

## The Influence of Main Technological Parameters on the Diameter of Poly(vinyl alcohol) (PVA) Nanofibre and Morphology of Manufactured Mat

Erika ADOMAVIČIŪTĖ<sup>1\*</sup>, Rimvydas MILAŠIUS<sup>1</sup>, Rimantas LEVINSKAS<sup>2</sup>

<sup>1</sup>Department of Textile Technology, Kaunas University of Technology, Studentų 56, LT-51424 Kaunas, Lithuania

<sup>2</sup>Lithuania Energy Institute, Breslaujos 3, LT-44403 Kaunas, Lithuania

Received 06 December 2006; accepted 06 May 2007

An electrospinning method was used to prepare poly(vinyl alcohol) (PVA) nanofibre from 12 % concentration of PVA solution. The influence of applied voltage and distance between electrodes on diameter of nanofibre and morphology of the manufactured mat was investigated. These characteristics were observed by scanning electron microscope (SEM). It was concluded that PVA nanofibre diameter tended to increase with increasing applied voltage and to decrease with increasing the distance between the electrodes. Morphology of the mat from PVA nanofibre was strongly affected by technological parameters, too.

*Keywords:* nanofibre, electrospinning.

### INTRODUCTION

Electrospinning is a process that employs electrostatic forces to produce nanofibres with diameter between 50 nm – 1000 nm from polymer solution or melt.

In traditional electrospinning equipment one electrode is placed into the spinning solution/melt and the other one is attached to the collector. The electric field is subjected to the end on the capillary tube that contains the solution fluid held by its surface tension. Thus a charge on the surface of the liquid is induced. As the intensity of electric field is increased, the hemispherical surface of the fluid at the tip of capillary tube elongates to form a conical shape known as the Taylor cone. As the repulsive electrostatic force overcomes the surface tension and the charged jet of the fluid is ejected from the tip of the Taylor cone [1]. As the jet moves toward a collecting screen, it elongates, solvent evaporates and a nonwoven fabric is formed on the collector [1, 2].

Electrospun mats from polymer fibres are drawing a great attention because of their unique properties such as high surface-to-volume ratio, high porosity, diameters in the nanoscale, flexibility in surface functionalities and superior mechanical performance [1, 3 – 5]. Many parameters influence the transformation of polymer solution into nanofibres: viscosity (concentration), conductivity, elasticity, temperature, surface tension of the solution, polymer molecular weight, applied voltage, distance between electrodes, humidity and air velocity in the electrospinning chamber [1, 6 – 16].

There is not common accordance about the influence of the main technological parameters, i. e. applied voltage and the distance between electrodes, on the diameter and morphology of nanofibres. S. Y. Gu et al. [6] reported that diameter of the electrospun PAN nanofibre did not change significantly over the range of applied voltage. M. M. Demir et al. [7] estimated that PU nanofibre diameter increased with increase of electrospinning

voltage. By contrary, X. M. Mo et al. [17] reported that P(LLA-CL) nanofiber diameter tended to decrease with increasing electrospinning voltage. J. M. Deitzel and et al. [8] analyzed the influence of applied voltage on morphology PEO nanofibre and estimated that with increasing applied voltage, the number of beads on fibre increased. Contrary assumptions were made analyzing PS nanofibre morphology [9].

The jet of polymer solutions grows longer and thinner due to bending instability until it solidifies and collects on the collector, i. e. if the distance between electrodes is short, electrospun fibres should be thicker. But according to some researchers [10], the distance does not have significant effect on the electrospun fibre geometrical characteristics.

It may be that the difference of used polymers, experimental conditions and equipments had influence on such variety of the results.

The aim of this paper is to define the influence of applied voltage ( $U$ , kV) and distance between the electrodes ( $L$ , cm) on the average of PVA nanofibre diameter and morphology of manufactured mat, spun on a “Nanospider” equipment.

### MATERIALS AND METHODS

A water solution of poly(vinyl alcohol) (PVA) was diluted using distilled water in order to get 12 % concentration of solution. To improve the conductivity of diluted polymer solution, 0.5 % of phosphoric acid ( $H_3PO_4$ ) was added.

