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(71) Applicant *(for all designated States except US)*: **STICHTING WETSUS CENTRE OF EXCELLENCE FOR SUSTAINABLE WATER TECHNOLOGY** [NL/NL]; Agora 1, NL-8934 CJ Leeuwarden (NL).

(72) Inventors; and

(75) Inventors/Applicants *(for US only)*: **DLUGOLECKI, Piotr** [NL/NL]; p/a Agora 1, NL-8934 CJ Leeuwarden (NL). **WESSLING, Matthias** [NL/NL]; p/a Agora 1, NL-8934 CJ Leeuwarden (NL). **NIJMEIJER, Kitty** [NL/NL]; p/a Agora 1, NL-8934 CJ Leeuwarden (NL). **METZ, Sybrand Jacob** [NL/NL]; p/a Agora 1, NL-8934 CJ Leeuwarden (NL).

(74) Agent: **VERDIJCK, Gerardus, J., C.**; Arnold+Siedsma, Sweelinckplein 1, NL-2517 GK Den Haag (NL).

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(54) Title: METHOD AND DEVICE FOR MEMBRANE CLEANING IN ELECTRO-MEMBRANE PROCESSES

(57) Abstract: The invention relates to a method for membrane cleaning and a device wherein this method can be applied. The method comprises the steps of : -operating a device for performing an electro-membrane process; and -applying inverse electrical pulses to the device. The device comprises a number of anode and cathode compartments provided with a number of electrodes, a number of cation exchange membranes and a number of anion exchange membranes, placed alternately between the compartments, and function generator means capable of providing inverse electrical pulses. Between or during the pulses, alternating or fluctuating current and /or voltage is applied.

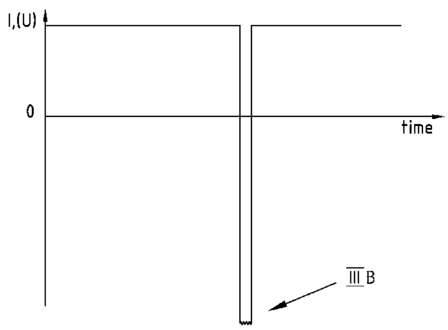

FIG. 3A

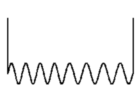

FIG. 3B

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METHOD AND DEVICE FOR MEMBRANE CLEANING IN ELECTRO-MEMBRANE PROCESSES

The present invention relates to cleaning of membranes in a device for performing electro-membrane processes. These processes include electro-dialysis (ED), reverse electro-dialysis (RED), membrane capacitive de-ionisation (CDI) and microbial fuel cells (MFC).

Existing electro-membrane processes are provided with ion exchange membranes that are membranes with fixed anionic or cationic exchange groups able to transport cations or anions. Specific properties of the ion exchange membranes of these charged groups, like membrane resistance, have a large influence on process performance, like the power output obtainable in a reversed electro-dialysis operation, for example.

In existing operations involving membranes, in use electrochemical layers start to build up on the membrane surface. These layers include a so-called electrical double layer and diffusion boundary layers. These layers increase resistance significantly, thereby, for example, reducing the power output obtainable in RED and MFC processes and increasing power consumption in other processes, like ED and CDI. In a continuous operation after a few hours other layers may become more pronounced, like fouling, scaling and bio-fouling layers. Also these layers reduce process efficiency significantly, like the power output obtainable by the process. Cleaning of scaling layers comprising calcium and magnesium salts is often performed using acid cleaning. This disrupts operation of the electro-membrane process for a time period, thereby reducing the overall efficiency of the process. In addition, acid cleaning is relatively expensive and not a very sustainable method. Organic fouling layers are formed by precipitation and/or settling on a membrane surface of organic compounds, like surfactants, colloidal silica and humid acid salts. After some days of operation, bio-fouling layers start to appear on the membrane surface. These layers increase the overall resistance, and also increase the pressure drop over the cell.

The object of the present invention is to improve the overall efficiency of an electro-membrane process.

This object is achieved with the method for membrane cleaning according to the invention, the method comprising the steps of:

- operating a device for performing an electro-membrane process; and
- applying inverse electrical pulses to the device.

Applying inverse electrical pulses to the device for performing electro-membrane process can arrange electro-osmotic instabilities to the membrane surface. As a result of these instabilities ions start to rotate and/or osculate thereby promoting turbulence that leads to breaking the undesired layers on the membrane surface. These undesired layers involve electrical double layers, diffusion boundary layers, scaling layers, organic fouling layers and/or bio-fouling layers.

When a current passes through the ion exchange membrane, charge is carried through the membrane by counter-ions (ions with an opposite charge relative to the fixed charge in the membrane) as a result of the Donan exclusion. In the bulk solution, current is carried by both positive and negative ions. The difference in ion transport number between the solution phase and the membrane leads to the building up of the diffusion boundary layer at the membrane surface. The resistance of this layer may, especially in case of low salt concentrations, form a significant contribution to the total system resistance thereby reducing the power output obtainable in RED and MFC processes and increasing power consumption in other processes, like ED and CDI.

By applying inverse electrical pulses the undesired layers decrease in size or can even be removed entirely, thereby obtaining a clean membrane surface and improving the overall efficiency of the electro-membrane processes. A further effect of applying inverse electrical pulses is that a cleaning effect is achieved without losing too much of the energetic performance of the process, so that this method is energetically favourable as compared to electrodialysis reversal (EDR), for example. EDR requires changing of the polarity of the electric field applied to the electrodialysis stack during a time interval ranging from a few minutes to several hours. In this reverse polarity mode the hydraulic flow streams are reversed simultaneously, i.e. the dilute cell becomes the brine cell and vice versa. Therefore, EDR requires significant power consumption for cleaning purposes, while applying inverse electrical pulses according to the invention shows a more energy-efficient effect on electro-membrane processes. Such electro-membrane processes include electro-dialysis, reverse electro-dialysis, membrane capacitive de-ionisation and microbial fuel cells.

