

Funda Cengiz,
*Izabella Krucińska,
*Eulalia Gliścińska,
*Michał Chrzanowski,
Fatma Göktepe

Suleyman Demirel University,
Engineering & Architecture Faculty,
Department of Textile Engineering
Cunur, 32260, Isparta, Turkey

*Technical University of Łódź,
Department of Fibre Physics
and Textile Metrology
ul. Żeromskiego 116, 90-924, Łódź, Poland
E-mail: klata@p.lodz.pl

Comparative Analysis of Various Electrospinning Methods of Nanofibre Formation

Abstract

In general, nanofibres can be formed using the electrospinning technique, which has been known for a long time in literature. However, there are still some problems related to this technique such as low production rate, capillary clogging, etc. In recent years, several new electrospinning methods have been developed to solve such problems. The main aim of this paper is to present the possibility of nanofibre formation by three various electrospinning methods. In this study nanofibres were produced from PAN/DMSO solution using three different electrospinning methods: a conventional method with one capillary, Jirsak's method and Yarin & Zussman's method. The properties of these nanofibres as well as the processing conditions were compared. It was observed that using the last two methods it is possible to increase the production rate because of the multiple jets. In the conventional method a single capillary should be replaced by a spinning head system with many capillaries. Using Jirsak's method, the nanofibre diameter decreases and a higher amount of fibre is obtained by increasing the rotary speed of the cylinder. Production at 10 wt % polymer concentration by Yarin & Zussman's method leads to much finer fibres than by other methods.

Key words: electrospinning, methods, nanofibres, polyacrylonitrile.

tion applications as their small diameter offers advantages for filtration and composite materials. Nanofibre webs with different filtration characteristics can be produced by controlling electrospinning parameters [15].

Generally, polymeric nanofibres are produced by electrospinning methods. Electrospinning is a technique that it is able to produce ultra-thin fibres ranging from tens to several hundred nanometers. This method has been known for a long time, but academic research and publications have only recently been on the increase. Formhals first invented an electrospinning method for polymer solution in the 1930's. He published a series of patents describing an experimental set up for the production of nanofibres using an electrostatic force [16]. After Formhals's studies, various research studies and patents were recorded about an electrospinning method with a single capillary. However, there are still some crucial problems such as low production rate, theoretical understanding of jet thinning, capillary clogging and solvent evaporation etc. To solve these problems, several new methods have been developed in recent years.

A high productivity capillaryless method was developed by Jirsak in 2003 [17]. In this method a rotating cylinder was used to obtain nanofibres and an increase in productivity was claimed. In the same year Kameoka and Craighead presented a method for the formation of oriented polymeric nanofibres using electrospinning deposition from an integrated microflu-

idic device. They studied the effect of the rotational speed of a counter electrode on the morphology of nanofibres and found that nanofibres were straightened by increasing the linear velocity during which the diameter of nanofibres does not significantly depend on the rotational speed [18]. In 2003 Lee et al. also patented a high speed apparatus for polymer web production by electrospinning [19].

In 2004, Chu et al. patented a multiple jet system allowing high production rates for a commercial process [8]. In this year, He et al. applied vibration technology to electrospinning for the first time. This system is of critical importance for a new generation of electrospinning apparatus. Using this technology it was possible to produce much finer nanofibres compared those manufactured by the conventional electrospinning method. The authors were able to produce finer nanofibres at a lower applied voltage and observed an increase in fibre strength by decreasing the applied voltage [20]. In 2005, Tomaszewski and Szadkowski constructed three types of multi-jet electrospinning heads (series, elliptic and concentric) for the electrospinning method. The head effectiveness was tested by using poly(vinyl alcohol)/water solution as the spinning liquid. The concentric electrospinning head was selected as the best type with respect to both the efficiency and quality of the process. It was able to produce 1 mg of dry PVA nanofibres from one spinning pipe during one minute. This head was examined for a longer duration of 4.5 hours [21].

Introduction

In fibre science literature, nanofibre is termed as a fibre with a diameter less than one micron [1]. In recent years, nanofibre production has become very important for scientists and companies because of its superior properties such as very small diameter, high surface area, small pore size, etc. Due to its small diameter, it has an extremely high ratio of surface area to weight compared to conventional fibres. It has many important application areas such as filtration [1 - 6], membrane [7 - 8], composites [9 - 11], wound dressing [12 - 13], and biomedical devices [14]. For instance, small pore sizes of nanofibres make them suitable especially for military and civilian filtra-

Another new capillaryless approach was proposed by Yarin and Zussman 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. As a result, vertical spikes of magnetic suspension perturbed the interlayer interface. The perturbations of the free surface became sites of jetting directed upward with the effect of an electric field. Therefore, a high production rate of multiple nanofibre electrospinning could be achieved without any capillaries [22].

More recently, 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 [23].

