

# (11) **EP 2 325 355 A1**

(12)

# **EUROPEAN PATENT APPLICATION**

(43) Date of publication:

25.05.2011 Bulletin 2011/21

(51) Int Cl.: **D01D** 5/00 (2006.01)

(21) Application number: 10014817.0

(22) Date of filing: 22.11.2010

(84) Designated Contracting States:

AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO PL PT RO RS SE SI SK SM TR

Designated Extension States:

**BA ME** 

(30) Priority: 24.11.2009 PL 38966009

(71) Applicant: Politechnika Lodzka 90-924 Lodz (PL)

(72) Inventors:

- Krucinska, Izabella 91-857 Lósz (PL)
- Gliscinska, Eulalia
   92-404 Lódz (PL)
- Chrzanowski, Michal 98-100 Lask (PL)
- (74) Representative: Kaczur-Kaczynska, Ewa Politechnika Lódzka Sekcja Patentów Dzialu Transferu Technologii UI. Zeromskiego 116 90-924 Lódz (PL)

#### (54) System for electrospinning fibres

(57) The electrospinning fiber throwing system is equipped with the spinning capillaries (1) fixed in the spinning head (2) from a non-conducting material, with the separator (3) from a conductive material inside the head, linked to each of the capillaries (1) and kitted in the high-voltage terminals. The head (2) is mounted in a slidable manner on the horizontal guide (5) whose ends are mounted slidably on the vertical arms (6) of the system's

load-bearing structure. Under the extrusion nozzles of the capillaries (1), the formed fiber take-up device (8) is located. The system is also equipped with the multi-threaded peristaltic pump (9), each thread of which being linked to one of the spinning capillaries (1), as well as with the reheating element located at the outlet of the formed fiber take-up device (8).

15

20

35

40

# [0001] The subject of the invention is the electrospin-

1

ning fiber throwing system.

[0002] Fiber throwing from polymer alloys or solutions using the electrospinning technique is based on the effects occurring in liquids and polymers under the influence of high voltage of specific polarity applied. In the first studies on fiber throwing through electrospinning, the system containing a single capillary with a nozzle, being an electrode feeding a polymer alloy or solution, connected to a high-voltage generator and a grounded collector electrode placed under it, was applied. The forces of the electrostatic field generated between the electrodes cause that a drop of polymer formed at the end of the nozzle undergoes a deformation into the so-called Taylor cone, which then, moving in the direction of the crown discharge current towards the grounded electrode, is accelerated and stretched to be finally collected on the surface of the collector electrode in the form of loosely packed fibers.

[0003] The work aimed at improving the efficiency of the electrospinning process focused on a change in the mode of taking up formed fibers as well as in the mode of feeding a polymer alloy or solution.

[0004] The Journal of Physics: Conference Series 142 (2008) 012027 Electrostatics 2007 describes an electrospinning system where the take-up of formed fibers is done by means of rotary rollers, made from a conductive material, which are covered with subsequent fiber layers until the assumed thickness thereof is reached, while the patent description US 2009/00910655 A1 presents an electro spinning system in which formed fibers are taken up by rotating discs.

[0005] In the first solutions to the improved spinning efficiency through the modified mode of feeding a polymer alloy or solution, the referenced alloy or solution was forced by compressed air or diffusion pumps. The foregoing techniques made it possible to isolate a polymer of high electric potential from the environment, thereby protecting system operators from electrocution, yet at the same time curbing the control of an amount of the extruded polymer and the time instability of the electro spinning process.

[0006] The Czech company Elmarko used an electrospinning system in which a spinning feed roller, immersed in a polymer solution, rotated, being covered with polymer film, while the voltage applied thereto caused that trickles of the polymer detached from the surface of the roller and formed fibers precipitated on an unwoven cloth belt traveling over the roller.

[0007] We also know an electrospinning system where the electro spinning process is intensified using a serratesurfaced feed roller. This system needs applying an extra carrier in the form of a fabric or unwoven cloth to take up formed fibers.

[0008] The patent description EP 1982735 A1 presents an electro spinning system in which a polymer is fed by several capillaries situated one by one in single file, while formed fibers are taken up by a rotating roller, located vertically.

[0009] The invented electro spinning fiber throwing system, equipped with vertically positioned spinning capillaries connected to a source of high voltage and a device taking up formed fibers, located under the extrusion nozzles of the capillaries and linked to a driving unit, is characterized by the solution according to which the spinning capillaries are fixed in a spinning head from a non-conducting material, inside which there is a separator from a conductive material, connected to each of the capillaries and kitted in high-voltage terminals. The head is mounted in a slidable manner, by means of a horizontal transport mechanism connected to the driving motor, on a horizontal guide whose ends are mounted slidably as well, by means of a vertical transport mechanism connected to the driving motor, on the upright arms of the system's load-bearing structure. In addition, the system is equipped with a multithreaded peristaltic pump, each thread of which being linked to one of the spinning capillaries placed on the horizontal arm of the load-bearing structure. The unit taking up formed fibers is an advantageously rotationally fixed roller or conveyor. The head contains minimum 32 capillaries, advantageously spaced in four rows, at an advantageous distance of 3 cm from each other in each row and 6 cm between the rows. The system contains an ancillary reheating element in the form of a drying tunnel, which makes it possible to evaporate dissolvent residue, collected at the outlet of the fiber take-up device, from formed fibers.

