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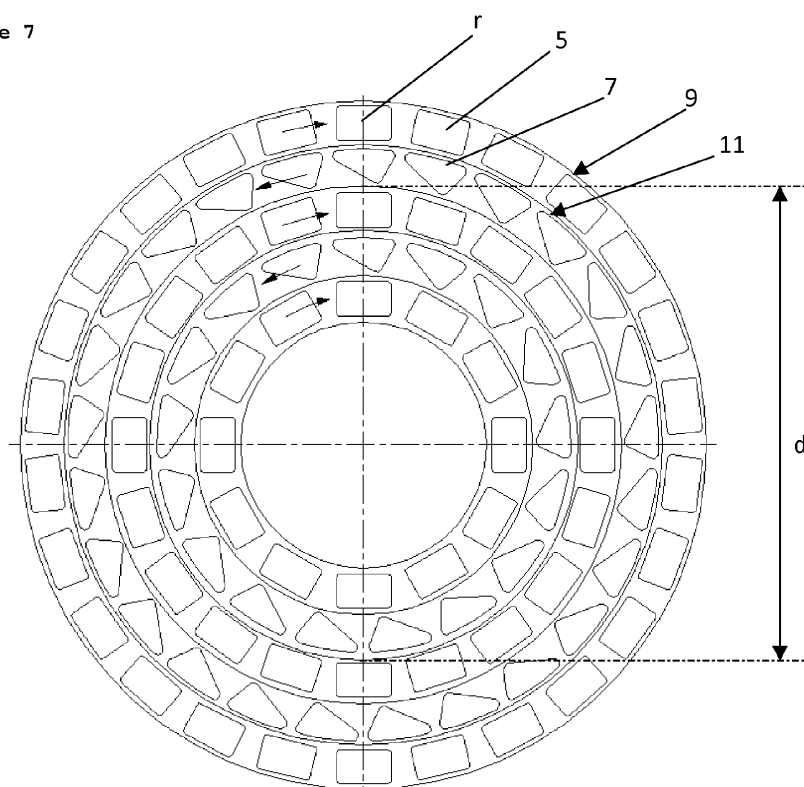
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(54) **Modified micronization device and use thereof**

(57) Device (1) for the micronization of substances comprising two rotors (3) driven in a direction (ω) opposite to each other, each rotor (3) carrying at least one row of multiple hitting elements (5, 7) forming a ring (9, 11), said rings (9, 11) being arranged concentrically in, the rings (9, 11) of the different rotors (3) engaging alternately with one another, the hitting elements (5, 7) being suitably arranged to provide transportation of the substance from

inside the rings (3) to the outside by effecting a suitable airflow, at least two, directly adjoining rings (9, 11) carrying hitting elements (5, 7) with different foot print wherein at least a first ring (9) is equipped with trapezoidal hitting elements (5) with trapezoidal foot print and at least one other ring (11), directly adjoining the first ring (9) is equipped with triangular hitting elements (7) with triangular foot print.

Figure 7



Description**Technical field**

5 [0001] The present invention relates to a device for micronization according to the preamble of claim 1.

Prior art

10 [0002] New physical properties, bioavailability and bio-efficacy of solid substances are often intrinsically related to the primary particle size and proportion of amorphous surface area. Particle size reduction by top-down processing i.e. milling is one of various strategies for improving solubility and reactive characteristics of poorly water-soluble ingredients. Thus, many attempts have been conducted to obtain good bioavailability achieved by creating an amorphous product.

[0003] Micronization is a highly effective kind of milling which allows a direct production of very fine particles, typically 5-50 μm , from relatively coarse-grained starting material, of particle size, e.g. 0,1-1 mm.

15 [0004] Technological operation of micronization is widely used in production of active substances and excipients for pharmaceutical, cosmetic, and agrochemical industries, in chemical industry (e.g. fillers, pigments), and in many other fields.

[0005] Micronization can be accomplished by a prolonged milling in various classical mills (ball mill, jet mill, disintegrators etc.), wherein the milling process is based mainly on collisions of particles between themselves or with hitting elements of a milling device

20 [0006] Disintegrators of various types are known from the prior art. Examples of such disintegrators are for example disclosed in US 4,406,409 A1 and HR 990 263 A2. Those disintegrators are principally based on the concept of two high-speed opposite rotating discs. The discs bear particular hitting elements, blades, which partially collide with particles directly, but mostly targeted to create an efficient airflow by mimicking turbines, driving particles into mutual collisions.

25 In literature, there are described devices with various shaped blades on the discs:

1. round-shaped blades;
2. blades in the shape of elongated plates; and
3. blades in the shape of slightly curved plates;

30 without or with additional particular mechanic details (e.g. indented hitting surface) which eventually improve a course of micronization process due affecting airflow.

[0007] Examples of such devices are shown in Figures 1 and 2;

35 **Short description of the invention**

[0008] The present invention provides an improved device for micronization based on the concept of desintegrator with two opposite rotating discs, known also as disintegration. The present device modified for significant improvement in micronization process. The main improvement of the present invention is a modification of particle hitting elements or blades, in positioning and shape. Along each of disc in two or several layers results in significant improvement of micronization. Discs are situated within the micronizer in a way that the layers of blades of first and second disc enter into each other's.

40 [0009] Particles of the material being micronized, carried by centrifugal force from the center of the device, pass through several layers of cubical and triangular shaped blades in the way to collide repeatedly with other particles and between rows of blades. Additionally, triangular blades, oppose to centrifugal particle flow by forcing them into repeated collisions, resulting in improved reduction of their size.

Detailed description of the invention

50 [0010] The present invention provides a modified device for an improved milling process, which is based on the concept of a disintegrator.

[0011] A device for micronization of substances according to the present invention comprises two rotors driven in a direction opposite to each other, each rotor carrying at least one row of multiple hitting elements forming a ring, said rings being arranged concentrically the rings of the different rotors engaging alternately with one another, the hitting elements being suitably arranged to provide transportation of the substance from inside the rings to the outside by effecting a suitable airflow, at least two directly adjoining rings carrying hitting elements with different foot print wherein at least a first ring is equipped with trapezoidal hitting elements with trapezoidal foot print and at least one other ring directly adjoining the first ring is equipped with triangular hitting elements with triangular foot print.

