Fabrication of a new type of polyvinyl alcohol/sulfosuccinic acid/glutaric anhydride cation exchange membrane for desalination using membrane capacitive deionization

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Abstract

Nowadays, the lack of clean water is becoming a serious problem, due to the increasing demand for clean water, along with global climate change. Desalination of sea water or brackish water has been considered as a practicable method to supply clean water. Among desalination technologies, membrane capacitive deionization has emerged for the last 15 years as an alternative method and received a lot of research attention since then. In this study, low-cost and ecofriendly polyvinyl alcohol/sulfosuccinic acid/glutaric anhydride composite membranes were fabricated through the crosslinking reaction of sulfosuccinic acid and glutaric anhydride with polyvinyl alcohol. The effect of sulfosuccinic acid and glutaric anhydride content was investigated. The presence of SSA in the cation exchange membrane, which was proven by FT-IR, helps improve its water uptake (98.5 % compared to 38.2 %) as well as cation exchange capacity (1.993 mM/g compared to 0.156 mM/g). The polyvinyl alcohol/sulfosuccinic acid/glutaric anhydride composite solution was coated on the electrode, and the membrane's ability to desalinate water was assessed. The membrane-coated electrode exhibited enhanced salt adsorption capacity and shorter desalination time (13.4 minutes compared to 85.3 minutes) in comparison with the uncoated commercial electrode, indicating that the polyvinyl alcohol/sulfosuccinic acid/glutaric anhydride membrane is a suitable candidate to be utilized in the membrane capacitive deionization systems.

1 Introduction

The lack of clean water is becoming more and more frequently nowadays, due to the increasing demand for clean water, along with serious global climate change [1]. Recently, numerous technologies supplying clean water have been researched all over the world, most of which are desalination technologies because of the sheer abundance of sea water as well as underground brackish water [2-4], specifically thermal distillation and reverse osmosis. Capacitive deionization (CDI) is also emerging as a prominent process, with a lot of advantages such as high efficiency, low energy consumption, simple operation, and reduced secondary pollution, especially with the brackish water with low salt content [5,6]. Further modification of the convention CDI, the membrane capacitive deionization (MCDI), comprises cation exchange membrane (CEM)

or anion exchange membrane (AEM) attached to the electrode surface [6, 7], which can help inhibit the movement of the ions by only allowing the similarly charged ions to pass, helping to keep the ions inside the electrode during the desalination process, and help prevent the movement of the co-ions during the regeneration process [8]. Compared to the traditional CDI, the MCDI exhibits greater salt removal efficiency [9-12], higher current efficiency [11-14], faster salt removal rate [10], lower energy consumption [13,15], and better energy recovery [16,17].

In MCDI technology, ion exchange membrane (IEM) is among the most important parts that determine the salt removal efficiency [18]. There are two factors affecting the properties of the membranes, namely the matrix and the charged functional groups of the membrane. The membrane matrix can modify water uptake, mechanical resistance, as well as thermal and chemical stability. The functional groups in the membrane can influence the ion adsorption capacity and ion selectivity [19].

Polyvinyl alcohol (PVA) is now widely utilized in membrane fabrication thanks to its strong film forming ability and high hydrophilicity. Furthermore, the thermal and mechanical properties of PVA can also be enhanced by thermal or chemical modification, by radiation, or by cross-linking [20,21]. However, as there is no charge in structure of pure PVA, it is necessary to attach the charged functional groups such as hydroxyl, amine, carboxylate, sulfonate, or quaternary ammonium to ameliorate the ion conductivity. All of the polyfunctional compounds that can react with hydroxyl groups can be employed in the cross-linking reaction with PVA to create polymers with network structure [22,23]. Furthermore, via cross-linking, the water solubility of PVA composites can be improved, from which their stability in water can be enhanced. PVA-based composites with various cross-linking agents such as sulfonate ($-SO_3H$) or carboxylate ($-COOH$) exhibit upgraded cation conductivity and selectivity. PVA/SSA cation exchange membrane helps improve the salt removal efficiency in MCDI [12]. There are many organic compounds that can take part in the cross-linking reaction with PVA, namely glutaraldehyde [22,24], formaldehyde [25], citric acid [26], acrylic acid [27],

sulfosuccinic acid [28], maleic acid [29,30], tartaric acid, or malic acid [31]. Besides, the membrane surface can also be modified with anionic functional groups such as sulfonate (SO_3^-) [32] or phosphate (PO_3^-).

