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Declarations under Rule 4.17:

— *as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii))*

— *of inventorship (Rule 4.17(iv))*

[Continued on next page]

(54) Title: VACUUM FILTRATION METHOD

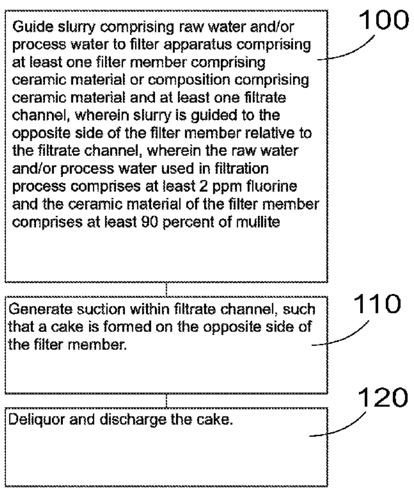

FIG. 6

(57) Abstract: A method of vacuum filtration comprises guiding (100) a slurry comprising raw water and/or process water to a filter apparatus (2) comprising at least one filter member (3) comprising a ceramic material or a composition comprising a ceramic material and at least one filtrate channel (7), wherein the slurry is guided to the opposite side of the filter member relative to the filtrate channel; generating (110) suction within the filtrate channel, such that a cake (5) is formed on the opposite side of the filter member; and deliquoring and discharging (120) the cake. The raw water and/or process water used in the filtration process comprises at least 2 ppm fluorine and the ceramic material of the filter member (3) comprises at least 90 percent of mullite.

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— *with international search report (Art. 21(3))*

— *before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments (Rule 48.2(h))*

Vacuum Filtration Method

FIELD OF THE INVENTION

The invention relates to filtration, and more particularly to a method of filtering solid particles from a fluid.

One of the disadvantages associated with filtration is the problem with filter plate breakages. A special problem rises from a broken filter plate breaking other filter plates in a domino effect type of a manner.

BACKGROUND

Filtration is a widely used process whereby a slurry or solid liquid mixture is forced through a filter member, with the solids retained on the filter member and the liquid phase passing through. This process is generally well understood in the industry. Examples of filtration types include depth filtration, pressure and vacuum filtration, and gravity and centrifugal filtration.

The most commonly used filter media for vacuum filters are filter cloths and coated media, e.g. the ceramic filter medium.

The use of a cloth filter medium requires heavy duty vacuum pumps, due to vacuum losses through the cloth during cake deliquoring. The ceramic filter medium, when wetted, does not allow air to pass through due to a capillary action. This decreases the necessary vacuum level, enables the use of smaller vacuum pumps and, consequently, yields significant energy savings.

BRIEF DESCRIPTION OF THE INVENTION

Viewed from an aspect, there can be provided a method of vacuum filtration comprising guiding a slurry comprising raw water and/or process water to a filter apparatus comprising at least one filter member comprising a ceramic material or a composition comprising a ceramic material and at least one filtrate channel, wherein the slurry is guided to the opposite side of the filter member relative to the filtrate channel; generating suction within the filtrate channel, such that a cake is formed on the opposite side of the filter member; and deliquoring and discharging the cake; and wherein the raw water and/or process water used in the filtration process comprises at least 2 ppm fluorine and the ceramic material of the filter member comprises at least 90 percent of mullite. Thereby a method can be provided, where a longer lifecycle of the filter elements in a process comprising fluorine can be achieved.

Some other embodiments are characterised by what is stated in the other claims. Inventive embodiments are also disclosed in the specification and drawings of this patent application. The inventive content of the patent application may also be defined in other ways than defined in the following claims. The inventive content may also be formed of several separate inventions, especially if the invention is examined in the light of expressed or implicit sub-tasks or in view of obtained benefits or benefit groups. Some of the definitions contained in the following claims may then be unnecessary in view of the separate inventive ideas. Features of the different embodiments of the invention may, within the scope of the basic inventive idea, be applied to other embodiments.

BRIEF DESCRIPTION OF THE DRAWINGS

In the following the invention will be described in greater detail by means of preferred embodiments with reference to the accompanying drawings, in which

Figure 1 is a perspective top view illustrating a disc filter apparatus;

Figure 2 is a side view illustrating the disc filter apparatus shown in Figure 1;

Figure 3 is a perspective top view illustrating another filter apparatus;

Figure 4 illustrates in a side view yet another filter apparatus;

Figures 5a to 5e illustrate phases of filtration seen on a filter element level; and

Figure 6 illustrates a method of vacuum filtration.

DETAILED DESCRIPTION OF THE INVENTION

Vacuum filtration is, thus, used for separating solid particles and liquids in a slurry or a solid liquid mixture. The cake formation in vacuum filtration is based on generating suction within the filtrate channels. The most commonly used type of a filter member for vacuum filters are filter cloths and coated media, such as a ceramic filter medium. Although several types of vacuum filter apparatuses 2 exist ranging from belt filters to drums, the specifics of rotary vacuum disc filters are explained here as an example.

