



# (11) EP 3 259 543 B1

# (12) EUROPEAN PATENT SPECIFICATION

(45) Date of publication and mention of the grant of the patent:01.04.2020 Bulletin 2020/14

(21) Application number: 16706830.3

(22) Date of filing: 22.02.2016

(51) Int Cl.: F26B 3/12 (2006.01)

F26B 3/12 (2006.01)
B01J 2/04 (2006.01)
B05B 7/06 (2006.01)
B05B 7/06 (2006.01)
B05B 12/18 (2018.01)

(86) International application number: PCT/EP2016/053643

(87) International publication number: WO 2016/131983 (25.08.2016 Gazette 2016/34)

# (54) AN APPARATUS AND A METHOD FOR GENERATING DROPLETS

VORRICHTUNG UND VERFAHREN ZUR ERZEUGUNG VON TRÖPFCHEN APPAREIL ET PROCÉDÉ PERMETTANT DE GÉNÉRER DES GOUTTELETTES

(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

(30) Priority: 20.02.2015 EP 15382069

(43) Date of publication of application: 27.12.2017 Bulletin 2017/52

(73) Proprietors:

- Ingeniatrics Tecnologias 41900 Camas (Sevilla) (ES)
- UNIVERSIDAD DE SEVILLA ES-41012 Sevilla (ES)
- (72) Inventors:
  - ASENSIO CAÑO, Miguel Angel 41900 Camas (Sevilla) (ES)

- TALAVERA ALMENA, Beatriz 41900 Camas (Sevilla) (ES)
- FLORES MOSQUERA, María 41900 Camas (Sevilla) (ES)
- GAÑÁN-CALVO, Alfonso Miguel 41012 Sevilla (ES)
- (74) Representative: Pons Glorieta Ruben Dario 4 28010 Madrid (ES)

(56) References cited:

EP-A1- 0 387 950 DE-A1- 2 148 051 DE-A1- 19 927 537 DE-U1-202007 008 121 FR-A5- 2 080 016 NL-A- 8 602 952

P 3 259 543 B1

Note: Within nine months of the publication of the mention of the grant of the European patent in the European Patent Bulletin, any person may give notice to the European Patent Office of opposition to that patent, in accordance with the Implementing Regulations. Notice of opposition shall not be deemed to have been filed until the opposition fee has been paid. (Art. 99(1) European Patent Convention).

20

25

30

40

45

50

#### Description

[0001] The invention relates to an apparatus and methods for generating droplets. The present disclosure relates to a method of generating a collimated stream of droplets from a flow focusing nozzle, and a method and an apparatus for preserving droplets when immersed (i.e. introduced) in to a drying or hardening environment and/or for protecting droplets during formation.

[0002] Techniques are known for producing collimated droplet streams, such as flow focusing. The droplets in the droplet streams may be subsequently dried to form a powder, for example, which is often referred to as spray drying. However, the number of droplets that are capable of being dried compared to the number of droplets that are produced is often low (e.g., below 80%), due to the droplets being attracted, and/or adhering, to the apparatus. Moreover, as the droplets exit from a droplet forming device, the initial properties/characteristics of the droplets may be lost or affected such that the droplet loses its form and may become agglomerated and/or polydispersed, or may not even be produced.

[0003] Figure 1 illustrates an example of a known system 1 that is used for spray drying. The system 1 includes a droplet forming device 2, such as a flow focusing nozzle. The illustrated example has been simplified, but it will be appreciated that the flow focusing nozzle is fed with one or more fluids 4 to produce a droplet stream at its outlet (not shown). The outlet of the device 2 is within a drying chamber, which is in this background example is an elongate cylindrical conduit 6. The conduit 6 is provided with an air stream via fluid inlet 8. The air stream is at a temperature greater than room temperature (i.e., 20°C to 26°C) such that as the droplet stream exits the device 2, the droplets are air dried. However, the air stream provided within the conduit 6 may prevent droplets being effectively produced or, as is mentioned above, may affect the droplets such that they become agglomerated and/or polydispersed.

[0004] Additionally, document DE202007008121 U discloses an arrangement for the distribution of process air in a drying tower useful in spray drying unit, which comprises ring chambers arranged below the tower covering between a feed pipe and outer wall of the drying tower towards lower product discharge opening. In the center, the drying tower protrudes from the upper tower cover by a coaxial feed tube with spraying unit for products to be dried in the drying tower. The heated process air is brought into the drying tower by the feed pipe present in the tower cover. The drying tower shows process air discharges. The ring chambers are formed by a ring area submerging in the drying area and in the outer wall. Each ring chamber shows a separate feed pipe for the process air. The internal ring chamber is provided with the process air of lower temperature than the external ring chamber during spray drying. The temperature of the process air is increased from the internal ring chamber to external ring chamber or decreased from the internal to external ring chamber using cool spraying procedure. The process air is discharged from the ring chambers with same or different discharge speeds. The ring chambers are separately or jointly connected with a boiler like heat exchanger and with a ventilator for introducing the process air.

[0005] Accordingly, it is an object of the present invention to improve the yield of droplets and/or powder produced by such spray drying apparatus, and to provide a stabilization environment for droplets exiting a droplet forming device before being air dried.

[0006] The present disclosure can be understood with reference to the description of the embodiments set out below, in conjunction with the appended drawings in which:

Figure 1 illustrates schematically an example of a known air drying system;

Figure 2 illustrates an apparatus for spray drying according to a first embodiment of the invention;

Figure 3 illustrates an expanded view of the apparatus illustrated in Figure 2;

Figure 4 illustrates schematically a cross-section through an apparatus according to the first embodiment of the invention;

Figure 5 illustrates, in an upper image, polydispersed, agglomerated droplets, and, in a lower image, monodispersed, dried droplets;

Figure 6 illustrates a partial cross-section through the apparatus according to the first embodiment of the invention;

Figure 7 illustrates a cross-section through a flow focusing nozzle according to the first embodiment of the invention;

Figure 8 illustrates an apparatus for spray drying according to a second embodiment of the invention;

Figure 9 illustrates an apparatus for spray drying according to a third embodiment of the invention; and

Figure 10 illustrates a multi-nozzle fluid dispenser which may be used with the second and third embodiments of the invention.

[0007] According to a first aspect of the invention there is provided an apparatus in accordance with claim 1. Preferred embodiments are defined in claims 2-7. Thus, the droplets during formation are protected from the reaction fluid flow and a greater number of the droplets produced using the liquid dispenser may be persevered. Accordingly, at least 99.5% of the droplets that are produced

will exit the first fluid flow device and can be subsequently transformed to form particles.

[0008] The liquid dispenser is configured to generate the droplet stream using flow focusing.

[0009] The liquid dispenser may be concentrically arranged within the first fluid flow device (i.e., the liquid dispenser and the first fluid flow device may share the same centre or axis, such that they are coaxial) and the first fluid flow device may be concentrically arranged within the second fluid flow device.

[0010] Each of the confinement fluid and/or the reaction fluid may be air.

[0011] The first and/or second fluid flow devices may comprise an elongate conduit and the first and/or second fluid flow devices may comprise a circular conduit (i.e., the conduit has a circular cross section). The conduits may also be non-circular (e.g. oval, elliptical or simple polygon). It will be understood that if a non-circular conduit is used its radius is taken to be half the largest distance between any pair of vertices.

**[0012]** The liquid outlet may comprise a plurality of nozzles, each configured to generate a droplet stream.

**[0013]** The temperature of the second fluid flow may be greater than the temperature of the droplet stream and/or the confinement fluid flow.

**[0014]** The reaction may comprise hardening the droplets in the droplet stream.

[0015] The first fluid flow device may be dimensioned to produce a laminar fluid flow, and may be dimensioned to prevent the droplet stream from being agglomerated.
[0016] According to a second aspect of the invention there is provided a system comprising an apparatus according to any one of the above-described apparatus and a first fluid flow generating device coupled to the first fluid flow device and configured to provide the confinement fluid to the first fluid flow device. The first fluid flow generating device and the first fluid flow device may be configured according to the following expression:

$$U_{min} \ge 0.2 \cdot \left(\frac{\mu_g^2 H^3}{\rho_a^2 D_1^5}\right)^{1/2}$$

where:  $U_{min}$  is the velocity of the flow rate of the confinement fluid;  $\mu_g$  is the viscosity of the confinement fluid; H is the distance between the liquid outlet of the liquid dispenser and the outlet of the first fluid flow device;  $\rho_g$  is the density of the confinement fluid; and  $D_1$  is the external diameter of the liquid dispenser.

