The invention relates to a device for producing nanofibers or microfibers from solutions, emulsions, liquid suspensions or melts containing a spun substance. The subject matter of the invention consists in that it comprises a chamber in which a hollow shaft is assembled, on which at least one rotating disc with an output gap is mounted. The chamber is generally provided with a source of the flowing gas and a collection area. In an alternative embodiment, the chamber is provided with a number of side by side arranged hollow shafts on which rotating discs are mounted. It is preferred that at least one hollow shaft is provided with two superposed rotating discs. At least one rotating disc is composed of two successive parts, wherein between the upper part and the lower part an outlet gap is formed around the circumference thereof. The size of the outlet gap between the upper part and the lower part of rotating disc may be formed by a spacer element, in particular a spacer ring.
A device for producing fibers or microfibers

Technical Field

The invention relates to a device for producing nanofibers or microfibers from solutions, emulsions, liquid suspensions or melts containing a spun substance.

Background Art

Currently there are numerous devices for electrostatic spinning of solutions, emulsions, liquid suspensions or melts containing a spun substance. Described are systems producing nanofibers comprising both nozzle and nozzleless arrangements. These devices are structurally demanding and their operation has a number of shortcomings, including clogging of nozzles, which leads to an interruption of the operation or reduction in productivity.

As far as the nozzleless devices are concerned, the formation of nanofibers occurs directly from the surface of spun solutions which can be in the form of a thin film. Two-layer systems are also used, wherein the lower layer is formed by a ferromagnetic suspension and the upper layer by a solution of spun polymer. After application of the
magnetic field, sharp vertical cones of the ferromagnetic liquid are formed that serve as nuclei from which the nanofibers are produced.

Other device is based on the aeration of a spun polymer solution in order to create a high concentration of bubbles on the surface of the solution, wherein a lowering of the surface tension takes place and the bubbles form seeds of nanofibers created by virtue of the electric field.

Another device is based on a slowly rotating cylinder which is partially immersed in a solution of the spun polymer. During the rotation of the cylinder, a deposit of a specific amount of the solution on the roller takes place, resulting in a formation of a continuous film from which on the upper part by virtue of a strong electric field so-called Taylor cones serving as nuclei of nanofibers are formed.

Electrostatic spinning methods are characterized by a low speed of the process; they are technically complicated and expensive. The electrostatic spinning is limited by the necessity of a high voltage electric field.

There are also devices used that are not based on the application of electrospinning. For the formation of nanofibers, these usually take advantage of centrifugal force or a gas stream. A rotating disk is used, on the surface of which a thin film of spun solution is produced by means of the centrifugal force.
Summary of the Invention

Said disadvantages of the devices for producing fibers or microfibers from solutions emulsions, liquid suspensions, or melts containing spun suspension can largely be removed by means of the solution according to the invention whose principle consists in that it comprises a chamber in which a hollow shaft is assembled, on which at least one rotating disc with an output gap is mounted.

The chamber is generally provided with a source of the flowing gas and a collection area. In an alternative embodiment, the chamber is provided with a number of side by side arranged hollow shafts on which rotating discs are mounted.

It is preferred that at least one hollow shaft is provided with two superposed rotating discs. At least one rotating disc is composed of two successive parts, wherein between the upper part and the lower part the outlet gap is formed around the circumference thereof. The size of the outlet gap between the upper part and the lower part of rotating disc may be formed by a spacer element, in particular a spacer ring.

It is preferred that at least one part of the rotating disc has a frustoconical shape. At least one rotating disc may be provided with a pressure element, such as pressure nut. At least one disc or discs disposed in a chamber which is made of heat resistant material may be provided with means for their heating.
The inner space of the hollow shaft is connected with the output gap of each of the rotating discs by means of openings and at the other end with a rotary unit for supplying the polymer and further with the drive motor.

The source of the flowing gas in the chamber is a compressor or a fan. The collecting area can be either a movable conveyor made of a breathable fabric or a rotating collector or a bag of a porous mesh. The collecting area may be electrically charged.

The present invention in comparison with the current state of the art prevents drying films of polymer solutions on the surface of rotating discs. It reduces the amount of defects of produced nanofibers and microfibrous layers, especially drops. It facilitates the centrifugal spinning of melts, because there occurs no cooling of the melt on the surface of the rotating elements.

