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(54) **Eljárás szilárd anyagok elválasztására, valamint foszfátoknak vizes szuszpenziókból való visszanyerésére**

Az európai szabadalom ellen, megadásának az Európai Szabadalmi Közlönyben való meghirdetésétől számított kilenc hónapon belül, felszólalást lehet benyújtani az Európai Szabadalmi Hivatalnál. (Európai Szabadalmi Egyezmény 99. cikk(1))

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METHOD FOR THE SEPARATION OF SOLIDS AND RECOVERY OF PHOSPHATES FROM AQUEOUS SUSPENSIONS

TECHNICAL FIELD OF THE INVENTION

The invention relates to a method for recovery of phosphates from a suspension of solids in an aqueous solution with the steps of concentrating phosphates in the aqueous solution, separating the solids from the aqueous solution and precipitating of the phosphates from the aqueous solution.

PRIOR ART

From DE 43 23 610 C1, a biorefuse cell is known which forms an intermediate deposit for soils or sludges to be cleaned and in which biological processes take place. The biorefuse cell serves for biological cleaning and/or mineralizing of soils, sludges and sewage waters which are contaminated with organic or inorganic pollutants. The biorefuse cell comprises a sealing in the shape of a watertight film against the ground. Its floor is covered with a drainage bed which is either equipped with iron compounds or consists completely of iron compounds. On the drainage bed, a water-permeable fleece is arranged above which the substances to be cleaned are brought into the biorefuse cell and form a soil or sludge layer contaminated with the organic and inorganic pollutants there. This layer is planted with suitable plants or corresponding rhizomes through the fine roots of which oxygen enters into the soil layer or sludge layer. Via a water-level control mounted into one side wall, a suitable water level in the biorefuse cell is set. The activity of aerobic microorganisms is enhanced or stabilized by the purposeful addition of nutrients and/or additives. By the impact of rain, a movement of not-converted pollutants and of conversion products of aerobic processes, e.g. sulphates, phosphates or heavy-metals, from an upper aerobic portion into a lower anaerobic portion of the biorefuse cell occurs. The phosphates react with the iron compounds of the drainage bed there, for example to produce FePO_4 and in this way are fixed in the drainage bed. The cleaned water is transported away via a system of ditches or a pump.



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DE 197 00 434 C1 describes a method for humification of effluent sludge avoiding chemicals. In order to achieve this, fluid effluent sludge is brought into a first cell up to a first filling level, the fluid effluent sludge is dried, fine-rooted, fast-growing plants are sowed, and a growing phase of the plants over a preset time span is induced. This is repeated until the complete filling of the cell and after again leaving it to rest the cell is emptied. In one embodiment of the known method, the single method steps are carried out in several cells with an offset in time. Preferably, three cells are used so that for example the introduction of the fluid effluent sludge into the first cell occurs parallel to the drying of the fluid effluent sludge and the sowing of the fine-rooted, fast-growing plants in the second cell and the growing phase of the plants in the third cell. Each cell is surrounded by walls and a ground plate, e.g. made of concrete, or a layer that is sealing against water or hardly water-permeable in the direction of the ground and side slopes. On the ground plate or the layer sealing towards the ground, there is a tube drainage or surface drainage covered by a layer of gravel. The layer of gravel is covered for example by a mineral mixture or concrete stones. Back-up water is carried away with the aid of a shaft structure on the wall. Drainage water and back-up water are fed to a sewage treatment plant via tubes.

For the recovery of phosphates in sewage treatment, the so-called PRISA method is known (Montag, 2008: Phosphorrückgewinnung bei der Abwasserreinigung - Entwicklung eines Verfahrens zur Integration in kommunale Kläranlagen, PhD thesis, RWTH Aachen), which comprises the features of the preamble of independent claim 1. This method comprises a thickening of the sludge in a pre-thickener and a post-thickener before and after an anaerobic sludge treatment. Subsequently, a mechanical de-watering of the sludge is carried out. A phosphate-rich back-up water of the pre-thickener, an ammonium-rich and phosphate-rich back-up water of the post-thickener and the sludge water from the mechanical sludge de-watering are filtered together, for example with the aid of a fabric filter. In a mixing container, magnesium oxide as a precipitant and sodium hydroxide for setting the pH value are added to the filtrate. In a crystallization container magnesium ammonium phosphate crystals form. The de-watered effluent sludge from the automatic sludge de-watering has to be disposed of separately. Because the realization of this method necessitates investments into a pre-thickener, a post-thickener and machines for sludge de-watering, it is only economically deployable in large sewage treatment plants.