[0012] Present research showed that the footprint of the hitting elements has significant influence on the results of micronization. It was found out that with reduced particle size the effect of inter particle collisions has less influence on the micronization result and thus conventional turbine-like footprints of the hitting elements get less effective. The hitting elements according to the present invention provide a suitable air flow to effect inter particle collisions but also provide improved collisions between the particles and the hitting elements as well as permanent milling between the hitting elements of directly adjoining rings.

[0013] In another embodiment of the device one side of the triangular hitting elements and/or one of the parallel sides of the trapezoidal hitting elements is perpendicularly oriented to a radius crossing the hitting element.

[0014] Due to the arrangement of the hitting elements perpendicular to a radius crossing the respective hitting elements are suitably arranged to provide parallel arranged surfaces for milling of the substance between adjoining rings.

[0015] In a further embodiment of the device the trapezoidal hitting elements are of rectangular foot print.

[0016] With a rectangular footprint the trapezoidal hitting elements may be of generally cubical shape. Compared to other shapes cubical hitting elements are easier to produce and thus cheaper in production.

[0017] It was unexpectedly found that the shape of the hitting elements of one rotating disc in a form of cubes and other in a form of tightly positioned triangular shapes (Figure 5), unexpectedly results in significantly improved efficacy of micronization.

[0018] In another embodiment of the present device the triangular hitting elements are of basically right angled triangular foot print.

[0019] The right angled triangular footprint allows manufacturing of the triangular hitting elements with rectangular base elements that are divided diagonally. It is thus possible to use base elements with rectangular footprint to manufacture both, the rectangular hitting elements and the triangular hitting elements.

[0020] In a preferred embodiment the perpendicularly oriented side of the triangular hitting elements is the longer cathetus. Preferably the longest cathetus is oriented to the outward circumference of its respective ring.

[0021] The longer cathetus of the triangular hitting elements is thus tangentially oriented and may serve for milling. The milling will take place between the tangentially oriented surface of the triangular hitting elements and surfaces of hitting elements of a circumferencing and directly adjoining ring. Furthermore the triangular hitting elements thus provide for sufficient air flow from the center of the rings to the outside.

[0022] In another embodiment the longest side of the triangular hitting elements is oriented in front in direction of rotation of the respective rotor.

[0023] Particularly in combination with right angled triangular hitting elements with the longest cathetus oriented to the outward circumference of the respective ring this embodiment directs the particles in backward loops, i.e. backward in substantially radial direction, thus resulting in extended time within the device and thus more inter particle collisions.

[0024] In another embodiment the hitting elements of directly adjoining rings are suitably arranged to provide milling between the hitting elements of adjacent rings.

[0025] As with shrinking particle size inter particle collisions get less important for size reduction of the particles, permanent milling between the hitting elements of adjacent rings becomes more relevant for the disintegration. By arranging the hitting elements of directly adjoining rings in this manner disintegration results are further enhanced.

[0026] Each ring may carry a number of hitting elements equivalent to the diameter in cm of the respective ring.

[0027] The number of hitting elements per ring obtained by this rule provides an optimal ratio of hitting elements to spaces between the hitting elements and thus further enhances the efficacy of disintegration.

[0028] The hitting elements may be made of stainless steel or ceramics and/or coated with industrial diamond or ruby.

[0029] The above materials and/or coatings provide high durability of the hitting elements as well as high availability of the used materials.

[0030] To achieve the above-mentioned advantage in manufacture of the hitting elements out of identical base elements, the triangular hitting elements and the trapezoidal hitting elements may have at least two sides with coinciding length.

[0031] Thus not only rectangular but also trapezoidal elements can serve as base elements for both, trapezoidal and triangular hitting elements.

[0032] The present invention hereinafter will be described with reference to the attached drawings representing preferred embodiments.

Fig. 1 shows a perspective view of a disintegrator,

Fig. 2 shows a sectional view of the disintegrator of Fig. 1,

Fig. 3 shows a first embodiment of hitting elements according to the state of the art,

Fig. 4 shows a second embodiment of hitting elements according to the state of the art,

Figs. 5 or 6 show enlarged sections of Fig. 3 and Fig. 4,

Fig. 7 shows a sectional view of the rotors according to an embodiment of the present invention,

5 Fig. 8 shows an enlarged section of Fig. 7,

Fig. 9 - 11 show the hitting elements according to Figs. 7 and 8 in enlarged view.

[0033] Figure 1 shows a perspective view of a disintegrator 1.

10 **[0034]** The disintegrator 1 according to figure 1 is equipped with two electro motors 20 arranged opposite to each other and rotating in opposite direction ω . Each electro motor 20 is coupled directly or indirectly via a gearbox to a rotor 3 that is equipped with multiple concentrically arranged rings 9, 11 of hitting elements 5, 7 (hereinafter also called blades). The rings 9, 11 of hitting elements 5, 7 of the two rotors 3 alternately engage with each other thus adjacent rings 9, 11 of hitting elements 5, 7 rotating in opposite direction ω . The rotors 3 are encapsulated in a casing 21 that may be opened
15 according to the illustration in figure 1.

[0035] The whole disintegrator 1 is located on a mount 22, e.g. a base frame that carries the electro motors 20 and the oppositely rotating rotors 3.

[0036] The disintegrator 1 exhibits a filler 23 with a hopper 24. As can be seen from figure 2 the filler 23 directs the material for micronization to the center of the rotors 3. A sufficient airflow generated by the rotors 3 rotating in opposite
20 direction ω transports the material from the center of the rotors 3 to an outlet 25 at the circumference of the rotors 3 causing multiple inter particle and particle-hitting-element collisions. At the outlet 25 the micronized material exits the casing 21 of the disintegrator 1 and either may be collected or again fed into the disintegrator 1.

[0037] Figure 2 clearly shows how the rings 9, 11 of hitting elements 5, 7 of the rotors 3 alternatively engage one another thus adjacent rings 9, 11 of hitting elements rotating in opposite directions ω .

25 **[0038]** Figures 3 to 6 show different views of hitting elements according to the prior art.

[0039] The hitting elements depicted in figures 3 and 4 are of blade-like footprint that mainly generates an airflow transporting particles from the center of the rotors 3 to the circumference, thus generating inter particle collisions. However due to the pitch of the blades the time the particles stay within the disintegrator 1 is quite low.

[0040] Figures 5 and 6 show exemplary traces of articles from the center of the rotors 3 to the outer circumference where the particle exit the disintegrator 1.
30

[0041] The improved device 1 according to the present invention has the similar device construction like the disintegrators 1 from the prior art [J.Durek: Disintegrator and the method for the operation thereof, US4406409 A], [T. Lelas: Device for micronizing materials, HR990263 A2 (1999)] shown in figures 1 and 2.

