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Controlled Deposition of Polyamide Nanofibers Through an Innovative Electrospinning Device

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Abstract

Electrospinning is a straightforward, cheap and unique method to produce novel fibers with diameter in the range of 100 nm and even less. Those nanofibers have a wide variety of applications such as: filters, membranes, composite reinforcement, drug delivery, protective barriers, sensors, wound dressings and tissue-engineered scaffolds where their unique properties contribute to product functionality. However, this process is characterized by a chaotic oscillation of the electrospinning jet which leads to the formation of beads an uneven nanofiber. This research work envisages the development of an apparatus to control the deposition of electrospun nanofibers through the use of a series of charged metal rings and the addition of a secondary power source, which enables a greater control over the polymer jet stream.

Keywords: Nanotechnology, Electrospinning, Nanofibres, Controled deposition, Polyamides

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1. Introduction

It is common knowledge that electrospinning is an effective process to produce nanofibers. Amidst many other emerging applications, the production of filters, membranes, composite reinforcement, drug delivery, protective barriers, sensors, wound dressings and tissue- engineered scaffolds produced with this technology is already a consolidated market. Nanofibers are valued for their small pore size and high surface area [1–3].

The electrospinning process results from the ejection of a polymer fluid jet, from a capillary by the action of a strong electrostatic force, and its deposition onto a grounded target. An electrospinning apparatus, in its basic form, is comprised by a support containing the polymer solution (pippete or syringe), a pump, two electrodes, a DC voltage power supply in the kV range (typically 5kv to 25kv) and a grounded collector medium.

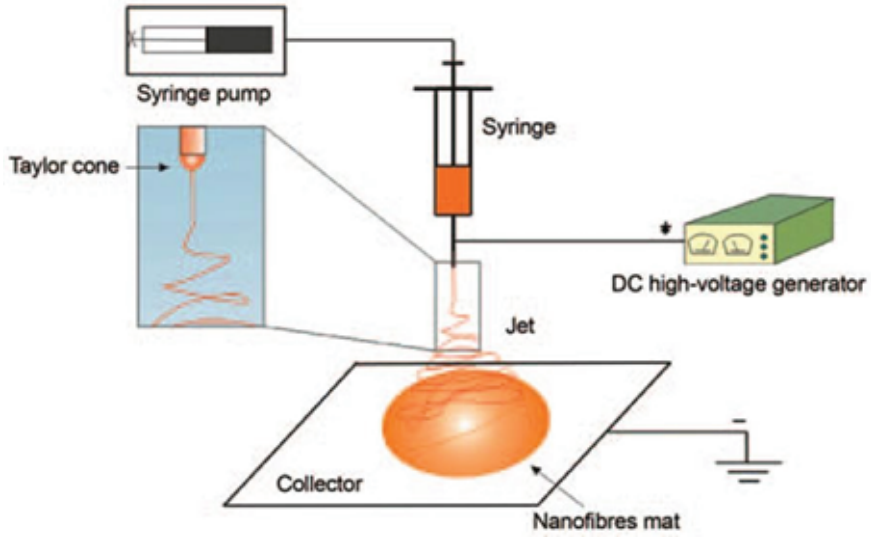

Figure 1: Schematic view of the electrospinning process

2. Experimental

In this study PA nanofibers[4], with novel morphological properties, were developed via a newly designed electrospinning apparatus which allows a greater control on the oscillation of the electrospinning jet.

PA 6 and PA 6,6 polymeric solutions were prepared by dissolving each polymer into a mixture of 50% ratio of formic acid (Merck) and 50% of acetic acid (Sigma Aldrich). Afterwards 1% of NaCl was added to increase the electrical conductivity of the attained solution [5,6].

The final mixtures were magnetically stirred (300rpm) and heated at 30°c and 60°c, respectively, to assure a homogeneous spinning solution. PA 11 was dissolved in 98% formic acid under magnetic stirring and heated at 90°c for two hours. All solutions were cooled at room temperature prior being used in the electrospinning process. The main features of the prepared solutions are described in table 1:

TABLE 1: Characterization of the polymeric solutions.