In this research, PVA/SSA/GA cation exchange membrane was created from the cross-linking reaction of PVA, SSA and GA, with GA acting as a cross-linking agent, and SSA acting mainly as a functional group donor but also as a cross-linking agent. The effect of GA percentage in the composite membrane on its mechanical and chemical characteristics as well as its electrochemical properties were thoroughly investigated.

2 Materials and methods

2.1 Materials and chemicals.

Polyvinyl alcohol [98 %, M = (146.000-186.000) $g \cdot mol^{-1}$] and glutaric anhydride (95 %) was acquired from Acros, Belgium. Commercial activated carbon was provided by Trabaco (Vietnam). Multi-walled carbon nanotube (MWCTN) was provided by nTherm, LLC (USA). Sulfosuccinic acid (SSA), concentrated nitric acid, concentrated sulfuric acid, hydrochloric acid (37 %), ammonia solution (25 %) were purchased from Sigma Aldrich (USA) and were used without any further purification. Graphite sheet (thickness of 200 μm) was obtained from Mineral Seal (USA).

2.2 Preparation of electrodes and cation-exchange membranes (CEM)

Preparation of the electrodes. 6 % PVA solution was prepared by stirring PVA powder in deionized water for 24 h at 90 ^{0}C. The solution was cooled down to room temperature, then glutaric anhydride (GA) was added and the mixture was kept stirring for 1 h. After that, MWCNT was added and the mixture was stirred for 1 more hour. The result slurry was homogenized in 10 minutes at 15,000 rpm with added AC following the published ratio [33]. A graphite sheet (200 mm × 300 mm, thickness of 200 μm) was used as a base for electrode coating using the doctor blade method. At the end, the electrodes were dried at 120 ^{0}C for 4 h.

Preparation of cation exchange membrane PVA/SSA/GA membrane. Firstly, a PVA solution was prepared similarly to the preparation of the electrode. Next, sulfosuccinic acid (SSA) solution was slowly added into the PVA solution, and the mixture was kept

stirring at 50 ^{0}C for 4 h. Then, GA was also added to the reaction mixture. This composite solution could either be directly coated on the electrode surface, or spread on a petri dish to receive the free-standing ion exchange membrane for membrane properties testing. Free-standing ion exchange membranes may be constructed by spreading the PVA-SSA/GA composite solution over a petri dish and drying it at 60 ^{0}C for 24 h, then heated to 80 ^{0}C for 1 h to establish the cross-linking process. Following that, the membrane was submerged in distilled water in 6 h for three times to completely remove unreacted components, and then dried for 4 h at 80 ^{0}C. The concentrations of GA and SSA were adjusted as described in Table 1. The characteristics of this free-standing ion exchange membrane were investigated, including mechanical properties, water absorption and ion exchange capacity. Additionally, the electrode was coated with a PVA/SSA/GA composite solution and the composite membrane electrode was dried at 120 ^{0}C in 4 h. The modified electrode was utilized for desalination investigation.

Table 1 Composition of the composite membranes

Sample	SSA concentration (%)	GA concentration (%)
GS1	1	
GS2.5	2.5	
GS5	5	5
GS7.5	7.5	
GS10	10	

2.3 Membrane characterization method

FT-IR spectra were acquired in the range of (4,000-650) cm^{-1} using the free-standing membranes in ATR mode on a Cary 630 FT-IR (Agilent Technologies Inc., Santa Clara, CA). SEM images were collected using JSM-IT500.

2.4 Water uptake capacity

Composite membrane (0.2 g) was soaked in 80 mL of water for 48 h at room temperature, then it was taken out and the excess water on the surface was blotted quickly. The soaked sample was weighed (m$_w$), then it was dried at 60 ^{0}C until reaching unchanged weight (m$_d$). Water uptake capacity of composite membrane was calculated based on the following formula:

$$WU(\%) = \frac{m_w - m_d}{m_d} \times 100 \qquad (1)$$

with m$_w$ and m$_d$ (g) being the mass of the wet and the dry membrane, respectively.