[0042] An embodiment of a blade design (design of the hitting elements) according to the present invention is shown in figures 5 to 11. In this present embodiment a first ring 9 of trapezoidal blades carries hitting elements 5 with rectangular footprint and thus ob generally cubic shape. Another ring 11, directly adjoining the first ring 9 carries triangular hitting elements 7 with a footprint of a right angled triangle. These triangular hitting elements 7 are tightly positioned and of generally prismatic shape. The different footprints of the blades 5, 7 are shown in figure 5.
35

[0043] Figure 5 shows a cross-section in the plane of rotation of the hitting elements 5, 7 thus depicting the different footprints of the hitting elements 5, 7 on the different rotors 3.
40

[0044] In the present embodiment the rotor 3 rotating in clockwise manner carries tree rings 9 of trapezoidal hitting elements 5 with rectangular footprint. The other rotor 3 (in the depicted view rotating counterclockwise) carries two rings 11 of triangular hitting elements 7 with triangular footprint. The rings 9, 11 of hitting elements 5, 7 engage alternately thus on a ring 9 rotating in clockwise manner follows a ring 11 rotating in opposite direction, i.e. counterclockwise manner.

45 **[0045]** Due to the different footprints of the hitting elements 5, 7 and due to their arrangement the present embodiment provides an elongated time of the particles within the disintegrator 1 and thus a enlarged number of inter particle collisions, permanent milling between the hitting elements 5, 7 of adjacent rings 9, 11 and a enlarged number of collisions between particles and the hitting elements 5, 7.

[0046] The trace of a particle being micronized in a disintegrator 1 according to the present embodiment is depicted in figure 8. Due to the arrangement of the hitting elements 5, 7 the particles move in a loope-like manner thus resulting in a prolonged time within the disintegrator 1.
50

[0047] The footprint of the triangular hitting elements 7 and their arrangement on their rotor 3 is depicted in figures 9 to 11.

[0048] Figure 9 shows an enlarged cross-section of a triangular hitting element 7 according to the present embodiment. The hitting element 7 is of basically triangular footprint with rounded edges. As depicted in figure 9 the footprint is in the shape of a right angled triangle. The hypotenuse 15 of the right angled triangle is oriented tangentially to the outward circumference of the ring 11 built by the hitting elements 7 of it's respective ring. 11
55

[0049] The arrow in figure 9 indicates the direction of rotation ω of the respective hitting element 7, the longer cathetus 13 of the right enabled triangle thus being forward oriented.

[0050] According to figure 10 a center of the hypotenuse 15 of the triangular shaped footprint is perpendicularly cut by a radius r of the respective ring 11. The longer cathetus 13 thus is inclined relative to the radius r by an angle α of 60 degrees. Accordingly (due to the fact that the triangle is right angled) the other cathetus 14 is inclined relative to the radius r by an angle β of minus 30 degrees.

[0051] Figure 11 depicts two triangular hitting elements 7 arranged in a ring like manner. The hitting elements 7 are spaced from one another by a distance A of 15 - 100% of the length of the hitting elements 7.

The longest side of the rectangular hitting elements 5 for example may be 30 mm. Accordingly the longer cathetus 13 of the triangular hitting elements 7 may be of the same length.

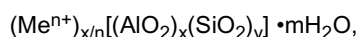
[0052] The course of the micronization process in the device 1 according to the present embodiment is actually the same as in the micronizer with hitting elements as disclosed in the prior art (shown in Figures 3 and 4). Material being micronized is added in the hopper 24 of the filler 23. The latter brings the material into a central part of rotating discs 3 beside their axes. Due to a strong centrifugal force, particles of material being micronized pass through two or several layers of rotating hitting elements 5, 7 (blades) of opposite discs rotating at high speed (about 5000 - 8000 rounds per minute) in opposite direction.

[0053] The influence of the shape of the blades on the efficiency of the micronization has been studied by the use of the micronization device shown in Figures 1 and 2 from the prior art of the following technical characteristics:

- (i) diameter of discs ($\varnothing$) of micronizer was 370 mm;
- (ii) number of layers of hitting elements (blades) on the discs was 3 on primary and 2 on secondary disc;
- (iii) number of blades on each of wreaths of discs was 20/16 (outer/inner wreath) from one side; and 18/16 (outer/inner wreath) from the other side of the micronizer;
- (iv) the rotation velocity was 10000 rpm;

[0054] The micronization device was equipped with two identical 20 kW power electro-motors that work at 220 V and 50 Hz.

[0055] Natural zeolite clinoptilolite, of the general formula



wherein Me = Na, K, Mg, Ca, Fe, Zn, Mn, Cr, was selected as a model substance of a hardness of 4 according to Mohs' scale. The starting material of an average particles size of 50-100 μm , was obtained from Zeocem a.s., Slovakia. The reason why zeolite is chosen is due to its NH_4^+ sorption and retention capacity. It has been already demonstrated that zeolite can have significant improvement in sorption capacity, if micronized below one μm .

[0056] 1 kg of each sample of same zeolite clinoptilolite was micronized by using the device according to the prior art and three different hitting elements as depicted in figures 5, 6 and 8. The disintegrators are characterized by the following data:

1. Plates formed according to US4406409 A (Figure 5; Micronization-1);
2. Slightly curved and indented plates according to HR990263 A2 (Figure 6; Micronization-2); and
3. The hitting elements according to the present invention (Figure 8; Micronization-3)

[0057] Prepared samples of micronized zeolite mineral were analyzed for particles size using a Malvern MasterSizer 2000 instrument and for NH_4^+ ion sorption capacity with aqueous NH_4Cl solution measured with an ion chromatograph DX-120 Dionex from the United States. The results of Micronization-1, Micronization-2 and Micronization-3 are shown in Table 1 and 2.

[0058] Table 1 shows the influence of the various shapes of hitting elements (blades) from different micronizing discs (Figures 5, 6 and 8) on efficacy of micronization of zeolite clinoptilolite of starting particle size 50-100 μm . The goal was to achieve larger amount of submicron particles.

