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Electrospinning: Nanofibre Production Method
Elektropredenje: postopek izdelave nanovlaken

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Abstract
Electrospinning is a method based on the use of electrostatic forces for producing continuous fibres with the diameter from between ten nanometres to some micrometres. Such fine fibres cannot be produced with conventional methods for the production of nonwovens. The nanofibres formed with electrospinning have an exceptionally large active surface area per mass unit (fibre surface at diameter of 100 nm equals 40 m^2/g) and the spinning process itself enables a planned formation of the web structure (e.g. planned size of pores in the web by adjusting the nanofibre diameter and fibre thickness). Nanofibres can be electrospun from synthetic or natural polymers and their blends, from polymers with various nanoparticles (metal, ceramic etc), active substances etc. We can fabricate individual fibres, as well as webs with a random or planned fibre arrangement. Fibres with a complex structure, e.g. core shell or hollow fibres, can be produced with a special electrospinning method. Regarding the advantages demonstrated by the nanofibres fabricated with electrospinning, this procedure has become an important part of research in several fields of use of technical textiles, e.g. shielding materials, air and oil filters in the car industry, agrotextiles and most of all medical textiles. The method can also be used in the production of batteries and photovoltaic cells. Apart from the apparatus designed for the research purpose in laboratories, pilot devices and the devices designed for the use in the industry can be found on the market. The paper comprises the introduction of the preparation procedure of nanofibres on an electrospinning apparatus, the morphological characteristics of fibres and the characteristics of electrospun webs in dependence of the conditions when forming fibres.
Keywords: nanotechnology, nanofibres, electrospinning, 3D-structure

Izvleček
Elektropredenje (electrospinning) je metoda, ki temelji na uporabi elektrostatskih sil za oblikovanje neskončnih vlaken s premerom med deset nanometri in nekaj mikrometri. Tako finih vlaken ni mogoče oblikovati s konvencionalnimi metodami izdelave netkanih tekstilij. Nanovlakna, oblikovana po postopku elektropredenja, imajo izjemno veliko aktivno površino na enoto mase (pri premeru 100 nm je površina vlaken 40 m^2/g), medtem ko sam postopek elektropredenja omogoča tudi načrtno oblikovanje strukture koprene (npr. načrtovanje velikosti por v kopreni z uravnavanjem premera nanovlaken in gostote vlaken). Po postopku elektropredenja lahko izdelamo nanovlakna iz sintetičnih ali naravnih polimerov in njihovih mešanic, polimerov z vključenimi različnimi nanodelci (kovinskimi, keramičnimi delci …), z vključenimi zdravilnimi učinkovinami itd. Oblikujemo lahko posamezna vlakna, kot tudi koprene z naključno ali načrtovano ureditvijo vlaken. Vlakna s kompleksno strukturo, kot na primer jedro-plašč ali votla vlakna, pa lahko proizvedemo po posebni metodi elektropredenja. Glede na prednosti, ki jih izkazujejo nanovlakna, oblikovana po postopku elektropredenja, je postal ta postopek zelo pomemben del raziskav na številnih področjih uporabe tehničnih tekstilij, kot so zaščitni materiali, zračni in oljni filtri za avtomobilsko industrijo, agrotekstilije in predvsem na področju medicine. Metoda je uporabna tudi za izdelavo materialov za baterije in fotovoltaične celice. Poleg naprav, ki so namenjene raziskavam v laboratorijskih obsegih,

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pa so na trgu že tudi pilotske naprave in takšne, ki so namenjene tudi industriji. V prispevku bomo predstavili pripravo nanovlaken na elektropredilni napravi, morfološke značilnosti vlaken in lastnosti izdelanih kopren v odvisnosti od pogojev oblikovanja vlaken.
Ključne besede: nanotehnologija, nanovlakna, elektropredenje, 3D-struktura