From US 3,236,766 A and US 4,141,822 A, a method also called the phostrip method for stripping phosphorus compounds from sewage is known. By treating raw or pre-treated sewage, a process fluid which is mostly free of phosphates and a phosphate-rich sludge result. In order to achieve this, the pH value is set into a region of approximately 6.2 to approximately 8.5. The raw or pre-treated sewage is mixed with sludge, which for the formation of a homogeneous mixed fluid as it is necessary for a high efficiency of the method necessitates heavy mixing. Oxygen is added to the mixed fluid and dissolved in it; phosphate-rich sludge is reclaimed and a mostly phosphate-free process fluid is produced. The phosphate-rich sludge for some hours is kept under anaerobic conditions to reduce the phosphate content in its solid phase. According to US 4,141,822 A, the transfer of the phosphate into the settling phosphate-rich fluid is accelerated by the addition of an auxiliary medium. Subsequently, part of the sludge is returned for mixing with sewage.

In a method described in DE 2442031 A1, during a resting time of 2 to 20 hours in a container a settling of the sludge occurs in which the soluble phosphates move into the upper portion of the settling sludge. In this way, between a sludge layer and a practically phosphate-free supernatant [sic] water a phosphate-enhanced aqueous layer results, which is subsequently removed from the settling zone and is further treated by mixing with an agent for precipitating the phosphates. The sludge layer is returned to the process, which, however, in practice leads to problems in the repeated settling of the sludge. As in the PRISA method, for this method a high procedural effort has to be coped with; special method steps are to be constructed and much energy is necessary e.g. for the stirring of the sludge. Therefore, this method, too, is only economical when employed on a large scale.

In DE 10 2008 038 886 A1, a method for the selective obtaining of phosphorus from heavy-metal and phosphate-rich solids is described. In this method, the heavy-metal and phosphorus-rich solid is treated with microorganisms which comprise leaching-active and polyphosphate-containing microorganisms. This is done under acidic aerobic conditions and serves for freeing heavy-metals and phosphate from the solid and for absorbing the freed phosphate by the polyphosphate-containing microorganisms. Subsequently, biomass enriched with phosphorus is separated. The accumulated phosphorus is available to plants and free of undesired heavy metals, so that the biomass can be used as a phosphate-rich fertiliser.

Horn, Sartorius, Tettenborn 2010: Technologievorausschau für Phosphorrecyclingtechnologien (Arbeitspaket 6) gives an overview of further methods for recovering phosphates.

WO 2012/150296 A2 discloses a method and a system for de-phosphatizing of sewage. The sewage is pre-treated in a biological pre-process. The pre-treated sewage in a collection
5 container is injected with coagulants for precipitating phosphates and if necessary stirred in order to distribute the coagulants better. Subsequently, the sewage is fed through a filter which is planted with rhizome plants. The precipitated phosphates are recovered from the surface of this filter. The injecting with coagulants in the collection container and the draining of the
10 sewage through the plant filter can be done subsequently in several steps. The disclosure especially has two subsequent steps with one collection container each and one plant filter each.

OBJECT OF THE INVENTION

It is the object of the invention to present a method with the features of the preamble of claim 1 with which phosphates can be recovered in a cost-efficient way from domestic and industrial
15 sewage even in sewage treatment plants of smaller dimensions for e.g. less than 100,000 residents.

SOLUTION

According to the invention, the object of the invention is solved by a method with the features of the independent claim. Additional preferred embodiments according to the invention are to be
20 seen in the dependent claims.

DESCRIPTION OF THE INVENTION

The invention relates to a method with which phosphates can be recovered from a suspension of solids in an aqueous solution. Such suspensions can for example be domestic or industrial sewage, but also waste material, for example from the food-processing industry or from farming.
25 In a humification basin a drainage layer is covered with a reactive organic layer, for example made of or on the basis of compost. The aqueous suspension is fed onto this reactive organic layer. Due to gravity, a separation of the aqueous solution - which can drain into the drainage