Rotary vacuum disc filter apparatuses are typically used for the filtration of relatively free filtering suspensions on a large scale, such as the dewatering of mineral concentrates. The dewatering of mineral concentrates requires large capacity in addition to producing a cake with low moisture content. The vacuum disc filter may comprise a plurality of filter discs arranged in line coaxially around a central pipe or shaft. Each filter disc may be formed of a number of individual filter sectors, called filter plates, that are mounted circumferentially in a radial plane around the central pipe or shaft to form the filter disc, and as the shaft is fitted so as to revolve, each filter plate or sector is, in its turn, displaced into a slurry basin and further, as the shaft of rotation revolves, rises out of the basin. When the filter member is submerged in the slurry basin where, under the influence of the vacuum, the cake forms onto the filter member. Once the filter sector or plate comes out of the basin, the pores are emptied as the cake is deliquored for a predetermined time which is essentially limited by the rotation speed of the disc. The cake can be discharged by a back-pulse of air or by scraping, after which the cycle begins again. Whereas the use of a cloth filter member requires heavy duty vacuum pumps, due to vacuum losses through the cloth during cake deliquoring, the ceramic filter member, when wetted, does not allow air to pass through which does not allow air to pass through, which further decreases the necessary vacuum level, enables the use of smaller vacuum pumps and, consequently, yields significant energy savings.

Figure 1 is a perspective top view illustrating a disc filter apparatus, and Figure 2 is a side view illustrating the disc filter apparatus shown in Figure 1.

The disc filter apparatus comprises a filter 15 consisting of several consecutive coaxial filter discs arranged in line coaxially around the central shaft 21 of the filter 15.

The filter 15 is supported by bearings on a frame of the filter apparatus and is rotatable about the longitudinal axis of the central shaft 21 such that the lower portion of the filter 15 is submerged in a slurry basin located below the filter 15. The filter is rotated by e.g. an electric motor.

The number of the filter discs may range from 2 to 20, for example. The filter apparatus shown in Figure 1 comprises twelve (12) filter discs. The outer diameter of the filter 15 may be ranging from 1.5 m to 4 m, for example. Examples of commercially available disc filters include Ceramec CC filters, models CC-6, CC-15, CC-30, CC-45, CC-60, CC-96 and CC-144 manufactured by Outotec Inc.

All the filter discs can be preferably essentially similar in structure. Each filter disc may be formed of a number of individual sector-shaped filter elements 1. The filter elements 1 are mounted circumferentially in a radial planar plane around the central shaft 21 to form an essentially continuous and planar disc surface. The number of the filter plates in one filter disc may be 12 or 15, for example.

As the central shaft 21 is fitted so as to revolve, each filter element 1 is, in its turn, displaced into a slurry basin and further, as the central shaft 21 revolves, rises out of the basin. As the filter member 3 is submerged in the slurry basin, the cake forms onto the filter member 3 under the influence of the vacuum. Once the filter element 1 comes out of the basin, pores of the filter member 3 are emptied as the cake is deliquored for a predetermined time which is essentially limited by the rotation speed of the disc. The cake can be discharged by e.g. scraping, after which the cycle begins again.

Operation of the disc filter apparatus may be controlled by a filter control unit, such as a Programmable Logic Controller, PLC.

Figure 3 is a perspective top view illustrating another filter apparatus 2. The filter apparatus 2 shown here is a drum filter apparatus comprising a drum-like filter 15. It is to be noted that the filter apparatus 2 is shown by dash lines in Figure 3 in order to clarify the structure of the filter 15.

In the drum filter apparatus the filter element 1 is a part of outer surface of the filter 15. The diameter of the filter 15 may be e.g. in range of 1.8 m – 4.8 m and length in axial direction 1 m – 10 m. The surface area of the filter 15 may be e.g. in range of 2 – 200 m2.

Examples of commercially available drum filters include CDF-6/1.8 manufactured by Outotec Inc.

Function of drum filter apparatus has already described in background part of this description.

Figure 4 illustrates in a side view yet another filter apparatus 2. The filter apparatus 2 here is a vacuum belt filter apparatus.

The filter 15 of the vacuum belt filter apparatus comprises an end-less belt comprising a multitude of individual filter elements 1 arranged one after another in the longitudinal direction of the belt. The filter elements 1 follow one after another along the whole length of the belt, but for sake of simplicity, all vacuum boxes have not been illustrated.

The filter element 1 comprises a vacuum box into which vacuum or underpressure is applied. The filter element 1 is submerged in the slurry basin and the cake forms onto the filter member 3 by the influence of the underpressure in the vacuum box.

The cake can be discharged by e.g. scraping, after which the cycle begins again.

In different types of vacuum filter apparatuses 2, the filter element 1, such as the filter plate, is affected by slurry particles and extraneous compounds, especially in the field of dewatering of mineral concentrates, and as the replacement of a plate can be expensive, the regeneration of the filter member becomes a critical factor when the time-in-operation of an individual filter plate needs to be increased. The filter member is periodically regenerated with the use of one or more of three different methods, for example: backwashing, ultrasonic cleaning, and acid washing. Whereas the regenerative effect of backwashing and ultrasound are more or less mechanical, regeneration with acids is based on chemistry. As another benefit of a ceramic filter member, the ceramic filter plate is mechanically and chemically more durable than, for example, filter cloths and can, thus, withstand harsh operating conditions and possible regeneration better than other types of filter members. These attributes allow for chemical regeneration of the filter plates with acids, whereas a cloth would have to be discarded, after being blinded by particles, and replaced several times during a year's operation.