1 Introduction

Electrospinning is a spinning technique with a unique approach using electrostatic forces to produce fine fibres from polymer solutions or melts. Fibres produced with electrospinning have a thinner diameter (from nanometre to micrometre) and a larger surface area than those obtained with conventional spinning processes. The electrospinning process is conducted at room temperature under atmospheric conditions. The typical set up of electrospinning apparatus basically consists of three major components (cf. Figure 1): feeding unit (e.g. pipette dip), high voltage power supply (15–25 kV) and a grounded collecting plate (usually a metal screen or plate that can be covered with a fabric or a rotating spindle) [1–3]. Before electrospinning, polymers are either completely dissolved in appropriate solvents or they are melted. The polymer fluid or melt is then introduced into the capillary tube for electrospinning. However, some polymers may emit unpleasant or even harmful smells, therefore, the processes should be conducted within chambers having a ventilation system [3, 4].

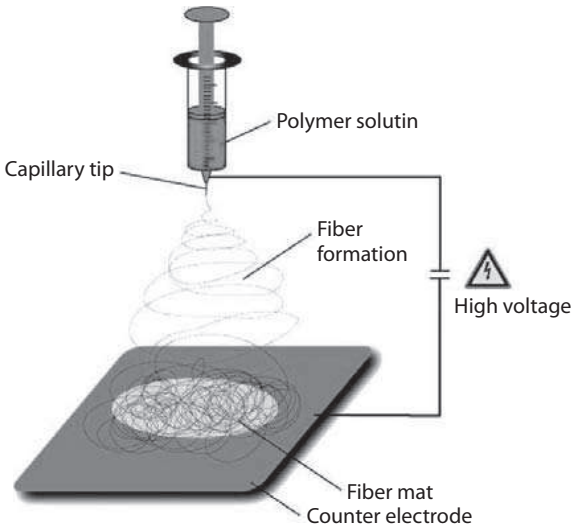

Figure 1: Set-up of electrospinning apparatus [7]

In the electrospinning process, a high voltage is used to create an electrically charged jet of a polymer solution or melt out of the pipette. The polymer is ejected from a needle with an inner diameter between 0.5–1.5 mm [5].

2 Jet initiation

When an electrical force is applied to an ejected polymer solution, its pendant droplet (cf. Figure 2) at the tip of the needle is deformed. Due to the application of the electrical field, a charge is induced on the surface of the drop. This charge offsets the forces of surface tension and the droplet changes its shape from hemispherical to conical. This conical shape of the ejected solution is typically referred to as Taylor cone. When the droplet is subjected to an external electric field, the conical surface is formed with an angle of 49.3° [2, 5, 6]. When the intensity of the electric field (V) attains the critical value (Vc), the electrostatic forces overcome the surface tension of the polymer solution and force the ejection of the liquid jet (cone-jet) from the tip of the Taylor cone. The highest charge density is present at the tip of the cone from where the jet is initiated. Cone-jet accelerates towards the grounded target collecting plate [2, 5, 6].

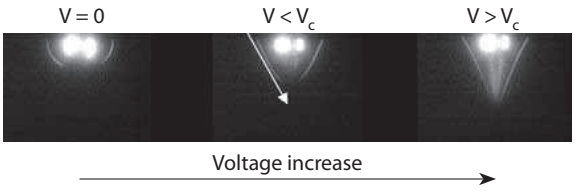

Figure 2: Formation of Taylor cone [5]

3 Jet thinning and jet instabilities

The discharged polymer solution or melt jet is only stable at the tip of the spinneret and after that, the jet undergoes an instable and a rapid whipping elongation process in the space between the capillary tip

and the collector, which allows the jet to become very long and thin (cf. Figure 3) [3, 4, 7]. On the way to the counter electrode, the solvent evaporates (or the melt solidifies) and solid fibres with diameters ranging from micrometres to nanometres are precipitating with high velocity (49 ms^{-1} or more) on the counter electrode [7].