layer - from the solids - which become part of the reactive organic layer - occurs without the necessity of the use of filter materials or energy. In the reactive organic layer, a biological degradation of phosphate-containing solids is achieved by bacteria, fungi or other destruent, also more highly developed ones, such as compost worms. In the reactive organic layer a high reduction of volume occurs so that with an interval of a few days each further layers of aqueous suspension can be added. Therefore, the humification basin can be operated over a time span of several years without being emptied, which is especially efficient. Due to the biological degradation, the ratio of phosphate in soluble form is heightened. This soluble phosphate can be washed from the reactive organic layer into the drainage layer with the aqueous solution. From there, the aqueous solution is drained whenever this appears favourable due to its enriched phosphate content. The concentration of phosphates in the drainage layer is continuously monitored; this for example can be done by periodic taking of samples or insertion of a probe. The aqueous solution is preferably drained when a concentration of phosphates is reached which is especially advantageous for the precipitation. The aqueous solution can be fed back into the humification basin when its concentration of phosphates is below a preset threshold value. From the drained aqueous solution the solved phosphates are precipitated and subsequently are available in a pure form which is suitable for example for use in fertilisers. Rests of phosphates which do not dissolve in the aqueous solution and permanently remain in the separated and highly volume-reduced solids are permanently accessible because of the above-ground deposition of the solids in the humification basin and if necessary can be brought to a further use by shaving (e.g. burning with subsequent recovery of phosphates from the combustion residue).

In addition to an existing humification basin and a container for precipitation no further devices are needed for the method according to the invention. No special procedural effort is necessary, either, since the separating of the aqueous suspension and the concentrating of the phosphates in the aqueous solution is done in one work step and the same humification basin. Humification basins are present anyway in some sewage treatment plants, especially smaller sewage treatment plants beginning at about 1,000 residents or are at least advantageous for such smaller sewage treatment plants. No special requirements are present for the container for precipitation; it does not take up much space, either.

The precipitation of the phosphates can be achieved for example by adding magnesium so that MAP precipitates. This is especially advantageous since the phosphate and ammonium

concentrations present in the aqueous solution lead to the precipitation of MAP even with very small additions of magnesium.

To guarantee that no environmental hazard occurs due to leaking substances, the humification basin is closed off in a fluid-tight way to the ground, for example by a film or a concrete basin
5 equipped with a fluid-tight coat. Contrary to that, the humification basin is usually open on its upper side. In this way, the degradation processes are aided by supply of air, solar irradiation and other weather influences.

To control the thickening of the suspensions, the humification basin according to necessity can be covered fully or partially, so that for example a thinning or the sluicing by rain, where the rain
10 water becomes part of the aqueous solution, is promoted or prevented. This can, for example, be done by a movable roof or a film. In this way, the drying-off of the solids or the reactive organic layer can be controlled by controlling the solar irradiation.

In one embodiment of the invention, the drying-off of the solids or the reactive organic layer can also be quickened by heating the drainage layer. In this way, ideal temperature conditions for
15 microbial degradation processes can be set. For heating, waste heat may be used, for example from a digestion plant, a sewage stream or other process heat accruing at the same sewage treatment plant, so that no additional demand of energy occurs.

Preferably, with a probe the redox potential of the aqueous solution from the drainage layer is monitored. For an ideal flow of operation of the microbial degradation processes in the reactive
20 organic layer, predominantly aerobic processes are desired. For this reason, predominantly oxidised compounds in the drained aqueous solution and therefore positive redox potentials in the region of more than 100 mV are aimed for. If the redox potential falls below this value, the respective partial region of the humification basin is preliminarily left to dry off so that more oxygen from the air can reach the reactive organic layer.

25 The aqueous solution, additionally, can be backed up so that it reaches into the landed solids. In this way, it can wash out dissolved phosphates there and carry them into the drainage layer.

The reactive organic layer can be planted to enhance the microbial degradation processes by forming a microbially populated root space. Through the root system, the layer also is aerated

and fine channels form so that the filter effect of the layer does not decrease. Additionally, increased input of oxygen into the layer can enhance aerobic destruction processes. In the same way, the plants serve as intermediate storage for different substances which they bind during their growing, such as carbon dioxide and phosphates. The concentrating of the phosphates from the aqueous solution is promoted by the plants. The plants are fertilised by the phosphates and incorporate them during their growth. The plants in this way form a temporary phosphates storage. When the plants die, they themselves become part of the organic layer. The stored phosphates then are degraded and in this way return to the aqueous solution in a concentrated form and are flushed out with the aqueous solution.

For the planting, C2 plants such as reed (*Phragmites australis*) are preferred. However, a planting with crop plants such as poplars or willows for wood production or rape for energy production is possible so that an additional use of the reactive organic layer as a cultivable surface is created, which further increases the economic viability of the process.

