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Electrostatic Field in Electrospinning with a Multicapillary Head – Modelling and Experiment

Abstract
Electrospinning is a process by which electrostatic field super thin fibres from polymer solution or polymer melt are produced. This study is a theoretical analysis of the distribution of the electrostatic field forming around spinning points in the area where polymer streams are formed and stretched. The model parameters were as follows: the number of capillaries, the distance between the capillaries, the capillary tip – including the electrode distance and the value of the supply voltage. The modelling was undertaken for 3 cm fixed length capillary tubes. The electrostatic field distribution was modelled with the use of Maxwell SV software. The increase in the distance between the capillary tip and the collector causes a drop in the strength value of the electrostatic field that forms in direct proximity to the capillaries, regardless of their position to one another. In the case of an increase in the supply voltage value, an increase in the strength value of the electrostatic field around the capillaries was observed. Next, an experiment was performed for selected modelling conditions. Using solutions of dibutyrylchitin, electrospinning tests were carried out. The influence of the process conditions and, consequently, the electrostatic field distribution on the fibre geometry was studied. For each test the transverse dimension values and the shape of the cross section area of the fibres were determined.

Key words: *electrospinning, microfibers, nanofibres, electrostatic field, computer simulation, dibutyrylchitin.*

Introduction

Nanofibres can be obtained in the electrospinning process, which consists in polymer stream forming, stream thinning and fibre collecting on the grounded collector. Electrospinning is a technique in which polymer solution or molten polymer, in the majority is pumped through electrode holes with diameters in the order of about 1 millimetre [1]. On applying the power supply voltage, electrical induction occurs and the solution becomes charged [2]. Consequently, the solution in its entire capacity is given a single fixed potential. Under the influence of the active electrostatic field and Coulomb forces present, the semispherical drop surface at the nozzle mouth of the capillary tube is strained into a conical shape (known as a Taylor cone) with an angle of 49.3° [3, 4], and then it is stretched into a stream and significantly thinned. The polymer is collected on the grounded collecting element – the collector [5]. The time of the mechanism forming the polymer stream is approximately 0.0125 ms. The uninominal charges that gather on the polymer stream surface, in combination with the stream surface tension, cause the stream to bend, the effect of which is that the distance covered by the polymer becomes extended. As a consequence of the active electrostatic field, fibres thus manufactured have diameters that vary from ten to several hundred nanometres. Such significant fibre thinning causes the product to be substantially extended and its properties to change. The first studies related to electrostatic field effects were carried out by J. Zeleny [6], and then by G.I. Taylor [7] and by S.A. Theron, A.L. Yarin, E. Zussman, E. Kroll [8].

The time it takes to electrospin a single polymer stream varies from 0.1 ml/h to 1ml/h. Such low process efficiency restricts the possible applications of electrospinning in industrial processes. Currently, the ongoing studies are intended to boost the process efficiency by multiplying the number of spinning points [9 - 11], as well as by other techniques, for example using the rotating-drum technique with its particular advantages and disadvantages [12 - 14]. The experiments have shown that in the case of a close proximity between a number of spinning points, when voltage is applied, the polymer stream deviates [15], and the size and direction of such a stream bend depend on the position and distances between the spinning points.

The ongoing studies show that apart from the polymer stream deviation, the angle of the Taylor cone changes due to the interaction between adjacent spinning points. For linear configuration of the capillaries at 1 cm intervals, the cone's angle, measured between two bright lines formed by the polymer stream, was within 25° and 30°. The measurements of the Taylor cone's angle in the perpendicular setup produced values ranging from 50°

to 75° in dependence on the geometrical form of the electrostatic field. This suggests that the cone, when in close proximity to subsequent spinning points, becomes flattened on the line that is perpendicular to the axes of the capillaries.

The phenomenon of deviation in polymer solution streams that flow out of adjacent capillaries may result from the deviation of the electrostatic field that forms around the nozzle mouths of the spinning points. The field distribution may be simulated with the use of calculation software. To experimentally assess the field distribution, sensors should be installed to detect the field distribution.

In this work, before setting up and performing multiple-spinning, a computer simulation of the electrostatic field between electrodes was made. The existence of this field decides the initiation and course of the electrospinning process. It can seem that the initiation and character of the electrostatic field depend on the distance between the capillary and collector, the distance between capillaries, their arrangement, as well as on the material from which the capillaries are made.