In recent years, the most important problem in electrospinning is the low production rate. Generally, it has been observed that it is very difficult to increase the process rate by the conventional method with a single capillary, while it is possible by Jirsak's and Yarin & Zussman's methods, where multiple polymer jets can be utilised. Usually, from a single capillary the productivity range is from 0.1 to 1.0 g/h of the polymer solution, but sometimes it is more or less, for example 0.02 g/h for 20 % solution of PA 6. In Jirsak's method the productivity is much higher and averages 1.5 g/min per 1 m of a rotating cylinder of 30 mm in diameter, and 1.8 g/min per 1 m of a cylinder of 50 mm in diameter, [23]. Using the Yarin and Zussman method, the productivity rate from the solution surface is 12 times higher than from the capillary arrangement.

As can be seen, there have been various researches and patents about the development of new electrospinning methods, although they have not been compared in terms of fibre properties and process parameters. For that reason, in this research work three different methods were explored and compared, i.e. their spinning possibilities depend on technological parameters, their productivity, and the influence of process parameters on fibre properties.

Experimental

Material

Polyacrylonitrile (PAN) powder including 5 % comonomers of acrylic acid and methyl methacrylate (MMA) was produced by Zoltek Rt, Hungary. The intrinsic viscosity of the polyacrylonitrile was equal to 1.3 ± 0.02 dl/g.

Pure dimethyl sulfoxide (DMSO) with a molecular weight $M_r = 78.13$ g/mol, used as a solvent, was produced by POCh S.A. Gliwice, Poland.

Preparation of the electrospinning solutions

PAN/DMSO solutions were used as spinning liquids. The polyacrylonitrile polymer was dissolved in dimethyl sulfoxide (DMSO) to obtain solutions with concentrations ranging as follows: 10, 13, 15, 17 wt %, respectively.

Electrospinning process – methods

Three different electrospinning methods were explored: the conventional method (Method A), Jirsak's method (Method B) and Yarin & Zussman's method (Method C), respectively.

For the experiments three laboratory prototype stands were used, for each method there was an individual stand.

Conventional method (Method A)

For this method the set up was a conventional electrospinning stand with one capillary. The most important basic components were a high voltage supplier, capillary tube with a single metal capillary, and a metal collector (**Figure 1**), [23]. The diameter of the capillary used was 0.8 mm.

The polymer solution was introduced into the capillary tube. Voltage was used to create an electrostatic field between the droplet of polymer solution at the tip of the capillary and the collector plate. This voltage was used to create an electrically charged jet of polymer solution out of the capillary.

In this set up two electrodes were the main elements. The positive conductor of the voltage source was connected to a syringe for extruding the polymer. The aluminium plate connected to the ground was used as a take-up electrode, called a collector electrode. Both electrodes are mutually insulated. Moreover, the whole

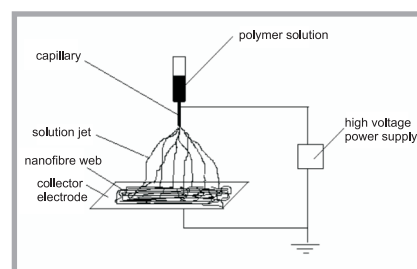


Figure 1. Schematic illustration of the set up for the conventional electrospinning method with a single capillary.

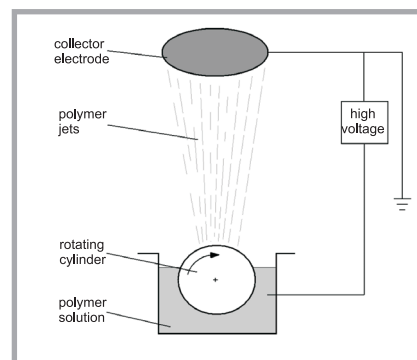


Figure 2. Schematic illustration of the set up for Jirsak's electrospinning method [5].

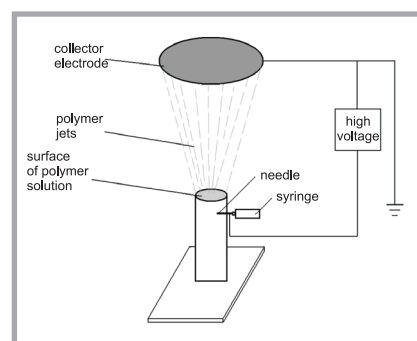


Figure 3. Schematic illustration of the set up for Yarin & Zussman's electrospinning method.

system was insulated from external electric fields by a screen. Mutual charge repulsion generated an electrostatic force, which created fluid drop elongation at the tip of the capillary. At this place the solution was formed into a conical shape, known as a Taylor cone [24]. As the voltage was increased between the anode and cathode, at its critical value, the electrostatic force overcame the surface tension of the polymer drop. The charged continuous jet of solution was ejected from the tip of the Taylor cone. The jet of solution was accelerated towards the collector electrode, whipping and bending wildly. On the surface of the grounded collector, randomly oriented solid nanofibres were deposited.

