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TITLE:

Ion-Exchange Membranes for the Fabrication of Reverse Electrodialysis Device

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membrane, reverse electrodialysis, conductivity, current density, power density

SUMMARY:

We demonstrate the fabrication of a reverse electrodialysis device using a cation-exchange membrane (CEM) and anion-exchange membrane (AEM) for power generation.

ABSTRACT:

Reverse electrodialysis (RED) is an effective way to generate power by mixing two different salt concentrations in water using cation-exchange membranes (CEM) and anion-exchange membranes (AEM). The RED stack is composed of an alternating arrangement of the cation-exchange membrane and anion-exchange membrane. The RED device acts as a potential candidate for fulfilling the universal demand for future energy crises. Here, in this article, we demonstrate a procedure to fabricate a reverse electrodialysis device using laboratory-scale CEM and AEM for power production. The active area of the ion-exchange membrane is 49 cm^2. In this article, we provide a step-by-step procedure for synthesizing the membrane, followed by the stack's assembly and power measurement. The measurement conditions and net power output calculation have also been explained. Furthermore, we describe the fundamental parameters that are taken into consideration for obtaining a reliable outcome. We also provide a theoretical parameter that affects the overall cell performance relating to the membrane and the feed solution. In short, this experiment describes how to assemble and measure RED cells on the same platform. It also contains the working principle and calculation used for estimating the net power output of the RED stack using CEM and AEM membranes.

INTRODUCTION:

Energy harvesting from natural resources is an economical method that is environmentally friendly, thereby making our planet green and clean. Several processes have been proposed until now to extract energy, but reverse electrodialysis (RED) has an enormous potential to overcome the energy crisis issue[1]. Power production from Reverse electrodialysis is a technological breakthrough for the decarbonization of global energy. As the name suggests, RED is a reverse process, where the alternate cell compartment is filled with the high-concentrated salt solution and low-concentrated salt solution[2]. The chemical potential generated by the salt concentration difference across the ion-exchange membranes, collected from the electrodes at the compartment end.

Since the year 2000, many research articles have been published, providing insight into the RED theoretically and experimentally[3,4]. Systematic studies on the operation conditions and reliability studies under stress conditions improved the stack architecture and enhanced the overall cell performance. Several research groups have diverted their attention toward RED's hybrid application, such as RED with desalination process[5], RED with solar power[6], RED with reverse osmosis (RO) process[5], RED with the microbial fuel cell[7], and RED with the radiative cooling process[8]. As mentioned earlier, there is a lot of scope in implementing RED's hybrid application to solve the energy and clean water problem.

Several methods have been adopted to enhance the RED cell's performance and the membrane's ion-exchange capacity. Tailoring the cation-exchange membranes with different types of ions using sulfonic acid group ($-SO_3H$), phosphonic acid group ($-PO_3H_2$), and carboxylic acid group ($-COOH$) is one of the effective ways to alter the physicochemical properties of the membrane. Anion-exchange membranes are tailored with ammonium groups ($-\mathrm{NR}_3^+, -\mathrm{NH}_3^+$)[9]. The high ionic conductivity of AEM and CEM without deteriorating the membrane's mechanical strength is the essential parameter for selecting an appropriate membrane for device application. The robust membrane under stress conditions provides mechanical stability to the membrane and enhances the device's durability. Here, a unique combination of high-performance free-standing sulfonated poly (ether ether ketone) (sPEEK) as cation-exchange membranes with FAA-3 as anion-exchange membranes are used in the RED application. **Figure 1** shows the flow chart of the experimental procedure.

PROTOCOL:

1. Experimental requirement

1.1. Purchase ion-exchange ionomer polymer, E-550 sulfonated-PEEK polymer fiber to prepare CEM and FAA-3 to prepare AEM. Ensure that all ionomer polymers are stored in a clean, dry, and dust-free environment before use.

1.2. Use high purity (>99%) solvents, including N-Methyl-2- pyrrolidone with molecular weight 99.13 g mol^{-1} and N, N-Dimethylacetamide with molecular weight 87.12, for preparing homogeneous ionomer solution. Ensure all analytical grade chemicals and solvents are used for membrane preparation as received without any further purification.

1.3. After the membranes' activation process, immediately immerse all membranes in a 0.5 M

NaCl solution for better performance. After activation of both membranes, drying is not required. Water with resistivity is 18.2 MΩ at room temperature was used throughout the synthesis of the membrane.

1.4. Characterize membrane properties using a dry membrane. The detailed description of the characterization techniques and their physicochemical properties such as ion-exchange capacity, ion-conductivity, thickness, thermal analysis, and surface morphology, are as presented in the literatures[10,11].

1.5. Use a cutter to shape the membrane for CEM and AEM to the RED stack size with an active area of 49 cm^2, as displayed in **Figure 2**.

1.6. For the RED stack fabrication, make an alternate CEM and AEM arrangement, separated by spacer and gasket; a real picture of the working RED stack is presented in **Figure 3a**, and its schematic diagram of each layer is illustrated in **Figure 3b**.

1.6.1. First, place the PMMA plate facing electrode upside; now, place the rubber gasket and spacer on it, then place the CEM. After that, place the silicone gasket with the spacer on the CEM then place the AEM on it. Similarly, add the silicon gasket and spacer on the top of AEM followed by CEM. Now, place the end PMMA plate, rubber gasket, and spacer followed with tightening using screw and nut bolts.

1.7. After assembling the RED stack, check the free flow of the high-concentration (HC), low-concentration (LC), and rinse the solutions one by one. Any crossflow or leakage is required to be eliminated before the measurement.

1.8. Prior to the current and voltage measurement, monitor the flow rate of salt solutions and pressure gauge reading and make sure it gets stabilized. Make sure all the connections are in the exact place before the measurement starts. Avoid touching the RED stack and its connecting tubes while the measurement is running.

NOTE: HC and LC solution flow from their compartments to discard the compartment through a peristaltic pump, pressure gauge, and RED stack, respectively.

1.9. Use galvanostat method for the measurement of current and voltage, the source meter instrument connected to the RED stack through crocodile clips.

[Place Figure 2 here]

[Place Figure 3 here]

2. Ion-exchange membrane preparation

NOTE: The amount of precursor material was optimized for obtaining a membrane with 18 cm diameter and ~50 µm thickness.

2.1. Cation-exchange membrane

2.1.1. Take 5 wt% of sulfonated-PEEK fibers in a 250 mL round bottom flask and dissolve the fibers in Dimethylacetamide (DMAc) as a solvent having molecular weight 87.12 g mol^{-1}. Shake the flask for 10 min so that all ionomer polymers settle down.

2.1.2. Place a magnetic bar in the flask and then keep the mixture in the silicon oil bath, followed by vigorously stirring at 500 rpm for 24 h at 80 °C to obtain a homogenous solution.

2.1.3. Filter the sulfonated-PEEK solution through a 0.45 µm pore size Polytetrafluoroethylene (PTFE) filter.

2.1.4. After that, pour the filtered solution onto a circular glass dish with a diameter of 18 cm. Ensure that all air bubbles are removed using an air blower before placing the Petri dish in the oven.

