



(12) **EUROPEAN PATENT APPLICATION**
published in accordance with Art. 153(4) EPC

(43) Date of publication:
19.08.2020 Bulletin 2020/34

(51) Int Cl.:
B02C 17/16 ^(2006.01) **B01F 3/12** ^(2006.01)
B01F 7/00 ^(2006.01) **B02C 17/00** ^(2006.01)
B02C 17/20 ^(2006.01)

(21) Application number: **18866551.7**

(22) Date of filing: **01.10.2018**

(86) International application number:
PCT/JP2018/036638

(87) International publication number:
WO 2019/073835 (18.04.2019 Gazette 2019/16)

(84) Designated Contracting States:
AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO PL PT RO RS SE SI SK SM TR
Designated Extension States:
BA ME
Designated Validation States:
KH MA MD TN

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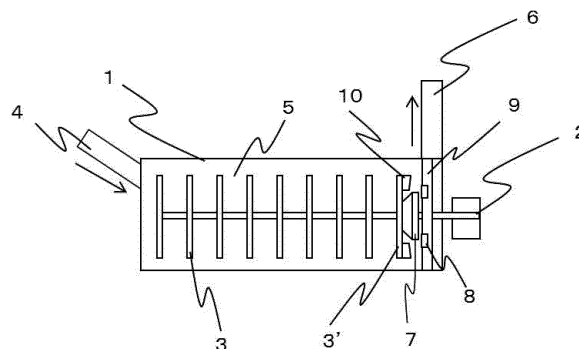
(54) **MEDIA TYPE DISPERSER AND LIQUID DISPERSOID MANUFACTURING METHOD**

(57) In order to provide a media-type disperser capable of improving productivity while suppressing damage due to abrasion of a screen or a gap separator, there is provided a media-type disperser that is a media-type wet disperser in which a drive shaft, a plurality of stirring blades that are arranged on the drive shaft and are simultaneously rotatable by rotation of the drive shaft, and media particles are housed in a cylindrical vessel having a supply port of a liquid mixture and an outlet of a liquid dispersion, in which the stirring blade nearest to the outlet

has a protrusion in a direction parallel to the drive shaft, and a thickness t (mm) of the protrusion, a diameter d (mm) of the media particle, and a distance L (mm) from the outlet side of the stirring blade nearest to the outlet to a rear end of the cylindrical vessel satisfy Formula (1) below,

$$10d \leq t \leq L - 10d \quad (1).$$

Fig. 1



Description

TECHNICAL FIELD

5 **[0001]** The present invention relates to a media-type disperser and a method for producing a liquid dispersion.

BACKGROUND ART

10 **[0002]** These days, media-type dispersers are frequently used as dispersers that finely disperse a liquid dispersion. A typical media-type disperser is one in which stirring blades (stirring discs) and media particles are housed in a cylindrical vessel; however, the abrasion of the stirring blade and the inner wall of the vessel has been a problem. Further, in general, a means for dividing of a liquid dispersion and media particles, such as a screen or a gap separator, is provided in a cylindrical vessel of a media-type disperser; however, there has been a problem of the clogging of the screen etc. due to media particles.

15 **[0003]** As a means for improving the abrasion resistance of a stirring blade placed in a cylindrical vessel and the inner wall of the vessel, for example, forming them out of a high molecular material having abrasion resistance and impact resistance is proposed (for example, see Patent Literature 1). Further, as a method for suppressing the clogging of a screen due to media particles, for example, a method is proposed in which the edge end of the stirring blade located most on the outlet side is extended up to near the side surface on the exit side of the outlet, thereby media that have
20 flowed in around a screen section are returned to the dispersing chamber by the rotation of the edge end, and thus a media separating chamber in which practically no media exist around the screen section is created (for example, see Patent Literature 2).

PRIOR ART DOCUMENTS

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PATENT DOCUMENTS

[0004]

30 Patent Document 1: Japanese Patent Laid-open Publication No. H7-24287
Patent Document 2: Japanese Patent Laid-open Publication No. 2000-171931

SUMMARY OF THE INVENTION

35 PROBLEMS TO BE SOLVED BY THE INVENTION

[0005] However, even in the technology described in Patent Document 1, as the viscosity of the liquid dispersion becomes higher, the fluidity of the media particles becomes lower around the screen or the gap separator that separates the media particles from the liquid mixture, and the speed difference between the screen or the gap separator and the
40 media particles becomes larger; hence, there has been a problem of the screen or the gap separator being damaged by abrasion. On the other hand, in the technology described in Patent Document 2, since practically no media particles exist around the screen section, damage due to abrasion of the screen or the gap separator is suppressed; however, the dispersing region in the cylindrical vessel cannot be utilized effectively, and hence there has been a problem of reduction in productivity.

45 **[0006]** Thus, an object of the present invention is to provide a media-type disperser and a method for producing a liquid dispersion that can improve productivity while suppressing damage due to abrasion of a screen or a gap separator.

SOLUTIONS TO THE PROBLEMS

50 **[0007]** In order to solve this object, the present invention mainly include any of the following configurations.

(1) A media-type disperser that is a media-type wet disperser in which a drive shaft, a plurality of stirring blades that are arranged on the drive shaft and are simultaneously rotatable by rotation of the drive shaft, and media particles are housed in a cylindrical vessel having a supply port of a liquid mixture and an outlet of a liquid dispersion, in
55 which the stirring blade nearest to the outlet has a protrusion in a direction parallel to the drive shaft, and a thickness t (mm) of the protrusion, a diameter d (mm) of the media particle, and a distance L (mm) from the outlet side of the stirring blade nearest to the outlet to a rear end of the cylindrical vessel satisfy Formula (1) below,

$$10d \leq t \leq L - 10d \quad (1).$$

(2) A method for producing a liquid dispersion includes a step of dispersing a liquid mixture containing at least filler particles each with a Mohs hardness of more than or equal to 4 and a solvent by using the media-type disperser.

(3) A method for producing a liquid dispersion, the method being a method of producing a liquid dispersion by stirring a liquid mixture by means of media particles and separating the media particles from the liquid mixture by means of a gap between a rotor being rotated by rotation of a drive shaft and a stator, in which an average relative speed V_n between a speed of the rotor and a speed in a rotation direction of the media particle existing in a position up to 1 mm from the stator toward the rotor in a direction parallel to the drive shaft satisfies Formula (2) below,

$$|V_n| \leq 5 \text{ (m/sec)} \quad (2).$$

EFFECTS OF THE INVENTION

[0008] According to the present invention, productivity can be improved while damage due to abrasion of a screen or of a gap separator in a media-type disperser is suppressed.

BRIEF DESCRIPTION OF THE DRAWINGS

[0009]

Fig. 1 is a schematic diagram showing an embodiment of a media-type disperser of the present invention.

Fig. 2 is an enlarged view of a stirring blade nearest to an outlet and the neighborhood of a gap separator in an embodiment of a media-type disperser of the present invention.

Fig. 3 is a schematic diagram showing another embodiment of a media-type disperser of the present invention.

Fig. 4 is a schematic diagram showing an example of a conventional media-type disperser.

Fig. 5 is a schematic diagram showing another example of a conventional media-type disperser.