Jirsak's method (Method B)

This modified electrospinning method, concerning fibre production directly from the polymer solution, was developed by Jirsak [17]. Unlike other methods, instead of a capillary, a rotating cylinder was used to form fibres. The length of the cylinder was 8 cm and its diameter was 2 cm. The polymer solution was poured into a plastic dish. The bottom of the rotating cylinder's body was partially immersed in the polymer solution. When the cylinder was rotating, a certain amount of the polymer solution moved to the top part of the cylinder where Taylor cones were created. An electric field was created by the potential difference between the charged cylinder electrode and counter electrode. The cylinder was indirectly connected to the voltage source (positive) via the polymer solution, into which the metal tip of the conductor was placed. The metal plate used as a counter collector electrode was connected to the ground, (Figure 2). Taylor cones were formed next to each other throughout the entire length of the cylinder. As the solvent evaporated, streams of polymer solutions were transformed, thus solid nanofibres were obtained before reaching the opposite collecting counter electrode.

Jarin & Zussman's method (Method C)

This method was similar to Yarin & Zussman's capillaryless method [22]. Nanofibres were obtained directly from the polymer solution surface (Figure 3).

In this method the set up was composed of two electrodes. One of them was a metal can connected to the voltage source (positive) by the metal tip of the conductor embracing a metal needle. This needle was submerged into polymer solution. The other one was the upper collector electrode connected to the ground. The distance between the surface of the spinning solution layer and the counter electrode was adjusted. When a high voltage was applied to the system, the polymer solution jets were directed upwards. The solvent evaporated, and the multiple electrified jets were exposed to strong stretching by the electric field. As the level of polymer solution in the dish decreased during the process, more polymer solution was added from a syringe. Finally, solidified nanofibres are deposited on the upper counter electrode.

All of the experiments were carried out at normal conditions ($22 \pm 1^\circ\text{C}$, RH = 38%) under normal atmospheric pressure.

Different voltage values of 10, 15, 20, 22.5, 30, 35, 40 kV and various distance values between the spinning surface and collector counter (5, 10, 15, 20, 25 cm) were used. In Method B three different cylinder speeds (the first speed was 62,73 turn/min, the second speed 174,98 turn/min and the third speed 199,3 turn/min) were used.

Methods of fibre characterisation

Scanning Electron Microscopy (SEM) was used to analyse the fibre properties. A Lucia G image analysis software program was used to determine the fibre diameter.

Results and discussion

In this research work, many different results were found because of using differ-

ent electrospinning methods. One of the differences between the three methods used was productivity. It was observed that it is possible to increase the production rate by Methods B and C, where the nanofibres are electrospun from multiple jets. In order to increase the productivity rate in the conventional method, it is necessary to develop a spinning head system with more capillaries placed at suitable distances from each other.

All of the process parameters applied during the experiments and fibre diameters obtained are summarised in **Tables 1 - 4**.

The effect of process parameters on fibre diameter is shown in **Figures 4** (see page 17), **8 - 10** (see page 18). The fibre diameters were determined on the basis of thirty measurements by using 'Lucia Programme'. In these figures the meas-

Table 1. Specification of technological parameters applied during the conventional electrospinning method – Method A.

Sample No	Solution concentration, wt %	Distance, cm	Voltage, kV	Average fibre diameter, nm	Coefficient of variation of fibre diameter, %	
A1	13	10	10	191.0	54.4	
A2			15	201.0	34.7	
A3			20	208.0	47.8	
A4			22.5	166.0	46.2	
A5		20	10	220.0	27.2	
A6			15	292.0	29.7	
A7			20	298.0	34.6	
A8			22.5	350.0	25.4	
A9		25	15	243.0	32.4	
A10			20	212.0	26.0	
A11			22.5	227.0	56.0	
A12	15	10	10	258.0	25.0	
A13			15	271.0	43.9	
A14			20	275.0	40.5	
A15			22.5	343.0	31.0	
A16		20	10	251.0	39.1	
A17			15	329.0	23.3	
A18			20	346.0	45.5	
A19			22.5	435.0	25.5	
A20		25	10	355.0	29.6	
A21			15	387.0	48.4	
A22			20	452.0	16.6	
A23			22.5	323.0	24.2	
A24			17	10	10	682.0
A25		15			471.0	42.4
A26		20			661.0	41.2
A27	22.5	500.0			35.5	
A28	20	10		644.0	46.5	
A29		15		522.0	31.1	
A30		20		566.0	27.8	
A31		22.5		397.0	32.7	
A32	25	10		462.0	25.8	
A33		15		521.0	37.7	
A34		20		393.0	40.1	
A35		22.5	361.0	39.7		

Table 2. Specification of technological parameters applied in Jirsak's electrospinning method – Method B; Speed 174.98 turn/min.

