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(54) **METHOD AND DEVICE FOR PRODUCING SIMPLE AND COMPOUND MICROMETRE-SIZED EMULSIONS**

VERFAHREN UND VORRICHTUNG ZUR HERSTELLUNG EINFACHER UND ZUSAMMENGESETZTER EMULSIONEN IM MIKROMETERGRÖSSENBEREICH

PROCÉDÉ ET DISPOSITIF DE GÉNÉRATION D'ÉMULSIONS MICROMÉTRIQUES SIMPLES ET COMPOSÉES

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(56) References cited:
WO-A1-2005/044434 **WO-A2-2013/006661**
WO-A2-2013/006661 **US-A1- 2009 131 543**
US-A1- 2009 131 543

- **A. S. UTADA: "Monodisperse Double Emulsions Generated from a Microcapillary Device", SCIENCE, vol. 308, no. 5721, 22 April 2005 (2005-04-22), pages 537-541, XP055020285, ISSN: 0036-8075, DOI: 10.1126/science.1109164**
- **VLADISAVLJEVIC ET AL.: 'Industrial lab-on -a chip: Design,applications and scale-up for drug discovery and delivery.' ADVANCED DRUG DELIVERY REVIEWS vol. 65, no. 11, 2013, pages 1626 - 1663, XP028775846 DOI: 10.1016/J.ADDR.2013.07.017**
- **SHAH ET AL.: 'Designer emulsions using microfluidics.' MATERIALS TODAY vol. 11, no. 4, 15 March 2008, pages 18 - 27, XP022535514 DOI: 10.1016/S1369-7021(08)70053-1**

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Description

Object of the invention

[0001] The invention described in this specification pertains to the Scientific and Technical Area of Microfluidics. The study of microfluids is a multi-disciplinary field that includes elements from Physics, Chemistry, Engineering and Biotechnology. It conducts research on the behaviour of micro-scale fluids, wherein the motion regime is laminar, the gravity and the inertia are negligible, and the viscosity and the surface tension are dominant. This scientific and technical area comprises designing systems - devices and processes - for the controlled production of simple and compound steady jets that break up due to capillary forces, to produce simple and compound droplets which, in turn, are used for the production of micrometre- and submicrometre-sized fibres, tubes and capsules.

[0002] It is well-known that various sectors of activity in different industries are interested in the generation of simple and compound emulsions formed by micrometre-size droplets with or without a coating. For example, the food industry (encapsulation of additives), the phytosanitary industry, the cosmetics industry, the pharmaceutical industry (selective transport of active principles), the chemical industry (manufacturing of detergents), and the materials industry (manufacturing of optical devices by means of liquid crystals), amongst others. In general, the process and the device for generating emulsions of the invention described in this specification have applications in those industrial sectors wherein the production of simple and compound monodisperse, homogeneous emulsions having micrometre-sized, controllable diameters and coatings is an essential part of the process.

Prior art

[0003] In recent years, numerous studies, inventions and applications related to the microscopic monitoring of fluid streams have arisen, and particularly noteworthy amongst these studies and inventions are those that involve *free surfaces* or interfaces between two immiscible fluids in order to obtain microscopic structures (micro-droplets, micro-bubbles, micro-capsules, etc.) in a reproducible, robust manner. It is worth mentioning two peculiar phenomena/inventions which are representative of the generation of micro-jets: (i) *electrospraying,* or the production of liquid micro-jets by means of electrostatic forces, which has been known for centuries, and (ii) capillary flow focusing, which uses pressure forces (purely mechanical) and a "focusing" orifice to generate the jet. Depending on the geometry, both methods present a genuine axial symmetry (axially symmetric) in the area of the interface wherein the jet is produced, although there exist embodiments of flow-focusing devices with geometries that are practically two-dimensional (Anna et al., Appl. Phys. Lett. (2003), 82, 364-366, Gordillo et al., Phys. Fluids (2004), 16, 2828-2834).

[0004] In the case of *electrospraying,* the main disadvantages originate from (i) the inherent, inevitable dependency of the phenomenon on the electrical properties of the liquid, which greatly limits the physical-chemical parametric freedom of the method (even though some highly relevant applications have appeared in biochemistry - mass spectrometry for biological molecules -), (ii) the low productivity of the method (very low mass flow rate) and the difficulty in "scaling" or "multiplexing" it, and (iii) the mediocre robustness of the method due to its great dependency on the surface conditions and the sizes of the liquid feeding tubes.

[0005] Although the disadvantages associated with the dependency on the electrical properties of the fluid are eliminated in axially symmetric flow focusing, there are still problems related to the alignment of the feeding tubes with respect to the focusing orifices. In implementations of the 2D flow focusing type, the main problem originates from the wetting of the surfaces that confine the fluid to be dispersed.

[0006] The reason why the controlled production of micro- and submicrometric particles is one of the most active lines of research within the field of Fluid Mechanics is the large number of scientific and technological applications arising therefrom. For example, as noted in the article "Micro- and nanoparticles via capillary flows", Barrero and Loscertales, Annual Review of Fluid Mechanics (2007), 39, 89-106, the efficient absorption of new drugs by tissues and organs requires that the active product be confined in droplets with sizes substantially smaller than 10 microns. Emulsions formed by micrometre-sized droplets also have applications in many other fields, such as the food industry and materials science (manufacturing of optical devices by means of liquid crystals), amongst others. Currently, there is a significant number of processes that make it possible to obtain this type of micro-emulsions, with characteristic droplet sizes of about ten microns. However, there is only one technique that is able to efficiently reduce the size below this limit: that of simple and compound electrosprays (Loscertales, Barrero et al., Science (2002), 295, 5560). Here we present a technique that dispenses with the use of electric fields or surfactants, and which has such a simple geometry that it eliminates the focusing problems associated with devices of the three-dimensional flow focusing type, Gañán-Calvo and Gordillo, Phys. Rev. Lett. (2001), 87, 274501, or the problems associated with the wetting of the adjacent surfaces, as in the case of those techniques that use flow-focusing devices created by means of soft-lithography methods (Anna et al., Appl. Phys. Lett. (2003), 82, 364-366). In addition to having a more complex geometry, due to the fact that the stream to be dispersed must be focused through an orifice or a channel with a smaller size than that of the injection needle, these methods are not capable of systematically obtaining droplet sizes smaller than 5 microns.

[0007] In recent years, there has been increasing interest in the food, pharmaceutical and chemical industries in generating capsules that contain an active principle in the interior and which are externally coated with a flexible or rigid housing. Numerous patents have registered processes for the production of capsules or emulsions. In the case of capsules for food industry applications, some examples are patents AU754712 and EP1263451. In regards to applications in the chemical industry (primarily companies devoted to the manufacturing of detergents), EP1288287 and WO03002160. Pharmaceutical industry applications are the most widespread and have numerous registrations, amongst them WO03004003, WO0041740, US6514526, and EP1151746. In most of these examples, the capsules are generated by means of chemical substance deposition on the surface of a droplet containing an active compound or principle. The purpose of the external cover, which is usually elastic or rigid, is to protect the active principle contained inside. There are processes, initially patented at the University of Seville, which follow a different method for encapsulating liquids or generating emulsions. Both are based on causing two immiscible fluid streams to flow in a coaxial manner. It is well-known that cylindrical jets break up into droplets due to the growth of capillary instability, also known as Rayleigh instability. When this break-up takes place simultaneously in the inner and outer jets, droplets are generated which contain other smaller-sized droplets inside. If the external cover is made solid by means of any process (for example, by using a photopolymer that increases its viscosity or becomes rigid with ultraviolet light as the outer fluid), solid capsules may be generated. Emulsions may be generated in these devices by simply injecting a liquid, using any of the aforementioned processes, into a bath containing a liquid that is immiscible with the injected fluid. The first process belongs to the family of devices known as flow focusing, which is protected by patents US 6174469, US 6187214 and US 6450189. In this case, similarly to what occurs with atomisers of the flow focusing type, the two concentric fluid streams are accelerated due to the favourable pressure gradient that exists between a gas-pressurised chamber and the exterior. The diameters of the inner and outer jets decrease and, finally, thanks to a primarily capillary mechanism, the compound droplets are generated. These compound droplets may have diameters of the order of 100 microns. On the other hand, using the technology known as Y-Flow, the concentric inner and outer jets are accelerated using an electric field. The capsules generated may have nanometric sizes (the capsules produced using this process are the smallest ever generated) and are protected by patents P200100231, PCT ES02/00047 and PCT US 02/02787. However, this process has a disadvantage as compared to flow focusing devices; namely, that electric fields are necessary and the liquid flow rates are of the order of 1000 to 100 times lower than those that may be used in flow focusing technology.