Table 1

	MICRONIZATION-1	MICRONIZATION-2	MICRONIZATION-3
Particles size (μm)	Volume (%) under	Volume (%) under	Volume (%) under
0.105	0.00	0.00	0.00
0.120	0.00	0.00	0.00
0.138	0.00	0.00	0.00

(continued)

	MICRONIZATION-1	MICRONIZATION-2	MICRONIZATION-3
Particles size (μm)	Volume (%) under	Volume (%) under	Volume (%) under
0.158	0.00	0.00	0.00
0.182	0.00	0.00	0.01
0.209	0.00	0.02	0.08
0.240	0.00	0.32	0.47
0.275	0.00	0.62	1.44
0.316	0.02	1.04	2.43
0.363	0.07	1.62	3.79
0.417	0.15	2.16	5.54
0.479	0.29	3.18	7.71
0.550	0.79	4.11	9.84
0.631	1.12	5.36	12.44
0.724	1.89	6.96	15.03
0.832	2.44	8.41	18.13
0.955	3.66	10.02	21.72
1.096	5.12	13.16	26.80

[0059] Table 2 shows the influence of the various shapes of hitting elements from different micronizing discs (Figures 5, 6 and 8) on particle specific surface area of zeolite clinoptilolite of starting particle specific surface area $0.9 \text{ m}^2/\text{g}$.

Table 2

	MICRONIZATION 1	MICRONIZATION 2	MICRONIZATION 3
Specific Surface Area m^2/g	1.9	3.2	6.7

[0060] Table 3 shows the influence of the various shapes of hitting elements from different micronizing discs (Figures 5, 6 and 8) on NH_4^+ sorption of zeolite clinoptilolite of starting capacity 0.23 mmol/g

Table 3

	MICRONIZATION-1	MICRONIZATION-2	MICRONIZATION-3
NH_4^+ Sorption Capacity mmol/g	0.48	0.78	0.94

[0061] The results show that the process of MICRONIZATION-3 (Figure 8) with cube shaped hitting elements on one disc and tightly positioned triangular hitting elements on the other disc is superior to the other processes. It is also shown that MICRONIZATION-3 particles have much larger specific surface and capacity of ion sorption than those processed by other micronization types.

[0062] Further experiments show how repeated micronization (5 times) in the same device, but with differently designed hitting elements affects particle size and crystalline surface deformation in order to enhance an amorphous portion of the surface and consequently improve solubility of poorly soluble pharmaceutically active ingredients.

[0063] 1 kg of each sample of ursolic acid (98% purity, Sigma Aldrich) of an average particle size of $30 \mu\text{m}$ was micronized five times in devices having different hitting elements. The discs were cooled through a feeder opening with liquid nitrogen spraying to avoid overheating of the heat-sensitive substance. The device used was according to the prior art while the hitting elements used are shown in Figures 5, 6 and 8.

[0064] The micronized samples were analyzed for particle size (Malvern MasterSizer 2000), while the extent of crystalline disorder of particles was quantified with isothermal calorimetry (IC TAM 3, TA instruments, USA). Data was recorded with proprietary software Digitam 4.2.

[0065] Table 4 shows the influence of the various shapes of hitting elements of different micronizing discs (Figures 5, 6 and 8) on ursolic acid particle size after five repeated micronization procedures.

Table 4

	MICRONIZATION-1	MICRONIZATION-2	MICRONIZATION-3
Particles size (μm)	Volume (%) under	Volume (%) under	Volume (%) under
0.105	0.00	0.00	0.00
0.120	0.00	0.00	0.00
0.138	0.00	0.00	0.00
0.158	0.00	0.00	0.28
0.182	0.00	0.00	1.38
0.209	0.00	0.12	2.73
0.240	0.00	0.46	3.69
0.275	0.01	1.19	5.39
0.316	0.06	1.97	8.42
0.363	0.23	2.83	11.76
0.417	0.50	4.09	14.01
0.479	0.87	5.36	19.32
0.550	1.12	7.01	25.19
0.631	2.02	9.13	31.12
0.724	3.29	11.51	36.14
0.832	4.78	13.61	42.72
0.955	6.12	16.29	50.02
1.096	8.44	19.17	59.80

[0066] Table 5 shows the influence of the various shapes of hitting elements from different micronizing discs (Figures 3, 4 and 5) on amorphous content of ursolic acid after five repeated micronization procedures.

Table 5

	MICRONIZATION-1	MICRONIZATION-2	MICRONIZATION-3
Amorphous Content (% w/w)	3.68	6.12	19.23

[0067] The tables above show that a reduction of particle size is not proportional to the number of repeated micronization processes; this is particularly notable in MICRONIZATION-1 and MICRONIZATION-2. It appears the smaller the particles are, the air friction affects them more, resulting in weaker collisions. This is predominant in discs having hitting elements with "turbine-like" design according to the state of the art devices. The hitting elements according to Figure 7 show superior results, efficiently reducing particle size and inducing significant proportion of amorphous surface content.

[0068] Generally, it seems that major reason for effective and useful micronization from the design described in figures 7-11 comes from the fact that the majority of particles is pushed back into compact repeated collisions (Figure 8) by the layers of tightly position hitting elements having extruded triangle shape as shown in figures 7-11. This way, particles return and collide repeatedly with other particles or hitting elements in the previous layer, while only a small amount of particles passes onto next flow layer. The flow of particles from this invention is shown in Figure 8. Such flow results in superior micronization compared to previous disintegrator devices, which relied mostly on turbine-like micronization effect. Particularly, airflow driven particle collisions, dominating in previous disintegrators are not sufficient for micronization below one (1) μm . Even after several repeated micronizing processes in prior art devices, particles do not reduce in size significantly, because airflow driven collisions become less important due to increased air friction and reduced kinetic energy of the particles. The smaller the particles become, the more air friction reduces impact velocity. The

present invention demonstrates the importance of tight contact with the blades, which is important for adequate size reduction. Airflow based particle movements is used as vehicle to enable particle exit from the device by centrifugal force. To our best knowledge, it seems that the present invention is accomplishing superior micronization through four (4) major factors a) minimal air friction, b) compact particle inter-collisions, c) collision of particles with disc blades and, thus permanent milling between the different layers of blades resulting in improved micronization compared to state of the art devices.

The use of the device for micronization from the present invention

[0069] The discs and hitting elements from the micronization device of the present invention can be built from various materials such as stainless steel 316, tungsten carbide or similar depending on the hardness of the materials to be micronized.

[0070] The micronization device of the present invention can be successfully used for milling of pure substances or mixtures of several substances, organic, inorganic or mixed compositions. Specifically it can be used for the processing of substances from the classes of raw materials, intermediates or final products in pharmaceutical, cosmetic, food, agrochemical or construction industry, in various kinds of chemical industries, agriculture, and in other fields of production.