Sample No	Solution concentration, wt %	Distance, cm	Voltage, kV	Average fibre diameter, nm	Coefficient of variation of fibre diameter, %
B1	13	10	10	410.0	37.0
B2			15	540.0	24.4
B3			20	660.0	23.9
B4		15	10	710.0	33.3
B5			15	480.0	26.4
B6			20	420.0	37.1
B7		20	10	720.0	24.3
B8			15	710.0	21.6
B9			20	625.0	24.6
B10		25	10	Not obtained	
B11			15	Not obtained	
B12			20	690.0	37.3
B13	15	10	10	190.0	67.1
B14			15	832.0	32.1
B15			20	349.0	25.5
B16		15	10	850.0	46.8
B17			15	819.0	48.6
B18			20	446.0	43.0
B19		20	10	540.0	38.0
B20			15	286.0	20.9
B21			20	463.0	36.6
B22		25	10	Not obtained	
B23			15	Not obtained	
B24			20	359.0	34.7
B25	17	20	20	1471.0	31.7

Table 3. Influence of the rotating cylinder speed on fibre diameter in Jirsak's electrospinning method – Method B; Solution Concentration - 15 wt %.

Sample No	Speed, turn/min	Distance, cm	Voltage, kV	Average fibre diameter, nm	Coefficient of variation of fibre diameter, %
B26	Speed 1 (62.73)	20	20	882.0	7.9
B27	Speed 2 (174.98)	20	20	463.0	36.6
B28	Speed 3 (199.3)	20	20	448.0	61.8

Table 4. Specification of the technological parameters applied during the Yarin & Zussman's electrospinning method – Method C.

Sample No	Solution concentration, wt %	Distance, cm	Voltage, kV	Average fibre diameter, nm	Coefficient of variation of fibre diameter, %	
C1	10	5	22.5	167.0	74.9	
C2		10	22.5	150.0	42.4	
C3	13	5	22.5	385.0	47.2	
C4		10	22.5	534.0	23.7	
C5		15	22.5	448.0	20.1	
C6	15	5	22.5	592.0	8.1	
C7		10		330.0	24.7	
C8	17	5	22.5	1107.0	9.3	
C9		10		950.0	63.0	
C10		15		1249.0	17.5	
C11		15	15	29.6	585.0	10.8
C12				30	235.0	22.5
C13				35	386.0	34.0
C14			20	37	866.0	20.4
C15				30.8	1097.0	15.6
C16				35	425.0	29.5
C17		25	40	464.0	30.2	
C18			30	480.0	16.8	
C19			35	799.0	21.2	
C20	40		474.0	13.5		

urement points are connected automatically.

On the basis of the test results presented in **Tables 1 - 4**, it can be seen that technological parameters of the electrospinning process have a considerable influence on fibre morphology.

The values of coefficient of variation of fibre diameter in Method A are more uniform for higher solution concentrations, and in Method B the values of coefficient increase with an increase in the rotating cylinder speed. Among the three methods studied, the values of coefficient are the lowest in Method C for higher solution concentrations.

Generally, solution concentration, the distance between electrodes and applied voltage control fibre diameter. If the concentration is too low, the solution can form droplets instead of fibres, whereas if the concentration is increased, the fibre diameter also increases irrespective of which method is applied, as was illustrated in Figure 4. For instance, in Method A (**Table 1**) at following identical conditions: distance between electrodes - 10 cm, voltage - 10 kV, the fibre diameter is 191 nm for 13 wt %, 258 nm for 15 wt % and 682 nm for 17 wt % solution concentration.

In literature [25] this phenomena is described in terms of a Berry number. The fibre diameter depends exactly on the polymer molecular conformation in the solution, defined by a Berry number $[\eta]c$ (where: $[\eta]$ is the intrinsic viscosity of the polymer, c is the concentration of the solution). The following equation shows the relationship between fibre diameter D and the Berry number, i.e. concentration of the solution:

$$D = 18.6([\eta]c)^{1.11} \text{ in nm} \quad (1)$$

The diameters of fibres obtained from the solution of lower concentration are smaller than diameters of fibres obtained at identical electrospinning conditions but from the solution of higher concentration.

In this study, during the electrospinning process, it was also observed that the character of relation between the fibre diameter, applied voltage and distance between electrodes depends on the solution concentration, which is illustrated in **Figure 5**.

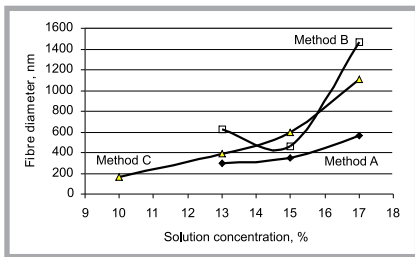


Figure 4. Effect of voltage on fibre diameter for Methods A and B (15 wt % polymer concentration, $d = 20$ cm).