In a preferred embodiment according to the present invention the inverse electrical pulses are applied to electrodes of the device.

Electro-membrane processes, like reverse electro-dialysis, use electrodes to operate the process. By applying the inverse electrical pulse to the electrodes the electrical field in the cell changes its direction as the polarity of the electrodes is changed. This inverse electrical pulse is preferably applied with a very high current that is higher than the limiting current density. Below this limiting current density there is an Ohmic relation between voltage and current. Above this limiting current density, at first, ions stop moving at a higher rate, while with a further increase of the voltage the migration of ions further increases in this over-limiting region. Surprisingly, this over-limiting region shows an improved mixing of the diffusion layer due to the provision of electro-osmotic instabilities by the inverse electrical pulses. This improves the cleaning of the membranes in the electro-membrane processes.

In a preferred embodiment according to the present invention the inverse electrical pulse is applied with a frequency between 10^{-5}-10^{-1} Hz and preferably between $3 \cdot 10^{-4}$ - 10^{-2} Hz.

The frequency of cleaning depends on a number of parameters, for example the dimensions of the device, the specific type of electro-membrane process, etc. Therefore, an inverse electrical pulse should be applied when the device is in use with a frequency of every second to every one day. Experiments have shown that the best effects on the efficiency of the overall process is in the range of every hundred seconds and one hour.

Preferably, the applied pulse is applied with a pulse width in a range of 10^{-2}-10^{2} seconds, and preferably between 1 and 10 seconds. This pulse width is the duration of one electrical pulse. The pulse width depends on several parameters, like the dimensions of the device and the type of electro-membrane process. Furthermore, it depends on the type of layer that is present at the membrane. This type of layer strongly depends on the solutions that are moved through the cell. Experiments have shown that applying pulses with a pulse width in the range of 10^{-2} to 10^{2} seconds duration provides sufficient cleaning. Shorter pulse widths do not appear to have a sufficient cleaning effect, while longer pulse widths do not significantly contribute to this cleaning anymore and, in addition, reduce the production time of the device. Further experiments have shown that a pulse width between 1 and 10 seconds is preferred for most electro-membrane processes.

In a further preferred embodiment according to the present invention applying the inverse electrical pulses comprises providing periodic high frequency alternating or fluctuating current and/or voltage.

Providing this high frequency alternating or fluctuating current and/or voltage has shown a significant effect on especially the bio-fouling layer. For the purpose of this application fluctuating current is defined as an alternating current over a direct current, so, in principle without a polarity difference.

In addition, providing this high frequency alternating or fluctuating current has an extra bonus effect on the other layers, as it improves mixing and extracts particles from the membrane. The periodic high frequency alternating or fluctuating current and/or voltage can in principle be applied at any time during the process.

In one embodiment according to the present invention this periodic high frequency is performed between applying the inverse electrical pulses. This means that this alternating or fluctuating current and/or voltage is applied when the device performing an electro-membrane process is in use.

In a presently preferred embodiment according to the present invention, the periodic high frequency alternating or fluctuating current and/or voltage is performed when applying the inverse electrical pulses. In this way, the effect of this high frequency alternating or fluctuating current and/or voltage is superposed on the effect of the inverse electrical pulse. This periodic high frequency can be applied during the entire electrical pulse width or a part thereof.

Preferably, the periodic high frequency alternating or fluctuating current and/or voltage is in the frequency range of 1 kHz to 1 MHz. It is shown that this frequency range has the largest effect on the killing rate of bacteria in the bio-fouling layer. This high frequency can arrange vibration of the ions, thereby killing the bacteria and viruses in the bio-fouling layer. In addition, there is a bonus affect as this vibration of ions increases breaking the scaling and organic fouling layers. Furthermore, it is shown that this high frequency is also acting as a preventive measure for bio-fouling, scaling and organic fouling layers in all electro-membrane processes.

The present invention also relates to a device for performing electro-membrane process, the device comprising:

- a number of anode and cathode compartments provided with a number of electrodes;
- a number of cation exchange membranes and a number of anion exchange membranes, placed alternately between the compartments; and
- function generator means capable of providing inverse electrical pulses as described above.

Such device provides the same effects and advantages as those stated with reference to the method.

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

- figure 1 illustrates a cation exchange membrane with additional layers;
- figure 2 illustrates an anion exchange membrane with additional layers;
- figure 3 illustrates an application of an inverse electrical pulse with a superposed high frequency alternating current;
- figure 4 illustrates the effect of high current densities on membrane surfaces;
- figure 5 illustrates an experimental setup of the device according to the invention;
- figure 6 illustrates the inverse electrical pulse applied to the device in figure 5; and
- figure 7 illustrates the measured potential as in time during the electrodialysis process.

For the purpose of explaining the behavior of the membranes in electro-membrane processes a possible configuration of a cation and anion exchange membrane with different layers will be discussed briefly. In the illustrated embodiments the different layers are represented by seperate layers. It will be understood that this is a simplification of reality.

A cation exchange membrane 2 (figure 1 with R_M the membrane resistance, R_{DL} the double layer resistance and R_{DBL} the diffusion boundary layer resistance) is in use covered by an electrical double layer 4 and a diffusion boundary layer 6 on the cation exchange membrane surface. The electrical double layers 4 are very thin, typically in the order of nanometers. The diffusion boundary layers 6 have a thickness in a range of 20-400 µm depending on the system and process conditions.

An anion exchange membrane 8 with scaling/fouling layers occurring at the anion/cation exchange membrane and in the layer adjacent to the membrane (figure 2 with R_M the membrane resistance, R_{SC} the scaling layer resistance, R_{OF} the organic fouling layer resistance and R_{BF} the bio-fouling layer resistance) is in use covered by a scaling layer 10, fouling layer 12, and bio-fouling layer 4 on the membrane surface.