In this study, for the selected technological conditions of the electrospinning process, a computer simulation of the electrostatic field distribution was made. In these conditions tests of electrospinning using dibutyrylchitin solution were carried out. Dibutyrylchitin is biodegradable polyester obtained from chitin. Introducing large groups derived from butyric acid to the polymeric chain causes dibutyrylchitin solubility. This polymer is easily soluble in many organic solvents, even in those used in medicine, such as dimethylsulfoxide and ethyl alcohol. In a dissolved state it can be transformed into film or fibres. Due to these features, textile fabrics made from dibutyrylchitin can be produced.

The methods of obtaining dibutyrylchitin fibres using spinning solutions from a water coagulating bath [16], as well as dry spinning from a solution [17], are well known. Moreover, nonwoven formed by the technique of polymer solution blowing has already been established [18]. In the above methods classical organic compounds (acetone, methanol, ethanol, dimethylformamide, dimethylacetamide or dimethylsulfoxide) are used as a solvent. An additional advantage is the possibility of transforming dibutyrylchitin into initial chitin by hydrolysis of ester groups. At the present time the subject of numerous investigations is the biological properties of dibutyrylchitin. Results hitherto obtained indicate that the biological characteristics of dibutyrylchitin is comparable with the characteristics of the initial chitin [19 - 25].

Model part

Electrostatic field

An electrostatic field (electric field) is a some state of space, in which forces act on the arbitrary charges. These forces can be determined by the electrostatic field strength. The electrostatic field strength is defined by the ratio of the force acting on the electric charge in the electrostatic field to the value of this electric charge [2].

The existence of the electrostatic field decides the initiation and course of the electrospinning process. The distance between the tips of the capillary tubes and the collector as well as the distance between the capillary tubes and their mutual arrangement influence the forming process and the character of the electrostatic field.

Simulation of the electrostatic field distribution

Usually, the methods of electrostatic field measurement are connected with the probe input to the field studied. Such a probe disturbs the field distribution and can be used to measure a field of large size, for example under a electrical supply line. For small sized fields, like the one formed between electrodes placed at a distance of several to tens of centimetres away, analytical methods are required.

In this study, computer simulation of the electrostatic field distribution around the capillaries in the electrospinning head applied in the electrospinning process, as shown in ***Figure 1***, was made.

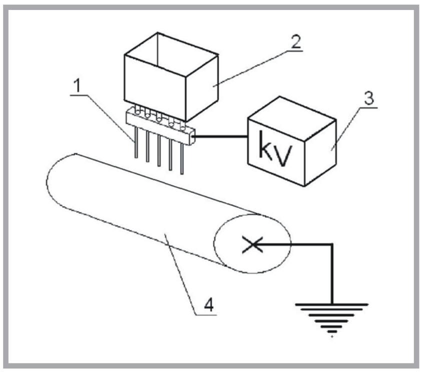

Figure 1. *Diagram of the multicapillary electrospinning setup. The drawing shows: 1- capillaries, 2 – polymer solution container, 3 – high voltage generator, 4 – collector.*

The electrostatic field distribution around the capillaries was modelled with the use of Maxwell SV software, applying the finite elements method. The model components consisted of acid-resistant steel capillaries with a fixed length of 30 mm and external diameter of 0.8 mm, with various distances between the capillaries. The internal diameter, which was equal to 0.5 mm, did not influence the modelling process. The distances of 1 and 6 cm were extreme and resulted from a setup construction. Five capillaries were used. The model reflected the variable distance of 15 – 25 cm between the tips of the capillaries and the collector, as well as the variable values of the supply voltage applied to the capillaries, which ranged from 15 to 25 kV. The ranges of distances and supply voltage values used in the computer simulation were determined on the basis of preliminary tests of the electrospinning process using dibutyrylchitin solution. There are many methods of interpreting results concerning the field distribution. In these calculations two methods were used. The maximum strength of the electrostatic field formed at the tips of the capillaries was determined, and the field strength distribution around the capillaries was shown as a drawing in the form of equipotential lines, e.g. lines highlighting the constant strength of the electrostatic field.

The field strength at a given point of the electrostatic field is the ratio of the electric force acting on the electric charge at this point to the value of this charge. The value of the electrostatic field strength depends on the distribution of the charge, which is the field source. When calculating the resultant electrostatic field strength, the superposition rule can be used, e.g. the vectors of strength at a given point of the field coming from all parts of the body should be added. This vectorial sum is the strength of the field coming from the whole body mentioned above.