Acid washing may especially problematic from regeneration point of view in filtering environments comprising fluorine, for instance in the raw water or process water. The fluorine reacts with the acids forming compounds such as hydrofluoric acid that may accelerate the wear of the filter plates considerably. There is a significant risk that breakage of one plate causes domino effect as the pieces of the broken plate may break neighbouring filter plates and so on, which may lead to a massive amount of broken filter plates and the filter apparatus may be out of operation for many days. In the worst case this might cost hundreds of thousands of dollars for the user due to cost of the filter plates and the shutdown time of the process.

Figures 5a to 5e illustrate phases of filtration seen on a filter element 1 level. Each filter element 1 moves in its turn into and through a basin 4. Thus, each filter element 1 goes through four different process phases during one rotation of the filter 15. In a cake forming phase, the liquid is passing through the element 1 when it travels through the slurry and into a filtrate channel 7, and a cake 5 is formed on the outer surface of the filter member 3, as illustrated in Figure 5a. Although each filter element 1 in Figures 5a to 5e comprises a structure comprising filter members on two opposite sides of the filter element 1, in some embodiments the filter element 1 may only comprise a filter member 3 on one side.

The filter element 1 enters the cake drying phase illustrated in Figure 5b after it leaves the basin 4. If cake washing is required, it is typically done in the beginning of the drying phase. In the cake discharge phase illustrated in Figure 5c the cake 5 is scraped off by scrapers 6. A thin cake 5 may be left on the filter elements 1 due to a gap between the scraper 6 and the surface of the filter member 3. In the backwash phase, also called backflush, of each rotation, water (filtrate) is pumped in a reverse direction through the filter element 1, as illustrated in Figure 5d. The backwash water washes off the residual cake and cleans the pores of the filter plate. Proper backwash is important for the filter operation. Backwash pressure may range from about 0.9 bar up to 2.5 bar, for example, depending on the application and the size of the filter discs and so on. As illustrated in Figure 5e, the filter element 1 is also periodically regenerated with the use of at least one of the following methods: backwashing, ultrasonic cleaning, and acid washing. A combined wash using both acid washing and ultrasonic cleaning is the most effective. The regeneration may be performed 1 to 3 times per day, for example.

Figure 6 illustrates a method of vacuum filtration. The method may comprise guiding 100 a slurry comprising raw water and/or process water to a filter apparatus 2 comprising at least one filter member 3 comprising a ceramic material or a composition comprising a ceramic material and at least one filtrate channel 7, wherein the slurry is guided to the opposite side of the filter member 3 relative to the filtrate channel 7. The method may further comprise generating 110 suction within the filtrate channel 7, such that a cake is formed on the opposite side of the filter member 3, and deliquoring and discharging 120 the cake. In the method, the raw water and/or process water used in the filtration process may comprise at least 2 ppm fluorine. In the method, the ceramic material of the filter member comprises at least 90 percent of mullite. In other words, according to an aspect, such a method of vacuum filtration may be used in an environment comprising raw water and/or process water used in the filtration process and comprising at least 2 ppm of fluorine, and the ceramic material of the filter member 3 may comprise at least 90 percent of mullite. According to an embodiment, the ceramic material of the filter member 3 comprises at least 95 percent of mullite. The use of a filter element 1 with a filter member 3 comprising a high percentage of mullite is beneficial in environments comprising fluorine as mullite is particularly tolerant to degradation caused by fluorine and fluorine compounds. Thereby, the expensive filtering of the fluorine from the raw water and/or the process water can also be avoided.

According to an embodiment, the raw water and/or process water may comprise 2 to 50 ppm of fluorine. According to a further embodiment, the raw water and/or process water may comprise 5 to 50 ppm of fluorine. The use of a filter element 1 with a filter member 3 comprising a high percentage of mullite is particularly beneficial in such processes where the high amount of fluorine often causes considerable degradation of the filter elements 1 comprising conventional materials.

According to an embodiment, the method may further comprise acid washing the filter member 3. According to an embodiment, at least one of the following acids is used in the acid washing: nitric acid, oxalic acid and sulphuric acid. According to a further embodiment, the pH of the acid washing medium may be less than or equal to 2. Acid washing may be particularly beneficial in connection with use of filter members 3 that are otherwise challenging to clean to a sufficient degree, such as in connection with microporous filter members 3 like filter members 3 forming a capillary filter, as the acid washing ensures a sufficient grade of degeneration of the filter element 1.

According to an embodiment, the method is used for filtration of at least one of the following: ferrochrome, iron ore or phosphate. The use of filter elements 1, wherein the ceramic material of the filter member 3 comprises at least 90 percent of mullite, may be particularly beneficial in such methods as fluorine is often present in filtration of ferrochrome, iron ore and phosphate. On the other hand, other factors, like the geographical factors, such as the geographical area where the filtration takes place, may also affect the presence of fluorine in raw water and/or process water.