An inverse e-pulse (figure 3) with a reverse polarity of the electrodes can be combined with high frequency alternating current. In the illustrated embodiment the high frequency alternating current is applied at the same time as the inverse electrical pulse. It will be understood that other shapes and values for the inverse e-pulse according to the invention are possible.

Applied voltages or currents obtaining inverse electrical pulses are preferably higher than limiting current density, and are preferably in the overlimiting region. The alternating current applied in addition to the inverse e-pulse preferably is in frequency range of 1 kHz to 1 MHz.

An ion exchange membrane 16 (figure 4) illustrates in use the electrical field lines 17 which generate electro-osmotic instabilities 18 at the membrane surface and in the solution adjacent to the membrane at high current densities (preferably in the overlimiting region). The layer thickness typically is about 20-500 µm, depending on the system, including spacer design and solution velocity, and the applied current and/or voltage.

A device 19 (figure 5) is made of PMMA poly(methyl 2-methylpropenoate) and comprises six separate compartments 20,22,24,26,28,30 with a total solution volume of 2 dm^3 per compartment. Device 19 further comprises a membrane 32 with an effective area of 2.835 cm^2. For the experiments discussed later, membrane 32 was equilibrated with the measuring solution for at least 24 hours. Using a smaller membrane area prevents membrane 32 bulging out and reduces the two current densities applied on the planar auxiliary electrodes, one acting as an anode 34 and the other acting as a cathode 36. The other four membranes 38,40,42,44 were auxiliary ion exchange membranes produced by Mega a.s. (Czech Republic). Membranes comprise cation exchange membranes (CEM) 38,40,44 and anion exchange membrane 42 (AEM). The area of the auxiliary membranes in the cell was 23.8 cm^2.

To enable measurements with the various sodium chloride concentrations with a potentiostat/galvanostat apparatus 46 (Iviumstat, The Netherlands) a fixed current density is applied. Simultaneously apparatus 46 measures the voltage drop over membrane 32. Both compartments 24,26 adjacent to central membrane 32, are provided with a solution 48 with a concentration in a range of 0.017 M to 1 M NaCl (analytical grade, Boom B.V., The Netherlands). For comparison, the standard concentrations we use to represent sea water 50 and/or river water are respectively 0.5M NaCl (sea water) and 0.017M NaCl (river water).

Experiments

Current-voltage curves and chronopotentiometry data were obtained with device 19 presented in Figure 5.

Inverse electrical pulses were applied during RED process and ED process. Figure 6 shows inverse electrical pulses in RED process and shows the measured potential as a function of time during the RED process with only one cation exchange membrane Neosepta CMX, and V being the potential difference over the Haber-Luggin capillaries (mV).

During the process as sea water we used 0.5M NaCl and as river water 1g/l NaCl. The open circuit potential over the membrane was 80mV. The maximum power density in reverse electrodialysis is obtained when the resistance of the external load equals the resistance of the stack (1/2 OCV condition).

In the RED process the first 150 seconds indicate building up the double and diffusion layer on the membrane surface until the plateau is reached. After 150 seconds a first 50mA inverse electrical pulse was applied. By doing this the double layer and the diffusion boundary layer were diminish/eliminated. Application of the pulse obtained energy indicated with the marked area in figure 6. We can observe again the formation of these layers on the membrane surface. Moreover, the second inverse pulse was maintained for two seconds and the third inverse pulse was maintained for three seconds. Also for these pulses the energy obtained by applying the pulses is marked in figure 6.

Figure 7 shows the potential as a function of time (in seconds) under the direct current condition in ED process, with only one cation exchange membrane Neosepta CMX with a surface area of 2.835 cm^2, and V being the potential difference over the Haber-Luggin capillaries (V).

The process was operated between 1 g/l NaCl and 1 M NaCl. The inverse electrical pulse value was set to 100 mA and the time of the pulse was 1 second. We applied constant current (8 mA) to desalinate river water. During the first 18 seconds ions were transported through the membrane, but after this time a significant voltage drop was observed.

This voltage drop indicates that on the membrane surface salt concentration reached zero. To enhance mass transfer by breaking the double layer and the diffusion boundary layer (concentration polarization layers) the inverse electrical pulse according to the invention was applied. The energy obtained by applying the pulses is marked in figure 7.

Surprisingly, the pulses into the region of ion dpeletion induce hydrodynamic instabilities. These instabilities are beneficial for membrane cleaning. Furthermore, if the pulse is provided with a high frequency fluctuation the pulse surprisingly can have a disinfecting effect.

The experimental results show that an inverse electrical pulse surprisingly lowers the overall resistance against mass transport, thereby breaking (removing) the electrical double layer and the diffusion boundary layer on the membrane surface during RED process for certain period of time. By breaking these layers the process efficiency and mass transfer increases. Moreover, inverse electrical pulses applied in combination with high frequency alternating current will be beneficial for breaking scaling/organic fouling and biofouling layers in all electro-membrane processes. Therefore, the inverse electrical pulse, in this case in combination with an alternating/fluctuating current, without requiring operation of the process in a reversed mode removes the layers fast and simultaneously from the membrane surface.

The present invention is by no means limited to the above described embodiments thereof. The rights sought are defined by the following claims, within the scope of which many modifications can be envisaged. For example, the method and device of the present invention can also be used for cleaning of electrodes, possibly in combination with cleaning of membranes. Furthermore, the alternating or fluctuating high frequency current and/or voltage can also be applied over the normal process current and/or voltage, so without an inverse electrical pulse, to achieve a disinfecting effect.

CLAIMS

1. Method for membrane cleaning, comprising the steps of:
   - operating a device for performing an electro-membrane process; and
   - applying inverse electrical pulses to the device.

