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- Bio-electrochemical system for recovery of components and/or generating electrical energy from a waste stream and method there for.
- The invention relates to a bio-electrochemical system and method for recovery of components or generating electrical energy from a waste stream. The system according to the invention comprises:
 - an anode compartment with an anode;
 - a cathode compartment with a cathode, wherein at least one of the anode and cathode is a bio-electrode;
 - a circuit connecting the anode and the cathode, the circuit comprising a power source for providing an electric current or a resistor;
 - an ion-exchange membrane separating the anode and cathode compartment; and
 - a flow channel defining hydrophobic membrane configured for gas extracting and/or reactant supply.

Bio-electrochemical system for recovery of components and/or generating electrical energy from a waste stream and method there for

The invention relates to a bio-electrochemical system (BES) for recovery of components and/or generating electrical energy from a waste stream. Such component may relate to ammonia (NH_3) and/or ammonium (NH_4^+) such that the system provides for ammonia and/or ammonium recovery. Waste stream may involve municipal waste water and industrial waste water, for example.

Conventional systems for recovering components involve the use of so-called strippers, for example for recovery of ammonia gas and/or ammonium solution. Such process is energy intensive and, furthermore, requires large amounts of chemicals.

WO 2013/105854 discloses a method for ammonia gas and/or ammonium solution recovery from an ammonium comprising fluid and bio-electrochemical system capable of performing such method. The method involves providing an anode compartment with an anode and a cathode compartment with a cathode, with both compartments being separated by an ion-exchange membrane, and extracting ammonia gas from the cathode compartment. This method enables ammonium-nitrogen (NH₃ and NH₄⁺) recovery from an ammonium comprising fluid.

An objective of the present invention is to provide a bio-electrochemical system for recovery of components from a waste stream that is more effective and more energy efficient as compared to conventional methods.

This object is achieved with the bio-electrochemical system (BES) for recovery of components or generating electrical energy from a waste stream according to the invention, the system comprising a reactor that comprises:

an anode compartment with an anode;

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- a cathode compartment with a cathode, wherein at least one of the anode and cathode is a bio-electrode;
- a circuit connecting the anode and the cathode, the circuit comprising a power source for providing an electric current or a resistor;
- an ion-exchange membrane separating the anode and cathode compartments; and
- a flow channel defining hydrophobic membrane configured for gas extraction and/or reactant supply.

In the case of component recovery, for example ammonia and/or ammonium recovery, involving the system according to the present invention, the term "ammonium" will be understood as NH_4^+ ions, and "ammonia" will be understood as NH_3 (for example in the gas phase (g) or in solution (aq)). The term "nitrogen recovery" will be understood as the recovery of a nitrogen comprising compound, such as ammonium and/or ammonia (NH_3) and/or nitrogen (N_2).

Waste streams may involve municipal waste waters and industrial waste waters, for example. This may involve ammonia and/or ammonium rich waste water streams such as the effluent of an anaerobic digester, urine treatment etc. Industrial waste waters may relate to waste waters from food processing, paper industry, and agriculture. It will be understood that also other waste water streams, preferably with a substantial amount of a component, such as ammonia and/or ammonium, can be treated in the bio-electrochemical system according to the present invention.

The at least one ion exchange membrane separating the anode and cathode compartments is preferably one or more of the following: a cation exchange membrane (CEM), an anion exchange membrane (AEM), a bipolar exchange membrane (BEM) or a charge mosaic membrane (CMM). In one of the presently preferred embodiments according to the invention, the membrane separating the anode compartment from the cathode compartment comprises a CEM, since transfer of NH_4^+ from the anode compartment is most efficient using a CEM.

In use, some of the cations are transported from the anode to the cathode, for example protons (H^+). For example, protons are produced in the anode compartment due to an oxidation reaction and pass through the membrane to the cathode compartment. Transport of cations other than H^+ and NH_4^+ will lead to an increase in the pH of the liquid in the cathode compartment, which will influence the equilibrium between ammonium and ammonia ($NH_4^+ + OH^- <--> NH_3 + H_2O$) resulting in a higher ammonia concentration once the pH is higher than the pKa of ammonia (pKa = 9.25).

The use of a flow channel defining hydrophobic membrane enables gas extraction from the compartment and/or reactant supply. It is shown that this significantly improves the overall efficiency of the reactions that take place in the bio-electrochemical system according to the present invention. Therefore, the efficiency of such system is improved due to the use of the one or more flow channel defining hydrophobic membranes.

More specifically, in the described embodiment of the present invention ammonium can be recovered by over the hydrophobic membrane as ammonia gas, involving the reaction $NH_4^+ + OH^- -> NH_3 + H_2O$. Additionally, the reason why there is a high pH in the cathode liquid is that the reduction process occurs under neutral to alkaline conditions and there is insufficient H^+ or NH_4^+ transport through the ion exchange membrane to compensate/buffer the production OH^- from the oxygen reduction reaction (MFC) or hydrogen evolution reaction (MEC). Oxygen reduction reaction (ORR) at neutral or alkaline conditions at the cathode involves $O_2 + 2H_2O + 4e^- --> 4OH^-$, and hydrogen evolution reaction (HER) at neutral or alkaline conditions at the cathode involves $2H_2O + 2e^- --> H_2 + 2OH^-$. Dependent of the type of membrane different ions are being transported through the anion exchange membrane (AEM), mostly OH^- from cathode to bonus compartment or anode compartment increasing the pH in the liquid. The cathode pH is also high at this point. CEM - cations, mostly NH_4^+ and other metal ions (Na^+ , K^+ , Mg^{2+} , Ca^{2+}) that may lead to a pH increase in

the bonus or cathode compartment. Once there is an equilibrium between the concentration of ions in anode compartment liquid and cathode compartment liquid the charge transport through the cation exchange membrane (CEM) will substantially/exclusively involve NH_4^+ and H^+ as $NH_4^+ + OH^- --> NH_3 + H_2O$, and ammonia is removed by the TMCS (hydrophobic membrane) and $H^+ + OH^- --> H_2O$. At this point the pH of the cathode should be stable and be defined by the buffer capacity and the most dominate species in the cathode, which should be ammonium/ammonia (pKa = 9.25) which leads to a pH around 9.25. This illustrates an operation with the system according to the present invention.

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In the system according to the invention the hydrophobic membrane can be placed at different positions in the system. The hydrophobic membrane can be placed in the cathode compartment with a cation exchanging membrane separating the anode compartment from the cathode compartment. In addition, or alternatively, the hydrophobic membrane can be positioned in the anode compartment with an anion exchange membrane separating the anode compartment and the cathode compartment. Furthermore, in addition or alternatively, the hydrophobic membrane can be positioned in another (intermediate) compartment. It will be understood that this specific design can be optimized in relation to relevant parameters including the type of waste water and the specific components and concentrations therein. Preferably, the hydrophobic membrane enables transmembrane chemisorption (TMCS).

In the presently preferred embodiment the hydrophobic membrane is provided as a number of tubular elements from a hydrophobic membrane material. Preferably, the tubular elements are shaped as hollow fiber flow channels, tubular members or straw like channels. The tubular elements can optionally be bundled. It will be understood that other designs for the hydrophobic membrane can also be envisaged in accordance with the present invention.

The bio-electrode is an electrode that is provided with micro-organisms, such as (electro-active) bacteria or electrogens. Often, the bio-electrode comprises a biofilm on the electrode. The micro-organisms catalyze the reactions at the anode and/or cathode, thereby improving the energy efficiency. The micro-organisms brake down "complex" organic compounds. Such organic compounds may comprise acetate or other fatty acids, creatinine, organic acids, creatine or sugars, for example.

The bio-electrochemical system according to the present invention is different from conventional microbial electrolysis cells (MECs) and conventional microbial fuel cells (MFCs).

The system according to the invention is capable of recovering components as was already described. For this purpose the system differs from conventional MECs, for example. In conventional MECs a voltage is applied for electrolysis of water to produce H_2 . Usually the anode is provided as a bio-electrode to oxidize organic compounds for electron production. The electrons are used to reduce protons (H^+ ions) or water (H_2O) at the cathode to hydrogen gas (H_2). The goal

of MECs is to produce a product at the cathode, in most cases hydrogen. In contrast, the system and method according to the invention is aimed at ammonia and/or ammonium recovery, for example in the form of ammonium sulphate, ammonium nitrate, ammonium phosphate, or ammonium chloride solution. For example, the recovery of ammonia and/or ammonium from a wastewater occurs in a final step, wherein the volatile ammonia is transported over the hydrophobic membrane and absorbed, in an absorption process, in an acid solution such as sulphuric, nitric, phosphoric and/or hydrochloric acid. Dependent on the used acid solution in the absorption process this results in a solution of ammonium -sulphate, -nitrate, -phosphate, -chloride.