Fig. 6 is a photograph of a rotor on which there are no traces of beads abrasion.

Fig. 7 is a photograph of a rotor on which there are traces of beads abrasion.

EMBODIMENTS OF THE INVENTION

[0010] In a media-type disperser of the present invention, a drive shaft, a plurality of stirring blades (stirring discs) that are arranged on the drive shaft and are simultaneously rotatable by the rotation of the drive shaft, and media particles are housed in a cylindrical vessel having a supply port of a liquid mixture and an outlet of a liquid dispersion. Here, in the present invention, the liquid mixture refers to a source material mixture that is passed through the media-type disperser, and the liquid dispersion refers to a dispersion of the source material mixture that has been passed through the media-type disperser, excluding the media. In the media-type disperser of the present invention, the stirring blade nearest to the outlet has a protrusion in a direction parallel to the drive shaft; and the thickness t (mm) of the protrusion, the diameter d (mm) of the media particle, and the distance L (mm) from the outlet side of the stirring blade nearest to the outlet to the rear end of the cylindrical vessel satisfy Formula (1) below.

$$10d \leq t \leq L - 10d \quad (1)$$

[0011] Fig. 1 shows a schematic diagram showing an embodiment of the media-type disperser of the present invention. The media-type disperser includes a drive shaft 2 in a cylindrical vessel 1, and includes, on the drive shaft 2, a plurality of stirring blades 3 that are simultaneously rotatable by the rotation of the drive shaft 2. Here, the center axis of the cylindrical vessel 1 and the drive shaft 2 are preferably coaxial. Each of the plurality of stirring blades 3 may basically be a platelike body of a circle or the like that is rotated by the rotation of the drive shaft 2; among these, a stirring blade 3' nearest to an outlet 6 has a protrusion 10 protruding in a direction parallel to the drive shaft 2. The protrusion is preferably provided on the outlet side of the stirring blade nearest to the outlet. Further, a rotor 7 rotatable by the rotation of the drive shaft 2 and a stator 8, which is a fixed ring, are provided still more on the outlet 6 side than the stirring blade 3' nearest to the outlet 6 in the cylindrical vessel 1. The stator 8 is integrated with a head cover 9 that is provided in a liquid-tight manner to an end portion on the outlet 6 side of the cylindrical vessel 1. Further, not-illustrated media particles

are provided in the cylindrical vessel 1.

[0012] In such a media-type disperser, a liquid mixture is caused to flow from a supply port 4 into a dispersing chamber 5 in which media particles are put, is finely dispersed by the collision etc. of media particles accelerated by the stirring blades 3 that are rotating and becomes a liquid dispersion, and is discharged from the outlet 6 to the outside of the cylindrical vessel 1. At this point, only the liquid dispersion passes through the gap between the rotor 7 and the stator 8, and thereby the liquid dispersion and the media particles can be separated. In Fig. 1, the arrow indicates the flowing direction of the liquid mixture and the liquid dispersion.

[0013] In a media-type disperser of the present invention like the above, the stirring blade nearest to the outlet of a liquid dispersion has a protrusion; thereby, the speed of media particles in a direction parallel to the rotation direction of the drive shaft is increased, and the difference between the peripheral speed of the rotor being rotated by the rotation of the drive shaft and the speed in a direction parallel to the rotation direction of the media particle in the vicinity of the rotor can be reduced; thus, damage due to abrasion of the screen or the gap separator can be suppressed.

[0014] Specifically, for example, when stirring a liquid mixture by means of media particles and separating the media particles from the liquid mixture by means of the gap between the rotor and the stator to obtain a liquid dispersion, the average relative speed V_n between the speed in the rotation direction of the media particle existing in a position up to 1 mm from the stator toward the rotor in a direction parallel to the drive shaft and the speed of the rotor being rotated by the rotation of the drive shaft can be caused to satisfy the relation of Formula (2) below.

$$|V_n| \leq 5 \text{ (m/sec)} \quad (2).$$

[0015] The speed of the media particle encompasses the speeds of all the media particles existing in the position described above and the speed of the rotor refers to the speed on the outer peripheral surface of the rotor, and the average value of the relative speeds between them is taken as the average relative speed V_n . However, when the speeds of the media particles existing in the position described above are not greatly different between the inner side and the outer side with respect to the diameter direction of the drive shaft and representation by media particles in a partial region alone is allowable, such representation may be employed.

[0016] Although the relation of Formula (2) above can be achieved also by reducing the viscosity of the liquid mixture, a dispersing apparatus like that mentioned above allows the relation to be achieved even when it is impossible to reduce the viscosity of the liquid mixture. Then, the frictional resistance between media particles and the stator etc. can be minimized, and the damage of the rotor, the stator, etc. can be suppressed.

[0017] Fig. 2 shows an enlarged view of the stirring blade nearest to the outlet and of the neighborhood of the gap separator in an embodiment of the media-type disperser of the present invention. In the media-type disperser of the present invention, the thickness t (mm) of the protrusion possessed by the stirring blade nearest to the outlet, the diameter d (mm) of the media particle, and the distance L (mm) from the outlet side of the stirring blade nearest to the outlet to the rear end of the cylindrical vessel satisfy Formula (1) below. Here, the thickness t of the protrusion is the length in a direction parallel to the drive shaft, and refers to the thickness of the thickest portion in a direction parallel to the drive shaft. The diameter d of the media particle refers to the number average value of the long diameters of 200 randomly selected media particles. The distance L from the outlet side of the stirring blade nearest to the outlet to the rear end of the cylindrical vessel refers to the distance from the outlet side of the portion excluding the protrusion 10 of the stirring blade 3' nearest to the outlet to the head cover 9.

$$10d \leq t \leq L - 10d \quad (1)$$

[0018] Studies by the present inventors have experimentally found out that, by a protrusion having a thickness t satisfying Formula (1) above, the speed of media particles in a direction parallel to the rotation direction of the stirring blade is increased and the difference between the peripheral speed of the rotor being rotated by the rotation of the drive shaft and the speed in a direction parallel to the rotation direction of the media particle in the vicinity of the rotor can be reduced, and thus damage due to abrasion of the screen or the gap separator (the rotor 7 and the stator 8 in Figs. 1 and 2) can be suppressed. If the thickness t of the protrusion is smaller than $10d$, the effect of the protrusion is not sufficiently obtained, and hence damage due to abrasion of the screen or the gap separator is likely to occur. On the other hand, if the thickness t of the protrusion is larger than $L - 10d$, media particles are likely to be caught in the gap between the protrusion of the stirring blade and the gap separator, and the fluidity of media particles is reduced and the internal pressure rises; consequently, productivity is reduced.

[0019] The thickness t of the protrusion is preferably more than or equal to $L/2$. By setting the thickness t of the protrusion to more than or equal to $L/2$, the speed in a direction parallel to the rotation direction of the media particle is

made larger, and damage due to abrasion of the screen or the gap separator can be suppressed more.