[0008] Document WO2013/006661A2 discloses a device according to the preamble of claims 1 and 2. It discloses multiple emulsions, as well as techniques for formation thereof. A multiple emulsion describes larger droplets that contain one or more smaller droplets therein. In some embodiments, the larger droplet or droplets may be suspended in a carrying fluid containing the larger droplets that, in turn, contain the smaller droplets. Multiple emulsions can be formed in one step in certain embodiments, with generally precise repeatability, as can be tailored in some embodiments to include a relatively thin layer of fluid separating two other fluids.

Description of the figures

[0009] In order to supplement the description of the invention, and to contribute to a better understanding of its characteristics, the present descriptive specification includes, as an integral part thereof, two diagrams which show, for illustrative, non-limiting purposes, two prototype models for generating simple and compound emulsions (Fig. 1 and Fig. 2, respectively), as well as two sets of images that show the actual production of emulsions, both simple and compound (Fig. 3 and Fig. 4), with prototypes of both classes.

[0010] *Figure 1:* The figure consists of a diagram of a prototype device for generating simple emulsions.

[0011] The device is composed of the injection tube (1), designed to inject the liquid to be emulsified, which has an inner diameter di and is coaxially aligned with, and separated by a distance h from, the extraction tube (3), which has a length l and a square cross-section with an inner side l_o, and an outlet to the exterior, contained inside a discharge chamber (4) equipped with an orifice (5) designed for the inlet of the emulsifying liquid.

[0012] Moreover, in order to illustrate the generation of simple emulsions in the device by means of the process of the invention, the figure shows the velocity profile (6) of the liquid to be emulsified flowing inside the injection tube, and the streamlines (10) and the velocity profile (8) of the emulsifying liquid flowing in the surroundings of the inlet section of the extraction tube and downstream in the interior thereof, respectively.

[0013] *Figure 2:* The figure is a diagram of a prototype device for generating compound emulsions.

[0014] The different parts that make it up are a composite injection tube formed by an inner capillary tube (1), which has a diameter di, is designed to inject the inner liquid and is coaxially centered inside a second outer capillary tube (2), with a diameter d_o, through which the intermediate liquid is injected, and which is coaxially aligned with, and separated by a distance h from, the extraction tube (3), which has a length l and a square cross-section with an inner side l_o, and an outlet to the exterior, contained inside a discharge chamber (4) equipped with an orifice (5) designed for the inlet of the emulsifying liquid.

[0015] In addition, in order to illustrate the generation of compound emulsions in the device by means of the process of the invention, the figure shows the velocity profile of the inner liquid (6) flowing inside the inner injection tube, the velocity profile of the intermediate liquid (7) flowing inside the outer injection tube, and the streamlines (10) and the velocity profile (8) of the emulsifying liquid flowing in the surroundings of the inlet section of the extraction tube and downstream in the interior thereof, respectively.

Description of the invention

[0016] According to the invention, a device for generating simple emulsions according to claim 1 and a device for generating compound emulsions according to claim 2 are provided. Further according to the invention, corresponding processes according to claim 6 and 8 are provided.

[0017] The object of the present invention is a process for generating simple and compound emulsions, from the formation of a simple or compound, as applicable, capillary micro-jet when a liquid or two immiscible or barely miscible liquids that flow coaxially are suctioned by means of a viscous outer liquid, which is immiscible or barely miscible with the simple or compound liquid to be emulsified, and which flows at the suitable velocity, as well as the device wherein and with which to perform said process.

[0018] According to one parametric determination, the specifications whereof are the essence of the process of the invention, in the case of the generation of simple emulsions, the suctioned liquid forms a steady capillary jet with a micrometre-sized, controllable diameter, due to the action of the co-flowing outer liquid and the favourable pressure gradient that it produces. This simple jet breaks up due to capillary instability, to produce droplets of a micro- and submicrometric size, with a limited size distribution.

[0019] In the case of the generation of compound emulsions, the suction causes a compound capillary jet, due to the action of the co-flowing outer liquid and the favourable pressure gradient produced on the intermediate liquid and, similarly, of the latter on the inner liquid. The interior of this compound jet breaks up into droplets due to capillary instability, which induces break-up of the intermediate liquid, to produce the coating of the inner liquid droplets and, consequently, the formation of compound micrometre-sized droplets.

[0020] The parametric determination in the generation of compound emulsions allows for control of both the size of the inner liquid droplets and the thickness of the coating with the intermediate liquid.

[0021] The process of the invention may be applied to all those technological demands that require the generation of simple monodisperse, micrometre-sized emulsions of the phase to be dispersed, as well as to those technological demands that require the generation of compound homogeneous emulsions, the dispersed phase whereof requires micrometre-sized, controllable droplet diameters and coating thicknesses.

[0022] The object of the present invention is a process and a device for generating simple and compound micrometre-sized emulsions.

[0023] The device that generates the emulsions is formed by the following elements: a discharge chamber with dimensions of the order of one centimetre, the interior whereof contains two coaxially aligned tubes, an extraction tube, with a diameter or inner side l_o of the order of one millimetre and a length l, and a simple capillary injection tube, with an inner diameter di of the order of one millimetre, or a compound capillary injection tube, formed by two concentric capillary tubes, the exterior whereof have an inner size d_o of the order of one millimetre. The outlet section of the simple or compound capillary tube is separated from the inlet section of the extraction tube by a distance h of the order of one millimetre. The geometrical dimensions di, d_o, l_o and h of the elements of the device constitute its geometrical configuration.

[0024] The circulation of a viscous outer liquid l_e, from the discharge chamber to the exterior through the extraction tube, produces the suction of the simple liquid l_i, injected into the chamber through the capillary tube where it flows, in the surroundings of the inlet section, to form a steady capillary jet, which narrows down downstream, to reach a constant micrometre-sized diameter. This jet is formed due to the action of the co-flowing outer fluid and the favourable pressure gradient that the outer fluid exerts on the simple capillary jet. This simple jet breaks up, due to capillary instability, into droplets of the same order of magnitude as that of the jet that originates them, to produce a simple monodisperse micrometre-sized emulsion.

[0025] If a composite capillary injection tube is used, the suction produces a capillary jet composed of the intermediate liquid, which forms the outermost layer of the jet, and the inner liquid, which is at the centre of the compound jet. The compound jet is formed due to the action of the co-flowing outer fluid and the favourable pressure gradient that the outer fluid exerts on the intermediate fluid. Likewise, the action of the outer co-flow and the favourable outer pressure gradient, jointly with the capillary pressure gradients exerted by the intermediate flow, lead to the formation of an inner liquid capillary jet which, due to capillary instability, breaks up into droplets that induce break-up of the intermediate liquid, thereby originating the coating thereof and, consequently, the formation of compound droplets, to produce a compound monodisperse emulsion having controllable, micrometre-sized inner and outer diameters and coating thickness.