[0017] According to a third aspect of the invention there is provided a method in accordance with claim 9. Preferred embodiments of the method are defined in claims 10-13.

**[0018]** The reaction fluid flow may harden the droplets to form particles/capsules and the method may comprise collecting the particles/capsules at an outlet of the second fluid flow device.

**[0019]** A minimum velocity of the confinement fluid flow is represented by the expression:

$$U_{min} \ge 0.2 \cdot \left(\frac{\mu_g^2 \ H^3}{\rho_g^2 D_1^5}\right)^{1/2}$$

where:  $U_{min}$  is the velocity of the flow rate of the confinement fluid;  $\mu_g$  is the viscosity of the confinement fluid; H is the distance between the liquid outlet of the liquid dispenser and the outlet of the first fluid flow device;  $\rho_g$  is the density of the confinement fluid; and  $D_1$  is the external diameter of the liquid dispenser.

[0020] In the

method of generating a collimated stream of droplets from a flow focusing nozzle comprising a discharge orifice, a first fluid dispenser and a second fluid dispenser, wherein the second fluid dispenser is arranged to accelerate, with a carrier fluid, a fluid is dispensed from the first fluid dispenser out of the discharge nozzle, the method comprising configuring the flow focusing nozzle to obtain a geometric standard deviation of the disbursement of droplets from the collimated stream of droplets of less than 1.6 according to the expression:

$$\left(\frac{D^2 \Delta P^2 \rho_l^2 Q_l}{\sigma \mu_l^3}\right)^{1/4} \le \frac{1 + 0.5 \left(\frac{\rho_l}{\rho_d}\right)}{1 + 0.018 \left(\frac{\mu_l}{\mu_d}\right)}$$

where D is the nominal diameter of the discharge orifice,  $\Delta P$  is the pressure of the carrier,  $\rho_l$  is the density of the fluid being dispensed,  $Q_l$  is the flow rate of the fluid being dispensed from the first fluid dispenser,  $\sigma$  is the surface tension between the fluid being dispensed and the carrier fluid,  $\mu_l$  is the viscosity of the fluid being dispensed,  $\rho_d$  is the density of the carrier fluid, and  $\mu_d$  is the viscosity of the carrier fluid.

**[0021]** The flow focusing nozzle may be configured to obtain a geometric standard deviation of the disbursement of droplets from the collimated stream of droplets of less than 1.4, or less than 1.3.

**[0022]** The first fluid dispenser may comprise an inner fluid dispenser configured to dispense a core fluid and an outer fluid dispenser configured to dispense a shell fluid and wherein  $\rho_{l}$ ,  $Q_{l}$ , and  $\mu_{l}$  are of the shell fluid.

[0023] Figure 2 illustrates an apparatus 10 for spray drying. The apparatus 10 can be separated into three main parts; a liquid dispenser or liquid dispensing device 12, a first fluid flow device or confinement/prevention device 14 and a second fluid flow device or reaction/drying device 16. Figure 3 illustrates the apparatus 10 in an expended view.

**[0024]** Figures 2 and 3 are now used to describe the apparatus 10. The liquid dispenser 12 is a form of flow focusing device or nozzle. However, it will be appreciated

that a flow focusing nozzle is used here as an example, but other types of nozzle (e.g., vibrating nozzle, ultrasonic nozzle, or ink-jet nozzle) may be used. The operation of the liquid dispenser 12 is described in more detail with reference to Figure 7. The liquid dispenser 12 includes two fluid inlets 18, 20 for introducing fluids to produce a droplet at a proximate end. For example, the first fluid inlet 18 may be fed with a core-droplet fluid, for example, an oily fragrance, and the second fluid inlet 20 may be fed with a shell-droplet fluid, for example, an aqueous polymeric solution (e.g., Arabic Gum or Shellac). The resultant droplet is in the form of a core-shell or capsule droplet (i.e., a droplet comprising a core and an outer shell). It will be appreciated that the liquid dispenser 12 may only include a single fluid inlet (i.e., one of two fluid inlets 18, 20) for feeding only a single fluid for the production of droplets. Other core or shell fluids include aqueous or organic suspensions, emulsions, organic polymeric solution, aqueous/organic solutions including ingredients, actives, pure liquids or solutions including mixtures of components such as plasticizers, additives, thickeners, and monomers. A third fluid inlet 22 is provided for feeding a carrier fluid such as air. Other gases may be used as a carrier fluid, such as argon or nitrogen. The fluid dispenser 12 includes a circular, elongate conduit or duct 24 having an external diameter of 5 mm which is coupled to each of the fluid inlets 18, 20, 22 and is terminated at a distal end with a nozzle, orifice or outlet 25. The nozzle 25 in this embodiment includes a single outlet orifice having a diameter of 350 µm (not shown). The duct 24 includes one or more internal ducts or conduits which are described in more detail with reference to Figure 7.

[0025] The first fluid flow device or confinement/prevention device 14 includes an elongate circular conduit or first conduit 26 having an internal diameter of 60 mm. The duct 26 has at its proximal end a cap, lid or cover (i.e., a closure device) 28 used to close the first conduit 26 at its proximal end except for an opening or inlet 32 to allow the liquid dispenser 12 to be inserted therein. The diameter of the first conduit 26 is greater than the diameter of the duct 24. The duct 24 of the liquid dispenser 12 is arranged such that at least a portion (i.e., a distal portion) extends through the opening 32 and within the first conduit 26, and a further portion (i.e., a proximal portion) extends outside of the first conduit 26. As can be seen in Figure 2, the outlet 25 is arranged part way along the length of the first conduit 26 (i.e., the liquid dispenser is terminated within the first conduit 26). The liquid dispenser 12 is arranged concentrically within the first fluid flow device 14. The closure device 28 includes an inlet 30 that is coupled to an internal channel 66 (see figure 4) to allow the passage of fluid from the inlet 30 through the internal channel 66 of the closure device 28. The closure device 28 also includes an opening 68 (see figure 4) coupled to the internal channel 66 to allow the passage of a fluid into the first conduit 26. The opening 68 within the closure device 28 may be circular to match the cross

sectional shape of the first conduit 26. The circular duct or first conduit 26 has an opening, exit or outlet 34 at its distal end to allow for the passage of droplets and the fluid fed to the fluid inlet 30.

[0026] The second fluid flow device or reaction device 16 includes an elongate circular second conduit 36 having an internal diameter of 147 mm. The second conduit 36 has at its proximal end a cap, lid or cover (i.e., a closure device) 38 that closes the duct 36 at its proximal end except for an opening or inlet 42 to allow the first conduit 26 of the first fluid flow device 14 to be inserted therein. The diameter of the duct 36 is greater than the diameter of the first conduit 26. The first conduit 26 of the first flow device 14 is arranged such that at least a portion (i.e., a distal portion) extends through the opening 42 and within the second conduit 36, and a further portion (i.e., a proximal portion) extends outside of the second conduit 36. As can be seen in Figure 2, the opening 34 is arranged part way along the length of the second conduit 36 (i.e., the first fluid flow device 14 is terminated within the second conduit 36). The first fluid flow device 14 is arranged concentrically within the second fluid flow device 16. The closure device 38 includes an inlet 40 that is coupled to an internal channel 71 (see figure 4) to allow the passage of fluid from the inlet 40 through the internal channel 71 of the closure device 38. The closure device 38 also includes an opening 73 (see figure 4) coupled to the internal channel 71 to allow the passage of a fluid into the second conduit 36. The opening 73 within the closure device 38 may be circular to match the cross sectional shape of the second conduit 36. The circular second conduit 36 has an opening, exit or outlet 44 at its distal end to allow for the passage of droplets and the fluid fed to the fluid inlets 30, 40. Alternatively, fluid may be drawn from the outlet 44 by creating a negative pressure at the outlet 44 using a suitable suction pump.

[0027] In this example, the second (reaction) fluid flow device 16 is manufactured from stainless steel (i.e., the closure device 38) and glass (i.e., the elongate second conduit 36), and the first (confinement) fluid flow device 14 is manufactured from similar materials. The liquid dispenser 12 is manufactured from plastics and stainless steel.

**[0028]** Glass has been used in this example to reduce any large scale fluctuations that could result from any internal roughness on the interior walls of the elongate conduits 26, 36.

[0029] Figure 4 illustrates schematically a cross-section through the apparatus 10 illustrated in Figure 2. The same reference numerals are used in Figure 4 as used in Figures 2 and 3 for like features. Figure 4 illustrates the internal channel 66 of the closure device 28 and its associated circular opening 68. Figure 4 also illustrates the internal channel 71 of the closure device 38 and its associated circular opening 73.

