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SHREDDING METHOD AND DEVICE FOR OBTAINING NANOCELLULOSE (54)

A shredding method and a device for obtaining (57)nanocellulose are proposed. By means of the combination of several steps through which a solution (1) passes, said solution consisting of a mixture of bleached cellulose diluted in water in a proportion of between 1% and 6% of bleached cellulose which is subjected to a pressure of between 250 and 600 bar, in order to accelerate the solution (1) so as to reach a speed of about 50 m/s-250 m/s in a nozzle (3) through the passage (4) thereof, the solution (1) subsequently being expanded and decompressed, causing it to collide against same, and thereby obtaining the nanocellulose. The device is made up of the combination of a compression chamber (2), including a compressor (12), and a nozzle (3) having a passage (4), wherein on the opposite side of the nozzle (3), there is arranged another receiving chamber (7) which has a dead center (8) and a decompressor (13).

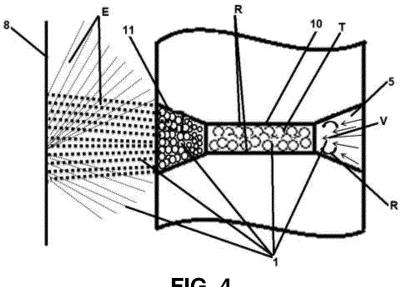


FIG. 4

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Technical Field

[0001] The object of the present invention belongs to the sector for obtaining nanocellulose. It relates to a method for obtaining nanocellulose by means of shredding through the combination of pressure, friction, turbulence, acceleration, speed, decompression, expansion, and collision of cellulose. Although this is performed by means of a mechanical method, it still constitutes an efficient alternative over what is known up until now for obtaining nanocellulose, proposing a method which is based on a highly homogenous cellulose solution in proportions of between 1% and 6% with the rest being water, said solution being subjected to a high pressure to then be passed through a characteristic nozzle in which the solution is subjected to great acceleration in order to reach a high speed (turbulent regimen), which in turn causes a strong friction and turbulence in the outlet of the nozzle, causing the corresponding expansion and decompression as well as the collision of the high-speed fluid such that nanocellulose is obtained with said method. Nanocellulose whose fibers are in turn with homogenous structure and elongated fiber, nano size that, once separated from the water in which it was diluted, by centrifugation process, the nanocellulose is obtained to be used as desired.

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Background of the Invention

[0002] Two manufacturers of nanocellulose which also produce said nanocellulose mechanically are known today and they are:

A) The company, MASUKO SANGYO Co. Ltd., which uses a grinding pulverization technique, i.e., friction grinding by means of a mechanical process involving grinding wheels which produces a high friction on cellulose fibers in order to reduce their size, leading to the gumming of the material during the exit thereof and difficulties in repeating the results obtained. Said process makes it necessary to previously dilute the solution in excess so as to reduce its size, in addition to requiring a long operation time, making it inefficient, as the fibers must be subjected to many passes until obtaining nanocellulose, with a high energy cost per kilogram that this entails in obtaining nanocellulose. Though this process is costly and slow, there are several patents relating to same. By way of example, reference can be made to the latest mill disclosed in JP2019037948; nevertheless, none of the company's patents specifies that the purpose is to obtain nanocellulose, rather they relate to the grinding of materials.

B) GEA NIRO SOAVI uses a system which is based on introducing cellulose fibers in a dilution through

a small cavity where a piston causes the closure of the cavity, increasing the pressure therein and reducing the size of the fiber. It appears that the passage of fibers through the cavity causes the machine to be readily gummed up, with continuous halts and disassembly for cleaning and resuming the processing, where said machine is unable to work with suspensions containing high-density fibers, limiting the use and causing this machine to obtain results that are far inferior to those obtained both with the present invention and with the solution indicated in point A). By way of example, reference can be made to the company's patents: CN102575751 entitled "Highpressure homogenizer with an epicyclic reduction gear unit", or US2010296363 entitled "Homogenizing valve".

