Introduction

The development of electrospinning technique [1, 2] as a method of obtaining fibrous materials with micro-and nanofibres has shown usefulness for many applications in the last twenty years. In this method, a drop of polymer solution or melt is strongly deformed and stretched longitudinally by the action of an electrostatic field forming a fibre with a degree of stretch dating back thousands of times. Over the course of fibre formation, the stream of spinning solution quickly releases the volatile solvent and solidifies, which is the result of lowering the temperature in the case of melt solidification. The electrospun fibres are usually arranged randomly on the receiver substrate forming a non-woven fabric with varying degrees of consolidation. If the fibres are so thin that their diameters are expressed on a nanometric scale, the material produced reaches a particularly large free surface, also having pores of nanometre size. The ability to use this technique with natural polymers and their derivatives, as well as synthetic polymers and different additives, makes manufactured materials attractive both technically and biologically. Currently, a few directions of application can be seen [2, 3], including medical prosthesis, filtration and protective materials, sensors, electronics and optics, composites reinforced with nanofibres, etc.. Above all, they are biomedical materials, which have seen a lot of review work, for example electrospun nanofibres for regenerative medicine [4], tissue engineering and drug-delivery [5, 6], and electrospinning approaches towards scaffold engineering [7]. Much information on the use of electrospinning techniques and appropriate materials for biomedical applications can also be found in numerous articles [8 - 10]. The technique of electrospinning of fibrous materials using the most commonly encountered device based on a medical syringe and a needle is inefficient. This yield can be improved by using more advanced technical solutions, such as [11, 12]. It also happens that the fibrous materials obtained through electrospinning have unfavourable properties, as the consolidation depends on the diameter and shape of fibres. In many cases, these properties can be improved by using simple methods [13]. However, in general, meeting the high requirements of fibrous biomedical materials needs not only the use of suitable polymers, but also the achievement of appropriate structures (multi-layer, with aligned fibres, patterned, porous, etc.) and often require carrying out a specific biochemical and/or mechanical treatment (modification of post-electrospun materials) [14, 15]. This follows on from the basic features observed during the propagation of the cells on different substrates, where the migration is most likely along the direction of chemical, structural and mechanical properties of such substrates. Obtaining appropriate architecture of scaffolds in two or three dimensions appears to be the key for the proper growth of young tissue. At the same time, the technique of electrospinning has emerged as an interesting way of arranging nano- and micro-fibres according to different patterns on microscopic and macroscopic levels. This direction will no doubt be further developed and should bring many innovative solutions. One of the particularly important forms of organization of fibres in scaffolds is their common orientation. This is due to the fact that many tissues, such as nerve, skeletal and cardiac muscle, tendons, ligaments, and blood vessels, contain cells oriented in a highly aligned arrangement. In recent years, electrospun fibrous substrates of this arrangement have been used in many studies to investigate the interaction, reproduction and migration of living cells [14 - 17].

In the past ten years, many attempts have been undertaken to modify electrospinning systems in order to receive fibres ordered in one direction. Due to the arrangement and types of electrodes, these modifications can be divided into two main groups:

A – Stationary; many different configurations of electrodes [18 - 21]

B – Mobile;

1) With a rotating drum, e.g. [22, 23]

2) With a rotating drum and additional electrodes, e.g. [24, 25]

3) With a rotating thin wheel, e.g. [26, 27]

4) With a rotating multiframe, e.g. [3]

5) When combining in yarn, e.g. [28 - 30].

Electrospinning of Aligned Fibrous Materials on an Inner Rapidly Rotating Cone Surface

Abstract

The paper presents a new way for producing fibrous mats with aligned micro- and nanofibres by electrospinning technique using a fast rotating filter basket of a properly adapted commercial centrifugal juicer as the collecting electrode. This method was used to obtain the fibrous materials from polylactide (PLA) and polyvinyl alcohol (PVA) with different degrees of fibres aligned depending on the linear velocity of the rotating substrate. The highest degree of the fibre alignment, measured by the planar orientation factor, with a value of 0.8 - 0.9, was obtained at the linear speeds of the rotating substrate in the range of 22 - 36 m/s. Using the gravimetric method, the velocity of fibre formation during electrospinning was tested under different conditions, i.e. at the immobile substrate as well as rapidly rotating, reaching values ranging from 4 to 200 m/s. The investigations suggest that electrospinning of the fibres on the rapidly rotating substrate is not only a modification of their receiving, but also significantly changes the nature of the fibre formation in this spinning technique.