**Brief Description of the Drawings**

An exemplary embodiment of the device for producing nanostructured and microstructured materials is shown in enclosed drawings, wherein

Figure 1 shows an overall diagram of the entire device,

Figure 2 shows a spindle with a hollow shaft and a disc in the perspective illustration and partial longitudinal section,
Figure 3 shows a specific embodiment of the disc according to the invention without a spacer, and

Figure 4 shows a specific embodiment of the disc according to the invention with a spacer.

Detailed Description of the Invention

Example 1. Preparation of nano- or microfibers from a polyvinyl alcohol solution

To prepare polyvinyl alcohol micro- or nanofibers, a commercial solution of polyvinyl alcohol Sloviol R16, 16% (wt./wt.) of solid content (FicHEMA) was used. The polyvinyl alcohol solution was pumped from the first liquid reservoir 17 by the first pump 18 through a connecting hose 19 via the first safety valve 20 and the first check valve 21 at a rate of 2-12 ml/min., and fed through an inlet 22 of liquid into the rotation unit 10 from which it further entered into a hollow shaft 3 disposed in a tube 5 of a spindle. Via openings 16 was the polyvinyl alcohol solution sprayed from the inner space 6 of the hollow shaft 3 into the inner space of the rotating disc 2 having a conical shape with a diameter of 120 mm, between the upper part 7 and the lower part 8 thereof. The output gap 4 of the conical disc 2 was set up using a spacer ring 13 on the width of 200 micrometers as shown in Figure 4. The rotating disc 2 was positioned over the base plate 23 with channels 32 for the distribution of the drying gas into the chamber 1 in the form of a tube 5 of a plexi-glass having the diameter
of 35 cm and the height 40 cm. In the base plate 23 entered at a rate of 0.7 m³/s drying air preheated to a temperature of 25°C from a source 11, which is formed by a compressor and a heater. The rotating disc 2 with the hollow shaft 3 was rotated via an intermediate transmission 15 by means of a drive motor 14 at a speed of 1 to 5000 revolutions per minute. The stream of preheated air carried away the nano- or microfibers generated by the centrifugal force on the edge of the outlet gap 4 into a collection chamber 12 provided with an orifice having a width of the slot 24 of 5 cm and a length of 35 cm and a sliding belt 25 formed by a permeable nonwoven Spunbond having a basis weight 18.8 g/m². The shift velocity was 10 cm/min. The nano- or microfibers were stored in the form of a continuous layer on the surface of the sliding belt 25 of the permeable nonwoven.

At a constant flow of the polyvinyl alcohol solution 10 ml/min. increased a rate of fiber formation with an increasing rotational speed of the disc in the range 1 to 3,000 revolutions per minute. Upon further increasing the rotation speed, the rate of the fiber formation did not further increase and the incidence of defects in the fibers network has increased in the form of droplets. Therefore, further experiments were carried out at a rotation speed of 3000 revolutions per minute. At this speed of rotation and other conditions described above has increased the basis weight of the fibers in a linear manner within the range of the polyvinyl alcohol solution flow from 2 to 8 ml/min. Maximum productivity was observed at the flow rate 10 ml/min. Further acceleration of the flow to 12 ml/min. under the conditions described above seemed to be counter-productive
already, because neither the fiber formation nor the exploitation of polyvinyl alcohol increased, conversely, they have even slightly declined. At the same time the incidence of defects in the form of microdroplets has increased. Under these conditions, it appeared as optimal the flow in the range of 8-10 ml/min. Under these conditions, the basis weight of the layer of polyvinyl alcohol fibers was in the range of 7-10 g/m². Maximum speed of the fiber formation at a flow rate of 8 ml/min., and a speed of rotation of 3000 revolutions per minute was 20g per hour. The distribution of the nanofibers was homogeneous both in the microscopic and macroscopic level across the whole belt width, which represented 35 cm. The diameter of the majority of fibers observed was in the range of 400 to 800 nanometers.

The above procedure was repeated with the exception that two rotating discs 2 of the same construction, located on one hollow shaft 3 superimposed with a spacing of 10 cm were used, and the flow rate of the polyvinyl alcohol solution and the shift of the belt 25 of permeable nonwoven were doubled. Under these conditions the rate of the fiber formation at a flow rate of 16 ml/min and the rotation speed of 3000 revolutions per minute managed to increase to 38 g per hour. There were no significant changes in the basis weight of the fibers and their quality.

Example 2. Preparation of nano- or microfibers from polyamide 6.

