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(72) Inventeurs/Inventors:
MUROFUSHI, KANJI, US;
NAGURA, SHIGEHIO, US

(73) Propriétaires/Owners:
SHIN-ETSU CHEMICAL CO., LTD., JP;
SHIN-ETSU BIO INC., US

(74) Agent: SMART & BIGGAR

(54) Titre : PROCEDE POUR L'OBTENTION DE GOMME XANTHANE SOLUBLE DANS LES SOLUTIONS SALINES

(54) Title: PROCESS FOR PRODUCTION OF SALINE-SOLUTION SOLUBLE XANTHAN GUM

(57) **Abrégé/Abstract:**

A process for saline-solution soluble xanthan gum which comprises the steps of precipitating xanthan gum by mixing, with stirring, an aqueous solution of xanthan gum with an organic solvent which is a non-solvent to xanthan gum but is hydrophilic, removing liquid from the precipitated xanthan gum to a liquid content of at most 50%, disintegrating the cake of xanthan gum after the liquid removal to an average grain size of from 0.3 to 2 cm in diameter, and drying the disintegrated xanthan gum by fluidizing the same at a temperature not exceeding 80°C.



ABSTRACT OF THE DISCLOSURE

A process for saline-solution soluble xanthan gum which comprises the steps of precipitating xanthan gum by mixing, with stirring, an aqueous solution of xanthan gum with an organic solvent which is a non-solvent to xanthan gum but is hydrophilic, removing liquid from the precipitated xanthan gum to a liquid
5 content of at most 50%, disintegrating the cake of xanthan gum after the liquid removal to an average grain size of from 0.3 to 2 cm in diameter, and drying the disintegrated xanthan gum by fluidizing the same at a temperature not exceeding 80°C.

PROCESS FOR PRODUCTION OF SALINE-SOLUTION SOLUBLE XANTHAN GUM

BACKGROUND OF THE INVENTION

The present invention relates to a process for modifying xanthan gum to obtain a xanthan gum having enhanced solubility in saline solution.

5 Xanthan gum is a naturally occurring high molecular weight polysaccharide and has found a wide scope of uses, particularly as a thickener or viscosity modifier for aqueous based compositions. For example, it has found extensive use in the food, cosmetic and oil industries. The ability of xanthan gum to effectively modify an aqueous solution is directly related to its solubility in the
10 aqueous medium. Since in such uses, the aqueous compositions generally contain sodium chloride or other salts, it is desired that the xanthan gum have as high a solubility in such salt solutions as possible. However, xanthan gum which is conventionally obtained from the fermentation of *Xanthomonas campestris* does not exhibit a particularly high solubility in saline solution, and in particular, saline
15 solutions having relatively high salt concentrations.

A process for obtaining a modified xanthan gum which exhibits enhanced solubility in aqueous solutions of salts, such as, sodium chloride, is disclosed in U.S. Patent 5,416,206. As described therein, and as referred to herein, enhanced solubility means that the xanthan gum exhibits a viscosity of not less than
20 800 mPa as determined on a 0.5% by weight solution thereof in a 12% by weight

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aqueous sodium chloride solution at 20°C at 30 rpm using a Brookfield BL* viscometer and having a ratio of this viscosity to that determined on a 0.5% by weight solution thereof in distilled water of not less than 1.5.

5 This process requires equipment for forming fine fibers of the precipitate while mixing an aqueous solution of xanthan gum with an organic solvent which is a non-solvent to xanthan gum but is hydrophilic. Japanese Patent Provisional Publication Nos. Sho 58-5301(5301/1983) and
10 Hei 1-318001(318001/1989) teach a method for drying xanthan gum directly as precipitated in the form of lumps rather than fine fibers. The dried lumps, despite subsequent grinding, give products sometimes with unfavorably affected properties, such as, low solubility in aqueous saline
15 solutions and other salts. In view of this, there has been need for a way of obtaining xanthan gum of good quality even through the conventional precipitation process.

SUMMARY OF THE INVENTION

 A first aspect of the present invention provides
20 an apparatus for recovering a polysaccharide from an aqueous solution thereof comprising: (a) precipitation means comprising a tank and a mixer therein and having means for introducing the aqueous solution of the polysaccharide and a hydrophilic nonsolvent for the polysaccharide thereto and
25 means for removing a mixture of liquid and precipitated polysaccharide therefrom; (b) cake-producing means for receiving the precipitated polysaccharide and the liquid mixture from the mixing means and for reducing a liquid content of the mixture to no more than 50% by weight to
30 produce a cake of the polysaccharide; (c) disintegrating

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means for disintegrating the polysaccharide cake into particles having a size from 0.3 to 2 cm in diameter; and (d) drying means for drying the particles of xanthan gum at a temperature of no more than 80°C.

5 A second aspect of the present invention provides a process for producing saline-solution soluble xanthan gum, which comprises the

steps of precipitating xanthan gum by mixing, with stirring, an aqueous solution of xanthan gum with an organic solvent which is a non-solvent to xanthan gum but is hydrophilic, removing liquid from the precipitated xanthan gum to produce a xanthan gum cake having a liquid content of at most 50%, disintegrating the cake of
5 xanthan gum after the liquid removal into particles having an average size of from 0.3 to 2 cm, and preferably, from about 0.6 to 1.5 cm in diameter, and drying the particles of xanthan gum at a temperature not exceeding 80°C.

According to the present invention, the cake of precipitated xanthan gum is broken into particles by conventional means so that specially designed
10 equipment is not required. The drying can be concluded within a short time period while the xanthan gum is maintained substantially in the particulate state, whereby a xanthan gum exhibiting excellent solubility in saline is obtained.