Results and discussion

Computer simulation of the electrostatic field distribution was carried out with 25 scale colours for the whole model mesh. The scale of the drawing field

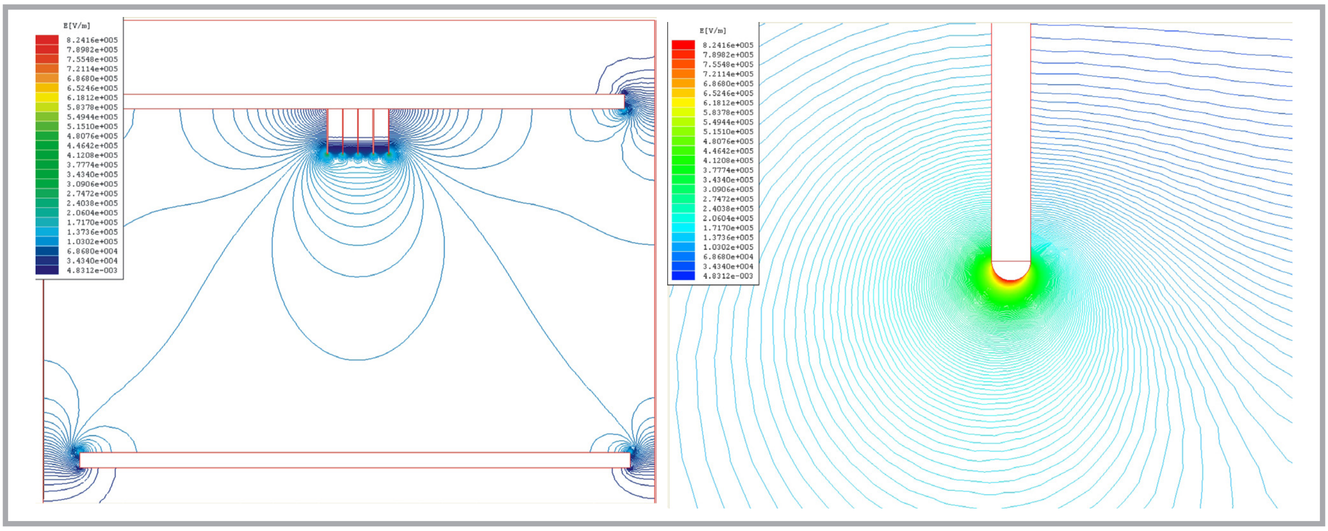

Figure 2. *Field strength distribution with a distance between capillaries of 1 cm, the distance between the capillary tip and collector 20 cm, and a supply voltage of 20 kV; a – field strength distribution around the capillaries, b - field strength distribution around the boundary capillary.*