For the preparation of micro or nanofibers polyamide 6 were used pellets of polyamide 6 (Rhodia Technyl). From these pellets was prepared a solution 15% (wt./wt.) in 85% (wt./wt.)
formic acid (Penta) at a temperature of 80°C. This solution was pumped from the first liquid reservoir 17 by the first pump 18 through the connecting hose 19 via the first safety valve 20 and the first check valve 21 at a rate of 6-16 ml/min., and fed through the inlet 22 of liquid into the rotation unit 10 from which it further entered into the hollow shaft 3 disposed in the tube 5 of the spindle. Via openings 16 was the polyamide 6 solution sprayed from the inner space 6 of the hollow shaft 3 into the inner space of the rotating disc 2 between the upper part 7 and the lower part 8 thereof. The disc 2 having a conical shape with a diameter of 120 mm provided with the pressure element 9 in the form of a nut was used, as it can be seen from Figure 3. The pressure of the presser nut was gradually changed so that an opening of the outlet gap 4 occurs at a pressure in the range of 4-400 bar. The rotating disc 2 was positioned over the base plate 23 with channels 32 for the distribution of the drying gas into the chamber 1 in the form of a tube 5 of a plexi-glass having the diameter of 35 cm and the height of 40 cm. In the base plate 23 entered at a rate of 0.6 m³/s drying air preheated to a temperature of 35°C from a source 11, which is formed by a compressor and a heater. The rotating disc 2 with the hollow shaft 3 was rotated via an intermediate transmission 15 by means of a drive motor 14 at a speed of 1 to 5000 revolutions per minute. The stream of preheated air carried away the nano- or microfibers generated by the centrifugal force on the edge of the outlet gap 4 into a collection chamber 12 provided with an orifice having a width of the slot 24 5 cm and a length 35 cm. At a height of 5 mm above the slit 24 of the aperture, a rotating collector of fibers in the form of a roller made of fine steel mesh having a diameter of 10 cm
and provided with a motor imparting the collector a rotation of 10 rpm., was positioned longitudinally horizontally.

Nano- or microfibers were deposited evenly over the whole surface of the rotary collector in the form of a continuous layer of a thickness of almost 3 mm in the form resembling a soft cotton wool. When increasing the pressure in the interior of the disc 2 between the upper part 7 and the lower part 8 thereof, a reduction in diameter of the fibers has occurred. While at a pressure of 4 bar the fiber diameter was in the range 600 to 900 nm, at a pressure of 400 bar the diameter was already in the range of 200 nm to 400 nm. The rate of the fibers formation was in the range of 50 g to 135 g per hour, depending on the conditions. The optimal flow rate was 14 ml/min.

In another experiment, a chamber 1 made of plexi-glass and having a cuboidal shape with a length of 2 m and a width and a height of 50 cm was used, in which two rotating discs 2 were placed side by side, with pressure elements 9 in the form of nuts. The discs 2 were placed at a distance of 1 m apart. Optimal conditions were used for spinning the polyamide 6 solution as identified in the above described experiment. The flow in each disk was 14 ml/min. The pressure in the inner space of the disk 2 between the upper part 7 and the lower part 8 thereof was 60 bar. The collection of the fibers in the collecting space 12 was carried out using a slot having a width of the aperture 24 of 20 cm and a length of 2 m and a sliding belt 25 of the permeable Spunbond nonwoven having a basis weight of 18.8 g/m², also with a width of 2 m. The speed of displacement of the belt was 10 cm/min. The nano- or microfibers have been stored in the form of a continuous
layer on the surface of the belt 25 of a permeable fabric. The distribution of the nanofibers was homogeneous both in the microscopic and macroscopic level across the whole belt width, which represented 35 cm. The diameter of the majority of fibers observed was in the range of 400 to 800 nanometers. The basis weight of the fibers was in the range of 4-6 g/m².