BRIEF DESCRIPTION OF THE DRAWINGS

The present invention will become more fully understood from the
15 detailed description given hereinbelow and the accompanying drawings which are given by way of illustration only, and thus are not limitative of the present invention and wherein:

FIG. 1 is a schematic view illustrating a stirred tank for use in the present invention;

20 FIG. 2 is a sectional view illustrating essential parts of a V-type disk press for use in the invention;

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FIG. 3 is a sectional view taken along line II-II in FIG. 2;

FIG. 4 is a front view of either screen 2a or 2b;

FIG. 5 is a sectional view taken along line III-III in FIG. 4;

FIG. 6 is a sectional view of a disintegrator for use in the invention;

5 and FIG. 7 is a side view of a dryer for use in the invention.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

Fermentation Step

The present invention in its practice may use an aqueous solution of xanthan gum, e. g., a fermentation broth containing dissolved xanthan gum as
10 produced by the fermentation of, for example, *Xanthomonas campestris*, and a solution obtained by dissolving xanthan gum which has been previously precipitated and separated and/or processed, e.g., subjected to clarification treatments, and the like, in an aqueous solution.

Xanthan gum is prepared by well-known fermentation processes. For
15 instance, it is produced by placing a bacterium belonging to the genus *Xanthomonas*, i.e., *X. campestris*, under appropriate fermentation conditions. (This compound and the process for producing the same are described in U.S. Patent 3,659,026.)

In producing xanthan gum, *Xanthomonas campestris* as a xanthan
20 gum-producing microorganism may be replaced by any other known *Xanthomonas*

species. Examples are *X. carotae*, *X. incanae*, *X. begoniae*, *X. papavericola*, *X. translucens*, *X. vasculorum*, and *X. hederiae*.

Precipitation Step

The xanthan gum to which the present invention applies is prepared
5 conventionally by preliminary fermentation and main fermentation of a xanthan gum-producing bacterium on a suitable culture medium. It is usually obtained as a xanthan gum fermentation broth.

In the practice of the invention, the fermented solution is sterilized (heat treated), and the resulting aqueous xanthan gum solution is mixed with an
10 organic solvent which is a non-solvent to xanthan gum but is hydrophilic by feeding them together to a tank equipped with agitating blades capable of high-speed agitation and mixing. The agitation-mixing in the tank causes precipitation of xanthan gum.

For the efficient mixing of a highly viscous aqueous xanthan gum and
15 a hydrophilic organic solvent and also for the satisfactory recovery of the precipitated xanthan gum, the use of an agitation system that involves high-speed reciprocating motion of the blades is recommended. Ordinary agitation using one-way revolution allows precipitating xanthan gum to stick to the blades and often the precipitate is difficult to recover. The blade diameter/tank diameter ratio
20 is desirably 0.5 or more. If the ratio is less than 0.5, stagnant regions develop along

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the inner walls of the tank, where the aqueous solution of xanthan gum and the hydrophilic organic solvent become immiscible.

The xanthan gum to be handled under the present invention is not specially limited. The invention can be used for any fermented solutions or broths
5 of xanthan gum with or without subsequent filtration or treatment with chemicals or the like, and aqueous solutions in which previously recovered xanthan gum has been redissolved. Also, the composition of the aqueous solution, as well as its xanthan gum concentration, ph, temperature, and concentrations of other additives are not limited.

10 The hydrophilic organic solvent incapable of dissolving xanthan gum that is used in precipitating xanthan gum may be an alcohol such as methanol, ethanol or isopropanol, or dioxane, acetone, tetrahydrofuran or the like. A combination of these solvents can also be used.

Liquid Removal Step

The xanthan gum precipitated by the process, at this stage contains at
15 least about 80% by weight liquid. Drying the precipitate in this state adds substantially to the energy cost and requires high temperature and much time. Moreover, it is prone to overdrying, which undesirably affects the solubility of the xanthan gum in saline solutions. To avoid these, it is important to remove liquid from the xanthan gum to reduce its liquid content to 50% or less.

In the liquid removal step where the present invention is embodied, a dispersion containing precipitated xanthan gum is fed to a V-type disk press having a pair of revolving disk-shaped screens, the space between which decreases as the screens revolve. The xanthan gum dispersion is thus squeezed by the pair of
5 disk-shaped screens, and its liquid content pressed out through the screens and recovered, while the xanthan gum is compressed into a cake which revolves with the screens until it is discharged for recovery.

Disintegration Step

The xanthan gum cake after the liquid removal takes the form of
10 small lumps or long cords. This precipitate can retain interior regions of high-liquid-content regions. To completely dry the xanthan gum in this form would require elevated temperatures and long drying times, which, in turn, adversely affects the saline-solution solubility of the product. In accordance with the present invention, the xanthan gum cake obtained is broken or disintegrated into small
15 grains or particles, which allows for the drying to be carried out at a sufficiently low temperature so as not to adversely affect the saline solubility of the product.

The disintegration method is not specially limited, but in the practice of the invention the use of a cutter provided with a screen is desirable. The average grain diameter of the xanthan gum thus broken out is preferably between 0.3 and 2
20 cm, and more preferably between 0.6 and 1.5 cm. A diameter larger than 2 cm is not recommended because it necessitates a high temperature treatment to dry up the interior of the xanthan gum grains. On the other hand, breaking down the cake

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into finer pieces of less than 0.3 cm in diameter is also undesirable since it can cause screen clogging or other problems which lead to reduced productivity.

Drying Step

The xanthan gum disintegrated by the afore-described method is dried
5 at a temperature not higher than 80°C. The drying method is not specially restricted but a system of the type that fluidizes the xanthan gum grains during the process of drying is desirable. The drying temperature should not exceed 80°C. Otherwise, the saline- solution solubility of the product will be undesirably decreased.

10 Grinding Step

In practicing the invention, it is possible to grind the dried xanthan gum to a sufficiently fine size to facilitate the dispersion in saline solutions. The grinding method may be any of the methods in common use, e.g., impact grinding.

The invention will now be described in more detail with reference to
15 the accompanying drawings showing a preferred embodiment thereof.

FIG. 1 illustrates a preferred form of tank for use in the precipitation step according to this invention. In FIG. 1, numeral 101 indicates drives including a motor for driving an agitator shaft, 102 indicates a stirring tank, and 103 indicates the agitator shaft 103. Agitator shaft 103 is provided with upper and lower
20 agitating blades 104 and 105, which, in opposing pairs, extend from agitator shaft

103 in the left and right directions as viewed in this figure. In the embodiment being described, each pair of agitating blades 104 or 105 has longitudinal axes on the same plane. Agitating blades 104 and 105 are triangular in cross section and are each tapered.