[0026] The viscosities of the inner, intermediate and outer liquids, and the surface tensions between the inner and outer liquids, in the case of the generation of simple emulsions, and between the outer and intermediate liquids, and the intermediate and inner liquids, in the case of the generation of compound emulsions, are essential physical properties for the production of the emulsions described in this invention and constitute what we will call the dimensional configuration thereof.

[0027] The flow rates of the inner, intermediate and outer liquids, qi, q_m and q_e, respectively, or, alternatively, the flow rates of the inner and intermediate liquids, q_i and q_m, respectively, and the manometric discharge pressure of the outer fluid Δp_e, which is related to the outer flow rate q_e according to the formula $\Delta p_e = Kq_e$, K being a constant that depends solely on the geometry of the device, are the operating control variables or parameters in the generation of emulsions produced by means of this technology and constitute what we will call the operational configuration thereof.

[0028] The process of the present invention involves the adequate selection of the aforementioned geometrical, dimensional and operating parameters or variables, i.e. the specification of the geometrical, dimensional and operational configurations.

[0029] The specification of the geometrical, dimensional and operational configurations define a parametric determination or a mode of production.

[0030] The following formulations express the physics of the phenomenon whereon the technology described above is grounded:

(i).- the characteristic Reynolds number of the outer and inner streams is lower than 1 and lower than 10, respectively; $\rho_o U_o D/\mu_o < 1$ and $\rho_i Q_i/(D\mu_o) < 10$, where U_o is the velocity of the outer fluid at the centre of the extraction tube;

(ii).- the capillary number is greater than 0.75; $\mu_o U_o/\sigma > 0.75$;

(iii).- the flow rate of the inner liquid and the velocity of the outer liquid are such that $[4Q_i/(\pi U_o)]^{1/2} < 1$ mm.

[0031] If the selected parametric determination or mode of production meets all of the aforementioned numerical ratios, the generation of simple emulsions by means of this device and this process is feasible. In this case, the scaling law that predicts the diameter of the droplets that form the emulsion is the following: $0.25[4Q_i/(\pi U_o)]^{1/2} < d < 4[4Q_i/(\pi U_o)]^{1/2}$.

[0032] The invention described in this specification has applications in those industrial sectors wherein the production of simple and compound monodisperse, homogeneous emulsions with controllable, micrometre-sized diameters and coatings is an essential part of the process.

Embodiment of the invention

[0033] *Embodiment of the invention for generating simple emulsions.*

[0034] Figure 1 presents a diagram of the device used for production of simple emulsions by means of this invention.

[0035] The prototype is composed of a discharge chamber (4) with inner dimensions of 3 cm x 3 cm x 3 cm, which contains, in the interior thereof, a glass extraction tube (3) that has a square cross-section, with an inner side l_o = 1 mm and a length l = 4 cm, and a stainless steel simple capillary injection tube (1), which has an inner diameter di = 450 μm. The spacing between both tubes is h = 1 mm. Several experiments were performed, wherein the geometrical configuration of the devices in four of the experiments is as follows: 1) l_o = 1 mm, d_i = 450 μm and h = 0.5l_o; 2).- d_i = 450 μm and h = 1.0 l_o; 3).- $d_i = l_o$ and h = 1.0 l_o; 4).- $d_i = l_o$ and h = 1.5 l_o.

[0036] The discharge chamber contains two pairs of windows, in order to allow for real-time control of the production of simple emulsions.

[0037] The outer and inner liquids used in this embodiment of the invention for generating simple emulsions are 10^{-4} m^2/s (100-cSt) liquid silicone oil and distilled water (with a viscosity of 10^{-6} m^2/s (1 cSt)), respectively, with a surface tension between them of 40 mN/m.

[0038] The flow rates of the outer and inner liquids, q_e and qi, were varied between 450 ml/h and 1400 ml/h, and 0.1 ml/h and 10 ml/h, respectively, to produce emulsions with droplets sizes ranging between 10 μm and 100 μm, and a production frequency ranging between 1000 Hz and 10000 Hz.

[0039] In the present embodiment, a simple emulsion has been produced according to the aforementioned specifications by injecting flow rates q_e = 1150 ml/h and q_i = 0.5 ml/h.

[0040] In order to evaluate the versatility of both the process and the device for generating simple emulsions, as well as the range of the parametric determination, emulsions were produced by modifying:

(i) the different variables that characterise the geometrical configuration of the elements thereof (d_o, h, l_o and l);

(ii) the viscosity of the outer and inner liquids in the ranges between 10^{-4} 10^{-4} m^2/s (100 cSt) and 10^{-3} m^2/s (1000 cSt), and between 10^{-6} m^2/s (1 cSt) and 10^{-6} m^2/s (1 cSt), respectively, and the surface tension between them in the ranges between 1 mN/m and 40 mN/m, replacing some liquids with others having different properties;

(iii) and the concrete, specific values of the operating control parameters for the production of simple emulsions, i.e. of the outer and inner liquid flow rates, in the ranges between 100 ml/h and 4000 ml/h, and 0.01 ml/h and 100 ml/h, respectively.

[0041] In all cases, a parameter window was found wherein the production of the emulsions is effective, continuous, uniform and stable.

[0042] *Embodiment of the invention for generating compound emulsions.*

[0043] Figure 2 shows a diagram of the device used for production of compound emulsions by means of the present invention.

[0044] The prototype is composed of a discharge chamber with inner dimensions of 3 cm x 3 cm x 3 cm, which contains, in the interior thereof, a glass extraction tube (4) that has a square cross-section, with an inner side l_o = 1 mm and a length l = 4 cm, and a composite injection tube, formed by a capillary tube (1) through which the inner liquid ciruclates, with a diameter di = 450 μm, contained and coaxially centered inside a second capillary tube (3), which has an inner diameter d_o = 1.20 mm and through which the intermediate liquid flows. The spacing between both tubes is h = 1 mm.

[0045] The discharge chamber contains two pairs of windows, in order to allow for real-time control of the production of simple emulsions.

[0046] The outer, intermediate and inner liquids used in this embodiment of the invention for generating compound emulsions are 10^{-3} m^2/s (1000-cSt) silicone oil, a mixture of glycerin and distilled water, with a viscosity of $4*10^{-4}$ m^2/s (400 cSt) at 25ºC, and silicone oil with a viscosity of 10^{-5} m^2/s (10 cSt), respectively, with a surface tension between both pairs of liquids (outer and intermediate liquids, and intermediate and inner liquids) of 50 mN/m.

[0047] The flow rates of the outer, intermediate and inner liquids, q_e, q_m and q_i, were varied between 100 ml/h and 200 ml/h, 0.1 ml/h and 10.0 ml/h, and 0.1 ml/h and 10 ml/h, respectively, to produce compound emulsions with droplets having inner diameters ranging between 10 μm and 100 μm, and coatings ranging between 10 μm and 40 μm, with a production frequency ranging between 100 Hz and 2000 Hz.

[0048] In the present embodiment, a compound emulsion according to the aforementioned specifications has been produced, in the case of q_e = 200 ml/h, q_m = 2 ml/h and q_i = 0.5 ml/h. Various experiments have been performed, for different inner liquid flow rates, ranging between 0.1 ml/h and 0.7 ml/h, without varying the rest of the specifications of the mode of production described above. In particular, some experiments relate to the flow rate of the outer liquid being 200 ml/h and the flow rate of the intermediate liquid is 2.00 ml/h, wherein the flow rate of the inner liquid q_i in four of the experiments is as follows: 1).- q_i = 0.10 ml/h; 2).- q_i = 0.30 ml/h; 3).- q_i = 0.50 ml/h; 4).- q_i = 0.70 ml/h.