[0020] The distance L from the outlet side of the stirring blade nearest to the outlet to the rear end of the cylindrical vessel is preferably more than or equal to the length of a separation mechanism in a direction parallel to the drive shaft and less than or equal to 1.2 times the length of the separation mechanism, in order to sufficiently obtain the effect of the protrusion. The length of the separation mechanism is the length in the direction of the drive shaft of a rotor in the case of the separation mechanism adopting a gap separator system like that shown in Figs. 1 and 2, and is the length in the direction of the drive shaft of a screen in the case of the separation mechanism adopting a screen system.

[0021] In the present invention, the thickness t (mm) of the protrusion and the distance L (mm) from the outlet side of the portion excluding the protrusion of the stirring blade nearest to the outlet to the rear end of the cylindrical vessel can be measured using vernier calipers. The diameter d (mm) of the media particle can be found by observing media particles with a microscope with a magnification of 100 times, measuring the long diameters of 200 randomly selected media particles, and calculating the number average value of them.

[0022] The protrusion may have any length in the diameter direction of the drive shaft, but it is preferable that the end on the outer peripheral side of the protrusion be provided more on the outer side than the middle position between the center and the outermost periphery of the stirring blade nearest to the outlet. When the end on the outer peripheral side of the protrusion is located more on the outer side than the middle position between the center and the outermost periphery of the stirring blade, the angular velocity of the protrusion is large, and the speed of media particles in a direction parallel to the rotation direction of the protrusion is large; thus, the effect of the protrusion can be sufficiently obtained.

[0023] It is preferable that a plurality of protrusions be provided. When the outer peripheral side of the protrusion has a larger area on the same circumference, the effect of the protrusion can be obtained more, but the passage of the liquid dispersion is reduced and a rise in the internal pressure is caused; thus, the protrusion may be divided into a plurality of pieces, and thereby a rise in the internal pressure can be suppressed. The protrusion more preferably has a tapered shape from the inner side to the outer side in the diameter direction of the stirring blade.

[0024] In the present invention, the media-type disperser preferably has, in the cylindrical vessel, a separation mechanism that separates the media particles and the liquid dispersion. Examples of the separation mechanism include a gap separator, a screen, a slit, a mesh, and the like. Among these, a gap separator system is preferable. Here, the gap separator system is a system that includes a rotor rotatable by the rotation of the drive shaft and a stator, which is a fixed ring, and that separates the liquid dispersion and the media particles by only the liquid dispersion passing through the gap between the rotor and the stator. The separation mechanism adopting a gap separator system can shorten the distance between the stirring blade nearest to the outlet and the separation mechanism; thus, the dispersing region in the cylindrical vessel can be utilized as much as possible, and productivity can be improved more.

[0025] In the present invention, examples of the material of the stirring blade include super hard alloy materials, ceramic materials, metals, materials each with a Young's modulus of 0.4 to 0.6 GPa, and the like. Examples of the super hard alloy material include WC-Co-based alloys, WC-TiC-Co-based alloys, WC-TaC-Co-based alloys, WC-TiC-TaC-Co-based alloys, and the like. Examples of the ceramic material include zirconia, alumina, silicon carbide, aluminum nitride, sialon, zirconia toughened alumina, and the like. Zirconia and zirconia toughened alumina are preferable from the viewpoints of abrasion resistance, the thermal conductivity, and the coefficient of thermal expansion. Examples of the metal include stainless steel, carbon steel, and the like.

[0026] From the viewpoint of more improving abrasion resistance to media particles, filler particles in a liquid mixture, etc., it is preferable that at least part of the stirring blade be made of a material with a Young's modulus of 0.4 to 0.6 GPa, and it is more preferable that a portion that media particles and a liquid mixture are in contact with be made of a material with a Young's modulus of 0.4 to 0.6 GPa. By using a material with a Young's modulus of 0.4 to 0.6 GPa, the abrasion of the stirring blade can be suppressed more, and the dispersibility of a liquid dispersion can be stabilized more. Here, the Young's modulus can be measured by a method prescribed by JIS K7161-1:2014 in the case of resin materials and by a method prescribed by JIS R1602:1995 in the case of ceramic and metal-based materials.

[0027] Examples of the material with a Young's modulus of 0.4 to 0.6 GPa include ultrahigh molecular weight polyethylene, polytetrafluoroethylene, and the like. Among these, ultrahigh molecular weight polyethylene is more preferable because it is light in weight and excellent in maintainability. Here, the ultrahigh molecular weight polyethylene refers generally to polyethylene with a weight-average molecular weight of more than or equal to one million. The molecular weight of the ultrahigh molecular weight polyethylene is preferably less than or equal to seven million.

[0028] The material of a portion of the stirring blade that media particles and a liquid mixture are not in contact with (for example, a portion other than the surface of the stirring blade) is preferably a metal and more preferably stainless steel from the viewpoint of mechanical strength.

[0029] Also the material of the drive shaft for rotating the stirring blade is preferably a metal from the viewpoint of mechanical strength. Those given as examples of the material of the stirring blade are given as the metal. However, the surface of the drive shaft is preferably covered with a ceramic or ultrahigh molecular weight polyethylene from the viewpoint of suppressing the mixing of metal powder due to abrasion into a liquid dispersion. For similar reasons, a

cover of a ceramic or ultrahigh molecular weight polyethylene may be provided on a metal drive shaft.

[0030] Examples of the material of the inner wall of the cylindrical vessel include super hard alloy materials, ceramic materials, stainless steel, materials each with a Young's modulus of 0.4 to 0.6 GPa, and the like. It is preferable that a ceramic material be selected when high thermal conductivity is required and a material with a Young's modulus of 0.4 to 0.6 GPa be selected when higher abrasion resistance is required. Those given as examples of the respective types of material of the stirring blade and the like are given as the super hard alloy material, the ceramic material, and the material with a Young's modulus of 0.4 to 0.6 GPa.

[0031] It is preferable that the inner wall of the cylindrical vessel have a structure capable of introducing a cooling or heating medium and that dispersion be performed while the temperature of the liquid dispersion is controlled by the cooling or heating medium. For example, in the case where the liquid mixture and the liquid dispersion have reactivity, thermal degradability, etc., a cooling medium may be used to keep the temperature of the liquid dispersion less than or equal to a certain temperature, and thereby the quality reduction of the liquid dispersion can be suppressed.

[0032] The media particle receives strong collision energy by the rotation of the stirring blade; thus, particles of zirconia, alumina, or the like are suitably used from the viewpoint of abrasion resistance. In the case of having the separation mechanism adopting a gap separator system, the gap between the rotor and the stator is generally more than or equal to 0.1 mm; thus, the diameter d of the media particle is preferably more than or equal to 0.3 mm from the viewpoint of the protection of the equipment.

[0033] The media-type disperser of the present invention preferably has a metering pump upstream of the supply port of a liquid mixture, and can perform continuous dispersion treatment by continuously supplying a liquid mixture. Further, the dispersibility of a liquid dispersion can be stabilized by quantitatively supplying a liquid mixture. Examples of the metering pump include centrifugal pumps such as a single-stage turbine pump and a bore hole, propeller pumps such as an axial flow pump and a mixed flow pump, viscous pumps such as a single-stage centrifugal pump and a multi-stage centrifugal pump, reciprocating pumps such as a lateral piston pump, a vertical piston pump, a horizontal piston pump, a horizontal plunger pump, a vertical plunger pump, a diaphragm pump, a tube pump, and a wing pump, rotary pumps such as an external gear pump, an internal gear pump, an eccentric screw pump, a vane pump, and a roller pump, and the like.