5 The number and cross sectional contour of agitating blades 104 and 105 are not limited to those embodied here but any other number and/or contour may be adopted unless it departs from the purposes of the invention.

 Agitating blades 104 and 105 are capable of stirring and mixing at high speeds, turning back and forth at every quarter of a revolution. Ordinary
10 agitation that involves one-way blade revolution often causes the precipitated xanthan gum to stick so fast to the blades that it can hardly be recovered. The above-mentioned reciprocating motion can be produced by a known mechanism for the transmission of rotation from the drive motor to the agitator shaft 103. It is, of course, possible to control the drives with the provision of microprocessor-based
15 controls so as to effect such reciprocating motion.

 The blade diameter/tank diameter ratio is desirably 0.5 or upwards. A ratio of less than 0.5 is undesirable because it forms a stagnant region close to the inner wall of tank 102, where an aqueous solution of xanthan gum and a hydrophilic organic solvent cannot be mixed. The tank is supplied with an organic solvent
20 (isopropanol) which is a non-solvent to xanthan gum but is hydrophilic and an aqueous solution of xanthan gum. Mixing them with stirring in the tank permits the

precipitation of xanthan gum. After the precipitation procedure, a dispersion containing the precipitated xanthan gum is fed to a V-type disk press shown in FIGS. 2 and 3.

FIGS. 2 and 3 illustrate a preferred form of the V-type disk press for use in the liquid removal step (referred to herein as a "V-type disk press"). In these figures, reference numeral 1 indicates the main body of the V-type disk press, 2a and 2b are screens, 3a and 3b are spindles, 4a and 4b are supporting arms, and 5 is a hydraulic cylinder (or spring-loaded cylinder).

The pair of screens 2a and 2b, as can be seen, are disk-shaped (or conical) and fixed to rotating supports 11a and 11b. The space between screens 2a and 2b is the widest on the raw material inlet side A and the narrowest on the opposite side B that is 180 degrees apart. Screens 2a and 2b are divided into 6 segments each when viewed from the front as in Fig. 4. Each segment consists of a punched metal plate 20 in which a large number of small openings 21 are made evenly throughout. Its relative open area is governed by the size (of the screen mesh) and number of the small openings 21. The punched metal plate 20 is suitably strengthened on the rear side by a reinforcing plate 22 to withstand the pressures during pressing (FIG. 5). The reinforcing plate 22 has larger, uniformly spaced openings 23.

In this embodiment, the size of the individual small openings (of the screen mesh) is desirably not greater than 1 mm^2 , and screens having circular or

slit-like openings are used. Meshes greater than 1 mm^2 are not desired because of large loss of xanthan gum through the screens upon pressing. As FIG. 3 shows, the spindles 3a and 3b are disposed in a pair, on the left and right, fixedly supported by supporting arms 4a and 4b, respectively, and connected rockably relative to each other by a center pin 6 at the center of main body 1. Center pin 6 is fixed at both ends to main body 1. Supporting arms 4a and 4b are L-shaped and fixedly support spindles 3a and 3b at one end in the manner described above and are connected to each other at the other end through hydraulic cylinder 5 or a spring-loaded cylinder. In the neighborhood of hydraulic cylinder 5, supporting arms 4a and 4b are displaceably connected to the main body with links 7a and 7b, respectively. Numeral 8 designates a pivot for link 7a and supporting arm 4a, and 9 designates a pivot for links 7a and 7b. Of course, this mechanism includes a pivot for link 7b and supporting arm 4b but it cannot be seen in FIG. 3. Pivot 9 can slide up and down within a recess 10 (thus constituting a slider).

A combination of main body 1, center pin 6, supporting arm 4a integral with spindle 3a, pivot 8, link 7a, pivot 9, and recess 10 forms revolving slider crank mechanism. Similarly, main body 1, center pin 6, supporting arm 4b integral with spindle 3b, pivot 8, link 7b, pivot 9, and recess 10 combinedly form a revolving slider crank mechanism. Owing to these mechanisms, it is made possible, by regulating the opening angle of links 7a and 7b through the lateral movement of hydraulic cylinder 5 or spring-loaded cylinder, to adjust the angle between spindles 3a and 3b cooperating with those elements and thereby control the degree of opening of screens 2a and 2b and hence the pressing force.

While revolving slider cranks are used in this embodiment as mechanisms for regulating the degree of opening of the screens, any other mechanism capable of regulating the spindle angle may be employed instead under the invention, such as quadric crank chains in which the sliders are replaced by
5 links.

As shown in FIG. 3, rotating supports 11a and 11b are rotatably supported by spindles 3a and 3b, respectively, via bearings and are rotated by driving forces transmitted to sprockets 12a and 12b integral with the supports (through endless chains). Alternatively, any of various other mechanisms, e.g.,
10 V-belt transmission, may be used for power transmission to rotating supports 11a and 11b.

The material fed to the raw material inlet A is held between screens 2a and 2b and is gradually squeezed as screens 2a and 2b revolve, with its water
15 content flowing out through the screens to their back side. The feed is subjected to maximum compression at point B, 180 degrees apart from the inlet, and then the spacing between screens 2a and 2b increases gradually, and a dewatered cake is discharged along a scraper 2013. In this way, a xanthan gum component in the form of a cake can be recovered.

20 The rate at which a dispersion of xanthan gum is fed to the V-type disk press is suitably chosen in consideration of the factors including the xanthan gum concentration in the dispersion and the state of precipitation of xanthan gum.

Unlike centrifugal separators that utilize the difference in specific gravity, the V-type disk press of this embodiment depends on compression. The degree of liquid removal can be adjusted with the compression pressure and the speed of screen revolution, whereby the xanthan gum is squeezed to a water
5 content of only 40-50% by weight. Consequently, the cleaning and purifying effect of the hydrophilic organic solvent is improved and the energy load on the ensuing drying step is lessened.

The V-type disk press shown in FIGS. 2 and 3 can squeeze the feed at low speeds of screen revolution, usually in the range of about 1 to 12 rpm (for
10 screens 0.5 to 1.5 m across). Compared with centrifugal separators that run at 500 to 2,000 rpm, it requires far less power for its operation. Another advantage is the higher degree of safety because there is no need of running the press at high speed in the presence of an organic solvent.