2.5 Cation-exchange capacity

The cation exchange capacity is an important electrochemical property of an ion-exchange membrane, and is a measure of the number of fixed charges per unit weight of the dry membrane. In order to determine CEC, the membrane (0.2 g) was immersed in 25 mL of 3 M NaCl for 24 h. 10 mL of the above solution was titrated with 0.0005 M NaOH in the presence of phenolphthalein. The cation exchange capacity (CEC) was calculated from the following equation:

$$CEC(\%) = \frac{C_{NaOH} \times V_{NaOH}}{m} mM \times 100 \qquad (2)$$

with m being the mass of the dry membrane, C$_{NaOH}$ being the concentration of the NaOH solution, V$_{NaOH}$ being the volume of the NaOH solution, and n = 2.5 being the dilution coefficient.

2.6 Electrochemical properties of the composite membrane electrode

Cyclic voltammetry (CV) was performed on Autolab 302 N using the three-electrode cell which included Pt as counter electrode, Ag/AgCl as reference electrode, and composite membrane electrode (3.0 × 2.5) cm as working electrode, in aqueous 0.5 M NaCl electrolyte. The electrodes were immersed in 0.5 M NaCl solution for 1 h before the experiment, each sample was measured three times from (-0.5 to 0.5) V at a scanning rate of 5 mV/s at room temperature. Specific capacitance of composite membrane electrode was calculated from the following formula:

$$C_S = \frac{1}{2 \times m \times v \times \Delta E} \times \int IdE \qquad (3)$$

where Cs (F g^{-1}) is the specific capacitance, $\int IdE$ is the integrated area of the CV curve, m (g) is the mass of the electrode, v (V/s) is the potential scan rate, ΔE (V) is the width of the potential window.
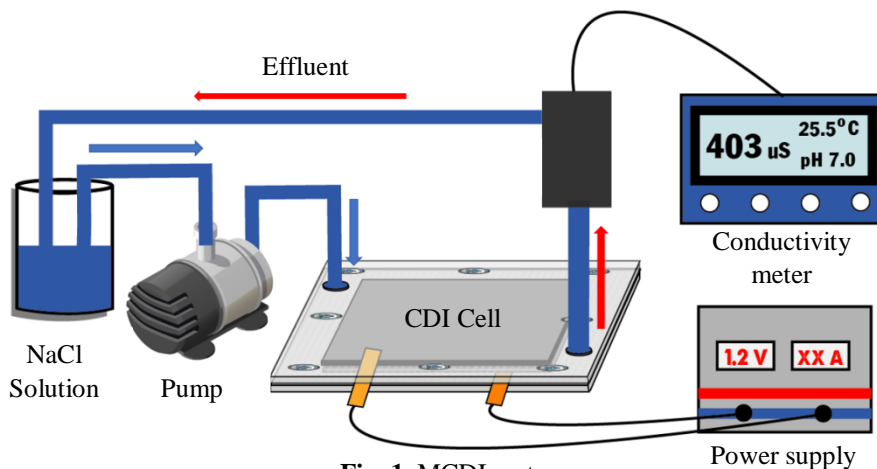
Fig. 1 MCDI system

2.7 Salt Adsorption on Composite Membrane Electrode

To investigate the desalination performance, the MCDI system was set up as described in Fig. 1, consisting of a CDI cell, a NKP-DCL-S10B peristaltic pump, and a Jenway 3540 conductivity meter. The CDI cell comprised of a pair of parallel electrodes (3.0×2.5) cm, thickness of $(100\text{-}300)$ μm separated from each other by an insulating plate. The feed water of 200 ppm NaCl solution was pumped through the CDI cell at a constant rate of 10 mL/min. The conductivity (G) of the inlet solution was observed until remaining unchanged (G_0). Next, the potential of 1.2 V was applied to the CDI cell, and the decreasing specific conductivity (G_t) was noted every 30 seconds until remain unchanged (G_c).