[0030] In operation, the apparatus 10 is coupled to a number of feeding systems, which may include compressors which are configured to pressurize air and feed the

25

40

45

pressurized air to various inlets of the apparatus 10. The fluid dispenser 12 is coupled to a compressor 50 via a pipe (line or passage) 52 at its inlet 22. The compressor 50 in this example feeds air to the dispenser 12 so that the air provides a carrier for droplets such that an aerosol is dispensed by the liquid dispense 12. However, a pump (e.g., a pressurized container, micropump, screw pump, or piezoelectric pump) could be used to feed the liquid dispenser 12 with a fluid (e.g., water if an organic solution is used) to form an emulsion. A pump 54 is coupled to the inlets 18, 20 via a pipe (line or passage) 56 to provide a core and shell-droplet material. It will be appreciated that only a single pump 54 is illustrated for simplicity, but when producing core-shell droplets, two separate pumps are used; one for each fluid inlet. Of course, droplets may also be produced using a single liquid such that only a single pump is required. A compressor 58 is coupled to the inlet 30 of the first fluid flow device 14 via a pipe (line or passage) 60 and a compressor 62 is coupled to the inlet 40 of the second fluid flow device 16 via a pipe (line or passage) 64. Although not shown in the figure, a suction pump may also be arranged at the outlet 44 of the second fluid flow device 16 to create a negative air pressure at the outlet 44. In this example, the compressors 58, 62 feed the apparatus 10 with air, but other gases may be used. For example, the compressor 62 may feed the second fluid flow device 16 with air which has a relative humidity of 60% or greater, for example, to react with the droplets containing cyanoacrylates, for example, exiting from the first fluid flow device 14. Furthermore, the compressor 62 typically feeds air to the apparatus having a temperate that is higher than room temperature (i.e., 20°C to 26°C). The compressor includes a heating element which is controlled to increase the temperature of the air flowing through the compressor. In this example, the temperature of reaction fluid is greater than the confinement fluid, which may be used for drying droplets, for example. However, it will be appreciated that the temperatures of the confinement and reaction flow may be different, such that the temperature of the confinement fluid is greater than the reaction fluid, for example. This may be achieved by increasing the temperature of the confinement fluid above room temperature while maintaining the temperature of the reaction fluid at room temperature, or alternatively, cooling the reaction fluid while maintaining the confinement fluid at room temperature. In an alternative configuration where a negative pressure is created at the outlet 44, the element labelled 62 in the figure could be replaced with a heating or cooling device only that allows fluid (e.g., air) to be drawn there through and in to the second fluid flow device 16 via inlet 40.

[0031] During use, the liquid dispenser 12 is operated and produces a collimated stream of droplets. In this example, the liquid dispenser 12 produces a single stream of droplets. The nozzle 25 dispenses droplets within the first conduit 26 of the first fluid flow device 14, whereby a movement of air (or a flow/stream of air) produced using pump 58 creates a preventative air flow that permits the

droplet stream to move generally along a trajectory that prevents the droplets from interacting (i.e., adhering) to an interior surface of the first conduit 26 of the first fluid flow device 14. The stream of air also permits the droplets to stabilize after exiting the dispenser. The droplet stream exits the opening 34 of the first fluid flow device 14, and enters the second conduit 36 of the second fluid flow device 16 and continues to move along a similar trajectory within the flow of air that is provided by the first fluid flow device 14 and the second fluid flow device 16, whereby a movement of air (or a flow/stream of air) produced using pump 62 creates a reaction air flow/stream that reacts with the droplets. In this example, the reaction air flow is an air flow at a temperature that is greater than room temperature which dries the droplets to form of powder. The first and second air streams will mix via diffusion, for example, such that the combined airstream will dry the droplets to form particles/capsules, or collectively a powder, which are collected at the opening 44. For example, the powder may be collected using different types of reservoirs, cyclones, and/or filters.

Two specific examples are now described using the apparatus 10 described in association with Figures 2, 3 and 4.

[0032] In example one, a solution of Shellac (15% w/v) is dispensed from a single nozzle of the fluid dispenser 12 at a volume flow rate of 30 mL/h (i.e., 8.333\*10<sup>-9</sup> m<sup>3</sup>/s) using pump 54, and a carrier phase is introduced at a pressure of 90 mbar using compressor 50. The velocity of the droplets exciting the fluid dispenser 12 is 100 m/s and the droplets have a diameter of 80  $\mu$ m (+/- 5  $\mu$ m). The air flow from the second fluid flow device 16, produced by compressor 62, is at a volume flow rate of 320 L/min (i.e., 0.0053 m<sup>3</sup>/s) at a temperature of 78.5°C, which equates to a Reynolds number of 2,182 (i.e., the flow is laminar). The air flow from the first or preventing fluid flow device 14, produced by compressor 58, is at a volume flow rate of 60 L/min (i.e., 0.001 m<sup>3</sup>/s) at room temperature, which equates to a Reynolds number of 1,150 (i.e., the flow is laminar). In example one, the nozzle 25 of the fluid dispenser 12 is terminated at a distance of 10 mm from the opening or fluid exit 34 of the first fluid flow device 14. The apparatus 10 is operated according to example one, produced dried particles with a yield of 90%. It is noted that particles are produced, since only a single liquid was dispensed from the liquid dispenser 12. [0033] In example two, a solution of Shellac (10% w/v) is dispensed from a single concentric nozzle of the fluid dispenser 12 at a volume flow rate of 30 mL/h (i.e., approximately 8.333\*10<sup>-9</sup> m<sup>3</sup>/s) using pump 54, and a carrier phase is introduced at a pressure of 90 mbar using compressor 50. In example two, the Shellac is a shell liquid and water is dispensed as a core liquid at a volume flow rate of 1 mL/h (i.e., approximately 2.77\*10<sup>-10</sup> m<sup>3</sup>/s). The velocity of the droplets exciting the fluid dispenser 12 is 100 m/s and the droplets have a diameter of 80  $\mu$ m  $(+/-5 \mu m)$ . The air flow from the second fluid flow device 16, produced by compressor 62, is at a volume flow rate

of 80 L/min (i.e., approximately 0.0013 m<sup>3</sup>/s) at a temperature of 70°C to 75°C, which equates to a Reynolds number of 1,982 (i.e., the flow is laminar). The air flow from the first or preventing fluid flow device 14, produced by compressor 58, is at a volume flow rate of 8 L/min (i.e., approximately 0.00013 m<sup>3</sup>/s) at room temperature, which equates to a Reynolds number of 543 (i.e., the flow is laminar). In example two, the nozzle 25 of the fluid dispenser 12 is terminated at a distance of 10 mm from the opening 34 of the first fluid flow device 14. That is to say that the distance travelled by the dispensed droplets is 10 mm within the first fluid flow device 14. During operation, the apparatus 10, operated according to example two, produced dried, monodispersed particles. It will be understood that as the fluid in the core is just water in this example, the water is evaporated and dried. Therefore, nothing is retained in the core and the final dried droplet is like a particle without a core. This is in contrast to a capsule which includes a core material.

[0034] The applicant has determined that the velocity of the confinement fluid in apparatus 10 such that at least 99.5% of the droplets produced using the liquid dispenser 12 are recoverable and are able to pass to the second, reaction fluid flow device 16, (0.5% of the droplets either adhere to the internal walls of the first fluid flow device 14 or become agglomerated, for example) is expressed according to the following formula:

$$U_{min} \ge k \left(\frac{\mu_g^2 H^3}{\rho_g^2 D_1^5}\right)^{1/2}$$

where  $U_{min}$  is the velocity of the confinement fluid, k is a constant in the range of 0.2 to 2.0, and includes 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9, 1.0, 1.1, 1.2, 1.3, 1.4, 1.5, 1.6, 1.7, 1.8, 1.9, 2.0 at least,  $\mu_g$  is the viscosity of the fluid fed by compressor 58 (i.e., the viscosity of the confinement fluid), H is the distance between a liquid outlet or nozzle 25 of the liquid dispenser 12 and a fluid exit 34 of the first fluid flow device 14,  $\rho_g$  is the density of the confinement fluid, and  $D_1$  is the external diameter of the liquid dispenser 12.