[0049] In order to evaluate the versatility of both the process and the device for generating compound emulsions, as well as the range of the parametric determination, emulsions were produced by modifying:

(i) the different variables that characterise the geometrical configuration of the elements thereof (d_o, h, l_o and l);

(ii) the viscosity of the outer liquid, in the range between 10^{-4} m^2/s (100 cSt) and 10^{-3} m^2/s (1000 cSt), the viscosity of the intermediate liquid between $5*10^{-5}$ m^2/s (50 cSt) and 10^{-3} m^2/s (1000 cSt), the viscosity of the inner liquid between 10^{-6} m^2/s (1 cSt) and $2*10^{-5}$ m^2/s (20 cSt), and the surface tension between both liquids ranging between 20 mN/m and 50 mN/m, replacing some liquids with others having different properties;

(iii) and the concrete, specific values of the operating control parameters for the production of the compound emulsions, i.e. the outer, intermediate and inner liquid flow rates, in the ranges between 100 ml/h and 4000 ml/h, 0.01 ml/h and 10 ml/h, and 0.01 ml/h and 100 ml/h, respectively.

[0050] In all cases, a parameter window was found wherein the production of the emulsions is effective, continuous, uniform and stable.

[0051] There are numerous materials with which the different elements that compose the generator of both simple and compound emulsions may be manufactured (metal, plastic, ceramics, glass), based primarily on the specific application of the device.

[0052] Any continuous feeding methods for the outer, intermediate and inner liquids may be used (pressure reservoirs, syringe pumps, etc.).

[0053] The aforementioned embodiments of the invention describe the process and the device, or individual cell, for generating simple or compound emulsions with the production limitations involved. If an increase in production is required, the device may be multiplexed. In this case, the flow rate of the inner liquid, or the intermediate and inner liquids, as applicable, should be as homogeneous as possible across the different cells, which may require discharge through multiple capillary needles, porous media, or any other medium capable of distributing a homogeneous flow rate across different feeding points.

Claims

1. Device for generating simple emulsions, hereinafter DGSE, composed of an injection tube (1), an extraction tube (3) and a discharge chamber (4), the injection tube (3) having an inner diameter di, and being coaxially aligned with, and separated by a distance h from, the extraction tube (3), the extraction tube (3)having a length l and a circular or square cross-section with a diameter or inner side l_o, and an outlet to the exterior, the injection tube (1) and the extraction tube (13) being contained partly inside the discharge chamber (4), wherein

1a.- di ranges between 0.4mm and 1.4 mm; **characterised in that**
1b.- l_o and l range between 0.7 mm and 1.2 mm, and 20 mm and 50 mm, respectively; and

1c.- h ranges between 0.5 mm and 1.5mm.

2. Device for generating compound emulsions, hereinafter DGCE, composed of a composite injection tube, an extraction tube (3) and a discharge chamber (4), the composite injection tube being formed by an inner capillary tube (1) and a second outer capillarity tube (2), wherein the inner capillarity tube (1) has an inner diameter di, and is coaxially centered inside the second outer capillary tube (2), and wherein the second capillarity tube (2) has an inner diameter d_o, and is coaxially aligned with, and separated by a distance h from, the extraction tube (3), which has a length I and a circular or square cross-section with a diameter or inner side l_o, and an outlet to the exterior, the composite injection tube and the extraction tube (3) being contained partly inside the discharge chamber (4), **characterised in that**

2a.- di ranges between 0.3 mm and 0.7 mm;
2b.- d_o ranges between 0.7mm and 1.4 mm;
2c.- l_o and I range between 0.7 mm and 1.2 mm, and 20 mm and 50 mm, respectively; and
2d.- h ranges between 0.5 mm and 1.5 mm.

3. Device for generating simple or compound emulsions, hereinafter DGE, according to claims 1 to 2, **characterised in that** the different parts that compose it are manufactured with various materials, such as metal, plastic, ceramic, glass or others.

4. DGE according to claims 1 to 3, **characterised in that** the discharge chamber is equipped with windows that allow for real-time control of the production of emulsions, as well as for studying the physical phenomenon whereon it is grounded.

5. Multiplexed device for generating emulsions, **characterised in that** each of the individual cells that compose it has the technical characteristics described in claims 1 to 4.

6. Process for generating simple micrometre-sized emulsions, hereinafter PGSE, by means of the effect of a co-flowing emulsifying liquid with a viscosity greater than that of the liquid to be emulsified, on the interface that they form, both being immiscible or barely miscible liquids, wherein

6a.- the viscosity of the liquid to be emulsified ranges between 10^{-6} m^2/s (1 cSt) and 10^{-3} m^2/s (1000 cSt), and the viscosity of the emulsifying liquid ranges between 10^{-6} m^2/s (1 cSt) and 10^{-2} m^2/s (10000 cSt);
6b.- the flow rate of the liquid to be emulsified ranges between 0.01 ml/h and 100 ml/h, and the flow rate of the emulsifying liquid ranges between 1 ml/h and 4000 ml/h; and
6c.- the process is implemented using a device for generating simple emulsions which has the technical characteristics described in claims 1, 3 and 4; or
6d.- the process is implemented using a multiplexed device which has the technical characteristics described in claim 5, the flow rates described in step 6b being those corresponding to an individual cell of the multiplexed device.

7. PGSE according to claim 6, **characterised in that**

7a.- the viscosity of the liquid to be emulsified ranges between 10^{-6} m^2/s (1 cSt) and $5*10^{-5}$ m^2/s (50 cSt), and the viscosity of the emulsifying liquid ranges between $5*10^{-5}$ m^2/s (50 cSt) and 10^{-3} m^2/s (1000 cSt);
7b.- the flow rate of the liquid to be emulsified ranges between 0.1 ml/h and 10 ml/h, and the flow rate of the emulsifying liquid ranges between 50 ml/h and 1000 ml/h.

8. Process for generating compound emulsions with micrometre-sized diameters and coatings, hereinafter PGCE, using two co-flowing immiscible or barely miscible liquids, by means of the effect, on the interface that they form, of a third co-flowing liquid which has greater viscosity than that of the exterior of those mentioned above, both liquids being immiscible or barely miscible, wherein

8a.- the viscosity of the inner liquid (l_i) of the compound jet to be emulsified ranges between 10^{-6} m^2/s (1 cSt) and 10^{-3} m^2/s (1000 cSt), the viscosity of the outer liquid of the compound jet (l_m) to be emulsified ranges between 10^{-6} m^2/s (1 cSt) and 10^{-3} m^2/s (1000 cSt), and the viscosity of the emulsifying liquid (l_e) ranges between 10^{-6} m^2/s (1 cSt) and 10^{-3} m^2/s (1000 cSt);
8b.- the flow rate of the inner liquid of the compound jet to be emulsified ranges between 0.01 ml/h and 100 ml/h, the flow rate of the outer liquid of the compound jet to be emulsified ranges between 0.001 ml/h and 1000 ml/h, and the flow rate of the emulsifying liquid ranges between 1 ml/h and 4000 ml/h; and
8c.- the process is implemented using a device for generating compound emulsions which has the technical characteristics described in claims 2, 3, and 4; or
8d.- the process is implemented using a multiplexed device which has the technical characteristics described in claim 5, the flow rates described in step 8b being those corresponding to an individual cell of the multiplexed device.

9. PGCE according to claim 8, **characterised in that**

9a.- the viscosity of the inner liquid l_i of the compound jet to be emulsified ranges between 10^{-6} m^2/s (1 cSt) and 10^{-5} m^2/s (10 cSt), the viscosity of the outer liquid l_m of the compound jet to be emulsified ranges between 10^{-5} m^2/s (10 cSt) and 10^{-4} m^2/s (100 cSt), and the viscosity of the emulsifying liquid l_e ranges between 10^{-4} m^2/s (100 cSt) and 10^{-3} m^2/s (1000 cSt);
9b.- the flow rate of the inner liquid l_i of the compound jet to be emulsified ranges between 0.1 ml/h and 10 ml/h, the flow rate of the outer liquid l_m of the compound jet to be emulsified ranges between 0.01 ml/h and 100 ml/h, and the flow rate of the emulsifying liquid l_e ranges between 50 ml/h and 1000 ml/h.