[0034] Next, a method for producing a liquid dispersion of the present invention is described. As described above, in the case where a conventional media-type disperser is used, as the viscosity of the liquid dispersion becomes higher, the screen or the gap separator has tended to be easily damaged by abrasion. The media-type disperser of the present invention can suppress damage due to abrasion of the screen or the gap separator, and can therefore be suitably used for the production of a liquid dispersion with high viscosity. Thus, the method preferably has a step of dispersing a liquid mixture containing at least filler particles and a solvent by using the media-type disperser described above.

[0035] Examples of filler particles include calcium carbonate particles, calcium phosphate particles, amorphous silica particles, crystalline glass filler, kaolin particles, talc particles, titania particles, alumina particles, silica-alumina composite oxide particles, barium sulphate particles, calcium fluoride particles, lithium fluoride particles, zeolite particles, molybdenum sulfide particles, mica particles, boehmite particles, zirconia particles, magnesium oxide particles, titanium oxide particles, silicon nitride, and the like. It is preferable that, in the liquid dispersion, the filler particles contained in the liquid dispersion be sufficiently dispersed up to the primary particle sizes while maintaining required particle sizes and surface characteristics. The Mohs hardness of the filler particle is preferably more than or equal to 4 in order to suppress surface modification due to pulverization of the filler particle by a media particle. Examples of the filler particle with a Mohs hardness of more than or equal to 4 include alumina particles, amorphous silica particles, magnesium oxide particles, titanium oxide particles, silicon nitride particles, zirconia particles, and the like.

[0036] Here, the Mohs hardness shows the hardness to minerals, and can be measured using a commercially available 10-grade Mohs hardness meter. Specifically, a material of interest and each of the minerals used as the ranks in the 10-grade Mohs hardness meter are rubbed together, and then the presence or absence of a flaw is visually observed. In the case where both of the material and the mineral of a specific grade are flawed or neither of them is flawed, the same grade as the grade of the mineral of the specific grade used is taken as the Mohs hardness of the material. In the case where only either of the material and the mineral of a specific rank is flawed among the minerals of all the grades, a value 0.5 higher than the grade of the mineral of the highest grade, among those used as the grades in the 10-stage Mohs hardness meter, by which the material is not flawed is taken as the Mohs hardness of the material.

[0037] In the case where the liquid dispersion is used for a member that electrical insulating properties are required of, the filler particles are preferably electrical insulating particles. Here, the electrical insulating properties mean that the volume resistivity of the material is more than or equal to $10^{14} \Omega \cdot \text{cm}$. The volume resistivity of the material can be measured by a method prescribed by JIS C2141:1992. Examples of electrical insulating particles include alumina particles, amorphous silica particles, magnesium oxide particles, silicon nitride particles, zirconia particles, and the like.

[0038] Examples of the solvent include Cellosolves such as methyl Cellosolve, ethyl Cellosolve, and butyl Cellosolve; alcohols such as isopropyl alcohol, methyl alcohol, ethyl alcohol, butyl alcohol, normal-propyl alcohol, benzyl alcohol, terpineol, and 3-methoxy-3-methyl-1-butanol; ketones such as methyl ethyl ketone, dioxane, acetone, cyclohexanone,

cyclopentanone, γ -butyrolactone, and N-methyl-2-pyrrolidone; esters such as ethyl lactate, methyl acetate, ethyl acetate, isopropyl acetate, normal-propyl acetate, isobutyl acetate, normal-pentyl acetate, 3-methoxy-3-methyl-1-butanol, 3-methoxy-3-methyl-butyl acetate, propylene glycol 1-monomethyl ether 2-acetate, diethylene glycol monobutyl ether acetate, ethylene glycol monobutyl ether acetate, 2,2,4-trimethyl-1,3-pentanediol monoisobutyrate, and 2,2,4-trimethyl-1,3-pentanediol diisobutyrate; hydrocarbons such as hexane, cyclohexane, toluene, and xylenes; and water and the like. Two or more of these may be contained.

[0039] As described above, as the viscosity of the liquid dispersion becomes higher, the screen or the gap separator tends to be easily damaged by abrasion. Thus, the present invention can obtain a more significant effect and is preferable particularly when it is attempted to obtain a liquid dispersion with a viscosity of more than or equal to 100 mPa·s, etc.

EXAMPLES

[0040] The evaluation of the amount of abrasion of the separation mechanism and the damage of the separation mechanism in each of Examples and Comparative Examples was performed by flow analysis using Eulerian multiphase flow (Granular model). The conditions used for the flow analysis are as follows.

- Density of the liquid mixture: 1,130 kg/m³
- Viscosity of the liquid mixture (that is, the viscosity of the liquid dispersion): 1,800 mPa·s
- True density of media particles: 4,000 kg/m³
- Bulk density of media particles: 2,400 kg/m³
- Rate of filling of media particles in the cylindrical vessel: 80 volume%
- Rate of rotation of the drive shaft: 1,239 rpm
- Diameter of the stirring blade: 185 mm
- Thickness of the stirring blade: 10 mm
- Inner diameter of the cylindrical vessel: 220 mm
- Full length of the cylindrical vessel: 175 mm
- Diameter of the rotation ring (rotor) of the separation mechanism: 125 mm
- Rate of supply of the liquid mixture: 75 kg/hr.

(Evaluation of amount of abrasion of separation mechanism by flow analysis)

[0041] The evaluation was performed by flow analysis. For each of Examples and Comparative Examples, the amount E of abrasion of the separation mechanism was calculated by Formula (3) below from the true density ρ (kg/m³) of media particles in the vicinity of the separation mechanism, the average relative speed V_n (m/s) between the speed of the rotor and the speed in the rotation direction of the media particle in the vicinity of the separation mechanism, the average speed V_r (m/s) in a direction perpendicular to the drive shaft of the media particle in the vicinity of the separation mechanism, the volume fraction α (%) of media particles in the vicinity of the separation mechanism, and a proportionality constant A. However, a relative value with respect to the amount of abrasion of Comparative Example 1 was used on the assumption that the amount of abrasion of Comparative Example 1 was 100. Here, the vicinity of the separation mechanism was, in the case of a gap separator system, a position that was in the cylindrical vessel and up to 1 mm from the stator toward the stirring blade in a direction parallel to the drive shaft, and was, in the case of a screen system, a position that was in the cylindrical vessel and up to 1 mm from the head cover toward the stirring blade in a direction parallel to the drive shaft.

$$E = A \cdot \rho \cdot V_n \cdot V_r \cdot \alpha \quad (3)$$

[0042] The abrasion of the separation mechanism was evaluated by the criteria mentioned below from the value of the amount E of abrasion of the separation mechanism.