Polymer	Concentration % (m/v)	Viscosity (mPA.s) at 23°C	Conductivity (mS/cm)
PA 6	8	11,9	2,47
	10	14,1	2,59
PA 6,6	8	12,2	2,94
	10	14,6	3,02
PA 11	8	13,2	3.55
	10	15,7	3,82

Two power supplies Glassman model's PS/ML40P07.5-22 and PS/ML40P07.G06 were used to apply voltages ranged from 8,3 Kv to 25Kv to the electrodes. Polymer solution was fed through a syringe Nordson model precision dispenser tip with 0,25 mm inner diameter using a Harvard Apparatus model PHD 2000 Infusion pump at a constant rate of 0,01 mL/min for the PA6 and PA6,6 polymer solutions and 0,03 mL/min for the PA 11 polymer solution. Three stainless steel rings with a diameter of 10cm each and a metallic fixed collector panel completed the experimental setup. During the experimental development two types of connections between the stainless-steel rings were used. Additionally, the parallel link was tested with two variants: increasing and decreasing the supplied high voltage. The particulars of those links are depicted in Figure 2 and quantitatively defined in table 2.

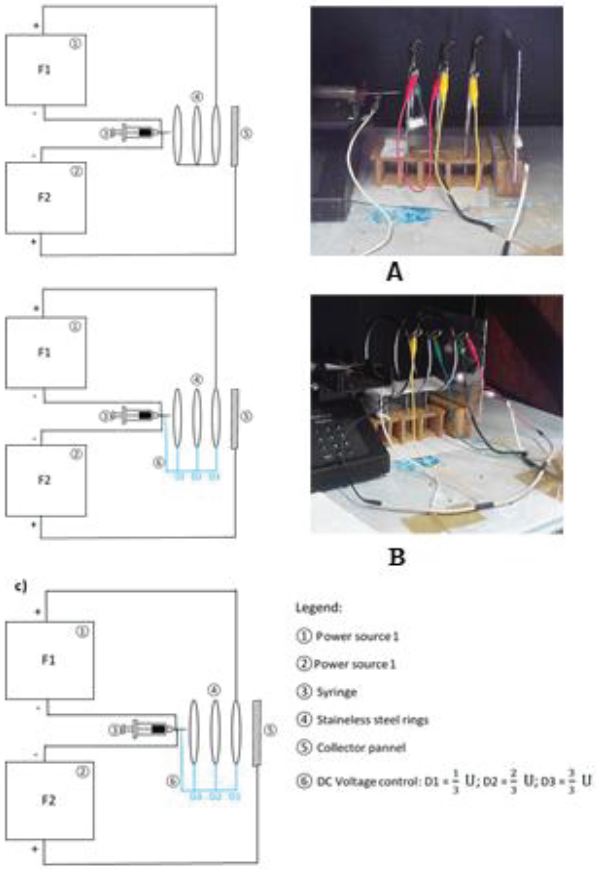

Figure 2: Electrospinning process variants

3. Results

Table 3 shows the variation of the nanofiber average diameter attained with different PAs and a different number of metal rings serially connected.

Table 2: Experimental setup

Source		Type of Rings Connection	Distance (cm)			Voltage (Kv)		
F2	F1		Ring 1	Ring 2	Ring 3	Ring 1	Ring 2	Ring 3
25KV	25KV	Serie	0 cm	4 cm	8 cm	25 KV	25KV	25KV
			0 cm	6 cm		25KV	25KV	
			0 cm			25KV		
25KV	25KV	Parallel (Increasing voltage)	0cm	4cm	8cm	(D1) ~8,3 KV	(D2) ~16,7 KV	(D3) 25 KV
			0cm	6cm		(D2) ~16,7 KV	(D3) 25 KV	
15KV	25KV	Parallel (decreasing voltage)	0cm	4cm	8cm	(D3) 25 KV	(D2) ~16,7 KV	(D1) ~8,3 KV
			0cm	6cm		(D3) 25 KV	(D2) ~16,7 KV	