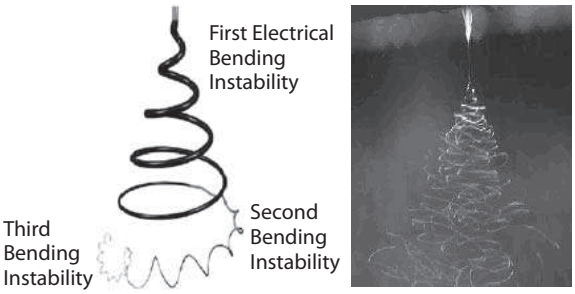

Figure 3: Scheme and photography of jet [7]

The jet follows a direct path towards the counter electrode only for a certain distance, while it then significantly changes its appearance. The straight jet turns laterally and forms a series of loops in the horizontal plane (cf. Figure 3). When the polymer jet becomes very long and thin, the time required for excess charge to redistribute itself along the full length of the jet becomes longer. The location of the excess charge then tends to change with the elongation. The repulsive Coulomb forces between the charges carried with the jet elongate the jet in the direction of its axis until the jet solidifies. This leads to an incredibly high velocity at the thin leading end of the straight jet. As a result, the jet bends and develops a series of lateral excursions that grow into spiral loops (cf. Figure 3) [6]. The loop diameters increase with time during the motion forming a cone opening towards the counter electrode. During this process, typical stretching ratios are in the range of 10^5 and stretching rates are up to 10^5 s^{-1} [7]. Such values are not accessible with any other methods, e.g. fibre extrusion followed by mechanical stretching. The high stretching rates are reflected in the morphology of the evolving fibres (e.g. high degree of molecular average and crystalline orientation).

The travelling liquid jet stream is subjected to a variety of forces with opposing effects; as a result, various fluid instabilities occur at this stage [6]. Hohman et al [8] and Shin et al [9] reported of three types of modes which are unstable, i.e. the Rayleigh mode, which is the axisymmetric extension of the classical Rayleigh instability when electrical effects are important, the axisymmetric conducting mode and the whipping conducting mode. Which instability dominates depends strongly on the surface charge density and the radius of the jet [10].

3.1 Jet splaying

Splaying occurs in the region of Rayleigh instability where the radical forces from the electrical charges carried by the jet become larger than the cohesive forces within the jet. The electrostatic repulsion of the charges in the jet tends to increase its surface area. On the other hand, surface tension tends to reduce the total surface area of the jet. Therefore, instability occurs, which causes the jet to break up into droplets, each with the surface area minimizing the spherical shape. A single jet divides into many charged jets (of approximately equal diameter and charge per unit length) before fibres 'loading' on the collector (cf. Figure 4). Splaying, together with the elongation due to the acceleration of the jet by electrical forces, forms unusually small diameter fibres [10].

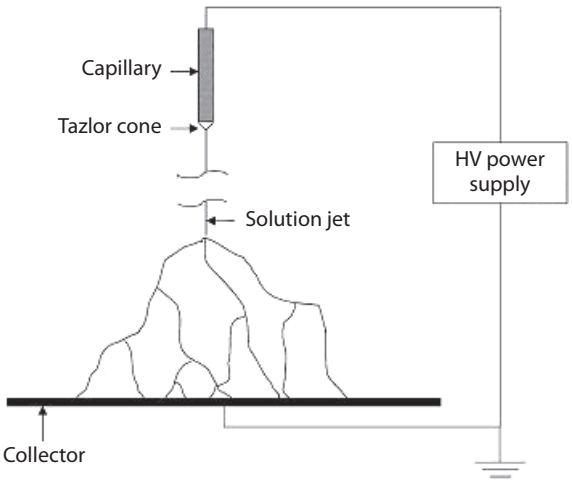

Figure 4: Jet splaying in region of Rayleigh instability [10]

3.2 Jet branching

During fibre formation, some additional irregularities can be observed. A thin branching that is nearly perpendicular to the thicker primary jet is shown in Figure 5. The formation of branches can be observed more frequently in more concentrated and more viscous solutions, and when the electric field higher than the minimum required for producing a single jet was used. A formation of many branches

was observed when the diameter of the jet was relatively large. The phenomenon was reported for electrospun solution of polycaprolactone in acetone (the concentration of polycaprolactone was 15%) (cf. Figure 5) [11].