[0003] There are other chemical methods and ways for obtaining nanocellulose; however, they are not in line with the present invention, giving rise to other types of circumstances and achievements which, although being studied continuously, have nothing to do with the methods, object of the present invention, used for obtaining nanocellulose.

[0004] The process of the present invention is an evolution of the existing mechanical methods, where it is much more efficient, and therefore entails a significant energy saving for obtaining same and a high degree of result repeatability, without generating any type of waste, utilizing 100% of the treated material.

[0005] Nanocellulose is produced after reducing cellulose fibers to a nanometric scale. To achieve a scale of between 50 and 100 nanometers, the original fiber must be greatly reduced, and this reduction is not performed efficiently with currently known methods and machines such as those described above.

[0006] KR 20170142836A discloses a method for producing cellulose fibers, and more particularly a method for producing nanocellulose fibers including the steps of subjecting a solution of cellulose in water to pressure in a chamber compressing the solution, passing the compressed solution through a nozzle with a frustoconical inlet experiencing a strong acceleration, passing through a more restricted area, where it increases the pressure of the solution and friction and collisions occur, giving rise to the shredding and precipitation of the solution in a collecting vessel, causing expansion.

Description of the Invention

[0007] The proposal of this invention for obtaining this solution is to use a new mechanical method that will pass the cellulose solution through a first compression step, to then be passed through a small opening arranged in the part which acts as an extrusion head that will be referred to as "nozzle" as it presents the characteristics typical of the passage with an angular inlet and outlet and a cylindrical central portion, causing the strong

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shredding of the cellulose solution to start in said step of the process.

[0008] Passage through said nozzle causes a strong acceleration in the solution, which causes a high speed of the solution with significant friction and achieving the turbulent regimen. All this together with the pressure with which it is introduced in the nozzle (between 250 and 600 bar at infeed plus Venturi effect) and significant friction with the walls thereof produces a combined shredding mechanism. Next, the solution moves to the subsequent step of exiting with a high negative pressure and of subsequent expansion, together with a large inertia due to the significant speed acquired to cause a final collision of said solution both against the actual walls of the outlet chamber and against an outlet front dead center. This process as a whole leads to obtaining nanocellulose (fibers of between 50 and 100 nm) the fibers of which furthermore have an elongated and loose fibrous structure, characteristics which result in a novel raw material, which enables a wide range of highly efficient applications and opportunities in various sectors.

[0009] The method object of the present invention is characterized by comprising several steps through which cellulose is converted into nanocellulose and, in that sense, it has:

- 1. An optional prior step which can fine tune and improve the main steps of the process object of the invention, where a prior step is recommendable for a good product result, increasing yield and efficiency. In this prior step, the raw material (bleached cellulose) is subjected to a process of mixing by shaking in a conventional device, such that a highly homogeneous starting solution is obtained, preventing shear in the fibers and facilitating the process to be performed later on in the other steps. This prior step consists of diluting the bleached cellulose in water, without any other component, in the desired consistency, leaving the preparation of the diluted cellulose solution in a proportion of between 1% and 6% to stand for 12 and 24 hours to then subject same to a shaking of between 7,000 and 12,000 revolutions per minute (rpm).
- 2. An initial step in which the cellulose diluted in water is subjected to a pressure of between 250 and 600 bar in a main working chamber. Homogenization of the cellulose solution at different concentrations of between 1% and 6% is performed in this chamber, which entails the need to resort to parts specially designed to withstand these pressures, for the purpose of ensuring process efficiency and directing the compressed solution to the nozzle.
- 3. An intermediate step, which is the most novel and original step, as it uses an innovative shredding of the cellulose diluted in water (in a proportion of between 1% and 6% and subjected to a pressure of