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The system according to the invention can be used in a configuration in which it is capable of generating electrical energy. In conventional MFCs the organic compounds are consumed by the bacteria to produce (bio-anode) electrons and/or consume (bio-cathode) electrons to produce a current. This requires providing oxygen to the cathode compartment. In the system according to the invention the oxygen can be supplied with the use of a flow channel defining hydrophobic membrane. This provides an effective and efficient way to supply oxygen to the cathode and generate electrical energy.

In a further preferred embodiment the system is used for both component recovery and generating electrical energy. Oxygen is supplied to the cathode compartment by an oxygen inlet and/or a hydrophobic membrane, thereby enabling the system to operate in an MFC configuration. The reaction components can be used for the recovery involving a flow channel defining hydrophobic membrane. Optionally, a hydrophobic membrane is used both for oxygen supply and component recovery.

The aforementioned applications of the system and method according to the invention will be described in more detail in relation to some specific embodiments that are illustrated in the drawings.

In use, in nitrogen recovery, at the anode of the bio-electrochemical system according to the present invention organic matter is oxidized, preferably resulting in the production of H^+ and CO_2 . At the cathode a reduction reaction is performed, preferably resulting in OH^- and H_2 production. This results in pH increase in the cathode compartment thereby shifting the equilibrium between ionic ammonium towards ammonia (aq). The ammonia is transported over the hydrophobic membrane. Preferably, in the flow channel defining hydrophobic membrane ammonia is subsequently chemosorbed in an acid, for example H_2SO_4 , to ammonium, for example producing ammonium sulphate.

The bio-electrochemical system according to the present invention enables an effective recovery of components, such as ammonia and/or ammonium. Due to the electrons production of the bacteria, the required voltage which has to be applied across the anode and the cathode is reduced, thereby decreasing the power consumption of the system. This achieves an efficient

system. Furthermore, the amount of chemicals that are required is significant reduced as compared to conventional system. Preferably no (additional) chemicals, for example for increasing the pH in the cathode compartment, are introduced in the system according to the invention.

As an alternative to providing the circuit connecting the anode and the cathode with a power source, a resistor can be provided. By providing the system with oxygen electrical energy can be generated. Oxygen can be provided with the flow channel defining hydrophobic membrane configured for reactant supply.

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As already mentioned a further interesting embodiment can be achieved by including a (additional) flow channel defining hydrophobic membrane enabling component recovery.

Preferably, the waste water supplied to the bio-electrochemical system according to the invention comprises providing urine as ammonium comprising fluid, or a flow wherein preferably the urine concentration is high, most preferably close to or equal to 100%. Urine comprises relatively high levels of nitrogen in the form of urea. Urea decomposes to ammonia and ammonium. For example, waste water treatment plants have to remove considerable amounts of ammonium and ammonia due to urine. In particular since approximately 80% of nitrogen in waste water originates from urine. The system according to the invention and the method associated therewith is in particular suitable for this task.

In a preferred embodiment according to the present invention the method comprises providing an ammonium comprising fluid having an ammonium-nitrogen concentration ≥ 0.5 g/l, preferably > 1 g/l, more preferably > 5 g/l and most preferably > 10 g/l.

Preferably, in use the applied voltage is in the range of 10 mV - 50 V, more preferably 50 mV - 10 V and most preferably 100 mV - 5 V, for example 1 V - 2 V. The voltage preferably is in the range of 0.6-1.2 V and supplied by a DC power supply or potentiostat.

In a presently preferred embodiment according to the invention the bio-electrochemical system further comprises an intermediate compartment between the anode and cathode compartments, the intermediate compartment comprising separating ion-exchange membranes.

The intermediate compartment preferably receives waste water. In a presently preferred embodiment, wherein the bio-electrochemical system recovers ammonia, the ammonia is removed from the waste water flow where after the waste water flow is provided to the anode compartment wherein organic manner is preferably oxidized involving a bio-catalyzed oxidation reaction. Cations like Na⁺, K⁺ are transported through the cation exchange membrane separating the intermediate from the anode compartment, and for example OH⁻ is transported through the anion exchange membrane separating the intermediate compartment from the cathode compartment resulting in a pH increase of the intermediate compartment thereby enhancing the deprotonation of ionic ammonium into ammonia (aq).

Preferably, the hydrophobic membrane is positioned in the intermediate compartment. Providing the flow channel defining hydrophobic membrane in the intermediate compartment enables diffusion transport of ammonia from the intermediate compartment towards the flow channel defined by the hydrophobic membrane. In this flow channel subsequently chemosorption of ammonia into an acid as ammonium takes place. This provides a compact design and improved performance of a bio-electrochemical system for component recovery.

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Optionally, an additional anode is provided in the reactor of the system of the present invention. Preferably, the additional anode is provided in an intermediate compartment enabling decomposing ammonia into nitrogen gas and, depending on the configuration, hydrogen and water. This improves the overall performance of the bio-electrochemical system according to the present invention. Further effects and advantages for such additional anode are described in WO 2013/105854 for such additional anode.

In a presently preferred embodiment according to the present invention the hydrophobic membrane is positioned adjacent to a separating ion-exchange membrane.

By positioning the hydrophobic membrane close to a separating ion-exchange membrane the flow channel provides at an optimal location for optimal ion concentrations, for example a location having a relatively high OH⁻ concentration and highest pH. This renders the bio-electrochemical system according to the invention even more effective.

Preferably, the hydrophobic membrane is integrated with a separating ion-exchange membrane. This further improves the efficiency of the hydrophobic membrane and provides an effective means to assemble the reactor according to the present invention involving ion-exchange membranes and at least one hydrophobic membrane.

In the presently preferred embodiment according to the present invention the anode comprises a bio-electrode.

Providing the anode as a bio-electrode results in a so-called bio-anode where electro-active micro-organisms at the bio-anode catalyze the anodic reaction, thereby improving the overall energy efficiency. This results in a more efficient oxidation of organic compounds at the anode and associated component recovery, for example ammonia and/or ammonium. As already mentioned, the bio-anode potential lowers the voltage which has to be applied across the anode and cathode. This decreases the power consumption of the system resulting in an efficient recovery.

In a further preferred embodiment according to the present invention the bioelectrochemical system further comprises a number of additional reactors with a flow channel defining hydrophobic membrane extending through more than one of the reactors.

By providing a number of reactors, for example a stack of reactors, an effective upscaling of the system can be achieved. By extending the hydrophobic membrane through more than one of the reactors an effective and efficient recovery system can be provided.

In a further preferred embodiment according to the present invention the anode and/or cathode is integrated with the hydrophobic membrane.

By integrating the cathode with the hydrophobic membrane, in one of the presently preferred embodiments oxygen can be supplied to the cathode. This obviates the need of dissolving/sparging/aeration of oxygen or air into the catholyte media in the cathode compartment. Preferably, the cathode is directly integrated with membrane fibers of the hydrophobic membrane, optionally using a carbon based catalyst, optionally enriched with noble metals, for direct integration. This enables oxygen reduction at the cathode in an effective manner.

In a further preferred embodiment according to the present invention the hydrophobic membrane is configured for extracting CO₂ from the electrolyte of the anode compartment and enriching electrolyte of the cathode compartment.

By providing a hydrophobic membrane in or connected to the anode compartment the electrolyte of the anode compartment can be brought into contact with the hydrophobic membrane, thereby enabling extraction/transfer of CO_2 . More specifically, the flow through the flow channel as defined by the hydrophobic membrane comprises electrolyte from the cathode compartment using the CO_2 to acidify/buffer/counteracting the hydroxyl ion production at the cathode by recycling the electrolyte of the cathode compartment through the flow channel defining hydrophobic membrane. This achieves extracting CO_2 from the electrolyte from the anode compartment and enriching the electrolyte of the cathode compartment.

In a further preferred embodiment according to the present invention, the bioelectrochemical system further comprises a fuel cell or engine configured for generating electricity with gasses removed from the reactor.

By generating electricity using hydrogen fuel, for example, electricity can be generated to further improve the overall energy efficiency of the bio-electrochemical system according to the present invention. This may even result in a stand-alone application that can be operated in remote areas.