Salt adsorption capacity (SAC) and maximum salt adsorption capacity (mSAC) were calculated from the following formulas:

$$SAC = \frac{(C_0 - C_1) \times V}{m}$$

(4a)

$$mSAC = \frac{(C_0 - C_c) \times V}{m}$$

(4b)

Salt adsorption rate (SAR) and average salt adsorption rate (ASAR) were determined by the following formulas:

$$SAR = \frac{SAC}{t}$$

(5a)

$$ASAR = \frac{mSAC}{t}$$

(5b)

where SAC (mg/g) is the salt adsorption capacity; ASAR (mg/g·min) is the average salt adsorption rate; C_0, C_t and C_c (mg/L) are concentration of the NaCl solution which were calculated from the conductivity of the solution at the beginning, at t (minute) and at the end (G_0, G_t and G_c), respectively; V (L) is the volume of the NaCl solution; m (g) is the mass of the electrode; and t (minute) is the adsorption time.

3 Results and discussions
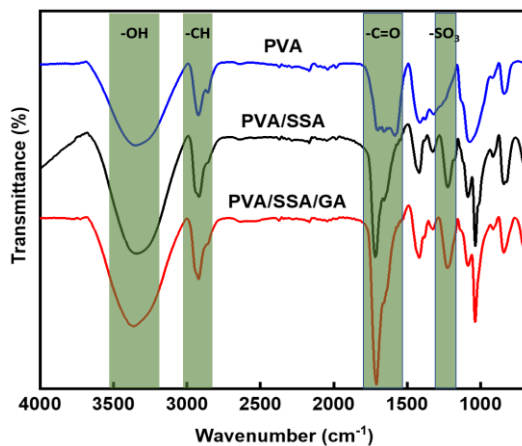
3.1 FT-IR analysis

Fig. 2 FT-IR PVA and PVA/SSA samples

The incorporation of SSA into the PVA chain was confirmed by Fourier-transform infrared spectroscopy (Fig. 2), as the peak at $(1,750\text{-}1,500)$ cm^{-1} was attributed to the COO-group of the crosslinking process between PVA and SSA, and the peak at 1,250 cm^{-1} was accredited to the $-SO_3$ group present in SSA. Other than that, the peaks at $(3,550\text{-}3,200)$ cm^{-1} and $(3,000\text{-}$

2,800) cm^{-1} were assigned to the –OH and the –CH groups of PVA, respectively.

3.2 Water uptake

The effect of SSA content on swelling behavior of the composite membrane was displayed in Fig. 3. Pure PVA exhibited high swelling behavior thanks to its hydroxyl (−OH) groups in its structure. Upon adding GA as crosslinking agent, the carboxyl (−COOH) groups reacted with the hydroxyl groups of PVA, reducing the number of hydroxyl groups and therefore lowering the water uptake capacity. When the SSA content increased, the number of hydrophilic –SO$_3$ groups of SSA incorporated in the PVA chain also increased, thus increasing the water uptake capacity of the cation exchange membrane (from 38.2 % at 1 % SSA to 98.5 % at 10 % SSA).
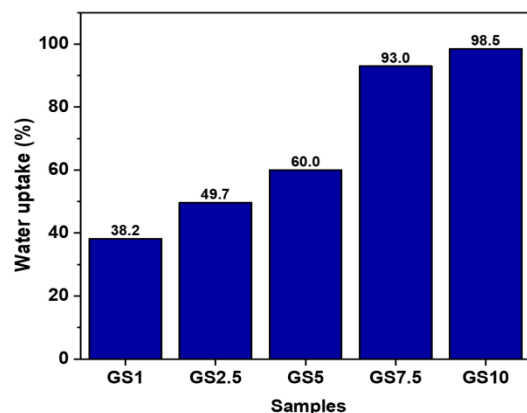

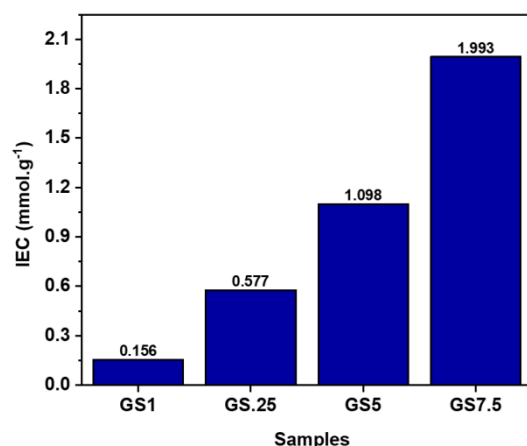**Fig. 3** Water uptake of the composite samples with different SSA content