[0071] A micronization process could induce defects in the crystalline network: these defects and increase of amorphous surface can improve the dissolubility of poorly soluble drugs. For example one such pharmaceutically active poorly soluble substance, is anti-ursodeoxycholic acid (UDCA). UDCA's solubility can be significantly improved by the use of the present device in a cost effective way, thus achieving better oral bioavailability.

[0072] The present invention can potentially enhance qualitative characteristics of various food ingredients, enabling cost-effective production processes and avoiding the need for chemical interventions. Micronizing macromolecular compounds can result in their more efficient processing, better solubility and oral bioavailability. Such modified molecules positively influence taste and nutritive characteristics. Micronized polysaccharides with high molecular mass, can also improve gelling characteristic and stability of gelatinous substances. Ratio of soluble fibers in food can be also increased by application of the present invention (breakage of chains, surface area increase) which is otherwise established only by addition of enzymes and implementation of heating process. During extraction of active ingredients from dry substances, the prior use of the present device on those substances can significantly improve related extraction time/quality and reduce the need for organic solvents due to smaller raw material particle size, increase of specific surface (better contact of solvent and raw material) and breakage of the bonds between active ingredient and raw material.

[0073] The present device can be also used for cost-effective processing of silica (including desert sand) to achieve more reactive nano size particles that can be used as advanced concrete additive for improvement of concrete properties or as added in certain percentage for brick production.

[0074] Herein mentioned examples of the use of the micronization device from the present invention are only illustrative and do not include all possible technical applications.

Examples

Example 1.

Preparation of pterostilbene nanoparticles for better solubility - bioavailability

[0075] 1.00 kg of Pterostilbene (98% purity, Organic Herb, China) 50 μm of crystalline particles as model substance were subjected to micronization from present invention with additional maintaining of low temperature (20 °C) of substance via slow liquid nitrogen stream flow addition through neck feeder. In this preparation discs with blades in the shape of cubes and extruded triangles were used (Figure 4), according to the present invention.

[0076] Such prepared samples of micronized Pterostilbene were subjected to particles size analyses and water solubility measurement. Average particle size after seven repeated processes of micronization were around 0.4 μm and significantly increased amorphous surface ratio (34% w/W). Solubility increased from 23 $\mu\text{g/ml}$, to 128 $\mu\text{g/ml}$.

Example 2.

Preparation of SiO_2 nanoparticles for concrete additive.

[0077] 1.00 kg of white SiO_2 sand from Drava River was commercially obtained from the store in Croatia. The average particle size was between 0.1-2 mm. In this preparation discs with blades in the shape of cubes and extruded triangles were used (Figure 4), according to the present invention, but all together reinforced with tungsten carbide coating. The average particle size of sand after nine repeated micronization processes were 0.35 μm .

List of reference numerals

[0078]

5	1	device for micronization / disintegrator
	3	rotors
	5	trapezoidal / rectangular hitting element
	7	triangular hitting element
	9	first ring
10		
	11	other ring
	13	longer cathetus
15	14	other cathetus
	15	hypotenuse
	20	electro motor
20		
	21	casing
	22	mount
25	23	filler
	24	hopper
	A	distance
30		
	d	diameter
	r	radius
35	ω	direction of rotation

Claims

- 40 1. Device (1) for the micronization of substances comprising
- two rotors (3) driven in a direction (ω) opposite to each other,
 - each rotor (3) carrying at least one row of multiple hitting elements (5, 7) forming a ring (9, 11), said rings (9, 11) being arranged concentrically in,
 - 45 - the rings (9, 11) of the different rotors (3) engaging alternately with one another,
 - the hitting elements (5, 7) being suitably arranged to provide transportation of the substance from inside the rings (3) to the outside by effecting a suitable airflow,
 - at least two, directly adjoining rings (9, 11) carrying hitting elements (5, 7) with different foot print **characterized in that**
 - 50 at least a first ring (9) is equipped with trapezoidal hitting elements (5) with trapezoidal foot print and at least one other ring (11), directly adjoining the first ring (9) is equipped with triangular hitting elements (7) with triangular foot print.
- 55 2. Device (1) according to claim 1,
characterized in that
one side of the triangular hitting elements (7) and/or one of the parallel sides of the trapezoidal hitting elements (5) is perpendicularly oriented to a radius (r) crossing the hitting element (5, 7).

3. Device (1) according to one of the preceding claims,
characterized in that
the trapezoidal hitting elements (5) are of rectangular foot print.
- 5 4. Device (1) according to one of the preceding claims,
characterized in that
the triangular hitting elements (7) are of basically right angled triangular foot print.
- 10 5. Device (1) according to claim 4,
characterized in that
the perpendicularly oriented side of the triangular hitting elements (7) is the longer cathetus (13) or hypotenuse (15) of the triangle.
- 15 6. Device (1) according to one of the preceding claims,
characterized in that,
the longer cathetus (13) or the longest side of the triangular hitting elements (7) is oriented in front in direction of rotation (ω) of the respective rotor (3).
- 20 7. Device (1) according to one of the preceding claims,
characterized in that
the hitting elements (5, 7) of directly adjoining rings (9, 11) are suitably arranged to provide mulling between the hitting elements (5, 7) of adjacent rings (9, 11).
- 25 8. Device (1) according to one of the preceding claims,
characterized in that
each ring (9, 11) carries a number of hitting elements (5, 7) equivalent to the diameter (d) in cm of the respective ring (9, 11).
- 30 9. Device (1) according to one of the preceding claims,
characterized in that
the hitting elements (5, 7) are made of stainless steel and / or ceramics and / or coated with industrial diamond and / or ruby.
- 35 10. Device (1) according to one of the preceding claims,
characterized in that
the triangular hitting elements (7) and the trapezoidal hitting elements (5) have at least two sides with coinciding length.
- 40 11. Use of the device (1) according to one of the preceding claims, for the micronization of solid inorganic or organic substances.
- 45 12. Use of the device (1) according to one of claims 1 to 10, wherein resulting particles have a particle size between 0,1 and 5 μm .
- 50 13. Use of the device (1) according to one of claims 1 to 10, for the micronization of crystalline substances, the resulting material having a surface with a portion of at least 9% preferably at least 14% amorphous surface.
- 55 14. Use of the device (1) according to claim 13, for the micronization of crystalline substances, the resulting material having a surface with a portion of at least 19%.

