



(51) International Patent Classification:

B02C 13/22 (2006.01) *D21B 1/14* (2006.01)
B02C 13/20 (2006.01) *D21D 1/20* (2006.01)

(21) International Application Number:

PCT/IB2013/058633

(22) International Filing Date:

18 September 2013 (18.09.2013)

(25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data:

61/703,472 20 September 2012 (20.09.2012) US

(71) Applicant: STORA ENSO OYJ [FI/FI]; Po 309, FI-00101 Helsinki (FI).

(72) Inventors: Nordström, Jan-Erik; Törnrosavägen 4, S-13147 Nacka (SE). Kostianen, Petteri; Aholantie 79, FI-03150 Huhmari (FI). Heiskanen, Isto; Kanava-aukio 10 as 13-14, FI-55100 Imatra (FI). Axrup, Lars; Lövnäsvägen 12, S-66341 Hammarö (SE).

(74) Agent: Magnusson, Ulf; Stora Enso AB, Group IP, Box 9090, S-65009 Karlstad (SE).

(81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM,

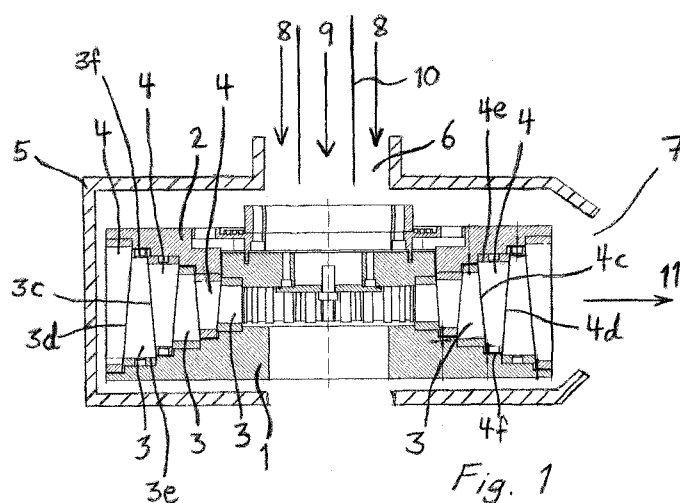
AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

Published:

- with international search report (Art. 21(3))
- before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments (Rule 48.2(h))

(54) Title: METHOD AND DEVICE FOR DEFIBRATING FIBRE-CONTAINING MATERIAL TO PRODUCE MICRO-FIBRIL-LATED CELLULOSE



(57) Abstract: Device for defibrating fibre-containing material to produce microfibrillated cellulose, wherein the device comprising a rotatably-mounted first rotor (1) equipped with collision surfaces (3a, 3b) mounted thereon in concentric conical rings (31, 32, 33; 51), and adapted to rotate in a first direction; a rotatably-mounted second rotor (2) equipped with collision surfaces (4a, 4b) mounted thereon in concentric conical rings (41, 42, 43; 1), and adapted to rotate in a second direction which is opposite to the first direction of the first rotor (1), said second rotor is in cooperating relationship with the said first rotor such that the collision surfaces of said first rotor and the collision surfaces of said second rotor are interspersed; and a housing (5) comprising said first and second rotors, and having feed inlet (6) and a feed outlet (7), said inlet is in communication with a centre of said first and second rotors and being sufficiently large that fibre-containing material (8) may be fed freely there through.

METHOD AND DEVICE FOR DEFIBRATING FIBRE-CONTAINING MATERIAL TO PRODUCE MICRO-FIBRILLATED CELLULOSE

Field of invention

The present invention relates to a device and a method for defibrating fibre-containing material to produce micro-fibrillated cellulose.

The concept "fibre-containing material" is later to be understood as a wide concept comprising wood chips, grass and other fibre-containing materials originating from the vegetable kingdom, which have been crushed into pieces of appropriate sizes so they can be fed into the device.

The concept "defibrating" covers separation of fibres from each other, wood or other raw materials in the vegetable kingdom. In this text, the concept also covers fibrillation of separate fibres.

The concept "microfibrillated cellulose" (MFC) is also known as nanocellulose. It is a material typically made from wood cellulose fibres, both from hardwood or softwood fibres. It can also be made from microbial sources, agricultural fibres such as wheat straw pulp, bagasse, bamboo or other non-wood fibre sources. In microfibrillated cellulose the individual microfibrils have been partly or totally detached from each other. A microfibrillated cellulose fibre is normally very thin (~20 nm) and the length is often between 100 nm to 10 µm. However, the microfibrils may also be longer, for example

between 10-200 μm , but lengths even 2000 μm can be found due to wide length distribution. Fibres that has been fibrillated and which have microfibrils on the surface and microfibrils that are separated and located in a water phase of slurry are included in the definition MFC. Furthermore, whiskers are also included in the definition MFC.

The present invention also relates to a paper or board product produced according to the process.

Prior art and background of the invention

US 6,202,946 discloses a method and a device for defibrating fibre-containing material. The device comprises a housing, which comprises a first rotor equipped with rectangular collision surfaces and a second rotor equipped with rectangular collision surfaces. The second rotor is concentrically arranged with the first rotor and is arranged to rotate in a direction opposite to the first rotor. The device further comprises a feed orifice in the housing and opening to the centre of the rotors and a discharge orifice on the housing wall and opening to the periphery of the outermost rotor. The fibre-containing material is led from the feed orifice to the housing and made to flow together with a liquid suspension through the rectangular collision surfaces of the nested rotors to the discharge orifice and further as a discharge flow out of the housing.

US 6,843,886 is a similar device for defibrating fibre-

containing material. In accordance with the device above, this device also discloses rotors equipped with rectangular collision surfaces.

WO 2013/072559 discloses a method for producing nanofibril cellulose through several counter-rotating rotors outwards in the radial direction with respect to the rotation axis of the rotors in such a way that the material is repeatedly subjected to shearing and impacting forces by the effect of rectangular blades.

There is a need for different types of nanocellulose and micro fibrillated cellulose for different end applications. For high quality (high strength potential) micro fibrillated cellulose, the target is to maintain the fibril length as long as possible, i.e. to produce as high length/thickness ratio as possible. For high quality micro fibrillated cellulose the ratio should always be >100, preferably >1000 and even more preferred >10000. This type of micro fibrillated cellulose is not possible to produce with normally used mechanical means (grinding/fluidization/homogenization etc.). If high length/thickness ratio is tried to be produced (for example with masuko grinder), fibril bundles (flocculation and mechanical binding between fibrils) becomes a major hindrance of the strength development. Production of large amounts of short and thick finer material is normally not considered positive in strength enhancement applications (such as paper making, different types of composites, films etc.). High amount of fines (i.e. low length/thickness ratio) will cause dewatering and retention problems in high speed paper making without

giving the best possible effect on strength properties. Simultaneously this type of low length/thickness ratio material efficiently reduces porosity of the materials where it has been added.

Thus, there is a need for a new type of machine arrangement, which makes it possible to produce high length/thickness ratio micro fibrils.

Thus, there is also a need for a machine arrangement, which makes possible to break down fibril bundles formed in MFC making for example grinding operations (for example masuko grinders) and fluidization/homogenization.