between 250 and 600 bar) in the process; in this step, the solution is passed through the nozzle which, by having a frustoconical-shaped inlet to facilitate the entry and guiding of the solution to the cylindrical central passage thereof with a very strong acceleration (upon acquiring a high fluid passage speed which can range between 50 meters/second and 250 meters/second), strong friction on the passage walls of the nozzle, with the subsequent turbulence in the solution which, along with the high pressure of the solution, produces the most important mechanism for shredding cellulose, with a significant part of the total nanocellulose being obtained in this intermediate step of the method. This arrangement and this step already allow obtaining different microfiber and nanofiber qualities, all this in combination of the pressure to which the solution is subjected and the use of the nozzle which is used in addition to the size of the central cylindrical passage area, where the diameter thereof can range between 0.2 millimeters and 2 millimeters and the length thereof between 3 and 100 millimeters, in combination of the sizes that can be imparted to the inlets and outlets of the nozzle and their frustoconical shapes made to that end, given that their angles and depth may vary according to the most suited entry onrush.

4. A final step of exiting the nozzle towards a collection chamber for collecting the solution with cellulose already partially transformed into nanocellulose. Nevertheless, it is in the final step, in the actual outlet of the nozzle, where the shredding is complemented by the solution being subjected to a strong expansion through the free decompression of the solution, or even aided by another additional decompression also of between the same 250 and 600 bar of the main chamber, but negative in the collection chamber itself. This, along with the speed with which the solution exits the nozzle, in turn causes a strong collision against the walls of the collection chamber and, very particularly, against the front outlet dead center thereof located between 15 and 150 millimeters (movable or non-movable), since the solution has a high speed and inertia.

[0010] Microcellulose and nanocellulose fibers are obtained with all these steps to which the solution is subjected which, based on the needs and purposes thereof, allows the use of virtually any device which will, however, always present a passage through the nozzle, reproducing the method as many times as deemed appropriate, and with as many passages the nozzle may have and the size thereof, in combination with the different pressures so as to allow obtaining the nanocellulose with more or less crystallization or transparency. All this is achieved, along with a better quality to reach a scale of between 50 and 100 nanometers, greatly reducing the original fiber with great homogeneity.

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[0011] Each of the steps has been optimized within the different envisaged options. In the initial cellulose feeding step and in the intermediate shredding step, different techniques and materials adapted to the dynamic behavior of microfibers have been tested. As a result of the foregoing, it has been observed that the different parameters of the arrangement used in each case must be adjusted: a) proportions for dissolving the solution with water of between 1% and 6% of cellulose, shaking the blend and leaving it to stand for between 12 and 24 hours in order to proceed with a new shaking of between 7,000 and 12,000 revolutions per minute; in combination with b) subjecting the solution in a chamber to a pressure of between 250 and 600 bar; in combination with c) providing a nozzle so that the solution passes through said nozzle having a central size with a diameter of between 0.2 and 2 millimeters and a length of between 3 and 100 millimeters; for finally passing into a receiving chamber in which the solution is subjected to an expansion with a negative decompression which can range between 250 and 600 bar, which can even be increased, depending on the geometry of the nozzle (Venturi effect and fluid dynamics), and to collision with the dead center which can be arranged more or less next to the outlet of the nozzle, between 15 mm and 150 mm, where it may be movable.

[0012] Due to the use of liquid solutions in the method, the device is subjected to the laws of fluid thermodynamics, being subjected to significant frictions and change of state of the solution with the corresponding energy transformations, causing heat to be given off (due to the very high friction with the walls of the nozzle), and this will lead to both the chambers and the nozzle itself being complemented with the corresponding cooling arrangements in order to withstand temperature changes with high pressures, frictions, turbulences, speeds, etc., occurring particularly inside the nozzle and during the passage of the solution therethrough with significant shredding during said passage.