[0035] Moreover, to achieve laminar flow in the first, confinement, fluid flow device 14, Reynolds number should be less than 4•10³, where Reynolds number is provided by the following known expression:

$$4 \cdot 10^3 \ge \frac{\rho_g U D_3}{\mu_g}$$

where U is the velocity of the confinement fluid and  $D_3$  is the internal diameter of the first fluid flow device. Accordingly, the velocity of the confinement fluid can be determined within the range of:

$$4 \cdot 10^3 \frac{\mu_g}{\rho_g D_3} \ge U \ge k \left(\frac{\mu_g^2 H^3}{\rho_g^2 D_1^5}\right)^{1/2}$$

[0036] In practical terms, the cross section of the protection device (i.e., the first fluid flow device) may be chosen/designed to obtain the required velocity using an airflow rate as low as possible while considering the confinement air is cold and will cool the hot drying air, which may decrease its drying capacity and avoiding droplet interaction with the internal walls of the protection device in order to achieve less than 0.5% losses.

[0037] Figure 5 illustrates two images of droplets. In the upper image, polydispersed, agglomerated droplets are illustrated that may be produced using the known system illustrated in Figure 1, for example. The droplets may have become agglomerated shortly after leaving the fluid dispenser 2. In the lower image, dried, monodispersed particles are illustrated, which are formed from core-shell droplets dispensed according to example two described above.

[0038] Figure 6 illustrates a partial cross-section through the apparatus illustrated in Figures 2, 3 and 4. In particular, a cross-section through the elongate second conduit 36 of the second fluid flow device 16, the elongate first conduit 26 of the first fluid flow device 14 and the fluid dispenser 12. The figure illustrates schematically the fluid flow from each of the devices and how it interacts. As can be seen from the figure, the fluid exiting the fluid dispenser 12 is travelling at a different velocity to the fluid flow from the first fluid flow device 14, such that there may be sheering where the two fluids meet, which may cause local turbulence. However, the first fluid flow device 14 is dimensioned so as to prevent any disturbance to the droplet stream exiting the fluid dispenser 12. Similarly, the fluid exiting the first fluid device 14 is typically travelling at a difference velocity to the fluid flow exiting from the second fluid flow device 16, such that there may be sheering forces where the two fluids meet, which may cause local turbulence. Any disturbance between the first and second (i.e., the confinement and reaction) fluid flows is less significant than any disturbance between the droplets exiting the fluid dispense 2 and the first fluid flow, since once the droplets have exited from the first fluid flow and into the second fluid flow, they are already typically stabilized. Once the first (confinement) fluid flow has exited the first flow device 14 into the second (reaction) fluid flow, the two fluids will gradually mix by diffusion as the fluids flow from the left to the right in the orientation shown in the figure, such that the increased temperature of the second fluid flow will effectively increase the temperature of the first fluid flow and dry the droplets to form a powder.

**[0039]** Figure 7 illustrates a cross-section through a flow focusing nozzle according to a second embodiment of the invention. The nozzle illustrated in Figure 7 forms the distal end of the liquid dispenser 12 illustrated in Fig-

ures 2 and 3. In this regard the duct 24 forms the outer channel or conduit of the nozzle. The nozzle is concentrically arranged and includes a first inner channel or fluid passage 51 (i.e., an outer fluid dispenser) arranged concentrically within the duct 24 and a second inner channel or fluid passage 53 (i.e., an inner fluid dispenser) arranged concentrically within the first inner channel 51. Although not illustrated in Figure 7, the channel or duct formed between the duct 24 and the first inner channel 51 is coupled to the fluid inlet 22 illustrated in Figures 2, 3 and 4 for feeding a carrier fluid 59, the channel or duct formed between the first inner channel 51 and the second inner channel 53 is coupled to the second fluid inlet 20 illustrated in Figures 2, 3 and 4 for feeding a shell-droplet fluid 55, and the second inner channel 53 is coupled to the first fluid inlet 18 illustrated in Figures 2, 3 and 4 for feeding a core-droplet fluid 57. Each of the first and second inner ducts 51, 53 are terminated within the duct 24 such that fluid exiting from these inner ducts interacts with the carrier fluid 59 within duct 24. The nozzle, orifice or outlet 25 allows the passage of the combined coreshell fluid 57, 55 and the carrier fluid 59 out of the device first as a stream, which subsequently forms droplets 61. It will be appreciated that the first inner wall 51 is optional, and is not typically used if a single fluid is used to generate the droplets (i.e., the generated droplets are not coreshell type droplets). Each of the fluids 55, 57, 59 illustrated in Figure 7 may be a gas or a liquid.

[0040] During operation, fluid is fed to each of the fluid inlets and travels along each respective duct to the orifice 25. As the fluids 55, 57 exit the first and second inner ducts 51, 53 they do not typically mix, such that the first fluid 55 forms an outer shell to the second fluid 57. The carrier fluid 59 accelerates the first and second fluids 55, 57 to generate a monodispersed beam of droplets.

[0041] The applicant has determined that a collimated stream of droplets having a geometric standard deviation of the disbursement of droplets from the collimated stream of droplets (i.e., a measure of the spread of droplets with respect to the droplet stream) of less than 1.6 can be achieved with a nozzle configured to according to the expression:

$$\left(\frac{D^2 \Delta P^2 \rho_l^2 Q_l}{\sigma \mu_l^3}\right)^{1/4} \le \frac{1 + 0.5 \left(\frac{\rho_l}{\rho_d}\right)}{1 + 0.018 \left(\frac{\mu_l}{\mu_d}\right)}$$

where D is the nominal diameter of the discharge orifice (i.e., orifice 25),  $\Delta P$  is the pressure of the carrier fluid,  $\rho_l$  is the density of the fluid being dispensed,  $Q_l$  is the flow rate of the fluid being dispensed from the first fluid dispensing device,  $\sigma$  is the surface tension between the fluid being dispensed and the carrier fluid,  $\rho_d$  is the density of the carrier fluid, and  $\mu_d$  is the viscosity of the carrier fluid. In the nozzle illustrated in Figure 7, two fluids are used to form a core-shell type droplet. In this case, the density

of the fluid being dispensed  $(\rho_l)$  is taken to be the greatest density of the two fluids being dispensed. In the nozzle illustrated in Figure 7, two fluids 55, 57 are dispensed to form core-shell droplets, such that the density and viscosity values (i.e.,  $\rho_d$  and  $\mu_d$ ) are typically of the dispensed fluid with the greatest fluid velocity, which is typically the shell forming fluid 55.

[0042] Preferably, a geometric standard deviation of the disbursement of droplets from the collimated stream of droplets of less than 1.4, and more preferably less than 1.3 can be achieved using a nozzle configured according to the above-expression. The angle of the fluid dispensed from a nozzle (angle 63 illustrated in Figure 7) is preferably less than or equal to 5 degrees.

[0043] Figure 8 illustrates an apparatus 70 for spray drying according to a second embodiment of the invention. Apparatus 70 is similar to apparatus 10 in that it includes the same second flow device 16. Apparatus 70 also includes a similar first fluid flow device 14' to that described in association with apparatus 10, except the opening 74 of the first fluid flow device 14' is greater than the opening 32 of the first fluid flow device 14 to accommodate a larger fluid dispensing device or fluid dispenser 72. The fluid dispensing device 72 is larger in diameter than the fluid dispenser 12, since it includes multiple nozzles to produce multiple droplet streams, as is described in association with Figure 10. The apparatus 70 is operated in the same manner as apparatus 10, except for the fluid dispenser 72, which is described in association with Figure 10.

[0044] The apparatus 70 illustrated in Figure 8 includes a multi-nozzle dispensing device 72. Using the expression for a single nozzle dispensing device presented above, it is possible to predict the velocity of the confinement fluid required in a multi-nozzle dispensing device to recover at least 99.5% of the droplets produced using the multi-nozzle dispensing device. This is done by replacing the value of H in the previously presented expression with the expression  $H/(1-D_2/D_3)$ , where H is the distance between a liquid outlet or nozzle of the liquid dispenser 72 and a fluid exit of the first fluid flow device 14',  $D_2$  is the smallest diameter of a virtual circle containing the centres of the discharge orifices/exits of the nozzles in the multi-nozzle dispensing device 72 and  $D_3$  is the internal diameter of the first fluid flow device 14'. The following expression is obtained:

$$U_{min} \ge k \left( \frac{\mu_g^2 \ H^3}{\rho_g^2 D_1^5 (1 - D_2/D_3)^3} \right)^{1/2}$$

where  $U_{min}$  is the velocity of the confinement fluid, k is a constant in the range of 0.2 to 2.0 and includes 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9, 1.0, 1.1, 1.2, 1.3, 1.4, 1.5, 1.6, 1.7, 1.8, 1.9, 2.0 at least,  $\mu_g$  is the viscosity of the confinement fluid, H is the distance between a liquid outlet or nozzle of the multi-nozzle dispensing device 72 and

a fluid exit of the first fluid flow device 14',  $\rho_g$  is the density of the confinement fluid,  $D_1$  is the external diameter of the multi-nozzle liquid dispenser 72,  $D_2$  is the smallest diameter of a virtual circle containing the centres of the discharge orifices/exits of the nozzles in the multi-nozzle dispensing device 72 and  $D_3$  is the internal diameter of the first fluid flow device 14'.