After the liquid removal step, the cake of xanthan gum is transferred
15 to a disintegrator as shown in FIG. 6, where it is broken up into xanthan gum grains from 0.3 to 2 cm in diameter.

FIG. 6 illustrates a preferred form of the disintegrator.

In FIG. 6, 601 is the main body of the disintegrator, 602 is a hopper, 603 is a treating tank, 604 is a rotary cutter, and 605 is a screen.

20 Treating tank 603 is built as a cylinder, in which rotary cutter 604 runs. Rotary cutter 604 has three revolving cutter blades 606. The cake of xanthan gum fed at the hopper 602 is broken up into pieces by the cutting blades 606 of

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rotary cutter 604 in treating tank 603. The pieces disintegrated to a given size or finer are discharged through screen 605 and then through an outlet 607. The size of the resulting xanthan gum grains can be controlled through the adjustment of the mesh size of screen 605. While the embodiment shown uses three revolving cutting
5 blades, the number of blades is not a limitation to this invention. The disintegrated xanthan gum is then sent to a dryer as shown in FIG. 7 for drying.

FIG. 7 illustrates a preferred form of the dryer. This dryer 701 is of a type which fluidizes xanthan gum grains during the process of drying, and is set to a drying temperature not exceeding 80°C. Dryer 701 includes a drying drum 702
10 which is rotated as a rotating shaft 703 is driven. Rotating shaft 703, shown at right, is driven with rotational forces transmitted from a drive motor 704. 705 is an inlet and 706 is an outlet. Hot water enters the dryer 701 from a pipe 707b on the right and flows through a pipe 711b into jackets 710 so as to adjust the temperature of dryer 701. After circulating through jackets 710, the hot water flows
15 out through a pipe 711a and finally through a pipe 707a. The temperature inside the dryer 701 can be regulated by setting the hot water temperature to a desired level.

Vapor-laden air is released under reduced pressure from a vent pipe 708 at the left end. This construction is realized by using a hollow rotating shaft
20 709 on the left and adopting other means known in the art. The dried xanthan gum is discharged from outlet 706, and it may be ground for easier dispersion in a saline solution. Impact grinding is preferred for that purpose.

Along with the embodiment thus far described, the process of the invention was tested. The results were as follows.

Testing Example 1

A liquid culture medium of the composition I given below was placed
5 in a 2,000-liter fermenter, inoculated with a culture of *Xanthomonas campestris* that had resulted from 24-hour preliminary cultivation, and incubated under aeration and agitation for 2 days, when a fermented solution containing 30 g/l of xanthan gum was obtained.

Culture medium composition I

10	Glucose	58 g/l
	Polypeptone	2 "
	KH ₂ PO ₄	2 "
	MgSO ₄ · 7H ₂ O	0.5 "
	Water	1,300 l

15 The fermented solution was heated at 70°C for 30 minutes to kill the xanthan gum-producing bacteria, and 480 l of the xanthan gum fermented solution and 720 l of aqueous isopropanol (water content: 15 wt%) were poured into a 1,600-liter precipitation tank of the construction described above in conjunction with FIG. 1. The charge was mixed with stirring by an agitation blade (the blade
20 diameter/inside diameter of the tank: 0.86, triangular blade, "Adzita* AG type" manufactured by Shimazaki Seisakusho K.K.) that turns reversely after running
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each quarter of a revolution at high speed within the tank, and xanthan gum was precipitated. The precipitated xanthan gum was in the form of a long cord about 30 to 40 cm in length having a diameter of from about 2 to 3 cm..

The dispersion containing the xanthan gum was fed at a rate of 1,500
5 liters per hour to a V-type disk press of the construction already described with reference to FIGS. 2 and 3 ("Asahi^{*} Press C-35," mfd. by Asahi Koki K.K., screen mesh size: 0.5 mm) for liquid removal. The liquid content in the product after primary liquid removal was 60% by weight. To 30 kg of the product after primary liquid removal was added one equivalent weight of isopropanol (water content:
10 15%) in a 500-liter stirred tank. After stirring, the resulting dispersion was fed again to the V-type disk press under the same conditions as described above for liquid removal. The xanthan gum product as a cake after the secondary liquid removal had a liquid content of 45% by weight.

The cake after the secondary liquid removal was broken into small
15 pieces using a disintegrator of the construction explained above in conjunction with FIG. 6. The disintegrated xanthan gum was in the form of grains from 0.6 to 1.5 cm in diameter.

In a 100-liter conical vacuum dryer of the construction illustrated in FIG. 7, 15 kg of the xanthan gum grains was dried at a drying temperature of 40 to
20 65°C and a drying pressure of 40 to 100 torr for 3.5 hours. The dry matter was

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ground to obtain a saline solution-soluble xanthan gum finely divided to 80 mesh or finer particles.

One gram of the xanthan gum thus obtained was placed in a 500 ml-tall beaker, and it was moistened by adding 3 g of 99% ethanol. Next, 196 g of
5 an aqueous solution of 12 wt% sodium chloride kept at 20°C was added, and the mixture was stirred using an anchor agitator at 1,000 rpm for 2 hours. Thorough dissolution of the xanthan gum and an increase in the viscosity of the aqueous sodium chloride solution were confirmed.

The resulting solution was allowed to stand at 20°C for one hour, and
10 then its viscosity was measured with a Brookfield BL* type viscometer. The viscometer speed was set to 30 rpm and the viscosity was measured with spindle No. 2, when the viscosity (1) was found to be 900 mPa·s. Then, the xanthan gum was dissolved in distilled water to form a solution at a concentration of 0.5 wt%. Its viscosity was measured using a Brookfield BL type viscometer at a speed of 30 rpm,
15 with spindle No. 2. The viscosity (2) was 500 mPa·s.

It was found that, in dissolving the xanthan gum in a saline solution, an agitation speed of 800 rpm or more gives good result. The configuration of the agitation blade is not specially limited; any shape is desirable if it permits the blade to fluidize the entire solution. Dissolution under these conditions produces a
20 solution, which attains a stable viscosity after about 60 minutes of agitation. It was

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also found that further agitation for 2 to 12 hours causes little change in the viscosity.