2. Method according to claim 1, wherein the inverse electrical pulses are applied to electrodes of the device.

3. Method according to claim 1 or 2, wherein the inverse electrical pulse is applied with a frequency between 10^{-5}-10^{-1} Hz, and preferably between $3 \cdot 10^{-4}$ - 10^{-2}Hz.

4. Method according to claim 1, 2 or 3, wherein the applied pulse is provided with a puls width in the range of 10^{-2} to 10^{2} seconds, and preferably between 1 and 10 seconds.

5. Method according to any of the claims 1-4, wherein applying the inverse electrical pulses comprises providing periodic high frequency alternating or fluctuating current and/or voltage.

6. Method according to claim 5, wherein applying periodic high frequency alternating or fluctuating current and/or voltage is performed between applying the inverse electrical pulses.

7. Method according to claim 5 or 6, wherein applying the periodic high frequency alternating or fluctuating current and/or voltage is performed when applying the inverse electrical pulses.

8. Method according to claims 5, 6 or 7, wherein the periodic high frequency alternating or fluctuating current and/or voltage is in the frequency range of 1 kHz to 1 MHz.

9. Device for performing an electro-membrane process, the device comprising:
   - a number of anode and cathode compartments provided with a number of electrodes;
   - a number of cation exchange membranes and a number of anion exchange membranes, placed alternately between the compartments; and
   - function generator means capable of providing inverse electrical pulses according to any of the claims 1-8.