In one embodiment of the invention, the aqueous suspension is fed into the humification basin via several feeding points distributed over the area of the humification basin. If each feeding point can be controlled individually, this enables use of several partial regions of the humification basin together or separately. If a humification basin for example has six feeding points, three neighbouring feeding points together can form a partial region of the humification basin which is uniformly operated. Two further neighbouring feeding points can be put into operation at a later point in time and serve a smaller partial region while the sixth feeding point due to a low utilization can continue to lie barren. If then, for example, in one partial region the redox potential falls below a desired value, this partial region can be left to dry off, while occurring sewage can still be processed in other partial regions. Especially, particular partial regions can serve for intermediate storage of bound substances, especially of phosphates bound in plants. It is also possible to aim for different substance concentrations or thickenings of the landed solids in the different partial regions according to their later use. It is also possible to put particular partial regions out of service and after a dry phase with maximum separation of the phosphate-rich aqueous solution take the landed solids from the humification basin and put them to further use. During this, the phosphates left in the landed solids and further recyclable fractions can be reclaimed, e.g. by burning the solids and reclaiming the recyclable fractions from the resulting ashes.

Advantageous developments of the invention result from the claims, the description and the drawings. The advantages of features and of combinations of a plurality of features mentioned in the description only serve as examples and may be used alternatively or cumulatively without the necessity of embodiments according to the invention having to obtain these advantages.

Without changing the scope of protection as defined by the enclosed claims, the following applies with respect to the disclosure of the original application and the patent: further features may be taken from the drawings, in particular from the illustrated designs and the relative dimensions of a plurality of components with respect to one another as well as from their relative arrangement and their operative connection. The combination of features of different embodiments of the invention or of features of different claims independently of the chosen references of the claims is also possible, and it is motivated herewith. This also relates to those features which are illustrated in separate drawings, or which are mentioned when describing them. These features may also be combined with features of different claims. Furthermore, it is possible that further embodiments of the invention do not have the features mentioned in the claims.

The reference signs contained in the claims do not limit the extent of the matter protected by the claims. Their sole function is to make the claims easier to understand.

BRIEF DESCRIPTION OF THE DRAWINGS

In the following, the invention is further explained and described with respect to preferred exemplary embodiments illustrated in the drawings.

Fig. 1 shows a humification basin with a separate container for precipitation of phosphates from an aqueous solution drained from the humification basin.

Fig. 2 schematically shows the flow of the aqueous solution through the humification basin and the container according to Fig. 1.

Fig. 3 is a top view onto a humification basin which is divided into several partial regions with one feeding point each.

DESCRIPTION OF THE DRAWINGS

Fig. 1 shows a humification basin 10 which is sealed with a watertight layer 7, for example a plastic film, towards the bottom and the sides, for example against the ground and earth walls. Above the watertight layer 7 there is a drainage layer 1, for example of a drainage gravel, onto which a reactive organic layer 2, for example based on compost, has been applied. Plants 3 have been planted into the reactive organic layer 2 and grow roots throughout the reactive organic layer 2. The plants 3 cause the input of oxygen O_2 into the reactive organic layer 2 and the exchange of carbon dioxide CO_2 . A pipeline 24, which at first runs below the watertight layer 7 and in the centre of the humification basin 10 rises through all layers to the height of the top of the humification basin, forms a feeding point 5. Via the feeding point 5, the suspension 4 of solids in an aqueous solution 6 is applied to the reactive organic layer 2. The aqueous solution 6 filtered off the aqueous suspension 4 due to gravity is drained into the drainage layer 1 and for the precipitation of phosphates is pumped into a separate container 8. The solids from the suspension 4 become part of the reactive organic layer 2. The humification basin 10 can be fully or partially covered with a roof 9.

Fig. 2 schematically shows the flow of the suspension 4 or the aqueous solution through the humification basin 10 and the container 8. The suspension 4 in its untreated state is brought into the humification basin 10. Optionally, to enhance its de-watering, polymers 19 may be added. The aqueous solution 6 filtered off by means of a pump 13 is pumped into the container 8, where magnesium ammonium phosphate 20 is precipitated by adding magnesium. From the container 8, the phosphate-free aqueous solution 14 can be brought into a sewage treatment plant 17. The aqueous solution 6 is monitored with a redox measuring probe 11. Should the redox potential fall below a guiding value of 100 mV and therefore an indication be present of a predominance of undesired anaerobic processes in the reactive organic layer 2, further feeding of the aqueous suspension 4 can be stopped. In this way, the plants 3 can recover and the input of oxygen rises again and stimulates the increase of aerobic processes. A phosphate-measuring probe 12 measures the phosphate concentration in the drained aqueous solution 6. If the phosphate concentration is below a desired value, the aqueous solution 6 can be fed back to the humification basin 10 as a recycle 16 instead of to the container 8, optionally by sprinkling 15 over the humification basin 10. Alkali or earth alkali metals 18 can be added to the recycle 16 to increase the pH value and obtain a composition of the aqueous solution 6 favourable for the precipitation of magnesium ammonium phosphate 20. The concentration of phosphate in the aqueous solution 6 can furthermore be controlled by the use of the roof 9. With

the roof, precipitation water or rainfall water H_2O can be blocked for thickening the suspension 4 brought into the humification basin 10 or inversely, an evaporation of H_2O by solar heat be inhibited.