Comparative Example 1

One kilogram of xanthan gum grains obtained in the same manner as
5 described in Example 1 was dried in an air-blast dryer above 85°C for 3.5 hours. It
was then ground to an 80 mesh or finer xanthan gum powder. This finely divided
xanthan gum was subjected to viscosity measurement by the same method as used
in Example 1. The viscosity in saline solution, referred to as (1), was 350 mPa·s,
the viscosity in distilled water (none-saline), referred to as (2), was 500 mPa·s, and
10 (1)/(2) was 0.70. In the saline solution, many particles remained undissolved even
after 2 hours of agitation. Continued agitation up to 12 hours still failed to dissolve
the undissolved particles.

Comparative Examples 2 and 3

The cake of xanthan gum obtained by liquid removal in the manner
15 described in Example 1 was disintegrated using screens with mesh sizes of 2 or 3
cm. Generally in conformity with the procedure of Example 1, the disintegration
products were dried until their water contents dropped to 10% or less. Grinding
after the drying produced fine xanthan gum powders of 80 mesh or more. These
xanthan gum products were subjected to viscosity measurements as in Example 1.
20 The results are shown below.

TABLE 1

	Mesh Size	Av.Part. Dia.	Drying Time	Drying Pressure	Drying Temp.	Viscosity (1)	(1)/(2)
Comp. Ex.2	2 cm	1 ~ 2 cm	5 hrs	40 ~ 100 torr.	40 ~ 80°C	800 mPa s	1.6
Comp. Ex. 3	3 cm	2 ~ 3 cm	10 hrs	40 ~ 100 torr.	40 ~ 80°C	500 mPa s	1.0

Comparative Example 4

A xanthan gum precipitate obtained similarly to Example 1 was dried without prior liquid removal or disintegration. The dried precipitate was ground to a fine xanthan gum powder 80 mesh or finer in size. This xanthan gum was
5 measured for its viscosity in the manner described in Example 1. The results are given below.

TABLE 2

	Mesh Size	Drying Time	Drying Pressure	Drying Temp	Viscosity	(1)/(2)
Comp. Ex. 4	-	20 hrs	40 ~ 100 torr.	40 ~ 90°C	300 mPa s	0.6

The invention being thus described, it will be obvious that the same may be varied in many ways. Such variations are not to be regarded as a departure from the spirit and scope of the invention, and all such modifications as would be obvious to one skilled in the art are intended to be included within the scope of
5 the following claims.

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CLAIMS:

1. A method for producing saline soluble xanthan gum comprising the steps of:

(a) producing precipitated xanthan gum by mixing, with stirring, an aqueous solution of xanthan gum with a hydrophilic organic solvent in which the xanthan gum is insoluble;

(b) removing liquid from the precipitated xanthan gum to form a cake having a liquid content of no more than 50% by weight;

(c) disintegrating the xanthan gum cake into particles having an average particle size of from 0.3 to 2 cm in diameter; and

(d) drying the xanthan gum at a temperature not exceeding 80°C.

2. The method of claim 1, wherein the liquid is removed in step (b) by squeezing the precipitated xanthan gum.

3. The method of claim 1 or 2, wherein the organic solvent is selected from the group consisting of methanol, ethanol, isopropanol, dioxane, acetone, tetrahydrofuran, and combinations thereof.

4. The method of any one of claims 1 to 3, wherein the average particle size diameter is from 0.6 to 1.5 cm.

5. The method of any one of claims 1 to 4, wherein the liquid content of the cake is from 40 to 50% by weight.

6. The method of any one of claims 1 to 5, wherein the xanthan gum particles are dried in step (d) at a

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temperature from 40 to 65°C and a pressure of from 40 to 100 Torr.

7. The method of any one of claims 1 to 6, wherein the xanthan gum produced exhibits a viscosity of not less
5 than 650 mPa as determined on a 0.5% by weight solution thereof in a 12% by weight aqueous sodium chloride solution at 20°C at 30 rpm using a Brookfield BL* viscometer and exhibits a ratio of this viscosity to that determined on a
10 less than 1.3.

8. The method of any one of claims 1 to 7, wherein the liquid removal is carried out in a V-type disk press.

9. The method of any one of claims 1 to 7, wherein the liquid removal is carried out in a V-type disk press
15 having revolving disk shape screens for pressing the precipitated xanthan gum therebetween.

10. The method of claim 9, wherein the screens have openings of not greater than 1 mm².

11. The method of claim 9, wherein the screens have a
20 diameter of from 0.5 to 1.5 meters and are rotated at a speed of from 1 to 12 rpm.

12. The method of any one of claims 1 to 11, wherein the mixing is carried out with a rotating stirrer and by repeated cycles of rotation of the stirrer, first in one
25 direction and then in the reverse direction.

13. The method of claim 12, wherein the cycle of rotation comprises rotating the stirrer between about 90 to 360 degrees in one direction and then rotating the stirrer

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in an equal amount in the reverse direction.

14. The method of any one of claims 1 to 11, wherein the mixing in step (a) is carried out in a cylindrical tank having an agitator shaft therein provided with upper and
5 lower opposing pairs of agitating blades extending from the agitator shaft and wherein a ratio of a diameter of a circle defined by the extended blades to a diameter of the tank is at least 0.5.

15. The method of any one of claims 1 to 14, wherein
10 the dried xanthan gum is ground to a mesh size of 80 mesh or finer.

16. The method of any one of claims 1 to 7, wherein the liquid is removed in step (b) by squeezing the precipitated xanthan gum in a V-shape press having revolving
15 disk-shaped screens fixed to rotatable supports to create a space of variable width therebetween, having a raw material inlet at a point where the space is the widest, the space being the narrowest 180 degrees opposite the inlet, the screens being divided into six segments, each segment having
20 a metal plate with a plurality of openings spaced evenly throughout the segment, means for rotating the supports and screens, means for regulating the width of the space between the screens such that when material having moisture therein is fed to the inlet, it is gradually moved by the rotation
25 of the screens to the narrowest width thereby squeezing the moisture in the material therefrom to form a cake, and scraper means for removing the cake from the screen.