Object of invention

An object of the present invention is to provide a device and a method which enables to produce high length/thickness ratio micro fibrils.

Another object is to provide a device and a method which enables to break down fibril bundles formed in MFC making for example grinding operations (for example masuko grinders) and fluidization/homogenization.

Yet another object is to provide a device and a method that enables an increase of the load between the rotating parts, such that, the shear-forces to fibrillate the fibres at a higher extent - where the goal is to get long fibrils that are not cut nor collected in lumps/bunches.

It is yet another object to provide a device and a method where the distance between the rotating part can be adjusted.

The defibrated material, made using the device or the method according to the invention, may be used in any micro cellulose application as paper and paperboard but also in various polymer/plastics engineering areas, food/feed areas, cosmetics area and pharmacy area.

The device, according to the invention, may also, when enabling these high shear forces, work as a dosing point of chemicals to get accurate blending of the chemicals properly to reduce the application amount and increase the efficiency of a certain chemical dosage.

Summary of the invention

The device according to the invention is characterized in that the device comprising:

- a) a rotatably-mounted first rotor equipped with collision surfaces mounted thereon in at least one, concentrically arranged, conical ring, and adapted to rotate in a first direction;
- b) a rotatably-mounted second rotor equipped with collision surfaces mounted thereon in at least one, concentrically arranged, conical ring, and adapted to rotate in a second direction, which direction is opposite to the first direction of the first rotor, said second rotor is in cooperating relationship with the first rotor

such that the collision surfaces of said first rotor and the collision surfaces of said second rotor and are interspersed; and

- c) a housing comprising said first and second rotors, and having feed inlet and a feed outlet, said inlet is in communication with a centre of said first and second rotors and being sufficiently large that fibre-containing material may be fed freely there through; said outlet is in communication with a periphery of an outermost ring.

The method according to the invention is characterized in that the method comprising the steps of:

- i) providing a defibrating device comprising
 - a) a rotatably-mounted first rotor equipped with collision surfaces mounted thereon in at least one concentric, conical ring, and adapted to rotate in a first direction;
 - b) a rotatably-mounted second rotor equipped with collision surfaces mounted thereon in at least one concentric, conical ring, and adapted to rotate in a second direction which is opposite to the first direction of the first rotor, said second rotor is in cooperating relationship with the said first rotor such that the collision surfaces of said first rotor and the collision surfaces of said second rotor and are interspersed; and
 - c) a housing comprising said first and second rotors, and having feed inlet and a feed outlet, said inlet is in communication with a

centre of said first and second rotors and being sufficiently large that fibre-containing material may be fed freely there through; said outlet is in communication with a periphery of an outermost ring;

ii) feeding said fibre containing material into said device such that the fibre-containing material is made to flow with the aid of air or liquid to said first and second rotors, and then through said collision surfaces to said outlet to produce microfibrillated cellulose; and
iii) collecting said microfibrillated cellulose from said outlet.

Detailed description of a preferred embodiment

In the following, the invention will be described in some preferred embodiments with reference to the drawings, wherein:

Figure 1 shows an elevation view of a first embodiment of a device for defibrating fibre, where the device comprises two rotatable rotors with blades which forms conical rings on the rotors.

Figure 2 shows a detailed view of the device in figure 1, where the upper rotor is in an upper position.

Figure 3 shows a top view of the rotors in Figure 1 and 2.

Figure 4 shows an alternative, second, embodiment of the device for defibrating fibre, wherein the blades is

replaced with a conical ring body which comprises radial holes.

Figure 5 shows an elevation view of the second embodiment in figure 4.

Figure 6 shows a top view of the second embodiment in figure 4.

Figure 7 shows a detailed view of the inner surface of the conical ring body.

The concept "tapered collision surface" will hereinafter be frequently used in this text, with reference to the first embodiment of the invention in figures 1-3. It should be noticed that "tapered collision surface", in this application, incorporates a geometric figure in the form of a truncated cone or a truncated pyramid or a wedge or similar geometric figures.

According to figure 1-3, the device of comprises a housing 5 (not shown in figure 2-3), which accommodates a rotatably-mounted, lower, first rotor 1 equipped with blades 3. The blades 3 of the first rotor 1 are arranged in concentric rings 31, 32, 33. Each ring 31, 32, 33 comprises a circumferential, inner surface 31a, 32a, 33a, which faces against the centre of the rotation axis of the first rotor 1, and a circumferential, outer surface 31b, 32b, 33b, which faces away from the circumferential, inner surface 31a, 32a, 33a. At least one of the circumferential, inner surface and circumferential, outer surface is inclining, such that, the cross section of each ring 31, 32, 33 is wedge shaped, i.e. the rings 31,

32, 33 are conical. The angle of each inclining surface is about 3-7°, preferably 5° from the rotation axis of the rotor 1. The first rotor 1 is adapted to rotate in a first rotation direction.

The housing 5 also accommodates a rotatably-mounted, upper, second rotor 2, which is concentric with the first rotor 1. The second rotor 2 is also equipped with blades 4. The blades 4 of the second rotor 2 are arranged in concentric rings 41, 42, 43. Each ring 41, 42, 43 comprises a circumferential, inner surface 41a, 42a, 43a, which faces against the centre of the rotation axis of the second rotor 2, and a circumferential, outer surface 41b, 42b, 43b, which faces away from the circumferential, inner surface 41a, 42a, 43a. At least one of the circumferential, inner surface and the circumferential, outer surface is inclining, such that, the cross section of each ring 41, 42, 43 is wedge shaped, i.e. the rings 41, 42, 43 are conical. The angle of each inclining surface is about 3-7°, preferably 5° from the rotation axis of the rotor 2. The second rotor 2 is adapted to rotate in a second rotation direction, which second rotation direction is opposite to the first rotation direction of the first rotor 1.

The conical rings 31, 32, 33 of the first rotor 1 and the conical rings 41, 42, 43 of the second rotor 2 are intermeshed, such that, the circumferential, inner surface 31a, 32a, 33a of a ring 31, 32, 33 of the first rotor 1 faces against a circumferential, outer surface 41b, 42b, 43b of a ring 41, 42, 43 of the second rotor 2 and the circumferential, outer surface 31b, 32b, 33b of a ring 31, 32, 33 of the first rotor 1 facing against an

inner surface 41a, 42a, 43a of a ring 41, 42, 43 of the second rotor 2. The first rotor 1 and the second rotor 2 can freely rotate in different directions.

The head of the housing 5 is provided with an inlet 6, which opens out to the centre of the rotors 1, 2 and is used as an inlet for fibre-containing material 8 to be fibrillated. The inlet 6 is also an inlet for dilution water and/or chemistry 9 which is located in a central pipe 10 inside the inlet 6. This feeding arrangement is possible, due to the shafts of the rotors 1, 2 are arranged within each other (not shown in figures). The inlet 6 is sufficiently large, such that, the fibre-containing material 8 may be fed freely at ambient pressure.

The wall of the housing 5 is provided with an outlet 7, which opens and is in communication with a periphery to the outermost blade ring 33 and is used as a delivery outlet of fibrillated material 11, i.e. microfibrillated cellulose 11.

Figure 3 shows the rotation directions of the rings 31, 32, 33; 41, 42, 43 of the rotors 1, 2. As shown in figure 3, the rotation direction of the two rotors are opposite to each other.

