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Effect of processing agents on production of ion exchange membranes

Eliška Stránská[a], Jan Praus[b], Kristýna Janegová[a,*]

[a]*MemBrain s.r.o., Pod Vinicí 87, 471 27 Stráž pod Ralskem, Czech Republic, Tel. +420 725 358 422; email: Kristyna.Janegova@membrain.cz*
[b]*University of Chemistry and Technology Prague, Technická 5, 166 28 Praha 6, Czech Republic*

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ABSTRACT

Processing agents are commonly used in plastic processing to improve processability, stability of production, reduce drive torque and head pressure during homogenization and extrusion, reduce the cleaning intensity of the equipment, including lowering drooling on the extrusion die. Heterogeneous ion exchange membranes consist of a polymeric binder and an ion exchange resin. They are also produced by extrusion. Therefore, processing agents have been tested in the production of heterogeneous membranes. Three different types of processing agents were tested in the production of anion and cation exchange resin with two types of polyethylene binder. Processing agents were dosed at the expense of polymer binder in a concentration of 0.2–1.25 wt.%. The lowest and the highest recommended dose limit values of the processing agents were used in testing. Drive torque and head pressure were studied during homogenization and subsequent extrusion of the heterogeneous membrane. The most significant decrease of the head pressure and drive torque, about 20% or more, occurred with the use of a 1 wt.% Struktol agent, which is a fatty acid metal soap, amide. In addition, the preparation of the ion exchange membranes has been stabilized overall, the equipment can be easily cleaned after production, and the process temperature can be reduced. The physical and electrochemical properties of the manufactured ion exchange membranes were determined. Significant changes in properties of ion exchange membranes were not noted when processing agents were used.

Keywords: Heterogeneous ion exchange membrane; Processing agent; Homogenization; Extrusion

1. Introduction

Ion exchange membranes (IEM) are separation barriers, which divide charged particles after electrical potential is applied. IEMs are divided into cation exchange membrane (CEM) and anion exchange membrane (AEM) according to the ions, which are transported by the membrane and which are retained. Heterogeneous IEMs are a type of IEMs that contain an inert polymeric binder and ion exchange resin particles; IEMs can be reinforced with a reinforcing fabric. Heterogeneous IEMs can be prepared by extrusion [1–3].

The technology of extrusion production is high-temperature production of IEMs, where no hazardous or costly materials are used. When selecting suitable materials and preparation conditions, it should be kept in mind that the ion exchange resins are temperature sensitive in terms of functional group degradation [4,5]. Optimization of the production of heterogeneous IEMs by extrusion is, therefore, relatively important [6,7].

Melt flow instabilities, referred to as "melt fracture" are phenomena limiting IEMs extrusion [8]. The occurrence of instabilities for plastics is known since 1945 [9]. The flow is stable at low shear rates, the extrudate is smooth. The instability becomes apparent when the shear rate exceeds the first critical value. Regular inhomogeneities are formed on the surface of the extrudate, which are called "shark skin" or "surface melt fracture". "Stick slip" instability

* Corresponding author.

becomes apparent when the second critical shear rate value is exceeded. Finally, at higher shear rates, the flow is again stable, but the extrudate shows gross, irregular and chaotic instabilities referred to as "gross melt fracture" [10].

Processing agents are one of the ways to improve the processability of the membrane mixture (reducing head pressure or drive torque), reduce waste and downtime due to cleaning, or guarantee production stability and reduce production costs.

Fluoropolymers, siloxanes, stearates, polyalkylene oxides, polyesters and their derivatives are the most commonly used processing agents for polyolefins [10,11]. In the early 1960s, DuPont Canada accidentally discovered that fluoropolymers added in a small amount to linear low-density polyethylene work as slip agents and processing agents to the shark skin instability. Processing agents based on siloxanes have been effectively employed for many years to improve melt flow and release in plastic moulding and extrusion. Stearates, such as calcium and zinc stearates, are present in many commercial PE resins at a level of about 1,000 ppm as heat stabilizers and lubricants. Increasing the agent level typically results in greater pressure reduction and some delay of shark skin instability [11].