Figure 1

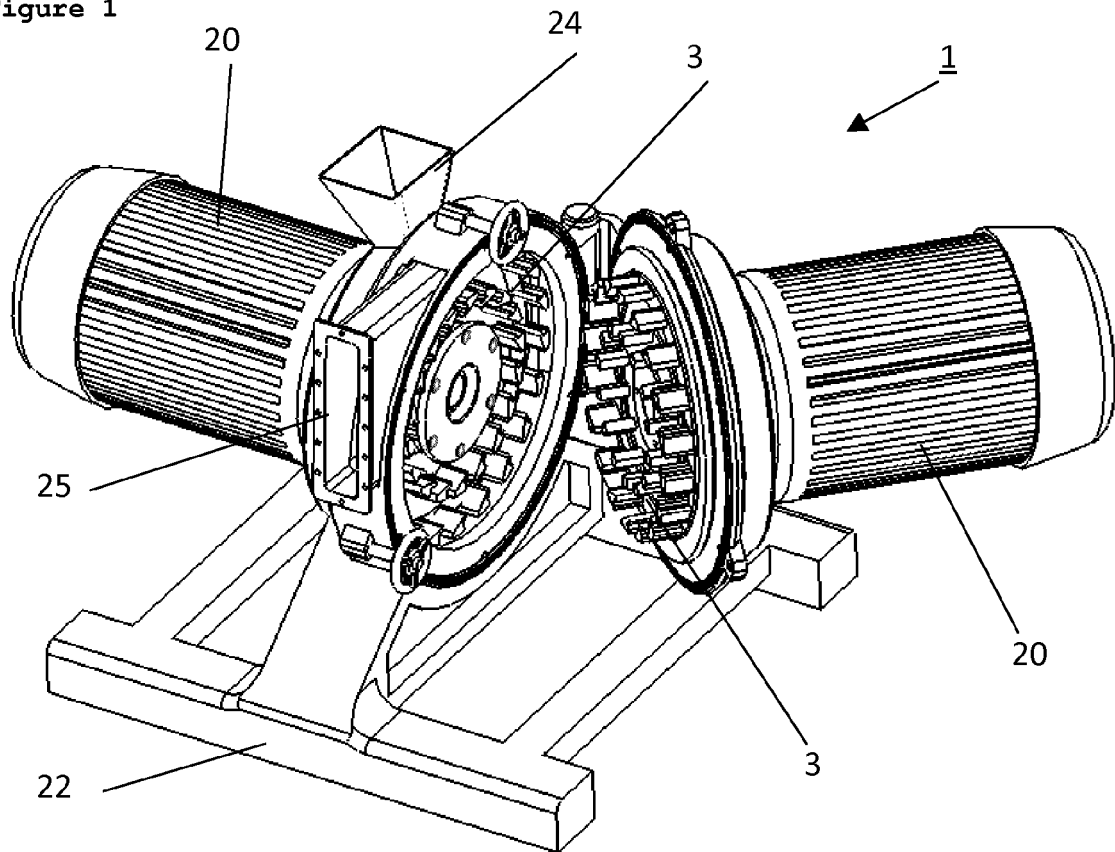


Figure 2

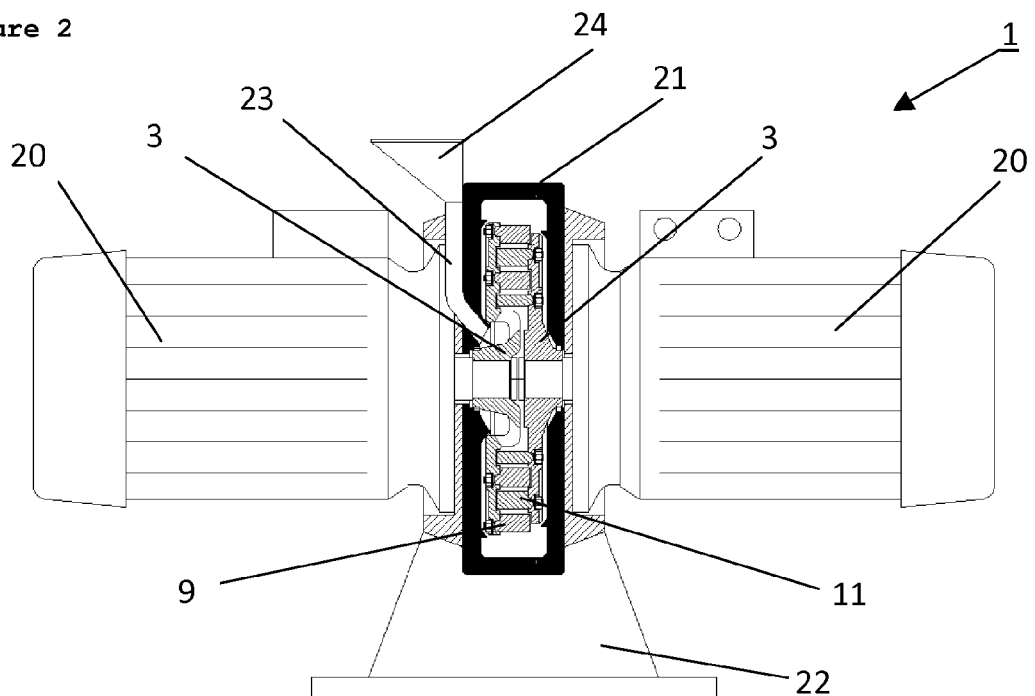


Figure 3

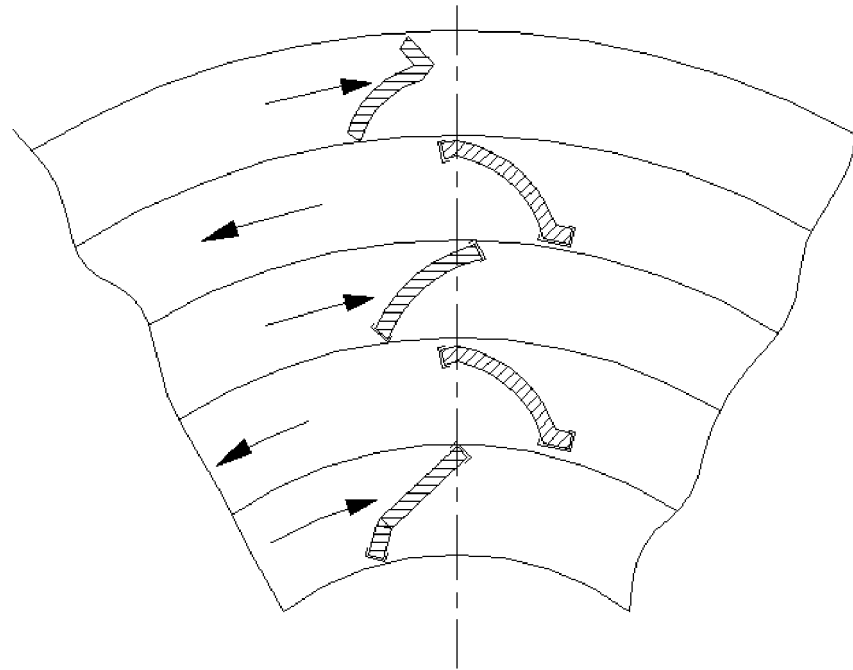


Figure 4

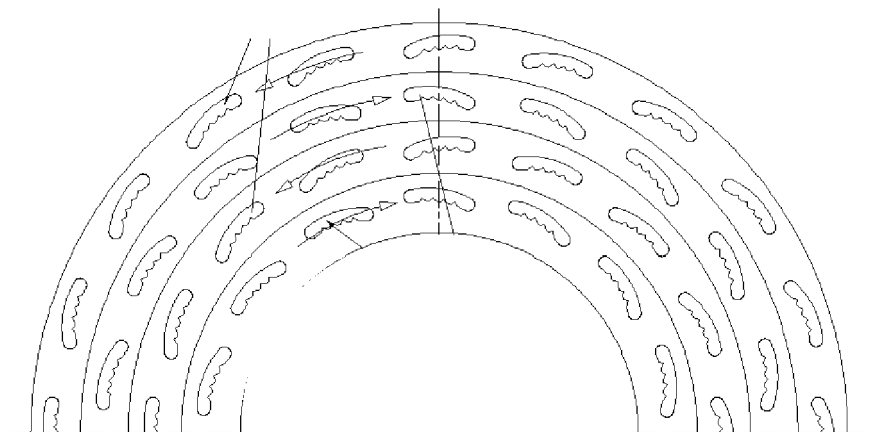


Figure 5

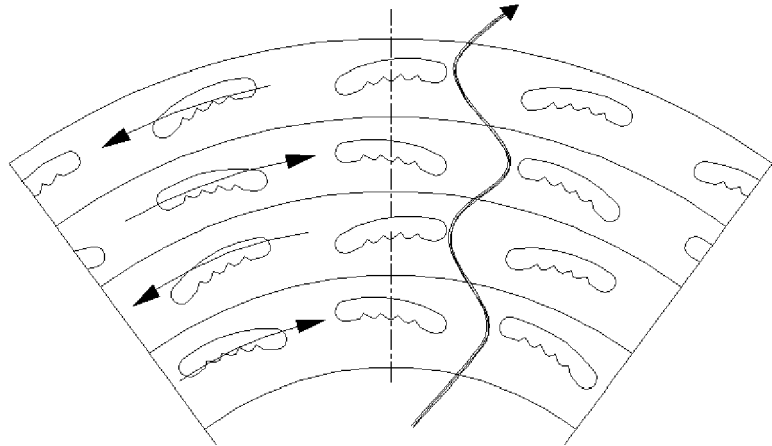


Figure 6

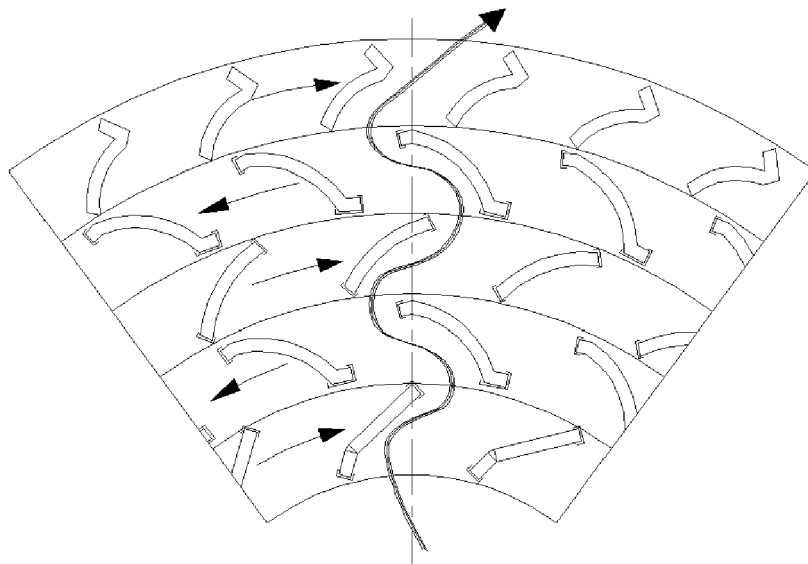