Each blade 3 in the rings 31, 32, 33 of the first rotor 1 comprises a first tapered collision surface 3a and a second collision surface 3b which faces away from the first collision surface 3a. The blades 3 are arranged, such that, the collision surfaces 3a, 3b facing in a

circumferential direction of the rings 31, 32, 33.

The blade 3 further comprising an inner, inclining first edge surface 3c and an outer, inclining second edge surface 3d, which edge surfaces 3c, 3d are arranged between the first collision surface 3a and the second collision surface 3b and faces away from each other (see Figure 1). The first edge surface 3c faces against the rotation axis of the rotors 1, 2 and the second edge surface 3d faces away from the rotation axis of the rotors 1, 2. The inclining edge surfaces 3c, 3d are perpendicular to the first and second collision surfaces 3a, 3b. The angle of each inclining edge surface is about 3-7°, preferably 5° from the rotation axis of the rotors 1, 2.

The inner, first edge surface 3c of the blades 3 forming the circumferential, inner surfaces 31a, 32a, 33a of the rings 31, 32, 33 of the first rotor 1. The circumferential, outer, second edge surface 3d of the blades 3 forming the circumferential, outer surface 31b, 32b, 33b of the rings 31, 32, 33 of the first rotor 1.

The blade 3 further comprises a base surface 3e, which is arranged between the first collision surface 3a and the second collision surface 3b. The blade 3 is connected to the first rotor 1 through the base surface 3e.

Finally, the blade 3 comprises a top surface 3f which is arranged between the first collision surface 3a and the second collision surface 3b. The top surface 3f faces away from the base surface 3e and against the second

rotor 2. The distance H between the top surface 3f and the second rotor 2 is about 1-3 mm, preferably 2 mm.

Each blade 4 in the rings 41, 42, 43 of the second rotor 2 comprises a first tapered collision surface 4a and a second collision surface 4b which faces away from the first collision surface 4a. The blades 4 are arranged, such that, the collision surfaces 4a, 4b facing in a circumferential direction of the rings 41, 42, 43.

The blade 4 further comprising an inner, inclining first edge surface 4c and an outer, inclining second edge surface 4d which edge surfaces 4c, 4d are arranged between the first collision surface 4a and the second collision surface 4b and faces away from each other (see Figure 1). The first edge surface 4c faces against the rotation axis of the rotors 1,2 and the second edge surface 4d faces away from the rotation axis of the rotors 1, 2. The inclining edge surfaces 4c, 4d are perpendicular to the first and second collision surfaces 4a, 4b. The angle of each inclining edge surface 4c, 4d is about 3-7°, preferably 5° from the rotation axis of the rotors 1, 2.

The inner, first edge surface 4c of the blades 4 forming the circumferential, inner surfaces 41a, 42a, 43a of the rings 41, 42, 43 of the second rotor 2. The outer, second edge surface 4d of the blades 4 forming the circumferential, outer surface 41b, 42b, 43b of the rings 41, 42, 43 of the second rotor 2.

The blade 4 further comprises a base surface 4e which is arranged between the first collision surface 4a and the

second collision surface 4b. The blade 4 is connected to the second rotor 2 through the base surface 4e.

Finally, the blade 4 comprises a top surface 4f which is arranged between the first collision surface 4a and the second collision surface 4b. The top surface 4f faces away from the base surface 4e and against the first rotor 1. The distance H between the top surface 4f and the first rotor 1 is about 1-3 mm, preferably 2 mm.

Since the rings 31, 32, 33 of the first rotor 1 and the rings 41, 42, 43 of the second rotor are intermeshed, a circumferential, inner surface 31a, 31b, 31c of a ring 31, 32, 33 of the first rotor 1 faces against a circumferential, outer surface 41b, 42b, 43b of a nearby ring 41, 42, 43 of the second rotor 2; and a circumferential, outer surface 31b, 32b, 33b of a ring 31, 32, 33 of the first rotor 1 faces against a circumferential, inner surface 41a, 42a, 43a of a nearby ring 41, 42, 43 of the second rotor 2. The gap L between the two nearby rings i.e. the gap between two nearby inclining surfaces of the first and second rotors 1,2, is 0.01-1 mm, preferably 0.1 mm.

The inclination of two nearby edge surfaces 3c and 4c are equal for the first rotor 1 and the second rotor 2.

The upper, second rotor 2 is movable in an axial direction upwards and/or downwards, such that, the gap L between two nearby rings 31, 32, 33, 41, 42, 43 of the first and second rotors 1, 2 can be changed thanks to the inclining surfaces of the rings. When the upper, second rotor 2 is in its highest position (figure 2), the gap L is about 1 mm. When the upper, second rotor 2 is in its

lowest position (figure 1) the gap L is about 0.1 mm. The level of the upper, second rotor can be varied about 10 mm in its axial direction to close/open the gap L between the rings. Hence, a movement of 0-10 mm gives a gap L between 0.1-1.0 mm between the conical rings.

This enables that the load between the rotating rings can be adjusted. The load can therefore be increased, such that, the shear-forces to defibrillate the fibres at a higher extension, where the goal is to get long fibrils that are not collected in lumps/bunches.

According to an advantageous embodiment of the invention, the device is constructed in such a way that the distance S (see figure 3) between the blades in the outermost rings is smaller than the distance between the blades in the inner rings.

The above-mentioned measures may be used to secure that also coarser fibre-containing material (coarse wood and/or fibres) may be fed into the device, and nevertheless well enough defibrated pulp may be achieved. An essential advantage is that the number blades in the rotor rings and the distances (tightness) between the rings are selected according to the need. The distance S between the rings, as well as the distance between the blades in the ring, may be arranged so that they decrease towards the outer ring. In this way the dispersing pieces of wood chips are led to tighter and tighter spaces before the established suspension is discharged from the device.

The tapered collision surfaces of the conical rings, whose cross-sectional-profile is triangular, are turned, in such a way, that the impact surfaces of the blades are radial. Furthermore, the blades of one or more rings may be turned, e.g. to increase the impact power, in such a way that the direction of their collision surfaces derivate from the radial direction.

The rotors of the device rotate at rates from 800 to approximate 3000 rpm, preferably about 1500 rpm. Thus, the outermost rings increase their peripheral speed as the ring diameter increases and thereby induce for each ring outwards an increased shear force to the material processed.

The present invention enables that the load between the rotating parts through the conical construction, increases thereby shear-forces to defibrillate the fibres at a higher extension, where the goal is to get long fibrils that are not collected in lumps/bunches.

The invention is not limited, in such a way, that the rings, with circumferential inclining surfaces, necessarily are formed by blades as described above.

In accordance with the invention the fibre material is introduced through several counter rotating collision-surfaces, outwards in the radial direction with respect to the axis of rotation of the rotors, in such a way, that the materials is repeatedly subjected to shear and impact forces by the effect of the different counter-rotating collision-surfaces, whereby it is simultaneously fibrillated.