2.1.5. Place the Petri dish inside an oven for drying out the solution at 90 °C for 24 h, resulting in ~50 µm thick free-standing membrane. Do this for extracting free-standing membrane: To peel off the membrane from the Petri dish, fill the Petri dish with warm distill water (~60 °C) and let it stand for 10 min untouched. The free-standing membrane will automatically come out.

2.1.6. For membrane activation, immerse the prepared free-standing membrane in 1 M sulfuric acid (H_2SO_4) aqueous solution, i.e., 98.08 g, in 1 L of distilled water, and incubate for 2 h at 80 °C.

NOTE: This step will ensure the removal of foreign particles and other chemicals such as solvents that will reduce the possibility of membrane from fouling.

2.1.7. Wash the soaked membrane with 1 L of distilled water for 10 min, at least three times at room temperature.

2.2. Anion-exchange membrane

2.2.1. Dissolve FAA-3 ionomer solution 10 wt.% in N-Methyl-2-pyrrolidone (NMP) solvent.

2.2.2. Keep the solution for stirring at room temperature for 2 h at ~500 rpm.

2.2.3. After that, filter the solution using the mesh with 100 µm pore size.

2.2.4. Pour ~30 mL filtered solution into a circular glass Petri dish with a diameter of 18 cm. Ensure that all air bubbles were removed using an air blower before placing the glass Petri dish in the oven. The drying process takes place at 100 °C for 24 h.

2.2.5. To obtain a free-standing membrane, pour hot distilled water into the glass Petri dish and keep it for at least 10 min. Now peel off the membranes and place in 1 liter of sodium hydroxide (NaOH) solution (concentration 1M and molecular weight 40 g mol^{-1}) for 2 h.

2.2.6. Then, wash the membrane thoroughly with 1 L of distilled water for 10 min, at least three times in ambient condition.

NOTE: All prepared membranes were stored in the 0.5 M NaCl solution overnight before using it in the RED stack. So that the membrane conductivity gets enhanced and can achieve stabilized output performance during the measurement of the RED stack. **Table 1** describes the membrane properties[10–11].

3. Fabrication of reverse electrodialysis

3.1. Assembly of RED stack

3.1.1. Prepare a model solution using 0.6 M NaCl for high concentration (HC) and 0.01 M NaCl for low concentration (LC) compartments[12].

NOTE: Here, river water is considered a low concentration salt solution, and seawater is represented as a high concentration salt solution.

3.1.2. Prepare 5 L of high concentration and low concentration solution in a large container connected with the tubes. Keep the solutions stirring at ambient conditions (room temperature) for at least 2 h before it is used in the RED stack.

3.1.3. Prepare the mixture of 0.05 M of $[Fe(CN)_6]^{-3}$/ $[Fe(CN)_6]^{-4}$ and 0.3 M NaCl in 500 mL water as a rinse solution for RED.

3.1.4. Connect all three solution containers with RED stack using rubber tubes through the peristaltic pump and pressure gauges. Use the tube of size L/S 16 for rinse solution, and use the tube of size L/S 25 for HC and LC solution.

3.1.5. To make a RED stack, take two endplates made-up of polymethyl methacrylate (PMMA). Connect both endplates horizontally face to face with nuts, bolts, and washers using 25 N·m force using a digital wrench driver. The thickness of PMMA endplates 3 cm, and the path of the flow channels was designed in plates for HC, LC, and rinse solution by a driller[2].

3.1.6. Place two mesh electrodes made from metal Titanium (Ti) coated with a mixture of Iridium (Ir) and Ruthenium (Ru) in a 1:1 ratio and place at the end of the PMMA plates. Both end electrodes are connected with the crocodile clip of the source meter.

NOTE: Both PMMA end plates are equipped with mesh electrodes, both electrodes were layered with a square shape spacer, and the PMMA endplate covered with a rubber gasket facing inside.

After that, CEM and AEM are placed alternatively, separated by silicone gasket and spacer, as shown in **Figure 3**.

3.1.7. Install silicon gaskets, polymer spacers, and ion-exchange membranes (CEM and AEM) layer by layer, as presented in the schematic diagram **Figure 4** and **Figure 5**. Ensure the active area of electrodes, both membranes, outer and inner spacer, outer and inner gasket is 7 x 7 = 49 cm^2.

3.1.8. Pass high-concentration and low-concentration solutions from respective compartments by peristaltic pumps, as displayed in the schematic diagram in **Figure 4**.

3.1.9. Circulate the rinse solution in the outer electrode and membrane compartments in recirculation mode using peristaltic pumps. The flow rate used for the rinse solution is 50 mL min^{-1}.

3.1.10. Fixed flow rate is used for analyzing the performance of each membrane. In this experiment, we have used 100 mL min^{-1} through a peristaltic pump.

[Place Figure 4 here]

[Place figure 5 here]

4. Measurement of reverse electrodialysis

4.1. Power calculation

4.1.1. Let the high concentration, low concentration, and rinse solution, run through the stack at least for 5 min. Measure the RED output performance by a source meter, which is connected to both electrodes of the RED stack[13].

4.1.2. Calculate the RED stack's current-voltage characteristics in terms of power density using the galvanostat method.

NOTE: In the galvanostat method, a constant current is applied across electrodes and measures the resulting current. The resulting current is the current generated due to the electrochemical reaction in the stack. The measurement is carried out under 0.05 V static voltage with a fixed sweep current that is 10 mA.

4.1.3. The maximum power density for the RED stack is measured with the help of the following equation 1.

$$P_{max} = \frac{U_{Stack} \cdot I_{Stack}}{A_{mem}} \qquad (1)$$

Here, P_{max} is the maximum power density of the RED stack (Wm^{-2}), U_{stack} is the voltage (V) produced by the membrane in the stack, I_{stack} is the recorded current (A), and A_{mem} is the active area of the membranes (m^2).

REPRESENTATIVE RESULTS:

Net power output

RED cell generally generates electrical energy from the salinity gradient of the salt solution, i.e., ions' movement in the opposite direction through the membrane. To assemble the RED stack correctly, one needs to align all the layers, including electrodes, gaskets, membranes, and spacers in the stack carefully, as demonstrated in the schematic diagram in **Figure 4** and **Figure 5**. If the stack is not perfectly aligned, two problems may arise: (i) HC and LC solution crossflow may occur in the stack and (ii) leakage of the solution in the stack may occur. It is necessary to eliminate both the problems before starting the actual measurement of power output. Other parameters need to be fixed, including the HC and LC solution's flow rate, pumping pressure, and applied voltage, to obtain efficient power output. To estimate the RED stack's net power, one needs to deduct the hydrodynamic power loss from obtained net power[10]. The maximum power output is obtained from the RED stack by multiplying the obtained voltage and current. In contrast, the active area and number of the membrane pairs must be divided to obtain the stack's actual power density, as given by equation 1[14,15]. The total power obtained from the RED stack is subtracted by a hydrodynamic power loss or pumping power loss generated by the pump and given by the following equation 2.

$$P_{net} = P_{max} - P_{loss} \qquad (2)$$

Here, P_{loss} is a hydrodynamic pumping power loss (W m^{-2}) produced in the RED stack by internal loss. P_{max} is the maximum power (W m^{-2}) obtained from the experiment. The highest net power output reported for RED is 1.2 W m^{-2} using river water and seawater by Vermaas[16]. Power loss is represented as a difference of pressure at inlet and outlet of HC and LC solution at the stack and given by pressure drop (ΔP), flow rate (Q), and pump efficiency (η_{pump})[17,18].

$$P_{loss} = \frac{\Delta P_H \, Q_H + \Delta P_L Q_L}{\eta_{pump}} \qquad (3)$$

Here, Q_H and Q_L are the flow rate (mL mim^{-1}) of a high-concentration solution and low concentration solution in mL min^{-1} and ΔP_H and ΔP_L is the pressure drop at the high - concentration side and low concentration compartment in Pa. Here, the measured pressure drop from the pressure gauge for the HC compartment is 11,790 Pa and LC compartment is 11,180 Pa. The calculated pumping power loss (P_{loss}) is 0.038 W m^{-2}.