One agent from each of these groups was selected and tested for homogenization of the membrane mixture (the polymer binder and the ion exchange resin). The influence on processing parameters and properties of IEMs was studied.

2. Experimental setup

2.1. IEMs preparation

The strong acid cation exchange resin and the strong base anion exchange resin with styrene-divinylbenzene matrix (both Dow Chemical, Michigan, USA) were used to produce heterogeneous non-reinforced IEMs. Low-density polyethylene LDPE 605BA (ExxonMobil, Texas, USA) and enhanced polyethylene (EPE) Elite 5811 (Dow Chemical, Michigan, USA) were used as polymer binders. The finely milled ion exchange resin was mixed on a HAAKE PolyLab OS Rheo Drive 16 machine (Thermo Scientific Company, Massachusetts, USA) with a polymer binder at 150°C. The ratio of ion exchange resin to binder was 3:2. Processing agents were tested in this step. Properties of selected processing agents are listed in Table 1. Drive torque and head pressure were studied during homogenization. Processing agents were dosed at the expense of polymer binder in a concentration of 0.2–1.25 wt.%. The lowest and the highest recommended dose limit values of the processing agents were used in testing. The membrane mixtures were extruded to form the flat IEM on the same equipment at 120°C–150°C. Head pressure and drive torque were recorded during processing. A Rheomex PTW 24/28 twin screw (diameter and length/diameter ratio are 24 and 28, respectively) was used for homogenization and a Rheomex 19/25 single screw for extrusion, both with a compression ratio of 2:1 (both from Thermo Scientific Company, USA) [12]. The extruders were always cleaned with CleanPlus HD N material (IMCD Co., Netherlands) between processing different membrane mixtures. IEMs with no processing agents were made as reference materials.

2.2. IEMs characterization

Physical (relative water content at swelling in demineralized water) and electrochemical (specific resistance and permselectivity) properties of IEMs were characterized. Processing agents should contribute to increase production, reduce overall costs and generally improve and facilitate the production of heterogeneous IEMs, but must not significantly affect the properties of the produced heterogeneous IEMs.

2.2.1. Specific resistance

IEMs for measuring specific resistance (R_s, Ω cm) were equilibrated with 0.5 mol dm^{-3} NaCl for 24 h. Electrochemical resistance was measured using a 0.5 mol dm^{-3} NaCl solution at 25°C in a special experimental cell (specially manufactured for MemBrain s.r.o.) using the compensation method [13]. Electrochemical resistance was measured between reference electrodes (calomel electrodes) and followed by the application of constant direct current (I = 10 mA) between platinum electrodes. Electrochemical resistance was determined from two measurements of potential difference, and the first measurement was performed in the solution without an IEM (U_{solution}), while the second was run with a 0.785 cm^2 (active area, S) IEM ($U_{\text{solution + IEM}}$) installed [14,15]. Specific resistance was determined by Eq. (1) as follows:

$$R_s = \frac{S\left(U_{\text{solution+IEM}} - U_{\text{solution}}\right)}{\text{th I}} \tag{1}$$

where "th" is the thickness of IEM measured after the resistance measurement.

2.2.2. Permselectivity

Permselectivity describes the ability of IEMs to prevent co-ions from passing through [16] and is often discussed with transport number [17]. Permselectivity (P, %) of IEMs was determined using Henderson's method in the same measuring cell as electrochemical resistance but using a KCl solution of 0.1 and 0.5 mol dm^{-3} in the separated parts and without direct current applied. Potential ($U_{\text{meas.}}$) was measured between reference (silver–silver chloride) electrodes. Permselectivity was determined by Eq. (2). The IEMs were equilibrated with 0.5 mol dm^{-3} KCl for 24 h before the measurement [18,19].

$$P = \frac{U_{\text{meas.}}}{U_{\text{theor.}}} \times 100 \tag{2}$$

where $U_{\text{theor.}}$ is a theoretical potential from Nernst's law.

