in recent years. The process of the basic needle electrospinning is well described in many papers. Various production methods have been developed recently for higher productivity. Lin et al. divided these methods in two groups such as downward multi-jet electrospinning and upward needleless electrospinning. In this work an upward needleless electrospinning is used. This technology is known as roller electrospinning system with the trade name Nanospider. The basic elements of electrospinning are simple to implement and explained in previous study. Roller electrospinning system is suitable for production in industrial scale.

As a consequence of both commercial and scientific interest in electrospinning and nanofibers, much effort has been made to understand and control the process and system parameters. The purpose of the current work is to look systematically at the effects of process parameters on the structure and morphology of electrospun polyethylene oxide (PEO) fibers. It was found that significant changes in fiber diameter, size distribution, morphology and productivity accompany changes in these variables.

2. MATERIAL AND METHODS

PEO was purchased from Sigma Aldrich. The weight average molecule weight (Mw) of PEO was 400.000. This material was dissolved in distilled water. 5% wt. of PEO solutions was prepared in various content of lithium chloride (LiCl) salts. LiCl salts were prepared according to molar ratio as 0, 0.024 and 0.062 and 0.124 mol/L. Conductivity (Radelkis OK-102/1), viscosity (HaakeRotoVisco 1 at 23 C°), surface tension
(Krüss K9) tests were done. Polymer solution was spun by using roller electrospinning system as nominated in Fig. 1.

Spinning conditions of PEO with and without salt content are shown in Table 1.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Distance between electrodes</th>
<th>Applied voltage</th>
<th>Rotating roller speed</th>
<th>Roller diameter</th>
<th>Temperature</th>
<th>Humidity</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>150 mm</td>
<td>42 kV</td>
<td>1.5 rpm</td>
<td>20 mm</td>
<td>25±1 ºC</td>
<td>25±1 %</td>
</tr>
<tr>
<td>0.024</td>
<td>150mm</td>
<td>42 kV</td>
<td>1.5 rpm</td>
<td>20 mm</td>
<td>25±1 ºC</td>
<td>25±1 %</td>
</tr>
<tr>
<td>0.062</td>
<td>150mm</td>
<td>42 kV</td>
<td>1.5 rpm</td>
<td>20 mm</td>
<td>25±1 ºC</td>
<td>25±1 %</td>
</tr>
<tr>
<td>0.124</td>
<td>150mm</td>
<td>42 kV</td>
<td>1.5 rpm</td>
<td>20 mm</td>
<td>25±1 ºC</td>
<td>25±1 %</td>
</tr>
</tbody>
</table>

The polymer throughput of the roller electrospinning process was calculated from the area weight and width of the nanofiber layer and from the velocity of the backing material, using formula \(^{16}\). The density of jets was calculated by using digital camera (Sony Full HD NEX-VG10E Handy cam). Distance between jets was calculated according to formula \(^{17}\). The voltage is measured by a 33401 A digital multimeter produced by Agilent and stored on a computer and has a resistance \(R\) is equal to 9811\(\Omega\). Current was calculated by using Ohm’s law.

### 3. RESULTS AND DISCUSSION

PEO solutions were electrospun in order to investigate the effects of process parameters of electrospinning. It was found that solution properties are the main factors influencing the diameter and spinning performance\(^{14,16}\). Surface tension, conductivity and viscosity of solution are tabulated in Table 2. Table 2 shows that adding salt influence only conductivity of solution. In this work the only solution parameter which has effective role on spinning result is conductivity.