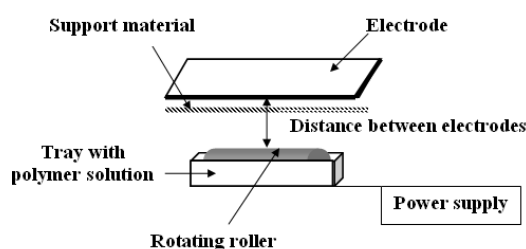
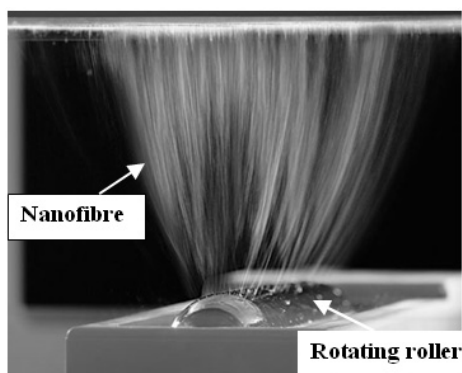


Fig. 1. The scheme of “Nanospider” spinning chamber

\*Corresponding author. Tel.: +370-655-41805; fax: +370-37-353989. E-mail address: erika.adomaviciute@stud.ktu.lt (E. Adomavičiūtė)



**Fig. 2.** Formation of nanofibre

Nanofibre of PVA solution was spun using “Nanospider” (Elmarco, Czech Republic). This new method of nanofibres production has been developed at the Technical University of Liberec and patented [18]. It differs from the traditional electrospinning techniques that the spinning head is a rotating roller (Fig. 1).

The rotating roller is sunk halfway in the polymer solution. Increasing the applied voltage, the Taylor cones (initiations of nanofibres) are created from polymer solution on the roller. In this way the support material is covered by the layer of nanofibre (Fig. 2).

The parameters of experiments are presented in Table 1.

**Table 1.** Parameters of the experiments

Number of experiments	The distance between electrodes $L$ , cm	Applied voltage $U$ , kV
1	11	45
2	11	50
3	11	55
4	11	60
5	11	65
6	11	70
7	11	75
8	12	65
9	13	55
10	13	65
11	13	75
12	14	65
13	15	55
14	15	65
15	15	75
16	16	65
17	17	75

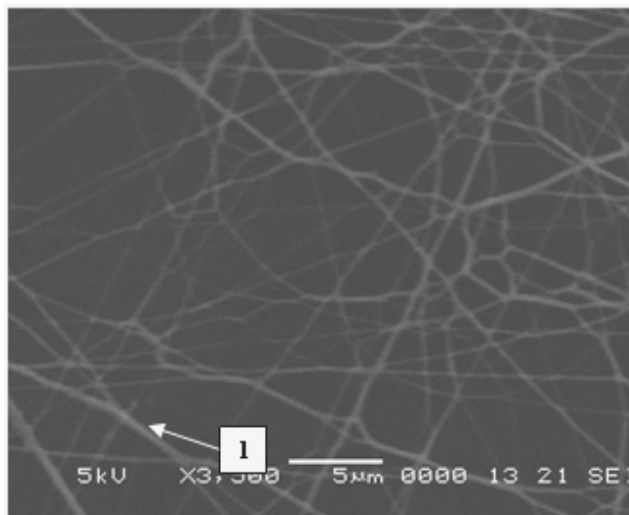
The morphology of electrospun PVA mats was observed with a scanning electron microscope (SEM) JOEL JSM 5600. The diameter of nanofibre was measured by LUCIA G software from SEM images. All nanofibres: single and stick were measured from every SEM image.