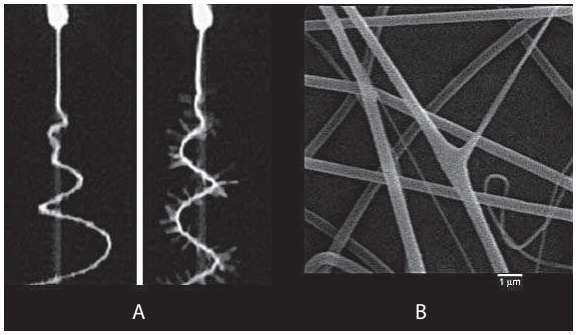

Figure 5: A) Branches on electrical bending coils of jet of polycaprolactone dissolved in acetone [11]; B) SEM image of branched fibre [7]

3.3 Fluid jets with thin skin

The formation of a skin on a jet is also a type of irregularity and is observed as a thin, mechanically distinct polymer skin on the liquid jet. After the skin is formed, the solvent inside evaporated. Atmospheric pressure tended to collapse the tube formed by the skin as the solvent evaporated. The circular cross section became elliptical and then flat, forming a ribbon with a cross-sectional diameter nearly the same as the diameter of the jet (cf. Figure 6 (a–d)). Sometimes, small tubes formed at each edge of the ribbon with the web made from the skin connected the two tubes (cf. Figure 6 (e)). As the collapse occurred, an electrical charge distributed uniformly on the cylindrical jet tended to flow to the edges of the ribbon, where it produced a lateral force that favoured a collapse. The web in Figure 6 (e)

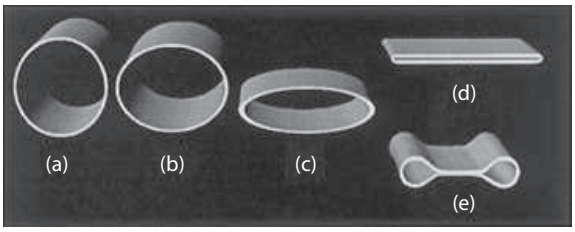

Figure 6: Collapsing of jet skin [12]

formed after atmospheric pressure forced the skin into contact, initially at separated points. Then, cohesive forces tended to increase the area of the web by sticking together the skin from opposite sides of the tube. The stiffness of the skin and the self-repulsion of the charge on each of the small tubes resisted the formation of flat ribbons of the sort shown in Figure 6 (d). These tubes might be hollow or filled [11, 12].

4 Nanofibre deformations

Nanofibres formed with electrospinning can have several deformations: fibres with beads arranged like pearls on a string, fibres like flat ribbons, very broad fibre diameter distribution. The deformations in fibre shapes and dimensions depend on a varied parameter, e.g. solution parameters, process parameters and ambient parameters (cf. Table 1).

As long as a polymer can be electrospun into nanofibres, ideal targets would be in that: (1) the diameters of the fibres are consistent and controllable, (2) the fibre surface is defect-free or defect-controllable, and (3) continuous single nanofibres are collectable. However, researchers so far have shown that these three targets are by no means easily achievable [13].