It will be understood that the different features that are described can be applied in the system, and method, according to the invention when applied for component recovery, electrical energy generation and/or a combination thereof.

The invention further relates to a method for recovery of components or generating electrical energy from a waste stream, comprising the steps of:

- providing a bio-electrochemical system as described above;
- supplying a waste stream to the reactor;
- supplying a reactant to and/or extracting a gas from the reactor with the flow channel defining hydrophobic membrane; and
- operating the reactor.

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The method provides the same effects and advantages as those described for a bioelectrochemical system.

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In a presently preferred embodiment the recovery of the components involves recovery of ammonia. For example, the method treats urine that comprises several organic compounds and having an ammonium-nitrogen concentration as high as 10 g/l, for example. Also other ammonium comprising waste streams can be treated. Furthermore, energy can be gained from the process, and organic material and ammonia and/or ammonium can be removed from the (waste) fluid.

In a further embodiment electrical energy is generated by providing oxygen to the reactor, preferably by a flow channel defining hydrophobic membrane.

In a further embodiment electrical energy is generated by providing oxygen to the reactor and recovering components, such as ammonia, thereby combining the two aforementioned embodiments.

Further advantages, features and details of the invention are elucidated on the basis of preferred embodiments thereof, wherein reference is made to the accompanying drawings, in which:

- Figure 1A shows a reactor with the hydrophobic membrane in the cathode compartment enabling component recovery;
- Figure 1B shows a reactor with the hydrophobic membrane in the cathode compartment enabling generating electrical energy;
- Figure 1C shows a reactor with the hydrophobic membrane in the cathode compartment enabling both component recovery and generating electrical energy;
- Figure 2 shows an alternative reactor according to the invention with the hydrophobic membrane in the anode compartment;
- Figure 3 shows an alternative embodiment of the hydrophobic membrane positioned in an intermediate compartment;
- Figure 4 shows a design of a hydrophobic membrane in a compartment;
- Figure 5 shows a number of stacked reactors according to the invention;
- Figure 6A shows a hydrophobic membrane integrated with the cathode in an embodiment for generating electrical energy;
- Figure 6B shows a hydrophobic membrane integrated with the cathode in an embodiment for generating electrical energy and component recovery;
- Figure 7 shows a reactor with a separate anode compartment for CO₂ removal with a hydrophobic membrane; and
- Figure 8 A-E illustrates some experimental results with a reactor of figure 1.

The figures comprise some of the relevant reactions that may take place in the system according to the invention. For illustrative purposes the reactions are presented with their components only, without showing the exact stoichiometric balanced reactions.

Bio-electrochemical system 2 (figure 1A) comprises reactor 4 with anode compartment 6 which comprises bio-anode 8 with bio film 10. Reactor 4 further comprises cathode compartment 12 with cathode 14. Anode 8 and cathode 14 are connected with circuit 16 involving power source 18a. Anode compartment 6 and cathode compartment 12 are separated with cation exchange membrane 20. Hydrophobic membrane 22 defining flow channel 24 is positioned in cathode compartment 12. Waste water inlet 26 is connected to anode compartment 6 that further comprising waste water outlet 28. Optionally gas outlet 30 is provided in cathode compartment 12.

In use, in system 2 as illustrated in figure 1A, is capable of component recovery. At anode 8 bio-catalyzed anode reactions take place that involve oxidation of organic matter resulting in a production of H⁺ and CO₂. Cations like NH₄⁺ and Na⁺ transfer through membrane 20 towards cathode compartment 12. At cathode 14 reduction reactions reduce water in OH and H₂. This increases the pH in cathode compartment 12. In cathode compartment 12 ionic ammonium is deprotonated into ammonia (aq) with OH. Due to the ammonia-ammonium equilibrium NH₃ and H₂O is produced, with NH₃ diffusing over hydrophobic membrane 22 into flow channel 24. Preferably, flow channel 24 receives acid through inlet 32 enabling chemosorbing ammonia into ammonium with H⁺ and leaving at ammonium outlet 34. It will be understood that other configurations can also be envisaged, for example including positioning hydrophobic membrane 22 outside cathode compartment 12 and involving a hydraulic connection between membrane 22 and compartment 12.

System 2 as illustrated in figure 1B is capable of generating electrical energy. Oxygen (O_2) is supplied to cathode compartment 12 by inlet 29 and/or hydrophobic membrane 24 to enable the oxygen reduction reaction $(O_2 + 2H_2O + 4e^- --> 4OH^-)$ producing OH $^-$. Unspecified ions Q+ and Q- are transported over the anion/cation selective membrane 20 in accordance with the electric field. In the illustrated embodiment system 2 acts as a MFC with resistor 18b included in circuit 16. This enables production of electrical energy.

By providing hydrophobic membrane 24 it is possible to combine electrical energy generation with component recovery, such as ammonia and/or ammonium, in system 2 (figure 1C). Oxygen reduction takes place $(O_2 + 2H_2O + 4e^- --> 4OH^-)$ in cathode compartment 12. In the illustrated embodiment oxygen is only supplied by membrane 24. It will be understood that oxygen can, alternatively or in addition thereto, be supplied with a separate inlet 29 and/or an additional flow channel defining hydrophobic membrane. At anode 8 bio-catalyzed anode reactions take place that involve oxidation of organic matter resulting in a production of H^+ and CO_2 . Cations like NH_4^+ and Na^+ transfer through membrane 20 towards cathode compartment 12. In cathode

compartment 12 ionic ammonium is deprotonated into ammonia (aq) with OH⁻. Due to the ammonia-ammonium equilibrium NH₃ and H₂O is produced, with NH₃ diffusing over hydrophobic membrane 22 into flow channel 24. Preferably, flow channel 24 receives acid through inlet 32 enabling chemosorbing ammonia into ammonium with H⁺ and leaving at ammonium outlet 34.

Further embodiments according to the invention will be described and/or illustrated disclosing further features. It will be understood that these features can be combined to provide even further embodiments. Also, it will be understood that these features can be applied to configurations capable of component recovery, electrical energy generation and/or the combination thereof.

Alternative system 42 (figure 2) comprises reactor 44 comprising similar components as mentioned relation to system 2 illustrated in figure 1A-C. The differences between system 42 illustrated in figure 2 and system 2 illustrated in figure 1 will be described. Anode compartment 6 and cathode compartment 12 are separated by anion exchange membrane 46. Hydrophobic membrane 22 is positioned in anode compartment 6. The same reactions can take place in reactor 44 as described in relation to reactor 4. OH is transferred over anion exchange membrane 46. In the illustrated embodiment the membrane fibers are positioned close to anion exchange membrane 46 where the OH concentration and the pH in the anode compartment is the highest, thereby allowing ammonia recovery from the anode compartment in an effective manner. This specific location for hydrophobic membrane 22 is preferably close to membrane 46 allowing for ammonia recovery from anode compartment 6 which is usually acidifying due to the anodic reactions. It will be understood that membranes 20, 46 can optionally be integrated with hydrophobic membrane 22.

System 52 (figure 3) comprises reactor 54 with similar components as described before in relation to systems 2, 42 that are illustrated in figures 1, 2. Reactor 54 comprises an additional intermediate compartment 56 that is separated with anion exchange membrane 58 from cathode compartment 12 and cation exchange membrane 60 from anode compartment 6. Hydrophobic membrane 22 is in the illustrated embodiment positioned in intermediate compartment 56. The same or similar reactions can take place in reactor 54 as described for reactors 4, 44. It will be understood that anions like OH may transfer over anion exchange membrane 58 and cations like Na⁺ can transfer over cation exchange membrane 60 towards intermediate compartment 56. This improves the overall performance of the bio-electrochemical system 52 and allows for a compact design.

Hydrophobic membrane 22 (figure 4) is preferably provided in a straw-type configuration. In the illustrated embodiment one straw-hydrophobic membrane 22 is shown in reactors 4, 44, 54. It will be understood that any number of hydrophobic membranes 22 can be provided in compartments of reactors 4, 44, 54. Optionally, additional hydrophobic membranes 22 can be provided in other compartments of reactors 4, 44, 54.

Bio-electrochemical system 72 (figure 5) comprises a number of reactors 74, 76 with one hydrophobic membrane 22 extending through reactors 74, 76. This enables upscaling of bio-electrochemical system 72 in an effective and efficient manner.

In a further alternative embodiment system 82 with reactor 84 (figure 6A) comprises cathode system 86. Cathode system 86 comprises cathode 88 and hydrophobic membrane 90 that have been integrated. This enables supply of oxygen (O₂) to cathode system 12.