According to an embodiment, the filter member 3 may form a capillary filter. A capillary filter refers to a filter, wherein the structure and/or the material of the filter, such as the filter member 3, enables a certain amount of liquid, such as water, to be kept in the filter by a capillary action. The liquid may be kept in micro-pores provided in the filter member 3, for example. Such a capillary filter enables the liquid to be filtered to easily flow through the filter member 3, but when all free liquid has passed through the filter member 3, the remaining liquid kept in the filter by the capillary action prevents flow of gas, such as air, through the wet filter member 3. The capillary action thus does not participate in the dewatering itself, for instance by sucking water out of the slurry. In other words, in a capillary filter liquid, usually water, may be kept in the micro-pores of the filter member 3 by capillary forces and no flow of gas takes place after the free water in the residue, such as the cake, has been removed. According to an embodiment, the filter member 3 formed as a capillary filter prevents air from entering the internal cavity. Acid washing may be particularly beneficial in connection with use of filter members 3 forming a capillary filter, as the acid washing ensures a sufficient grade of degeneration of the filter element 1.

According to an embodiment, the bubble point of the filter member 3 is at least 0.2 bar. In this context, the bubble point refers to an effective bubble point. The effective bubble point describes a pressure difference between a first filter surface 9a of the filter member 3 directed towards the filtrate channel 7 and the second filter surface 9b of the filter member 3 opposite to the first filter surface 9a and directed towards the cake 5, at which 1 liter of air flows through one square meter of the second filter surface 9b during a one minute time. In other words, when a 0.2 bar pressure difference is provided, in such a filter member 3, between the outside of the filter element 1 and the inside of the filter element 1, such as within the filtrate channel 7, a maximum of 1 liter of air should be able to pass through a square meter of the second filter surface of the filter member 3 during a one minute time. If a flow of air through the filter member 3 at 1 liter per minute requires a pressure difference of 0.2 bar or greater, the bubble point of the filter member 3 is thus at least 0.2 bar. Thereby, in embodiments where it is not practical to block the flow of air completely, only a very minor amount of air may be able to flow through the filter member 3 when the cake is being dried. When the cake is being dried, an underpressure is provided within the filter element 1, such as within the filtrate channel 7, which means than the pressure inside the filter element 1 is lower that the pressure outside the filter element 1.

According to an embodiment, at least 600 liter of water per an hour and per one square meter of the second filter surface 9b may be able to pass through the filter member 3 when a pressure difference of 1 bar is provided between the first filter surface 9a and the second filter surface 9b. Thus, a sufficient amount of water may flow through the filter member 3 to provide efficient filtering of the slurry, especially when the actual filtering takes place. During filtering, an underpressure is provided within the filter element 1, such as within the filtrate channel 7, which means that the pressure inside the filter element 1 is lower than the pressure outside the filter element 1.

The pressure difference between the inside of the filter element 1 and the outside of the filter element 1 may be greater during the actual filtering than during the drying of the cake. The drying of the cake may take place for instance in a disc filter apparatus 2 when the filter element 1 in question has passed the filtering position, such as the lowest position in the filter 15 and rotated back upwards. In other words, a specific filter element 1 participates in the actual filtering at a different point of time and at a different position in the filter apparatus 2 than in the drying of the cake. Thus, the relevant pressure difference for the actual filtering and the drying of the cake may be different from one another.

The structure of the filter member 3, such as the mean pore size of the filter member 3, affects both the effective bubble point and the flow of water through the filter member 3.

According to an embodiment, the filter apparatus 2 comprises a disc filter apparatus, a belt filter apparatus or a drum filter apparatus.

It will be obvious to a person skilled in the art that, as the technology advances, the inventive concept can be implemented in various ways. The invention and its embodiments are not limited to the examples described above but may vary within the scope of the claims.

CLAIMS

1. A method of vacuum filtration comprising

guiding (100) a slurry comprising raw water and/or process water to a filter apparatus (2) comprising at least one filter member (3) comprising a ceramic material or a composition comprising a ceramic material and at least one filtrate channel (7), wherein the slurry is guided to the opposite side of the filter member relative to the filtrate channel,

generating (110) suction within the filtrate channel, such that a cake (5) is formed on the opposite side of the filter member, and

deliquoring and discharging (120) the cake, **characterized** by

the raw water and/or process water used in the filtration process comprising at least 2 ppm fluorine and

the ceramic material of the filter member (3) comprising at least 90 percent of mullite.

2. A method according to claim 1, wherein the raw water and/or process water comprises 2 to 50 ppm of fluorine.

3. A method according to claim 1 or 2, wherein the raw water and/or process water comprises 5 to 50 ppm of fluorine.

4. A method according to any one of claims 1 to 3, wherein the ceramic material comprises at least 95 percent of mullite.

5. A method according to any one of claims 1 to 4, wherein the method further comprises acid washing the filter member.

6. A method according to claim 5, wherein the pH of the acid washing medium is less than or equal to 2.

7. A method according to claim 5 or 6, wherein at least one of the following acids is used in the acid washing: nitric acid, oxalic acid and sulphuric acid.