[0045] A specific example is now described using the apparatus 70 described in association with Figure 8.

[0046] In example three, a solution of Arabic Gum (10% w/v) is dispensed from a multiple nozzle including 8 nozzles of the fluid dispenser 72 at a volume flow rate of 30 mL/h per nozzle (i.e., approximately 8.333\*10-9 m<sup>3</sup>/s), and a carrier phase is introduced at a pressure of 85 mbar. The droplets have a diameter of 82  $\mu$ m (+/- 5  $\mu$ m). The air flow from the second fluid flow device 16 is at a volume flow rate of 550 L/min (i.e., approximately 0.0092 m<sup>3</sup>/s) at a temperature of 70°C to 75°C, which equates to a Reynolds number of 4,330 (i.e., the flow is NOT laminar). The air flow from the first or preventing fluid flow device 14' is at a volume flow rate of 50 L/min (i.e., approximately 0.00083 m<sup>3</sup>/s) at room temperature, which equates to a Reynolds number of 982 (i.e., the flow is laminar). In example three, the nozzles 25 of the fluid dispenser 72 are terminated at a distance of 10 mm from the opening of the first fluid flow device 14'. That is to say that the distance travelled by the dispensed droplets is 10 mm within the first fluid flow device 14'. The first fluid device 14' has a diameter of 60 mm. During operation, the apparatus 70, operated according to example three, produced dried, monodispersed particles.

[0047] Figure 9 illustrates an apparatus 80 for spray drying according to a third embodiment of the invention. Apparatus 80 is similar to apparatus 70 and includes four fluid flow devices 14', each having a fluid dispenser 72. The second fluid flow device 16' is also similar to the second fluid flow device 16 of apparatus 10, but as can be seen it is greater in diameter and includes four openings 82 to accommodate the four fluid flow devices 14'. The fluid dispensing device 72 is described in association with Figure 10. The apparatus 80 is operated in the same manner as apparatus 70.

[0048] Figure 10 illustrates a multi-nozzle fluid dispenser 72 which may be used with the second and third embodiments of the invention. The multi-nozzle fluid dispenser 72 includes 8 nozzles 25 in this example. Each of the nozzles 25 includes an associated duct 24, first inner channel 51, and second inner channel 53, as is illustrated in Figure 7. These are not illustrated in Figure 10 for simplicity. Each of the ducts for each nozzle is also coupled to an associated compressor or pump. It will be appreciated that the same compressor/pump may feed all of the respective ducts 24, first inner channels 51, and second inner channels 53 depending on the type of droplets being formed.

**[0049]** It will be appreciated that the dimensions provided for the apparatus described herein are only examples and should not be considered to limit the scope of

the invention. In this regard, the outer diameter of the fluid dispenser may range from 5 to 100 mm. Furthermore, the internal diameter of the first fluid flow device may range from 5.5 mm to 1000 mm and the internal diameter of the second fluid flow device may range from 50 mm to 2000. Moreover, the ratio between the outer diameter of the fluid dispenser and the internal diameter of the first fluid flow device is from 1:1.1 to 1:100. It is noted that the values for density and viscosity mentioned herein are with respect to room temperature (i.e., 20°C to 26°C). The viscosity mentioned herein is a dynamic viscosity, and the density and viscosity values mentioned herein have been determined using the standard test method for dynamic viscosity and density of liquids by Stabinger Viscometer, for example, in accordance with the ASTM standards.

**[0050]** The examples are typically illustrated in a vertical orientation such that the droplets are introduced at the top and exit at the bottom of the apparatus. This is the typical orientation of the apparatus during use.

[0051] In the examples described herein, the confinement flow device is a single elongate conduit. However, this can be increased to include multiple elongate conduits arranged concentrically. The multiple elongate conduits will also have different lengths, such that the shortest conduit will terminate within the next longest conduit and so forth. Thus, a nested, or telescopic, confinement device is achieved.

#### **Claims**

1. An apparatus (10, 70, 80) for generating droplets, the apparatus (10, 70, 80) comprising:

a liquid dispenser (12, 72) comprising a liquid outlet (25) adapted to generate a collimated droplet stream;

a first fluid flow device (14, 14') configured to generate a confinement fluid flow for confining a trajectory of the droplet stream therein, the first fluid flow device comprises an outlet (34) arranged to allow the droplet stream to exit therefrom, and further comprising a first conduit (26) wherein the liquid outlet (25) is arranged so as to dispense the droplets within the first conduit (26) of the first fluid device (14,14'),

a second fluid flow device (16, 16') configured to generate a reaction fluid flow for reacting with droplets in the droplet stream, the second flow device (16, 16') comprising a second conduit (36) and the first fluid flow device (14, 14') is arranged within the second fluid flow device (16, 16') so that the droplet stream exits in the second conduit (36) of the second fluid flow device (16,16')

2. The apparatus (10, 70, 80) of claim 1, wherein the

55

35

45

liquid dispenser (12, 72) is concentrically arranged within the first fluid flow device (14, 14'), and/or the first fluid flow device (14, 14') is concentrically arranged within the second fluid flow device (16, 16').

- 3. The apparatus (10, 70, 80) of claim 1 or claim 2, wherein each of the confinement fluid and/or the reaction fluid is air.
- 4. The apparatus (10, 70, 80) of any one of claims 1 to 3, wherein the first (14, 14') and/or second (16, 16') fluid flow devices comprise an elongate conduit (24, 26), and the elongate conduit (24, 16) may be circular
- 5. The apparatus (10, 70, 80) of any one of the preceding claims, wherein the liquid outlet (25) comprises a plurality of nozzles, each configured to generate a droplet stream.
- **6.** The apparatus (10, 70, 80) of any one of the preceding claims, wherein the liquid dispenser (12, 72) is configured to generate the droplet stream using flow focusing.
- 7. The apparatus (10, 70, 80) of any one of the preceding claims, wherein the first fluid flow device (14, 14') is dimensioned to produce a laminar fluid flow.
- 8. A system comprising the apparatus (10, 70, 80) of any one of claims 1 to 7 and a first fluid flow generating device coupled to the first fluid flow device (14, 14') and configured to provide the confinement fluid to the first fluid flow device (14, 14').
- 9. A method of generating droplets using an apparatus according to any one of claims 1 to 8, the method comprising:

generating a droplet stream from a liquid dispenser (12, 72);

generating a confinement fluid flow within a first fluid flow device (14, 14') to confine the trajectory of the droplet stream, wherein the droplet stream is generated within the confinement fluid flow and exits from an outlet (34) of the first fluid flow device (14, 14'); and

generating a reaction fluid flow within a second fluid flow device (16, 16') for reacting with droplets in the droplet stream, wherein the confinement fluid flow is generated within the reaction fluid flow.

**10.** The method of claim 9, wherein the reaction fluid flow hardens the droplets to form particles and the method comprises collecting the particles at an outlet (44) of the second fluid flow device (16, 16').

**11.** The method of claim 9 or claim 10, wherein a minimum velocity of the confinement fluid flow is represented by the expression:

 $U_{min} \ge 0.2 \cdot \left(\frac{\mu_g^2 \ H^3}{\rho_g^2 D_1^5}\right)^{1/2}$ 

where:  $U_{min}$  is the velocity of the flow rate of the confinement fluid;

 $\mu_g$  is the viscosity of the confinement fluid; H is the distance between the liquid outlet (25) of the liquid dispenser (12, 72) and the outlet (34) of the first fluid flow device (14, 14');  $\rho_g$  is the density of the confinement fluid; and  $D_1$  is the external diameter of the liquid dispenser (12, 72).

- 12. The method of anyone of claims 9-11, wherein the temperature of the second fluid flow is greater than the temperature of the droplet stream and/or the confinement fluid flow.
- 25 13. The method of anyone of claims 9-12, wherein the reaction comprises hardening the droplets in the droplet stream.