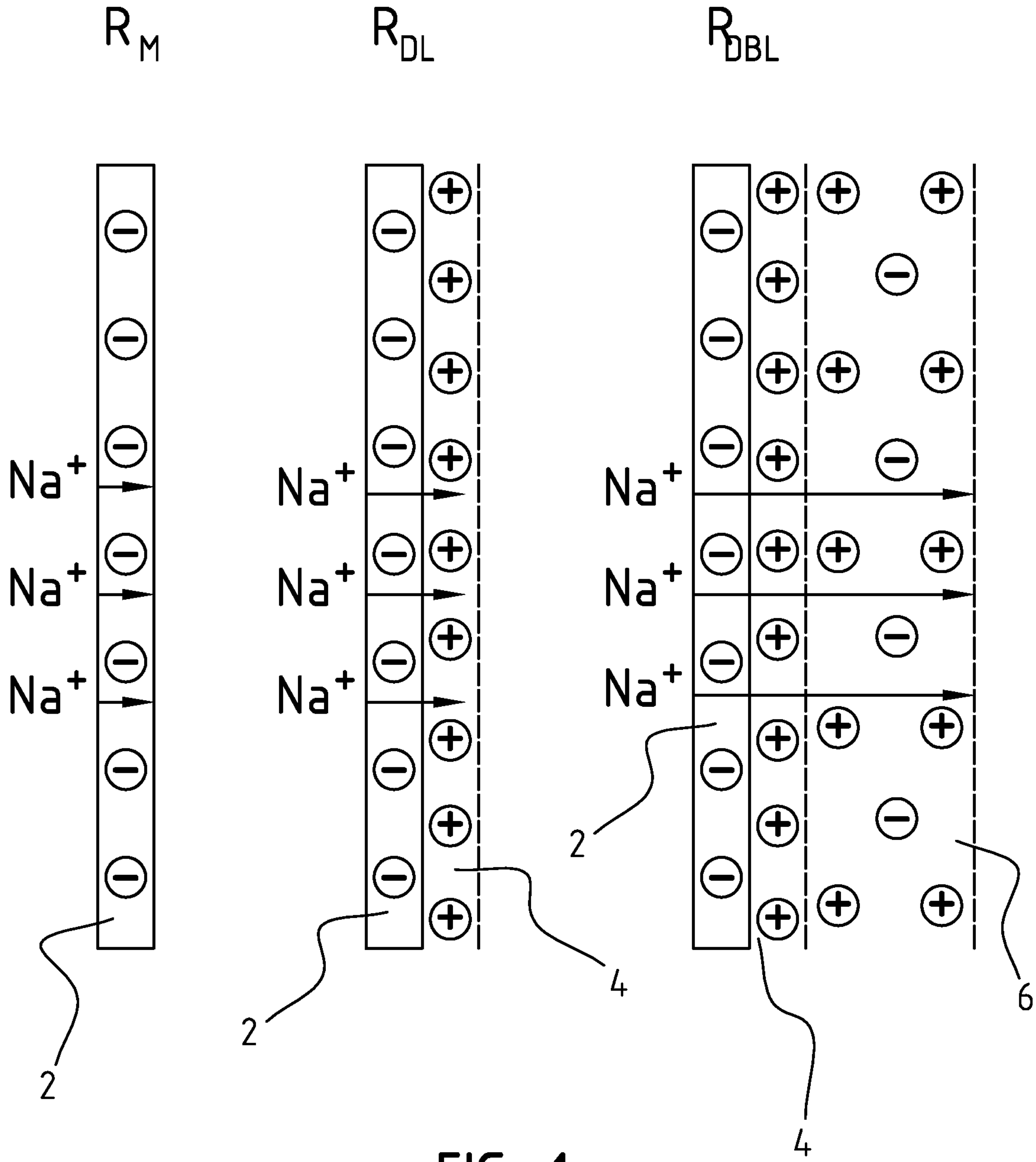


FIG. 1

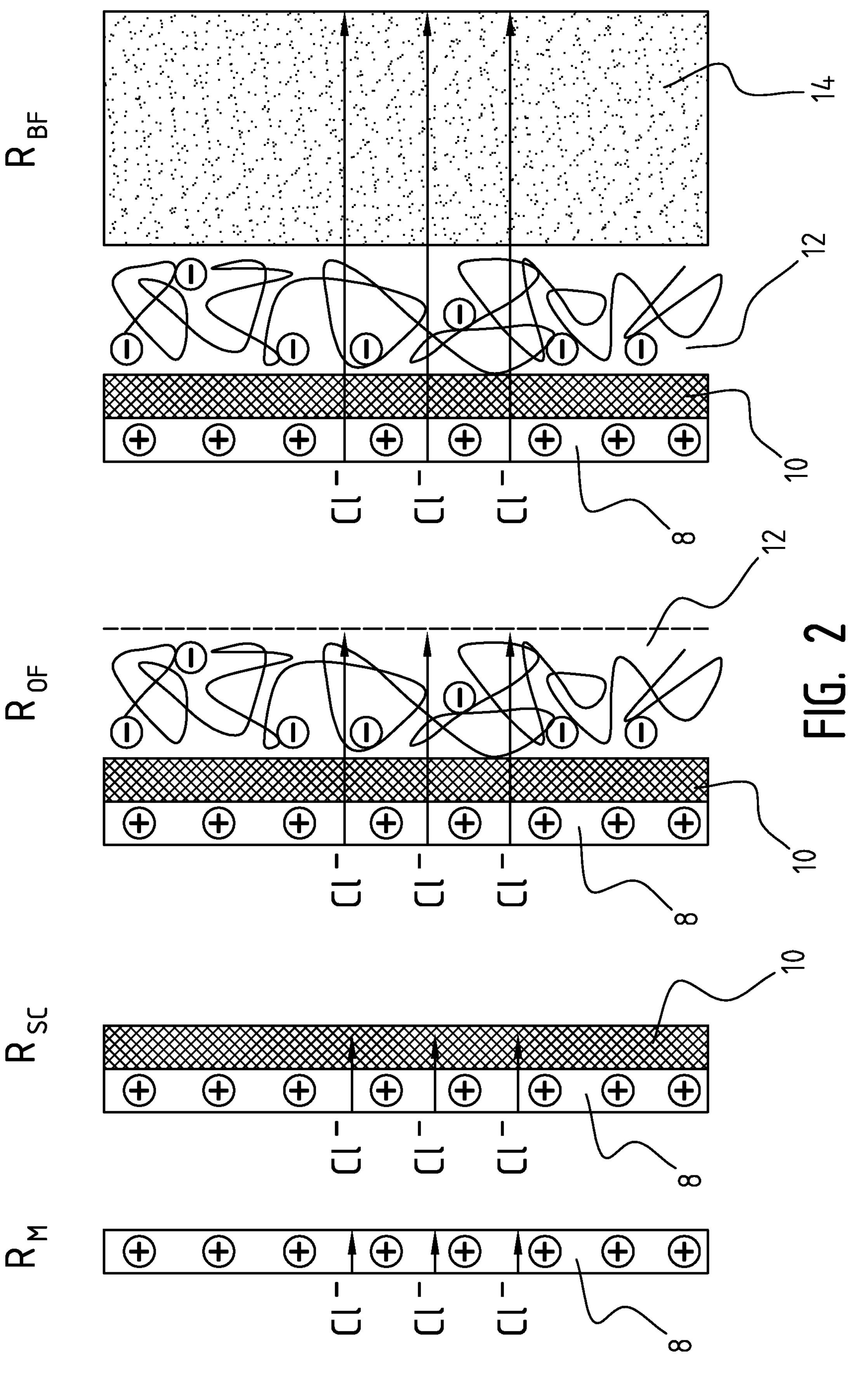


FIG. 2

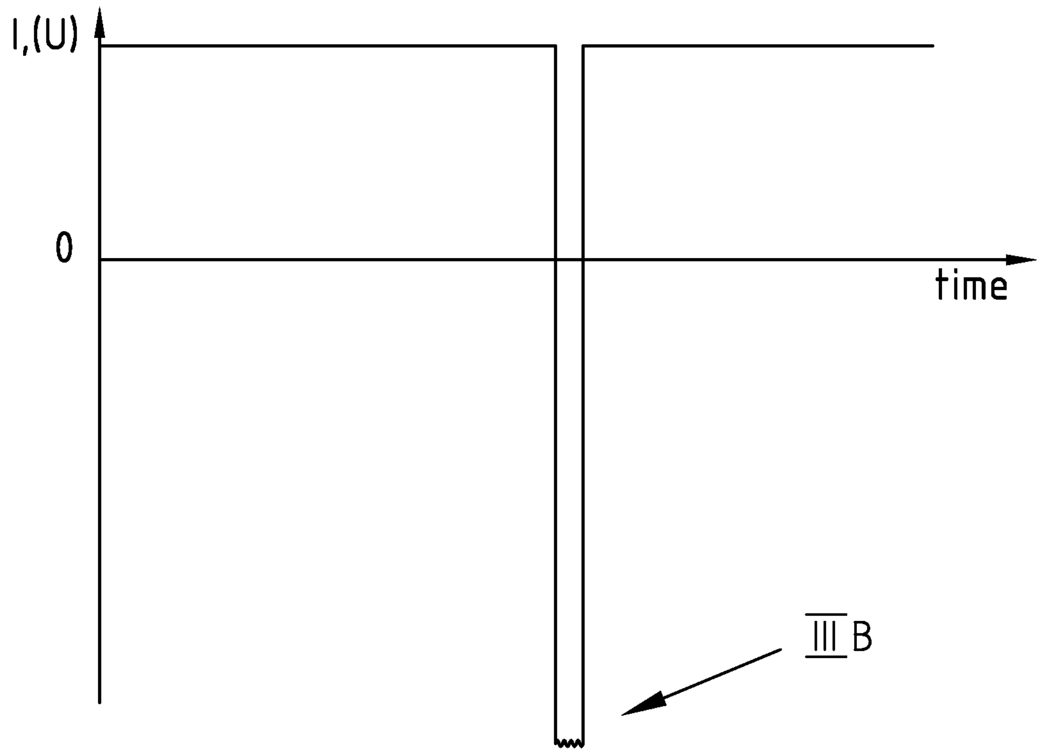