2.2.3. Physical properties

A piece of IEM was dried to constant weight in a hot air oven at 75°C. This weight was referred to as dry weight of IEM (m_{dry}). IEM was then immersed for 24 h in demineralized water. Subsequently, the IEM was weighed (m_{wet}). The relative water content (Δm, %) was determined by Eq. (3), as follows:

$$\Delta m = \frac{m_{wet} - m_{dry}}{m_{dry}} \times 100 \quad (3)$$

3. Results and discussion

All processing agents were tested during homogenization at the recommended dosage. The IEMs were extruded, and electrochemical and physical properties were measured. The Struktol agent appeared to be the best of all tested processing agents as there was a significant improvement in processability and produced IEMs did not show different properties [20]. Therefore, only data using Struktol agent and LDPE 605BA binder and Elite 5811 binder with cation exchange resin are presented in the paper. All other measured data are given in the supplementary material (Tables S1–S3).

Figs. 1a and b show head pressure and drive torque records from homogenization. At the beginning of material mixing, there is a gradual increase in head pressure and drive torque, after about 10 min, the values are stable. From these values, graphs of frequency vs. head pressure and drive torque (Figs. 1c and d) are plotted. From these graphs, we can see the effect of the processing agent Struktol on the mentioned process parameters. Head pressure and drive torque decreased. The stabilization of values, that is, stabilization of production of IEMs, is also clearly visible. Drive torque reduction is advantageous in view of lowering the load on the ion exchange resin during the process. Reducing both parameters allows for greater variability in other manufacturing parameters, such as the speed of the extruder screw, respectively production lines, amount of processed material per hour. Reduction of material residence time in the extruder is important due to the thermal degradation of the ion exchange resin.

In this context, it is possible to reduce the processing temperature. Both parameters (drive torque and head pressure) increased, but the head pressure and drive torque were still lower than when the processing agent Struktol was not used. The measurement took place at selected mixtures (Table 2) of CEM LDPE 605 + 1 wt.% Struktol during extrusion. The temperature of each zone of the extruder and the head was reduced by 5°C and then by 10°C compared with the extrusion temperatures originally used. When the temperature decreased, the head pressure increased. Drive torque increased only slightly with decreased temperature.

Another benefit of using Struktol agent is the disappearance of the so-called shark skin effect on the surface of the strings (Fig. 2a), which are extruded after homogenization of the polymeric binder and ion exchange resin [10,11]. A great benefit is the elimination of drooling on the extrusion die (Fig. 2b). Die drool in unwanted as the material can be cut off from the extrusion head by the drool build-up and the produced material can be destroyed. It is necessary to stop production regularly and clean the extrusion head. Loss of material will occur, production may not be stable, and the performance of the entire equipment will be reduced. When using the Struktol agent in 1 wt.% concentration, the shark skin on the string completely disappeared and die drool was reduced significantly (Fig. 2c).