## RESULTS AND DISCUSSION

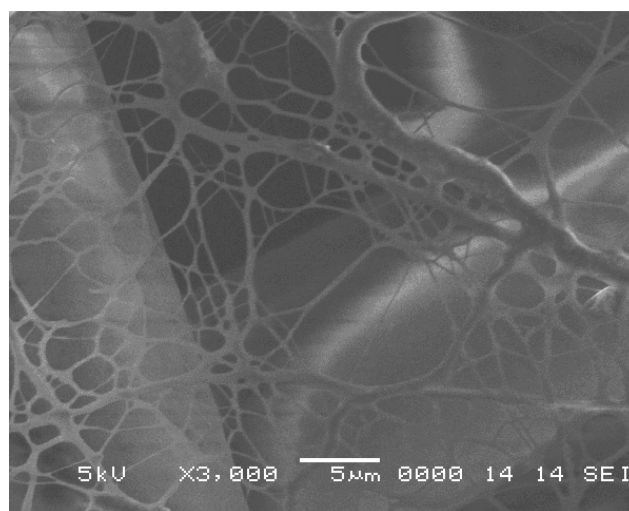
**The influence of applied voltage.** The main series of experiments were carried out at the distance between the

electrodes 11 cm while the applied voltage was varied from 45 kV to 75 kV. Also experiments were carried out at the distances – 13 and 15 cm and applied voltages – 55, 65, 75 kV.

45 kV is the lowest voltage value by which it was possible to create nanofibre from 12 % concentration of PVA solution, when the distance between electrodes is 11 cm. 75 kV is the highest voltage, which was possible to set up by the “Nanospider”.



a



b

**Fig. 3.** SEM images of mat from PVA nanofibre, electrospun at the distance between electrodes 11 cm; the applied voltage: a – 45 kV; b – 50 kV

As nanofibre moves from roller to support material, the solvent (distilled water) evaporates and nanofibre solidifies. If the distance is short and applied voltage is very high, nanofibres tend to stick to each other due to incomplete solvent evaporation (Fig. 3, a, number 1 indicates the stick nanofibre).

Analyzing the morphology of manufactured mats, three types of mats were determined: mat from uniform nanofibres; stick nanofibres (i.e. mat from stick and uniform nanofibres) and “spidery” mat (only from stick nanofibres). From SEM images presented in Figure 3, we can see that support material (spunbond of PES filaments) is covered by mat from uniform PVA nanofibre (Fig. 3, a),

while applied voltage is 45 kV. But 50 kV voltage is too high, at the distance between electrodes 11 cm. In this case when all nanofibres are stuck, the “spidery” mat is formed (Fig. 3, b). Further increasing voltage to 75 kV, the morphology of mats are similar as is presented in Figure 3, b.

Support material is covered by mat from uniform nanofibre (SEM image is similar as in Fig. 3, a), while the voltage is 55 kV and the distance 15 cm. In order to create mat from uniform nanofibre, increasing the distance between the electrodes, applied voltage should be increased too. It is possible to suppose that mat from uniform PVA nanofibre will be at the distance 13 cm, while applied voltage 50 kV. Mats from stick nanofibres are created at the distance 13 cm, while applied voltages 55, 65 kV and at the distance 15 cm, voltage – 65 kV (Fig. 5, b). “Spidery” mats (similar to Fig. 3, b) are formed at the distance 13 cm, while applied voltages 65, 75 kV and at the distance 15 cm, voltage – 75 kV.