Fig. 3 shows a humification basin 10 which is divided into separately operated partial regions 21 to 23 by several separately controllable feeding points 5, which here are six separately controllable feeding points 5. Three feeding points 5 neighbouring each other define a first partial region 21 of the humification basin, while two further neighbouring feeding points 5 supply a smaller second partial region 22 of the humification basin. A smallest, third partial region 23 of the humification basin is defined by the sixth feeding point 5. Though the aqueous solution 6 is centrally drained from the humification basin 10, when only one of the partial regions 21 to 23 is used, it can be correlated with this partial region. In this way, some aqueous solution 6 can be purposely fed to the container 8 for the precipitation of phosphates when it originates from the feeding of a partial region 21 to 23 in which the plants 3 are currently dying and therefore setting free phosphates bound during their growth into the aqueous solution 6 in a concentrated form.

LIST OF REFERENCE NUMERALS

- 1 Drainage layer
- 2 Reactive organic layer
- 3 Plants
- 4 Aqueous suspension
- 5 Feeding Point
- 6 Aqueous solution
- 7 Watertight layer
- 8 Container
- 9 Roof
- 10 Humification basin
- 11 Redox measuring probe
- 12 Phosphate measuring probe
- 13 Pump
- 14 Phosphate-free aqueous solution
- 15 Sprinkling
- 16 Recyclate
- 17 Sewage treatment plant
- 18 Alkali metals or earth alkali metals
- 19 Polymers
- 20 Magnesium ammonium phosphate (MAP)
- 21 Partial region
- 22 Partial region
- 23 Partial region
- 24 Pipeline

Eljárás szilárd anyagok elválasztására valamint foszfátoknak vizes szuszpenziókból való visszanyerésére

SZABADALMI IGÉNYPONTOK

1. Eljárás foszfátoknak egy olyan szuszpenzióból (4) való visszanyerésére, amely egy vizes oldatban (6) szilárd anyagokat tartalmaz,

- a foszfátoknak a vizes oldatban (6) való koncentrálásával,
- a szilárd anyagoknak a vizes oldattól (6) való elválasztásával (6) és
- a foszfátoknak a vizes oldatból (6) való kicsapásával,

azzal jellemezve,

- **hogy** a koncentrálás és az elválasztás magában foglalja
 - egy lefolyó rétegnek (1) és egy reaktív szerves rétegnek (2) egy humuszképző medencében (10) való elhelyezését,
 - a szuszpenzió (4) a humuszképző medencébe (10) való betáplálását, a reaktív szerves réteg (2) felett,
 - a reaktív szerves rétegben (2) oldható formában található foszfátok hányadának, a foszfáttartalmú szilárd anyagok biológiailag lebontásával megvalósított növelését,
 - a reaktív szerves rétegből (2) származó oldható foszfátoknak, a vizes oldattal (6), a lefolyó rétegbe (1) való belemosását,
 - a foszfátkoncentrációnak a figyelését a lefolyó réteg (1) tartományából származó vizes oldatban (6) és
 - a vizes oldatnak (6) a lefolyó réteg (1) tartományból való időszakos elvezetését és
- **hogy** a növelt foszfáttartalommal rendelkező vizes oldat térfogat-elvezetéseiben található foszfátoknak a kicsapását akkor kezdjük el, amikor a foszfátkoncentrációnak egy előre meghatározott küszöbértékét elérjük.

2. Az 1. igénypont szerinti eljárás, **azzal jellemezve, hogy** a humuszképző medencének (10) az alaptalaj irányába történő zárása cseppmentes, és a humuszképző medencébe (10) betáplált szuszpenziókat (4) kitesszük az időjárásnak.

3. Az előző igénypontok bármelyike szerinti eljárás, **azzal jellemezve, hogy** figyeljük a lefolyó réteg (1) részből származó vizes oldat (6) redox-potenciálját.

4. Az előző igénypontok bármelyike szerinti eljárás, **azzal jellemezve, hogy** azt az elvezetett vizes oldatot (6), amelynek a foszfáttartalma egy előre megadott küszöbérték alatt van, visszavezetjük a humuszképző medencébe (10).