Brief Description of the Drawings

[0013] The different steps of the method, parts, and arrangements of the device for shredding and obtaining nanocellulose object of the invention are explained below by means of the drawings complementing the specification, illustrating the preferred example, and helping to better understand the invention, consisting of an embodiment of said invention that is, however, in no case limiting thereof.

[0014] The foregoing and other features and advantages will be better understood based on the following detailed description of an embodiment in reference to the drawings of the attached figures, in which:

Fig. 1 shows a view of the device with the nozzle in combination with the compression and inlet chambers, with the chamber for decompressing and re-

ceiving the solution with its collision dead center, and the device for compressing and driving the solution.

Fig. 2 shows a view of the nozzle, its frustoconical inlet and outlet portions, and its cylindrical central passage.

Fig. 3 shows a view of the nozzle in the moment in which the solution passes through all the portions thereof.

Fig. 4 shows a view of the solution and the passage thereof through the nozzle, indicating the friction, speed, turbulence, with decompression and expansion with collision being caused in the receiving chamber and its dead center as the solution exits the nozzle.

Fig. 5 shows a view of the wholly similar device in full operation with the entry, passage, and exit of the solution subjected to the steps of the method object of present invention.

Fig. 6 shows a view of the method in which the functions of P=Pressure; S=Speed; F=Friction; T=Turbulence, and E=Expansion with collision are represented.

Description of the Different Elements of the Invention

[0015]

- 1. Solution of cellulose diluted in water in a proportion of 1% to 6%.
- 2. Compression chamber for compressing the solution (1), which is arranged facing the inlet of the nozzle (3) and has a compressor (12).
- 3. Nozzle having a passage (4) for the solution (1) which is arranged in combination with the compression chamber (2) on one side, and with the receiving chamber (7) on the other side.
- 4. Cylindrical tubular passage of the nozzle through which the solution (1) passes causing a strong turbulence (T) with friction (F) at speed (S), and which is limited at the ends thereof by the corresponding frustoconical inlet (5) and likewise frustoconical outlet (6).
- 5. Frustoconical inlet of the nozzle (3).
- 6. Frustoconical outlet of the nozzle (3).
- 7. Receiving chamber for receiving the solution (1).
- 8. Dead center of the receiving chamber (7) against

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which the solution (1) collides.

- 9. Wall of the frustoconical inlet (5) against which the solution (1) hits.
- 10. Perimetral wall of the passage (4) of the nozzle (3) against which the solution (1) hits.
- 11. Wall of the frustoconical outlet (6) against which the solution (1) hits.
- 12. Compressor device of the compression chamber (2) which furthermore directs the solution towards the nozzle (3).
- 13. Decompressor device of the receiving chamber (7) to increase the expansion of the solution (1).
- 14. Cooling device.

[0016] E. Expansion of the solution (1) in the receiving chamber (7) due to decompression.

[0017] P. Pressure on the solution (1) in the compression chamber (2) with the compressor (12).

[0018] R. Friction of the solution (1) produced on the frustoconical inlet and outlet walls (9) and (11), as well as on the wall of the passage (4) of the nozzle (3).

[0019] T. Turbulence of the solution (1) as it goes through the nozzle (3) through the passage (4) thereof and the frustoconical inlets (5) and outlets (6).

[0020] V. Speed reached by the solution (1) of between 50 m/s and 250 m/s.

Detailed Description of an Embodiment

[0021] The attached figures show the preferred embodiment of the shredding method and arrangement for obtaining nanocellulose object of the present invention, consisting of:

- 1. As an optional prior step or embodiment, obtaining a solution (1) which, using a raw material of bleached cellulose, is subjected to a process of mixing by shaking in a conventional device such that a starting solution that is as homogeneous as possible is obtained. To that end, the cellulose is diluted with water in a mean proportion of 2.5% (depending on the qualities to be obtained, where said proportion may vary between 1% and 6%), left to stand for between 12 and 24 hours, and then subjected to shaking of between 7,000 and 12,000 rpm.
- 2. Once the solution (1) is obtained, the real method object of the invention begins, said method consisting of subjecting the solution (1) in a compression chamber (2) to a mean pressure (P) of +/-425 bar. Said chamber will be arranged in contact with and limited on one of the walls thereof by the nozzle (3),

and the chamber will in turn have the compressor (12) which directs the solution (1) to said nozzle (3) and to its passage (4).