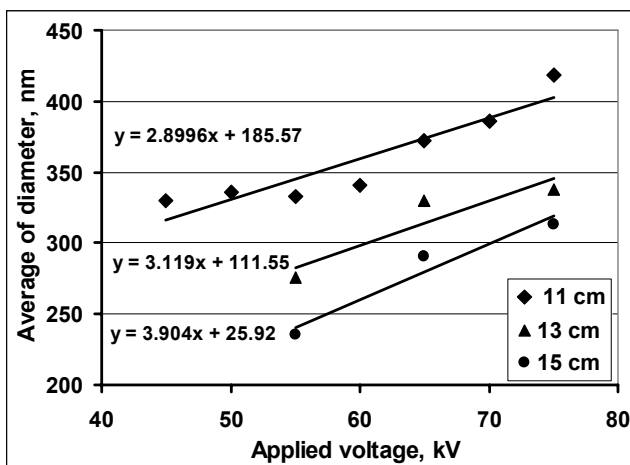


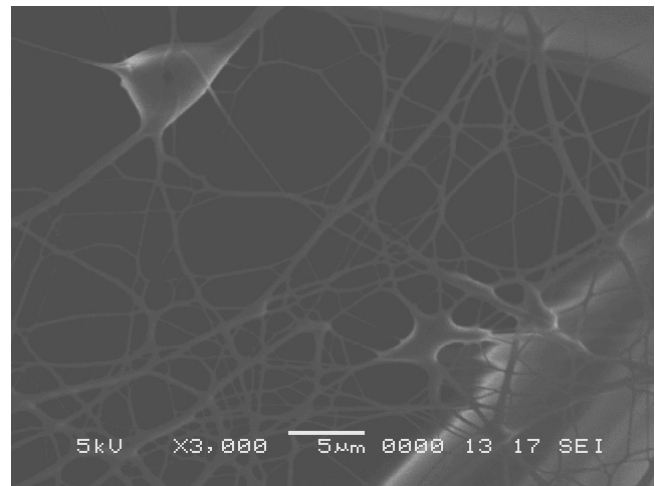
Fig. 4. Dependence of the average of PVA nanofibre diameter  $d$  upon the applied voltage  $U$ , at distances between electrodes  $L$  are 11 cm, 13 cm and 15 cm

The dependence of the average of PVA nanofibre diameter  $d$  (nm) upon the applied voltage  $U$  (kV) is presented in Figure 4. The average of PVA nanofibre diameter 25 % increases (average of relative error  $\bar{\delta} = 12\%$  at confidence level  $t_{\alpha} = 95\%$ ) when the distance between electrodes is 11 cm. When distances are 13 cm and 15 cm, increasing applied voltage from 55 kV to 75 kV (Fig. 4) causes the increase in the average of nanofibre diameter too, respectively 18 % ( $\bar{\delta} = 10\%$ ) and 25 % ( $\bar{\delta} = 8\%$ ).

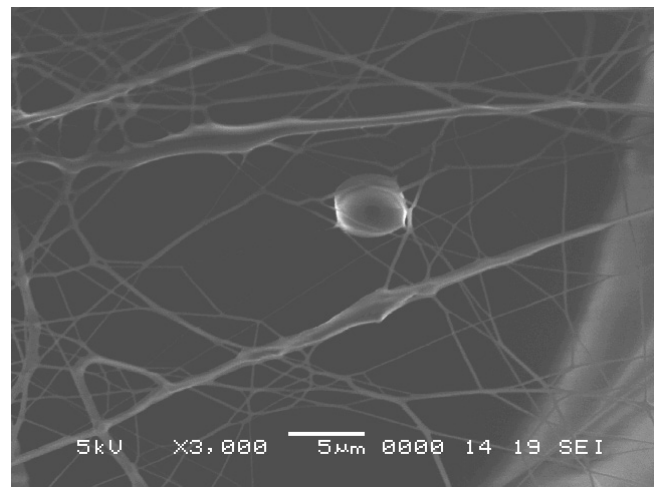
Increasing applied voltage, the Taylor cone (initiator of nanofibre) is formed from more amount of polymer solution, i.e., is formed thicker nanofibre. Also there are a lot of stick nanofibres on the support material, when the distance between electrodes is small and the applied voltage is high. These reasons influence on the increase of PVA nanofibre diameter.

**The influence of the distance between electrodes.** In this case the main part of experiments was carried out when applied voltage was 65 kV and the distance between electrodes varied from 11 cm to 17 cm.