As a matter of great importance, the fibre material is repeatedly impacted by the blades or ribs of the rotors striking it from opposite directions when the blades rotate at the rotating speed and at the peripheral speed determined by the radius (distance to the rotation axis) in opposite directions. Because of the fibre material is transferred outwards in the radial direction, it crashes onto the collision surfaces, coming one after each other at a high peripheral speed from opposite directions, in other words, it receives several successive impacts from opposite directions. Also, at the edges of the wide surfaces of the blades, i.e. collision surfaces, which edges forms the gap L with the opposite edge of the nest rotor blade, shear forces occur, which contribute to the fibrillation.

Figure 4-7 discloses an alternative, second embodiment of the invention, where the rings 51 are formed by a continuous ring body 52. The figures 4-7 does only show one single ring 51, but in accordance with the first embodiment of the invention, the rings 51 are likewise concentrically arranged in the housing 5 onto the first and second rotors 1,2. Each ring 51 comprises a circumferential, inner surface 51a, which faces against the centre of the rotation axis of the first and second rotors 1, 2 and a circumferential, outer surface 51b, which faces away from the circumferential, inner surface 51a. At least one of the inner and outer surfaces is inclining, such that, the cross-section of each ring 51 is wedge shaped, i.e. the ring 51 is conical. The angle of each inclining surface is about 3-7°, preferably 5° from the rotation axis of the rotors 1, 2.

Each conical ring body 52 comprises circular holes 53, which are uniformly distributed over the inner surface 51a and outer surface 51b, for transport of material to be fibrillated through the conical rings 51. Each hole 53 is arranged in a radial direction through the inner and outer surface. Each hole 53 has two diameters through the ring body, such that, the hole diameter closest to the inner surface 51a is larger than the hole diameter closest to the outer surface 51b. The hole diameter closest to the inner surface 51a is preferably about 16 mm and the hole diameter closest to the outer surface is preferably about 7 mm. The circumferential, inner surface of each hole 53, forms the collision surface 53a. The collision surface 53a of the hole 53, has an equal purpose as the collision surfaces 3a, 3b, 4a, 4b of the blades 3, 4 described above. Hence, for the invention it is possible to replace the conical rings 31, 32, 33 of the first embodiment (rings formed by tapered blades) with the conical rings 51 of the second embodiment (rings formed by a conical ring body comprising holes). For a person skilled in the art it is obvious that the holes 53, of each ring 51, may have various sizes and shapes. The holes may for example be round, oval or square shaped.

A benefit with the second embodiment is that long fibres is oriented and fibrillated better during passage through the hole 53.

A great benefit with the invention is that high amount of energy can be transformed into the fibres without any or

very minor fibre cutting. This will have several positive effects:

- Chemical can be absorbed into fibres via mechanical agitation very efficiently.
 - These chemicals include a large amount of different type of chemicals.
 - Anionic chemicals, such as anionic polymers such as CMC, A-PAM
 - Cationic chemicals, such as alum, H₂SO₄, HCl and NaOH.
 - Enzymes such as cellulases and hemicellulases.
 - Chemicals known to improve fibrillation for MFC production such as CMC, OBA, and C-PAM.
- "Activate fibres", changing internal structure of the fibres
 - Including strength of individual fibres
 - Including internal pore structure
- Changing surface of the fibres
 - Including open surface and surface fibrillation.
- The device can be used to mix enzymes and thereafter grind the enzyme-treated fibres.

The design of the blades 3, 4 that forms the conical rings 31-33, 41-43 (first embodiment) and the design of the conical ring body 52 that forms the ring 51 (second

embodiment), gives some major benefit in comparison to the known prior art when defibrillating fibres. In the dynamic loading situation it is not possible to know the preferred gap L between the rings due to dynamic changes in the structure of the equipment. The gap L is known to be different from a static situation and will change even more when fibres are present in the flow. For this reason the mechanical arrangement, in accordance with the invention, is done so, that the gap L can be decreased during loading situation. The dynamic gap L can also be smaller than the static loading of zero. The distance of the gap L can be controlled for example via energy or vibration measurements.

In the foregoing, the invention has been described on the basis of two specific embodiments. It is appreciated, however, that other embodiments and variants are possible within the scope of the following claims, for example:

A person skilled in the art realises that instead of moving the upper, second rotor 2 in an axial direction to adjust the gap L, it would, of course, also be possible to move the lower, first rotor 1 in order to adjust the gap L.

The number of concentric conical rings in the device do not necessary need to be six (i.e. three rings 31, 32, 33, 51 on the first rotor 1 and three rings 41, 42, 43, 51 on the second rotor). In theory, the number of rings could be from two rings (one ring on each rotor) up to a finite number of rings.

The inclination of the conical rings 31-33, 41-43, 52, i.e. the inclination from the axis of the rotors of the rings circumferential inclining surfaces does not necessary need be in the interval 3-7°. In theory the inclination may be anything from 1-45°.

The rotors of the device do not necessarily need to rotate at rates from 800 up to 3000 rpm. In theory, the rate can be anything from 400 up to 6000 rpm.

C L A I M S

1. Device for defibrating fibre-containing material to produce microfibrillated cellulose, **characterized in that** the device comprising:

- a) a rotatably-mounted first rotor (1) equipped with collision surfaces (3a, 3b; 53a) mounted thereon in at least one, concentrically arranged, conical ring (31, 32, 33; 51), and adapted to rotate in a first direction;
- b) a rotatably-mounted second rotor (2) equipped with collision surfaces (4a, 4b; 53a) mounted thereon in at least one, concentrically arranged, conical ring (41, 42, 43; 51), and adapted to rotate in a second direction, which direction is opposite to the first direction of the first rotor (1), said second rotor (2) is in cooperating relationship with the first rotor such that the collision surfaces (3a, 3b) of said first rotor (1) and the collision surfaces (4a, 4b; 53a) of said second rotor and are interspersed; and
- c) a housing (5) comprising said first and second rotors, and having feed inlet (6) and a feed outlet (7), said inlet is in communication with a centre of said first and second rotors and being sufficiently large that fibre-containing material (8) may be fed freely there through; said outlet (7) is in communication with a periphery of an outermost ring.

2. Device according to claim 1, **characterized in that** the conical ring (31, 32, 33; 41, 42, 43; 51) has a cross-section which is wedge-shaped.

3. Device according any of claims 1-2, **characterized in that** each conical ring (31, 32, 33; 51) of the first rotor 1 comprising a circumferential, inner surface (31a, 32a, 33a; 51a), which faces against the rotation axis of the first rotor (1), and a circumferential, outer surface (31b, 32b, 33b; 51b), which faces away from the inner surface (31a, 32a, 33a; 51a).

4. Device according any of claims 1-3, **characterized in that** each conical ring (41, 42, 43; 51) of the second rotor 2 comprising a circumferential, inner surface (41a, 42a, 43a; 51a), which faces against the rotation axis of the second rotor (2), and a circumferential, outer surface (41b, 42b, 43b; 51b), which faces away from the inner surface (41a, 42a, 43a; 51a).

5. Device according to any of claims 3-4, **characterized in that** at least one of the inner surface (31a, 32a, 33a; 41a, 42a, 43a; 51a) and the outer surface (31b, 32b, 33b; 41b, 42b, 43b; 51a) inclines with respect to the rotation axis of the rotors (1, 2).

6. Device according to claim 5, **characterized in that** the inclining surface inclines 1-45° relative to the rotation axis of the rotors (1, 2).