Theoretical parameter estimation

Basically, A RED system is made up of two different types of ion-exchange membranes, gasket, pump, spacers, and electrode. The pressure drop across the RED stack is estimated theoretically using the Darcy-Weisbach equation[11,19]. In an ideal RED system, a laminar flow of solution in an infinite wide uniform channel is used for calculating the pressure drop.

$$\Delta P = \frac{12\ \mu\cdot L^2}{t_{res}\cdot\frac{1}{4}\cdot d_h^2} \qquad (4)$$

Here, d_h (m) is the channel's hydraulic diameter, whereas the hydraulic diameter for an infinite wide channel is 2h. Other parameters μ is the viscosity of water (Pa·s), t_{res} is the residence time (s), L is the length of the membrane (cm). In RED stack, sPEEK as CEM and FAA-3 as AEM is used, and the distance between both membranes is given by the term b, which is directly proportional to the hydraulic diameter's value in the case of the profiled membrane, and *"h"* is the intermembrane distance (m), is given by equation 5[20].

$$d_h = \frac{4b\cdot h}{2b+2h} \qquad (5)$$

For an infinite wide channel, the value calculated from equation 6 is usually much lower than the finite wide channel's value. The values obtained are low in magnitude, which is due to the non-uniformity of inlet and outlet of feed solutions. The spacer mesh restricts the flow of aqueous salt solutions due to the spacer shadow effect, resulting in an increment in pumping power. Placing the value obtained from the ratio of surface to volume (S_{sp}/V_{sp}) of spacer mesh in the formula, ε is the porosity, one can estimate the thickness of spacer-filled channels from equation 6[21,22].

$$d_h = \frac{4\varepsilon}{\frac{2}{h}+(1-\varepsilon)\cdot S_{sp}/V_{sp}} \qquad (6)$$

The spacer thickness and the other parameters, including open ratio, mesh opening, and wire diameters, are kept constant in all the compartments. Both HC and LC compartments used the same solution (NaCl) with different concentrations. Therefore, it is easy to initialize the parameters, and theoretical pumping loss can be given by equation 7[23].

$$P_{loss} = \frac{\Delta P\cdot Q}{A} = \frac{12\ \mu\cdot L^2\cdot h\cdot\varepsilon}{t_{res}{}^2\cdot\frac{1}{4}\cdot d_h^2} \qquad (7)$$

Where, A is the active membrane area in m^2 and Q feed solution flow rate in $m^3\ s^{-1}$. Here, μ is the viscosity of water measured in Pa·s, L is the *l*ength of membrane given by cm, and t_{res} is a residence time in second.

The performance of the RED stack

The RED stack's output performance was investigated using one cell pair at a fixed flow rate of 100 mL min^{-1}. The feed solution's concentration was also kept fixed for a higher concentration (0.6 M), and a lower concentration (0.01 M) prepared from NaCl salt. It is observed that the maximum power density is 0.69 W m^{-2} at 100 mL min^{-1}, and the net power density is 0.66 W m^{-2} as presented in **Figure 6**. Higher flow rate and high ion-exchange capacity play a significant role in obtaining better cell performance because ions' transport is more active at a higher flow rate. On the other hand, it decreases the diffusion-boundary-layer resistance at the interface. The difference in the salinity gradient of the salt concentration gives rise to the open-circuit voltage, as illustrated in **Figure 6**. This voltage depends on the internal resistance of the RED stack and other parameters. It is noted that as the current density increases, the voltage start decreases whereas, initially the power density of the cell increases obtaining maxima at a certain current density value and then drops down. This decrease in the power density is due to an increase in the stack's internal resistance, as shown in **Figure 6**.

FIGURE LEGENDS:

Figure 1: Procedure chart. The flow chart presents the procedure adopted for the preparation of ion-exchange membrane followed by the process for measurement of reverse electrodialysis.

Figure 2: Size and shape of the prepared membrane, gasket, and spacer for the fabrication of reverse electrodialysis. (**a**) outer silicone gasket, (**b**) outer spacer and inner spacer, (**c**) inner silicone gasket, (**d**) cation-exchange membrane, (**e**) anion-exchange membrane, and (**f**) gasket and membrane assembly.

Figure 3: Reverse electrodialysis stack. (**a**) setup of reverse electrodialysis stack with connecting tubes, and (**b**) schematic illustration of different layers, including PMMA endplates, electrodes, gasket, spacer, CEM, and AEM.

Table 1: Membranes properties. Summary of both cation-exchange and anion-exchange membrane properties.

Figure 4: Schematic representation of the tube connection with reverse electrodialysis stack. Connection of reverse electrodialysis with peristaltic pumps, high-concentration solution container, low-concentration solution container, rinse solution container, and discard solution container. It also shows the spacer's alignment with both an anion exchange membrane (AEM) and cation exchange membrane (CEM).

Figure 5: Schematic diagram of different layers in the reverse electrodialysis setup. (**a**) Cross-section view of a schematic illustration of reverse electrodialysis shows the flow direction of the high-concentration solution, low-concentration solution, and electrode rinse solution. Other components such as electrodes, outer and inner gaskets, outer and inner spacers, cation-exchange membrane, and anion-exchange membrane. (**b**) Front view of the stack, which shows the flow direction of a solution.

Figure 6: Output performance of the reverse electrodialysis device: (**a**) variation of output voltage with varying current, and (**b**) net power density with a varying current density of the RED stack.

DISCUSSION:

The RED's working principle is mainly dominated by the membrane’s physicochemical properties, which is a crucial part of the RED system, as illustrated in **Figure 3**. Here, we describe the fundamental characteristics of the membrane for delivering a high-performance RED system.

Membrane's specific ion permeability makes it pass one type of ions through their polymer nanochannel. As the name suggests, CEM can pass cation from one side to another and restricts anion, whereas AEM can pass anion and restricts cation. As shown in **Figure 2**, all membranes were shaped into a RED stack size containing inlet and outlet passage for flow solution. The amount of ion exchanged through the membrane is directly proportional to the membrane's conductivity and, therefore, the power output of the stack[24]. The movement of ions in the ion-exchange membrane works on the Donnan exclusion principle[25]. The charge group attached with the polymer backbone repels the same charge present in the solution. Thus, higher the charge density greater will be the repulsion, which usually depends on the perm selectivity. Generally, in RED cells, ions' movement takes place through the membrane from higher concentration to lower concentration of the solution. This ion transport from one compartment to another through the membrane gives an open circuit voltage and current values, which is used to calculate the net power output of the cell[26].