FIG. 3A

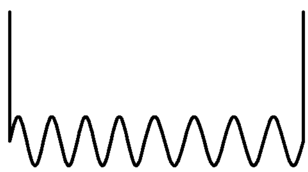


FIG. 3B

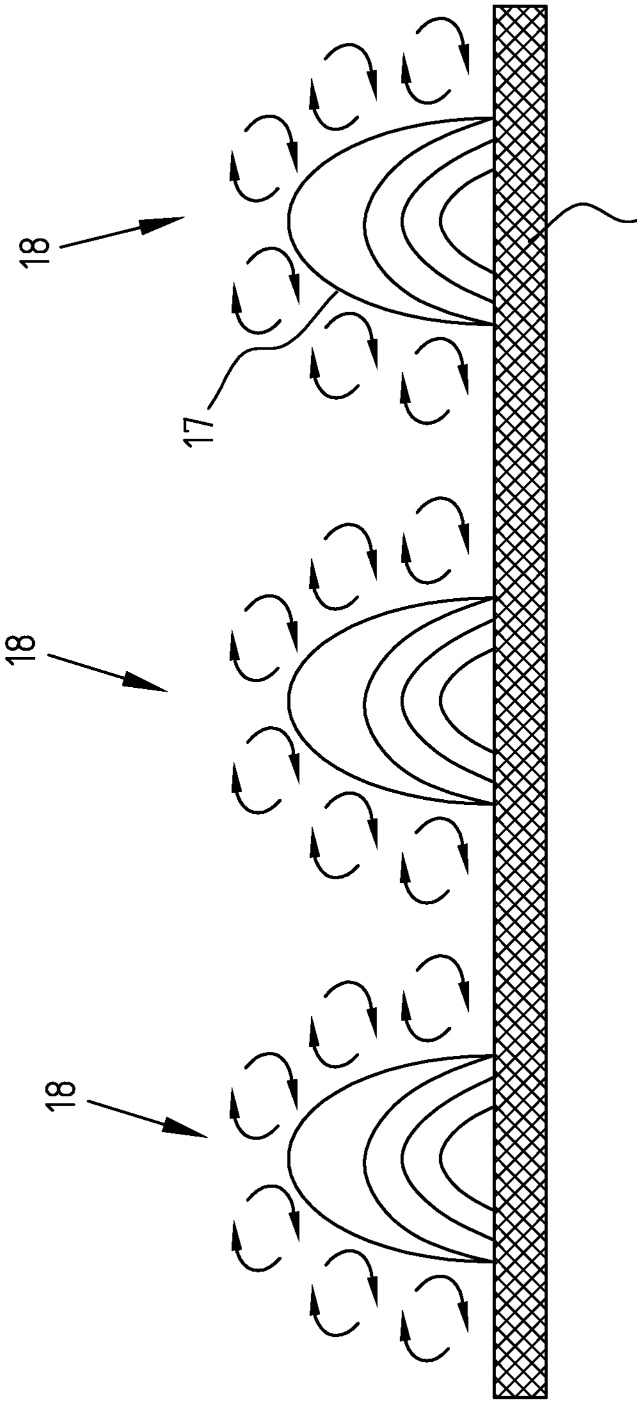
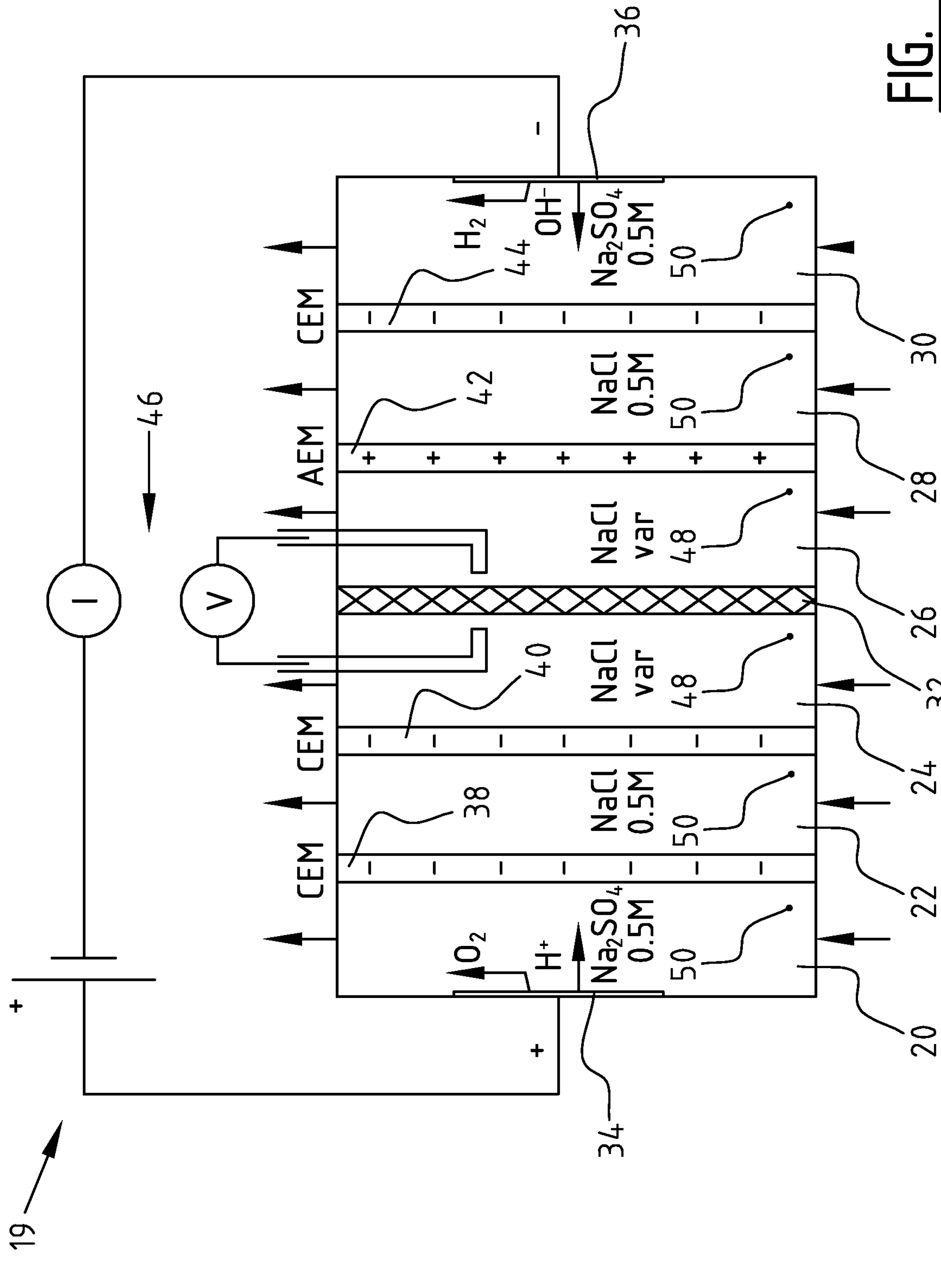