#### 30 Patentansprüche

 Vorrichtung (10, 70, 80) zum Erzeugen von Tröpfchen, wobei die Vorrichtung (10, 70, 80) Folgendes umfasst:

eine Flüssigkeitsabgabevorrichtung (12, 72), umfassend einen Flüssigkeitsauslass (25), der geeignet ist, einen kollimierten Tröpfchenstrom zu erzeugen:

eine erste Fluidströmungsvorrichtung (14, 14'), die zum Erzeugen einer Beschränkungsfluidströmung zum Beschränken einer Trajektorie des Tröpfchenstroms darin konfiguriert ist, wobei die erste Fluidströmungsvorrichtung einen Auslass (34) umfasst, der angeordnet ist, um einem Tröpfchenstrom das Austreten daraus zu ermöglichen, und ferner eine erste Leitung (26) umfasst, wobei der Flüssigkeitsauslass (25) angeordnet ist, um die Tröpfchen innerhalb der ersten Leitung (26) der ersten Fluidvorrichtung (14,14') abzugeben,

eine zweite Fluidströmungsvorrichtung (16, 16'), die zum Erzeugen einer Reaktionsfluidströmung zum Reagieren mit Tröpfchen in dem Tröpfchenstrom konfiguriert ist, wobei die zweite Strömungsvorrichtung (16, 16') eine zweite Leitung (36) umfasst und wobei die erste Fluidströmungsvorrichtung (14, 14') innerhalb der

25

zweiten Fluidströmungsvorrichtung (16, 16') angeordnet ist, sodass der Tröpfchenstrom in der zweiten Leitung (36) der zweiten Fluidströmungsvorrichtung (16,16') austritt.

- 2. Vorrichtung (10, 70, 80) nach Anspruch 1, wobei die Flüssigkeitsabgabevorrichtung (12, 72) konzentrisch innerhalb der ersten Fluidströmungsvorrichtung (14, 14') angeordnet ist und/oder die erste Fluidströmungsvorrichtung (14, 14') konzentrisch innerhalb der zweiten Fluidströmungsvorrichtung (16, 16') angeordnet ist.
- 3. Vorrichtung (10, 70, 80) nach Anspruch 1 oder Anspruch 2, wobei jedes des Beschränkungsfluids und/oder des Reaktionsfluids Luft ist.
- 4. Vorrichtung (10, 70, 80) nach einem der Ansprüche 1 bis 3, wobei die erste (14, 14') und/oder zweite (16, 16') Fluidströmungsvorrichtung eine längliche Leitung (24, 26) umfassen und die längliche Leitung (24, 16) kreisförmig sein kann.
- **5.** Vorrichtung (10, 70, 80) nach einem der vorhergehenden Ansprüche, wobei der Flüssigkeitsauslass (25) mehrere Düsen umfasst, die jeweils zum Erzeugen eines Tröpfchenstroms konfiguriert sind.
- 6. Vorrichtung (10, 70, 80) nach einem der vorhergehenden Ansprüche, wobei die Flüssigkeitsabgabevorrichtung (12, 72) zum Erzeugen des Tröpfchenstroms unter Verwendung von Strömungsfokussierung konfiguriert ist.
- 7. Vorrichtung (10, 70, 80) nach einem der vorhergehenden Ansprüche, wobei die erste Fluidströmungsvorrichtung (14, 14') zum Herstellen einer laminaren Fluidströmung bemessen ist.
- 8. System, umfassend die Vorrichtung (10, 70, 80) nach einem der Ansprüche 1 bis 7 und einer ersten Fluidströmungs-Erzeugungsvorrichtung, die an die erste Fluidströmungsvorrichtung (14, 14') gekoppelt ist und zum Bereitstellen des Beschränkungsfluids an die erste Fluidströmungsvorrichtung (14, 14') konfiguriert ist.
- Verfahren zum Erzeugen von Tröpfchen unter Verwendung einer Vorrichtung nach einem der Ansprüche 1 bis 8, wobei das Verfahren Folgendes umfasst:

Erzeugen eines Tröpfchenstroms aus einer Flüssigkeitsabgabevorrichtung (12, 72); Erzeugen einer Beschränkungsfluidströmung innerhalb einer ersten Fluidströmungsvorrichtung (14, 14'), um die Trajektorie des Tröpfchenstroms zu beschränken, wobei der Tröpfchenstrom innerhalb der Beschränkungsfluidströ-

mung erzeugt wird und aus einem Auslass (34) der ersten Fluidströmungsvorrichtung (14, 14') austritt; und

Erzeugen einer Reaktionsfluidströmung in einer zweiten Fluidströmungsvorrichtung (16, 16') zum Reagieren mit Tröpfchen in dem Tröpfchenstrom, wobei die Beschränkungsfluidströmung innerhalb der Reaktionsfluidströmung erzeugt wird.

- 10. Verfahren nach Anspruch 9, wobei die Reaktionsfluidströmung die Tröpfchen verfestigt, um Partikel zu bilden und wobei das Verfahren das Sammeln der Partikel an einem Auslass (44) der zweiten Fluidströmungsvorrichtung (16, 16') umfasst.
- 11. Verfahren nach Anspruch 9 oder Anspruch 10, wobei eine Mindestgeschwindigkeit der Beschränkungsfluidströmung durch folgenden Ausdruck dargestellt wird:

$$U_{min} \ge 0.2 \cdot \left(\frac{\mu_g^2 \ H^3}{\rho_g^2 D_1^5}\right)^{1/2}$$

wobei:  $U_{min}$  die Geschwindigkeit der Strömungsrate des Beschränkungsfluids ist;  $\mu_g$  die Viskosität des Beschränkungsfluids ist; H der Abstand zwischen dem Flüssigkeitsauslass (25) der Flüssigkeitsausgabevorrichtung (12, 72) und dem Auslass (34) der ersten Fluidströmungsvorrichtung (14, 14') ist;  $\rho_{\rm g}$  die Dichte des Beschränkungsfluids ist; und  $D_1$  der Außendurchmesser der Flüssigkeitsabgabevorrichtung (12, 72) ist.

- 12. Verfahren nach einem der Ansprüche 9 bis 11, wobei die Temperatur der zweiten Fluidströmung höher als die Temperatur des Tröpfchenstroms und/oder der Beschränkungsfluidströmung ist.
- **13.** Verfahren nach einem der Ansprüche 9 bis 12, wobei die Reaktion das Verfestigen der Tröpfchen in dem Tröpfchenstrom umfasst.

#### Revendications

**1.** Appareil (10, 70, 80) pour la génération de goutte-lettes, l'appareil (10, 70, 80) comprenant :

un distributeur de liquide (12, 72) comprenant une sortie de liquide (25) adaptée pour générer un flux de gouttelettes collimaté; un premier dispositif d'écoulement de fluide (14, 14') configuré pour générer un écoulement de fluide de confinement pour confiner une trajec-

20

30

toire du flux de gouttelettes à l'intérieur, le premier dispositif d'écoulement de fluide comprend une sortie (34) agencée pour permettre au flux de gouttelettes de sortir de celui-ci, et comprenant en outre un premier conduit (26) dans lequel la sortie de liquide (25) est agencée de manière à distribuer les gouttelettes dans le premier conduit (26) du premier dispositif à fluide (14, 14'),

un deuxième dispositif d'écoulement de fluide (16, 16') configuré pour générer un écoulement de fluide de réaction pour réagir avec des gout-telettes dans le flux de gouttelettes, le deuxième dispositif d'écoulement (16') comprenant un deuxième conduit (36) et le premier dispositif d'écoulement de fluide (14, 14') est agencé dans le deuxième dispositif d'écoulement de fluide (16, 16') de sorte que le flux de gouttelettes sort dans le deuxième conduit (36) du deuxième dispositif d'écoulement de fluide (16, 16').