Key words: electrospinning, fibrous mat, polylactide, polyvinyl alcohol, rotating cone.
Each of these methods has advantages and disadvantages, which are presented in more detail in a previous review [31]. Probably the highest degree of ordering of fibres is obtained in devices such as in group A, where the electrodes are fixed in place and properly shaped. In practice, if a small sheet of paper with a slit cut to denote the first fibres is put into the electrospinning field, the predominant direction of their arrangement is perpendicular to the slit. In this way, a sample of single micro- or nanofibres can be prepared for further research, as is the case in our laboratory. However, this feature is maintained only at the initial stage of receiving fibres when the rising fibrous mat is still very thin. Further, with increasing thickness of the mat, the arrangement of the fibres reaches the standard chaotic character. Moreover, placing samples of other materials between the collecting electrodes in order to cover them with a layer of oriented fibres usually distorts the electrostatic field and makes it difficult to achieve good results. However, this method is interesting and deserves further study; in particular, as the reason for the appearance of fibre orientation has been paid little attention here [32].

Technical solutions contained in group B are based on forcing the parallel stacking of the fibres by snatching them in the direction of a fast-moving surface of the receiver. More precisely, this is the case for B1 and B3. Type B2 solutions are more complex, since they contain additional electrodes modifying the electrostatic field, as in group A. In turn, the methods of type B5 (and also B4) relate to the orientation of many fibres when they are mechanically combined into a beam in the form of yarn from the state of a random pile formed during electrospinning. Hence, the linear speeds of the moving parts in this method are usually not large. For example, in [28] the linear take-up speed was 5.79 m/hour (a yarn consisted of 100 fibres), and also [29] used a motorised take-up roller with a linear take-up speed of 0.05 m/s (a yarn consisted of 3720 fibres). However, the yarn was received much faster in [30], because the rate was from 0.9 to 14.9 m/s. Rotating equipment for the collection of the aligned fibres during electrospinning (method B, 1 - 3), used by different researchers, reached linear speeds of 1.4 m/s [22] and 11 m/s [27] up to 22 m/s [26], and they observed that the use of higher speeds gave a higher degree of ordering of the fibres. On the other hand, already at a linear speed 45 m/min (0.75 m/s) scattering of the fibres outside the receiving device was observed [23], probably as a result of air movement, or centrifugal force. It can also be expected that, if the substrate is moving too fast, the fibres will finally break. Such a case was seen at the breaking of single nanofibres during yarn receiving [30] and indirectly in the form of the multiple necking of nanofibres by too rapidly rotating the accumulating wheel [33]. It may seem obvious that the degree of orientation of received fibres is not only the result of the linear speed of the receiving device, but also the speed achieved by these fibres at the moment just before reaching the substrate, and more precisely, the mutual relationship between them. However, verification of this relationship is now hindered, because the velocity of electrospinning has not been systematically studied. According to the data found, measurements using fluorescent particles, found speeds at the initial stage of stretching of the spinning solution jet during electrospinning of 1 - 2.5 m/s [34] and 0.5 - 5 m/s [35]. Hence, one can only conclude that, at a later stage of the process, these speeds had to be much higher. Indeed, the average linear speed while collecting electrospun fibres, as estimated by the gravimetric method in [29], had a value of 186 m/s.

The aim of the present work is to investigate the possibilities of obtaining nanofibrous mats with aligned fibres from the use of a new system based on the rapidly rotating substrate in order to reduce the disadvantages described above. Studies refer also to the velocity of fibre formation in the final stage of electrospinning while collecting fibres on both mobile and immobile substrates, which should expand the limited knowledge on the subject.

Materials and methods

Electrospinning solutions

Two types of spinning solutions were prepared for electrospinning trials: 6 wt% polylactide (PLA) (Cargill, Mw = 98,000 g/mol) in a mixture of chloroform/dimethyl sulphoxide (DMSO) at a weight ratio of 90/10 and 10 wt% polyvinyl alcohol (PVA) (POCH SA, Mw = 72,000 g/mol) in distilled water. More details about these solutions can be found in our previous works [11, 36].