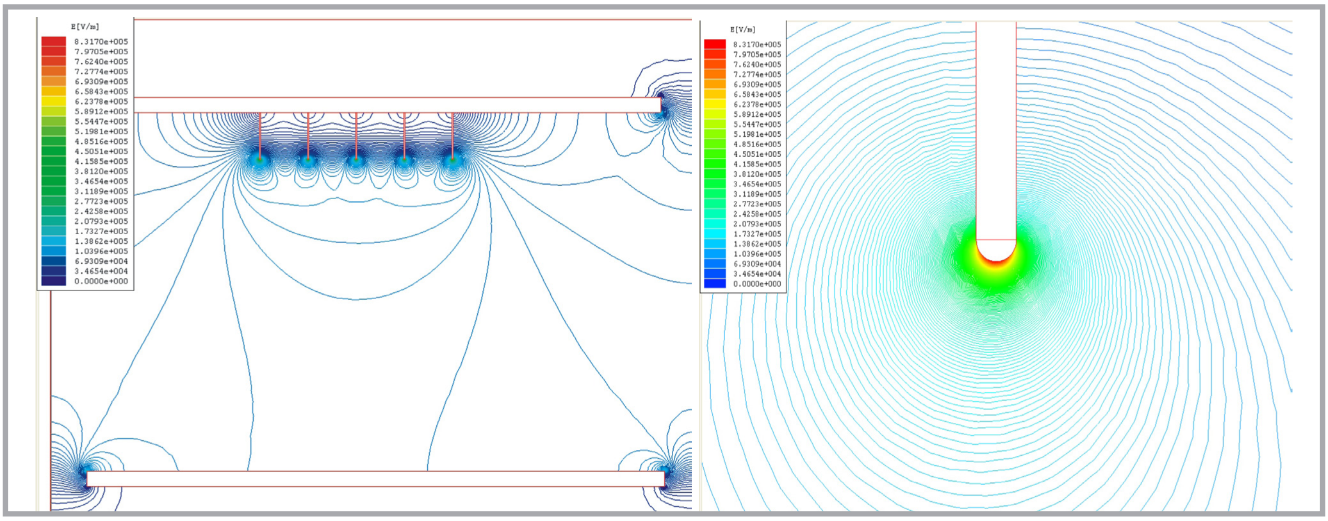

Figure 3. *Field strength distribution with a distance between capillaries of 3 cm, the distance between the capillary tip and collector 20 cm, and a supply voltage of 20 kV; a – field strength distribution around the capillaries, b - field strength distribution around the boundary capillary.*

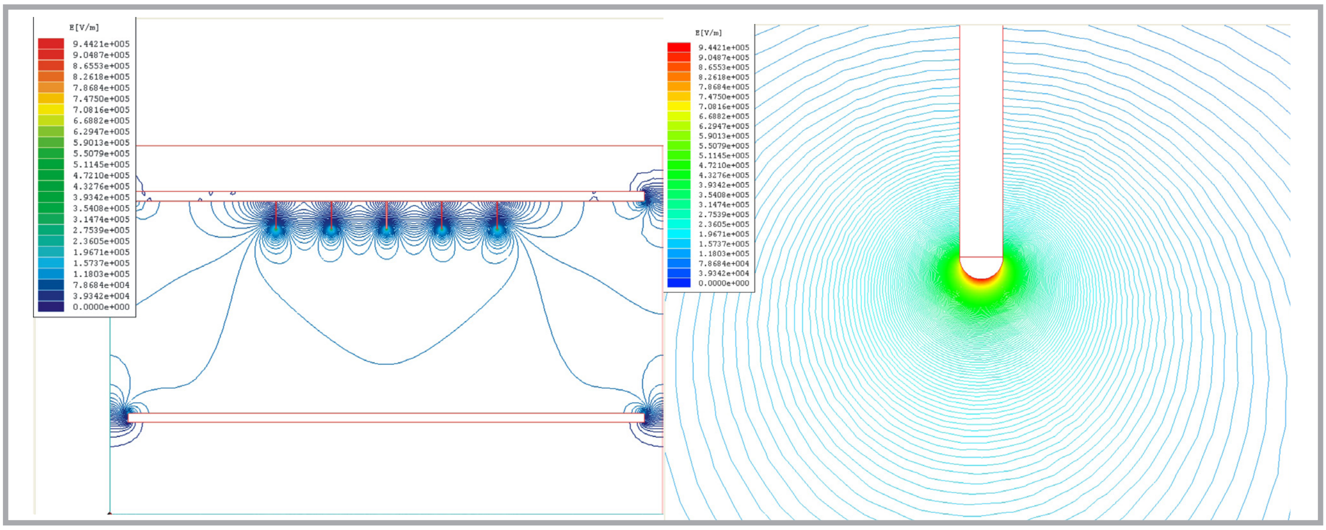

Figure 4. *Field strength distribution with a distance between capillaries of 6 cm, the distance between the capillary tip and collector 20 cm, and a supply voltage of 20 kV; a – field strength distribution around the capillaries, b - field strength distribution around the boundary capillary.*

was established at D = 160, with a linear value distribution. Examples of the field strength distribution are illustrated in ***Figures 2 - 4***, and the field strength values at the tips of the capillaries are shown in ***Tables 1 - 2. Table 1*** shows the dependence of the electrostatic field strength on the number of capillaries and the distance between them at 20 kV. ***Table 2*** shows the dependence of the electrostatic field strength on the distance between capillaries, the distance between the capillary tip and the collector and on the voltage supplied to the system.