Fig. 4 Cation exchange capacity of the composite samples with different SSA content

3.3 Cation exchange capacity

As displayed in Fig. 4, pure PVA possesses neither negative charge nor the ion exchange functional groups, thus it exhibits no cation exchange capacity (CEC). The CEC value increased from (0.156 to 1.993) mM/g when increasing the SSA content from (1 to 7.5) %, as more –SO$_3$ ion exchange groups were introduced to the composite membrane. The CEC value of GS7.5 sample was 12 times higher than that of GS1 sample and 1.81 times higher than that of GS5 sample.
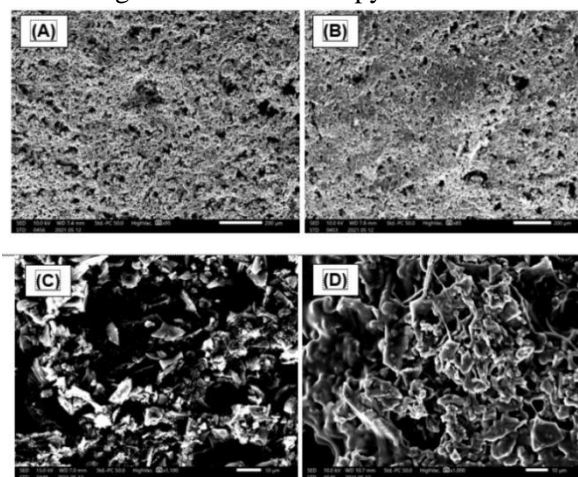
3.4 Scanning Electron Microscopy

Fig. 5 SEM images of the uncoated (A), (C) and coated electrode (B), (D) with different magnification of 90 (A), (B) and 1,000 (C), (D)
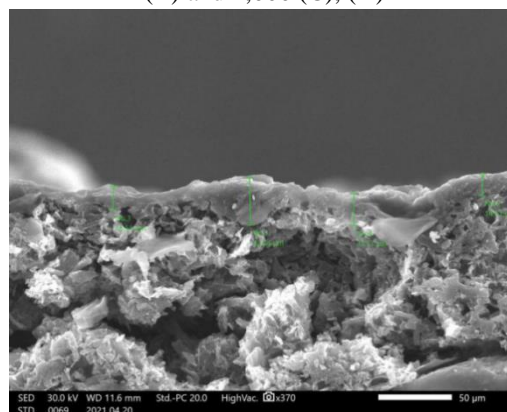

Fig. 6 SEM cross section image of the composite electrode

The morphology of the composite-membrane-coated electrode was investigated using scanning electron microscopy (SEM), and the images were presented in Fig. 5. The high porosity of activated carbon electrode (Fig. 5a and Fig. 5c) remained unchanged after being coated with the cation exchange membrane (Fig. 5b and Fig. 5d). At higher magnification, it can be seen

clearly that the carbon electrode was fully covered by the composite membrane (Fig. 5d).

SEM cross section image (Fig. 6) showed an equal distribution of the composite membrane on the electrode surface. Also, the thickness of the composite membrane was ununiformed, ranging of (10-30) μm due to the characteristic structure of activated carbon.