Standard research techniques

- The rotational speed of the movable element in the receiving device for aligned fibres (filter basket) was measured using a non-contact device ‘ZYLKOMAT I’ (Gossen’s, Germany).
- The thermal properties of the electrospun fibres from PLA were studied using a differential scanning calorimeter DSC ‘Diamond’ equipped with Pyris software, Perkin-Elmer (USA).
- The studies by scanning electron microscopy (SEM) for electrospin fibrous samples, after being gold sputter-coated, were performed using an electron microscope (Quanta 200, Fei Co., USA), equipped with the computer program, analySIS Docu (USA).

Gravimetric method for assessing the speed of electrospinning

The assessment of electrospinning speed by gravimetric methods can be easily performed, but the fulfilment of many assumptions can be embarrassing and lead to significantly reduced accuracy. If the fibres, electrospun from a single capillary, have a stable circular cross-section, characterised by a homogeneous supermolecular structure, and the stream of the spinning solution is not separated during stretching in the electrostatic field, making this the source of a single fibre, then the average value of the linear electrospinning speed can be obtained from the following Equation 1:

$$\frac{\Delta \rho}{\Delta \pi} = \frac{4}{\pi \cdot \rho \cdot t} \frac{\Delta m}{\Delta t}$$

where:
- $\Delta \rho/\Delta \pi$ and $\Delta m/\Delta t$ – the average speed of electrospinning in m/s and of the growth of weight of the received fibres in kg/s, respectively;
- $\rho$ - an apparent density of the fibres kg/m$^3$;
- $d$ - mean diameter of the fibres m;
- $\pi$ - constant, 3.14;
- if a spinneret with N capillaries was used for electrospinning, the result must be divided by $N$;
- if the stream of the spinning solution stretched during electrospinning was divided into $M$ parts, the result must be divided by $M$.

Using Equation 1 on the data obtained by us previously for electrospinning of PVA fibres [11], it can be estimated that in the sample where the average diameter was 0.53 $\mu$m, the speed of electrospinning was 60 m/s. However, in other tri-
als, where the PVA fibres had an average diameter of 0.23 µm, the electrospinning speed could be much higher. Of course, these results and the earlier cited values are credible only on the assumption that only one fibre is formed from one capillary, which is not always easy to determine.

Electrospinning technique for collecting aligned fibres based on the use of the inner surface of the rotating cone

As noted earlier, obtaining fibrous materials with aligned fibres by the electrospinning technique using a fast-rotating device causes the problem of maintaining the collected fibres on the surface. This problem increases in the case of coverings with aligned fibres, as larger samples of other materials allows many more fibres to be thrown out from the surface of the rotating element. Also, it must be added that the good results obtained for aligned fibres on the edge of the rotating disc [26, 27] apply only to very narrow samples, and the use of this method for additional items to be installed in such a way would be problematic. It now seems that, at least in part, these problems can be solved using the inner surface of a rapidly rotating cone. In particular, various commercial centrifugal juicers (produced for households) containing a rapidly rotating filter basket, which is well balanced, respectively-bearing and made of metal, with significant chemical resistance, can be adapted for this purpose. The principle of this form of collection of fibres during electrospinning is shown in Figure 1.

In the currently presented method, the centrifugal force significantly increases the emphasis of the sample on the inner lateral surface of the rotating filter basket, and thus increases the friction force, which significantly limits the possibility of ejecting the sample from the device, as shown in Figure 1. In the previously described methods for collection, aligned fibres used the outer surfaces of the rotating components, where the balancing of centrifugal force was a problem. Obviously some drawbacks can also be found in this new system, such as the possibility of the occurrence of air turbulence at high speeds, hindering a high degree of alignment of the fibres being obtained. The second drawback is the inconstancy of the linear speed of the substrate. The linear speed of the sample, \( V \), during rotation of the filter basket, depends on its location on the surface and, measured in m/s, is given by the Equation 2:

\[
V = 2 \cdot \pi \cdot r \cdot f
\]

where:
- \( r \) - radius of rotation measured by the distance of the sample from the axis of rotation of the filter basket in m;
- \( f \) - frequency of the filter basket rotation (revolutions per second) in 1/s;
- \( \pi \) - constant, 3.14

Thus, at large sample sizes, the degree of alignment of fibres on its surface may not be homogeneous. On the other hand, a situation where the occurrence of such
a gradient with regard to the arrangement of fibres could be beneficial cannot be excluded. The occurrence of a shielding effect of the electrostatic field inside the filter basket can also be expected if its shape is inappropriate, i.e. there will be a relatively small diameter and a large depth (Faraday shield). As a result, the electrospun fibres cannot be deposited at the bottom of such a filter basket. In our study we observed no such effect.