It will be understood that cathode system 86 can also be provided to the other bioelectrochemical system configurations 2, 42, 52, 72.

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In the illustrated embodiment cathode system 86 supplies oxygen to the cathode compartment, thereby enabling reduction at cathode 86. Unspecified ions Q+ and Q- are transported over the anion/cation selective membrane in accordance with the electric field. In the illustrated embodiment system 82 acts as a MFC with a resistor included in the circuit. This enables production of electrical energy. By providing an additional hydrophobic membrane, as shown in one or more of the other embodiments, recovery of components, such as ammonia and/or ammonium can be achieved.

In an alternative configuration (figure 6B) cathode system 86 is combined with flow channel defining hydrophobic membrane 24 as illustrated in figure 1A, for example. This enables an effective electrical energy generation with cathode system 86 in combination with component recovery as described in relation to figure 1A and figure 1C.

A further alternative system 92 (figure 7) provides reactor 94 with separate membrane module 96. Membrane module 96 is connected with input 98 receiving electrolyte from anode compartment 6 and returning treated electrolyte through exit 100 to anode compartment 6. Hydrophobic membrane 102 is positioned in module 96 and is, in use, supplied with electrolyte from cathode compartment 12 at its input 104. The (enriched) electrolyte leaves membrane 102 through exit 106 to cathode compartment 12. System 92 is advantageously used for acidifying/buffering the cathode counteracting the hydroxyl ion production at the cathode and thereby lowering losses due to an increasing pH.

As already mentioned features of embodiments described and/or illustrated can be combined and/or applied in other configurations according to the invention. For example, more than one hydrophobic membrane 24 can be provided in a system according to the invention.

Experiments have been performed with reactors shown in figures 1-7. In an ammonia removal experiment from human urine as waste stream with bio-electrochemical system 2 illustrated in figure 1, the current was measured during operation (figure 8A) during a time period of more than 20 days showing measured currents in the range of 0.2-0.35A. The recovery percentage of nitrogen from the influent in the experiment lies between 30%-55% (figure 8B) and COD removal between 20%-50% (figure 8D). The so-called coulombic efficiency (figure 8C)

determined from the COD removal and the measured current lies between 50%-100%. The transport efficiency over cation exchange membrane 20 (figure 8E) is between 40% and 100% during the experiment based on transported charge (coulombs) in the form of ammonium and current.

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The experiment illustrates the possible use of the configurations according to the present invention. The use of hydrophobic membrane 22 provides improved results of the process in bio-electrochemical system 2 as compared to a configuration without any hydrophobic membrane 22.

The present invention is by no means limited to the above described preferred embodiments thereof. The rights sought are defined by the following claims within the scope of which many modifications can be envisaged. For example, the bio-electrochemical system 2, 42, 52, 72, 82, 92 can be operated batch wise or continuously with a waste stream that may comprise ammonium.

Clauses

- 1. Bio-electrochemical system for recovery of components or generating electrical energy from a waste stream, the system comprising a reactor that comprises:
- an anode compartment with an anode;

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- a cathode compartment with a cathode, wherein at least one of the anode and cathode is a bio-electrode;
- a circuit connecting the anode and the cathode, the circuit comprising a power source for providing an electric current or a resistor;
- an ion-exchange membrane separating the anode and cathode compartments; and
 - a flow channel defining hydrophobic membrane configured for gas extracting and/or reactant supply.
- Bio-electrochemical system according to clause 1, wherein the flow channel defining
 hydrophobic membrane comprises a tubular element.
 - 3. Bio-electrochemical system according to clause 1 or 2, further comprising an intermediate compartment between the anode and cathode compartments, the intermediate compartment comprising separating ion-exchange membranes.

4. Bio-electrochemical system according to clause 3, wherein the hydrophobic membrane is positioned in the intermediate compartment.

- 5. Bio-electrochemical system according to one or more of the foregoing clauses, wherein the hydrophobic membrane is positioned adjacent to a separating ion exchange membrane.
 - 6. Bio-electrochemical system according to clause 5, wherein the hydrophobic membrane is integrated with a separating ion-exchange membrane.
- Bio-electrochemical system according to one or more of the foregoing clauses, wherein the anode comprises a bio-electrode.
 - 8. Bio-electrochemical system according to one or more of the foregoing clauses, further comprising a number of additional reactors with the flow channel defining hydrophobic membrane extending through more than one of the reactors.

- 9. Bio-electrochemical system according to one or more of the foregoing clauses, wherein the anode and/or cathode is integrated with the hydrophobic membrane.
- Bio-electrochemical system according to one or more of the foregoing clauses, wherein the
 hydrophobic membrane is configured for extracting CO₂ from the electrolyte of the anode compartment and enriching electrolyte of the cathode compartment.
 - 11. Bio-electrochemical system according to one or more of the foregoing clauses, further comprising a fuel cell or engine configured for generating electricity with gasses removed from the reactor.
 - 12. Method for recovery of components or generating electrical energy from a waste stream, comprising the steps of:
 - providing a bio-electrochemical system according to one or more of the foregoing clauses:
- supplying a waste stream to the reactor;
 - supplying a reactant to and/or extracting a gas from the reactor with the flow channel defining hydrophobic membrane; and
 - operating the reactor.

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20 13. Method according to clause 12, wherein the recovery of the components involves recovering of ammonia and/or ammonium.

Conclusies

- 1. Bio-elektrochemisch system voor terugwinning van componenten of genereren van elektrische energie uit een afvalstroom, het systeem omvattende een reactor welke omvat:
 - een anode compartiment met een anode;

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- een kathode compartiment met een kathode, waarin ten minste één van de anode en kathode een bio-elektrode is;
- een circuit voor het verbinden van de anode en de kathode omvattende een voedingsbron voor het voorzien van een elektrische stroom of een weerstand;
- een ion-uitwisselingsmembraan scheidend het anode compartiment en het kathode compartiment; en
 - een stromingskanaal definiërend hydrofoob membraan geconfigureerd voor gasextractie en/of reactant toevoer.
- 15 2. Bio-elektrochemisch systeem volgens conclusie 1, waarin het stromingskanaal definiërend hydrofoob membraan een buisvormig element omvat.
 - 3. Bio-elektrochemisch systeem volgens conclusie 1 of 2, verder omvattende een additioneel compartiment tussen de anode en kathode compartimenten, waarbij het additioneel compartiment scheidende ion-uitwisselingsmembranen omvat.
 - 4. Bio-elektrochemisch systeem volgens conclusie 3, waarin het hydrofobe membraan gepositioneerd is in het additionele compartiment.
- 5. Bio-elektrochemisch systeem volgens een of meer van de voorgaande conclusies, waarin het hydrofobe membraan gepositioneerd is nabij een scheidend ionuitwisselingsmembraan.
- 6. Bio-elektrochemisch systeem volgens conclusie 5, waarin het hydrofobe membraan 30 geïntegreerd is met een scheidend ion-uitwisselingsmembraan.
 - 7. Bio-elektrochemisch systeem volgens een of meer van de voorgaande conclusies, waarin de anode een bio-elektrode omvat.

- 8. Bio-elektrochemisch systeem volgens één of meer van de voorgaande conclusies, verder omvattende een aantal additionele reactors met het stromingskanaal definiërend hydrofoob membraan zich uitstrekkend door meer dan één van de reactoren.
- 5 9. Bio-elektrochemisch systeem volgens één of meer van de voorgaande conclusies, waarin de anode en/of kathode geïntegreerd is met het hydrofobe membraan.
 - 10. Bio-elektrochemisch systeem volgens één of meer van de voorgaande conclusies, waarin het hydrofobe membraan geconfigureerd is voor het extraheren van CO₂ uit het elektrolyt van het anode compartiment en het verrijken van het elektrolyt van het kathode compartiment.
 - 11. Bio-elektrochemisch systeem volgens één of meer van de voorgaande conclusies, verder omvattende een brandstofcel of motor geconfigureerd voor het genereren van elektriciteit met gassen verwijderd uit de reactor.
 - 12. Werkwijze voor het terugwinnen van componenten of het genereren van elektrische energie uit een afvalstroom, omvattende de stappen:
 - het voorzien van een bio-elektrochemisch systeem volgens één of meer van de voorgaande conclusies;
 - het toevoeren van een afvalstroom aan de reactor;
 - het toevoeren van een reactant aan en/of het extraheren van een gas uit de reactor met een stromingskanaal definiërend hydrofoob membraan; en
 - het bedrijven van de reactor.