8. A method according to any one of claims 1 to 7, wherein the method is used for filtration of at least one of the following: ferrochrome, iron ore or phosphate.

9. A method according to any one of claims 1 to 8, wherein the filter member forms a capillary filter.

10. A method according to any one of claims 1 to 9, wherein the bubble point of the filter member is at least 0.2 bar.

11. A method according to any one of claims 1 to 10, wherein the filter apparatus (2) comprises a disc filter apparatus, a belt filter apparatus or a drum filter apparatus.

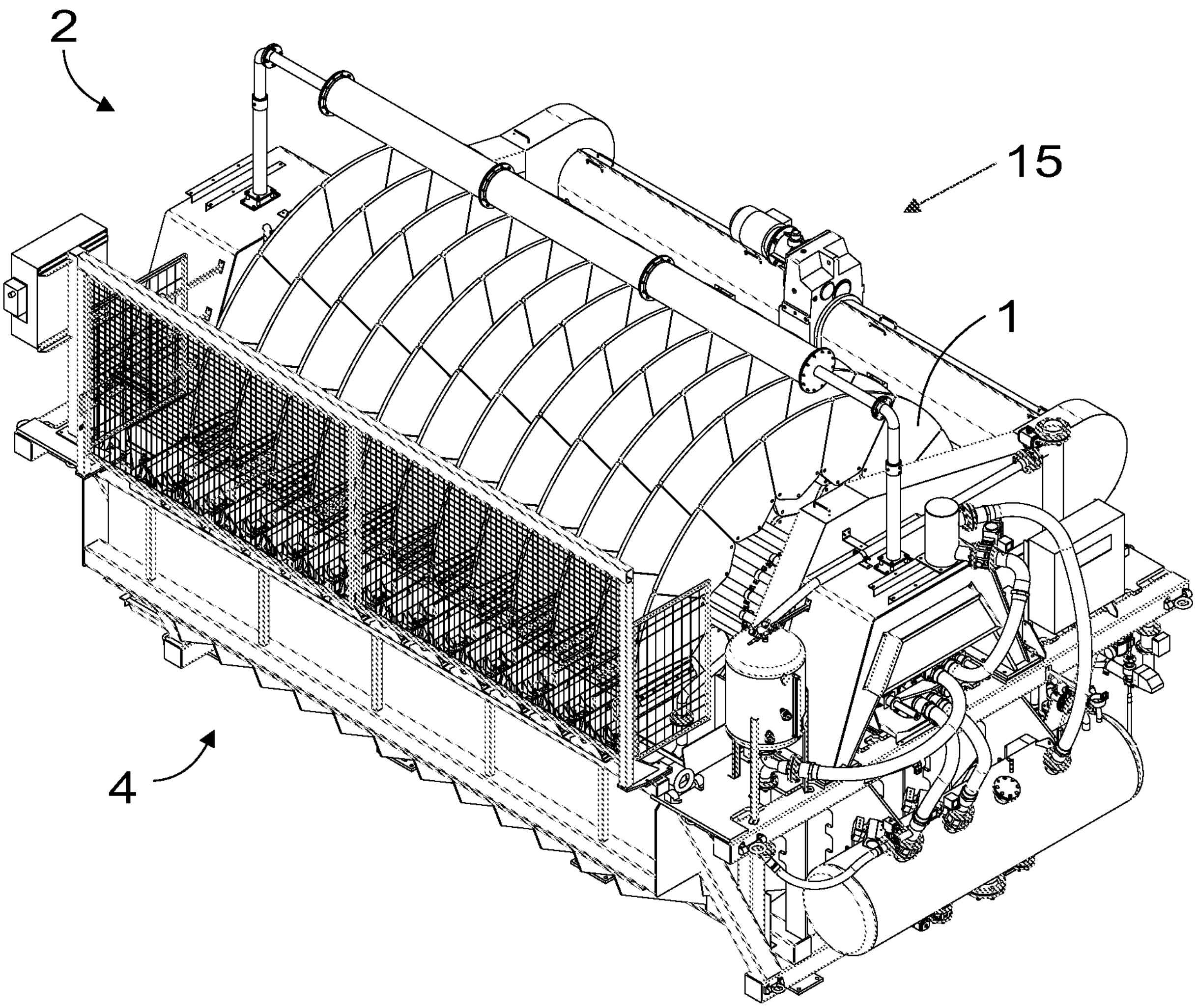

FIG. 1

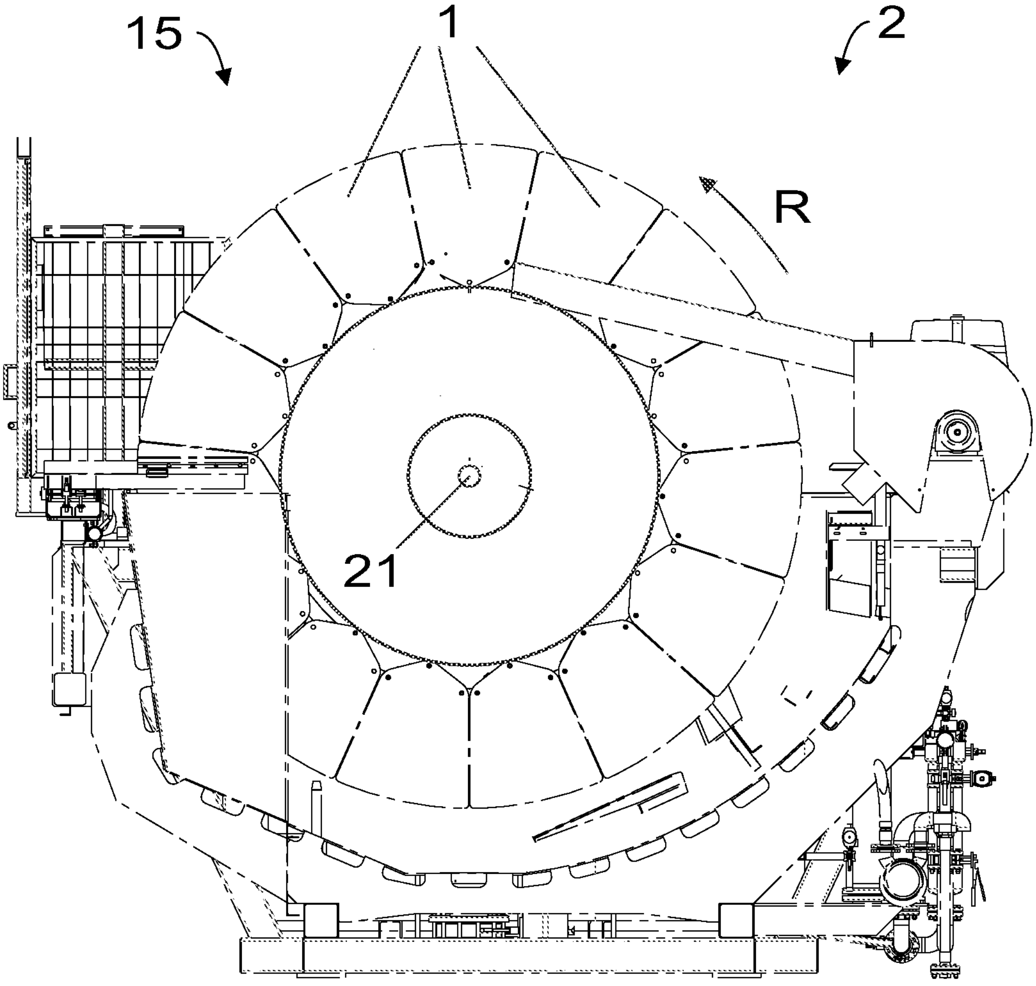

FIG. 2

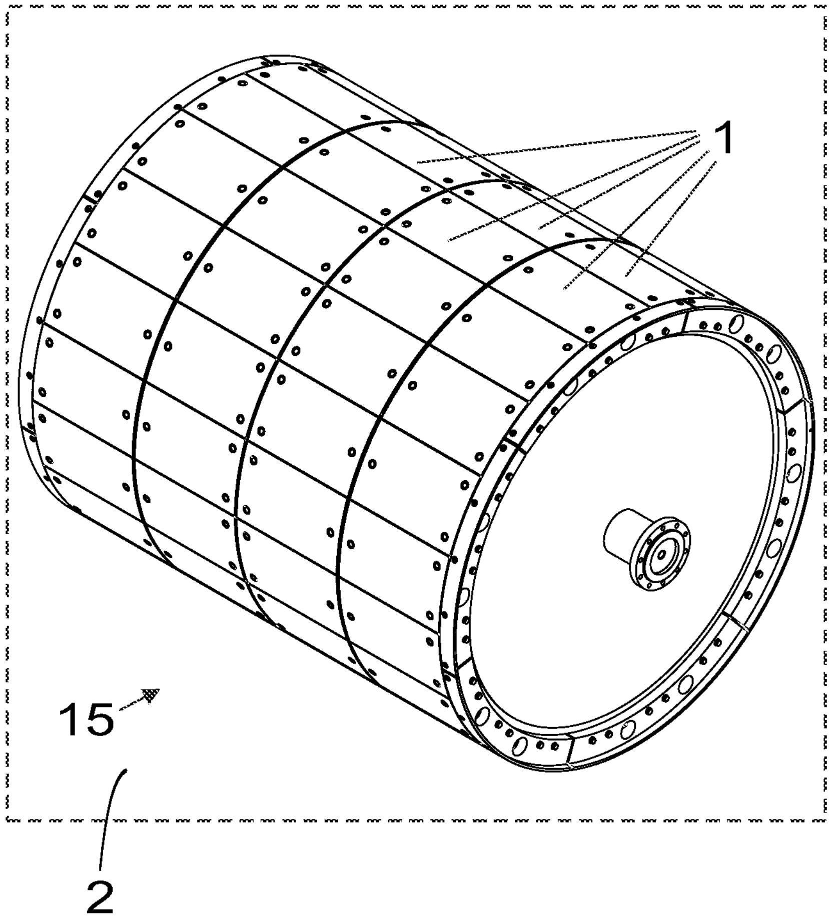