17. The method of any one of claims 1 to 16, wherein the xanthan gum cake is disintegrated in step (c) using a
30 disintegrator comprising a treating tank formed from a cylindrical wall, a portion of the wall being a screen, a rotary cutter disposed essentially in the cylindrical tank

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for chopping up xanthan gum which has been introduced to the cylindrical tank, the disintegrated xanthan gum being discharged from the cylinder through the screened wall portion.

5 18. The method of any one of claims 1 to 17, wherein the disintegrated xanthan gum is dried in a fluidized air dryer.

19. The method of any one of claims 1 to 17, wherein the precipitated xanthan gum is dried in a dryer comprising
10 a rotatable drying drum rotatable on a driven rotating shaft, the drum having an inlet and an outlet, and having an outer jacket for receiving hot liquid to heat the interior of the drum.

20. An apparatus for recovering xanthan gum from an
15 aqueous solution thereof comprising:

(a) precipitation means comprising a tank and a mixer therein and having means for introducing the aqueous solution and a hydrophilic nonsolvent for xanthan gum and means for removing a mixture of liquid and precipitated
20 polysaccharide therefrom;

(b) a V-type disk press having a pair of revolving disk-shaped screens and being adapted to squeeze the liquid from the mixture of the liquid and the precipitated xanthan gum due to a decrease of a space between the screens as the
25 screens revolve;

(c) disintegrating means for disintegrating the cake of xanthan gum into particles having a size from 0.3 to 2 cm in diameter; and

(d) drying means for drying the particles of
30 xanthan gum at a temperature of no more than 80°C.

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21. The apparatus of claim 20, wherein the V-type press (b) comprises:

a pair of revolving disk-shaped screens fixed to rotatable supports to create a space of variable width
5 therebetween;

a raw material inlet at a point where the space is widest, the space being narrowest 180 degrees opposite the inlet, the screens being divided into six segments, each segment having a metal plate with a plurality of openings
10 spaced evenly throughout the segment;

means for rotating the supports and screens;

means for regulating a width of the space between the screens such that when material having moisture therein is fed to the inlet, it is gradually moved by rotation of
15 the screens to the narrowest width thereby squeezing the moisture in the material therefrom to form the cake; and

scraper means for removing the cake from the screen.

22. The apparatus of claim 20 or 21, wherein the
20 disintegrating means (c) is a disintegrator comprising:

a treating tank formed from a cylindrical wall, a portion of the wall being a screen, and

a rotary cutter disposed essentially in the cylindrical tank for chopping up xanthan gum which has been
25 introduced to the cylindrical tank, whereby the disintegrated xanthan gum is discharged from the cylinder through the screened wall portion.

23. The apparatus of any one of claims 20 to 22,

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wherein the precipitation means (a) comprises an agitation system that involves a high-speed reciprocating motion of blades at a blade diameter/tank diameter ratio of 0.5 or more.