3. It is in the main intermediate step of the present invention where the solution (1), subjected to a pressure (P) of +/-425 bar, is directed to the nozzle (3) through its frustoconical inlet (5), at which time a strong acceleration is caused on the solution (1) since, in a span of +/-5 millimeters that is the depth of the frustoconical inlet (5), the solution goes from a speed of +/-0.02 m/s to a speed (S) of between 50 m/s and 250 m/s (flow speed limit conditioned by friction with the walls and the diameter of the nozzle) with which it will go through the passage (4) of the nozzle (3). In this case, the passage has a diameter of +/-0.6 mm and a length of +/-8 mm to begin the exit towards the frustoconical outlet portion (6), which will have a depth of +/-5 mm in which a slight expansion and decompression begins. All this causes, during said passage through the nozzle, a strong friction (F) of the compressed (P) solution (1) at speed (S) on the wall (9) of the frustoconical inlet (5), on the perimetral wall (10) of the passage (4), and finally on the wall (11) of the frustoconical outlet (6), which friction (F) will in turn cause a strong turbulence (T) in the solution (1), leading to major cellulose shredding in this intermediate step and in combination with the nozzle (3) and the arrangement thereof. Accordingly, a significant amount of nanocellulose will be obtained since, with the acceleration from +/-0.02 m/s to between 50 m/s and 250 m/s in a span of +/-5 millimeters, the solution (1) would experience an acceleration at the molecular level that is so significant that, in the passage (4) with friction (F) on the perimetral wall (10), mainly longitudinal shredding is caused from the inlet to the outlet for the solution (1), and particularly in the bleached cellulose diluted in the solution (1).

[0022] It should be indicated that a nozzle (3) can have one or more passages (4) with their corresponding frustoconical inlets (5) and outlets (6), respectively.

[0023] 4. In order to reach a final complementary step of the process for obtaining nanocellulose, in the solution (1), when said solution (1) exits the nozzle (3) through the frustoconical outlet (6) to a receiving chamber (7) which, in this case of a preferred embodiment, has zero pressure, but in which the solution which exits the nozzle (3) at a mean pressure (P) of +/-425 bar together with a mean speed of between 50 m/s and 250 m/s faces a total decompression, causing a strong expansion (E) of the solution (1) which will collide against all the walls of the receiving chamber (7), and very particularly and to a large extent, against the dead center (8) arranged in the receiving chamber (7), this as a result of the high speed and the actual inertia with which the solution (1) exits the nozzle (3) through the frustoconical outlet (6).

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[0024] 5. Steps two, three, and four can be repeated as many times as deemed appropriate and necessary for obtaining a more homogenous nanocellulose, where the third intermediate step is the step in which the greatest shredding occurs and nanocellulose is obtained. The last step through the conventional (centrifugation) methods separates the solution (1) into water on one hand and nanocellulose on the other. The method for obtaining nanocellulose will thus be completed, obtaining nanocellulose with the characteristics that are deemed appropriate and necessary.