Table 3: Average nanofiber diameter attained for serial connection

SERIAL CONNECTION	NANOFIBRE AVERAGE DIAMETER (nm)		
	PA 6	PA 6,6	PA 11
1 Ring	68.5	82.5	354.1
2 Rings	61.3	75.4	304.6
3 Rings	60.4	84.2	263.8

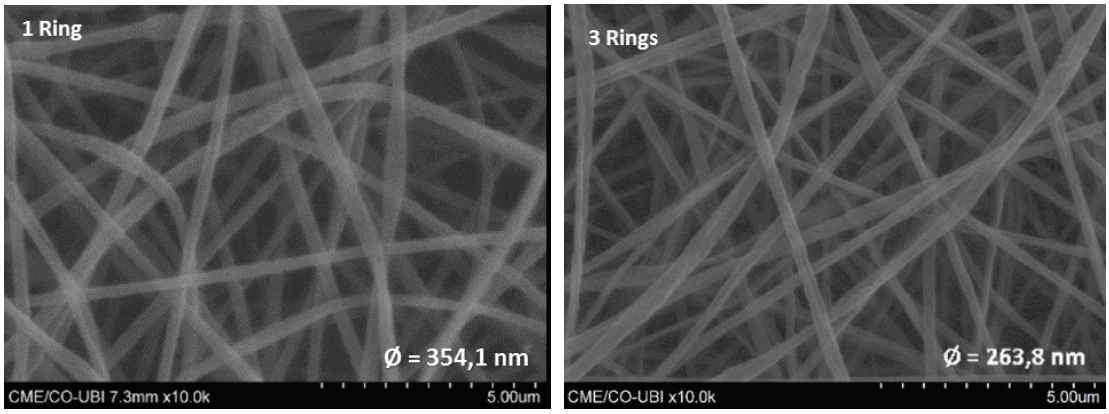

Figure 3: PA 11 nanofibres produced with a serial connection and with 1 and 3 metal charged rings.

The figures demonstrate that the number of rings has no particular influence in the electrospinning process of PA 6 and PA 6,6 whereas PA 11 evidenced a continuous diameter reduction with the addition of metal rings in the electrostatic field.

Hence, it is possible to infer that, under the present conditions, the electrospinning process of PA 11 impart a higher drafting resulting in a more controlled deposition. It is

possible to think that this phenomenon is due to the higher electrical conductivity of that polymer.

In the second part of the experiment the connection between the stainless-steel rings shifted to parallel. Table 4 shows the variation of the nanofiber average diameter attained with different PAs under the described conditions:

Table 4: Average nanofiber diameter attained for parallel connection

PARALLEL CONNECTION		**NANOFIBRE AVERAGE DIAMETER (nm)**		
High Voltage Application	Number of Rings	PA 6	PA 6,6	PA 11
Increasing	2	89,6	86,8	152,9
	3	67,3	78,2	-
Decreasing	2	73,5	83,7	206,9
	3	62,4	80,8	244,1

In accordance with our findings for the serial link, results seems to indicate that the increasing in the number of rings and the insertion of a second power source, led to a decrease in the average diameter of PA 6 and PA 6,6 nanofibers, whereas PA11 presents a completely different behaviour.

The full understanding of this phenomenon requires more studies to be carried out in the future. Eventually, this can be explained by the higher molecular weight of the polymer. Our trying to connect a third ring in the electrospinning process of PA 11 (increasing tension) failed since the polymer could not be successfully electrospun.

4. Conclusions

Our discoveries demonstrated that the use of the novel electrospinning drafting system - experimental variant with increasing voltage - proved to be successful in the obtainment of thinner nanofibers with lower standard deviation and few beads formations.

This fact can be explained by the tight control of the electrostatic field which, in turn, exerts a greater control over the inherent instability of the polymer jet along his travel from the needle tip to the collector panel and, consequently, ensuring a more controlled nanofiber deposition.

The use of multiple charged metal rings acting as “lens” modify the electric filed from the very beginning – polymer jet initiation - until the deposition over the collector panel. Thus, a possible explanation for the phenomenon lies on the convergence of the electrostatic field towards a centreline on the collector panel.

Additionally, the polarity difference enhancement assures a continuous increase – “drafting” - in the downward force of the polymer jet stream.

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