At low solution concentration values (13%, 15%), the fibre diameter increases with an increase in the applied voltage and distance between electrodes simultaneously; for instance, for 13 wt % solution and the following conditions: distance between electrodes – 10 cm, voltage – 10 kV, the fibre diameter is 191 nm, but for these conditions: distance between electrodes – 25 cm, voltage – 22.5 kV, the fibre diameter is 227 nm. The situation is similar for 15 wt % solution, where at the following conditions: distance between electrodes – 10 cm, voltage – 10 kV, the fibre diameter is 258 nm, but at these conditions: distance between electrodes – 25 cm, voltage – 22.5 kV, the fibre diameter is 323 nm. For polymer so-

lutions with a higher concentration, e.g. 17 wt %, the situation is opposite; for instance, for 17 wt % solution and the following conditions: distance between electrodes – 10 cm, voltage – 10 kV, the fibre diameter is 682 nm, but for these conditions: distance between electrodes – 25 cm, voltage – 22.5 kV, the fibre diameter is 361 nm. The values of distance between electrodes and applied voltage necessary to produce nanofibres depend on the polymer solution concentration. As can be surmised from the literature [26, 27], increasing voltage causes a higher mass flow rate and higher strain rate in the solution. For low-viscosity (low concentration) solutions, increasing voltage increases fibre diameter because the mass flow rate increases while the strain rate is constant. For high-viscosity (high concentration) solutions with increasing voltage, fibre diameter decreases because frictional forces are high, which limit the mass flow rate, causing the strain rate to increase, and consequently the fibres obtained are thinner. Based on the results presented, it is possible to draw the conclusion that for each characteristic of polymer solution, the values of two variable voltage and the distance between electrodes should be optimised.

A similar observation concerning fibre diameter was made in relation to Method B. In Method B (Table 2) sample B27, made from 15 wt % solution at a speed of 2 with a distance of 20 cm and voltage of 20 kV, has a fibre diameter equal to 463 nm, while sample B25, made from 17 wt % solution at a speed of 2 with a distance of 20 cm and voltage of 20 kV, has the highest fibre diameter – 1471 nm. 17 % polymer concentration in the solution is too high to produce fibres easily by Method B. Moreover, the results presented in Table 3 indicate that the diameter of fibres decreases with an increase in the rotating cylinder speed. Figure 6 illustrates SEM images of nanofibres electrospun from 15 wt % solutions of PAN polymer at 20 kV and at 20 cm distance, at different speeds of the cylinder.

The comparison of the processability of Methods A and B indicates that Method A allows to produce thinner fibres than Method B. A clear difference in fibre diameter produced from 13 wt % solutions of PAN polymer at 20 kV and 20 cm distance can be seen in Figure 7 (see page 18). Coarser fibres and rougher fibre surface are obtained by Method B compared to Method A. For the same

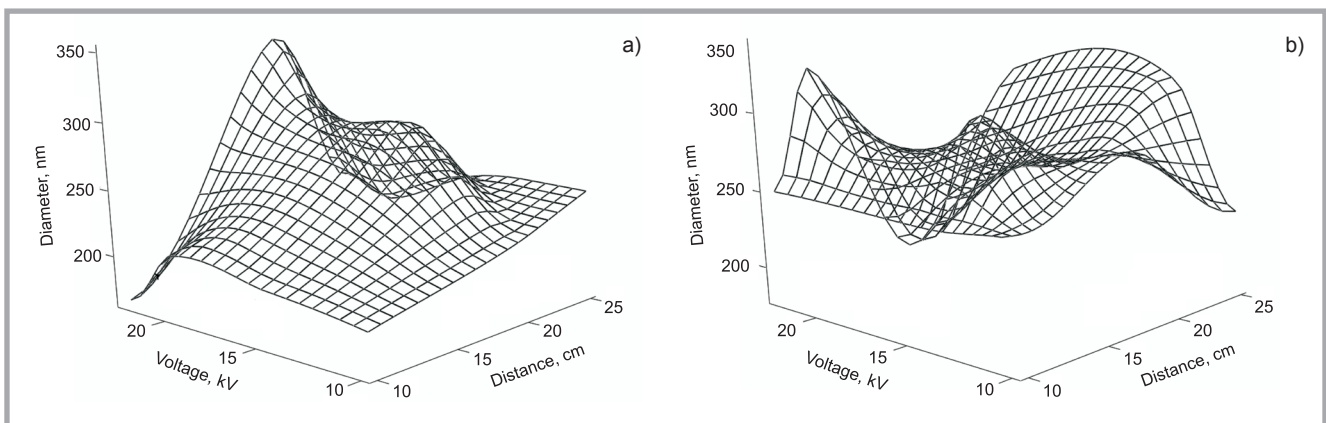


Figure 5. Comparison of the character of changes in diameter of fibres in relation to solution concentration, applied voltage and distance between both electrodes for Method A: a) relationship for a solution concentration of 13%, b) relationship for a solution concentration of 17%.