- 2. Appareil (10, 70, 80) selon la revendication 1, dans lequel le distributeur de liquide (12, 72) est agencé concentriquement dans le premier dispositif d'écoulement de fluide (14, 14'), et/ou le premier dispositif d'écoulement de fluide (14, 14') est agencé concentriquement dans le deuxième dispositif d'écoulement de fluide (16, 16').
- 3. Appareil (10, 70, 80) selon la revendication 1 ou la revendication 2, dans lequel chacun parmi le fluide de confinement et/ou le fluide de réaction est de l'air.
- 4. Appareil (10, 70, 80) selon l'une quelconque des revendications 1 à 3, dans lequel les premier (14, 14') et/ou deuxième (16, 16') dispositifs d'écoulement de fluide comprennent un conduit allongé (24, 26), et le conduit allongé (24, 16) peut être circulaire.
- Appareil (10, 70, 80) selon l'une quelconque des revendications précédentes, dans lequel la sortie de liquide (25) comprend une pluralité de buses, chacune étant configurée pour générer un flux de gouttelettes.
- 6. Appareil (10, 70, 80) selon l'une quelconque des revendications précédentes, dans lequel le distributeur de liquide (12, 72) est configuré pour générer le flux de gouttelettes en utilisant la focalisation en écoulement.
- 7. Appareil (10, 70, 80) selon l'une quelconque des revendications précédentes, dans lequel le premier dispositif d'écoulement de fluide (14, 14') est dimensionné pour produire un écoulement de fluide laminaire.
- 8. Système comprenant l'appareil (10, 70, 80) selon

l'une quelconque des revendications 1 à 7 et un premier dispositif de génération d'écoulement de fluide couplé au premier dispositif d'écoulement de fluide (14, 14 ') et configuré pour fournir le fluide de confinement au premier dispositif d'écoulement de fluide (14, 14').

**9.** Procédé de génération de gouttelettes en utilisant un appareil selon l'une quelconque des revendications 1 à 8, le procédé comprenant :

la génération d'un flux de gouttelettes à partir d'un distributeur de liquide (12, 72);

la génération d'un écoulement de fluide de confinement dans un premier dispositif d'écoulement de fluide (14, 14 ') pour confiner la trajectoire du flux de gouttelettes, dans lequel le flux de gouttelettes est généré dans l'écoulement de fluide de confinement et sort d'une sortie (34) du premier dispositif d'écoulement de fluide (14, 14'); et

la génération d'un écoulement de fluide de réaction dans un deuxième dispositif d'écoulement de fluide (16, 16 ') pour réagir avec des gouttelettes dans le flux de gouttelettes, dans lequel l'écoulement de fluide de confinement est généré dans l'écoulement de fluide de réaction.

- 10. Procédé selon la revendication 9, dans lequel l'écoulement de fluide de réaction durcit les gouttelettes pour former des particules et le procédé comprend la collecte des particules à une sortie (44) du deuxième dispositif d'écoulement de fluide (16, 16').
- 11. Procédé selon la revendication 9 ou la revendication 10, dans lequel une vitesse minimum de l'écoulement de fluide de confinement est représentée par l'expression :

$$U_{min} \ge 0, 2 \left(\frac{\mu_g^2 H^3}{\rho_g^2 D_1^5}\right)^{1/2}$$

où :  $U_{\min}$  est la vitesse du débit du fluide de confinement ;

 $\mu_g$  est la viscosité du fluide de confinement ; H est la distance entre la sortie de liquide (25) du distributeur de liquide (12, 72) et la sortie (34) du premier dispositif d'écoulement de fluide (14, 14') ;

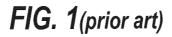
 $\rho_g$  est la densité du fluide de confinement ; et  $D_1$  est le diamètre externe du distributeur de liquide (12, 72).

12. Procédé selon l'une quelconque des revendications 9-11, dans lequel la température du deuxième écoulement de fluide est supérieure à la température du flux de gouttelettes et/ou de l'écoulement de fluide

50

de confinement.

**13.** Procédé selon l'une quelconque des revendications 9-12, dans lequel la réaction comprend le durcissement des gouttelettes dans le flux de gouttelettes.



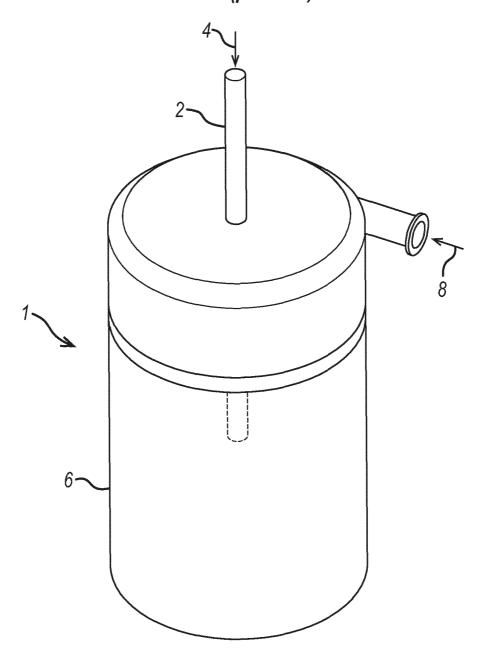
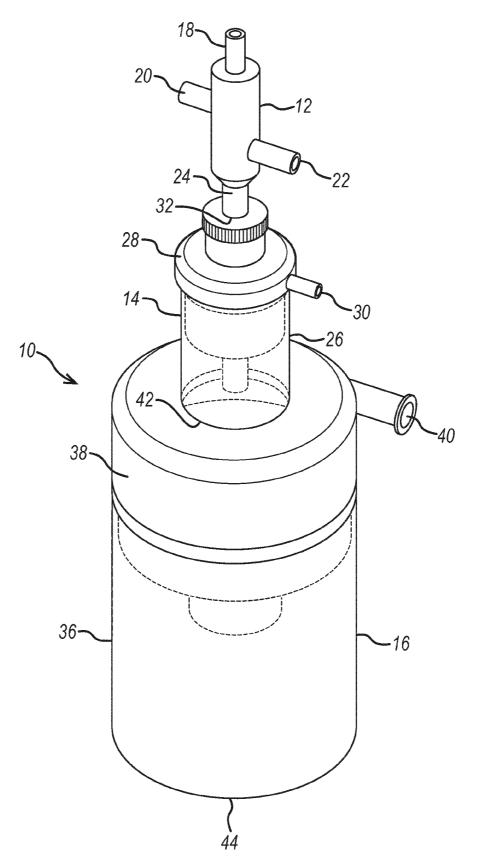