FIG. 3

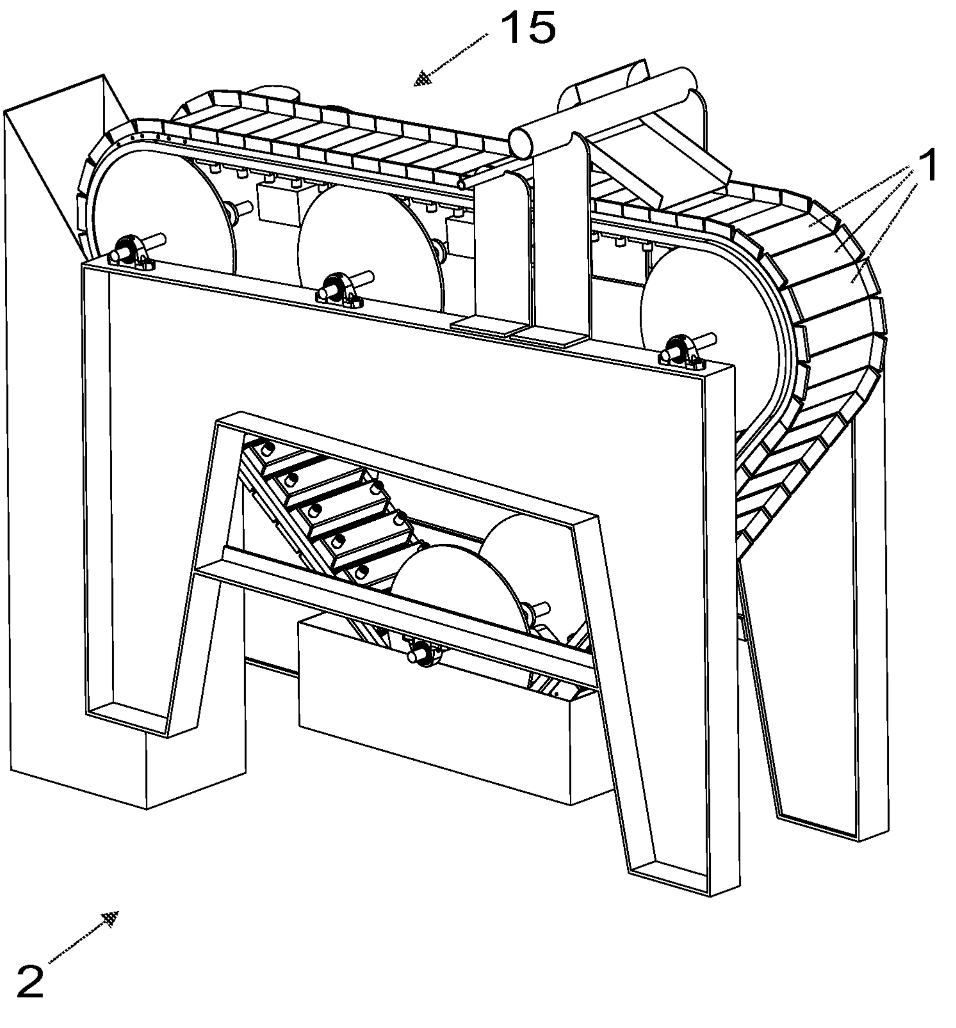

FIG. 4

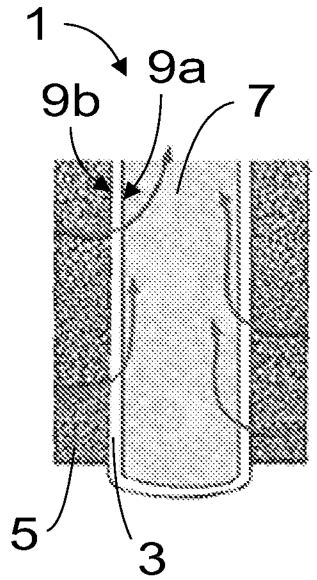

FIG. 5a

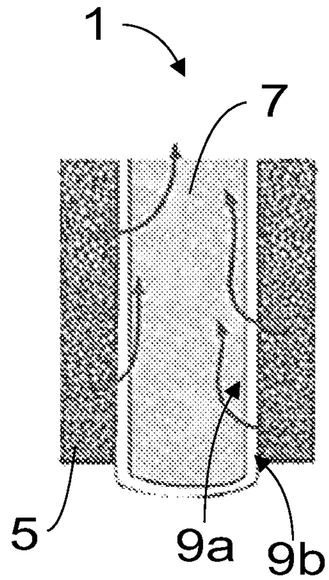

FIG. 5b

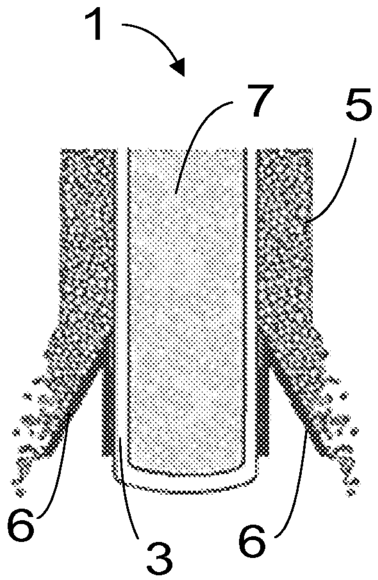

FIG. 5c

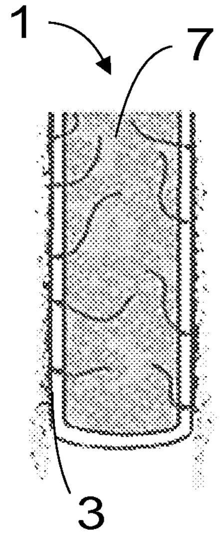

FIG. 5d

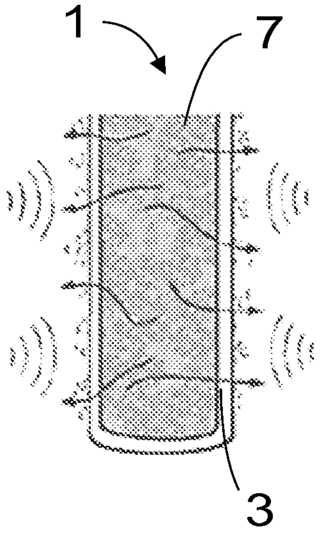

FIG. 5e

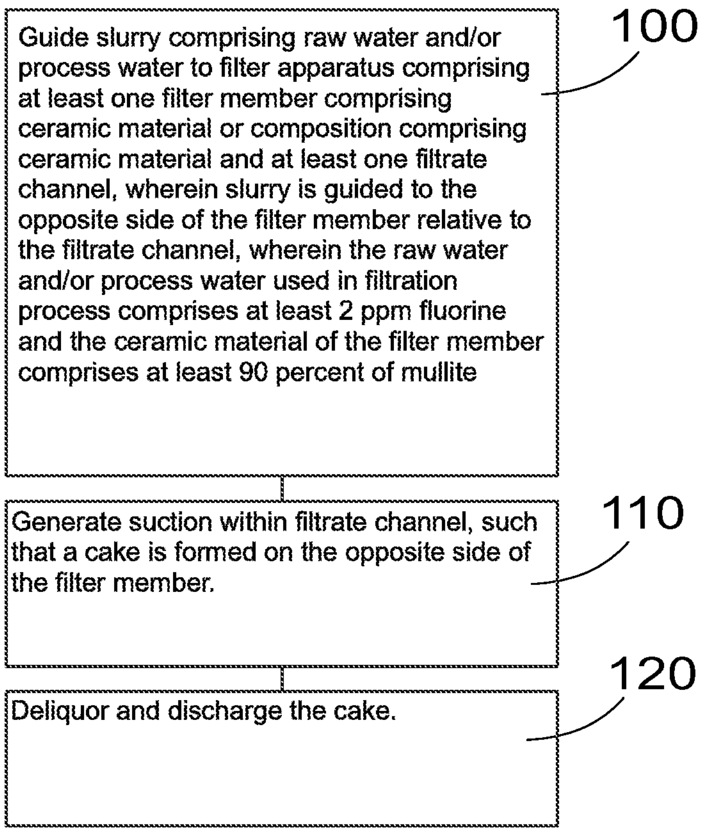
Guide slurry comprising raw water and/or process water to filter apparatus comprising at least one filter member comprising ceramic material or composition comprising ceramic material and at least one filtrate channel, wherein slurry is guided to the opposite side of the filter member relative to the filtrate channel, wherein