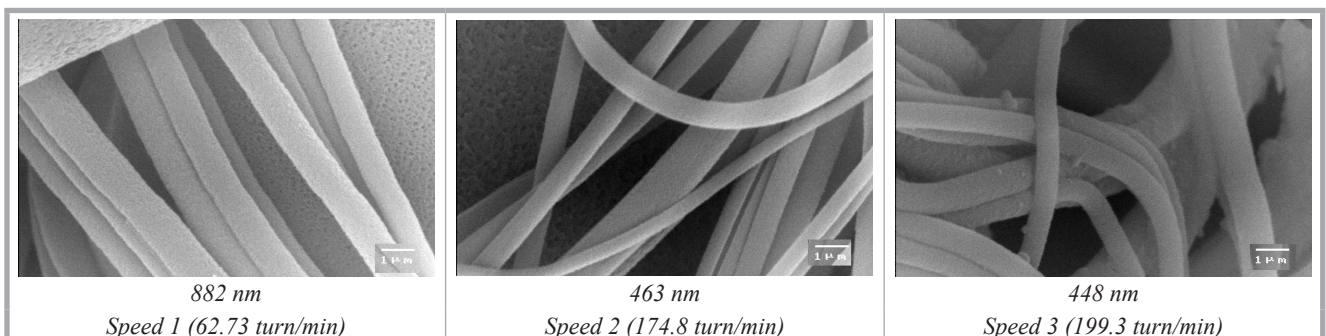


Figure 6. SEM images of nanofibre samples B26, B27 and B28 respectively, obtained by using Method B ($\times 10,000$).

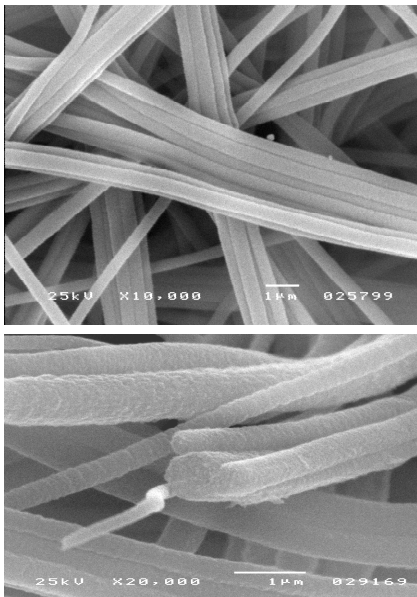


Figure 7. Comparison of SEM images of nanofibre samples A7 and B9 obtained by using Methods A and B ($\times 10\,000$); a) Method A, b) Method B.

conditions but different methods the fibre diameters are different.

Table 4 (see page 16) shows that also in Method C sample C2 obtained from 10 wt % solution has the lowest fibre diameter, while sample C10 obtained from 17 wt % solution has the highest (micro fibre) diameter. In this method the voltage values should be relatively high and distances between the solution surface and collector electrode should be relatively short. The voltage value used depends on the solution concentration, for higher concentrations the voltage must be higher. The electrospinning also proceeds from solutions of low concentration, even from 10 wt % solution. A further increase in solution concentration causes an increase in the fibre diameter.

The results presented allow to draw the conclusion that in all three methods the coarsest fibres are obtained from 17 wt % polymer solution. Especially for Methods B and C it is evident that 17 wt % solution concentration is not suitable for producing nanofibres. Moreover, it is possible to produce nanofibres from 10 wt % polymer solution by Method C, while this is not possible by Methods A and B. Moreover, to produce the nanofibres using Method C, the lowest concentration of PAN/DMSO solution can be used.

Moreover, it is observed that higher voltage values such as >20 kV are necessary for Method C, while voltage values of

the following range: 10, 15, 20 kV are enough for Methods A and B, as shown in **Figure 8**.

It is known from the literature [26, 28] that a voltage change at a constant distance between electrodes does not significantly change the fibre diameter; but in general a higher applied voltage decreases the fibre diameter especially for highly concentrated solutions.

Similarly, as in literature [29], experimental results show various dependences, for some variants of process parameters with increasing voltage the fibre diameter increases, for other variants the situation is opposite. It results from the fact that the applied voltage decides many factors influencing fibre diameter in a different way, such as the flow rate being dependent on solution concentration, the morphology of the jet, the elongation level of the jet by an electrical force, etc. The fibre diameter is a result of these factors, for example, shows that increasing voltage leads to higher fibre diameter in Method A, while in Method B the fibre diameter decreases between 10 and 15 kV and in Method C between 22.5 and 30 kV. Generally, for each method there exists a certain range of voltage values due to which the fibre diameter increases.

The effect of distance on fibre diameter is depicted in **Figures 9** and **10**. It is reported in the literature [28, 30] that by increasing the distance between the electrodes, the fibre diameter decreases. Quite the opposite is reported in literature [26] concerning a method with a single needle in which the fibre diameter decreases with an increasing electric field due to a change in the working distance. If the distance between the electrodes is shorter, the intensity of the electric field is higher and the fibre diameter should be smaller. The results of this relationship were confirmed by simulations carried out on multi-capillary equipment [31]. However, in **Figure 9**, for 15 % solutions the fibre diameter increases by increasing the distance, but in Method B one can observe that over a distance of 20 cm, the fibre diameter decreases. **Figure 10** shows a similar shape of the curve of the fibre diameter – the relationship of the distance between electrodes in Method A and C for 13 % solutions. The fibre diameter increases by increasing the distance up to 10 cm in Method C, but in Method A the fibre diameter increases as the distance increases up to 20 cm. In addition,