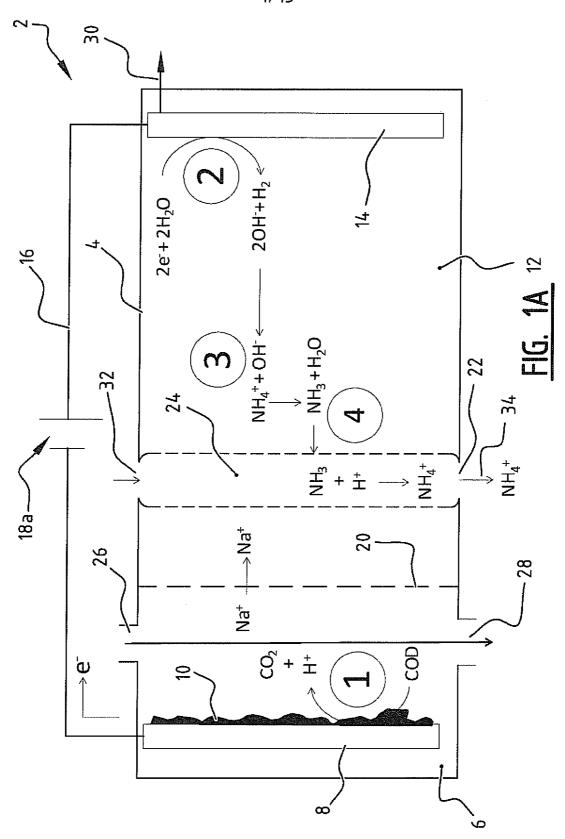
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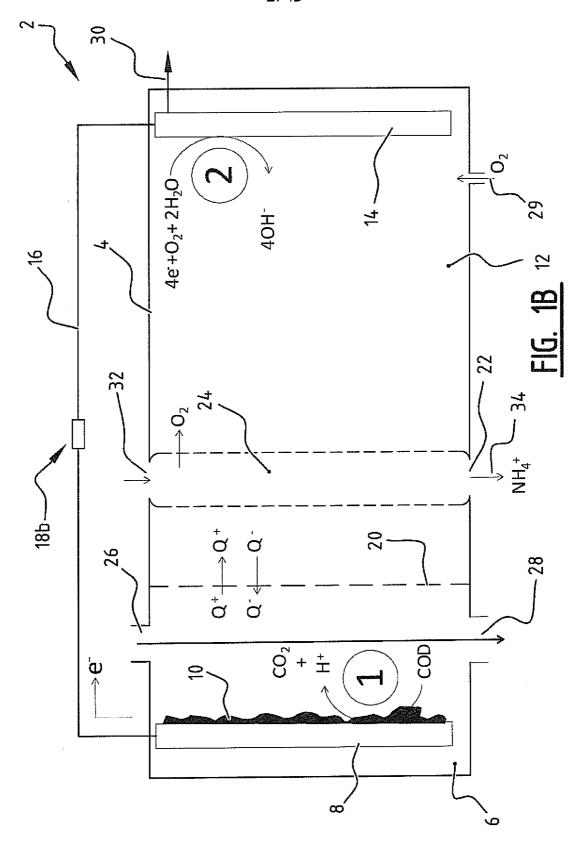
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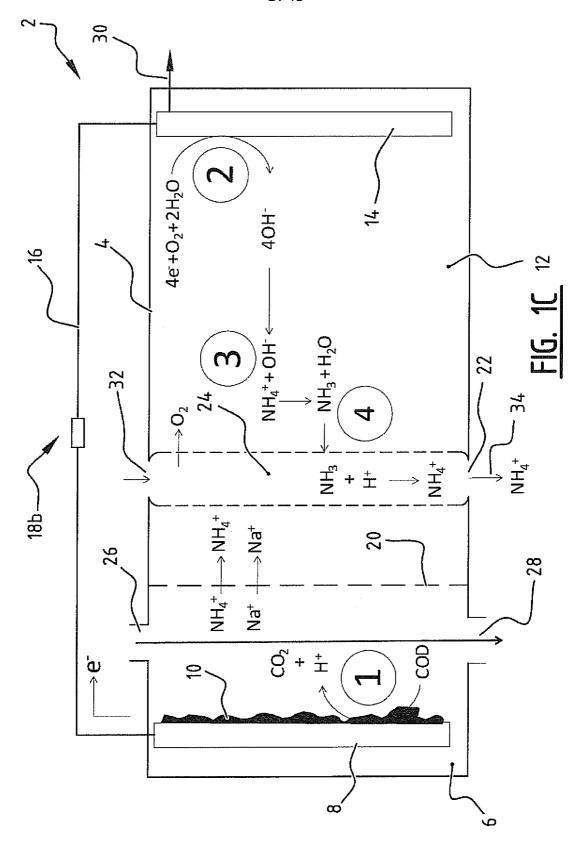
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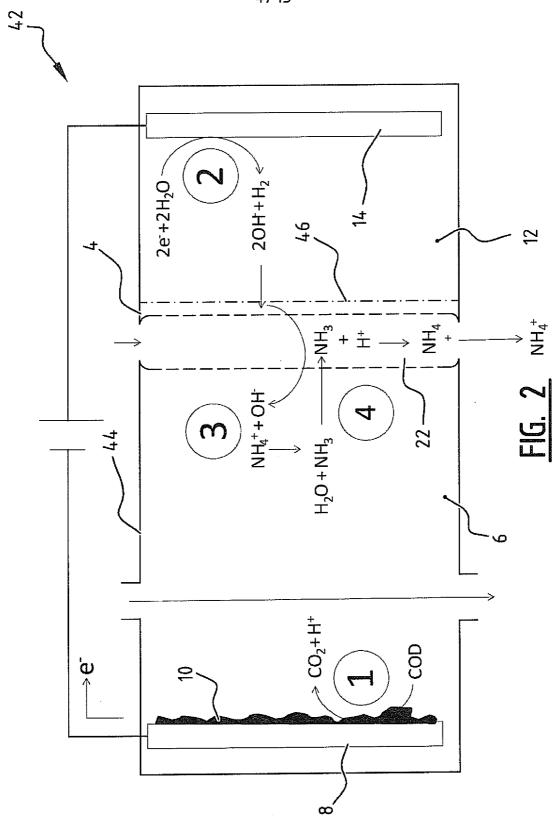
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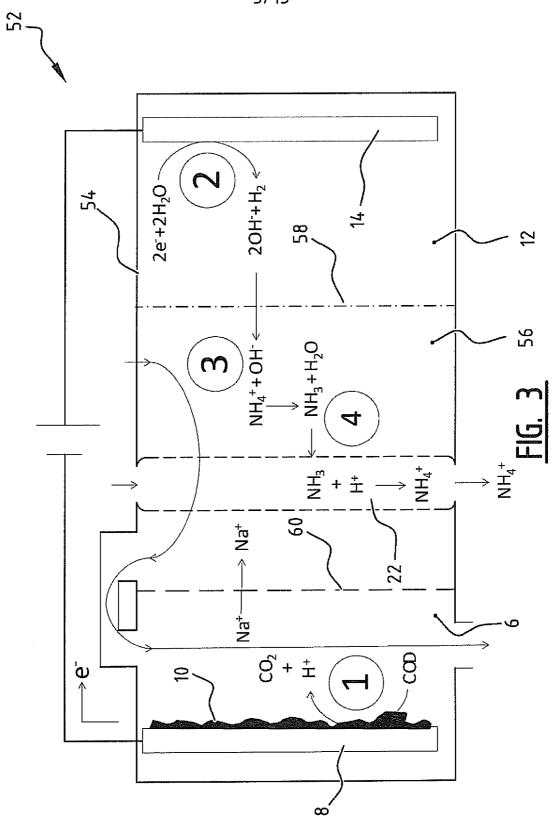
13. Werkwijze volgens conclusie 12, waarin we terugwinnen van de componenten het terugwinnen van ammonia en/of ammonium omvat.