The RED stack's performance mainly depends on the ion exchange capacity and swelling density of CEM- and AEM-based membranes[27]. It is observed that the higher the ion-exchange capacity of the CEM and AEM, the better is the conductivity. However, the higher ion-exchange capacity of the membrane leads to high swelling, easily deteriorating the membrane's mechanical strength. Thus, it is essential to optimize swelling density and the membranes' conductivity for better and reliable cell performance. On the other hand, it is also crucial to optimize the stack resistance with the function of the feed solution's flow in both the compartments. As the flow rate increases, the stack resistance decreases, and the output cell performance increases. Theoretically, RED stack resistance is given by equation 8.

$$R_{stack} = \frac{N}{A_{mem}}\left(R_A + R_C + \frac{d_c}{k_c} + \frac{d_d}{k_d}\right) + R_e \qquad (8)$$

N is the number of cell pairs (alternate arrangement of anion- and cation-exchange membranes), A is the effective area of both the membranes (m^2), R_A is the anion exchange membrane resistance (Ω m^2), R_C is the cation exchange membrane resistance (Ω m^2), d_c is the thickness of the compartment with the concentrated solution (m), k_c is its ionic conductivity (S m^{-1}), d_d is the thickness of the compartment with the diluted solution (m), k_d is its ionic conductivity (S m^{-1}), and R_e is the electrode resistance (Ω). Reducing the stack resistance is an essential factor for enhancing the net output power, but other factors also influence the cell performance[28], which also need to be considered. The spacer shadow effect, the flow of feed solution, compartment width, and concentration of feed solution, the schematic illustration of the RED cell are presented in **Figure 5**.

In RED cells, the membrane acted as a limiting factor and required a stable high conducting membrane. Apart from that, both CEM and AEM are required to have comparable ion-conducting properties so that the cell can produce an efficient and optimized power output. Degradation of ion-exchange capacity and salt accumulation also need to be taken into account for reliable RED performance. Novel membrane material and state-of-the-art device architecture may further improve cell performance in the coming future and will pave a path for future research direction.

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DISCLOSURES:

The authors declare no conflicts of interest.

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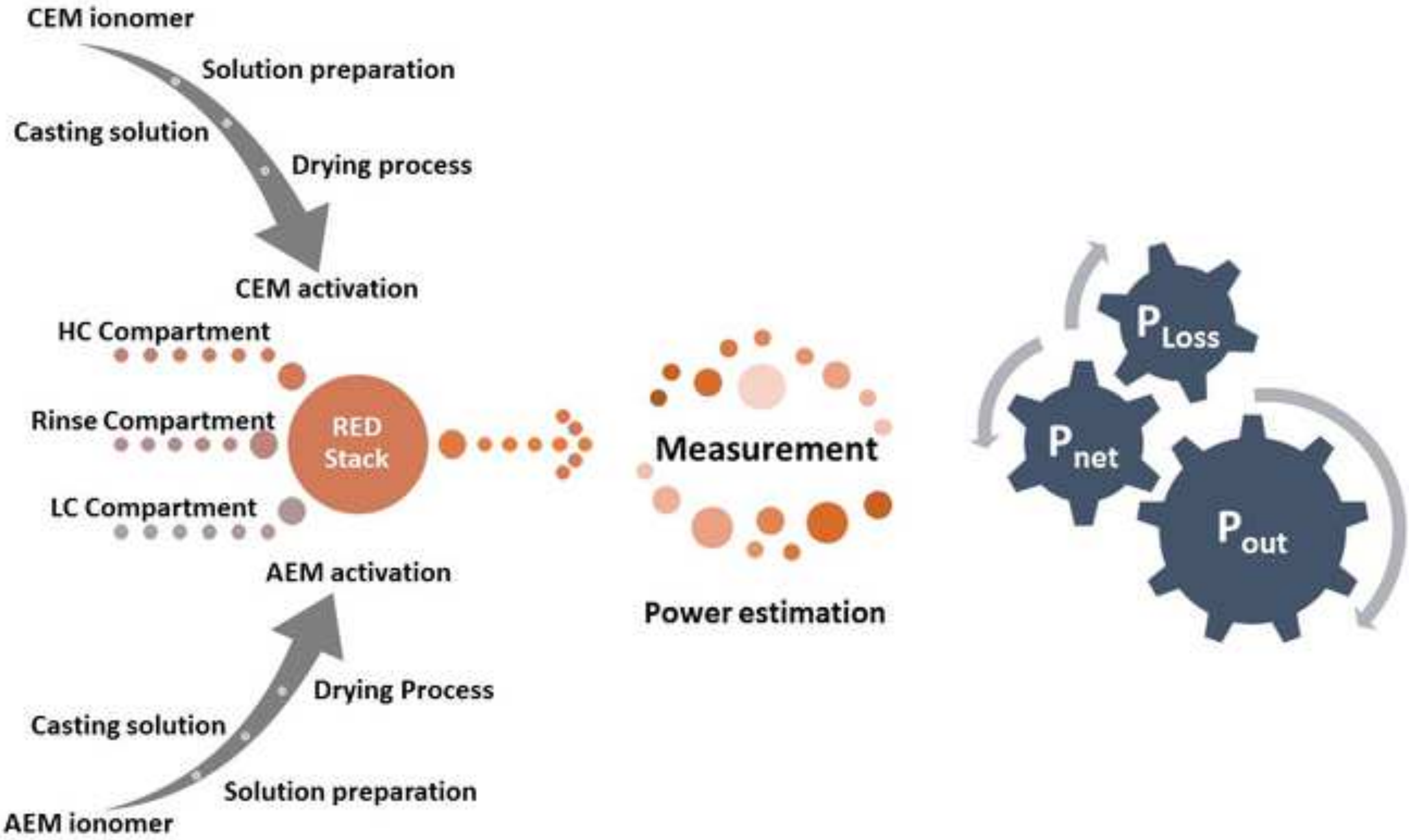

CEM ionomer
Solution preparation
Casting solution
Drying process
CEM activation
HC Compartment
Rinse Compartment
LC Compartment
RED Stack
AEM activation
Drying Process
Casting solution
Solution preparation
AEM ionomer
Measurement
Power estimation
P_{Loss}
P_{net}
P_{out}