[0010] According to the invention, the system is marked by a considerably higher efficiency of the fiber throwing process compared to the systems known hitherto. Furthermore, the hazard of operators' electrocution has been eliminated from the system.

[0011] The subject of the invention is represented as a built model in a figure showing the system from a front view

**[0012]** The system is equipped with the 32 vertically situated spinning capillaries 1, fixed in the Teflon spinning head 2. The capillaries 1 are positioned in the four rows, at a distance of 3 cm from each other in each row and 6 cm between the rows. The head 2 has the built-in steel wool separator 3, linked to each of the capillaries 1 and kitted in the high-voltage terminals. The head 2 is mounted in a slidable manner, by means of the horizontal transport mechanism 4 connected to the driving motor, on the horizontal guide 5 whose ends are mounted slidably, by means of the vertical transport mechanism 7 connected to the driving motor, on the upright arms 6 of the system's load-bearing structure. Under the extrusion nozzles of the capillaries 1, there is a metallic formed fiber take-up roller 8, fixed rotationally in a holder providing a galvanic connection to the grounding and linked to the driving unit. The system is also equipped with the 32-thread peristaltic pump 9, each thread of which being connected to one of the spinning capillaries 1 placed on the horizontal arm

55

10

15

20

40

45

10 of the system's load-bearing structure. The system contains the drying tunnel situated along the path of fibers leaving the roller 8.

[0013] In order to produce nanofibers, the peristaltic pump 9 is filled with the polymer and the separator 3 is connected to the source of high voltage, thus starting the pumping of the polymer into the capillaries 1. When the capillaries 1 are filled with the polymer, the guide 5 with the head 2 is positioned at an adequate distance from the take-up roller 8 by means of the vertical transport mechanism 7. Next, the horizontal motion range of the head 2 along the guide 5 so it can extend over the entire work area of the roller 8 is set up by means of the horizontal transport mechanism 4 and the unit driving the roller 8 is activated. Once the roller 8 is set in rotary motion, the head 2 is set in horizontal motion along the guide 5 by means of the horizontal transport mechanism 4. Formed nanofibers are taken up on the grounded roller 8 in the form of unwoven cloth. The obtained layer of unwoven cloth, desirably thick, passes through the drying tunnel whose outlet releases the finished product.

**[0014]** The system was used for nanofiber throwing in the form of unwoven fabric from:

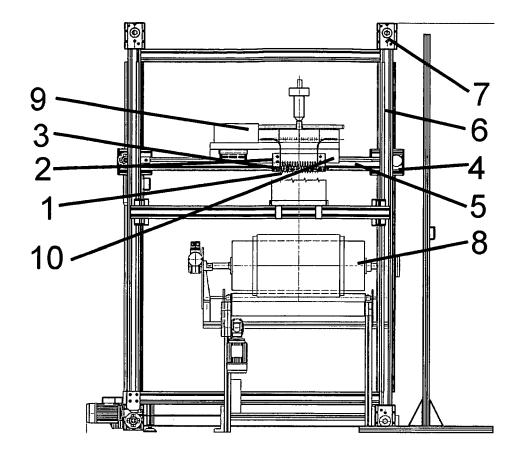
- 15% polyacrylonitrile (PAN) solution in dimethyl sulfoxide (DMSO) at a temperature of 20°C and a velocity of the peristaltic pump 9 of 0.5 rpm, in the electrostatic field generated by the voltage 20 kV, at a distance of 20 cm between the head 2 and the roller 8 and a rotational speed of the roller 8 of 30 rpm; obtained nanofibers with a diameter of 500 nm;
- 17% PAN solution in DMSO at a temperature of 20°C and a velocity of the peristaltic pump 9 of 0.5 rpm, in the electrostatic field generated by the voltage
- 20 kV, at a distance of 20 cm between the head 2 and the roller 8 and a rotational speed of the roller 8 of 30 rpm; obtained nanofibers with a diameter of 400 nm;
- 20% polycaprolactone solution in dichloromethane at a temperature of 20°C and a velocity of the peristaltic pump 9 of 0.5 rpm, in the electrostatic field generated by the voltage 25 kV, at a distance of 20 cm between the head 2 and the roller 8 and a rotational speed of the roller 8 of 30 rpm; obtained nanofibers with a diameter of 1,200 nm.

