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Advanced centrifugal electrospinning setup

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ABSTRACT

An advanced centrifugal electrospinning setup is proposed for the fabrication of non-woven textiles from polymer solutions using a pivotal feed unit and stationary collectors of large diameters and different designs: cylindrical collectors or collectors consisting of circularly arranged metal strips. The use of a collector composed of strips enables the production of aligned fibers. The feed reservoir is supplied with easy to handle interchangeable nozzles with the possibility of varing the nozzle-to-collector gap distance. The simultaneous use of four nozzles results in enhancement of the production rate and shortens the time for fabrication of a denser mat with a large surface area and enhanced exploitation properties. The special construction of the reservoir provides for its negligible dead volume.

1. Introduction

Micro- and nanofibers possess remarkable properties owing to their ultrafine structure. Mats can be prepared by electrospinning of solution or melt and they find versatile applications such as drug carriers, wound dressings, cell and tissue engineering scaffolds, filters, etc. [1]. Depending on the collector type, randomly deposited (stationary collector [2] and revolving disc [3]) or aligned fibers (rotating drum collector [4]) have been obtained. Aligned fibers have been successfully obtained using a static collector made up of two parallel blades that have been designed to increase the transversal electric field across the gap [5]. Laboratory scale electrospinning has gone through stages of improvement. Nonetheless, a major drawback of the process is its relatively low productivity. The preparation of mats with adequate thickness and a surface area of ca. 500–600 cm^2 takes about 6–8 h. One of the latest novelties is electrospinning combining the use of an electrical and a centrifugal field. The combined use of the electrical and the centrifugal field was reported for the first time by Liao et al. [6]. The centrifugal field ensures additional stretching of the spinning jet, thus leading to further orientation of the polymer chains in the nanofibers. That apparatus has not been commercialized and has several disadvantages. The reservoir containing the solution is connected to a high voltage power supply through a contactor, which can generate sparks upon rotation as a result of friction, thus creating an important risk of inflammation of volatile solvents. A single spinneret is utilized in that apparatus, and at higher rotational speed this can break the balance of the feeding unit. The mode of attachment of the spinneret to the reservoir body (by means of a wire) does not meet the technical standards. Nor the issue of easy control on the nozzle-to-collector distance, nor the use of multiple-needle feed unit still has been solved.

In this study we propose an advanced apparatus for electrospinning in an electrical and a centrifugal field. When 2, 3 or 4 nozzles with appropriate disposition are used, a perfect balance of the reservoir is achieved. The attachment of the needles to the reservoir body is achieved through counter nuts, ensuring the absence of undesired and uncontrolled leakage of the polymer solution. The use of a greater number of nozzles can shorten the time for fabrication of the mats. A feeding unit with an optimal design providing negligible dead volume and optimal disposition of the nozzles is described.

2. Experimental

Polyacrylonitrile (PAN) of $\overline{M}_v = 45{,}000$ g/mol was used. The electrospinning was performed at a nozzle-to-collector distance of 13 or 17 cm, a voltage of 40 kV, a rotational speed of the reservoir of 1900 rpm using 1, 2, 3 or 4 nozzles and a PAN concentration of 17 wt% in dimethylformamide (DMF). The fiber morphology was observed by scanning electron microscopy (SEM, Philips SEM 515) and assessed using reported criteria [7]. The mean fiber diameter was determined by measuring at least 20 objects from a SEM micrograph using Image J [8]. The tensile characteristics of strips of mats (20×60 mm^2) were measured longitudinally using a Zwick/Roell Z 2.5 apparatus load cell of 2 mV/V, type Xforce P, nominal force of 2.5 kN, test Xpert I at a