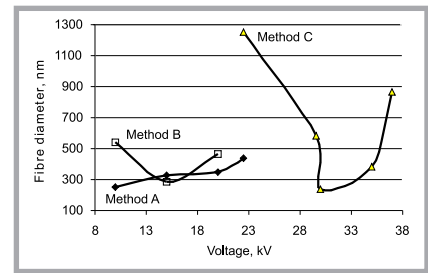


Figure 8. Effect of voltage on fibre diameter for Methods A and B (15 wt % polymer concentration, $d = 20$ cm), and Method C (17 wt % polymer concentration, $d = 15$ cm)

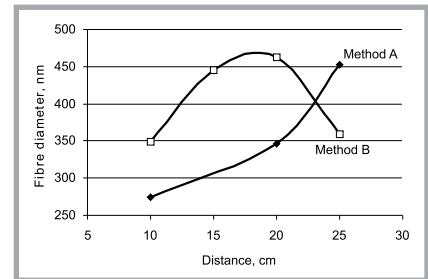


Figure 9. Effect of distance on fibre diameter for Methods A and B (15 wt % solution concentration, 20 kV).

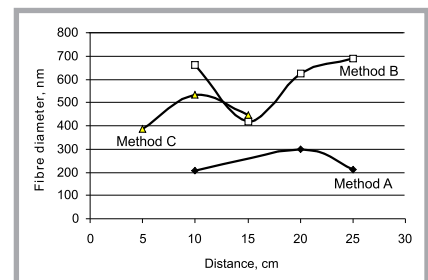


Figure 10. Effect of distance on fibre diameter for Methods A and B (13 wt % solution concentration, 20 kV) and Method C (13 wt % solution concentration, 22.5 kV).

tion, the distance between the electrodes in Method C, equal to 5 cm, seems to be too low for Method A to obtain dry fibres. However, by Method C it is only possible to produce fibres at 22.5 kV up to 15 cm distance.

Conclusions

In this research work three different electrospinning methods were compared: the conventional method (Method A), Jirsak's method (Method B) and Yarin & Zussman's method (Method C), respectively. The effect of various process parameters on fibre properties was investigated, mainly the fibre diameter. It was observed that it is possible to increase the production rate by Methods B and C, where the nanofibres are elec-

trospun from multiple jets. In Method A with a single capillary development of the spinning head system, it seems to be a solution for increasing productivity. The electrospinning head should contain more capillaries.

Generally, the solution concentration, the distance between electrodes and the applied voltage control the fibre diameter in each method investigated. If the concentration is too low, the solution can form droplets instead of fibres, and if the concentration increases, the fibre diameter also increases irrespective of which method is applied. It was also observed that the character of relation between the fibre diameter, applied voltage and distance between electrodes depends on the solution concentration. For each polymer solution characterised by a Berry number, the combination of values of two variable voltage and the distance between the electrodes should be optimised.

Moreover, it was observed that in Method B the diameter of the nanofibres decreases by increasing the speed of the rotating cylinder. Another interesting result obtained is the possibility of producing fibre at 10 wt % polymer concentration by Method C, leading to much finer fibres than in Methods A and B. Moreover, higher voltage values such as >20 kV are necessary for Method C, while voltage values such as 10, 15, 20 kV are enough for Methods A and B.



Acknowledgments

- These investigations were carried out within project No 3 T08E 082 27 sponsored by the Polish State Committee for Scientific Research.
- The authors would like to thank, Ph.D. A. Komisarczyk and MSc. H. Wrzosek who provided the Scanning Electron Microscopy (SEM) images of nanofibres, from the Technical University of Lodz.

References

1. Grafe T., Graham K., 'Polymeric Nanofibres and Nanofibre Webs: A New Class of Nonwovens', INTC, Atlanta, Georgia, 2002.
2. Simm W., 'Apparatus for the Production of Filters by Electrostatic Fiber Spinning', US Patent 3 994 258, 1976.
3. Chung H. Y., Hall J. R. B., Gogins M. A., Crofoot D., G., Weik, T. M., 'Polymer, Polymer Microfiber, Polymer Nanofiber and Applications Including Filter Structures', US Patent 6 924 028, 2005.