FIG. 4


CEM
CEM
CEM
AEM
CEM
O_2
H^+
Na_2SO_4
0.5M
NaCl
0.5M
NaCl
var
NaCl
var
NaCl
0.5M
Na_2SO_4
0.5M
H_2
OH^-
I
V
+
+
-
19
20
22
24
26
28
30
32
34
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38
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42
44
46
48
50

FIG. 5

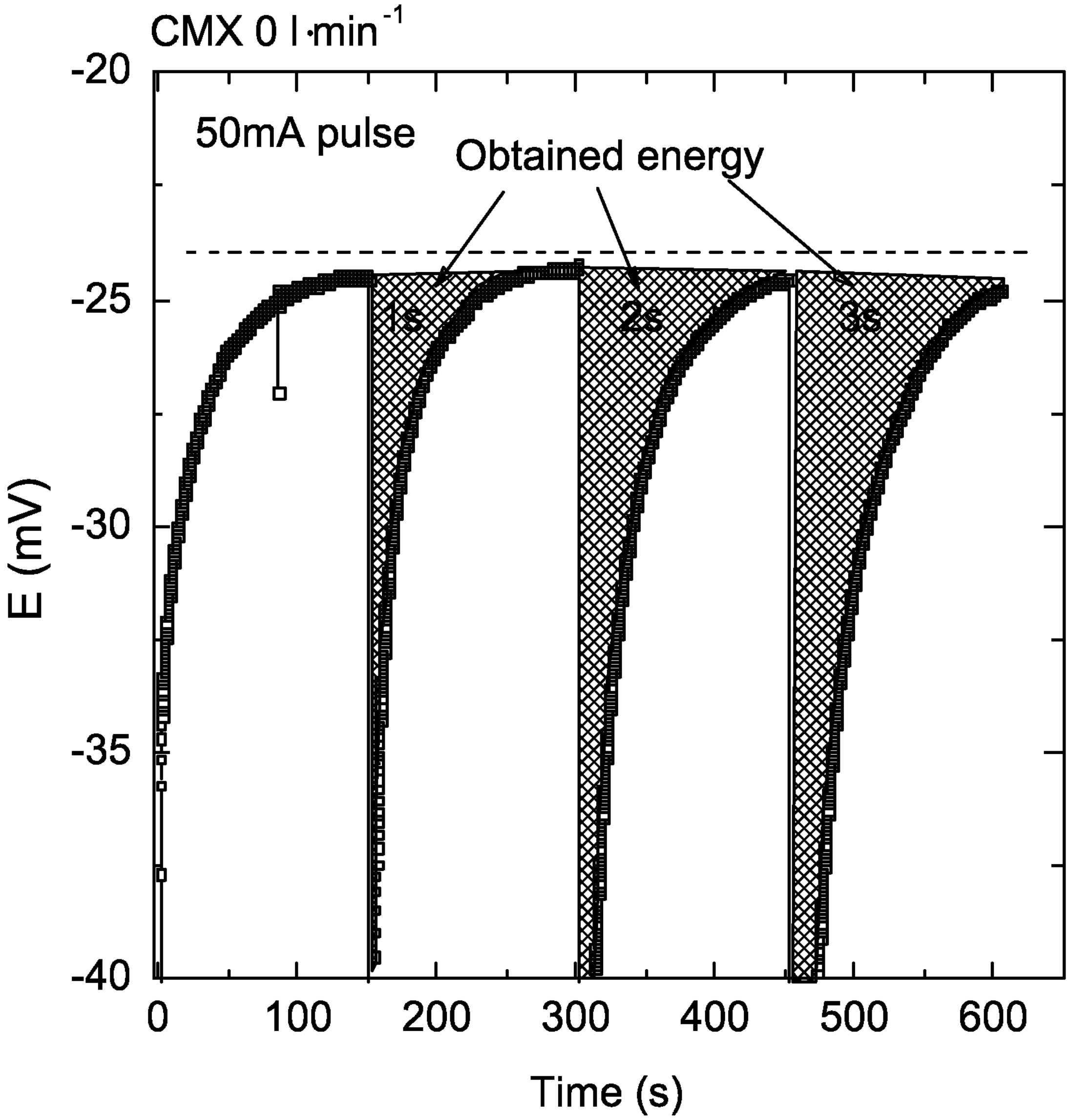
CMX 0 l·min-1
50mA pulse
Obtained energy
1s
2s
3s
E (mV)
Time (s)
-20
-25
-30
-35
-40
0
100
200
300
400
500
600