5. Az előző igénypontok bármelyike szerinti eljárás, **azzal jellemezve, hogy** a humuszképző medencében (10) felgyűlt szilárd anyagok megkülönböztethető szártási foka egymást követően beállításra kerül.

6. Az 5. igénypont szerinti eljárás, **azzal jellemezve, hogy** a vizes oldatnak (6) a lefolyó réteg (1) régiójából való elvezetése előtt, a vizes oldatot (6) a felgyűlt szilárd anyagok egy régiójában felduzzasztjuk.

7. Az előző igénypontok bármelyike szerinti eljárás, **azzal jellemezve, hogy** a növelt foszfáttartalmú vizes oldatok (6) térfogataihoz magnéziumot adunk, azzal a céllal, hogy magnézium-ammónium-foszfát csapódjék ki.

8. Az előző igénypontok bármelyike szerinti eljárás, **azzal jellemezve, hogy** a reaktív szerves réteget (2) növényekkel beültetjük.

9. A 8. igénypont szerinti eljárás, **azzal jellemezve, hogy** a reaktív szerves réteget (2) C2 növényekkel ültetjük be.

10. A 8. vagy 9. igénypont szerinti eljárás, **azzal jellemezve, hogy** a reaktív szerves réteget (2) újrahasznosítható források nyerése végett haszonnövényekkel ültetjük be.

11. Az előző igénypontok bármelyike szerinti eljárás, **azzal jellemezve, hogy** a reaktív szerves réteg (2) számára egy komposztot alkalmazunk.

12. Az előző igénypontok bármelyike szerinti eljárás, **azzal jellemezve, hogy** szelektív módon tápláljuk be a vizes szuszpenziót (4) a humuszképző medence (10) azon különböző részrégióiba (21-23), amelyek a humuszképző medence (10) kiterjedésében terjednek szét.

13. Az előző igénypontok bármelyike szerinti eljárás, **azzal jellemezve, hogy** a humuszképző medencét (10) melegítjük.

A meghatalmazott:



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F/16381/B0

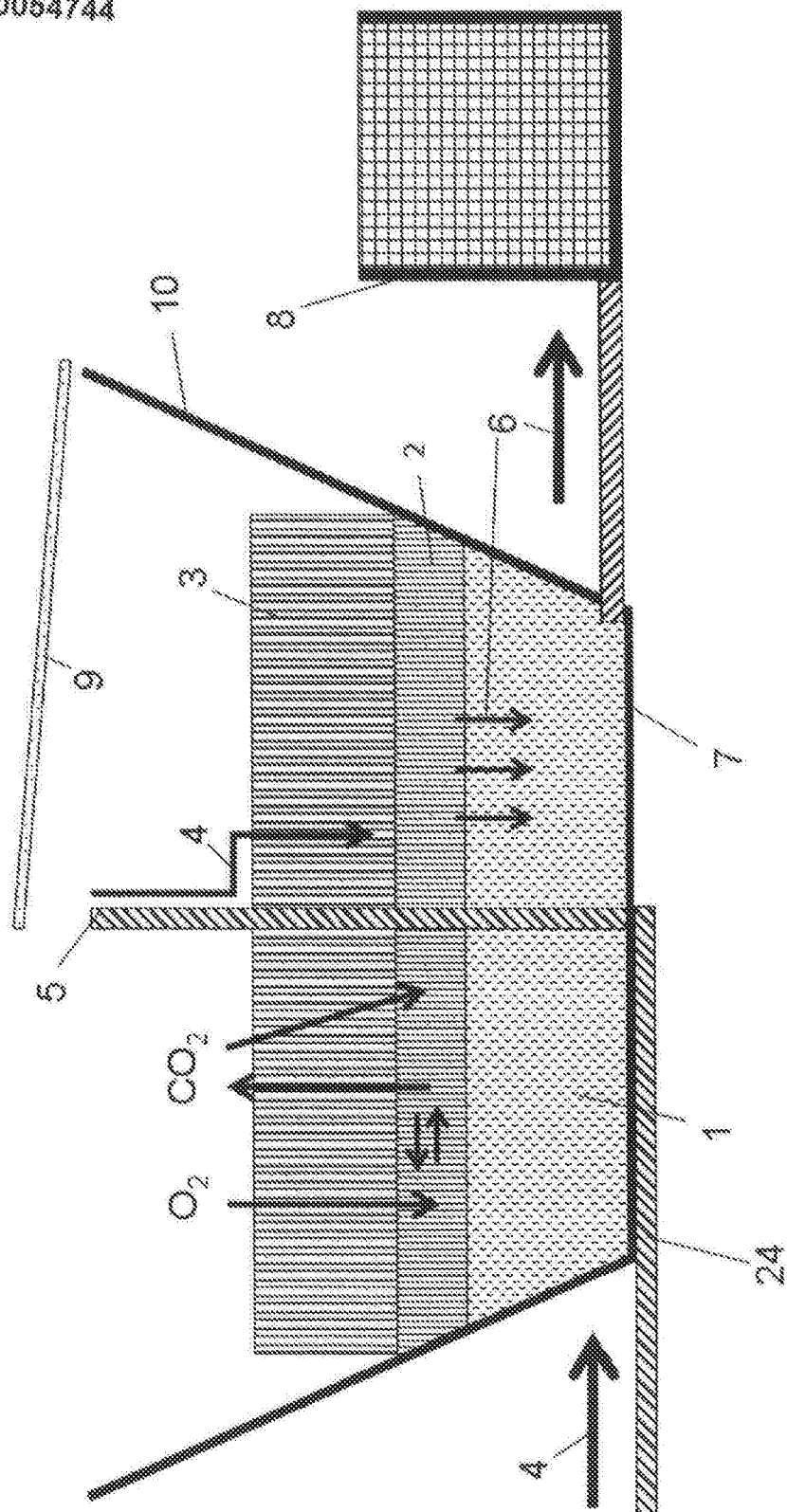