4. Koslow E. E., 'Nanofibre Filter Media' US Patent 6 872 311, 2005.
5. Krucinska I., Klata E., Chrzanowski M., 'New Textile Materials for Environmental Protection', NATO Advanced Research Workshop-Intelligent Textiles for Personal Protection and Safety, 2005.
6. Shin C., Chase G. G., Reneker, D. H.; 'Recycled Expanded Polystyrene Nanofibres Applied in Filter Media'. *Colloids and Surfaces A: Physicochem Eng. Aspects*, vol. 262, (2005) pp. 211-215.
7. Liu H., Hsieh Y.; 'Ultrafine Fibrous Cellulose Membranes From Electrospinning of Cellulose Acetate', *Journal of Polymer Science, Part B: Polymer Physics*, vol. 40, (2002) pp. 2119-2129.
8. Chu, B., Hsiao, B. and Fang, D., 2005. 'Apparatus and Methods for Electrospinning Polymeric Fibers and Membranes', US Patent 6, 713, 011.
9. Balkus, K.J.JR., Ferraris, J.P. and Madhugiri, S., 2003. 'Electrospinning of Polymer and Mesoporous Composite Fibers', US Patent 20030168756.
10. Giannelis, E.P., 1998. 'Polymer-Layered Silicate Nanocomposites: Synthesis, Properties and Applications', *Applied Organometallic Chemistry*, vol. 12, p. 675-680.
11. Adanur S. and Ascioğlu B., 2005. 'Challenges and Opportunities in Nanofibre Manufacturing and Applications', II. International Technical Textiles Congress, p. 29-31, Istanbul, TURKEY.
12. Smith, D.J., Reneker, D.H., McManus, A.T., Schreuder-Gibson, H.L., Mello, C. and Sennett, M.S., 2004. 'Electrospun Fibers and an Apparatus Therefore', US Patent 6, 753, 454.
13. Krucinska, I., Blasinska, A. Komisarczyk, A., Kiekens, P., Chrzanowski, M., Szosland L., and Shoukens G., 2005. 'Review of Techniques for Manufacturing Dibutylchitin Nonwoven Biomaterials', II. International Technical Textiles Congress, p. 1-9, Istanbul, TURKEY.
14. Scopelianos, A.G., 1996. 'Piezoelectric Biomedical Device', US Patent 5, 522, 879.
15. Schreuder-Gibson, H.L. and Gibson, P., 2004. 'Cooperative Charging Effects of Fibers from Electrospinning of Electrically Dissimilar Polymers', *INJ Winter*, p. 39-45.
16. Formhals, A., 1934. 'Process and Apparatus for Preparing Artificial Threads', US Patent No. 1, 975, 504.
17. Jirsak, O., 2003. 'A Method of Nanofibres Production From a Polymer Solution Using Electrostatic Spinning and a Device for Carrying Out The Method', Cz. Patent, 2414 (2, 994, 274).
18. Kameoka, J. and Craighead, H.G., 2003. 'Fabrication of Oriented Polymeric Nanofibres on Planar Surfaces by Electrospinning', *Applied Physics Letters*, vol. 83, p. 371-373.
19. Lee, W.S., Jo, S. M., Go, S. G. and Chun, S. W., 2003. 'Apparatus of Polymer Web by Electrospinning Process', US Patent 6, 616, 435.
20. He, J., Wan, Y. and Yu, J., 2004. 'Application of Vibration Technology to Polymer Electrospinning', *International Journal of Nonlinear Sciences and Numerical Simulation* vol. 5, p. 253-262.
21. Tomaszewski, W. and Szadkowski, M., 2005. 'Investigation of Electrospinning with the Use of a Multi-jet Electrospinning Head', *Fibres&Textiles in Eastern Europe*, vol. 13, Nr 4 (52), p. 22-26, 2005.
22. Yarin, A.L. and Zussman, E., 2004. 'Upward Needleless Electrospinning of Multiple Nanofibres' *Polymer*, vol. 45, p. 2977-2980.
23. Dosunmu, O.O., Chase, .G.G., Kaptaphinan and Reneker, D.H., 2006. 'Electrospinning of Polymer Nanofibres from Multiple Jets on a Porous Tubular Surface', *Nanotechnology*, vol. 17, p. 1123-1127.
24. Taylor, G. I., 1964. 'Disintegration of water drops in an electric field', *Proc. R. Soc. London, Ser. A.*, p. 280,383.
25. Tao, J., Shivkumar, S., 2007. "Molecular weight dependent structural regimes during the electrospinning of PVA", *Materials Letters*, vol. 61, p. 2325-2328.
26. Wang, T., Kumar S., 2006. "Electrospinning of polyacrylonitrile nanofibers", *Journal of Applied Polymer Science*, vol. 102, p. 1023-1029.
27. Mitchell, S.B., Sanders, J.E., 2006. "A unique device for controlled electrospinning", *Journal of Biomedical Materials Research Part A*, vol. 78 A, Issue 1, p. 110-120.
28. Zufan, R. and Megaridis, C., 2005. 'Electrospinning of Nanofibres', Final 2005 RET Report.
29. Tan, S-H., Inai, R., Kotaki, M., Ramakrishna, S., 2005. "Systematic parameter study for ultra-fine fiber fabrication via electrospinning process", *Polymer*, vol. 46, p. 6128-6134.
30. Doshi, J. and Reneker, D. H., 1995. 'Electrospinning Process and Applications of Electrospun Fibres', *Journal of Electrostatics*, vol. 35, p. 151-160.
31. Krucińska, I. Komisarczyk, A. Chrzanowski, M. Wrzosek, H. Gliścińska, E., "Modelling The Electrostatic Field Forming Around Multispinneret Head for Electrospinning of Polymer Solutions", IMTEX 2007.

Received 26.09.2007 Reviewed 08.10.2008