Evaluation criteria

[0043] $E \leq 90$: ◎

$90 < E \leq 95$: ○

$95 < E$: ×

(Evaluation of damage of separation mechanism by flow analysis)

[0044] The evaluation was performed by flow analysis. For each of Examples and Comparative Examples, the amount W of the liquid mixture passing through the media-type disperser until damage occurred was calculated by Formula (4) below, which was found experimentally. However, a relative value with respect to the value of Comparative Example 1 of 100 was used.

$$W = 1/E \quad (4)$$

[0045] The damage of the separation mechanism was evaluated by the criteria mentioned below from the value of the amount W of the liquid mixture passing through the media-type disperser until damage occurred.

Evaluation criteria

[0046] $111 \leq W$: ◎

$105 \leq W < 111$: ○

$W < 105$: ×

(Evaluation of abrasion of stirring blade)

[0047] A disc with a diameter of 80 mm and a thickness of 10 mm was produced using the material for forming the stirring blade used in each of Examples and Comparative Examples. The obtained disc was applied to stirring for 30 hours at a rate of rotation of 2,000 rpm in a container in which 400 g of water and 1,440 g of media particles with a true density of 4,000 kg/m³ were housed, using Three-One Motor ST-200 manufactured by AS ONE Corporation. For the disc after stirring, the presence or absence of abrasion or deformation was visually observed, and the abrasion of the stirring blade was evaluated by the criteria mentioned below.

Evaluation criteria

[0048] There was no abrasion/deformation: ○

There was abrasion: △

There was deformation: ×.

(Evaluation of productivity)

[0049] A liquid mixture in which a polyvinylidene fluoride-hexafluoropropylene copolymer (PVdF/HFP = 92/8 (weight ratio), with a weight-average molecular weight of one million), alumina particles (average particle size: 0.5 μm; volume resistivity: 10¹⁵ Ω·cm), and N-methyl-2-pyrrolidone were blended at a weight ratio of 5:12:83, respectively, was passed through a media-type disperser of the configuration shown in each of Examples and Comparative Examples, via a diaphragm pump. The stirring blade was rotated under the condition of a peripheral speed of 12 m/s, and productivity was evaluated by the criteria mentioned below from the residence time in the disperser vessel until the average particle size of the alumina particle in the liquid dispersion reached 0.5 μm. The average particle size of the alumina particle in the liquid dispersion was measured using MT-3300 manufactured by MicrotracBEL Corp.

Evaluation criteria

[0050] The residence time being less than 10 minutes: ○

The residence time being more than or equal to 10 minutes: ×.

(Evaluation of abrasion of rotor)

[0051] Traces of beads abrasion on the rotor after 0.8 t of a liquid mixture was continuously passed under the same conditions as those of the evaluation of productivity described above were evaluated by visual inspection.

(Evaluation of amount of abrasion of stator)

[0052] The shape of the stator after 0.8 t of a liquid mixture was continuously passed under the same conditions as those of the evaluation of productivity described above was measured using VR-3000 manufactured by KEYENCE Corporation, and was evaluated by the cross-sectional area of the abrasion portion.

(Example 1)

[0053] In a media-type disperser of the configuration shown in Figs. 1 and 2, the thickness t of the protrusion of the stirring blade nearest to the outlet was set to 19 mm, the diameter d of the media particle was set to 0.5 mm, the distance L from the outlet side of the portion excluding the protrusions of the stirring blade nearest to the outlet to the rear end of the cylindrical vessel was set to 24 mm, the distance r from the center of the stirring blade to the end on the outer peripheral side of the protrusion was set to 74 mm, and the number of protrusions of the stirring blade was set to 4; and flow analysis was performed using Eulerian multiphase flow. Here, as shown in Fig. 1, the head cover 9 was located at the rear end of the cylindrical vessel 1. Ultrahigh molecular weight polyethylene (UHMwPE) with a Young's modulus of 0.5 GPa was used as the material of the stirring blade. The results of evaluation performed by the methods described above are shown in Table 1. Further, Fig. 6 shows a photograph of the rotor after the evaluation of productivity; on this rotor, no traces of beads abrasion were seen.

(Example 2)

[0054] The procedure was similar to that of Example 1 except that the material of the stirring blade was changed to zirconia with a Young's modulus of 200 GPa. The results are shown in Table 1.

(Example 3)

[0055] The procedure was similar to that of Example 2 except that the separation mechanism was changed to the one adopting a rotary screen system, the thickness t of the protrusion 10 of the stirring blade 3' nearest to the outlet 6 was changed to 34 mm, and the distance L from the outlet side of the portion excluding the protrusions of the stirring blade nearest to the outlet to the rear end of the cylindrical vessel was changed to 39 mm (see Fig. 3). The results are shown in Table 1.

(Example 4)

[0056] The procedure was similar to that of Example 1 except that the material of the stirring blade was changed to low density polyethylene (LDPE) with a Young's modulus of 0.3 GPa. The results are shown in Table 1.

(Example 5)

[0057] The procedure was similar to that of Example 1 except that the material of the stirring blade was changed to high density polyethylene (HDPE) with a Young's modulus of 0.7 GPa. The results are shown in Table 1.

(Example 6)

[0058] The procedure was similar to that of Example 1 except that the thickness t of the protrusion of the stirring blade nearest to the outlet was changed to 15 mm. The results are shown in Table 2.

(Example 7)

[0059] The procedure was similar to that of Example 1 except that the thickness t of the protrusion of the stirring blade nearest to the outlet was changed to 11 mm. The results are shown in Table 2.

(Example 8)

[0060] The procedure was similar to that of Example 1 except that the thickness t of the protrusion of the stirring blade nearest to the outlet was changed to 10 mm and the diameter d of the media particle was changed to 1.0 mm. The results are shown in Table 2.

(Example 9)

[0061] The procedure was similar to that of Example 1 except that the distance r from the center of the stirring blade to the end on the outer peripheral side of the protrusion was changed to 69 mm. The results are shown in Table 2.

(Example 10)

[0062] The procedure was similar to that of Example 1 except that the distance r from the center of the stirring blade to the end on the outer peripheral side of the protrusion was changed to 47 mm. The results are shown in Table 2.

(Example 11)

[0063] The procedure was similar to that of Example 1 except that the distance r from the center of the stirring blade to the end on the outer peripheral side of the protrusion was changed to 23 mm. The results are shown in Table 2.

(Comparative Example 1)

[0064] The procedure was similar to that of Example 1 except that no protrusion was provided on the stirring blade nearest to the outlet (see Fig. 4). The results are shown in Table 3. Further, Fig. 7 shows a photograph of the rotor after the evaluation of productivity; on this rotor, traces of beads abrasion were seen.

(Comparative Example 2)

[0065] The procedure was similar to that of Example 1 except that the thickness t of the protrusion of the stirring blade nearest to the outlet was changed to 4 mm. The results are shown in Table 3.

(Comparative Example 3)

[0066] The procedure was similar to that of Example 1 except that the thickness t of the protrusion of the stirring blade nearest to the outlet was changed to 5 mm and the diameter d of the media particle was changed to 2.0 mm. The results are shown in Table 3.