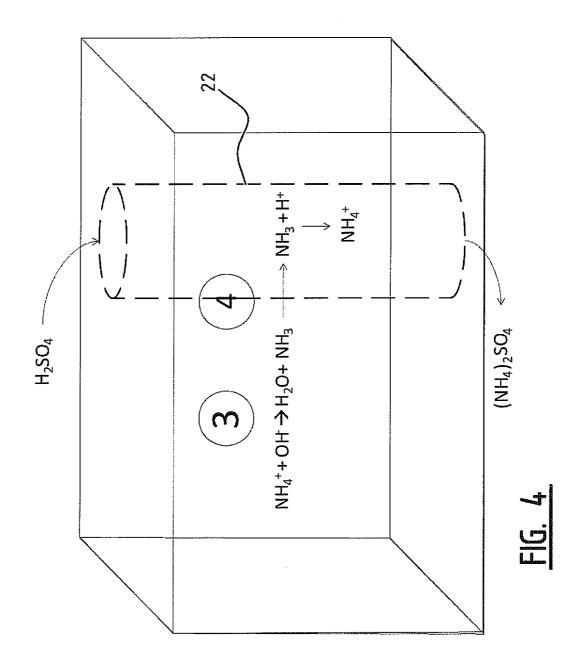












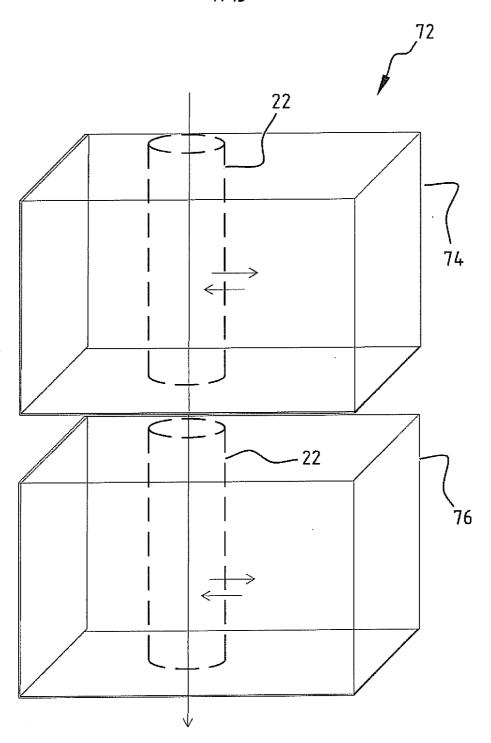
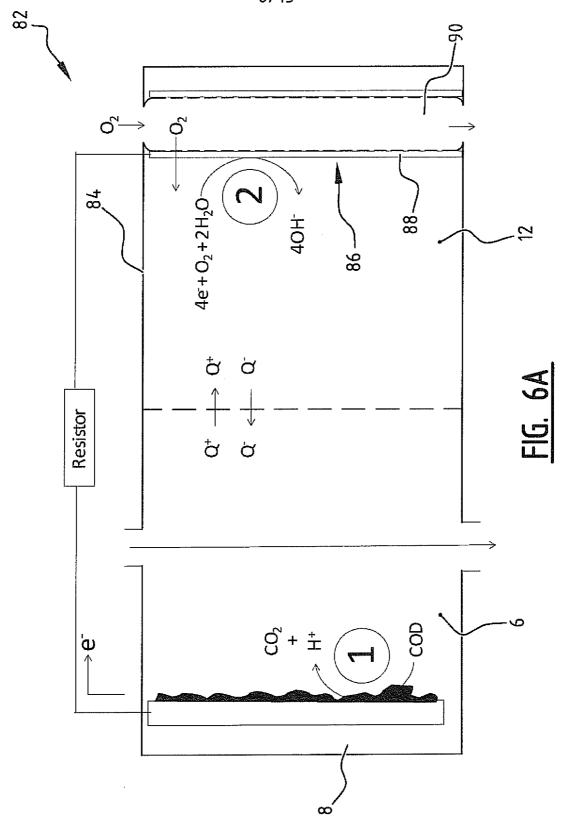
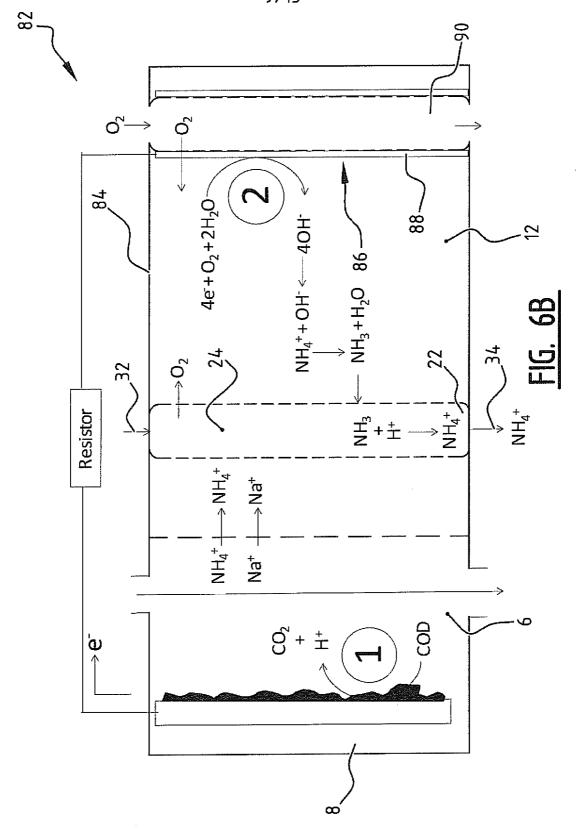
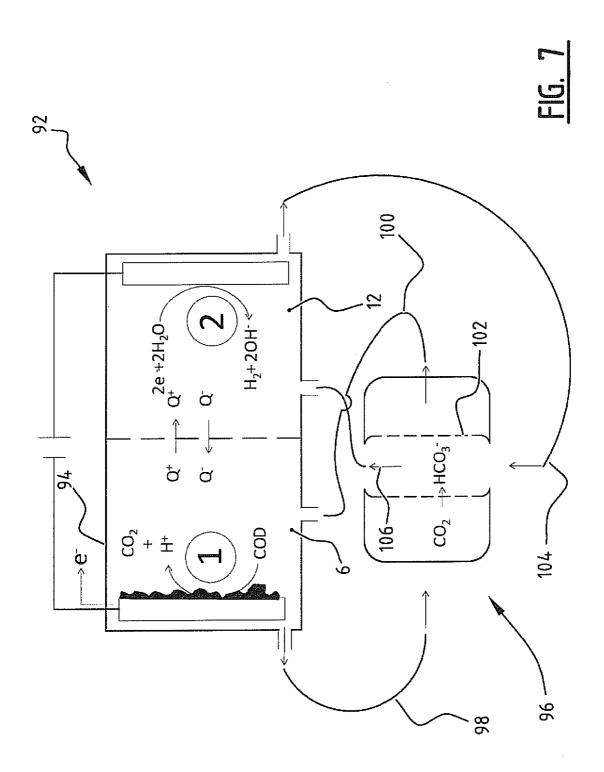
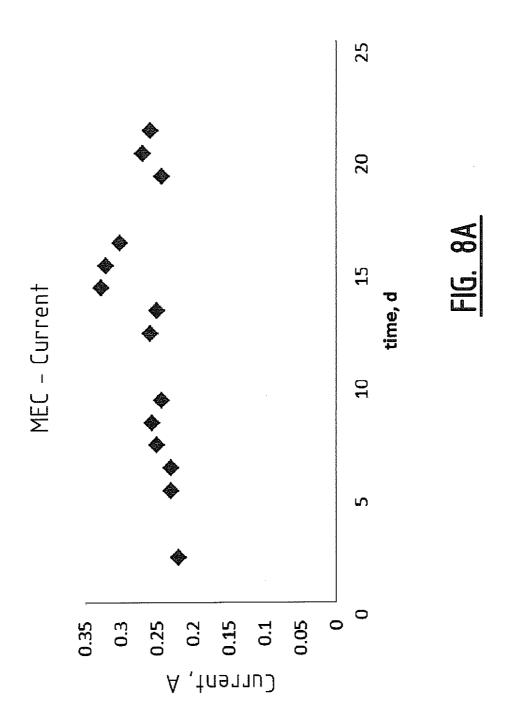