Example 3. Encapsulation of probiotic bacteria into gelatin microfibers

For the encapsulation of probiotic bacteria, a 10% (wt./wt.) suspension of the microbial preparation BA (1.10⁹ CFU/g) (Milcom) containing the probiotic strains of genera Lactobacillus acidophilus and Bifidobacterium bifidum freeze-dried with powdered milk in distilled water was used. Further, a solution of 30% (wt./wt.) of pig skin gelatin, 300 bloom, type A (Sigma-Aldrich) in 40% (vol./vol.) acetic acid was used. The gelatin solution at a temperature of 45°C was pumped from the first reservoir 17 of liquid by means of the first pump 18 via the first connecting pipe 19 through the first safety valve 20 and the first check valve 21 at a rate of 5 ml/min. Simultaneously, a bacterial suspension was pumped from the second reservoir 26 of liquid by means of the second pump 27 via the connecting hose 19 through the second safety valve 28 and the second check valve 29 at a rate of 5 ml/min. The gelatin solution and bacterial suspension were mixed in a mixing chamber 30 having a volume of 5 ml. The resulting bacterial suspension in the gelatin solution was fed through the inlet 22 of liquid into a rotation unit 10 from which it further entered the hollow shaft 3 disposed in the tube 5 of the spindle. Through the openings 16, the suspension was
further sprayed from the inner space 6 of the hollow shaft 3 into the inner space of the rotating disc 2 of a conical shape with a diameter of 120 mm, between its upper part 7 and the lower part 8. The output gap 4 of the conical disc 2 was set using the spacer element 13 to the width of 150 microns, as shown in Figure 4. The rotating disc 2 was positioned over the base plate 23 with channels 32 for the distribution of the drying gas into the chamber 1 in the form of a tube made of fine steel mesh 35 cm in diameter and the height 40 cm. Into the base plate 23 entered the drying air preheated to a temperature of 40°C from a source 11, which is formed by a compressor and a heater, at a velocity of 0.8 m³/s.

The rotating disc 2 with the hollow shaft 3 was rotated via an intermediate transmission 15 by means of a drive motor 14 at a speed of 3500 revolutions per minute. The stream of preheated air carried away the nano- or microfibers generated by the centrifugal force on the edge of the outlet gap 4 of the rotating disc 2 into a collection chamber 12 provided with an orifice having a width of the slot 24 of 10 cm over which a bag made of a permeable nonwoven Spunbond having a basis weight 18.8 g/m². The shift velocity was 10 cm/min. The microfibers were stored in this bag in the form resembling a soft cotton wool.

The yield was 80 g of the microfibers with the encapsulated bacterial culture in one hour of the operation. A microscopic analysis confirmed the presence of bacterial cells encapsulated within the microfibers having a diameter of between 5 and 10 microns. Standard methods for microbiological analysis showed that there was only
a small decrease in vitality of the original bacterial culture, expressed as a number of colony-forming units (CFU), by one order. Microbiological tests confirmed a significant protective effect of the encapsulating against a simulated acidic environment of the stomach and against the action of bile acids.

Example 4. Preparation of fibers from the melt the polyhydroxyalkanoate

A melt of polyhydroxy alkanoate (Nanjing Huichen Co., Ltd., China) was prepared in the first reservoir 14 of a solution equipped with an induction heating, and maintained at a temperature of 300 degrees Celsius. The entire device was thermally insulated.

The melt was pumped from the first reservoir 17 of a solution by the first pump 18 via the insulated connecting hose 19 made of a profiled steel strip through the first safety valve 20 and the first check valve 21 at a velocity of 10 ml/min., and fed through the inlet 22 of a liquid into the rotation unit 10 from which it further entered into the hollow shaft 3 disposed in the tube 5 of the spindle. Through openings 16 were the melt sprayed from the inner space of the hollow shaft 6 into the inner part of the rotating disc 2 of a conical shape, with a diameter of 120 mm, between the upper part 7 and the lower part 8 thereof. The output gap 4 of the conical disc 2 has been set using the spacing element 13 to the width of 50 microns, as it is apparent from Figure 4. The rotating disc 2 was positioned over the base plate 23 equipped with channels 32 for the distribution of the drying gas into the chamber 1 in the form
of an insulated steel tube having the diameter of 35 cm and the height of 40 cm. Into the base plate 23, the drying air preheated to 250°C from a source 11, which was formed by a compressor and a heater, entered at a velocity of 1 m³/s. The rotating disc 2 with the hollow shaft 3 was rotated via an intermediate transmission 15 by means of a drive motor 14 at a speed of 1 to 5 000 revolutions per minute. The stream of preheated air carried away the nano- or microfibers generated by the centrifugal force on the edge of the outlet gap 4 into a collection chamber 12 provided with an orifice having a width of the slot 24 of 5 cm and a length of 35 and a sliding belt made of a fine steel mesh.

The basis weight of the fibers was in the range of 8-10 g/m². The distribution of the nanofibers was homogeneous both in the microscopic and macroscopic level across the whole belt width, which represented 35 cm. The diameter of the majority of fibers observed was in the range of 400 to 800 nanometers. The rate of fibres production was 600 g per hour.