Fig.1

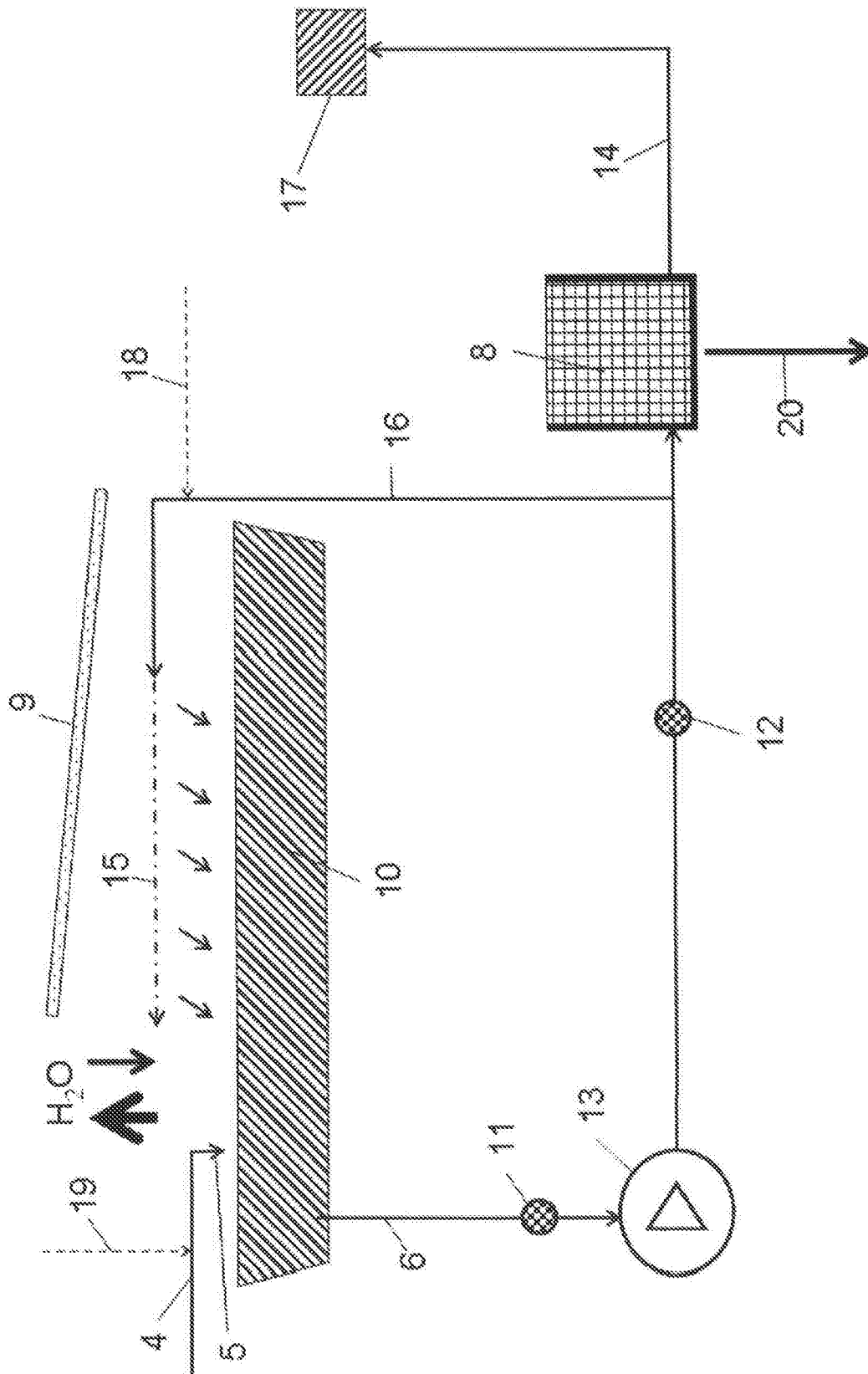
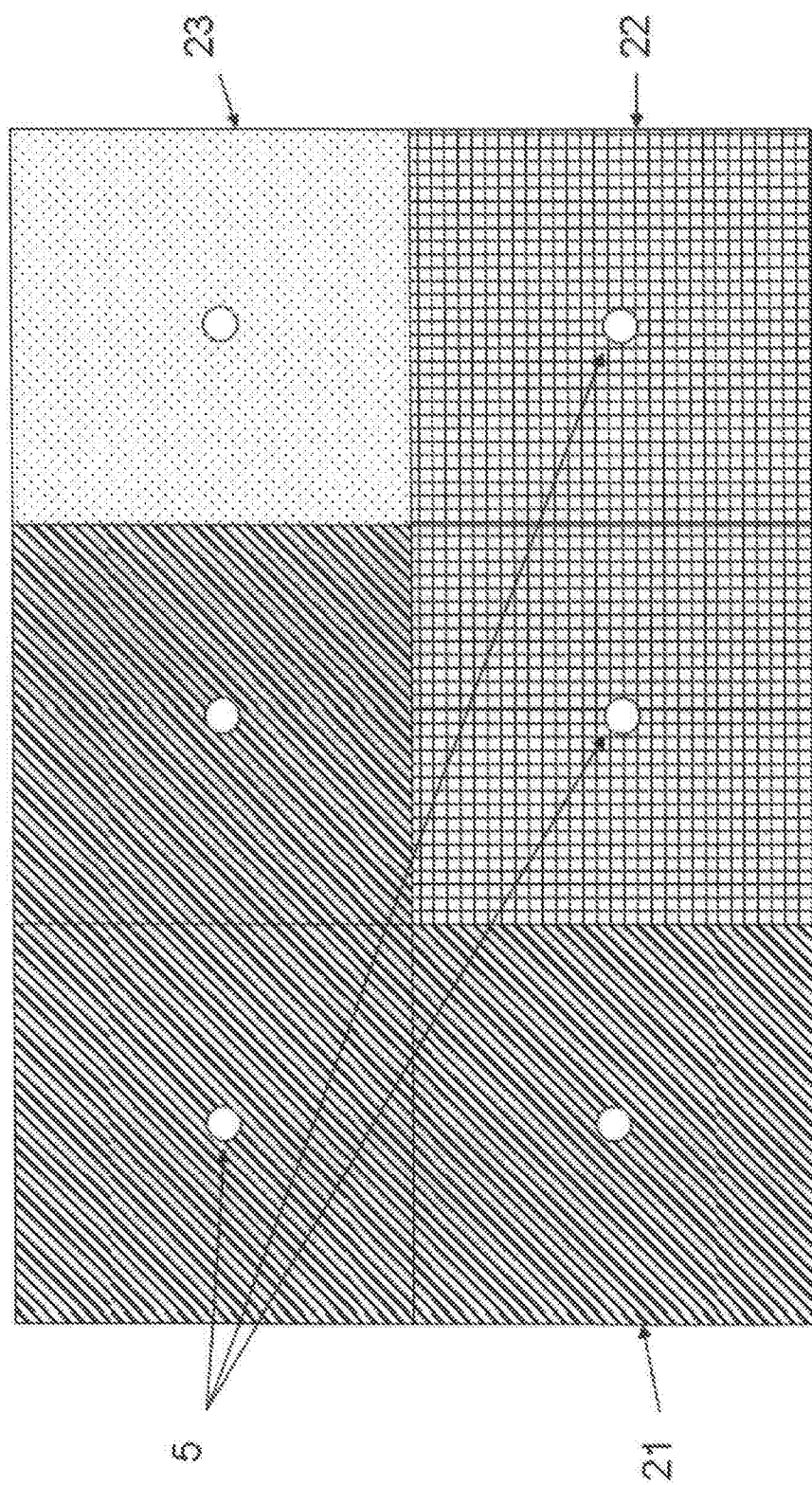


Fig. 2

**Fig. 3**