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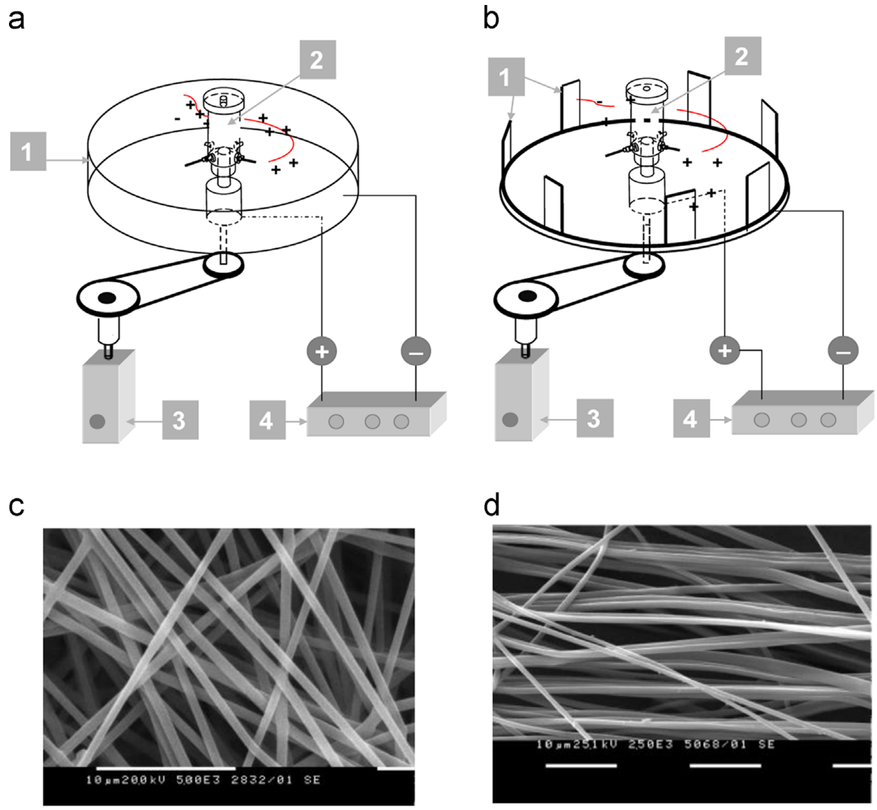

Fig. 1. Schematic of the electrospinning setup, apparatuses with stationary collectors (1) and a rotating feed unit (2), electric motor (3) and a high-voltage power supply (4); cylindrical collector (a) and collector composed of strips (b). SEM micrographs of mats deposited on the cylindrical collector (c) and obtained between the strips (d).

stretching rate of 20 mm/min and a temperature of 21 °C on at least 10 measurements.

3. Results and discussion

Centrifugal electrospinning setup: The feeding unit consists of a reservoir for the polymer solution supplied with 1, 2, 3 or 4 nozzles. The rotary motion of the reservoir is achieved by a belt-coupled electric motor (up to 3000 rpm). When higher rotational speed is required, the reservoir axis can be connected through a special gear to a high speed electric motor. The axis of the reservoir is attached through a bearing to the console mounted on the stand of the electrospinning setup. The power supply is connected to the metal console, which ensures that high voltage is transmitted to the reservoir containing the polymer solution (Fig. 1a and b).

Fiber morphology: In the present study we have obtained fibers deposited on two types of interchangeable stationary collectors, the first one being a cylindrical collector with a diameter of 45 cm for the fabrication of non-woven textile with a large area of 2200 cm^2. The collector is manufactured from an aluminum alloy sheet and aluminum foil can be fixed on its internal side for recovery of the mat (Fig. 1c). The collector is suitable for one-step fabrication of composite mats: electrospinning of dispersions of nano-sized metal or oxide particles in polymer solutions. At higher rotational speed a substantial part of the fibers is likely to be aligned in the direction of rotation of the reservoir. The second one is a collector consisting of circularly arranged metal strips (8 or 16; 35 × 150 mm^2), which is used for the preparation of mats composed of aligned fibers (Fig. 1d). The number of the strips and the distance between them can be easily altered, thus allowing the preparation of mats of aligned fibers. Combining these methods enables the fabrication of new generation, large-sized and multi-layered fibrous materials of a unique 3D hierarchy of successively deposited random or aligned nanofibers.

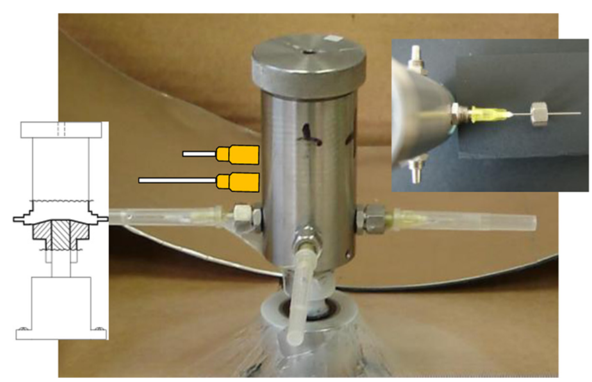

Fig. 2. Photograph of the stainless steel reservoir with negligible dead volume (left) and tips with needles and mounted counter nuts (right).