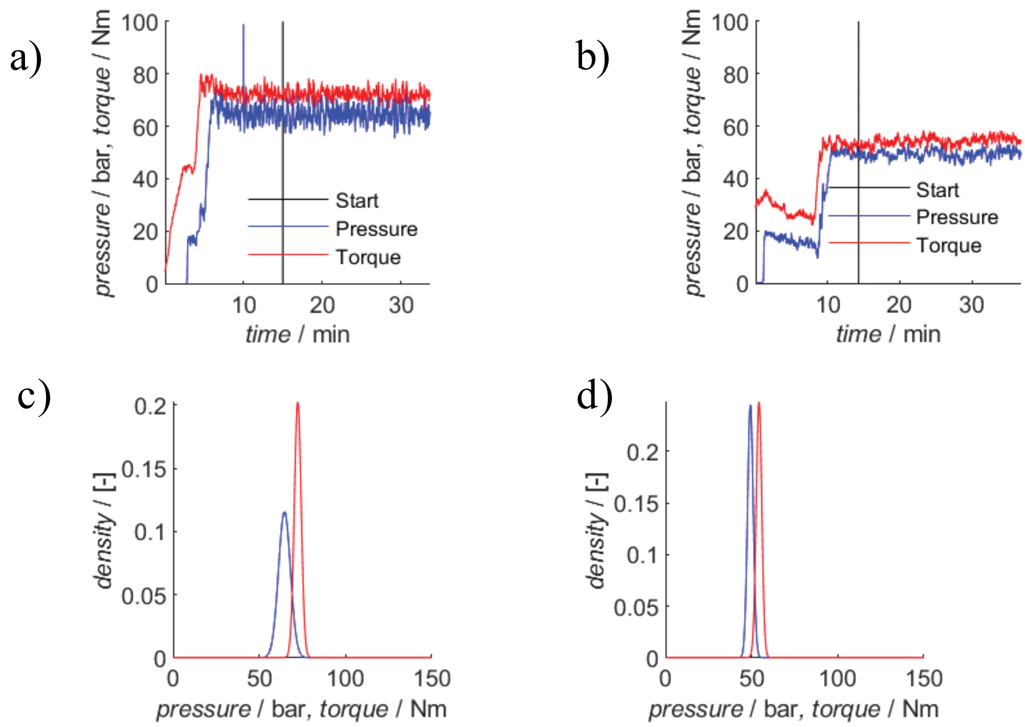

Fig. 1. Time dependence of head pressure and drive torque for (a) CEM LDPE 605 and (B) CEM LDPE 605 + 1 wt.% Struktol during homogenization; normal distributions of head pressure and drive torque during homogenization of (c) CEM LDPE 605 and (d) CEM 650 + 1 wt.% Struktol.

Summary of changes of head pressure and drive torque during homogenization and extrusion using 1 wt.% Struktol agent is shown in Fig. 3. Both anion and CEMs with LDPE 605BA and EPE Elite 5811 polymer binders are listed. There was a 21%–30% decrease in head pressure and a 18%–25% decrease in drive torque during homogenization. In the extrusion of IEMs, the drop of drive torque was even more pronounced, in the range of 28%–35%, the drop of head pressure varied from 8% to 20% depending on the type of mixture.

Improvements in process parameters must not have a negative impact on the resulting properties of manufactured IEMs. Figs. 4 and 5 show that there were no significant changes in electrochemical or physical properties. On the other hand, when using Elite 5811 binder, the specific resistances of IEMs decreased due to the use of Struktol. The effect of processing agents on the microstructure or crystallinity of IEMs was not studied yet. It will be subject of further research.

4. Conclusions

The paper deals with the use of processing agents in the production of heterogeneous ion exchange membranes (IEMs). Three types of processing agents were selected for IEMs with polyethylene binder. The processing agents were mixed with the ion exchange resin and the polymer during homogenization. Drive torque and head pressure were studied during homogenization and extrusion of IEMs. The best results were achieved using 1 wt.% of Struktol, which significantly reduced head pressure and drive torque, reduced drooling on the extrusion die, and eliminated the shark skin effect on the surface of membrane strings. There was 21%–30% decrease in head pressure and 18%–25% decrease in drive torque during homogenization. In the extrusion of IEMs, the drop of torque was even more pronounced, in the range of 28%–35%, the drop of head pressure varied from 8% to 20% depending on the type of mixture. Drive torque reduction is advantageous in view of lowering the load on the ion exchange resin during the process.

Table 1
Processing agents (LDPE – low-density polyethylene, UHMW – ultra high molecular weight)

Processing agent	Manufacturer	Chemical composition	Dosing
Struktol® TR 16	Struktol Co. – Ohio, USA	Fatty acid metal soap, amide	0.25–1.00 wt.%
Pearlene SiPE MB01	Momentive Performance Materials – Netherlands	UHMW polysiloxane, LDPE	0.20–1.25 wt.%
Maxithen® HP 7A8770PPA	Gabriel-Chemie Group – Austria	Fluorinated elastomer, LDPE	1.00 wt.%

Table 2
Process parameters of CEM LDPE 605 during extrusion at reduced temperatures