A legend for the colours is placed in the left part of the figures. It is visible that the highest strength and the biggest field unevenness occur in the vicinity of the tips of the capillaries. From the pictures obtained for different combinations, it is visible that the closer the capillary tube, the higher the field strength gradient and strength value are. Probably, a too big distance between the tips of the capillaries and the collector can cause a field strength reduction or even a field collapse at the collector, which can make electrospinning initiation difficult and cause electrostatic interferences during the process. Distances of 6 cm between capillaries are favourable conditions for electrostatic field interference in the space above the level of polymer solution outflow. It can be seen that too short distances between capillaries cause mutual interference. Bigger distances, even 6 cm, cause that the equipotential lines of the field arrange themselves in a similar fashion around all the capillaries. The considerable deflection of equipotential lines around the boundary capillaries fades, and electrostatic field strength values are higher and similar around all capillaries. ***Table 1*** confirms that from the results it can be seen that the big difference between values of the electrostatic field strength near the central capillary and of the electrostatic field strength near the successive boundary capillaries fades. It indicates the possibility of uniform electrospinning using all the capillaries without any substantial deflection of the polymer streams coming from boundary capillaries. Therefore, for the system with more capillaries, the electrostatic field strength was determined in the close proximity to the capillaries and was placed beside each one as identical.

Table 2 shows results of the electrostatic field strength for the system, not only for a variable distance between capillaries but also for a variable distance between the capillary tip and the collector, and a variable supply voltage value. The increase in distance between the capillary tip and the collector and the increase in the supply voltage value are not factors which decide the field deformation around the boundary capillaries. The increase in distance between the capillary tip and collector causes a decrease in the strength value of the electrostatic field forming in close proximity to the capillaries, apart from their mutual arrangement. In the case of an increase in the supply voltage value, an increase in the strength of the electrostatic field forming around the capillaries was observed. The correlation was calculated for the electrostatic field distributions and experiment results obtained for the nonwoven forming process in model conditions.

Table 1. *Electrostatic field strength near the tips of the capillaries, (at a supply voltage of 20 kV), depending on the number of capillaries and the distance between them;* $E_{central}$ *– field strength near central capillary,* E_1, E_2 *– field strength near successive capillaries - symmetrical system, the values are only for one side of the system.*

		Distance between capillaries, cm				
		1	**2**	**3**	**4**	**5**
Electrostatic field strength, kV/cm (supply voltage: 20 kV)		**1 capillary**				
	$E_{central}$	10.41				
		3 capillaries				
	$E_{central}$	5.93	7.04	7.52	8.84	9.33
	E_1	7.73	8.25	8.71	9.04	9.46
		5 capillaries				
	$E_{central}$	4.77	6.05	6.68	7.59	8.18
	E_1	5.26	6.22	7.10	7.88	8.62
	E_2	7.41	7.90	8.60	9.01	9.22

Table 2. *Electrostatic field strength near the tips of the capillaries in a multicapillary system (nine capillaries) – dependence on the distance between capillaries, the distance between the capillary tip and collector, and the supply voltage.*

No	Distance between capillaries, cm	Distance between capillary tip and collector, cm	Supply voltage, kV	Electrostatic field strength in the close proximity to capillaries, kV/cm
1	1	20	20	8.242
2	3	20	20	8.317
3	6	20	20	9.442
4	3	15	20	12.711
5	3	20	20	8.317
6	3	25	20	8.018
7	3	20	15	7.37 5
8	3	20	20	8.317
9	3	20	25	12.291

Experimental part

Material

In this study a solution of dibutyrylchitin in 5% wt. ethanol was used. The dibutyrylchitin was synthesised using krill chitin. For polymer studied the mean weight value of the molecular weight was 8800 g/mol and the intrinsic viscosity, measured in DMAc at a temperature of 25 °C, was 1.4 dl/g. The rheological characteristic of the polymer solution was evaluated using a rheotest RV2 for different temperatures and concentrations of the polymer.

Fibre forming from dibutyrylchitin solution by electrospinning

At the Department of Fibre Physics and Textile Metrology of the Technical University of Łódź, a design was made and a laboratory setup constructed for fibre production by electrospinning. A scheme of the setup is shown ***Figure 1***.

The polymer solution is forced through by pressure from a tank to the capillary tubes, to which a voltage of 10 - 30 kV is supplied from a high voltage (***Figure 1***, 3) generator. Due to the fact that the metal collector is earthed, between the capillaries and collector there occurs a potential difference which generates the electrostatic field. The fibres formed are gathered on the collector, [6 - 9, 15, 20].