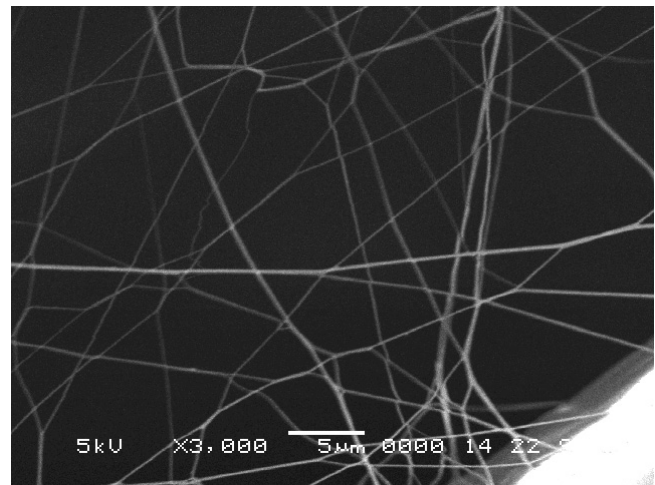
Some SEM images, which present the effect of the distance between the electrodes on morphology of PVA nanofibre mats, at voltage 65 kV, are presented in Figure 5.



a



b



c

Fig. 5. SEM images of mat of PVA nanofibre electrospun while applied voltage 65 kV, the distance: a – 12 cm; b – 15 cm; c – 17 cm

“Spidery” mats from PVA nanofibre are formed when the distances are 12 cm (Fig. 5, a). Support material is covered by the layer of stick nanofibres at distances 13 cm – 16 cm (Fig. 5, b). In all these cases there are many stick nanofibres due to incomplete solvent evaporation, because distances are too short for 65 kV voltages. Uniform mat

from PVA nanofibre is formed only at distance 17 cm (Fig. 5, c).

The dependence of the average of PVA nanofibre diameter upon the distance between electrodes is presented in Figure 6.

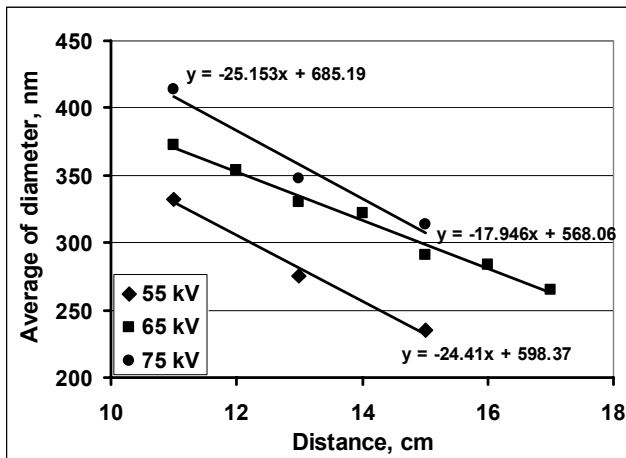


Fig. 6. Dependence of the diameter  $d$  of PVA nanofibre upon the distance  $L$  between electrode, at applied voltages  $U$  55 kV, 65 kV, 75 kV

Increasing the distance between electrodes, at voltage 65 kV (Fig. 6), the diameter of PVA nanofibre decreases 40 % ( $\bar{\delta} = 11$  %). In those cases as applied voltages are 55 kV and 75 kV, the average of nanofibre diameter respectively decrease 42 % ( $\bar{\delta} = 9$  %) and 32 % ( $\bar{\delta} = 12$  %).

As it was mentioned above, when a nanofibre moves from roller to support material, it elongates and solidifies. Therefore increasing the distance between electrodes, the average of PVA nanofibre decreases and nanofibres do not stick due to complete solvent evaporation (when voltages are 45, 55 and 75 kV and the distances respectively 11 cm (Fig. 3, a), 15 cm and 17 cm (Fig. 5, c)).

## CONCLUSIONS

The influence of applied voltage and the distance between electrodes on the diameter of PVA nanofibre and morphology of mat was studied. It was found, that the average of nanofibre diameter increases, increasing applied voltage and decreases, increasing the distance between the electrodes. Varying the main technological parameters, the morphology of PVA nanofibres mat is changing too. To cover support material by a mat from uniform nanofibre with increase of the distance between electrodes, applied voltage should be increased too.