The average diameter of PAN fibers decreases upon increasing the tip-to-collector gap from 700 ± 85 nm to 550 ± 90 nm at collecting distances of 13 cm and 17 cm, respectively. The mat area increases upon increasing the number of the nozzles. When using 4 nozzles, the time required for the preparation of a mat with large surface area (2200 cm^2) can be reduced to less than 20 min. The reservoir ensures maximum utilization of the spinning solution with minimal “dead volume”, easily changeable nozzles and regulation of the tip-to-collector distance. The reservoir (43 cm^3) is supplied with a cover in order to avoid evaporation of the volatile solvents. A small aperture on the cover allows adding fresh polymer solution without opening the reservoir. Nozzle mounting is easily performed – standard medical needles

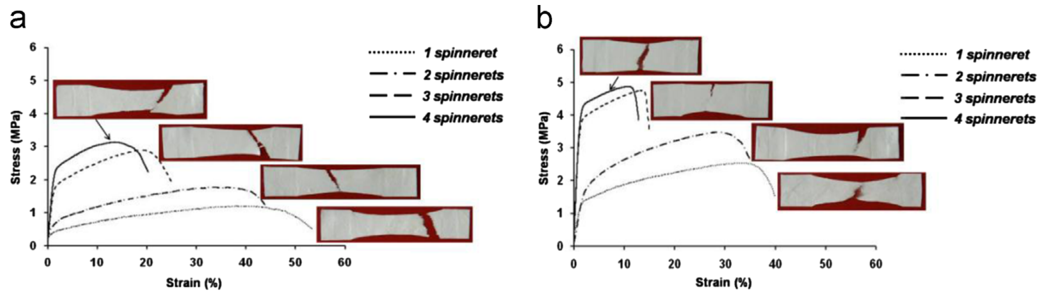

Fig. 3. Stress/strain curves of PAN mats prepared by centrifugal electrospinning at a tip-to-collector distance of 13 cm (a) and 17 cm (b).

are placed on cone-like tips and are fixed to the reservoir body via threaded nuts (Fig. 2).

Mechanical properties: The performed studies demonstrate that when 1 or 2 nozzles are used at a nozzle-to-collector distance of 13 cm, the mats undergo significant deformation at break (Fig. 3a), 55% and 45%, respectively. Mats obtained using 3 or 4 nozzles have greater strength and break at a smaller deformation of 20%. The mats prepared using 4 nozzles manifest the greatest strength with Young's modulus of 170 ± 12.4 MPa and breaking stress of 2.3 MPa. In terms of strength, they are followed by 3-nozzle mats (Young's modulus 146 ± 10.7 MPa, breaking stress 2.1 MPa) and 2 nozzle mats (Young's modulus 53 ± 3.9, breaking stress 1.2 MPa). The mats fabricated using 1 nozzle are characterized by the greatest strength with 26 ± 1.9 MPa Young's modulus and 0.5 MPa breaking stress. This result is not an unexpected one because at a higher number of nozzles, the number of entangled fibers increases.

The increase in the tip-to-collector distance allows mats with enhanced strength to be obtained (Fig. 3b). Mats collected at a distance of 17 cm have smaller elongation at break compared to those at 13 cm. An increase in the number of nozzles reduces the deformation at break of the mats, at 40%, 35%, 15% and 12% for 1, 2, 3, or 4 nozzles mats, respectively. The strength of the mats increases upon increasing the number of the nozzles. Mats obtained using 4 nozzles demonstrate the greatest strength (Young's modulus 250 ± 18.3 MPa, breaking stress 3.7 MPa) compared to Young's modulus 230 ± 17.2 MPa, 94 ± 7.1 MPa and 70 ± 5.1 MPa and a maximal elongation at break of 3.5 MPa, 2.7 MPa and 1.5 MPa, for 3, 2, and 1 nozzle(s) mats, respectively. It is known that under the combined action of an electrical and a centrifugal field the jet stability increases [6] and significant stretching and orientation of the polymer macromolecules along the axis of the fibers takes place. Fibers with such orientation are likely to display better tensile properties even in cases when their diameters are smaller.

4. Conclusion

In this study, we report an improved apparatus for preparing micro- and nanofibrous mats by centrifugal electrospinning. Two stationary collectors of large-diameter design are proposed in order to obtain mats of random or aligned fibers. The multi-nozzle electrospinning feed unit leads to a significant increase in fiber productivity for shorter time. The designed reservoir allows maximum utilization of the spinning solution with negligible dead volume. The use of 3 or 4 nozzles or the increase in the tip-to-collector distance enhances the strength of the mats.

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