7. Device according to claim 5, **characterized in that** the inclining surface inclines 3-7° relative to the rotation axis of the rotors (1, 2).

8. Device according to any of claims 1-7, **characterized in that** the gap (L) between a conical ring (31, 32, 33; 51) of the first rotor (1) and an immediate conical ring (41, 42, 43,; 51) of the second rotor (2), may be changed by moving one of the rotors (1, 2) in an axial direction.

9. Device according to claims 8, **characterized in that** the gap (L) is adjustable from 0.01 mm to 2 mm.

10. Device according to claims 8, **characterized in that** the gap (L) is adjustable from 0.1 mm to 1 mm.

11. Device according to claims 10, **characterized in that** a movement of one of the rotors (1, 2) of 0-10 mm results in that the gap (L) is adjustable from 0.1 mm to 1 mm.

12. Device according to any of the claims 1-11, **characterized in that** the conical ring (31, 32, 33) of the first rotor is formed by blades (3) having a tapered collision surface (3a, 3b) and that the conical ring (41, 42, 43) of the second rotor (2) is formed by blades (4) having a tapered collision surface (4a, 4b).

13. Device according to any of claims 1-11, **characterized in that** the conical ring (51) of the first and second rotors (1, 2) are formed by a conical ring body (52) having radial holes (53), wherein the inner surface of the holes (53) forming the collision surface (53a).

14. Device according to claim 13, **characterized in that** the holes are round.

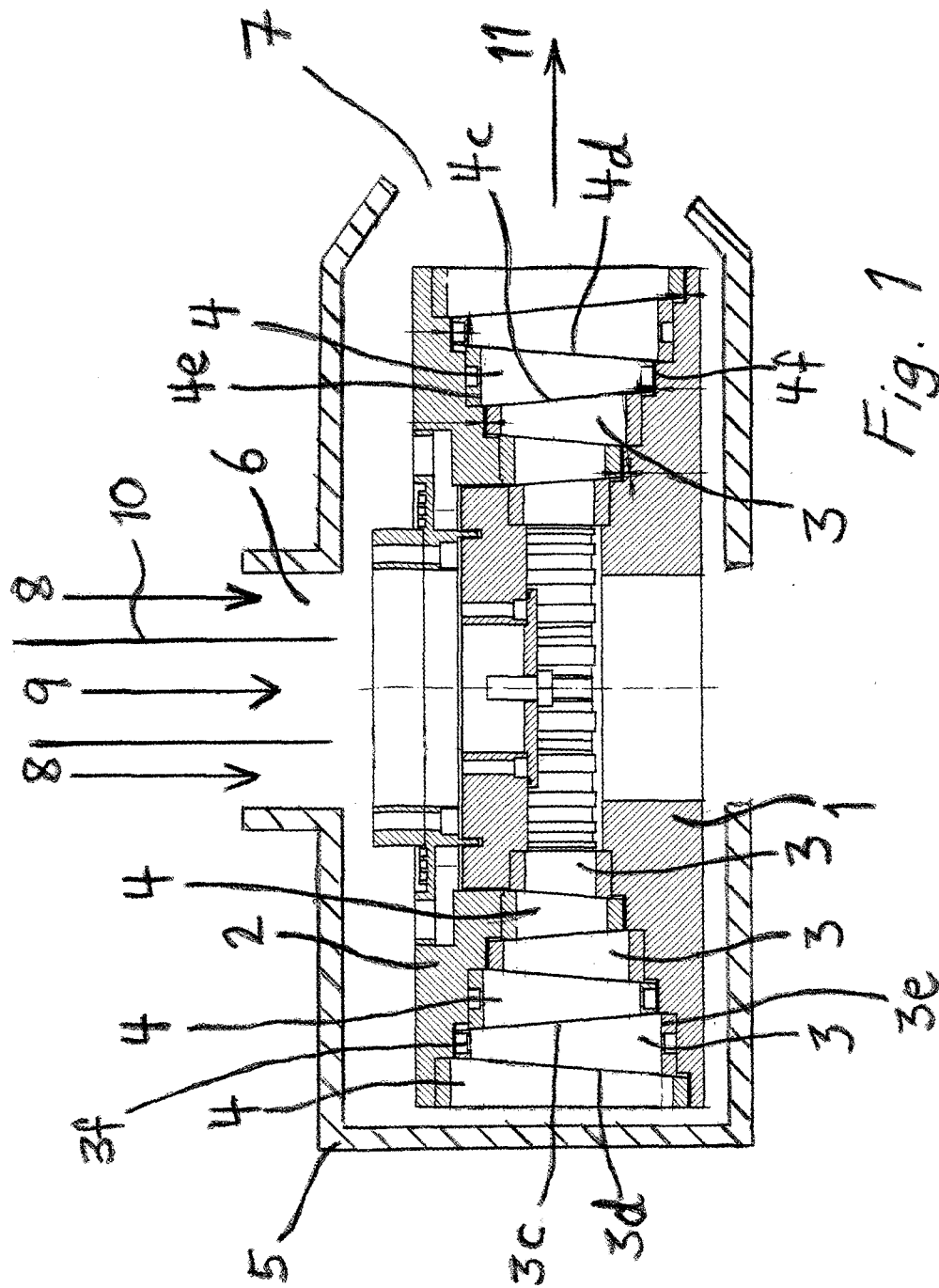
15. Device according to claim 13, **characterized in that** the holes are square formed.

16. A method for defibrating fibre-containing material to produce microfibrillated cellulose, **characterized in that** the method comprising the steps of:

- i) providing a defibrating device comprising
 - a) a rotatably-mounted first rotor (1) equipped with collision surfaces (3a, 3b; 53a) mounted thereon in at least one concentric, conical ring (31, 32, 33; 51), and adapted to rotate in a first direction;
 - b) a rotatably-mounted second rotor (2) equipped with collision surfaces (4a, 4b; 53a) mounted thereon in at least one concentric, conical ring (41, 42, 43), and adapted to rotate in a second direction which is opposite to the first direction of the first rotor (1), said second rotor (2) is in cooperating relationship with the said first rotor such that the collision surfaces (3a, 3b) of said first rotor (1) and the collision surfaces (4a, 4b) of said second rotor and are interspersed; and
 - c) a housing (5) comprising said first and second rotors, and having feed inlet (6) and a feed outlet (7), said inlet is in communication with a centre of said first and second rotors and being sufficiently large that fibre-containing material (8) may be fed freely there through; said outlet (7) is in communication with a periphery of an outermost ring;
- ii) feeding said fibre containing material (8) into said device such that the fibre-containing material (8) is

made to flow with the aid of air or liquid to said first and second rotors (1, 2), and then through said collision surfaces to said outlet (7) to produce microfibrillated cellulose (11); and

iii) collecting said microfibrillated cellulose (11) from said outlet (7).