**Industrial Applicability**

Due to preventing the drying of films of polymer solutions on the surface of rotating discs and a reduced quantity of defects fibrous layers, a device for the production of fibers or microfibres from solutions, emulsions, melts or liquid suspensions containing a spinnable polymer according to the invention may be advantageously used to produce nanofibers or microfibers, where a high productivity work is required. The device also facilitates the centrifugal spinning
of melts, because no cooling of the melt on the surface of the rotating elements takes place.
CLAIMS

1. A device for producing nanofibers or microfibers from solutions, emulsions, liquid suspensions or melts containing a spun substance, characterized in that it comprises a chamber (1) in which a hollow shaft (3) is assembled on which at least one rotating disk (2) with an output gap (4) is mounted.

2. The device for producing nanofibers or microfibers as defined in claim 1, characterized in that the chamber (1) is provided with a source (11) of the gas flow and a collection area (13).

3. The device for producing nanofibers or microfibers as defined in claim 1, characterized in that the chamber (1) is provided with a number of side by side arranged hollow shafts (3) on which rotating discs (2) are mounted.

4. The device for producing nanofibers or microfibers as defined in claim 1, characterized in that at least one hollow shaft (3) is provided with at least two superimposed rotating discs (2).

5. The device for producing nanofibers or microfibers as defined in claim 1 and 4, characterized in that least one rotating disc (2) is composed of two successive parts (7,8), wherein between the upper part (7) and the lower part (8) an outlet gap (4) is formed around the circumference thereof.
6. The device for producing nanofibers or microfibers as defined in claim 5, characterized in that the size of the outlet gap (4) between the upper part (7) and the lower part (8) of the rotating disc (2) is formed by a spacer element (13), for example a spacer ring.

7. The device for producing nanofibers or microfibers as defined in claim 1 and 5, characterized in that at least one part of the rotating disc (2) has a frustoconical shape.

8. The device for producing nanofibers or microfibers as defined in claim 1, 4 and 5, characterized in that at least one rotating disc (2) is provided with a pressure element (9).

9. The device for producing nanofibers or microfibers as defined in claim 6, characterized in that the pressure element (9) is a pressure nut.

10. The device for producing nanofibers or microfibers as defined in claim 1 and 3, characterized in that the inner space (6) of the hollow shaft (3) is interconnected via openings (16) with the inner space of each of the rotating discs between their upper part (7) and the lower part (8) and the output gap (4).

11. The device for producing nanofibers or microfibers as defined in claim 1, characterized in that the hollow shaft (3) is interconnected with a rotary unit (10) allowing the supply of a liquid or a melt comprising the spun substance, and further with a drive motor (14).
12. The device for producing nanofibers or microfibers as defined in claim 1, characterized in that the source (11) of the gas flow in the chamber (1) is a compressor or a fan.

13. The device for producing nanofibers or microfibers as defined in claim 2, characterized in that the collection area (12) is a movable conveyor (25) made of a nonwoven fabric or a rotating collector or a bag of a porous mesh.

14. The device for producing nanofibers or microfibers as defined in claim 2 and 11, characterized in that the collection area (12) is electrically charged.

15. The device for producing nanofibers or microfibers as defined in claim 1 and 3, characterized in that the rotating disc or discs (2) located in the chamber (1), made of a heat resistant material, are fitted with means for their heating.
List of reference numbers

1 - chamber
2 - disc
3 - hollow shaft
4 - output gap
5 - tube
6 - inner space
7 - upper part
8 - lower part
9 - pressure element
10 - rotary unit
11 - source of the gas flow
12 - collection area
13 - spacer element
14 - drive motor
15 - intermediate transmission
16 - openings
17 - first reservoir
18 - first pump
19 - connecting hose
20 - first safety valve
21 - first check valve
22 - liquid inlet
23 - base plate
24 - slot
25 - sliding belt
26 - second reservoir
27 - second pump
28 - second safety valve
29 - second check valve
30 - mixing chamber
31 - heating device
32 - channel for gas distribution
**INTERNATIONAL SEARCH REPORT**

**A. CLASSIFICATION OF SUBJECT MATTER**

INV. D01D5/00  D01D5/18

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

D01D  D04H

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, WPI Data

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

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<td>US 5 326 241 A (ROOK ROBERT H [US] ET AL) 5 July 1994 (1994-07-05) column 4, lines 15-24; figure 9 column 6, line 16 - column 7, line 22</td>
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Further documents are listed in the continuation of Box C. See patent family annex.

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Date of the actual completion of the international search: 15 December 2015

Date of mailing of the international search report: 23/12/2015

Name and mailing address of the ISA/ European Patent Office, P.B. 5018 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-3040, Fax: (+31-70) 340-3016

Authorized officer: Van Beurden-Hopkins
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