SMART & BIGGAR

OTTAWA, CANADA

PATENT AGENTS

FIG. 1

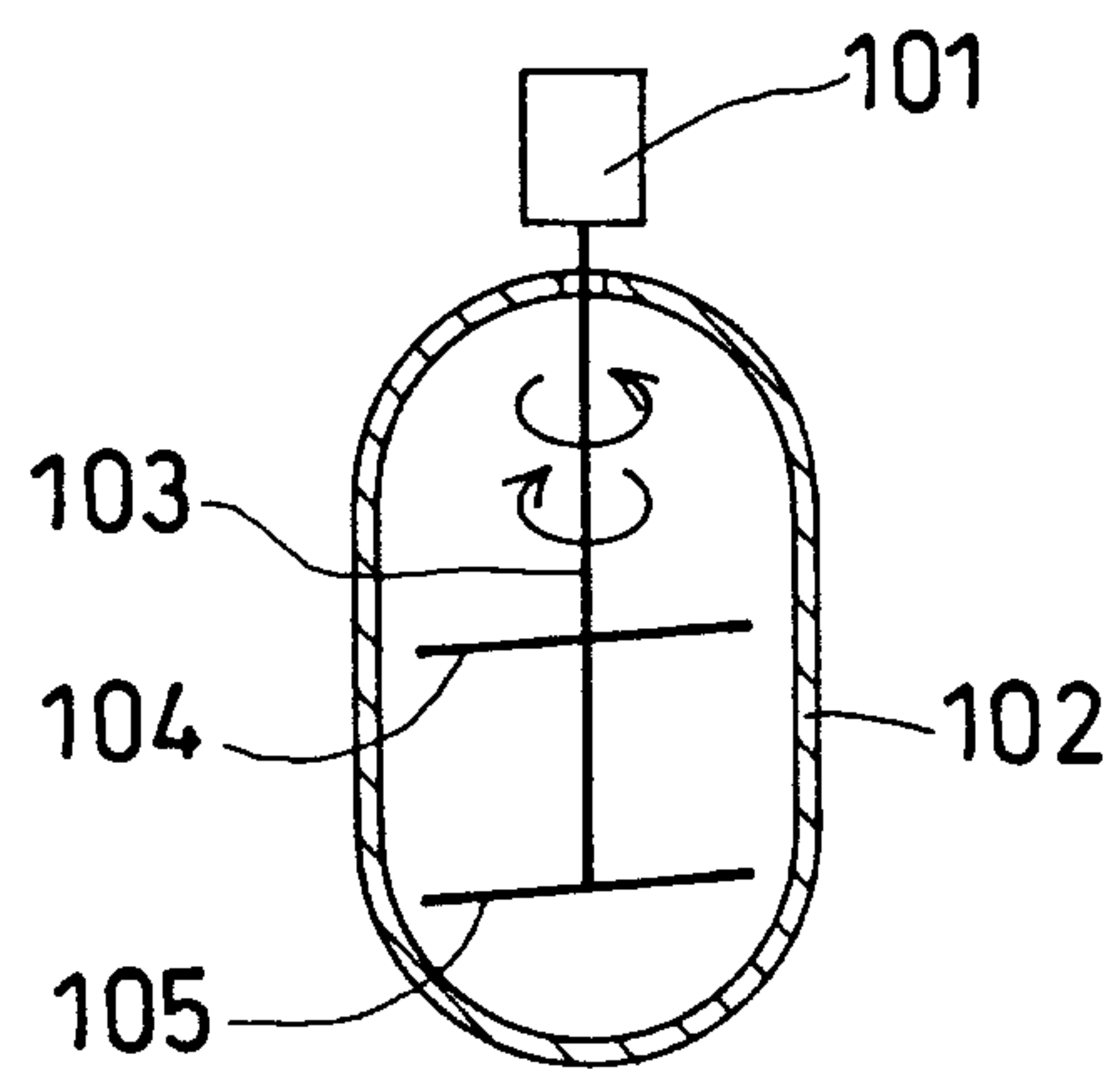
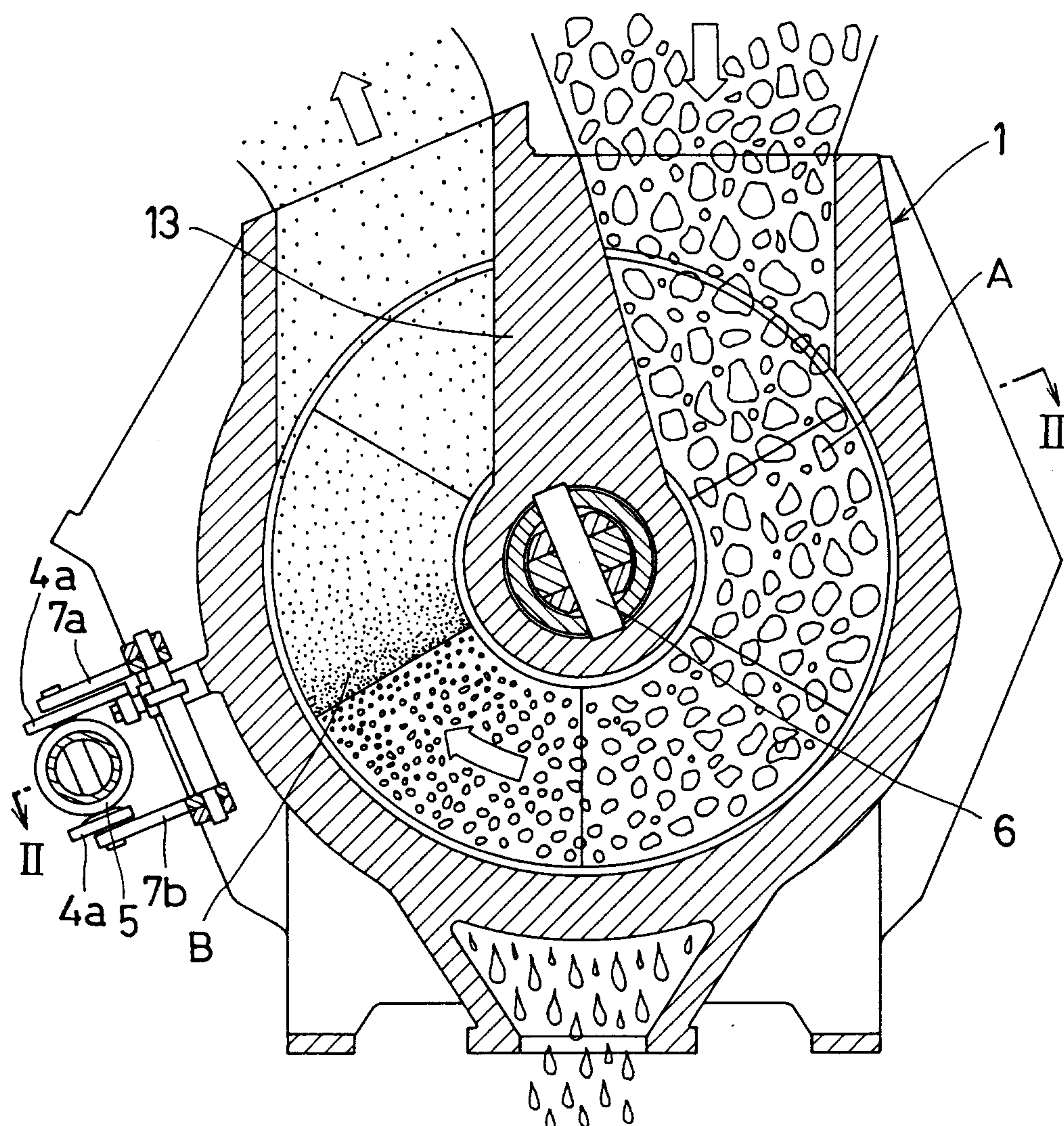