In this setup, regulation of the distance between the capillary tip and the collec-

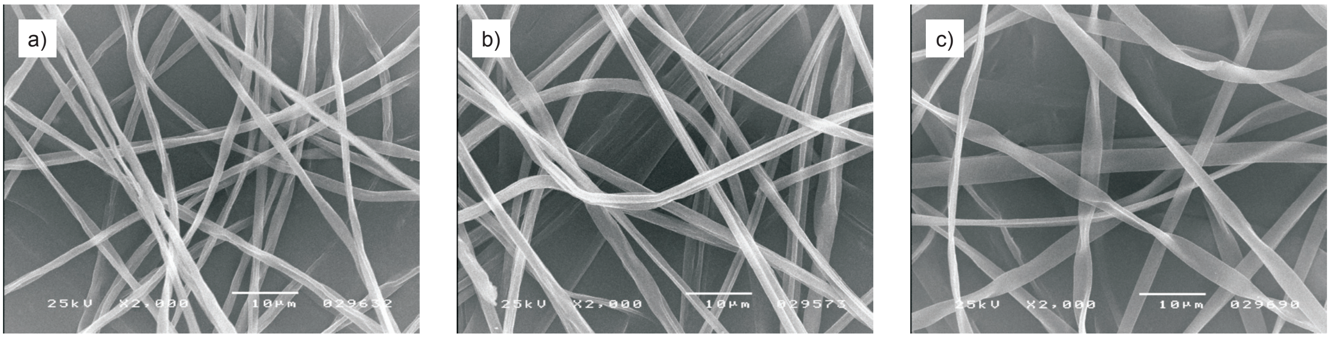

Figure 5. *Relationship between the fibre transverse dimensions and distance between the capillaries; a - 1 cm; b- 3 cm; c – 6 cm distance between the capillary tip and collector – 20 cm, supply voltage – 20 kV.*

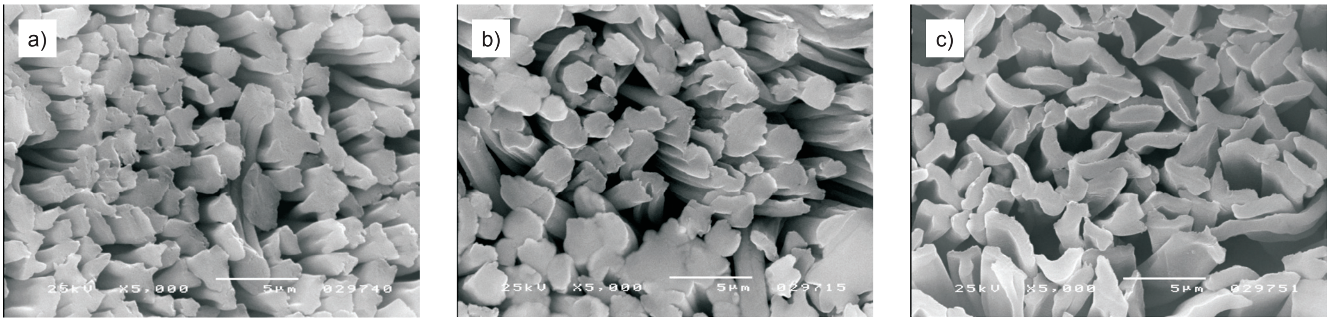

Figure 6. *Cross-section of the fibres obtained at different distances between capillaries; a - 1 cm; b- 3 cm; c – 6 cm.*

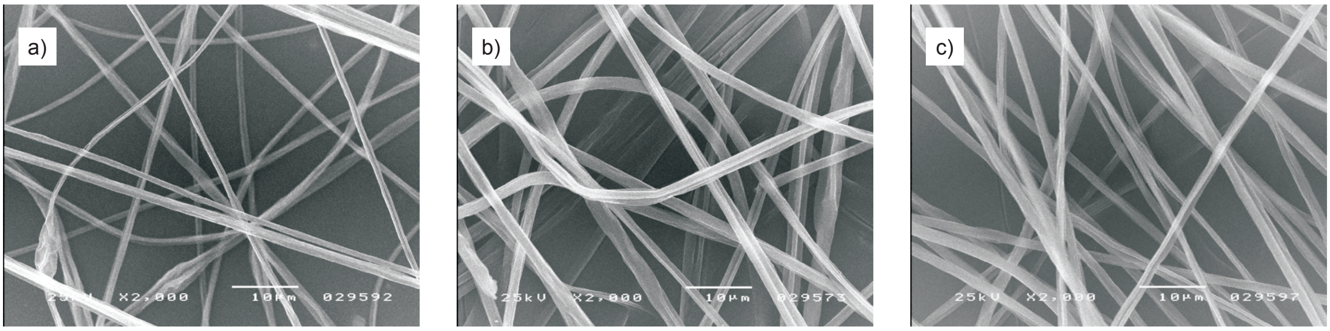

Figure 7. *Relationship between the fibre transverse dimensions and distance between the capillary tip and collector; a - 15 cm; b - 20 cm; c - 25 cm, distance between capillaries - 3 cm, supply voltage - 20 kV.*