the raw water and/or process water used in filtration process comprises at least 2 ppm fluorine and the ceramic material of the filter member comprises at least 90 percent of mullite
100
Generate suction within filtrate channel, such that a cake is formed on the opposite side of the filter member.
110
Deliquor and discharge the cake.
120

FIG. 6

INTERNATIONAL SEARCH REPORT

International application No
PCT/FI2017/050026

A. CLASSIFICATION OF SUBJECT MATTER
INV. B01D33/073 B01D33/21 B01D33/333 B01D33/46 B01D33/54
B01D33/60 C02F1/00
ADD. C02F101/10 C02F101/20 C02F101/22 C02F101/14

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)
B01D C02F

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

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

EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X Y	WO 2014/191634 A1 (OUTOTEC FINLAND OY [FI]) 4 December 2014 (2014-12-04) figures 1-5 page 4, line 21 - page 9, line 30 -----	1-4,8-11 5-7
Y	WO 2014/170533 A1 (OUTOTEC FINLAND OY [FI]) 23 October 2014 (2014-10-23) figures 1-13 page 10, line 35 - page 11, line 6 -----	5-7
A	JP H06 134267 A (NORITAKE CO LTD; SHINKO PANTEC CO LTD) 17 May 1994 (1994-05-17) paragraphs [0001], [0034] -----	1-11

☐ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

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

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