Figure 2

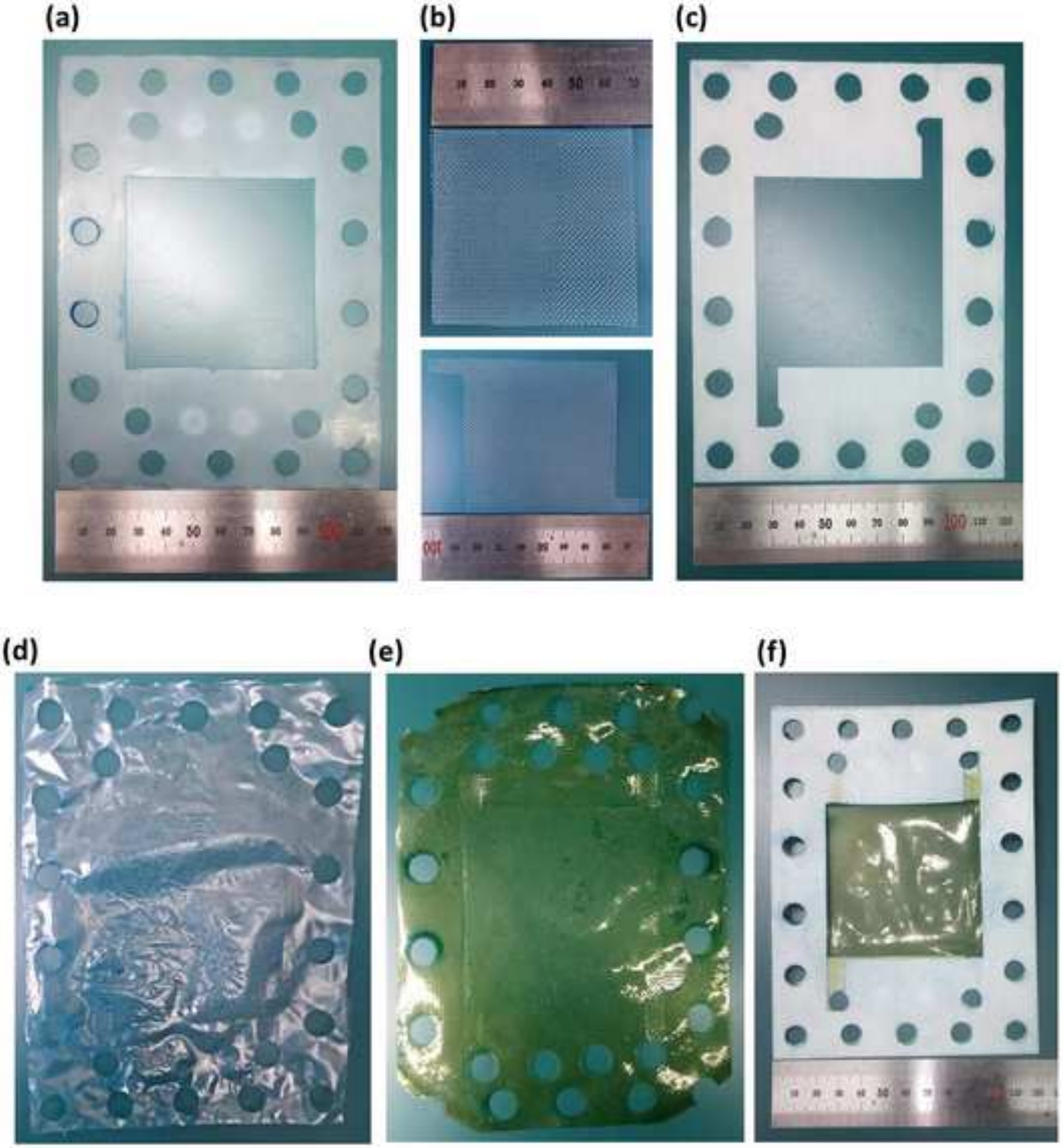
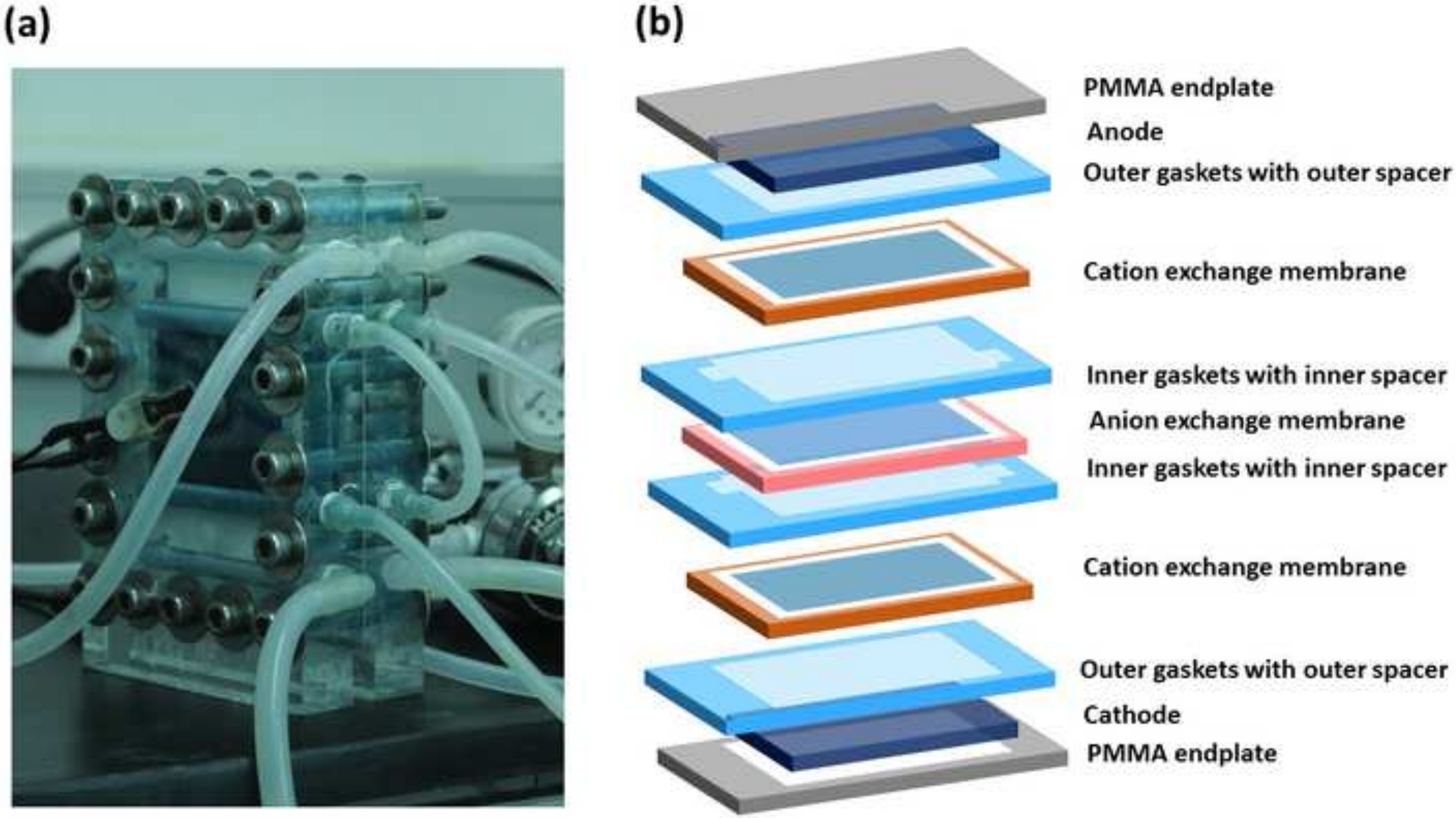


Figure 3


(a)
(b)
PMMA endplate
Anode
Outer gaskets with outer spacer
Cation exchange membrane
Inner gaskets with inner spacer
Anion exchange membrane
Inner gaskets with inner spacer
Cation exchange membrane
Outer gaskets with outer spacer
Cathode
PMMA endplate