To build the device for receiving aligned fibres by electrospinning, a commercial centrifugal juicer (type 377 produced by Zelmer, Poland) was used. The filter basket in this device is characterised by the inlet diameter of about 110 mm and the inclination angle of the conical lateral surface, α (Figure 1), of approximately 55°. An adaptation of the juicer to the needs of electrospinning consisted mainly of the electrical connection between the metal parts of the basket and the metal drive axle, which was electrically grounded with the help of a sliding connection. In order to control the rotational speed of an electric motor, the device was combined with the power grid by the autotransformer. This solution reduces the engine power at lower voltages, but it is not important for the current application. The resulting relation between the rotational speed of the filter basket and the voltage of the power supply was determined experimentally using a non-contact instrument for measuring the rotational speed. The obtained data show that this speed grows linearly with supply voltage, with a small error, reaching more than 12,000 r.p.m. at a standard voltage of 230 V in the power grid. The final design of the device, after placing it in an enclosure, is shown in Figure 2.A.

Results and discussion

Electrospinning speed for the standard chaotic fibre production

Measurements of the speed of electrospinning at standard chaotic collection were performed for fibres from PLA and PVA using the gravimetric method. During electrospinning using a single needle (size 0.8 × 40 mm, pressure of extrusion of spinning solution in the range of 0 - 392 Pa (4 cm H₂O)) the applied voltage was 20 - 25 kV at a distance of 20 - 25 cm between the tip of the needle and the flat aluminium foil, which was used as the collecting electrode. The received fibre mass was obtained as the average linear speeds of electrospinning of the fibres, the samples of obtained fibrous mats were examined by SEM. Examples of SEM images of the surface, along with the histograms of measured fibre diameters, are shown in Figure 3. Based on the results obtained for the fibre diameters, and taking into account the fact that 0.0483 g of the PLA fibres, and 0.0303 g of PVA fibres were obtained during the one-hour exposure and, after adoption, that the apparent density of both types of fibres is approximately the same (1250 kg/m³), the average linear speeds of electrospinning of the fibres can be calculated using Equation 1. The speed for the PLA fibres is 4.1 m/s, and for the PVA fibres is 66.2 m/s. It should be noted that the PLA fibres are porous, as shown in the SEM image of their surface, in Figure 4. In turn, the DSC test performed on a sample of freshly electrospun PLA fibres, Figure 4, testifies to their low crystallinity, because the difference of their enthalpies of melting and cold crystallisation is as low as 8.5 J/g; hence, almost the entire crystalline phase was formed during heating of the sample in the calorimeter. Therefore, the above adopted density of the PLA microfibres corresponds to the amorphous state. However, in the case of PVA nanofibres, similar DSC determinations were not performed, since this poly...

Figure 3. SEM images of the electrospun fibrous mats and fibre diameter histograms for: PLA - top, PVA - bottom.

Figure 4. A - SEM image of the electrospun PLA fibres with visible pores. B - DSC curve of the PLA fibres at the first heating at a rate of 20 K/min.
mer is atactic and does not melt. These two features of the electrospun PLA fibres, and especially their porosity, make it accepted that the apparent density is overvalued to a certain extent for them, and thus the calculated speed of electrospinning should be higher. On the other hand, the fibres contain a lot of thickenings, which are not included in the estimation of their average diameter. In addition, fibre diameter in Equation 1 occurs in the square, which further increases the impact of the latter feature on reduction of the calculated speed. Finally, it appears that the impact of these factors can be largely abolished and the estimated speed of electrospinning of the PLA fibres is not burdened with too much error.