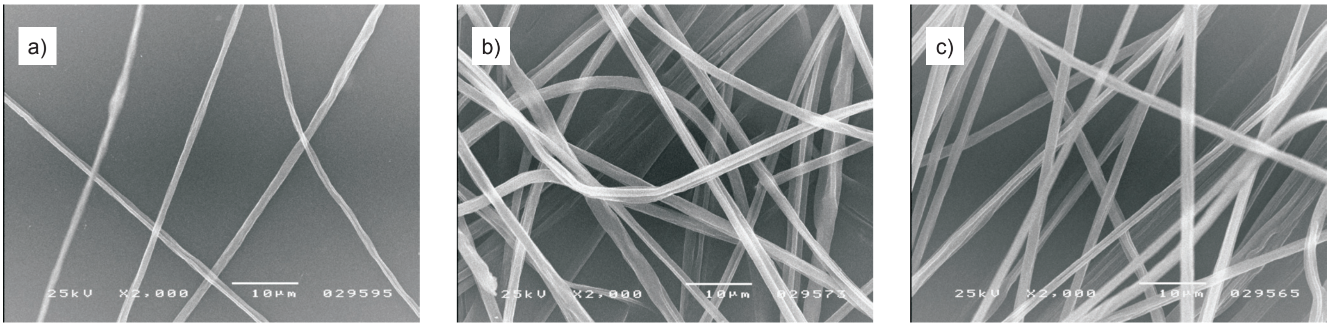

Figure 8. *Relationship between the fibre transverse dimensions and supply voltage; a - 15 kV; b - 20 kV; c - 25 kV cm; distance between the capillaries - 3 cm, distance between the capillary tip and collector - 20 cm.*

tor, the distance between capillaries and the supply voltage value is possible. The setup has a spinning slat with five capillaries placed in a linear system.

Experiments of fibre forming by electrospinning were carried out in the model conditions selected, for which simulation of the electrostatic field distribution was made. Fibres were formed at distances of 1, 3 and 6 cm between capillaries, at distances of 15, 20 and 25 cm between the capillary tip and the collector and at a supply voltage value of 15, 20 and 25 kV. The geometry of the capillaries was the same as in the modelling process.

All of the experiments were carried out at room temperature (21 - 22 °C) and air relative humidity of 35%. The temperature of the polymer solution was about 20 °C.

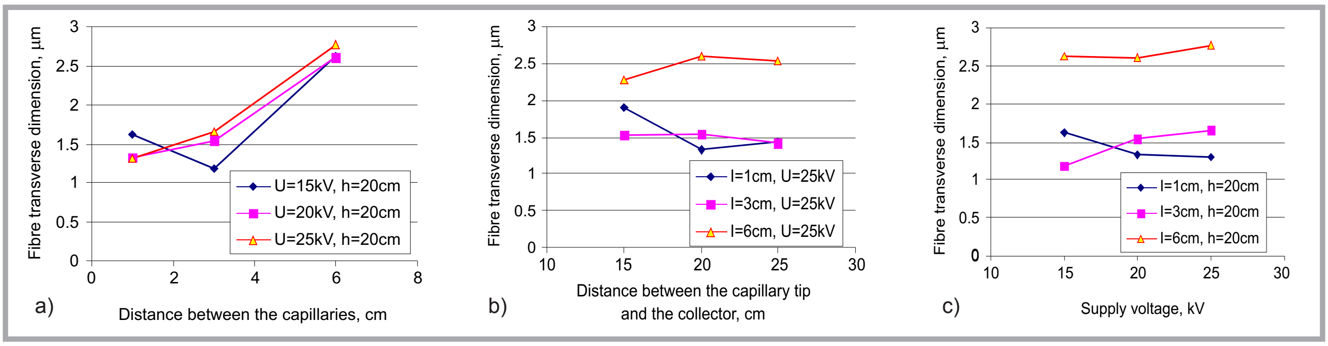

Figure 9. *Relationship between the fibre transverse dimension and a) distance between the capillaries for different supply voltages, b) distance between the capillary tip and collector for different distances between capillaries, c) supply voltage for different distances between capillaries; l – distance between capillaries, h – distance between the capillary tip and collector, U – supply voltage.*