(Comparative Example 4)

[0067] The procedure was similar to that of Example 1 except that the separation mechanism was changed to the one adopting a fixed screen system, the thickness t of the protrusion 10 of the stirring blade 3' nearest to the outlet 6 was set to 50 mm, the distance L from the outlet side of the portion excluding the protrusions of the stirring blade nearest to the outlet to the rear end of the cylindrical vessel was set to 52 mm, and a structure in which the protrusion 10 is extended so as to cover a screen 7' was employed (see Fig. 5). The results are shown in Table 3. The protrusion of the stirring blade was longer, and the area of the effective dispersing region was reduced; consequently, the residence time to obtain a desired particle size was longer, and this resulted in a reduction in productivity.

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[Table 1]

[Table 1]						
		Example 1	Example 2	Example 3	Example 4	Example 5
Configuration of media-type disperser	Thickness t of protrusion (mm)		19	19	34	19
	Distance L from stirring blade to rear end of cylindrical vessel (mm)		24	24	39	24
	Diameter d of media particle (mm)		0.5	0.5	0.5	0.5
	Distance r from center of stirring blade to protrusion (mm)		74	74	74	74
	Number of protrusions		4	4	4	4
	Separation mechanism		Gap separator system	Gap separator system	Screen system	Gap separator system
	Stirring blade	Material	UHM _w PE	Zirconia	Zirconia	LDPE
		Young's modulus (GPa)	0.5	200	200	0.3
Results of flow analysis	Density ρ of media particles (kg/m ³)		4000	4000	4000	4000
	Average relative speed Vn (m/s)		4.3	4.3	4.3	4.3
	Average speed Vr in direction perpendicular to drive shaft of media particle (m/s)		1.4	1.4	1.4	1.4
	Volume fraction α of media particles (%)		48	48	48	48
	Amount of abrasion of separation mechanism (E)		⊙ (84)	⊙ (84)	⊙ (84)	⊙ (84)
	Damage of separation mechanism (W)		⊙ (119)	⊙ (119)	⊙ (119)	⊙ (119)
Abrasion/deformation of stirring blade ○ : absent, Δ : abrasion, × : deformation		○	Δ	Δ	Δ	Δ
Productivity (average residence time (min))		○ (7)	○ (7)	○ (9)	○ (7)	○ (7)
Beads abrasion of rotor		Absent	-	-	-	-
Amount of abrasion of stator (mm ²)		0.01	-	-	-	-

[Table 2]

[Table 2]		Example 6	Example 7	Example 8	Example 9	Example 10	Example 11
Configuration of media-type disperser	Thickness t of protrusion (mm)	15	11	10	19	19	19
	Distance L from stirring blade to rear end of cylindrical vessel (mm)	24	24	24	24	24	24
	Diameter d of media particle (mm)	0.5	0.5	1.0	0.5	0.5	0.5
	Distance r from center of stirring blade to protrusion (mm)	74	74	74	69	47	23
	Number of protrusions	4	4	4	4	4	4
	Separation mechanism	Gap separator system	Gap separator system	Gap separator system	Gap separator system	Gap separator system	Gap separator system
Results of flow analysis	Stirring blade	Material	UHM _w PE	UHM _w PE	UHM _w PE	UHM _w PE	UHM _w PE
		Young's modulus (GPa)	0.5	0.5	0.5	0.5	0.5
	Density ρ of media particles (kg/m ³)	4000	4000	4000	4000	4000	4000
	Average relative speed Vn (m/s)	4.5	4.8	4.8	4.5	4.8	5.0
	Average speed Vr in direction perpendicular to drive shaft of media particle (m/s)	1.4	1.4	1.4	1.4	1.4	1.4
	Volume fraction α of media particles (%)	48	48	48	48	48	48
Abrasion/deformation of stirring blade	Amount of abrasion of separation mechanism (E)	○ (88)	○ (94)	○ (95)	○ (88)	○ (94)	× (98)
	Damage of separation mechanism (W)	○ (114)	○ (106)	○ (105)	○ (113)	○ (106)	× (102)
	Abrasion/deformation of stirring blade ○ : absent, Δ : abrasion, × : deformation	○	○	○	○	○	○

(continued)

[Table 2]						
	Example 6	Example 7	Example 8	Example 9	Example 10	Example 11
Productivity (average residence time (min))	○ (7)	○ (7)	○ (7)	○ (7)	○ (7)	○ (7)
Beads abrasion of rotor	-	-	-	-	-	-
Amount of abrasion of stator (mm ²)	-	-	-	-	-	-

[Table 3]

[Table 3]						
			Comparative Example 1	Comparative Example 2	Comparative Example 3	Comparative Example 4
Configuration of media-type disperser	Thickness t of protrusion (mm)		0	4	5	50
	Distance L from stirring blade to rear end of cylindrical vessel (mm)		24	24	24	52
	Diameter d of media particle (mm)		0.5	0.5	2.0	0.5
	Distance r from center of stirring blade to protrusion (mm)		-	74	74	74
	Number of protrusions		-	4	4	4
	Separation mechanism		Gap separator system	Gap separator system	Gap separator system	Screen system
	Stirring blade	Material	UHMw PE	UHMw PE	UHMw PE	Zirconia
		Young's modulus (GPa)	0.5	0.5	0.5	200
Results of flow analysis	Density ρ of media particles (kg/m³)		4000	4000	4000	4000
	Average relative speed Vn (m/s)		5.5	5.5	5.4	10.0
	Average speed Vr in direction perpendicular to drive shaft of media particle (m/s)		1.3	1.3	1.3	1.8
	Volume fraction α of media particles (%)		48	48	48	48
	Amount of abrasion of separation mechanism (E)		× (100)	× (100)	× (98)	× (252)
	Damage of separation mechanism (W)		× (100)	× (100)	× (102)	× (40)
Abrasion/deformation of stirring blade ○ : absent, Δ: abrasion, x: deformation			○	○	○	Δ
Productivity (average residence time (min))			○ (7)	○ (7)	○ (7)	× (12)
Beads abrasion of rotor			Present	-	-	-
Amount of abrasion of stator (mm²)			0.06	-	-	-

INDUSTRIAL APPLICABILITY

[0068] The present invention can be suitably used to finely disperse a liquid mixture containing filler particles and a solvent; for example, can be suitably used for a dispersion of slurry for a separator of a battery, or the like.