[0025] The device which would be used for putting this method into practice and which is thus deduced from the description made is the combination of:

- A compression chamber (2) which would have, on one side, the compressor (12) which would in turn drive the solution (1). On the other side, it would be facing a nozzle (3) which would have a passage (4) towards which the solution (1) would be directed once compressed.
- A nozzle (3) which, in combination with the compression chamber (2), would receive the solution (1) with the pressure (P) through its frustoconical-shaped inlet (5) which, like a funnel, would direct the solution (1) in an accelerated manner towards the passage (4) through which the solution (1) would pass at a speed (S) of between 50 m/s and 250m m/s, with strong friction (F) on its perimetral wall (10), which would in turn cause turbulence (T) so that, through the frustoconical outlet (6), the solution would go to the receiving chamber (7) with which the nozzle (3) is also arranged in combination on the other side.
- A receiving chamber (7) which, in combination with the nozzle (3) and the solution (1), receives the solution with a speed (S) of between 50 m/s and 250 m/s with a compression of +/-425 bar, in order to change it to zero pressure, which will cause decompression and corresponding expansion (E) of the solution (1) which, as a result of inertia itself, will collide against the dead center (8) of the receiving chamber (7).

[0026] This device can be complemented with a cooling system (14) for cooling the entire assembly, i.e., the compression chamber (2), the nozzle (3), and the receiving chamber (7), due to the occurrence of heating which may be too high in some cases, taking into account the pressure (P) and frictions (F) in combination with turbulence (T), the speed (S), as well as the expansion (E) and decompression, so the entire device can or should be cooled to ensure the proper operation thereof.

[0027] This will be the device with the basic elements which put the method into practice in the main and intermediate steps of the present invention.

[0028] On the whole, the invention relates to a method

and device object of the invention for obtaining nanocellulose by means of shredding through the combination of pressure, acceleration, speed, friction, turbulence, expansion, decompression, and collision, in a mechanical and non-chemical manner.

Claims

1. A shredding method for obtaining nanocellulose of the types which are mechanical and based on a bleached cellulose diluted in water, the bleached cellulose being subjected in a combined manner to pressure (P), acceleration, speed (S), friction (F), turbulence (T), expansion (E), characterized in that the method is based on a solution (1) comprising a bleached cellulose diluted in water in a percentage from 1% to 6%, said solution (1) being subjected to:

> - a pressure (P) of between 250 and 600 bar in a compression chamber (2) in order to force the passage of the solution (1) through a nozzle (3). - forced passage of the compressed solution (1) through a nozzle (3) in which:

a) in a frustoconical inlet (5) having a length of +/-5 mm with an outer ring perimeter matching the compression chamber (2), greater than an inner ring perimeter which will match the perimeter of the passage (4), the solution (1) goes from a speed (S) of +/-0.02 m/s in the compression chamber (2) to a speed of between 50 m/s and 250 m/s in the nozzle (3), giving rise to a first shredding of the solution (1).

- b) in its cylindrical passage (4) and with the solution (1) being at a pressure (P) of between 250 and 600 bar and at a speed (S) of between 50 m/s and 250 m/s, a friction (F) occurs in the perimetral wall (10) of the tubular passage (4), which causes a turbulent regimen (T) in the solution (1), causing a second shredding.
- c) in a frustoconical outlet (6) of the nozzle (3), arranged inversely with respect to that of the inlet (5), the start of an expansion (E) and decompression of the solution (1) is caused,
- expansion (E), decompression, and collision of the solution (1) at the outlet of the nozzle (3) where the solution (1) rushes into a receiving chamber (7) which is at a pressure of between zero to between less than 250 and less than 600 bar, which causes an expansion (E) and decompression of the solution (1) which, along with the speed (S) with which the solution exits the nozzle (3), causes the collision of the solution (1)

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against all the walls of the receiving chamber (7), and particularly against a wall functioning as a dead center (8), which is arranged facing the frustoconical outlet of the nozzle at a distance of between 15 mm and 150 mm, which again causes a third shredding of the solution (1), thereby obtaining the shredding and the nanocellulose.

2. The method according to claim 1, characterized in that it includes a prior step in which said solution (1):

> is subjected to a prior mixing by shaking in order to obtain the diluted solution (1) of bleached cellulose in water in a proportion of between 1% and 6% in a homogeneous manner.