Results

In the course of the process, the deviation of the boundary polymer solution streams in relation to the streams formed by the inner capillaries was observed. Concurrently, the fibres collected from each capillary produced independent, non-overlapping fields. The deviation of polymer streams on the boundary capillaries corresponds to the modelled electrostatic field distribution, which indicates a shift of the equipotential lines for these capillaries. The nonwoven structure and morphology of fibres obtained by electrospinning in diversified conditions are illustrated in ***Figures 5 - 8***, showing sample pictures taken with the use of a JEOL SM 522 LV scanning microscope. Measurements of the fibre transverse dimensions were carried out by means of a computer program for image analysis, Lucia G. The biggest transverse dimension of the fibre cross-section was measured. For one sample measurements of 60 fibres were made on the basis of the longitudinal view of the fibres. The shape of the fibre cross-section area varied depending on the electrospinning conditions. In most cases the shape was irregular, similar to a circular or oval form. When the distance between capillaries was increased up to 6 cm, a flattening of the fibre cross-section and the forming of a ribbon was observed, as illustrated in ***Figures 5.c*** & ***6.c***. Moreover, separate ribbons were already observed in samples obtained at a distance of 3 cm between capillaries, as shown in ***Figure 5.b***. It was found that the supply voltage value and distance between the capillary tip and the collector did not influence the shape of the fibre cross-section area. Mean values of the fibre transverse dimensions are shown in ***Figure 9***. The mean fibre transverse dimension was equal to more than 1 µm, and the finest fibres, with a mean transverse dimension of 1.08 µm, were obtained in the following electrospinning conditions: distance between the capillary tip and collector – 15 cm, supply voltage – 15 kV, distance between capillaries – 3 cm.

The highest value of the fibre transverse dimension was observed in a sample obtained in the following electrospinning conditions: distance between the capillary tip and collector – 25 cm, supply voltage – 25 kV, distance between capillaries – 6 cm.

From ***Figure 9*** it can be concluded that an increase in the distance between capillaries causes an twofold rise in fibre transverse dimensions in comparison with values for fibres obtained at distances of 1 and 3 cm between capillaries. A larger average fibre transverse dimension can result from a change in the shape of the fibre cross-section area.

Conclusions

Larger distances between capillaries, even 6 cm, cause equipotential lines of the field to arrange themselves around all the capillaries in a similar fashion. The considerable deflection of lines around the boundary capillaries fades, and the electrostatic field strength values are higher and similar around all capillaries. Concurrently, the six-centimetre distance between the capillaries is conducive to generating interferences in the field in the area above the level of the polymer solution outflow. An increase in the distance between the capillary tip and the collector and an increase in supply voltage values do not essentially affect the field deformation around the boundary capillaries. An increase in the distance between the capillary tip and the collector causes a drop in the strength value of the electrostatic field that forms in direct proximity to the capillaries, regardless of their position to one another. In the case of an increase in the supply voltage value, an increase in the strength value of the electrostatic field around the capillaries was observed. The model distribution of the electrostatic field is reflected in how the electrospinning process proceeds and in results of the average fibre transverse dimension. In this study it was observed that the supply voltage value and distance between the capillary tip and the collector cause a change in the average fibre transverse dimension, but the character of these changes is difficult to define on the basis of three experimental points. For determination of the character of the changes, it is necessary to conduct more extensive investigations. The influence of these two parameters on the morphology of the fibres formed was not found. The biggest changes in the average fibre transverse dimension and shape of the fibre cross-section area were caused by changes in the distance between capillaries. At an increased distance between capillaries, fibres were obtained in form of ribbons.

Additionally, regardless of the distances between capillaries, fibres lie on the collector producing independent and non-overlapping fields. This phenomenon can be eliminated by changing the collector design.

The investigations carried out indicated without doubt that the shape of the electric field influences the character of the fibre obtained; however, to draw detailed, unequivocal conclusions further investigation is necessary as well as an analysis of the electric field distribution obtained by computer simulation and of the results of electrospinning.

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