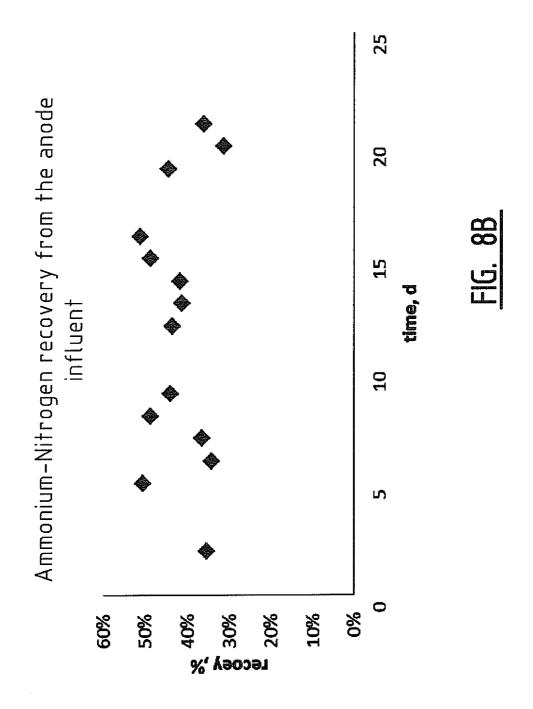
FIG. 5

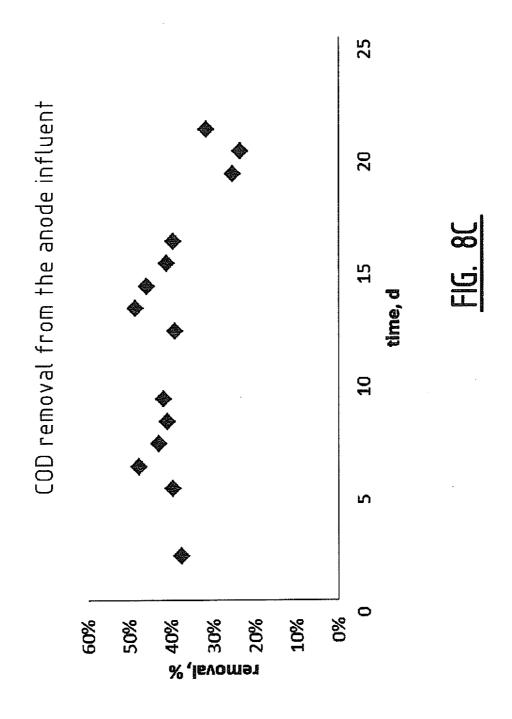


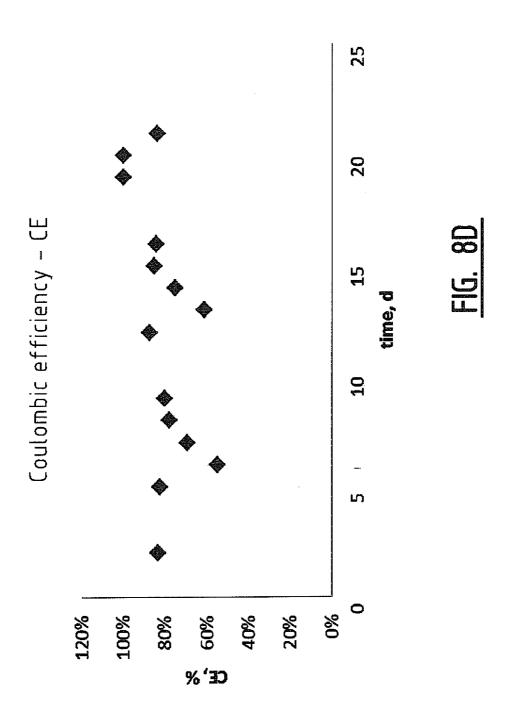


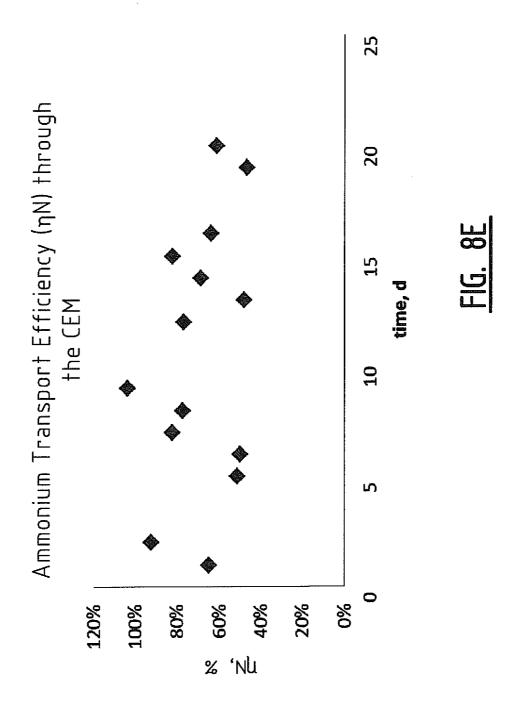












Abstract

The invention relates to a bio-electrochemical system and method for recovery of components or generating electrical energy from a waste stream. The system according to the invention comprises:

- an anode compartment with an anode;

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- a cathode compartment with a cathode, wherein at least one of the anode and cathode is a bio-electrode;
- a circuit connecting the anode and the cathode, the circuit comprising a power source for providing an electric current or a resistor;
- an ion-exchange membrane separating the anode and cathode compartment; and
- a flow channel defining hydrophobic membrane configured for gas extracting and/or reactant supply.

SAMENWERKINGSVERDRAG (PCT)

RAPPORT BETREFFENDE NIEUWHEIDSONDERZOEK VAN INTERNATIONAAL TYPE

IDENTIFICATIE VAN DE NATIONALE AANVRAGE		KENMERK VAN DE AANVRAGER OF VAN DE GEMACHTIGDE 2L/2RE39/CAK/67		
Nederlands sanvrasg nr.		indisningsdatum		
2014797		12-05-2015		
		Ingercepen voorrangsdatum		
Assyrager (Naam) Stichting Wetsu Technology	ıs, European Ceni	lre of Excellence for Sustair	nable Water	
Oahen van hat verzoek voor:	een onderzoek van	Toor de Instantie voor Internationaal Onderzoek aan		
internationasi type		het verzoek voor een onderzoek van loegekend m:	internaliowasi lype	
20-06-2015		SN 64346		
I. CLASSIFICATIE VAN HET	ONDERWERP (bi) tospa	ssing van verschillende classificaties, alle cl	assificatiesymbolen opgeven)	
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III. T GEEN ONDERZOEK	MOGELIJK VOOR BEP	AALDE CONCLUSIES (namerkinnen	cp aanvulingsbiasi	
IV. GEBRIEK AAN EENH			op aanvullingabled)	
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Automore van het versteek dit een ondersteek naar de stand van de techniek

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Desbois, Valérie

Recommen your het verocoek com even condenzasek maar de stood van de technisek

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Categorie :	Geobende documentes, eventuel met auroboling van epeciaal van belang zijnde paesigee	Van belang voor sonolopie in
X	US 2006/011491 A1 (LOGAN BRUCE [US] ET AL) 19 januari 2006 (2006-01-19) * alinea (0084] * * alinea (0085); figuur 4 * * alinea (0091); figuur 9 * * alinea (0039); figuur 12 *	1,2,7-9, 11,12 13
¥ A	WO 2013/105854 A1 (STICHTING WETSUS CT EXCELLENCE SUSTAINABLE WATER TECHNOLOGY [NL]) 18 juli 2013 (2013-07-18) * het gehele document *	13 1,3,10,
	20 30 Jan 20 20 30 30 20 20 20 20 20 20 20 20 20 20 20 20 20	12
Å	EP 0 247 748 Al (ICI PLC [GB]) 2 december 1987 (1987-12-02) * kolom 3. regel 28 - kolom 5. regel 4 * * kolom 7. regels 37-48 *	2
۸	US 2005/000798 Al (FAÏTA GIUSEPPE [IT] ET AL) 6 januari 2005 (2005-01-06) * conclusies 1,2 *	5,6
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WO 2012001061	Al 05-91-2012	CA 2797474 A1 CN 102918690 A DK 2548246 T3 EP 2548246 A1 ES 2546885 T3 HR P20150922 T1 JP 20135396541 A KR 20130098148 A RU 2012144242 A SI 2548246 T1 US 2013101906 A1 WO 2012001061 A1	08-11-201 05-01-201 06-02-201 14-09-201 23-01-201 29-09-201 19-09-201 10-08-201 30-11-201 25-04-201	
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WRITTEN OPINION