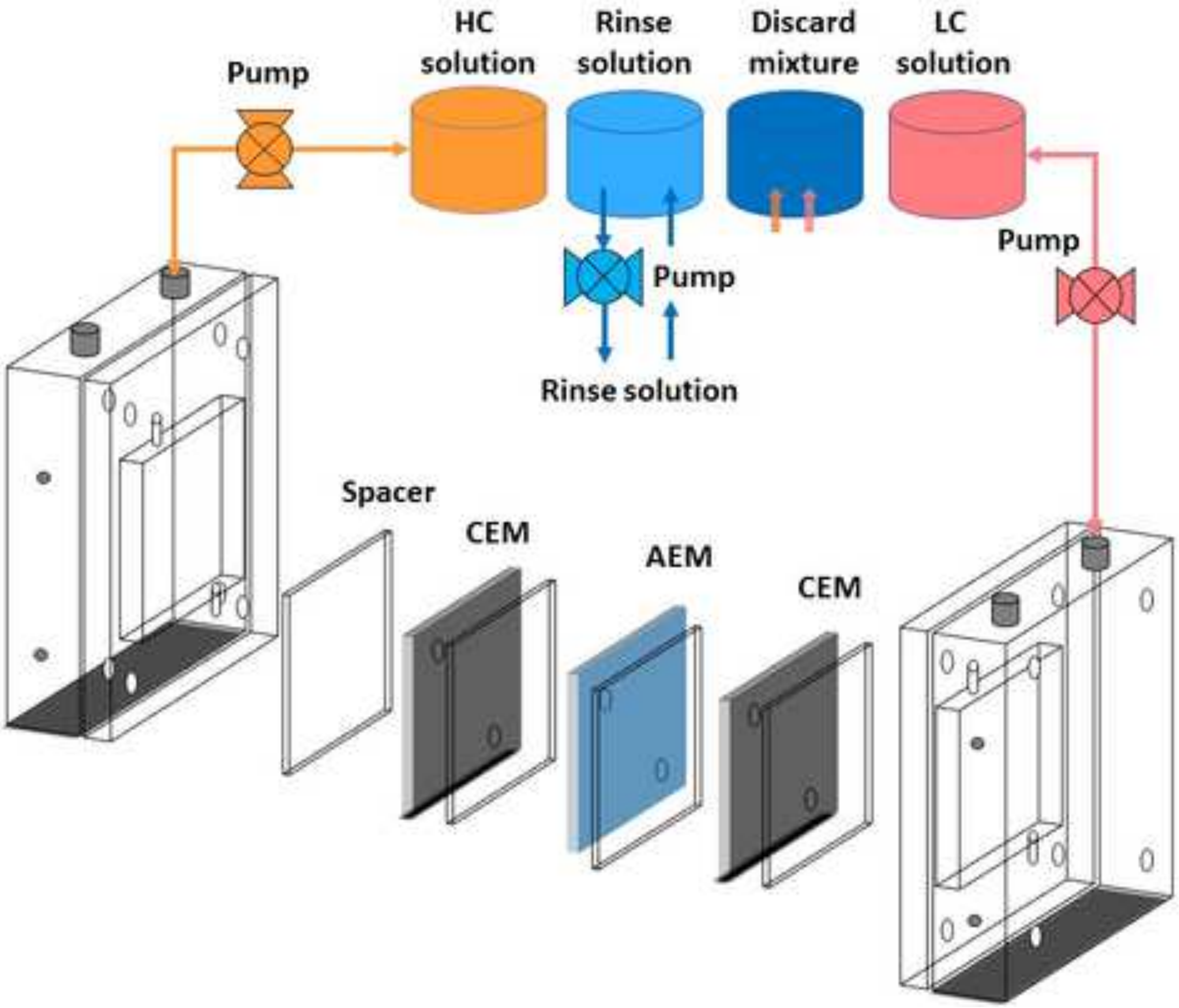
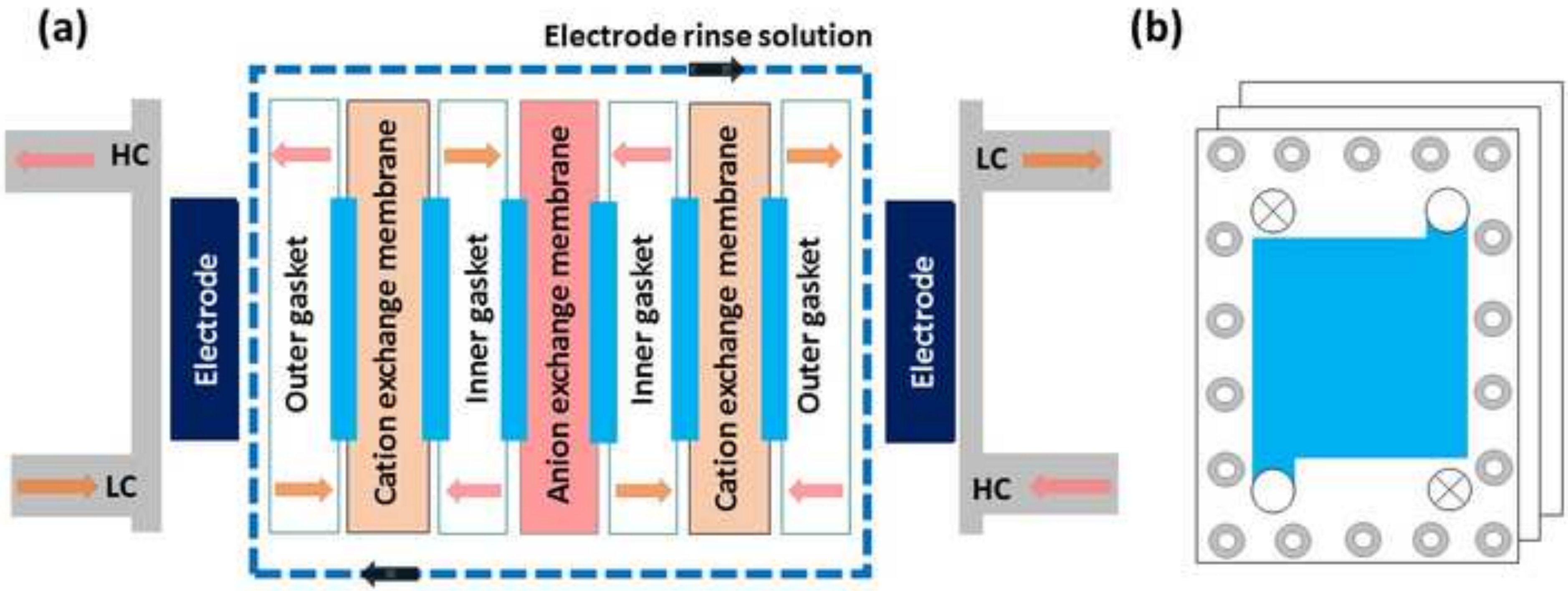

Pump
HC solution
Rinse solution
Discard mixture
LC solution
Pump
Pump
Rinse solution
Spacer
CEM
AEM
CEM