FIG. 6

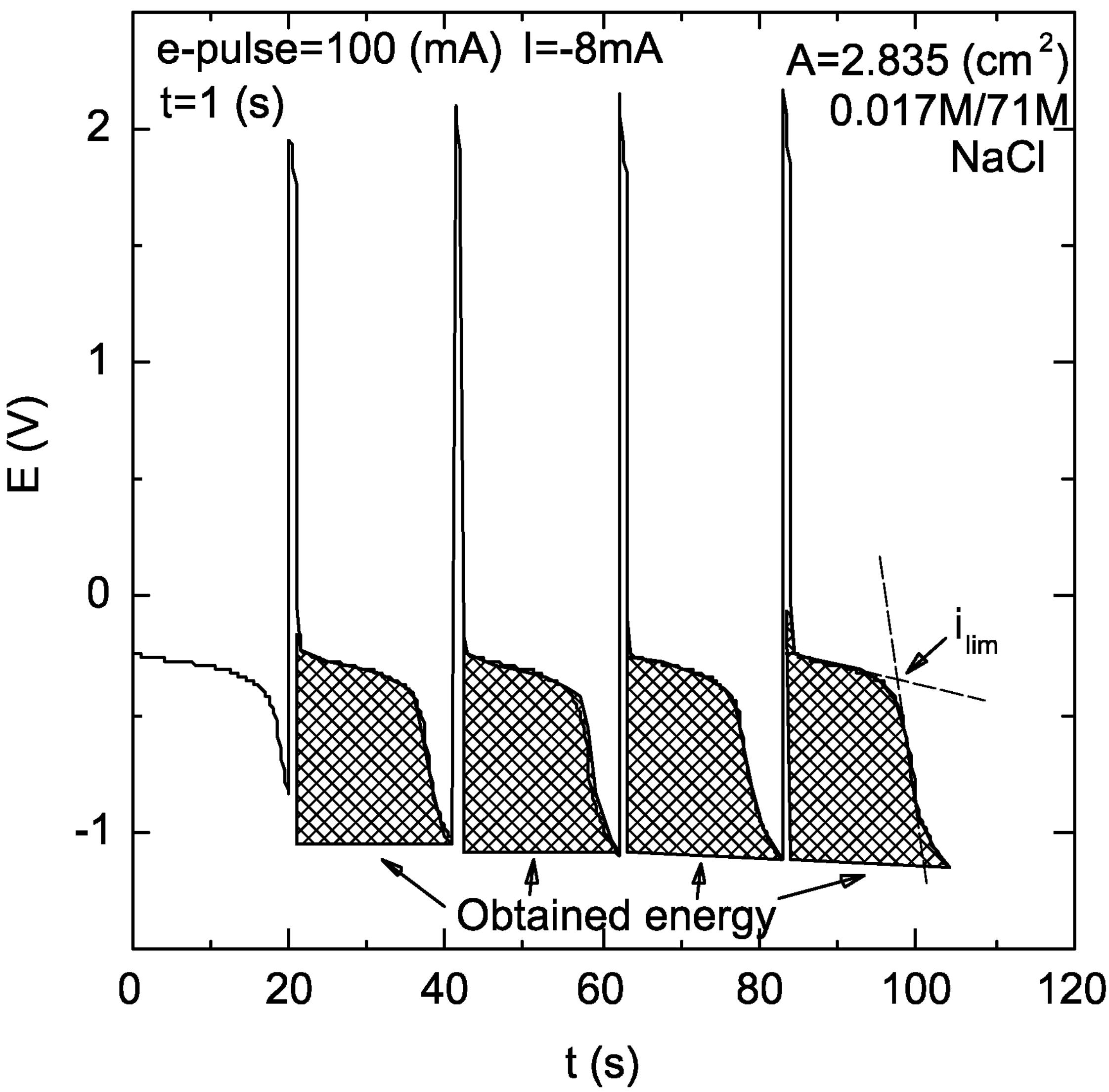
e-pulse=100 (mA) I=-8mA
t=1 (s)
A=2.835 (cm²)
0.017M/71M
NaCl
i_lim
Obtained energy
E (V)
t (s)
2
1
0
-1
0
20
40
60
80
100
120

FIG. 7

INTERNATIONAL SEARCH REPORT

International application No
PCT/NL2010/050335

A. CLASSIFICATION OF SUBJECT MATTER
INV. B01D61/52 B01D61/54 C02F1/469
ADD.

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 practical, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 3 029 196 A (REUVEN MATZ ET AL) 10 April 1962 (1962-04-10) column 2, line 7 - line 39; figures 1,3,4,5,6,7; examples 1,2,4,5	1-9
X,P	WO 2009/082573 A1 (GEN ELECTRIC [US]; DU YU [US]; LIU YUNFENG [CN]; MAO SAIJUN [CN]; YUAN) 2 July 2009 (2009-07-02) the whole document	1-9
X	DE 198 12 005 A1 (LAESER LORENZ [DE]) 30 September 1999 (1999-09-30) column 3, line 32 - column 4, line 12; figure 1; example 4	1-9

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[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

* Special categories of cited documents :

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

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"O" document referring to an oral disclosure, use, exhibition or other means

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"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
7 September 2010	14/09/2010

Name and mailing address of the ISA/	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31–70) 340–2040, Fax: (+31–70) 340–3016	Goers, Bernd

INTERNATIONAL SEARCH REPORT

International application No
PCT/NL2010/050335

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X A	GB 2 290 086 A (ATOMIC ENERGY AUTHORITY UK [GB] ATOMIC ENERGY AUTHORITY UK [GB]; AEA T) 13 December 1995 (1995-12-13) page 5, line 21 - page 6, line 5; figure 1	1-4 9
X	US 2006/231406 A1 (FREYDINA EVGENIYA [US] ET AL) 19 October 2006 (2006-10-19) paragraphs [0071] - [0073]; figures 1,8,9	1,2,9
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
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