Composition	Temperature profile (°C)	p (bar)	σ p (bar)	Torque (Nm)	σ torque (Nm)
CEM 605	120, 130, 140, 140	91.6	1.8	26.2	1.2
CEM 605 + Struktol 1.00 wt.%	120, 130, 140, 140	84.5	0.5	18.7	0.5
CEM 605 + Struktol 1.00 wt.% –5°C	115, 125, 135, 135	87.3	0.6	18.9	0.6
CEM 605 + Struktol 1.00 wt.% –10°C	110, 120, 130, 130	90.6	0.8	19.1	0.6

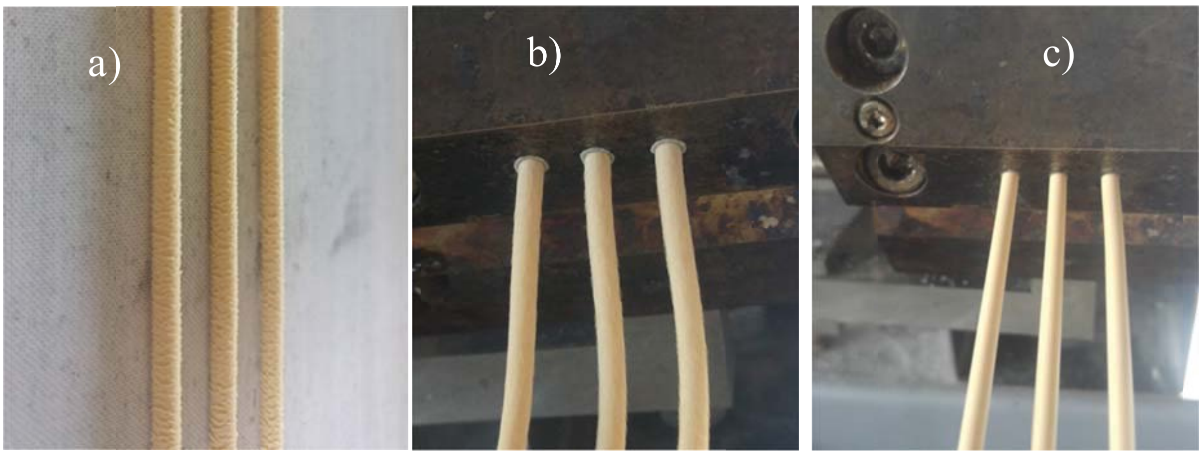

Fig. 2. (a) Membrane strings after homogenization with distinctive shark skin, (b) demonstrating drooling at the extrusion die during homogenizing of polymer with the ion exchange resin, and (c) reducing drooling and shark skin effect using 1 wt.% Struktol.

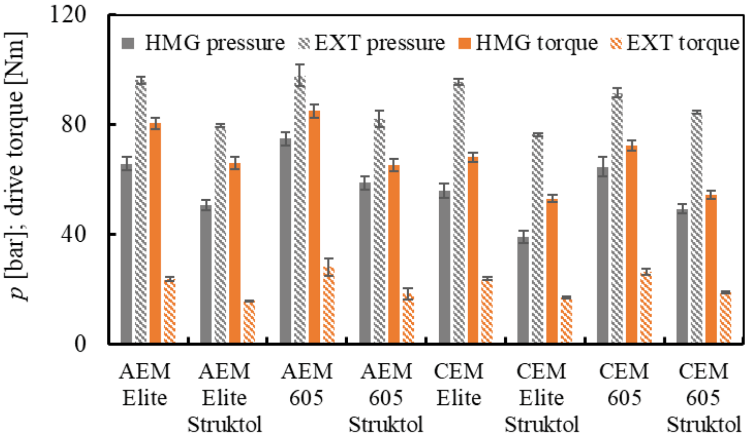

Fig. 3. Comparison of head pressure and drive torque during homogenization (HMG) and extrusion (EXT) for selected IEMs without agents (gray) and with 1 wt.% Struktol processing agent (orange).