3.5 Desalination performance

In order to evaluate the effect of the composite membrane, desalination performance was assessed using the uncoated (DC) and the coated electrode (GS7.5). Fig. 7 displayed an adsorption/desorption cycle of the desalination process, approximately 13.4 minutes for GS7.5 and 85.3 minutes for DC. When adsorption reached saturation, in case of the uncoated electrode, the desorption process took place spontaneously just by stopping the circuit, while in case of the coated electrode, by alternating the current, it took less time for the desorption process to proceed. Besides, in the presence of the cation exchange membrane, the adsorption capacity of the electrode was enhanced due to its ability to adsorb ions as well as to prevent the co-ion effect at the micropores of the electrode. Therefore, it can be concluded that the cation exchange membrane in MCDI system helps reduce the time of the desalination process and ameliorate the ion adsorption capability of the electrode.
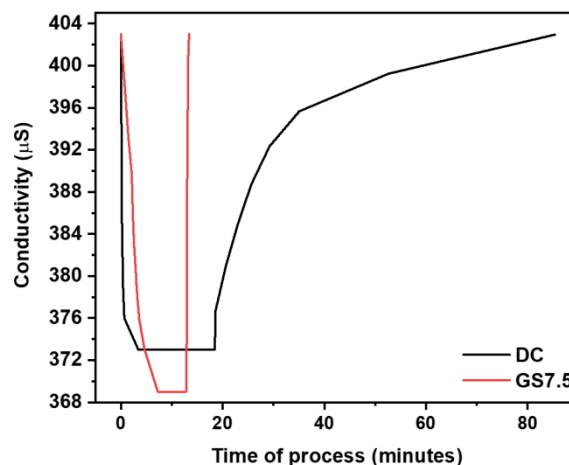

Fig. 7 Salt adsorption process using uncoated (DC) and coated electrode (GS7.5)

4 Conclusion

Cation exchange membrane was successfully fabricated from PVA and the crosslinking agents such as SSA and GA. The addition of GA helps enhance the strength as well as lower the water uptake capacity of the composite membrane. The membrane with SSA:GA ratio of 7.5 : 5 exhibited the best mechanical properties and the highest cation exchange capacity of 1.993 mM/g. The PVA/SSA/GA gel was also coated on the activated carbon electrode, which exhibited improved salt adsorption capacity and shorter process time compared to the conventional uncoated electrode.

Conflict of Interest
The authors declare that there is no conflict of interest.

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Chế tạo màng trao đổi cation polyvinyl alcohol/sulfosuccinic acid/glutaric anhydride ứng dụng cho quá trình khử mặn bằng công nghệ điện dung khử ion kết hợp màng trao đổi ion

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Tóm tắt Vấn đề thiếu nước sạch sinh hoạt hiện nay đang ngày càng trở nên nghiêm trọng, do nhu cầu sử dụng nước ngày càng tăng cùng với tác động của biến đổi khí hậu toàn cầu. Trước tình hình này, việc khử mặn các nguồn nước biển hay nước lợ được xem như một giải pháp khả thi. Công nghệ điện dung khử ion kết hợp màng trao đổi ion (MCDI) đã nhận được nhiều sự quan tâm trong vòng 15 năm qua, do có nhiều ưu điểm hơn so với các công nghệ khử mặn truyền thống như thẩm thấu ngược (RO) hay công nghệ điện thẩm tách (ED), bằng cách sử dụng một điện thế áp vào giữa hai điện cực được phủ màng trao đổi ion. Nghiên cứu này đã thành công trong việc chế tạo màng composite PVA/SSA/GA, một loại màng trao đổi cation rẻ tiền và thân thiện với môi trường, bằng phản ứng khâu mạng giữa sulfosuccinic acid (SSA) và glutaric anhydride (GA) với polyvinyl alcohol (PVA). Qua việc khảo sát ảnh hưởng của hàm lượng SSA và GA trong màng, ta thấy rằng sự có mặt của SSA trong màng trao đổi cation giúp cải thiện độ trương nở của màng (98,5 % so với 38,2 %), đồng thời tăng cường khả năng trao đổi cation (1,993 mM/g so với 0,156 mM/g). Bằng việc khảo sát khả năng khử mặn của điện cực có phủ hỗn hợp composite PVA/SSA/GA, ta thấy được việc sử dụng điện cực có phủ màng trao đổi cation giúp tăng cường hiệu quả hấp phụ muối và giảm thời gian khử muối (13,4 phút so với 85,3 phút) khi so sánh với điện cực không có phủ màng, chứng tỏ rằng màng composite PVA/SSA/GA là một ứng cử viên sáng giá có thể được áp dụng vào công nghệ MCDI.

Từ khóa Màng trao đổi cation, điện dung khử ion, polyvinyl alcohol, sulfosuccinic acid, glutaric anhydride