Table 1: Parameters affecting electrospun nanofibre formation

Solution parameters	Process parameters	Ambient parameters
Solution viscosity	Shape of collector	Environment temperature
Solution elasticity	Geometry of spinneret	Environment humidity
Solution surface tension	Solution flow rate	Air velocity
Conductivity of polymer, solvent, solution	Tip to collector distance	
Solution temperature	Applied voltage	
Added salts to solution		
Vapour pressure of solution		

4.1 Solution concentration and viscosity

It has been found out that the polymer concentration affects the formation of beads. Fong et al [14] have reported that a higher polymer concentration resulted in fewer beads. In their experiments with the PEO polymer, the polymer concentrations of 1–4.5 wt% were used. Figure 7 shows the representative images of beads and beaded fibres for the solutions with a range of viscosity (from 13 mPas to 1.25 Pas). The lowest viscosity 13 mPas corresponds to the lowest concentration 1 wt% PEO concentration, whereas the highest viscosity, i.e. 1.25 Pas, corresponds to 4 wt% concentrations. Beads and beaded fibres are less likely to be formed for the more viscous solutions. The diameter of the beads becomes bigger and the average distance between beads on the fibres longer as the viscosity increases. Meanwhile, the shape of the beads gradually changes from spherical to spindle-like.

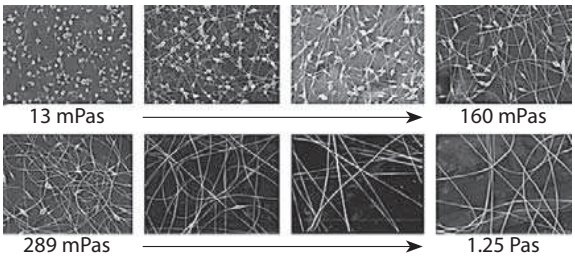

Figure 7: Effect of polymer concentration on nanofibres formation [14]

The solution viscosity has been strongly related to the concentration of the solution. An increase in solution viscosity or concentration gives rise to a large and more uniform fibre diameter. In electrospinning, the viscosity of a solution plays an important role in determining the range of concentrations from which continuous fibres can be obtained. For the solution of low viscosities, surface tension is the dominant factor and just beads or beaded fibres are formed while above critical concentration, a continuous fibrous structure is obtained and its morphology is affected by the concentration of the solution [3].

4.2 Solution molecular weight

The molecular weight of the polymer has a significant effect on the rheological and electrical properties, e.g. viscosity, surface tension, conductivity and dielectric strength. It has been observed that a too low molecular weight solution tends to form beads rather than fibres and a high molecular weight solution gives fibres with large average diameter. The molecular weight of the polymer reflects the number of entanglements of polymer chains in a solution, thus solution viscosity. Chain entanglement plays an important role in the processing of electrospinning (cf. Figure 8) [15]. Gupta et al have synthesized PMMA varying in molecular weight from 12.47 to 365.7 kDa to investigate the effect of molecular weight of the polymer, and they found that as the molecular weight increased, the number of beads and droplets decreased [16].

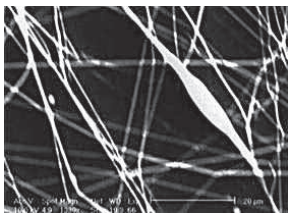

Figure 8: SEM of variable diameter formation in electrospun nanofibres [15]

4.3 Voltage dependence

Deitzel et al [17] investigated the influence of the electrical charge which was applied for electrospinning on the morphology of PEO nanofibres. They reported that with the increase of the electrical potential, the resulting nanofibres became rougher. These results are shown in Figure 9. It can be seen that an increasing electrospinning voltage changed the shape of the surface from which the electrospinning jet originates. This shape change, which corresponds to a decrease in the stability of the initiating jet as the voltage is increased, has been correlated with an increase in the number of bead defects forming along the electrospun fibres.