#### Table 2. Solution properties of PEO with and without salt.

<table>
<thead>
<tr>
<th>5%PEO+salt</th>
<th>Viscosity (Pa.s)</th>
<th>Conductivity (mS/cm)</th>
<th>Surface tension (mN/m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.167±0.1</td>
<td>0.158±0.1</td>
<td>59.2±2</td>
</tr>
<tr>
<td>0.024</td>
<td>0.184±0.1</td>
<td>3.027±0.1</td>
<td>60.8±2</td>
</tr>
<tr>
<td>0.062</td>
<td>0.155±0.1</td>
<td>5.928±0.1</td>
<td>62.2±2</td>
</tr>
<tr>
<td>0.124</td>
<td>0.162±0.1</td>
<td>10.944±0.1</td>
<td>61.5±2</td>
</tr>
</tbody>
</table>
Fig. 2 shows that spinning performance is decreasing with content of salts. Number of jets shows the same tendency as spinning performance. It means that number of jets decrease and it effects the productivity. This case can be explained by the leaky dielectric model which was first proposed by Melcher and Taylor. It is important to know that if the changes in conductivity resulting from salt additions are large enough to alter the behavior of the fluid from that of a leaky dielectric to that resembling a conductor, then the tangential component of the electrical stress that accelerates the fluid is likely to diminish and the flow process be stopped. In this limit the electrical stress will be balanced by the alteration of the shape of the interface and surface tension only. Herein, we can assume that high conductivity of PEO solutions shows a behavior as conductor under the electrical field. The other process parameters such as current, current/jet, spinning area, distance between jets and spinning performance/jet is tabulated in Table 3.

Table 3. Effect of salt on dependent parameters of PEO electrospun fibers.

<table>
<thead>
<tr>
<th>5%PEO + salt</th>
<th>Number of jets</th>
<th>Current (µA)</th>
<th>Current/jet (µA)</th>
<th>Spinning Area x 10² (m²)</th>
<th>Distance between jets x 10³ (m)</th>
<th>Spinning performance/jet (g/h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>105</td>
<td>48</td>
<td>0.457</td>
<td>1.177</td>
<td>3.348304</td>
<td>0.2245</td>
</tr>
<tr>
<td>0.024</td>
<td>75</td>
<td>110</td>
<td>1.467</td>
<td>1.177</td>
<td>3.961766</td>
<td>0.0508</td>
</tr>
<tr>
<td>0.062</td>
<td>46</td>
<td>120</td>
<td>2.609</td>
<td>1.121</td>
<td>4.936804</td>
<td>0.0466</td>
</tr>
<tr>
<td>0.124</td>
<td>45</td>
<td>160</td>
<td>3.556</td>
<td>1.177</td>
<td>5.114618</td>
<td>0.0475</td>
</tr>
</tbody>
</table>

Current per one jet increased with salt content. It shows number of ions which are transported with jet increases due to increase in conductivity. As a result total current increase. On the other hand spinning area did not change with salt content, distance between jets increase. It could be because of highly charged jet repulse each other and distance between jets increases. Spinning performance per one jet for 5%PEO without any additive is extremely high. PEO without any salt has very high spinning performance and number of jets. However, only polymer solution is transported to collector with forming few fibers. 5% PEO (without any salt) electrospun web shows a surface with non-fibrous area around 90%. Non-fibrous area means there is no fiber only polymer droplets are forming. Adding salt helps to create thinner jet and highly splitting jet. As a result fibers are forming by adding salt and non-fibrous area is eliminated. The fibers and diameter distribution is shown in Fig. 3. In case of 5% PEO without any additives, samples were prepared from non-fibrous area to compare diameter of fibers with salt solution.
This work shows that adding salt has a positive effect to produce thin nanofibers and better surface of web. On the other hand, salt content decreases spinning performance.
CONCLUSION

Ultrafine PEO fibers without non-fibrous area were obtained from electrospinning. Above a certain content of salt, fibers without non-fibrous area are electrospun and thinner fibers are obtained from a polymer solution with higher net charge density. Increase conductivity favors the formation of fibers without non-fibrous area. At high content of salt as Fig.3 (d), diameter of fibers increase and productivity decrease. It is necessary to arrange content of salt in a desired amount to control fiber diameter, non-fibrous area and spinning performance. This work shows that even at very low content of salt (0.024M ≃ 0.1% wt.) shows low fiber diameter and good web surface. The content of LiCl salt can be adjustable according to desired properties web.

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REFERENCES