Figure 5



Cross-section view

Front view

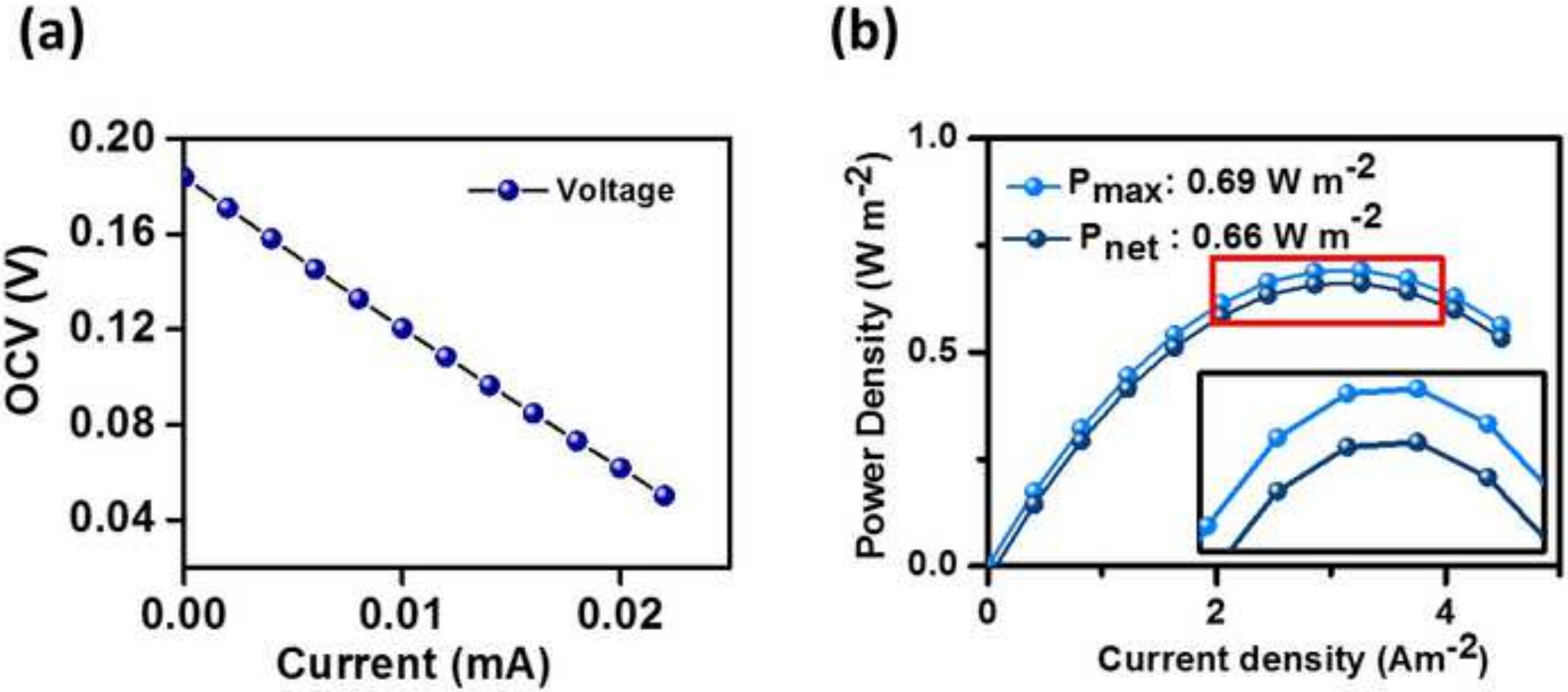

(a)
0.20
0.16
0.12
0.08
0.04
OCV (V)
Voltage
0.00
0.01
0.02
Current (mA)
(b)
1.0
0.5
0.0
Power Density (W m-2)
Pmax: 0.69 W m-2
Pnet : 0.66 W m-2
0
2
4
Current density (Am-2)

Table 1 Membranes properties: Summary of both cation-exchange and anion-excha

Specification	**Unit**	**CEM**
Swelling degree	%	5±1
Charge density or Ion exchange capacity	meq/g	1.8
Mechanical properties (Tensile strength)	MPa	>40
Elongation to Break	%	~42
Young Modulus (MPa)		1500±100
Conductivity at room temperature	S/cm	~0.03
Permselectivity	%	98-99
Thickness	µm	50±2
Solvent	-	Dimethylacetamide (DMAc)

ange membrane properties.

AEM
1±0.5
~1.6
40-50
30-50
1000-1500
~0.025
94-96
50±3
N-methyl-2-pyrrolidone (NMP)

Click here to access/download;Table of Materials;JoVE_Table_of_Materials.xlsx

Name of Material/Equipment	Company	Catalog Number	Comments/Description
AEM based membrane	Fumion	P1810-194	Ionomer
CEM based membrane	Fumion	E550	Ionomer
Digital torque wrench	Torqueworld	WP2-030-09000251	wrench
Labview software	Natiaonal Instrument	-	Software
Laptop	LG	-	PC
Magnetic stirrer	Lab Companion	-	MS-17BB
N, N-Dimethylacetamide	Sigma aldrich	271012	Chemical
N-Methyl-2- pyrrolidone	Daejung	872-50-4	Chemical
Peristaltic pump	EMS tech Inc	-	EMP 2000W
Potassium hexacyanoferrate(II) trihydrate	Sigma aldrich	P3289	Chemical
Potassium hexacyanoferrate(III)	Sigma aldrich	244023	Chemical
Pressure Gauge	Swagelok	-	Guage
Reverse electrodialysis setup	fabricated in lab	-	Device
RO system pure water	KOTITI	-	Water
Rotary evaporator	Hitachi	YEFO-KTPM	Induction motor
Sodium Chloride	Sigma aldrich	S9888	Chemical
Sodium Hydroxide	Merk	1310-73-2	Chemical
Source meter	Keithley	-	2410
Spacer	Nitex, SEFAR	06-250/34	Spacer
Sulfuric acid	Daejung	7664-93-9	Chemical
Tube	Masterflex tube	96410-25	Rubber tube

Dear All

We thank all the reviewers and editor of the journal for the detailed, constructive feedback. Below, we have responded line-by-line to each question. We believe this process has helped to significantly improve the quality of our manuscript.

Thank You

Reviewers' comments:

Reviewer #1:

Manuscript Summary:

This manuscript presents a relevant and interesting research topic concerning green energy production -- namely, membrane synthesis and engineering processes. The manuscript describes the preparation process for both the ion-exchange membranes and the reverse electrodialysis cell stack, and then estimates the overall system performance. The discussion does a good job of relating the membrane properties to the characteristics of the reverse electrodialysis stack, presenting a final calculation of the theoretical resistance and performance of the latter.

Major Concerns:

The basic properties of the membranes were added to the final manuscript incorporating essential and important information to the work presented.

Minor Concerns:

The minor concerns were handled, thus improving the manuscript quality. An extended and detailed protocol was added to the manuscript allowing a better understanding of the membrane preparation, assembly of the RED stack, and the power calculation.

Reply: We want to thank reviewer 1 for their positive comment, spending time in studying our manuscript, and providing insightful comments with constructive feedback.