## REFERENCES

- Huang, Z.-M., Zhang, Y.-Z., Kotaki, M., Ramakrishna, S. A Review on Polymer Nanofibres by Electrospinning and Their Applications in Nanocomposites *Composite Science and Technology* 63 2003: pp. 2223–2253.
- Zeng, J., Xu, X., Chen, X., Liang, Q., Bian, X., Yang, L., Jing, X. Biodegradable Electrospun Fibers for Drug Delivery *Journal of Controlled Release* 92 2003: pp. 227–231.
- Tomaszewski, W., Szadkowski, M. Investigation of Electrospinning with the Use of a Multi-jet Electrospinning Head *Fibre & Textile in Eastern Europe* 13 (4) 2005: pp. 22–26.
- Chen, C., Chuanbao, C., Xilan, M., Yin, T., Hesun, Z. Preparation of Non-woven Mats from All-Aqueous Silk Fibroin Solution with Electrospinning Method *Polymer* 47 2006: pp. 6322–6327.
- Min, B.-M., Lee, S. W., Lim, J. N., You, Y., Lee, T. S., Kang, P. H., Park, W. H. Chitin and Chitosan Nanofibres: Electrospinning of Chitin and Deacetylation of Chitin Nanofibres *Polymer* 45 2004: pp. 7137–7142.
- Gu, S. Y., Ren, J., Vancso, G. J. Process Optimization and Empirical Modeling for Electrospun Polyacrylonitrile (PAN) Nanofiber Precursor of Carbon Nanofibres *European Polymer Journal* 41 2005: pp. 2559–2569.
- Demir, M. M., Yilgor, I., Yilgor, E., Erman, B. Electrospinning of Polyurethane Fibers *Polymer* 43 2002: pp. 3303–3309.
- Deitzel, J. M., Kleinmeyer, J., Harris, D., Beck Tan, N. C. The Effect of Processing Variables on the Morphology of Electrospun Nanofibres and Textiles *Polymer* 42 2002: pp. 261–272.
- Jarusuwannapoom, T., Hongrojjanawiwat, W., Jitjaicham, S., Wannatong, L., Nithitanakul, M., Pattamaprom, C., Koombhongse, P., Rangkupan, R., Supaphol, P. Effect of Solvents on Electro-spinnability of Polystyrene Solutions and Morphological Appearance of Resulting Electrospun Polystyrene Fibers *European Polymer Journal* 41 2005: pp. 409–421.
- Zhang, C., Yuan, X., Wu, L., Han, Y., Sheng, J. Study of Morphology of Electrospun Poly(vinyl alcohol) Mats *European Polymer Journal* 41 2005: pp. 423–432.
- Theron, S. A., Zussman, E., Yarin, A. L. Experimental Investigation of the Governing Parameters in the Electrospinning of Polymer Solutions *Polymer* 45 2004: pp. 2017–2030.
- Jia, Y.-T., Gong, J., Gu, X.-H., Kim, H.-Y., Dong, J., Shen, X.-Y. Fabrication and Characterization of Poly(vinyl alcohol)/ Chitosan Blend Nanofibres Produced by Electrospinning Method *Carbohydrate Polymers* 2006.
- Yu, J. H., Fridrikh, S. V., Rutledge, G. C. The Role of Elasticity in the Formation of Electrospun Fibers *Polymer* 47 2006: pp. 4789–4797.
- Son, W. K., Youk, J. H., Lee, T. S., Park, W. H. Effect of pH on Electrospinning of Poly(vinyl alcohol) *Materials Letters* 59 2005: pp. 1571–1575.
- Lee, K. H., Kim, H. Y., Bang, H. J., Jung, Y. H., Lee, S. G. The Change of Bead Morphology Formed on Electrospun Polystyrene Fibers *Polymer* 44 2003: pp. 4029–4034.
- Han, S. O., Son, W. K., Youk, J. H., Lee, T. S., Park, W. H. Ultrafine Porous Fibers Electrospun from Cellulose Triacetate *Materials Letters* 59 2005: pp. 2998–3001.
- Mo, X. M., Xu, C. Y., Kotaki, M., Ramakrishna, S. Electrospun P(LLA-CL) Nanofiber: a Biomimetic Extracellular Matrix for Smooth Muscle Cell and Endothelial Cell Proliferation *Biomaterials* 25 2004: pp. 1883–1890.
- Jirsak, O. CZ Patent, 2003-2414 (294274).