FIG. 2



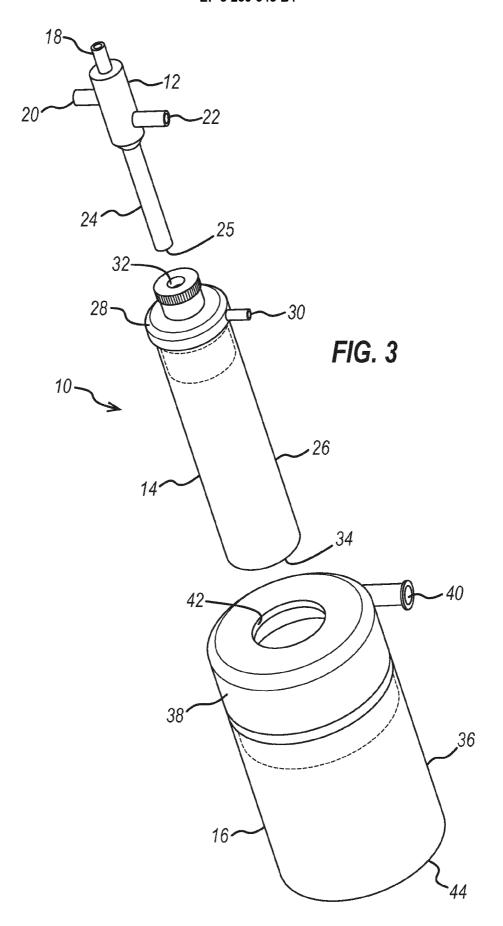


FIG. 4

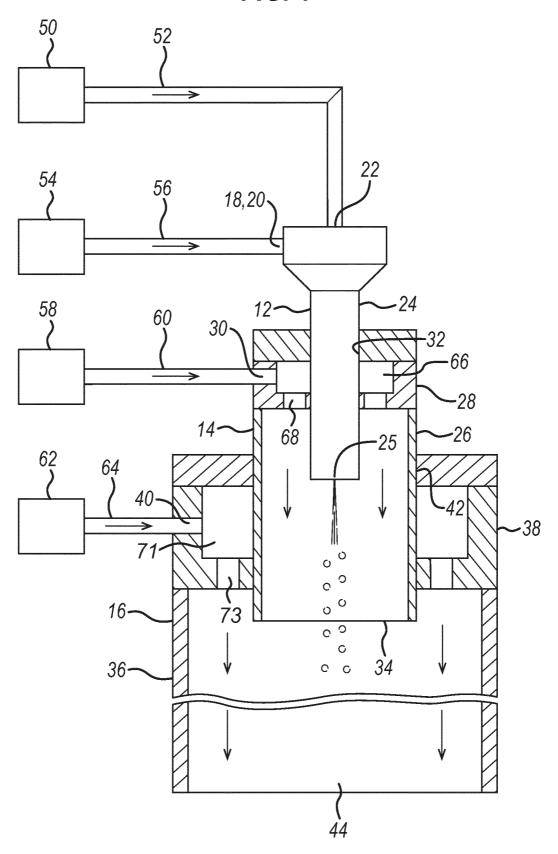
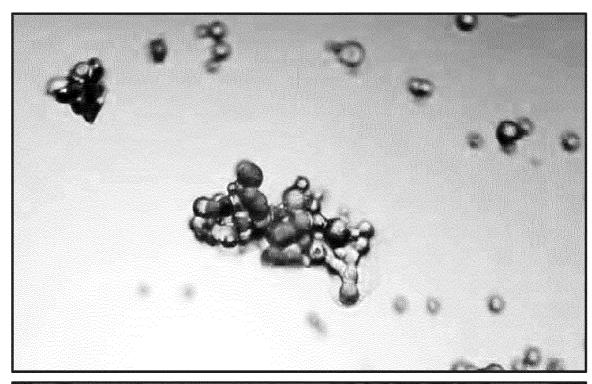
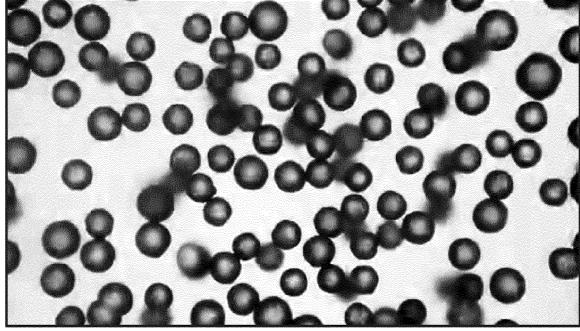


FIG. 5





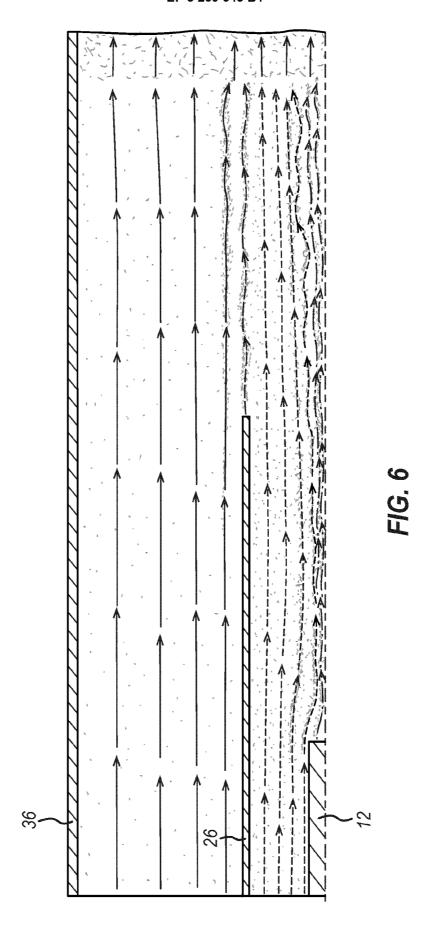


FIG. 7

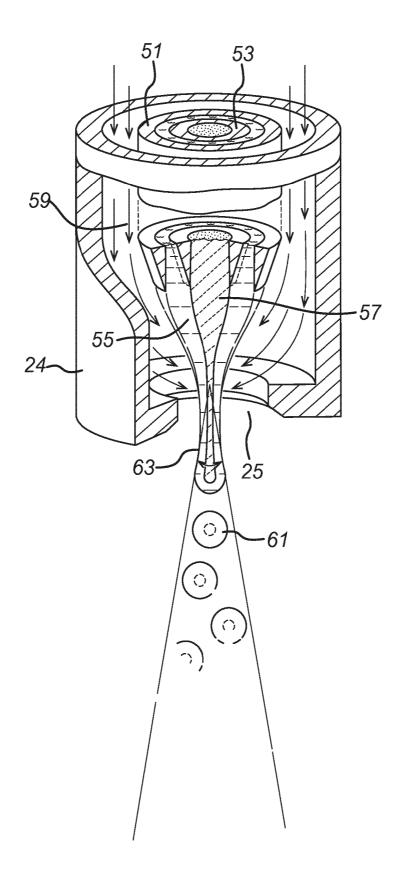
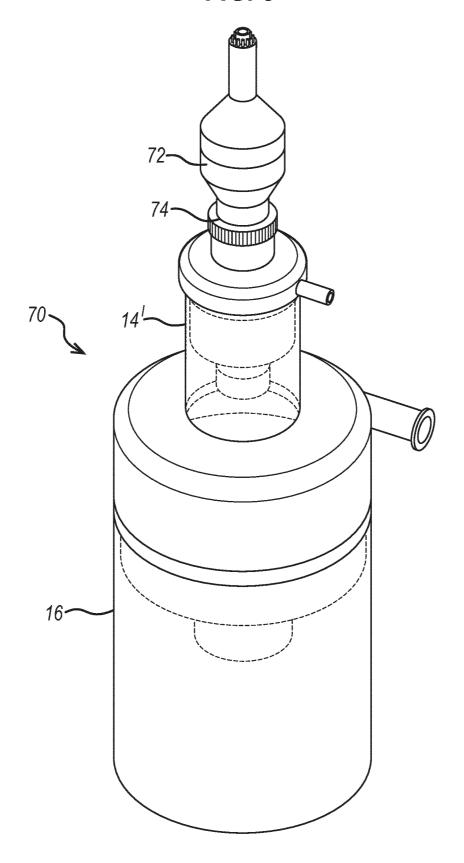


FIG. 8



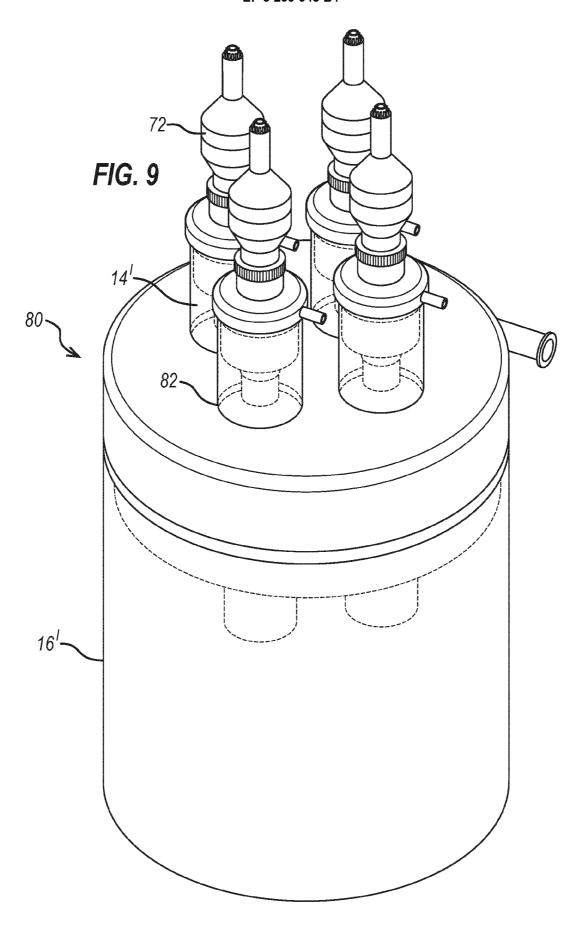
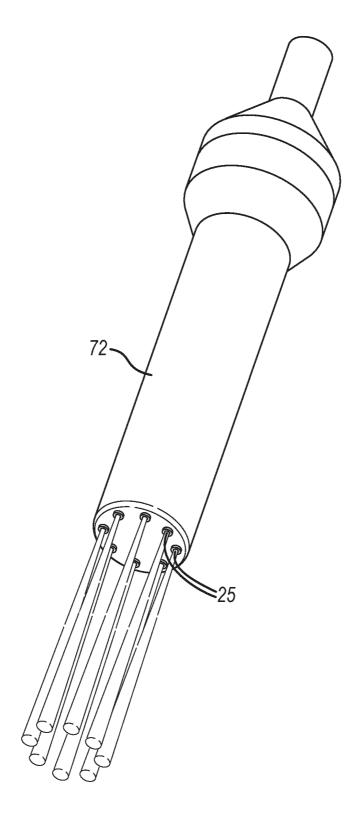


FIG. 10



# EP 3 259 543 B1

#### 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

• DE 202007008121 U [0004]