Reviewer #2:

Manuscript Summary:

The revision R1 has introduced some marginal imprvement, but still is not very useful for potential readers. Much more detailed experimental details and calculations on reverse electrodialysis have been published already (including videos on yotube).

Major Concerns:

My main point raised for the original manuscript has not been answered in R1. What I asked was about presenting all calculations / mass balances... Otherwise, all the equations presented in the results section (except for that for gross power) should be presented in the introduction or even better addresing the readers to appropriate reference(s).

It appears that the only calculation that the authors have done in their study is to multiply the voltage by current and then divide this by the total exposed membrane area...

Even this is not presented clearly enough, since in order to obtain the power density one normally uses a range of external loads in order to obtain the power versus current density curves (with OCV with OCV with no load).

It is not clear what external loads (if any) were used to produce, in fact, the only experimental graph (Fig. 5a), which can be obtained in a very short time (minutes range)...

What about Ploss? How was it calculated? What is the net power obtained? What are the respective resistances, etc.?

Reply: This is an interesting question, and it could be a subject for further study because when we started writing this manuscript our main focus is to make this article for the educational purpose for the student who just jumpstarts their research.

We agree with the referee that there are already reported articles on RED cell preparation and videos are reported on other platforms. But, here our main objective is to provide basic information about ion-conducting membrane preparation and its application in reverse electrodialysis at a single platform. So that, students around the globe can attract to this field and can understand the fundamentals of how membrane chemistry affects RED cell performance. We are specifically motivated towards the basic research of ion-conducting membrane therefore, we restrict ourselves going towards the use of in-depth study on any of the topic presented in this particular article (including, membranes chemistry, and device physics, etc.) and consider as out of scope for this manuscript.

In this article, we emphasize how we can enhance the understanding of membrane synthesis and its device fabrication at the initial level (beginners). We believe that the basic research demonstrated or carried out in this manuscript is focused on the basic principle and deals with acquiring knowledge that has universal applicability. We are confident that our revised manuscript expands scientific knowledge in understanding the RED systems for educational purposes and there is no other financial or competent interest. This type of research doesn't show enormous potential at the cutting-edge but enhancing our awareness of science to the beginner at school and university level. We think that our manuscript will be used to help us in understanding a real-world problem or will research further for our general intelligence. This manuscript not only bridging the theoretical and experimental aspects of the membranes and their application but also contributes to paving a path for developing a basic understanding of this real-world problem.

At last, we hope the reviewer will consider our description for the revised manuscript.

Some changes have been made in the manuscript, which is as follows:

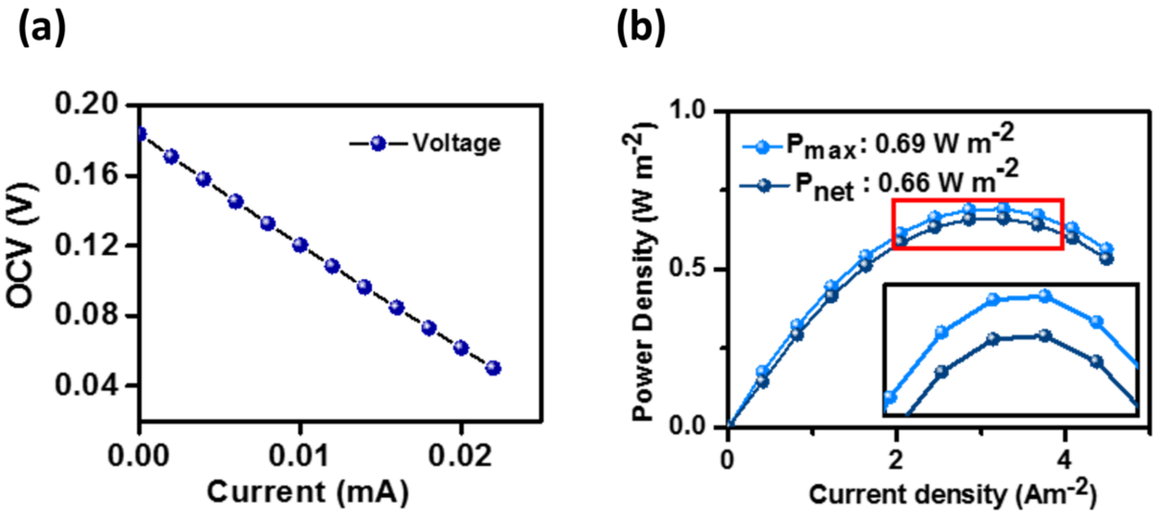


Figure 6 Output performance of the revers electrodialysis device: (a) variation of output voltage with varying current, and (b) net power density with a varying current density of the RED stack.

Regarding applied load: All measurement is carried out under 0.05 V static voltage with fixed sweep current which is 10 mA.

Changes made in the manuscript:

Note: In the galvanostat method, a constant current is applied across electrodes and measure the resulting current. The resulting current is the current generated due to the electrochemical reaction in the stack. The measurement is carried out under 0.05 V static voltage with a fixed sweep current which is 10 mA.

Regarding Power loss calculation: The hydrodynamic power loss or pumping power loss generated by the pump is subtracted from the maximum power and given by following equation 2.

$$P_{net} = P_{max} - P_{loss} \tag{2}$$

Here, P_{loss} is a hydrodynamic pumping power loss (W m^{-2}) produced in the RED stack by internal loss. P_{max} is the maximum power (W m^{-2}) obtained from the experiment.

Power loss is represented as a difference of pressure at inlet and outlet of HC and LC solution at the stack and given by pressure drop (ΔP), flow rate (Q), and pump efficiency (η_{pump}).

$$P_{loss} = \frac{\Delta P_H\, Q_H + \Delta P_L Q_L}{\eta_{pump}} \tag{3}$$

Here, Q_H and Q_L are the flow rate (mL mim^{-1}) of a high-concentration solution and low concentration solution in mL min^{-1} and ΔP_H and ΔP_L is the pressure drop at the high - concentration side and low concentration compartment in Pa. Here, the measured pressure drop from the pressure gauge for the HC compartment is 11790 Pa and LC compartment is 11180 Pa. The calculated pumping power loss is 0.038 W m^{-2}.

The bulk resistance of both CEM and AEM are calculated from the EIS workstation and the resistance of the solution HC and LC are estimated from the conductivity meter. However, The membrane resistance in the stack predominately affects the overall performance of the cell therefore we have provided the calculated conductivity of the respective membrane in table 1.

Minor Concerns:

A number of grammatical problems and wrong dimensions can still be found in R1.

Also there is wrong numbering of sub-sections... Please, check and correct them accordingly.

Reply: We apologies for any inconvenience experienced. Now changes have been made in the main manuscript. We have gone through each paragraph, line by line, all modified portion is marked with yellow color. (Please see the revised main manuscript and other supplementary files)

Changes made in the main manuscript:

In "1 Experimental requirement:" sections the last point "1.8" is replaced by "1.9"

In "4. Measurement of Reverse electrodialysis:" section the last point "5.1.3" is replaced by "4.1.3".

Reviewer #3:

Manuscript Summary:

The authors have well-addressed all the necessary comments.

Reply: We thank reviewer 3 for carefully reading our work, and recognizing its promise.