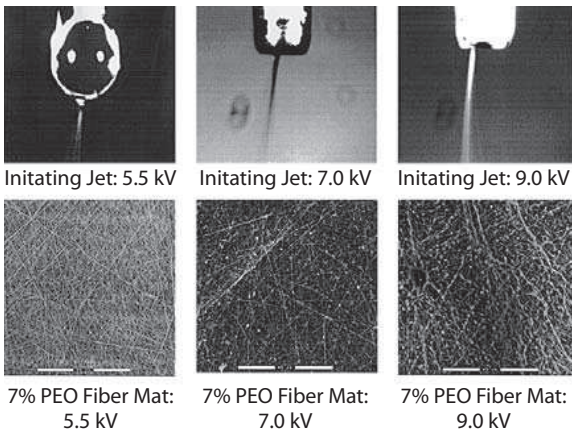

Figure 9: Effect of applied voltage on nanofibre formation [17]

4.4 Tip to collector distance

The distance between the tip and the collector controls the fibre diameter and morphology. A minimum distance is required to give the fibres sufficient time to dry before reaching the collector, otherwise with distances that are either too close or too far, beads have been observed [18]. The effect of the tip and collector distance on the fibre morphology is not as significant as other parameters; nevertheless, an optimum distance between the tip and collector which favours the evaporation of a solvent from nanofibres is still required.

4.5 Ambient parameters

Apart from the solution and processing parameters, there are also ambient parameters that include humidity, temperature etc. Studies have been conducted to examine the effects of ambient parameters (i.e. temperature and humidity) on the electrospinning process. The effect of temperature on polyamide-6 fibres has been investigated from 25 °C to 60 °C and it has been established that with an increase in temperature, there is a yield of fibres with decreased fibre diameter, and they attributed this decline in diameter to the decrease in the viscosity of the polymer solution at increased temperature [19].

The effect of humidity on the formation of polystyrene fibres has been studied by Casper et al [20]. They established that the variation in humidity while spinning polystyrene solutions has shown that by increasing humidity there is an appearance of small circular pores on the fibre surface; a further increase in humidity leads to the pore coalescence. At very low humidity, volatile solvent dries rapidly since the evaporation of the solvent is faster.

5 Large scale nanofibre production

Given the advantages of the high surface area of nanofibres for various applications, there is a huge interest in the nanofibre production at a commercial scale. Important criteria for determining the rate of fibre production include (i) concentration, (ii) the volume of solution forming spinning jet and (iii) the density of spinning jets [21]. The concentration of the solution determines the mass of fibres that can be produced per spinning jet. It is also important to consider the volume of the solution, since excess solution that does not take part in the spinning will either coagulate at the spinneret tip or drip onto the collector. The density of spinning jets refers to the number of spinning jets per unit area.

The formation of nanofibres by electrospinning from a single syringe has a low production rate (typical rates are 0.1–1 g/h); therefore, the industrial devices for large-scale nanofibres production were developed [22]. The scale-up possibilities can base on the multiplication of the jets using multi-nozzle construction (cf. Figure 10). In conventional spinning, the density of spinning jets is limited by the number of orifices that can be forced in the spinneret. In electrospinning, there is a limit to the number of orifices that can be placed in a given space, which is imposed by the bending instability of the electrified jet. When multiple orifices are used, the electrospinning jets repel each other increasing the distance between the deposited fibres [21]. However, the number of jets needed to reach economically acceptable productivity is very high, typically thousands, which can cause several problems with reliability, quality, consistency and machine maintenance (cleaning).

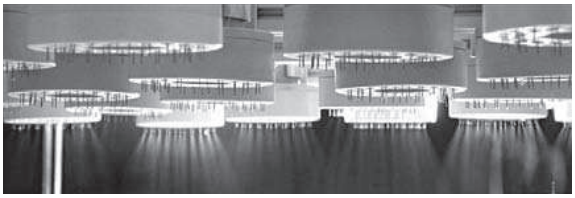

Figure 10: Multi-nozzle construction [24]