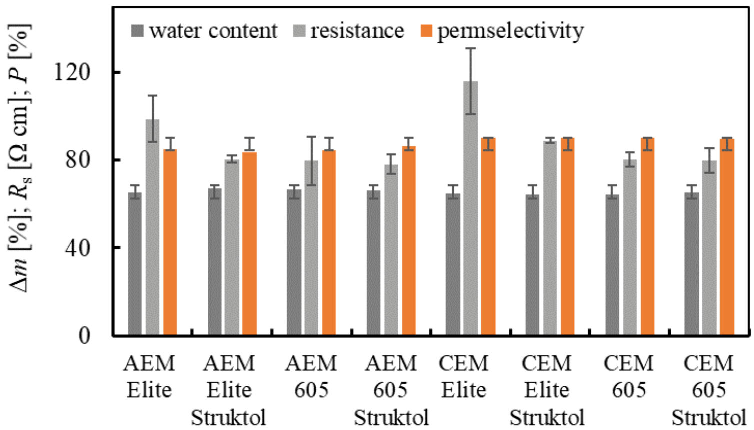

Fig. 4. Comparison of IEMs' properties (water content Δm, resistance R_s and permselectivity P) with 1 wt.% Struktol and properties of IEMs produced without processing agent.

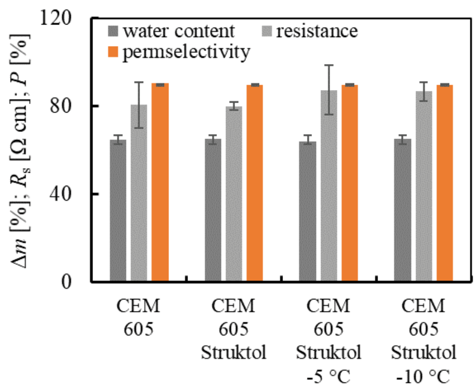

Fig. 5. Properties (water content Δm, resistance R_s and permselectivity P) of CEM LDPE 605 and CEM LDPE 605 +1 wt.% Struktol at extrusion with temperature reduction.

Reducing both parameters allows for greater variability in other manufacturing parameters, such as the speed of the extruder screw or the amount of processed material per hour. Reduction of material residence time in the extruder is important due to the thermal degradation of the ion exchange resin. The use of processing agents generally did not affect the physical and electrochemical properties of produced IEMs compared with IEMs without processing agent.

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Supplementary information

Table S1
Average head pressure p, drive torque and standard deviations σ in their normal distribution during homogenization of polymeric matrix with ion exchange resin

Composition	p (bar)	σ p (bar)	Torque (Nm)	σ Torque (Nm)
AEM Elite 5811	65.7	2.5	80.5	2.1
AEM Elite 5811 + Struktol 1.00 wt.%	50.7	1.8	66.0	2.1
AEM LDPE 605BA	74.8	2.6	85.0	2.5
AEM LDPE 605BA + Maxithen 1.00 wt.%	73.5	2.5	83.1	2.1
AEM LDPE 605BA + Pearlene 0.20 wt.%	73.9	2.9	83.4	2.6
AEM LDPE 605BA Pearlene 1.25 wt.%	73.7	2.4	80.8	2.4
AEM LDPE 605BA Struktol 0.25 wt.%	67.3	2.9	74.6	2.9
AEM LDPE 605BA Struktol 1.00 wt.%	58.7	2.3	65.3	2.2
CEM Elite 5811	55.8	2.5	68.1	1.8
CEM Elite 5811 + Struktol 1.00 wt.%	39.0	2.1	53.0	1.3
CEM LDPE 605BA	64.6	3.5	72.3	2.0
CEM LDPE 605BA + Pearlene 1.25 wt.%	63.5	3.3	67.8	2.5
CEM LDPE 605BA + Maxithen 1.00 wt.%	65.8	3.2	71.6	2.0
CEM LDPE 605BA + Pearlene 0.20 wt.%	64.1	3.7	71.6	2.2
CEM LDPE 605BA + Struktol 0.25 wt.%	61.9	3.0	64.9	2.6
CEM LDPE 605BA + Struktol 1.00 wt.%	49.3	1.6	54.3	1.6