Figure 7

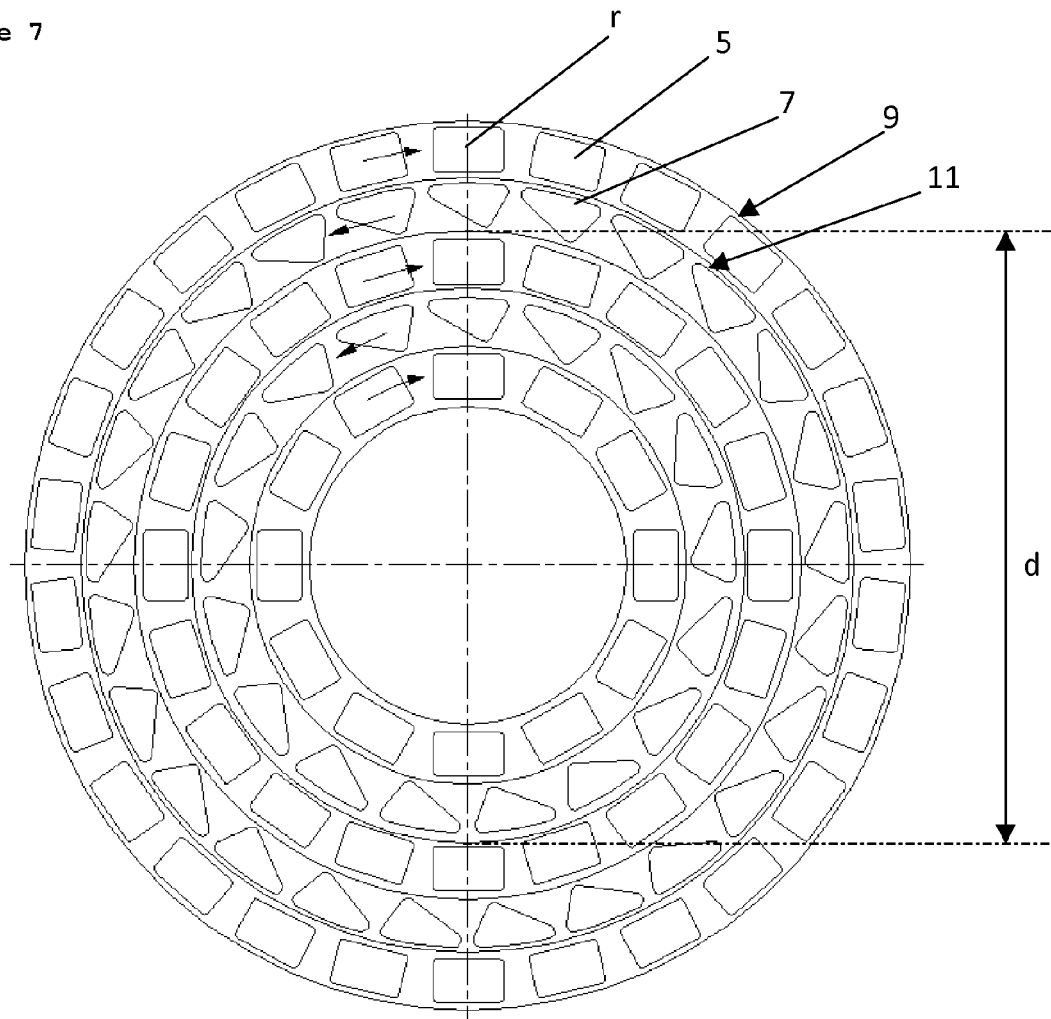


Figure 8

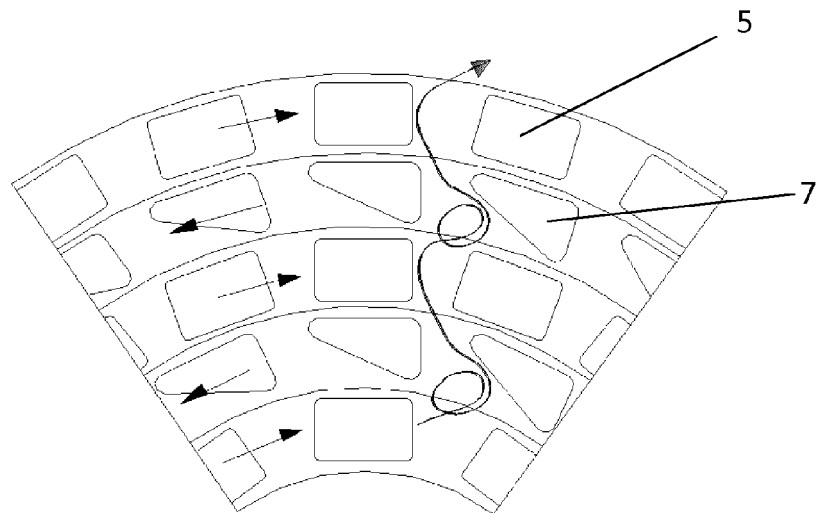


Figure 9

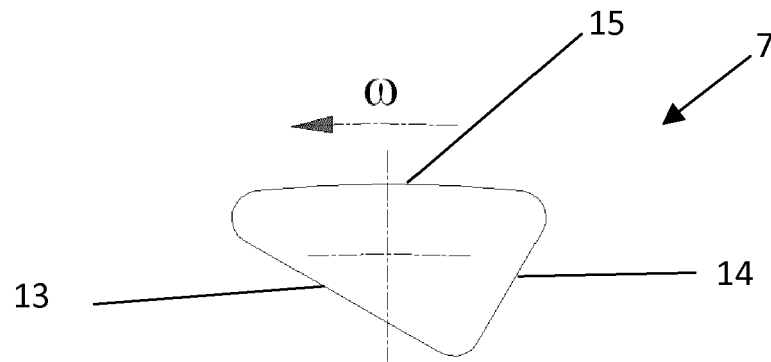


Figure 10

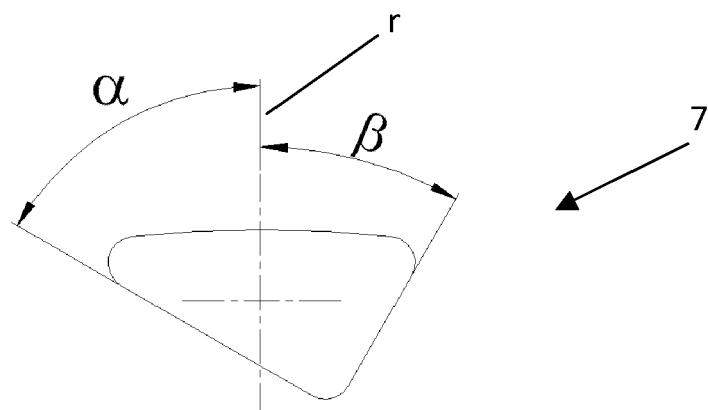
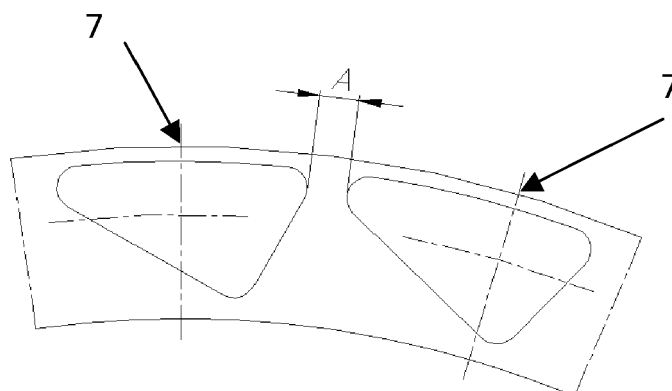


Figure 11





EUROPEAN SEARCH REPORT

Application Number
EP 12 17 7709

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Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
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CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			

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