10. PGCE according to claim 8, **characterised in that**

10a.- the viscosity of the inner liquid l_i of the compound jet to be emulsified ranges between 10^{-6} m^2/s (1 cSt) and 10^{-5} m^2/s (10 cSt), the viscosity of the outer liquid l_m of the compound jet to be emulsified ranges between 10^{-4} m^2/s (100 cSt) and $4{*}10^{-4}$ m^2/s (400 cSt), and the viscosity of the emulsifying liquid $l_e$ranges between $4{*}10^{-4}$ m^2/s (400 cSt) and 10^{-3} m^2/s (1000 cSt);
10b.- the flow rate of the inner liquid l_i of the compound jet to be emulsified ranges between 0.1 ml/h and 10 ml/h, the flow rate of the outer liquid l_m of the compound jet to be emulsified ranges between 0.01 ml/h and 100 ml/h, and the flow rate of the emulsifying liquid l_e ranges between 50 ml/h and 1000 ml/h.

11. PGCE according to claim 8, **characterised in that**

11a.- the viscosity of the inner liquid l_i of the compound jet to be emulsified ranges between 10^{-6} m^2/s (1 cSt) and 10^{-5} m^2/s (10 cSt), the viscosity of the outer liquid l_m of the compound jet to be emulsified ranges between $4{*}10^{-4}$ m^2/s (400 cSt) and $7{*}10^{-4}$ m^2/s (700 cSt), and the viscosity of the emulsifying liquid l_e ranges between $7{*}10^{-4}$ m^2/s (700 cSt) and 10^{-3} m^2/s (1000 cSt);
11b.- the flow rate of the inner liquid l_i of the compound jet to be emulsified ranges between 0.1 ml/h and 10 ml/h, the flow rate of the outer liquid l_m of the compound jet to be emulsified ranges between 0.01 ml/h and 100 ml/h, and the flow rate of the emulsifying liquid l_e ranges between 50 ml/h and 1000 ml/h.

Patentansprüche

1. Vorrichtung zum Erzeugen von einfachen Emulsionen, im Folgenden DGSE, bestehend aus einem Injektionsschlauch (1), einem Extraktionsschlauch (3) und einer Ablasskammer (4), wobei der Injektionsschlauch (3) einen Innendurchmesser di aufweist und koaxial mit dem Extraktionsschlauch (3) ausgerichtet und von diesem in einem Abstand h getrennt ist, wobei der Extraktionsschlauch (3) eine Länge I und einen kreisförmigen oder quadratischen Querschnitt mit einem Durchmesser oder einer Innenseite l_o aufweist, und einen Auslass zur Außenseite, wobei der Injektionsschlauch (1) und der Extraktionsschlauch (13) teilweise in der Ablasskammer (4) enthalten sind,
wobei

1 a.- d_i in einem Bereich zwischen 0,4 mm und 1,4 mm liegt; **dadurch gekennzeichnet, dass**
1 b.- l_o und I in einem Bereich zwischen 0,7 mm und 1,2 mm bzw. 20 mm und 50 mm liegen; und
1 c.- h in einem Bereich zwischen 0,5 mm und 1,5 mm liegt.

2. Vorrichtung zum Erzeugen von Verbundemulsionen, im Folgenden DGCE, bestehend aus einem Verbundinjektionsschlauch, einem Extraktionsschlauch (3) und einer Ablasskammer (4), wobei der Verbundinjektionsschlauch aus einem inneren Kapillarschlauch (1) und einem zweiten äußeren Kapillarschlauch (2) gebildet ist, wobei der innere Kapillarschlauch (1) einen Innendurchmesser di aufweist und koaxial in dem zweiten äußeren Kapillarschlauch (2) zentriert ist und wobei der zweite Kapillarschlauch (2) einen Innendurchmesser do aufweist und koaxial mit dem Extraktionsschlauch (3) ausgerichtet ist und von diesem in einem Abstand h getrennt ist, der eine Länge I und einen kreisförmigen oder quadratischen Querschnitt mit einem Durchmesser oder einer Innenseite l_o aufweist, und einen Auslass zur Außenseite, wobei der Verbundinjektionsschlauch und der Extraktionsschlauch (3) teilweise in der Ablasskammer (4) enthalten sind, **dadurch gekennzeichnet, dass**

2a.- d_i in einem Bereich zwischen 0,3 mm und 0,7 mm liegt;
2b.- d_o in einem Bereich zwischen 0,7 mm und 1,4 mm liegt;
2c.- l_o und I in einem Bereich zwischen 0,7 mm und 1,2 mm bzw. 20 mm und 50 mm liegen; und
2d.- h in einem Bereich zwischen 0,5 mm und 1,5 mm liegt.

3. Vorrichtung zum Erzeugen von einfachen oder Verbundemulsionen, im Folgenden DGE, nach einem der Ansprüche 1 bis 2, **dadurch gekennzeichnet, dass** die unterschiedlichen Teile, aus denen diese bestehen, hergestellt werden aus verschiedenen Materialien, wie Metall, Kunststoff, Keramik, Glas oder anderen.

4. DGE nach einem der Ansprüche 1 bis 3, **dadurch gekennzeichnet, dass** die Ablasskammer mit Fenstern ausgestattet ist, die eine Echtzeitkontrolle der Emulsionsherstellung ermöglichen, sowie das Studium des physikalischen Phänomens, auf dem dieses gründet.

5. Multiplexvorrichtung zum Erzeugen von Emulsionen, **dadurch gekennzeichnet, dass** jede der einzelnen Zellen, aus denen diese besteht, die technischen Eigenschaften aufweist, die in den Ansprüchen 1 bis 4 beschrieben sind.

6. Verfahren zum Erzeugen von einfachen mikrometergroßen Emulsionen, im Folgenden PGSE, mittels der Wirkung einer mitfließenden emulgierenden Flüssigkeit mit einer Viskosität, die größer als die der zu emulgierenden Flüssigkeit ist, auf die Grenzfläche, die sie bilden, wobei beide nicht mischbare oder kaum mischbare Flüssigkeiten sind, wobei

6a.- die Viskosität der zu emulgierenden Flüssigkeit in einem Bereich zwischen 10^{-6} m^2/s (1 cSt) und 10^{-3} m^2/s (1000 cSt) liegt und die Viskosität der emulgierenden Flüssigkeit in einem Bereich zwischen $10^{-2}m^2/s$ (1 cSt) und $10^{-2}m^2/s$ (10000 cSt) liegt;
6b.- die Fließgeschwindigkeit der zu emulgierenden Flüssigkeit zwischen 0,01 ml/h und 100 ml/h liegt und die Fließgeschwindigkeit der emulgierenden Flüssigkeit zwischen 1 ml/h und 4000 ml/h liegt; und
6c.- das Verfahren unter Verwendung einer Vorrichtung zum Erzeugen einfacher Emulsionen implementiert wird, welche die in den Ansprüchen 1, 3 und 4 beschriebenen technischen Eigenschaften aufweist; oder
6d.- der Prozess unter Verwendung einer Multiplexvorrichtung implementiert wird, welche die in Anspruch 5 beschriebenen technischen Eigenschaften aufweist, wobei die in Schritt 6b beschriebenen Fließgeschwindigkeiten diejenigen sind, die einer einzelnen Zelle der Multiplexvorrichtung entsprechen.