FIG. 2



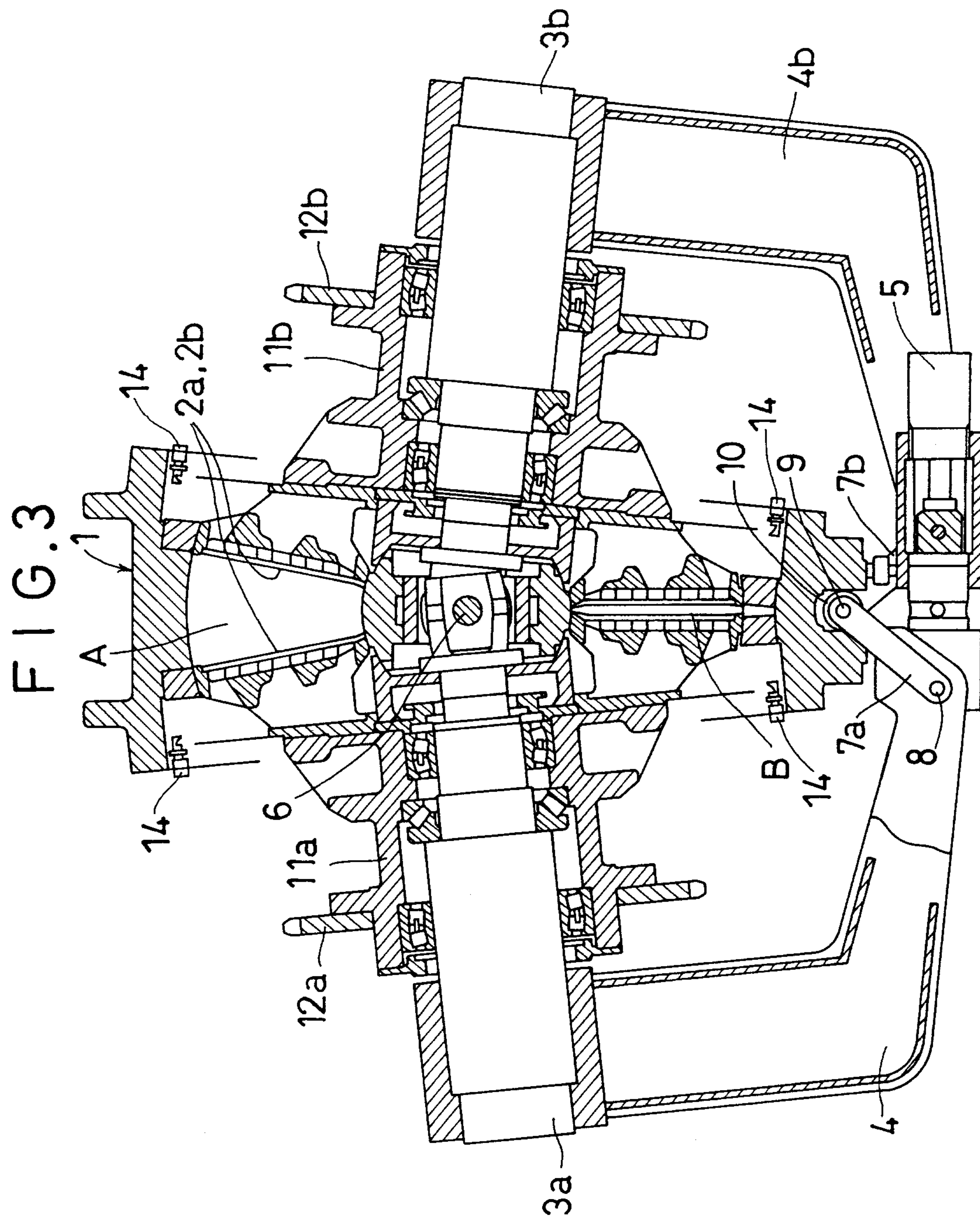


FIG. 4

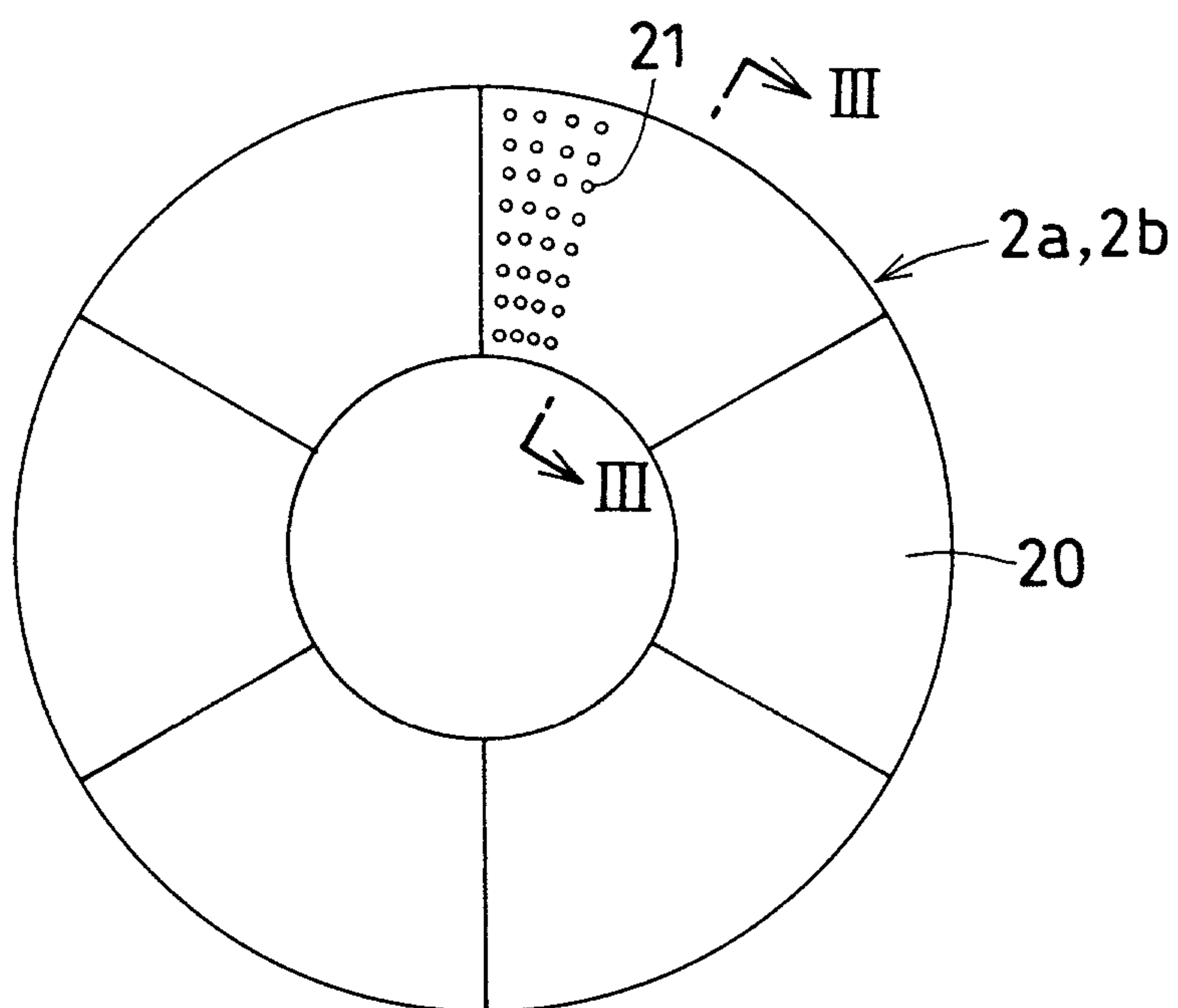


FIG. 5