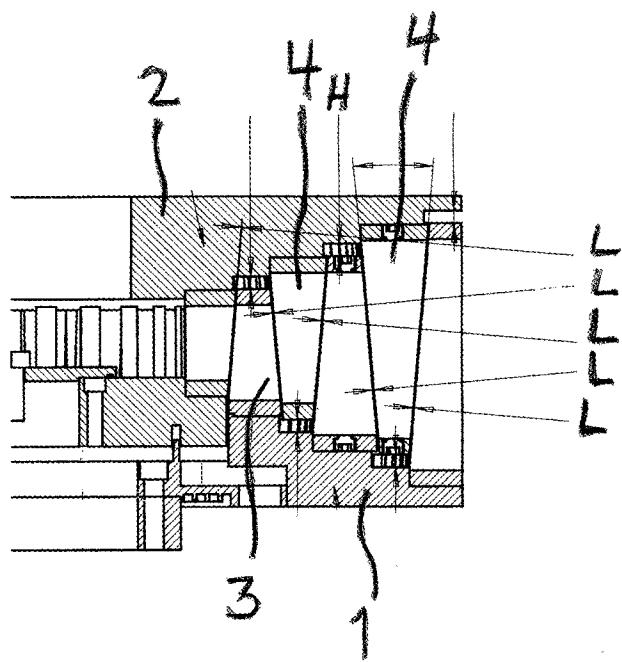
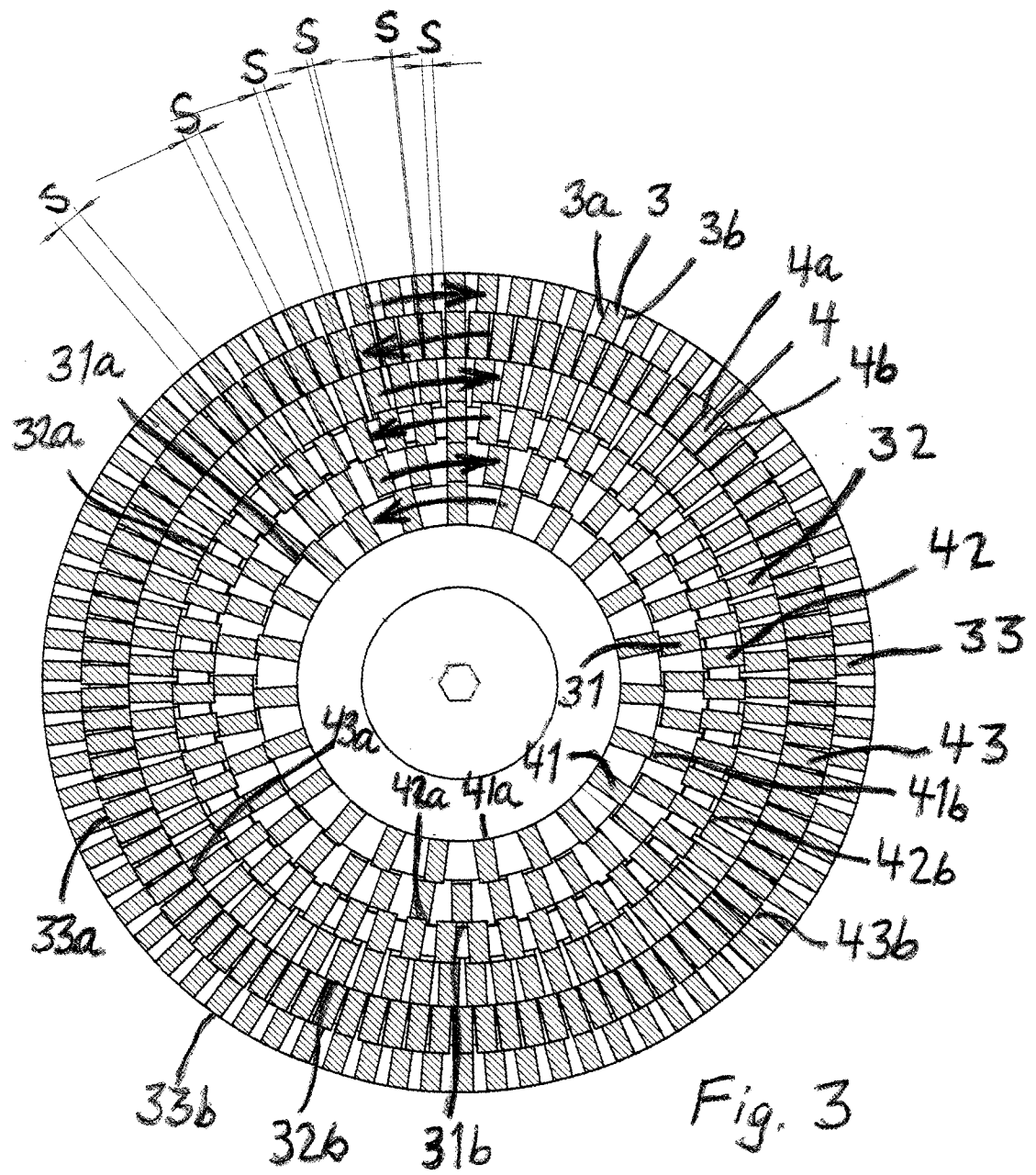
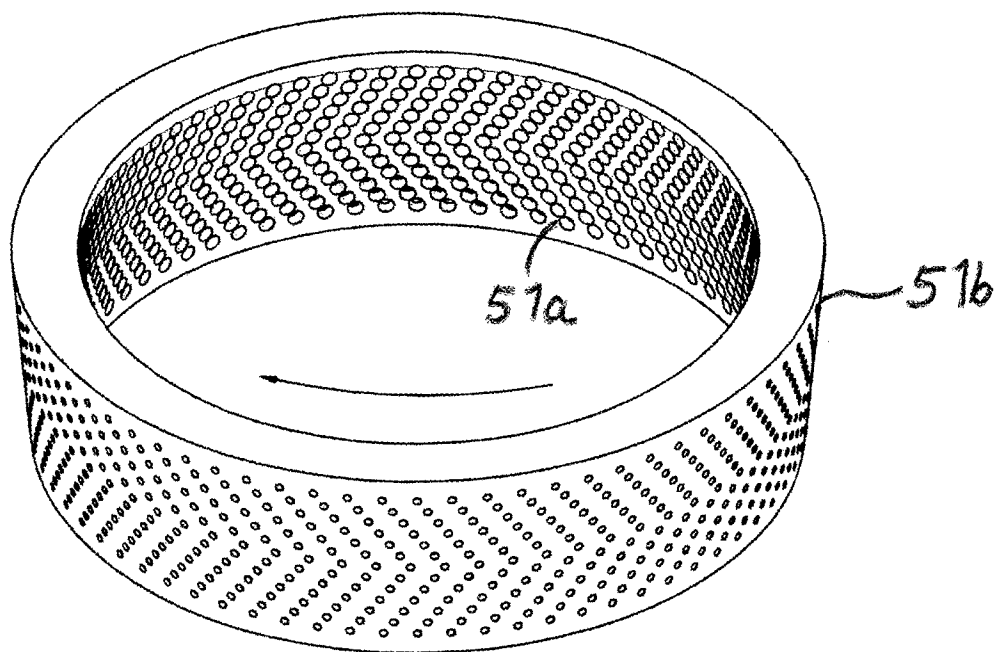
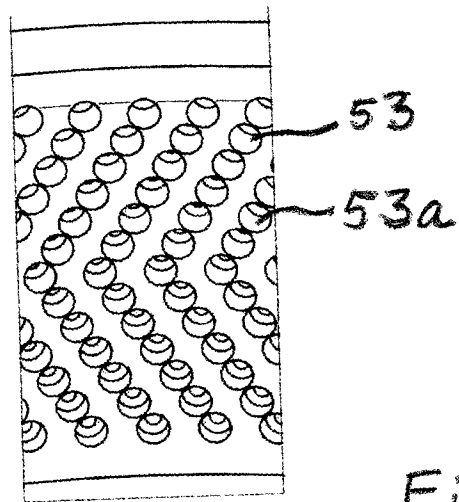