7. PGSE nach Anspruch 6, **dadurch gekennzeichnet, dass**

7a.- die Viskosität der zu emulgierenden Flüssigkeit in einem Bereich zwischen 10^{-6} m^2/s (1 cSt) und $5*10^{-5}$ m^2/s (50 cSt) liegt und die Viskosität der emulgierenden Flüssigkeit in einem Bereich zwischen $5*10^{-5}m^2/s$ (50 cSt) und $10^{-3}m^2/s$ (1000 cSt) liegt;
7b.- die Fließgeschwindigkeit der zu emulgierenden Flüssigkeit zwischen 0,1 ml/h und 10 ml/h liegt und die Fließgeschwindigkeit der emulgierenden Flüssigkeit zwischen 50 ml/h und 1000 ml/h liegt.

8. Verfahren zum Erzeugen von Verbundemulsionen mit mikrometergroßen Durchmessern und Beschichtungen, im Folgenden PGCE, unter Verwendung von zwei zusammenfließenden nicht mischbaren oder kaum mischbaren Flüssigkeiten durch die Wirkung einer dritten zusammenfließenden Flüssigkeit auf der Grenzfläche, die sie bilden, die eine höhere Viskosität als die der Außenseite der vorgenannten aufweist, wobei beide Flüssigkeiten nicht mischbar oder kaum mischbar sind, wobei

8a.- die Viskosität der inneren Flüssigkeit (li) des zu emulgierenden Verbundstrahls in einem Bereich zwischen 10^{-6} m^2/s (1 cSt) und 10^{-3} m^2/s (1000 cSt) liegt, die Viskosität des zu emulgierenden Verbundstrahls (l_m) in einem Bereich zwischen 10^{-6} m^2/s (1 cSt) und 10^{-3} m^2/s (1000 cSt) liegt und die Viskosität der emulgierenden Flüssigkeit (l_e) in einem Bereich zwischen $10^{-6}m^2/s$ (1 cSt) und $10^{-3}m^2/s$ (1000 cSt) liegt;
8b.- die Fließgeschwindigkeit der inneren Flüssigkeit des zu emulgierenden Verbundstrahls in einem Bereich zwischen 0,01 ml/h und 100 ml/h liegt, die Fließgeschwindigkeit der äußeren Flüssigkeit des zu emulgierenden Verbundstrahls zwischen 0,001 ml/h und 1000 ml/h liegt und die Fließgeschwindigkeit der emulgierenden Flüssigkeit zwischen 1 ml/h und 4000 ml/h liegt; und
8c.- das Verfahren unter Verwendung einer Vorrichtung zum Erzeugen von Verbundemulsionen implementiert wird, welche die in den Ansprüchen 2, 3 und 4 beschriebenen technischen Eigenschaften aufweist;
oder
8d.- der Prozess unter Verwendung einer Multiplexvorrichtung implementiert wird, welche die in Anspruch 5 beschriebenen technischen Eigenschaften aufweist, wobei die in Schritt 8b beschriebenen Fließgeschwindigkeiten diejenigen sind, die einer einzelnen Zelle der Multiplexvorrichtung entsprechen.

9. PGCE nach Anspruch 8, **dadurch gekennzeichnet, dass**

9a.- die Viskosität der inneren Flüssigkeit l_i des zu emulgierenden Verbundstrahls in einem Bereich zwischen 10^{-6} m^2/s (1 cSt) und 10^{-5} m^2/s (10 cSt) liegt, die Viskosität der äußeren Flüssigkeit l_m des zu emulgierenden Verbundstrahls in einem Bereich zwischen 10^{-5} m^2/s (10 cSt) und 10^{-4} m^2/s (100 cSt) liegt und die Viskosität der emulgierenden Flüssigkeit l_e in einem Bereich zwischen $10^{-4}m^2/s$ (100 cSt) und $10^{-3}m^2/s$ (1000 cSt) liegt;

9b.- die Fließgeschwindigkeit der inneren Flüssigkeit l_i des zu emulgierenden Verbundstrahls in einem Bereich zwischen 0,1 ml/h und 10 ml/h liegt, die Fließgeschwindigkeit der äußeren Flüssigkeit l_m des zu emulgierenden Verbundstrahls zwischen 0,01 ml/h und 100 ml/h liegt und die Fließgeschwindigkeit der emulgierenden Flüssigkeit l_e zwischen 50 ml/h und 1000 ml/h liegt.

10. PGCE nach Anspruch 8, **dadurch gekennzeichnet, dass**

10a.- die Viskosität der inneren Flüssigkeit l_i des zu emulgierenden Verbundstrahls in einem Bereich zwischen 10^{-6} m^2/s (1 cSt) und 10^{-5} m^2/s (10 cSt) liegt, die Viskosität der äußeren Flüssigkeit l_m des zu emulgierenden Verbundstrahls in einem Bereich zwischen 10^{-4} m^2/s (100 cSt) und $4*10^{-4}$ m^2/s (400 cSt) liegt und die Viskosität der emulgierenden Flüssigkeit l_e in einem Bereich zwischen $4*10^{-4} m^2/s$ (400 cSt) und $10^{-3} m^2/s$ (1000 cSt) liegt;
10b.- die Fließgeschwindigkeit der inneren Flüssigkeit l_i des zu emulgierenden Verbundstrahls in einem Bereich zwischen 0,1 ml/h und 10 ml/h liegt, die Fließgeschwindigkeit der äußeren Flüssigkeit l_m des zu emulgierenden Verbundstrahls zwischen 0,01 ml/h und 100 ml/h liegt und die Fließgeschwindigkeit der emulgierenden Flüssigkeit l_e zwischen 50 ml/h und 1000 ml/h liegt.

11. PGCE nach Anspruch 8, **dadurch gekennzeichnet, dass**

11a. - die Viskosität der inneren Flüssigkeit l_i des zu emulgierenden Verbundstrahls in einem Bereich zwischen 10^{-6} m^2/s (1 cSt) und 10^{-5} m^2/s (10 cSt) liegt, die Viskosität der äußeren Flüssigkeit l_m des zu emulgierenden Verbundstrahls in einem Bereich zwischen $4*10^{-4}$ m^2/s (400 cSt) und $7*10^{-4}$ m^2/s (700 cSt) liegt und die Viskosität der emulgierenden Flüssigkeit l_e in einem Bereich zwischen $7*10^{-4}$ m^2/s (700 cSt) und $10^{-3} m^2/s$ (1000 cSt) liegt;
11b. - die Fließgeschwindigkeit der inneren Flüssigkeit l_i des zu emulgierenden Verbundstrahls in einem Bereich zwischen 0,1 ml/h und 10 ml/h liegt, die Fließgeschwindigkeit der äußeren Flüssigkeit l_m des zu emulgierenden Verbundstrahls zwischen 0,01 ml/h und 100 ml/h liegt und die Fließgeschwindigkeit der emulgierenden Flüssigkeit l_e zwischen 50 ml/h und 1000 ml/h liegt.

Revendications

1. Dispositif de génération d'émulsions simples, ci-après DGES, composé d'un tube d'injection (1), d'un tube d'extraction (3) et d'une chambre de décharge (4), le tube d'injection (3) ayant un diamètre interne d_i, et étant coaxialement aligné avec, et séparé d'une distance h du, tube d'extraction (3), le tube d'extraction
(3) ayant une longueur l et une section transversale circulaire ou carrée avec un diamètre ou
côté interne l_o, et une sortie vers l'extérieur, le tube d'injection (1) et le tube d'extraction (13) étant contenus en partie à l'intérieur de la chambre de décharge (4),
dans lequel

1 a. - d_i est compris entre 0,4 mm et 1,4 mm ;
caractérisé en ce que
1 b.- l_o et l sont compris entre 0,7 mm et 1,2 mm, et 20 mm et 50 mm, respectivement ; et
1 c.- h est comprise entre 0,5 mm et 1,5 mm.