DESCRIPTION OF REFERENCE SIGNS

[0069]

- 5 1: Cylindrical vessel
- 2: Drive shaft
- 3: Stirring blade
- 3': Stirring blade nearest to an outlet
- 4: Supply port
- 10 5: Dispersing chamber
- 6: Outlet
- 7: Rotor
- 7': Screen
- 8: Stator
- 15 9: Head cover
- 10: Protrusion

Claims

- 20
1. A media-type disperser that is a media-type wet disperser in which a drive shaft, a plurality of stirring blades that are arranged on the drive shaft and are simultaneously rotatable by rotation of the drive shaft, and media particles are housed in a cylindrical vessel having a supply port of a liquid mixture and an outlet of a liquid dispersion, wherein the stirring blade nearest to the outlet has a protrusion in a direction parallel to the drive shaft, and a thickness t (mm) of the protrusion, a diameter d (mm) of the media particle, and a distance L (mm) from the outlet side of the stirring blade nearest to the outlet to a rear end of the cylindrical vessel satisfy Formula (1) below,
- 25

$$10d \leq t \leq L - 10d \quad (1).$$

- 30
2. The media-type disperser according to claim 1, wherein the protrusion is provided such that an end on an outer peripheral side is more on an outer side than a middle position between a center and an outermost periphery of the stirring blade in a diameter direction of the drive shaft.
- 35
3. The media-type disperser according to claim 1 or 2, comprising a plurality of protrusions.
4. The media-type disperser according to any one of claims 1 to 3, comprising, in the cylindrical vessel, a separation mechanism adopting a gap separator system for separation between media particles and a liquid dispersion.
- 40
5. The media-type disperser according to any one of claims 1 to 4, wherein at least part of the stirring blade is made of a material with a Young's modulus of 0.4 to 0.6 GPa.
- 45
6. The media-type disperser according to claim 5, wherein the material with a Young's modulus of 0.4 to 0.6 GPa is ultrahigh molecular weight polyethylene.
7. The media-type disperser according to any one of claims 1 to 6, wherein at least part of the drive shaft and part of the stirring blade are made of metal.
- 50
8. A method for producing a liquid dispersion, comprising: a step of dispersing a liquid mixture containing at least filler particles each with a Mohs hardness of more than or equal to 4 and a solvent by using the media-type disperser according to any one of claims 1 to 7.
9. The method for producing a liquid dispersion according to claim 8, wherein the filler particles are insulating particles.
- 55
10. The method for producing a liquid dispersion according to claim 8 or 9, wherein a viscosity of the liquid dispersion is more than or equal to 100 mPa·s.

11. A method for producing a liquid dispersion, the method being a method of producing a liquid dispersion by stirring a liquid mixture by means of media particles and separating the media particles from the liquid mixture by means of a gap between a rotor being rotated by rotation of a drive shaft and a stator, wherein an average relative speed V_n between a speed of the rotor and a speed in a rotation direction of the media particle existing in a position up to 1 mm from the stator toward the rotor in a direction parallel to the drive shaft satisfies Formula (2) below,