Fig. 2





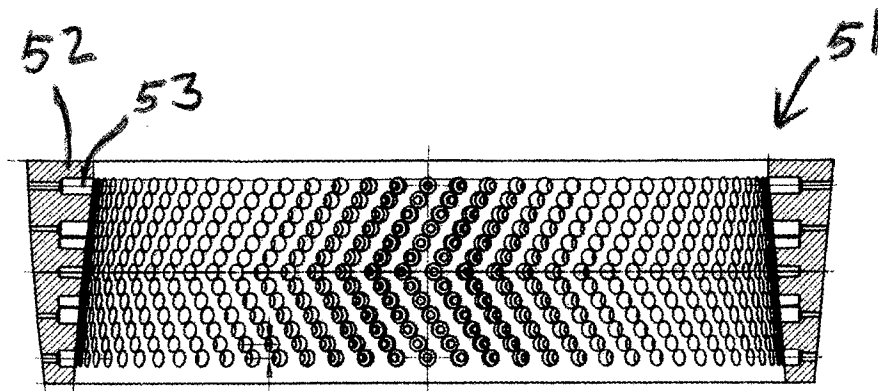


Fig. 5

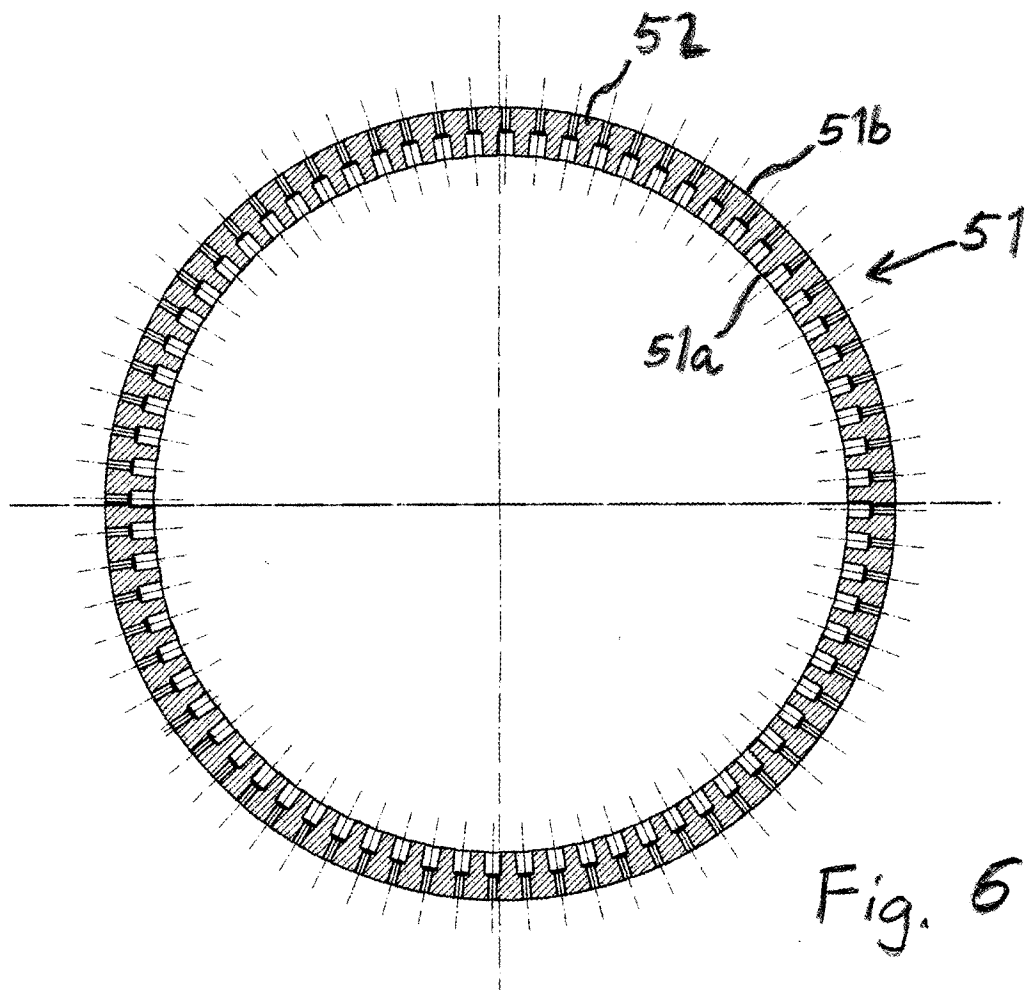


Fig. 6

Continuation of: Box No. III

collision surface in the first embodiment. ("Tapered collision surface" incorporates a geometric figure in the form of a truncated cone or a truncated pyramid or a wedge or similar geometric figures according to lines 16-19 on page 8 in the description.)

2: Claims 13-14 directed to a device and method for defibrating fibre-containing material to produce microfibrillated cellulose with a device comprising two counter-rotating rotors equipped with collision surfaces mounted thereon in at least on concentrically arranged conical ring. These claims relates to the second embodiment of the invention in which the conical rings are formed by conical ring bodies having radial holes, thereby long fibres are better oriented and fibrillated.

The present application has been considered to contain 2 inventions which are not linked such that they form a single general inventive concept, as required by Rule 13 PCT for the following reasons:

The single general concept of the present application is the teaching that the device for defibrating fibre-containing material should be equipped with conical rings in order to produce high length/thickness ratio micro fibrils and to break down fibril bundles due to an increased load between the rotating parts resulting in higher shear forces.

Document D1 discloses a pair of co-operating refining elements (10, 11) intended for a refiner for the working of lignocellulosic fiber material in a refining gap between two opposed refining discs rotating relative to each other. The refining elements are provided with pins which are arranged in several rows in the circumferential direction, see the abstract and figures 1-3. The pins can have a conical shape, see line 3 on page 3.

It is implicit that the refiner mentioned in D1 has an inlet, an outlet and a housing in accordance with part c) of claim 1.

It is not stated in D1 that microfibrillated cellulose is produced. However, the device described in D1 is considered to be suitable for producing MFC since there is no difference between the device described in claim 1 and the device described in D1.

Consequently, the subject matter of claim 1 is previously known and therefore lacks novelty.

Thus, the single general concept is known and cannot be considered as a single general inventive concept in the sense of Rule 13.1 PCT.

No other features can be distinguished which can be considered as the same or corresponding special technical features in the sense of Rule 13.2 PCT.