5.1 Nozzle-less technology

Recently, nozzle-less electrospinning appeared as an alternative electrospinning technology with the purpose of producing nanofibres on a large scale from a compact space. Nozzle-less electrospinning is featured as electrospinning of nanofibres directly from an open liquid surface. Numerous jets are formed simultaneously from the needleless fibre generator without the influence of capillary effect that is normally associated with needle-like nozzles. Since the jet initiation in nozzle-less electrospinning is a self-organized process which happens on a free liquid surface, the spinning process is hard to control [23]. The method that introduces the formation of many Taylor cones from a free liquid surface (nozzle-less electrospinning) was patented by Jirsak et al and the machines called NanoSpider are developed by the

Table 2: Comparison between nozzle electrospinning and nozzle-less electrospinning [21]

Process	Nozzle electrospinning	Nozzle-less electrospinning
Advantages	✓ Spinning solution with a wide range of viscosity ✓ Spinning at relatively low voltage ✓ Collector can be placed in any direction relative to the nozzle ✓ Fabrication of fibres with various configurations (e.g. core shell, multicomponent, hollow fibre) ✓ Easy to translate experimental data from spinning with a single needle nozzle	✓ Easy maintenance ✓ Easy to provide sufficient solution
Disadvantages	– Electrical field interference between nozzles – Difficult to maintain (cleaning of nozzle) – Difficult to maintain a uniform feed rate through each orifice	– Very high voltage required – Difficult to maintain consistent solution viscosity owing to solvent evaporation

ElMarco company (Liberec, Czech Republic). These NanoSpiders have the production rate of ~100 g/h per meter [22].

The main advantage of this technology is that the jets are initiated naturally in the optimal positions. The jet formation in needleless electrospinning has been proposed to follow four steps: (1) a thin layer of polymer solution is formed on the spinneret surface as a result of its partially immersion in the solution and rotation; (2) the rotation also causes perturbations on the solution layer thus inducing the formation of conical spikes on the solution surface; (3) when a high voltage is applied, the spikes concentrate electric forces thus intensifying the perturbations to form "Taylor cones"; (4) jets are stretched out from the "Taylor cones" and finally result in fibres (cf. Figure 11) [23]. The jets are distributed over the electrode surface with certain periodicity. This is one of the main advantages of nozzle-less electrospinning: the number and location of jets is set up naturally in their optimal positions. In the case of multi-needle spinning heads, the jet distribution is made artificially. The mismatch between the 'natural' jet distribution and the real mechanical structure leads to instabilities in the process, and to the production of nanofibre layers which are not homogenous [22]. NanoSpider has shown the capability of producing nanofibres from both polymer solutions and polymer melts.

Figure 11: Electrospinning using rotating drum dipped in polymer solution [22]

Several types of rotating electrodes for free liquid surface electrospinning have been developed (cf. Figure 12). However, the drum type is still one of the most productive.

Figure 12: Different rotating electrodes for nozzle-less electrospinning device [22]

The nozzle-less principle using rotating electrodes has been developed into commercially available laboratory and industrial scale-up (cf. Figure 13).

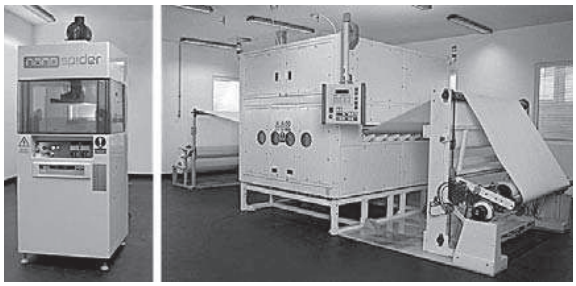

Figure 13: Laboratory and industrial scale-up electrospinning apparatus [22]

6 Conclusion

Electrospinning is a promising and effective method which offers several possibilities for the creation of high added value products for various applications. The possibility of using several different polymers for the fibre formation enables potential applications of electrospun fibres in diverse fields, e.g. tissue engineering, medical textiles, filtration materials etc.

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