$$|V_n| \leq 5 \text{ (m/sec)} \quad (2) .$$

Fig. 1

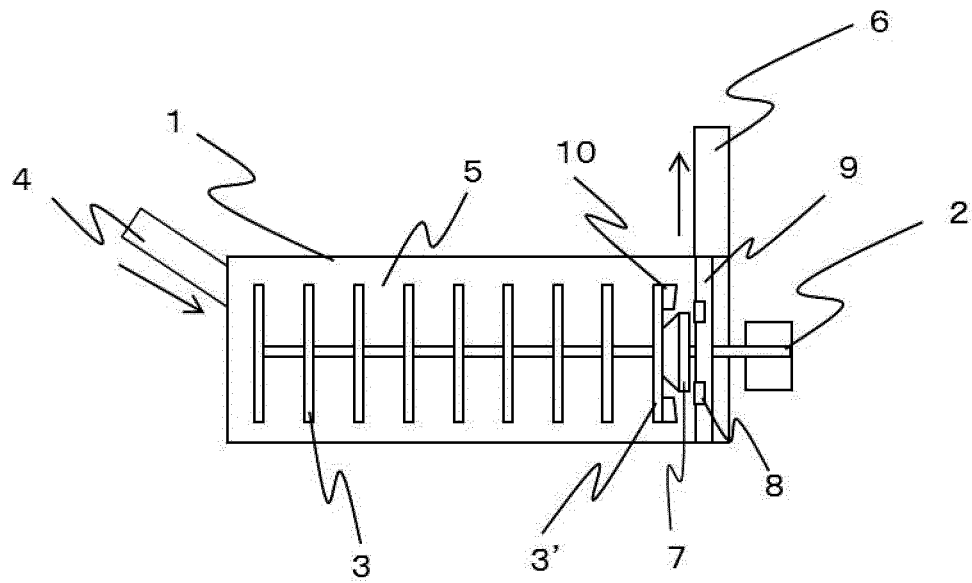


Fig. 2

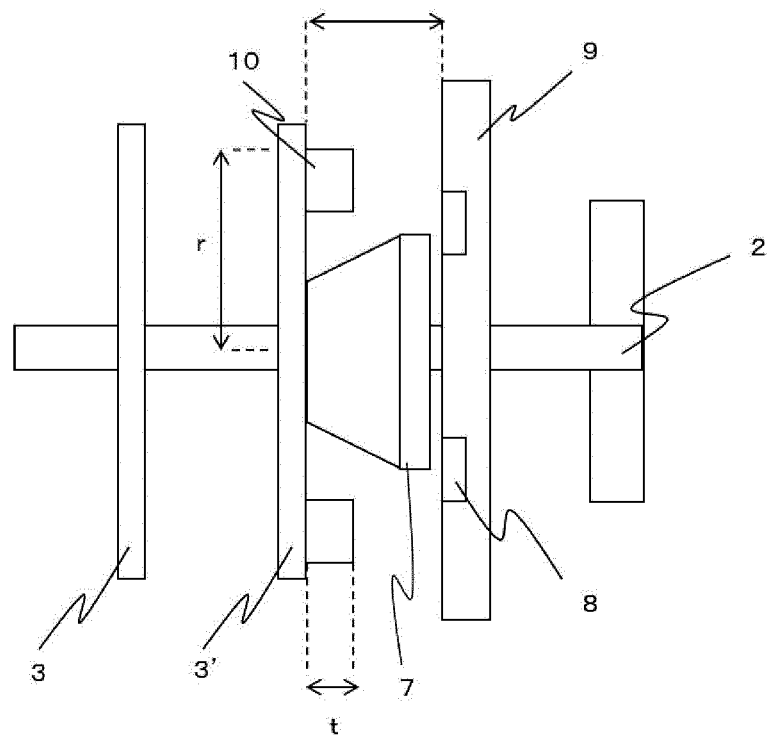


Fig.3

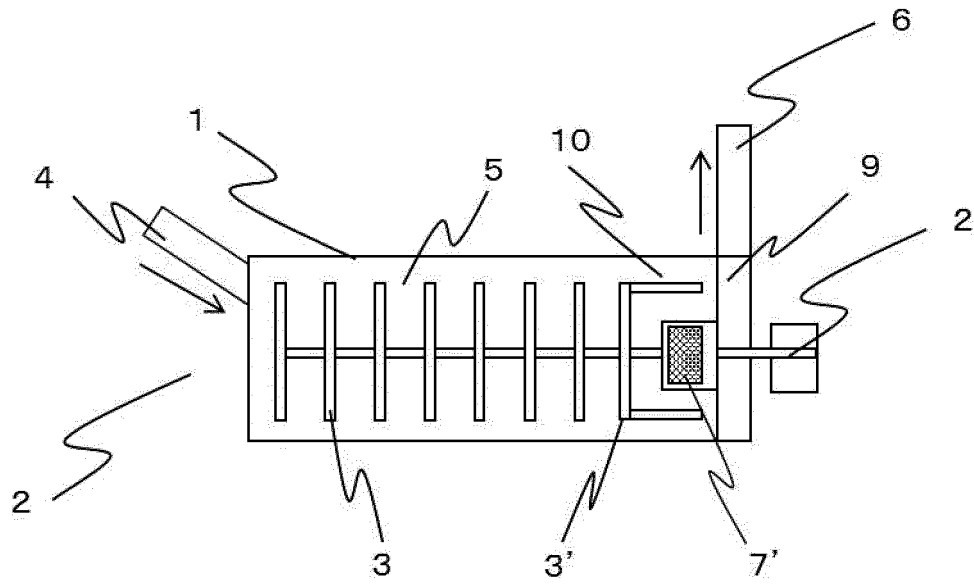


Fig. 4

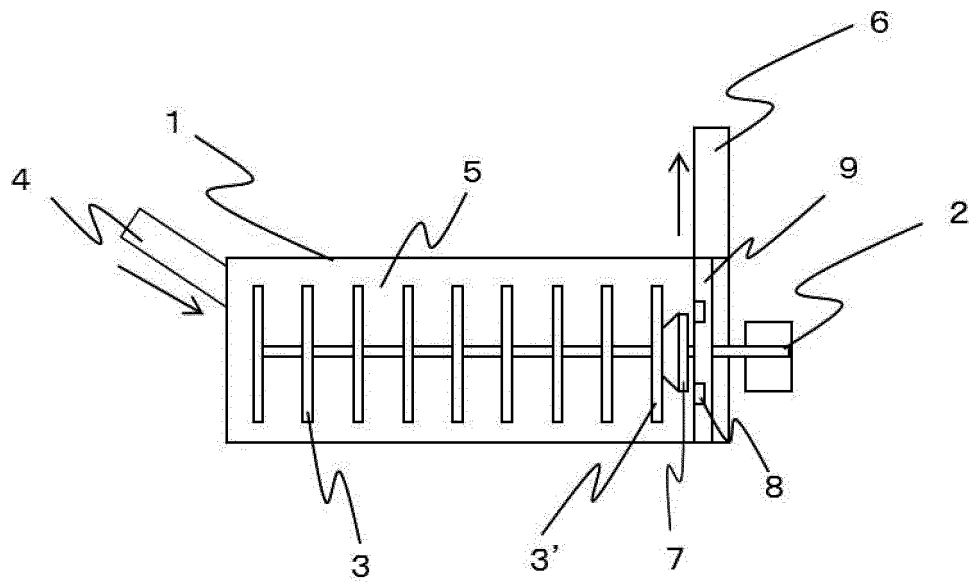


Fig. 5

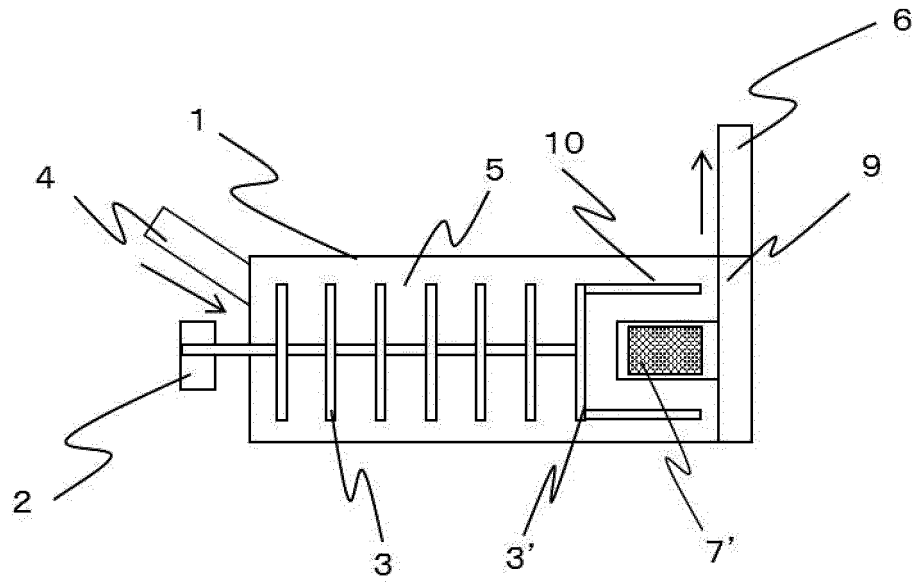


Fig. 6

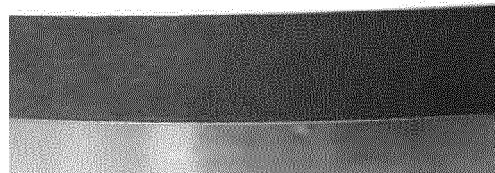
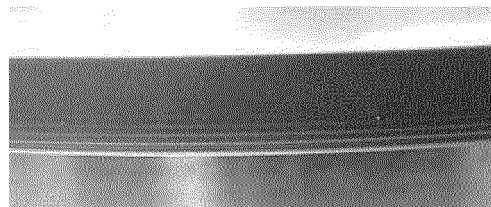


Fig. 7



INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2018/036638

A. CLASSIFICATION OF SUBJECT MATTER

Int. Cl. B02C17/16 (2006.01) i, B01F3/12 (2006.01) i, B01F7/00 (2006.01) i, B02C17/00 (2006.01) i, B02C17/20 (2006.01) i

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)

Int. Cl. B02C17/16, B01F3/12, B01F7/00, B02C17/00, B02C17/20

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

Published examined utility model applications of Japan 1922-1996

Published unexamined utility model applications of Japan 1971-2018

Registered utility model specifications of Japan 1996-2018

Published registered utility model applications of Japan 1994-2018

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	JP 2014-42867 A (SUMITOMO OSAKA CEMENT CO., LTD.)	1-4, 11
Y	13 March 2014, claims, paragraphs [0002], [0029], [0036]-[0050], fig. 1-6 (Family: none)	4-10
Y	JP 2005-292422 A (ZEON CORPORATION) 20 October 2005, claims, paragraphs [0007], [0038] (Family: none)	4-10
Y	JP 60-227821 A (GENERAL SIGNAL CORP.) 13 November 1985, claims, page 5, upper right column, line 12 to lower right column, line 7 & US 4571090 A, claims, column 3, lines 28-64 & EP 158048 A2 & KR 920000538 B1	5-10



Further documents are listed in the continuation of Box C.



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Date of the actual completion of the international search
13.11.2018

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2018/036638

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	JP 2011-147866 A (NIPPON COKE & ENGINEERING CO., LTD.) 04 August 2011, paragraph [0046], fig. 1 (Family: none)	7-10
Y	JP 6-277479 A (TIOXIDE SPECIALTIES LTD.) 04 October 1994, claims, paragraphs [0017]-[0023] & EP 611738 A1, claims, column 4, line 49 to column 6, line 15	8-10
Y	JP 2005-111357 A (MITSUI MINING CO., LTD.) 28 April 2005, paragraphs [0053], [0054] (Family: none)	8-10
Y	JP 11-179179 A (RICOH CO., LTD.) 06 July 1999, paragraphs [0024]-[0044] (Family: none)	10
A	JP 2005-169340 A (AIMEX CO., LTD.) 30 June 2005 (Family: none)	1-11

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REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- JP H724287 B [0004]
- JP 2000171931 A [0004]