2. Dispositif de génération d'émulsions composées, ci-après DGEC, composé d'un tube d'injection composite, d'un tube d'extraction (3) et d'une chambre de décharge (4), le tube d'injection composite étant constitué d'un tube capillaire interne (1) et d'un deuxième tube de capillarité externe (2), dans lequel le tube de capillarité interne (1) a un diamètre interne d_i, et est coaxialement centré à l'intérieur du deuxième tube capillaire externe (2), et dans lequel le deuxième tube de capillarité (2) a un diamètre interne d_o, et est coaxialement aligné avec, et séparé d'une distance h du, tube d'extraction (3), qui a une longueur l et une section transversale circulaire ou carrée avec un diamètre ou un côté interne l_o, et une sortie vers l'extérieur, le tube d'injection composite et le tube d'extraction (3) étant contenus en partie à l'intérieur de la chambre de décharge (4), **caractérisé en ce que**

2a. - d_i est compris entre 0,3 mm et 0,7 mm ;
2b.- d_o est compris entre 0,7 mm et 1,4 mm ;
2c.- l_o et l sont compris entre 0,7 mm et 1,2 mm, et 20 mm et 50 mm, respectivement ; et
2d.- h est comprise entre 0,5 mm et 1,5 mm.

3. Dispositif de génération d'émulsions simples ou composées, ci-après DGE, selon les revendications 1 à 2, **caractérisé en ce que** les différentes pièces qui le composent sont fabriquées avec différents matériaux, comme le métal, le plastique, le céramique, le verre ou autres.

4. DGE selon les revendications 1 à 3, **caractérisé en ce que** la chambre de décharge est équipée de fenêtres qui permettent un contrôle en temps réel de la production d'émulsions, ainsi que l'étude du phénomène physique sur lequel elle est fondée.

5. Dispositif multiplexé de génération d'émulsions, **caractérisé en ce que** chacune des cellules individuelles qui le composent a les caractéristiques techniques décrites dans les revendications 1 à 4.

6. Procédé de génération d'émulsions simples de taille micrométrique, ci-après PGES, au moyen de l'effet d'un liquide émulsifiant en co-écoulement avec une viscosité supérieure à celle du liquide à émulsionner, sur l'interface qu'ils forment, tous deux étant des liquides non miscibles ou à peine miscibles, dans lequel

6a.- la viscosité du liquide à émulsionner est comprise entre 10^{-6} m^2/s (1 cSt) et 10^{-3} m^2/s (1 000 cSt), et la viscosité du liquide émulsifiant est comprise entre $10^{-2} m^2/s$ (1 cSt) et $10^{-2} m^2/s$ (10 000 cSt) ;
6b.- le débit du liquide à émulsionner est compris entre 0,01 ml/h et 100 ml/h, et le débit du liquide émulsifiant est compris entre 1 ml/h et 4 000 ml/h ; et
6c.- le procédé est mis en œuvre en utilisant un dispositif de génération d'émulsions simples qui a les caractéristiques techniques décrites dans les revendications 1, 3 et 4 ; ou
6d.- le procédé est mis en œuvre en utilisant un dispositif multiplexé qui a les caractéristiques techniques décrites dans la revendication 5, les débits décrits à l'étape 6b étant ceux correspondant à une cellule individuelle du dispositif multiplexé.

7. PGES selon la revendication 6, **caractérisé en ce que**

7a.- la viscosité du liquide à émulsionner est comprise entre 10^{-6} m^2/s (1 cSt) et $5*10^{-5}$ m^2/s (50 cSt), et la viscosité du liquide émulsifiant est comprise entre $5*10^{-5} m^2/s$ (50 cSt) et $10^{-3} m^2/s$ (1 000 cSt) ;
7b.- le débit du liquide à émulsionner est compris entre 0,1 ml/h et 10 ml/h, et le débit du liquide émulsifiant est compris entre 50 ml/h et 1 000 ml/h.

8. Procédé de génération d'émulsions composées avec des diamètres et des revêtements micrométriques, ci-après PGEC, utilisant deux liquides en co-écoulement non miscibles ou à peine miscibles, au moyen de l'effet, sur l'interface qu'ils forment, d'un troisième liquide en co-écoulement qui a une viscosité supérieure à celle de l'extérieur de ceux mentionnés ci-dessus, les deux liquides étant non miscibles ou à peine miscibles, dans lequel

8a.- la viscosité du liquide interne (l_i) du jet composé à émulsionner est comprise entre 10^{-6} m^2/s (1 cSt) et 10^{-3} m^2/s (1 000 cSt), la viscosité du liquide externe du jet composé (l_m) à émulsionner est comprise entre 10^{-6} m^2/s (1 cSt) et 10^{-3} m^2/s (1 000 cSt) et la viscosité du liquide émulsifiant (l_e) est comprise entre 10^{-6} m^2/s (1 cSt) et 10^{-3} m^2/s (1 000 cSt) ;
8b.- le débit du liquide interne du jet composé à émulsionner est compris entre 0,01 ml/h et 100 ml/h, le débit du liquide externe du jet composé à émulsionner est compris entre 0,001 ml/h et 1 000 ml/h, et le débit du liquide émulsifiant est compris entre 1 ml/h et 4 000 ml/h ; et
8c.- le procédé est mis en œuvre en utilisant un dispositif de génération d'émulsions composées qui a les caractéristiques techniques décrites dans les revendications 2, 3 et 4 ;
ou
8d.- le procédé est mis en œuvre en utilisant un dispositif multiplexé qui a les caractéristiques techniques décrites dans la revendication 5, les débits décrits à l'étape 8b étant ceux correspondant à une cellule individuelle du dispositif multiplexé.

9. PGEC selon la revendication 8, **caractérisé en ce que**

9a.- la viscosité du liquide interne l_i du jet composé à émulsionner est compris entre 10^{-6} m^2/s (1 cSt) et 10^{-5} m^2/s (10 cSt), la viscosité du liquide externe l_m du jet composé à émulsionner est compris entre 10^{-5} m^2/s (10 cSt) et 10^{-4} m^2/s (100 cSt), et la viscosité du liquide émulsifiant l_e est comprise entre $10^{-4} m^2/s$ (100 cSt) et $10^{-3} m^2/s$ (1 000 cSt) ;
9b.- le débit du liquide interne l_i du jet composé à émulsionner est compris entre 0,1 ml/h et 10 ml/h, le débit du liquide externe l_m du jet composé à émulsionner est compris entre 0,01 ml/h et 100 ml/h, et le débit du liquide émulsifiant l_e est compris entre 50 ml/h et 1 000 ml/h.

10. PGEC selon la revendication 8, **caractérisé en ce que**

10a.- la viscosité du liquide interne l_i du jet composé à émulsionner est compris entre $10^{-6} m^2/s$ (1 cSt) et $10^{-5} m^2/s$ (10 cSt), la viscosité du liquide externe l_m du jet composé à émulsionner est compris entre 10^{-4} m^2/s (100 cSt) et $4*10^{-4}$ m^2/s (400 cSt), et la viscosité du liquide émulsifiant l_e est comprise entre $4*10^{-4}$ m^2/s (400 cSt) et 10^{-3} m^2/s (1 000 cSt) ;
10b.- le débit du liquide interne l_i du jet composé à émulsionner est compris entre 0,1 ml/h et 10 ml/h, le débit du liquide externe l_m du jet com-

posé à émulsionner est compris entre 0,01 ml/h et 100 ml/h, et le débit du liquide émulsifiant l_e est compris entre 50 ml/h et 1 000 ml/h.