Thus, the application lacks unity of invention.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/IB2013/058633

A. CLASSIFICATION OF SUBJECT MATTER

IPC: see extra sheet

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC: B02C, C08L, D21B, D21D, D21H

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

SE, DK, FI, NO classes as above

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

EPO-Internal, PAJ, WPI data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 9926725 A1 (SUNDS DEFIBRATOR IND AB ET AL), 3 June 1999 (1999-06-03); abstract; page 3, line 1 - line 3; figures 1-3 --	1-7
Y	WO 2010092239 A1 (UPM KYMMENE OYJ ET AL), 19 August 2010 (2010-08-19); abstract; page 7, line 23 - line 28 --	1-12, 16
Y	EP 2243879 A2 (ANDRITZ INC), 27 October 2010 (2010-10-27); paragraphs [0010], [0024]; figure 4 --	1-13, 15-16
Y	SE 436139 B (OBBOLA LINERBOARD AB), 5 March 1983 (1983-03-05); page 1, line 9 - line 32; figure 1 --	1-12, 16



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents:

“A” document defining the general state of the art which is not considered to be of particular relevance

“E” earlier application or patent but published on or after the international filing date

“L” document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

“O” document referring to an oral disclosure, use, exhibition or other means

“P” document published prior to the international filing date but later than the priority date claimed

“T” later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

“X” document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

“Y” document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

“&” document member of the same patent family

Date of the actual completion of the international search

21-02-2014

Date of mailing of the international search report

24-02-2014

Name and mailing address of the ISA/SE

Patent- och registreringsverket
Box 5055
S-102 42 STOCKHOLM
Facsimile No. + 46 8 666 02 86

Authorized officer

Karin Bengtsson

Telephone No. + 46 8 782 25 00

INTERNATIONAL SEARCH REPORT

 International application No.
 PCT/IB2013/058633

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 2009097963 A2 (VOITH PATENT GMBH ET AL), 13 August 2009 (2009-08-13); abstract; page 5; figure 5; claim 2 --	13, 15
Y	US 5904308 A1 (SCHNELL HANS), 18 May 1999 (1999-05-18); abstract; column 6, line 27 - line 41; figure 3a --	13, 15
A	US 4366929 A (DE LOS SANTOS FEDERICO), 4 January 1983 (1983-01-04); whole document --	13-16
P, Y	WO 2013072559 A1 (UPM KYMMENE CORP), 23 May 2013 (2013-05-23); whole document --	1-12, 16
A	GB 891152 A (PETER WILLEMS), 14 March 1962 (1962-03-14); abstract; figures 3, 6; claim 10 --	1-12, 16
A	US 4269362 A1 (BERGGREN LEIF A), 26 May 1981 (1981-05-26); column 1, line 21 - line 43; column 3, line 1 - line 7; figures 4, 8 --	1-12, 16
A	WO 9954045 A1 (MEGATREX OY ET AL), 28 October 1999 (1999-10-28); whole document --	1-12, 16
A	EP 1538257 A1 (JAPAN ABSORBENT TECH INST -(B1) DSG INTERNAT LTD [CN]), 8 June 2005 (2005-06-08); abstract; paragraph [0026] -- -----	1-12, 16

Continuation of: second sheet

International Patent Classification (IPC)

B02C 13/22 (2006.01)

B02C 13/20 (2006.01)

D21B 1/14 (2006.01)

D21D 1/20 (2006.01)

INTERNATIONAL SEARCH REPORT

International application No.
PCT/IB2013/058633

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1: Claims 1-12 and 16 directed to a device and method for defibrating fibre-containing material to produce microfibrillated cellulose with a device comprising two counter-rotating rotors equipped with collision surfaces mounted thereon in at least on concentrically arranged conical ring. The conical shape of the ring enables to produce high length/thickness ratio micro fibrils and to break down fibril bundles due to an increased load between the rotating parts resulting in higher shear forces. The application discloses two embodiments of the invention. The conical rings are formed by blades having a tapered

.../...

1. ☒ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.
2. ☐ As all searchable claims could be searched without effort justifying additional fees, this Authority did not invite payment of additional fees.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.
- ☐ The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.
- ☒ No protest accompanied the payment of additional search fees.

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/IB2013/058633

WO	9926725 A1	03/06/1999	AR	017671 A1	12/09/2001
			AT	386593 T	15/03/2008
			AU	728371 B2	11/01/2001
			AU	1358499 A	15/06/1999
			BR	9815040 A	03/10/2000
			CA	2311532 C	31/10/2006
			DE	69839154 T2	12/06/2008
			EP	1034041 B1	20/02/2008
			JP	2001523777 A	27/11/2001
			NO	321836 B1	10/07/2006
			NO	20002650 A	07/07/2000
			NZ	504779 A	28/03/2002
			SE	9704350 L	26/05/1999
			SE	510948 C2	12/07/1999
			US	6499682 B1	31/12/2002
			ZA	9810725 A	31/05/1999
WO	2010092239 A1	19/08/2010	CA	2750082 A1	19/08/2010
			CN	102317542 A	11/01/2012
			EP	2396470 A4	25/07/2012
			FI	20095140 A0	13/02/2009
			JP	2012518050 A	09/08/2012
			KR	20110116054 A	24/10/2011
			RU	2011136555 A	20/03/2013
			US	20120043039 A1	23/02/2012
EP	2243879 A2	27/10/2010	AU	2010201349 A1	11/11/2010
			BR	PI1001319 A2	28/01/2014
			CA	2699801 A1	23/10/2010
			CN	101871175 A	27/10/2010
			JP	2010255172 A	11/11/2010
			NZ	584565 A	28/10/2011
			RU	2010116047 A	27/10/2011
			US	8342437 B2	01/01/2013
			US	20100269991 A1	28/10/2010
SE	436139 B	05/03/1983	SE	8105273 L	05/03/1983
WO	2009097963 A2	13/08/2009	DE	102008007888 A1	13/08/2009
US	5904308 A1	18/05/1999	AT	199577 T	15/03/2001
			CA	2189901 A1	11/05/1997
			DE	19541891 A1	22/05/1997
			EP	0773316 A1	14/05/1997
US	4366929 A	04/01/1983	NONE		
WO	2013072559 A1	23/05/2013	FI	20116130 A	15/05/2013
GB	891152 A	14/03/1962	FR	1207479 A	17/02/1960

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/IB2013/058633

US	4269362 A1	26/05/1981	DE	2931275 A1	28/02/1980
			GB	2031299 A	23/04/1980
			JP	55026291 A	25/02/1980
			SE	7808423 A	08/02/1980
			SE	435942 C	07/02/1985
WO	9954045 A1	28/10/1999	AU	3423199 A	08/11/1999
			CA	2328205 A1	28/10/1999
			EP	1087840 A1	04/04/2001
EP	1538257 A1	08/06/2005	AT	524601 T	15/09/2011
			AU	2003281587 A1	09/02/2004
			BR	0305572 A	28/09/2004
			CN	1665984 A	07/09/2005
			CN	1325725 C	11/07/2007
			ES	2370151 T3	13/12/2011
			JP	4305766 B2	29/07/2009
			KR	20050021512 A	07/03/2005
			KR	100985399 B1	06/10/2010
			MX	PA04012799 A	31/03/2005
			US	7381294 B2	03/06/2008
			US	20050194477 A1	08/09/2005
			WO	2004009902 A1	29/01/2004