F% No SN6434	16	Fäng date (day/month)-ear) 12.05.2015	Priority data (day/month/year)	Application No. NL2014797
			C258500 H01M8/16 H01M8/02:	3 H01M8/0252 H01M8/04089
Applicant Stichtin		μιοφείαη Centre of Excellence	for Sustainable Water Technok	Y0Y
Tì	is opinion co	intains indications relating to f	the following items:	
63	Box No. i	Basis of the opinion		
O	Box No. II	Priority		
O	Box No. III	Non-establishment of opinion w	iffi regard to novelly, inventive step	and industrial applicability
O	Box No. IV	Lack of unity of invention		
83	Bax No. V	Reasoned statement with regar applicability; citations and expla	d to novelty, inventive step or indus mations susporting such statement	trial Can
O	Box No. Vi	Cedain documents cited	en kalandaria. Makabahda ina nakabah masada kilabahnak walabi mari ina kaba. Wina	
	Box No. VII	Certain defects in the application	86	
a	Box No. VIII	Certain observations on the app	plication	
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			Examinar	
			Cashnie Valèrie	

Form NL237A (Dekblad) (July 2005)

WRITTEN OPINION

ست	Box M	No. I Basis of this opinio	<u> </u>					
 1				basis of the	he latest set of claims filed before the start of the search.			
	With regard to any nucleotide and/or amino acid sequence disclosed in the application and necessary to the claimed invention, this opinion has been established on the basis of:							
	a, type of material:							
	O	a sequence listing						
		table(s) related to the seq	uence l	sting				
	b. format of material:							
	O	Cl on paper						
	O	in electronic form						
	c. time of filing/furnishing:							
	a	Contained in the application as filed.						
	O	C filed together with the application in electronic form.						
	O	C3 furnished subsequently for the purposes of search.						
3.	h O	has been filed or furnished, t	he requ	red staten	sion or copy of a sequence listing and/or table relating thereto nents that the information in the subsequent or additional lied or does not go beyond the application as filled, as			
4.	Additional comments:							
cococ	Box N	No V - Reasoned statems	est weits	regard to	novelty, inventive step or industrial applicability;			
		ons and explanations sup	porting	such stat	eucuj			
1,	States	Statement						
	Novel	Мy	Yes:	Claims	4-6, 8, 10, 11, 13			
	udu vitu (Mathinu)		No:	Claims	1-3, 7, 9, 12			

4-6, 10

1-13

1-3, 7-9, 11-13

Yes: Claims No: Claims

Yes: Claims

No: Claims

2. Citations and explanations

Industrial applicability

see separate sheet

Inventive step

Re Item V

Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

Reference is made to the following documents:

- D1 WO 2014/150415 A1 (SOLINA BRENT A [US]) 25 september 2014 (2014-09-25)
- D2 WO 2012/001061 A1 (VITO NV [BE]; ALVAREZ GALLEGO YOLANDA [BE]; VERMEIREN PHILIPPE [BE]; C) 5 januari 2012 (2012-01-05)
- D3 US 2006/011491 A1 (LOGAN BRUCE [US] ET AL) 19 januari 2006 (2006-01-19)
- D4 WO 2013/105854 A1 (STICHTING WETSUS CT EXCELLENCE SUSTAINABLE WATER TECHNOLOGY [NL]) 18 juli 2013 (2013-07-18)

2 The present application does not meet the criteria of patentability, because the subject-matter of claims 1 and 12 is not new.

2.1 D1 discloses a :

Bio-elektrochemisch system voor genereren van elektrische energie uit een afvalstroom (paragraph [0093]), het systeem omvattende een reactor welke omvat:

- een anode compartiment met een anode (paragraph [0106], Fig. 1, ref. 102);
- een kathode compartiment met een kathode, waarin ten minste één van de anode en kathode een bio-elektrode is (paragraphs [0105]-[0106], Fig. 1, ref. 106);
- een circuit voor het verbinden van de anode en de kathode omvattende een weerstand (paragraph [0106]);
- een ion-uitwisselingsmembraan scheidend het anode compartiment en het kathode compartiment (paragraph (0101)); en
- een stromingskanaal definiërend hydrofoob membraan geconfigureerd voor gasextractie en/of reactant toevoer (paragraph [0104]).

2.2 D2 discloses a:

Bio-elektrochemisch system voor genereren van elektrische energie uit een afvalstroom (Fig. 10 and 11, p. 9, l. 12-21; claim 7), het systeem omvattende een reactor welke omvat:

een anode companiment met een anode (claim 8);

- een kathode compartiment met een kathode (fig. 11, ref. 4 ; claim 8), waarin ten minste één van de anode en kathode een bio-elektrode is (claim 10);
- een circuit voor het verbinden van de anode en de kathode omvattende een weerstand (claim 8);
- een ion-uitwisselingsmembraan scheidend het anode compartiment en het kathode compartiment (Fig. 11, ref. 6 ; claim 7) ; en
- een stromingskanaal definièrend hydrofoob membraan geconfigureerd voor gasextractie en/of reactant toevoer (Fig. 10, ref. 3 ; p. 15, l. 12-18 ; claim 6).
- 2.3 The subject-matter of **claim 1** is not distinguishable from either teaching of D1 or D2.
- 2.4 The same reasons applying mutatis mutandis, the subject-matter of claim 12 concerning the generation of electricity is not distinguishable from either teaching of D1 or D2.
- 3 The present application does not meet the criteria of patentability, because the subject-matter of claims 1 and 12 does not involve an inventive step.
- 3.1 D3, which is considered to represent the most relevant state of the art to the subject-matter of claim 1 concerning the winning of a compound, discloses a "bio-elektrochemisch system voor terugwinning van componenten" (paragraph [0091], Fig. 5) from which the subject-matter of claim 1 differs in that it comprises a "ion-wisselingsmembraan scheidend het anode compartiment en het kathode compartiment".
- 3.2 The problem to be solved by the present invention may therefore be regarded as avoiding the contact of the analyte with the cathode or vice-versa.
- 3.3 The solution proposed in claim 1 of the present application cannot be considered as involving an inventive step:
 D1 suggests to optionally use a membrane for a similar apparatus (paragraph [0085], Fig. 4). Therefore, the skilled person, according to the electrochemical reactions being involved, would separate the analyte from the catholyte by an ion-exchange membrane, without the exercise of inventive skills.
- 3.4 The same reasons applying mutatis mutandis, the subject-matter of claim 12 concerning the winning of a compound, which is therefore not considered inventive.

- 4 Dependent claims 2, 3, 7-11, 13 do not contain any features which, in combination with the features of any claim to which they refer, meet the requirements of novelty and/or inventive step, see:
- 4.1 Of discloses a "hydrofoob membraan" comprising a "buisvormig element" (paragraph [0091] "tubular modules"), according to claim 2.
- 4.2 D1 discloses an "additioneel compartiment" (paragraph [0091]) "one or more membranes"), according to claim 3.
- 4.3 D1 discloses an anode comprising a "bio-elektrode" (paragraph [0105] "electrons are produced by an oxidation (...) by the electroactive biofilm"), according to claim 7.
- 4.4 D1 and D2 disclose a cathode which is "geïntegreerd (...) met het hydrofobe membraan" (D1, paragraph [0104] and D2, Fig. 10), according to claim 9.
- 4.5 Therefore the subject-matter of claims 2, 3, 7 and 9 is not novel.
- 4.6 D3 discloses a "hydrofoob membraan" comprising a "buisvormig element" (paragraph [0039], Fig. 12), according to claim 2.
- 4.7 D3 discloses an anode comprising a "bio-elektrode" (paragraph [0085], Fig.4), according to claim 7.
- 4.8 In **claim 8** a slight constructional change in the bio-electrochemical system of claim 1 is defined which comes within the scope of the customary practice followed by persons skilled in the art, especially as the advantages thus achieved can readily be foreseen.
- 4.9 D3 discloses a cathode which is "geïntegreerd (...) met het hydrofobe membraan" (paragraph [0091], Fig. 9), according to claim 9.
- 4.10 D3 discloses a system comprising "een motor geconfigureerd voor het genereren van elektriciteit met gassen verwijderd uit de reactor" (paragraph [0084]), according to claim 11.
- 4.11 O4 discloses the "terugrwinnen van ammonia en/of ammonium" in a procesusing a bio-electrochemical system (claim 2), according to **claim 13**.
- 4.12 Therefore the subject-matter of claims 2, 7-9, 11 and 13 is not inventive.
- The combination of the features of dependent claim(s) 4 and 5 is neither known from, nor rendered obvious by, the available prior art. The reasons are as follows:

5.1 There is neither a disclosure nor a suggestion in the available prior art to locate the hydrophobic membrane in a intermediate compartiment or adjacent to the ion-exchange membrane.