**Claims** 

The electrospinning fiber throwing system equipped with the vertically situated spinning capillaries connected to the high-voltage source and with the formed fiber take-up device located under the extrusion nozzles of the capillaries and connected to the driving unit, characterized by the solution according to which the spinning capillaries (1) are fixed in the spinning head (2) from a non-conducting material, with the separator (3) from a conductive material in-

side the head, linked to each of the capillaries (1) and kitted in the high-voltage terminals, in addition to which the head (2) is mounted in a slidable manner, by means of the horizontal transport mechanism (4) connected to the driving motor, on the horizontal guide (5) whose ends are mounted slidably, by means of the vertical transport mechanism (7) connected to the driving motor, on the upright arms (6) of the system's load-bearing structure; furthermore, the system is equipped with the multithreaded peristaltic pump (9), each thread of which being linked to one of the spinning capillaries (1) placed on the horizontal arm (10) of the system's load-bearing structure, as well as with the reheating element in the form of the drying tunnellocated at the outlet of the fiber take-up device (8).

- 2. As per claim 1, the system characterized by the head (2) containing minimum 32 capillaries advantageously spaced in the four rows, at an advantageous distance of 3 cm from each other in each row and 6 cm between the rows.
- As per claim 1, the system characterized by the formed fiber take-up device (8) in the form of the advantageously rotationally mounted metal roller or conveyor.





# **EUROPEAN SEARCH REPORT**

Application Number EP 10 01 4817

	DOCUMENTS CONSID	ERED TO B	E RELI	EVANT		
Category	Citation of document with ir of relevant pass		appropriat	э,	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
A	EP 1 967 617 A1 (UN 10 September 2008 ( * figure 2 *				1-3	INV. D01D5/00
	* paragraphs [0064]	, [ 0066]	*			
A	WO 2006/009854 A2 ( ALESSANDRO [US]; KL WEIWE) 26 January 2 * figure 1 * * page 26, lines 6-	EMIC JAMES 2006 (2006-	F [ÚS	S]; DENG	1-3	
A	WO 2008/069795 A1 ( ROBERTSON JOHN A [U [US]) 12 June 2008 * figure 6 * * claim 6 *	S]; SCOTT	<b>ASHLEY</b>		1-3	
A	WO 2006/043968 A2 (ANDRADY ANTHENSOR DAVID S [US] RES TRIANGLI AND) 27 April 2006 (2006-04-27)				1-3	
	* figures 2,8,9A,9B * claims 23-26 * * page 14, line 21	- page 15, line 14 *		14 *		TECHNICAL FIELDS SEARCHED (IPC)  D01D
А	WO 2004/016839 A1 ( [KR]; CHUN SUK-WON [KR]) 26 February 2 * figures 1,3 * * page 8, line 20 -	[KR]; PARK 2004 (2004-	( JONG- 02-26)	·SU	1-3	
	The present search report has Place of search		r all claim			Examiner
	The Hague	14	March	2011	Ve	erschuren, Jo
X : parti Y : parti docu A : tech O : non	ATEGORY OF CITED DOCUMENTS cularly relevant if taken alone cularly relevant if combined with anotiment of the same category nological background written disclosure mediate document	her	E : ea aft D : do L : do  & : me	eory or principle rlier patent docu er the filing date cument cited in cument cited for ember of the sar	underlying the ument, but pub the application other reason	e invention olished on, or n

## ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO.

EP 10 01 4817

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

14-03-2011

	Patent document ed in search report		Publication date		Patent family member(s)		Publication date
EP	1967617	A1	10-09-2008	AU 20 EP EP EP WO 20 US 20	008225883 008252985 1992721 2126164 2148945 008110538 010112020 010215939	A1 A2 A2 A2 A2 A1	18-09-200 27-11-200 19-11-200 02-12-200 03-02-201 18-09-200 06-05-201 26-08-201
WO	2006009854	A2	26-01-2006	NONE			
WO	2008069795	A1	12-06-2008	CA CN 1 EP JP 20 US 20	006351464 2671719 .01610884 2099595 010511808 010071619 008131615	A1 A A1 T A1	12-06-200 12-06-200 23-12-200 16-09-200 15-04-201 25-03-201 05-06-200
WO	2006043968	A2	27-04-2006	US 20	1756338 007532791 005224998 011031638	T A1	28-02-200 15-11-200 13-10-200 10-02-201
WO	2004016839	A1	26-02-2004	JP JP 20 KR 200	003247181 4129261 005534828 040016320	B2 T A	03-03-200 06-08-200 17-11-200 21-02-200 20-10-200

For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

#### EP 2 325 355 A1

#### REFERENCES CITED IN THE DESCRIPTION

This list of references cited by the applicant is for the reader's convenience only. It does not form part of the European patent document. Even though great care has been taken in compiling the references, errors or omissions cannot be excluded and the EPO disclaims all liability in this regard.

## Patent documents cited in the description

US 200900910655 A1 [0004]

• EP 1982735 A1 [0008]

#### Non-patent literature cited in the description

 The Journal of Physics: Conference Series, 2008, vol. 142, 012027 [0004]