> the solution (1) is left to sit between 12 and 24 hours, to then subject same to a shaking of between 7,000 and 12,000 rpm.

- 3. The method according to claim 1, characterized in that the method described in claim 1 is repeated until obtaining a more homogeneous nanocellulose on a scale of between 50 and 100 nanometers.
- 4. The method according to claim 1 or 3, characterized in that the solution (1), after the steps of pressure in the compression chamber (2), forced passage through the nozzle (3), expansion, decompression, and collision in the receiving chamber, is separated in a conventional way by centrifugation or decantation of the water and of the obtained nanocellulose in the proportions in which it had been diluted from 1% to 6%.
- 5. A shredding device for obtaining nanocellulose of the types which are mechanical, characterized in that it comprises in combination:

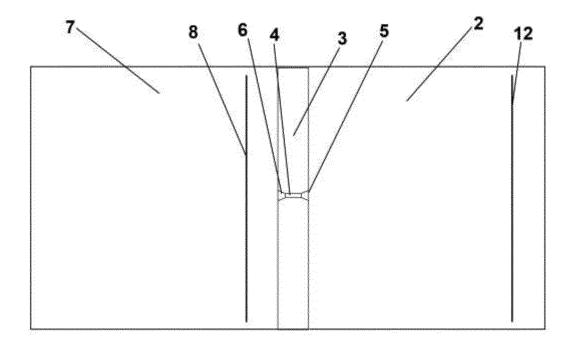
a) a compression chamber (2), equipped with a compressor (12), for compressing the solution (1) between 250 and 600 bar, directing the solution (1) towards a nozzle (3);

b) a nozzle (3) through which the solution (1) passes through a frustoconical inlet (5) having a length of +/-5mm, the larger perimeter of which is always arranged next to the compression chamber (2) and the smaller perimeter thereof always matches the perimeter of a central passage (4) having a cylindrical shape the diameter of which is between 0.2 mm and 2 mm and the length thereof ranges between 3 mm and 100 mm; and a frustoconical outlet (6) which is the same as the frustoconical inlet (5) but arranged inversely, the smaller perimeter thereof being of 55 the same size as that of the passage (4); and c) a receiving chamber (7) which is arranged on the other side of the nozzle (3) and receives the

frustoconical outlet (6), having a dead center (8) which is arranged at a distance of between 15 mm and 150 mm from the frustoconical outlet (6) of the nozzle (3), in which chamber the solution (1) is decompressed and expanded colliding against all the walls thereof and preferably against the dead center (8).

- 6. The device according to claim 5, characterized in that said dead center (8) is suitably movable to provide greater or less collision in combination with the decompression in the receiving chamber (7).
- 7. The device according to claim 5, characterized by having a decompression device (13) which, regardless of the dead center (8), causes a decompression of less than zero, that is, a negative compression, in order to increase the expansion of the solution (1) in the receiving chamber (7).
- 8. The device according to claim 5, characterized by having a refrigeration (14) for cooling the compression chamber (2), the nozzle (3), and the receiving chamber (7).
- The device according to claim 5, characterized by the nozzle (3) having one or more passages (4) with their corresponding frustoconical inlets (5) and outlets (6), respectively.