**Electrospinning of aligned fibres**

The electrospinning tests of fibrous mats with aligned fibres, using a rotating filter basket of a juicer, were carried out for the solutions of PLA and PVA. The device...
was set so that the needle was at a height of 20 cm above the basket in a middle position between its centre and the edge of its side, as shown in Figure 1. Electrospinning was usually conducted for about 40 min at a voltage of 20 kV. Other electrospinning parameters were the same as in the case of standard chaotic fibre receiving. In Figures 2.B and 2.C, pictures of the filter basket when the resulting electrospun fibrous material is removed and a sample of this material are shown, respectively. Additionally, an SEM image of the sample of standard non-woven polypropylene (PP) coated with aligned poly(ethylene oxide) (PEO) nanofibres is shown in Figure 2.D, as an example of the possibility of using this device for the surface modification of textile materials by nanofibrous coatings. During electrospinning, the linear speed at the selected location on the surface of a rotating filter basket increases linearly with the increase of its distance from the axis of rotation (Equation 2) and it can be expected that this will have a significant effect on the ordering of the fibres during collection. The relevant studies were carried out for electrospinning of PVA fibres at the filter basket rotational speed of 5860 r.p.m. In Figure 5, SEM images of fibrous samples taken from different places on the filter basket characterised by the linear velocity from 0 to 32.6 m/s are shown. The researches show that the electrospinning of PVA fibres by such a modified technique allows a noticeable alignment of the fibres at a linear velocity of receiving order of 20 m/s, and a satisfactory alignment at a velocity slightly above 30 m/s. These velocities are at least two times smaller than previously determined for the electrospinning of PVA fibres with chaotic collecting (66.2 m/s). If we assume that the final position of the fibres on the filter basket surface is a result of submission of their chaotic motion during formation in an electrostatic field and the rotation of the substrate, such a clear alignment of these fibres at these linear velocities should not be expected to occur. To examine the impact of the speed of fibre formation in electrospinning on the level of their alignment on the surface of the rotating filter basket, appropriate tests were performed for solutions of PLA and PVA, for which the speeds of electrospinning may vary by at least ten-fold. At the same time, the tests were performed at a wide range of rotating velocities of the filter basket. The results of SEM studies of the samples of fibres from PLA and PVA, taken from the same point in the basket, marked in Figure 5.D, are shown respectively in Figures 6 and 7. The process of progressive orientation of the fibres with increasing linear velocity of the rotating substrate, which is noticeable in the images, can be described quantitatively. However, due to difficulties in the precise determination of the direction of the linear velocity of the substrate during the observation of SEM, it was decided to refer a single fibre’s orientation to the direction of a designated arithmetic average of the angles for the entire population of fibres visible in the photograph. For this purpose, after measuring all of the angles for the population of fibres in the photo, the mean value was subtracted from each of them. Statistical results of the orientation of the fibres from PLA and PVA for different linear speeds of the substrate are shown.

**Figure 8.** Normalised histograms of the orientation angle - α, of the electrospun: a) PLA and b) PVA fibres at different linear velocities of the rotating substrate.

**Figure 9.** Planar orientation factor of the PLA and PVA fibres against the linear velocity of the rotating substrate.

**Figure 10.** Electrospinning speed and average PVA fibre diameter as a function of the linear velocity of the rotating substrate.
in Figure 8, respectively, in the form of histograms. To facilitate opportunities to compare, histograms were drawn after normalising the count frequency of fibres oriented in a specified angular range, $f_{\alpha}$, according to the Equation 3:

$$ f_{\alpha} = \frac{f_i}{\sum f_i} \times 100\% $$

where:

$$ \sum f_{\alpha} = 100\% $$

$f_i$ - number of fibres in the range of $\pm 10^\circ$ around the angle $\alpha$ from the set {$-100^\circ;\ldots;-20^\circ;0^\circ;+20^\circ;\ldots;+100^\circ$}.