11. PGEC selon la revendication 8, **caractérisé en ce que**

11a. - la viscosité du liquide interne l_i du jet composé à émulsionner est compris entre 10^{-6} m^2/s (1 cSt) et 10^{-5} m^2/s (10 cSt), la viscosité du liquide externe l_m du jet composé à émulsionner est compris entre $4*10^{-4}$ m^2/s (400 cSt) et $7*10^{-4}$ m^2/s (700 cSt), et la viscosité du liquide émulsifiant l_e est comprise entre $7*10^{-4}$ m^2/s (700 cSt) et 10^{-3} m^2/s (1 000 cSt) ;
11b. - le débit du liquide interne l_i du jet composé à émulsionner est compris entre 0,1 ml/h et 10 ml/h, le débit du liquide externe l_m du jet composé à émulsionner est compris entre 0,01 ml/h et 100 ml/h, et le débit du liquide émulsifiant l_e est compris entre 50 ml/h et 1 000 ml/h.

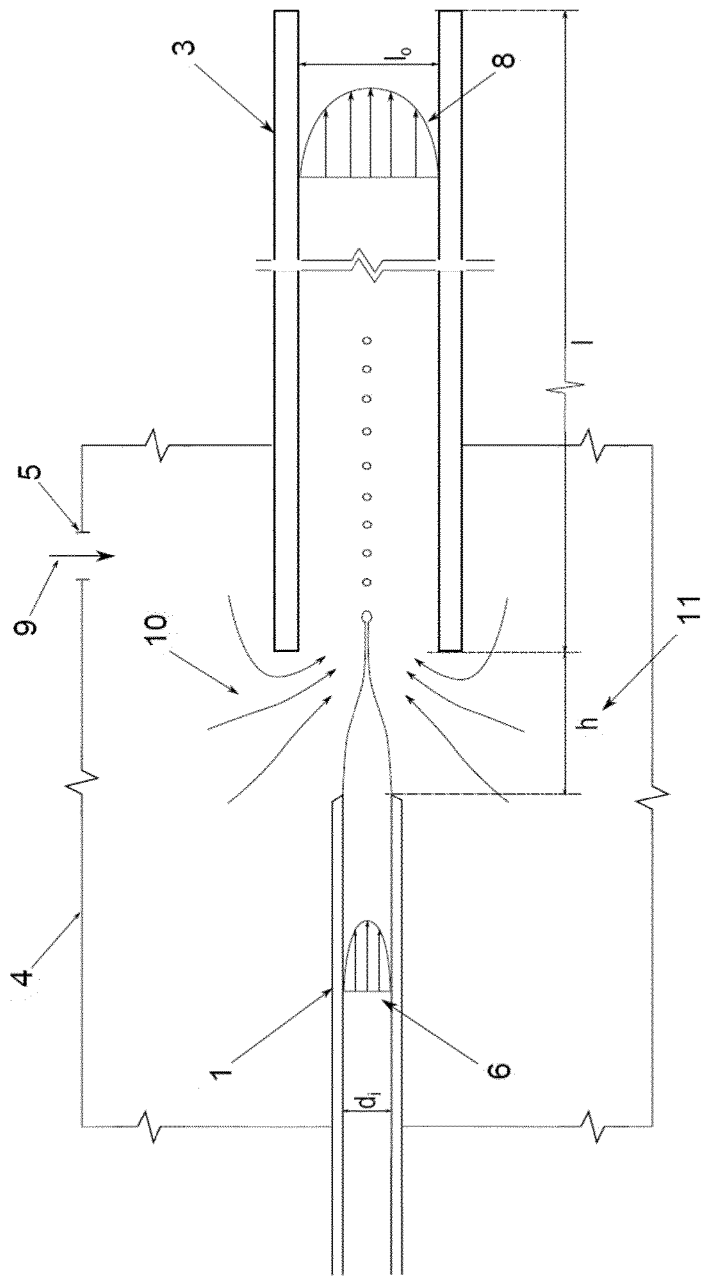

Figure 1

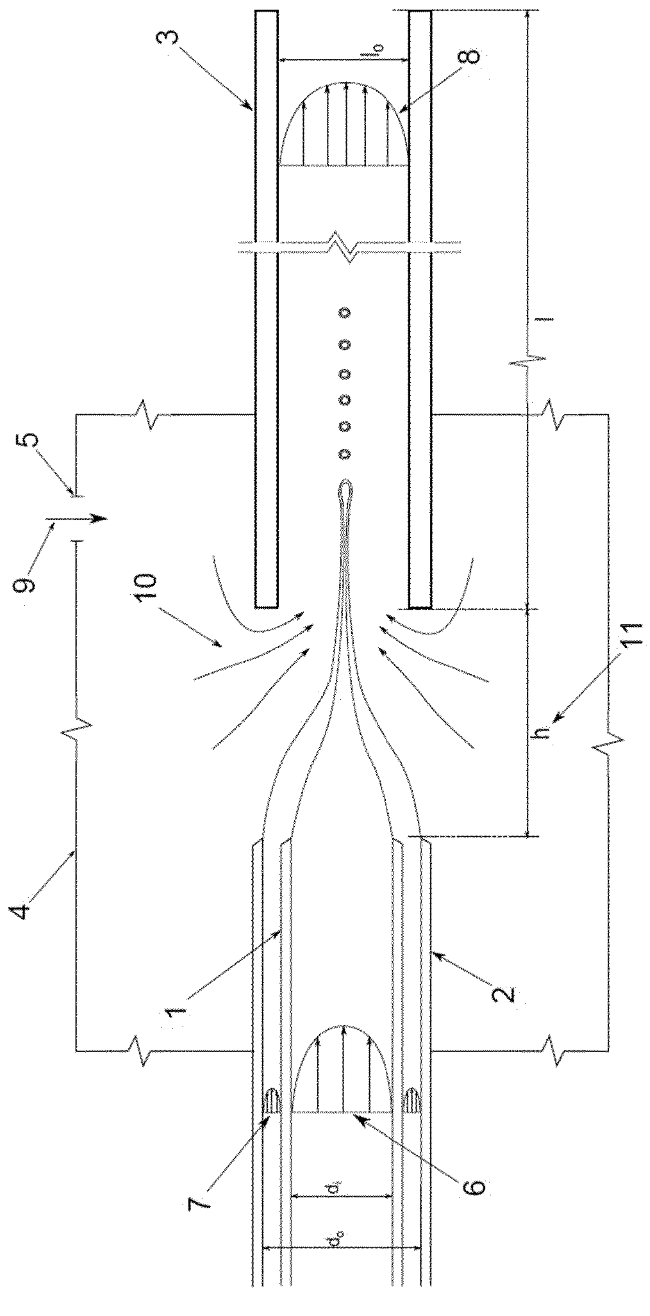

Figure 2

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- AU 754712 **[0007]**
- EP 1263451 A **[0007]**
- EP 1288287 A **[0007]**
- WO 03002160 A **[0007]**
- WO 03004003 A **[0007]**
- WO 0041740 A **[0007]**
- US 6514526 B **[0007]**
- EP 1151746 A **[0007]**
- US 6174469 B **[0007]**
- US 6187214 B **[0007]**
- US 6450189 B **[0007]**
- ES P200100231 **[0007]**
- ES 0200047 W **[0007]**
- US 0202787 W **[0007]**
- WO 2013006661 A2 **[0008]**

Non-patent literature cited in the description

- **ANNA et al.** *Appl. Phys. Lett.,* 2003, vol. 82, 364-366 **[0003] [0006]**
- **GORDILLO et al.** *Phys. Fluids,* 2004, vol. 16, 2828-2834 **[0003]**
- **BARRERO ; LOSCERTALES.** Micro- and nanoparticles via capillary flows. *Annual Review of Fluid Mechanics,* 2007, vol. 39, 89-106 **[0006]**
- **LOSCERTALES ; BARRERO et al.** *Science,* 2002, vol. 295, 5560 **[0006]**
- **CALVO ; GORDILLO.** *Phys. Rev. Lett.,* 2001, vol. 87, 274501 **[0006]**