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<u>FIG. 1</u>

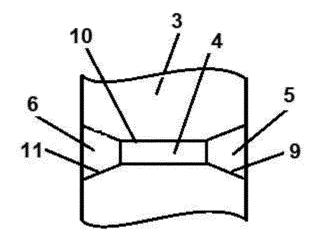


FIG. 2

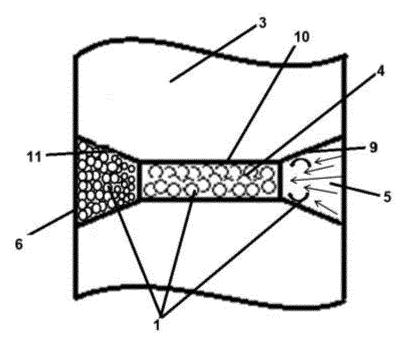
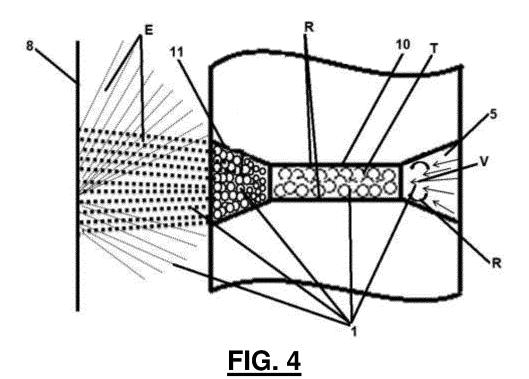


FIG. 3



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FIG. 5

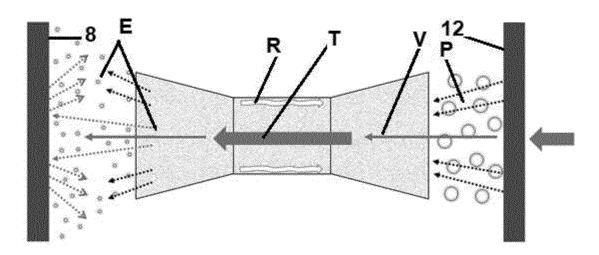


FIG. 6

International application No. INTERNATIONAL SEARCH REPORT PCT/ES2020/070733 5 A. CLASSIFICATION OF SUBJECT MATTER See extra sheet According to International Patent Classification (IPC) or to both national classification and IPC B. FIELDS SEARCHED 10 Minimum documentation searched (classification system followed by classification symbols) D21B, D21H Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched 15 Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) EPODOC, INVENES, WPI C. DOCUMENTS CONSIDERED TO BE RELEVANT 20 Category* Citation of document, with indication, where appropriate, of the relevant passages Relevant to claim No. KR 20170142836 A (ELECTRONICS & TELECOMMUNICATIONS RES 1-9 Α INST) 28/12/2017, figure 1; paragraph [3]; paragraphs [5 - 6]; paragraphs [15 - 17]; 25 paragraph [22]; paragraphs [24 - 25]; ABDUL KHALIL H P S et al. 30/11/2013, 1-9 A Production and modification of nanofibrillated cellulose using various mechanical processes: A review. Carbohydrate Polymers 2014 Elsevier Ltd Gbr., Vol. 99, pages 649 - 665, ISSN 0144-8617 (print), 30 <DOI: 10.1016/j.carbpol.2013.08.069> pages 651 - 653; WO 2010131016 A2 (IMERYS MINERALS LTD ET AL.) 18/11/2010, 1-9 Α page 4, lines 1 - 26; page 20, line 22 - page 21, line 13; page 24, lines 12 - 29; US 5964983 A (DINAND ELISABETH ET AL.) 12/10/1999, A 1-9 35 column 3, lines 6 - 9 and 56 - 67; column 5, lines 9 - 45; ☑ Further documents are listed in the continuation of Box C. See patent family annex. 40 later document published after the international filing date Special categories of cited documents: document defining the general state of the art which is not or priority date and not in conflict with the application but considered to be of particular relevance. cited to understand the principle or theory underlying the invention earlier document but published on or after the international filing date document which may throw doubts on priority claim(s) or "X" document of particular relevance; the claimed invention which is cited to establish the publication date of another cannot be considered novel or cannot be considered to 45 involve an inventive step when the document is taken alone citation or other special reason (as specified) document referring to an oral disclosure use, exhibition, or "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the other means. document is combined with one or more other documents, such combination being obvious to a person skilled in the document published prior to the international filing date but later than the priority date claimed document member of the same patent family 50 Date of the actual completion of the international search Date of mailing of the international search report (17/02/2021)02/02/2021

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