Table S2
Average pressures *p*, drive torque and standard deviations in their normal distribution during extrusion of IEMs

Composition	*p* (bar)	σ *p* (bar)	Torque (Nm)	σ torque (Nm)
AEM Elite 5811	96.2	1.3	23.6	0.6
AEM Elite 5811 + Struktol 1.00 wt.%	79.6	0.5	15.6	0.3
AEM LDPE 605BA	97.9	3.9	28.0	3.2
AEM LDPE 605BA + Maxithen 1.00 wt.%	96.7	3.9	27.7	3.1
AEM LDPE 605BA + Pearlene 0.20 wt.%	94.4	4.3	27.5	2.9
AEM LDPE 605BA + Pearlene 1.25 wt.%	96.9	4.1	33.1	4.0
AEM LDPE 605BA + Struktol 0.20 wt.%	95.6	5.6	26.0	3.9
AEM LDPE 605BA + Struktol 1.00 wt.%	82.2	3.0	18.3	2.0
CEM Elite 5811	95.6	1.1	23.8	0.6
CEM Elite 5811 + Struktol 1.00 wt.%	76.4	0.6	16.9	0.3
CEM LDPE 605BA	91.6	1.8	26.2	1.2
CEM LDPE 605BA + Maxithen 1.00 wt.%	90.8	1.6	24.8	1.7
CEM LDPE 605BA + Pearlene 0.20 wt.%	90.4	2.5	25.1	2.0
CEM LDPE 605BA + Pearlene 1.25 wt.%	86.3	8.7	28.0	5.2
CEM LDPE 605BA + Struktol 0.20 wt.%	90.1	5.7	23.1	4.2
CEM LDPE 605BA + Struktol 1.00 wt.%	84.5	0.5	18.7	0.5
CEM 605BA + Struktol 1.00 wt.% –10°C	90.6	0.8	19.1	0.6
CEM 605BA + Struktol 1.00 wt.% –5°C	87.3	0.6	18.9	0.6

Table S3
Relative water content (Δm) and electrochemical (specific resistance R_s with standard deviation, permselectivity *P*) properties of IEMs

IEMs	Δm (%)	R_s (Ω cm)	σ R_s (Ω cm)	*P* (%)
AEM LDPE 605	66.7	79.8	11.3	84.7
AEM 605 + Maxithen 1.00 wt.%	64.4	82.1	7.5	84.9
AEM 605 + Pearlene 0.20 wt.%	64.8	70.3	2.5	85.5
AEM 605 + Pearlene 1.25 wt.%	63.1	74.9	13.7	85.3
AEM 605 + Struktol 0.25 wt.%	65.7	96.7	29.2	85.9
AEM 605 + Struktol 1.00 wt.%	66.2	78.2	4.4	86.3
AEM Elite 5811	65.5	98.9	10.6	85.1
AEM Elite 5811 + Struktol 1.00 wt.%	67.1	80.6	1.8	83.8
CEM LDPE 605	64.6	80,5	3.2	90.3
CEM 605 + Maxithen 1.00 wt.%	67.1	79.5	0.4	89.7
CEM 605 + Pearlene 0.20 wt.%	65.6	83.0	7.4	90.4
CEM 605 + Pearlene 1.25 wt.%	65.5	80.0	0.3	89.5
CEM 605 + Struktol 0.25 wt.%	65.4	87.8	4.3	90.0
CEM 605 + Struktol 1.00 wt.%	65.3	80.0	5.7	89.6
CEM 605 + Struktol 1.00 wt.% –10°C	65.2	86.7	0.6	89.6
CEM 605 + Struktol 1.00 wt.% –5°C	63.9	87.4	3.1	89.7
CEM Elite 5811	64.7	116.2	10.0	90.3
CEM Elite 5811 + Struktol 1.00%	64.6	89.0	1.1	90.3

AEM: anion exchange membrane; CEM: cation exchange membrane.