In the case of normal distribution, all histograms would be symmetrical with respect to the value of $\alpha = 0^\circ$, and the normalised frequency, $f_{\alpha}$, should fall within the range of $\pm 90^\circ$. However, in practice, the events on the individual fibres cannot be missed, whose positions are close to the perpendicular in the most likely direction for the population of fibres visible in the photograph. In this case, a certain asymmetry in the distribution of events can occur, where the average and most probable angle will not overlap. Events beyond the angular range of $\pm 90^\circ$ can be then observed, but always only on one side of the distribution. Such situations occur for example in Figure 8, respectively, at linear velocities of 17.7 m/s and 10.7 m/s (also 32.6 m/s). To assess the degree of fibre alignment in the fibrous materials obtained, the single-numerical estimator was also used, which allows plotting function relationships. For this purpose, the flat orientation factor, $f_0$, was used here [37], calculated by the Equation 4:

$$ f_0 = 2 \times \langle \cos \alpha \rangle - 1 $$

where:

$$ \langle \cos \alpha \rangle $$ - arithmetic mean of the cosines of the angles of fibre orientation of the population. The $\alpha$ angles, as previously, were referenced to their arithmetic mean.

The planar orientation factor $f_p$, Equation 4, expresses the state of fibre orientation distribution in 2D; in cases where the fibre orientation is isotropic, the planar orientation factor value is $f_p = 0$, in cases of being in only one-direction (anisotropy), the value is $f_p = 1$, and when it is arranged in a rotation direction of $90^\circ$ for the direction of $f_p = 1$, then it is $f_p = -1$. It should be noted that a similar function of orientation known as Hermans factor was developed for uniaxial orientation, but it is not applicable here; its' possible use leads to incorrect results. The resulting alignment degree of the PLA and PVA fibres, depending on the linear speed of the substrate, is shown in Figure 9. It seems that comparison of the degree of the alignments of the PLA and PVA fibres based on organoleptic assessment of the SEM images and analytical results is consistent and leads to the conclusion that the state of the fibre orientation increases with the speed of the linear substrate, but only to a certain value, above which it begins to decrease. As shown in Figure 10, as outlined above, these relations are well described by quadratic functions. According to these approximations, the best alignment for PLA fibres, with an $f_p$ value of 0.80, was obtained at linear velocity of 36.5 m/s, and for PVA fibres, with an $f_p$ factor of 0.90, at 21.8 m/s. These figures confirm the previous observations of a lack of influence of the speed of fibre formation obtained in electrospinning with a chaotic method of fibre collection on the conditions at which the fibres are aligning. Therefore, the speed of electrospinning of PVA fibres was measured when collecting on a rotating filter basket, using the method according to the Equation 1. These results together with measured mean values of diameters of these fibres are shown in Figure 10. The relationships shown here indicate a three-fold increase in the velocity of fibre formation during electrospinning on the rotating substrate relative to the system when the substrate is fixed, and a simultaneous one and a half times reduction of the diameter of the fibres. Hence, it must be concluded that electrospinning of the fibres on the rapidly rotating substrate is not just another form of collection of these fibres, but significantly changes the nature of the fibre formation in this spinning technique.

Conclusions

The paper presents a new way for producing fibrous mats with aligned micro- and nano-fibres using an electrospinning technique with a fast rotating filter basket of a properly adapted commercial centrifugal juicer as the collecting electrode. This method allowed the fibrous materials from PLA and PVA to be obtained with different degrees of fibre alignment dependent on the linear velocity of the rotating substrate. The highest degrees of the fibre alignment, measured by the planar orientation factor, with a value of $0.8 - 0.9$, were obtained at linear speeds of the rotating substrate in the range of $22 - 36$ m/s. By using the gravimetric method, the velocity of fibre electrospinning was tested under different conditions. When the receiving substrate was immobile, the velocity of electrospinning was 4.1 m/s for PLA fibres and 66.2 m/s for PVA fibres, but this velocity exceeded 200 m/s for PVA fibres. The investigations suggest that electrospinning of the fibres on the rapidly rotating substrate is not only a modification of obtaining fibres, but significantly changes the nature of the fibre formation in this spinning technique. When using the device, over 100 cm$^2$ of mat with aligned fibres can be obtained at once. Moreover, during this electrospinning a beneficial system of forces acting on the produced fibrous material arises, which allows the layer of aligned fibres to cover other materials attached to the rotating substrate.

Acknowledgments

This work was carried out as part of the research project PBZ-MNiSW-01/II/2007 and the statutory project (P23) IBWCh/2012, both of which are supported by the Ministry of Science and Higher Education, Poland.

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Received 13.12.2010 Reviewed 29.06.2012

FIBRES & TEXTILES in Eastern Europe 2012, Vol. 20, No. 6B (96)