



## Selective electrochemical removal of monovalent and divalent ions from seawater

Utsav Raj Dotel<sup>1,2</sup>, Erik Dirdal<sup>1,2</sup>, Magne Olav Sydnes<sup>1</sup>, Ingunn Westvik Jolma<sup>3</sup>,  
Hans Urkedal<sup>2</sup>, Tor Hemmingsen<sup>1</sup>

<sup>1</sup> Department of Mathematics and Natural Science, University of Stavanger, NO-4036 Stavanger, Norway.

<sup>2</sup> Deox AS, Prof. Olav Hanssens vei 7A, NO-4021 Stavanger, Norway.

<sup>3</sup> Energy Department, International Research Institute of Stavanger (IRIS), Prof. Olav Hanssensvei 15, NO-4021 Stavanger, Norway.

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### Abstract

Selective removal of monovalent and divalent ions from ternary solution, synthetic seawater, and real seawater is studied in an electrodialysis cell with iridium oxide as anode and cathode. The experiments were performed at two different current densities, 10 A m<sup>-2</sup> and 100 A m<sup>-2</sup>. Sulphate was found to be difficult to remove at the lower current density (10 A m<sup>-2</sup>) while the cations were removed more efficiently at both current densities. The size, charge, and concentration of ions were found to be the main factors influencing the selective removal of monovalent or divalent ions. Scale formed in the recirculated electrolyte in the concentrate chamber could be removed upon treatment with 0.1 M citric acid.

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**Keywords:** Electrodialysis; Seawater; Ions removal; Membranes; Applied current density.

### 1. Introduction

Less than 3% of earth's water is fresh water, and the remaining water is saltwater which is not directly utilizable for drinking or household purpose without desalination [1]. The purification of brackish or seawater is an important issue to consider in areas suffering from fresh water scarcity [2]. Some industrial applications also require desalination in order to avoid problems, such as scale deposition or corrosion. Based on the applications, the presence of certain ions in seawater can either be problematic or beneficial [3]. Within the petroleum industry the effect of salts in injected water, the tendency of scale formation, and the need of treatment of discharged or reinjected water has received much attention [4-6]. Scale may be formed by mixing incompatible water, such as formation water which is enriched with ions from sedimentary minerals, and seawater that is enriched with sulphate. The formed scale will lead to reduced permeability in the reservoir and near-well area, or in the production lines [4]. In addition, change in temperature or pressure conditions may lead to precipitation in seawater [5]. Produced water separated from oil that will be reinjected in the well in order to improve oil recovery needs treatment prior to such use [6]. Historically, water flooding has mainly been used to maintain reservoir pressure because of the water's efficiency in displacing oil [7]. Less attention has been paid to the effects of water-rock interactions. Research has shown that the ionic composition of the water phase can influence the wettability

of the reservoir rock pore surface [8]. Sulphate ions have been found to make chalk and clay-rich formations more waterwet, and thereby expel more oil. Sulphate in combination with calcium or magnesium have been found to increase the oil recovery even more [9]. Although the presence of sulphate ions may improve the oil recovery, it can also lead to increased souring of the reservoir, as it can feed sulphate-reducing bacteria. This again can lead to increased production of  $H_2S$ , which is a huge problem in many oil fields today [10]. The optimal chemistry of the injection water will depend on the type of formation, the composition of the formation brine, and the type of oil in the reservoir. Water salinity can also affect the performance of typical enhanced oil recovery (EOR) chemicals. Polymer solution is one group of EOR chemicals that increases viscosity with decreasing water salinity, mainly due to interactions between the polymer and the divalent ions in the brine. For surfactants, salinity will determine the type of microemulsion a surfactant can form [11]. Thus, the salinity and specific ionic composition play a significant role for various industrial applications and the development of methods for the alteration of ions composition can be beneficial for different applications.

There are three major existing technologies for desalination [12]: distillation, reverse osmosis (RO), and electrodialysis (ED), where RO and ED are membrane based technologies. The membranes for RO operate under high pressure whereas the membranes for ED, anion exchange membranes (AEM), and cation exchange membranes (CEM), operate under the influence of electric potential. This study focuses on the alteration of ionic composition of seawater with ion exchange membranes using ED. The principle of ED involves the application of an electrical driving force to remove the ionic components from an aqueous solution through ion exchange membranes [13]. A typical ED cell consists of a series of AEM and CEM arranged in an alternating pattern separated by spacers and gaskets that makes up several compartments. The two end compartments are for anode and cathode, and they are responsible for generating the driving force for ions to separate. When a DC current is applied between two electrodes, the negatively charged anions migrate towards the anode by passing through the positively charged AEM, but cannot pass through negatively charged CEM. Similarly, the positively charged cations start to migrate towards the cathode and pass through the negatively charged CEM, but not through positively charged AEM. This results in an increase of ion concentration in adsal compartments with a simultaneously decrease of ion concentration in other desal compartments. Thus, desalinated streams (diluate) and concentrated streams (concentrate) are created in alternating channels [14-17]. The anode and cathode compartments are rinsed with an electrolyte solution in order to transfer electric current and to remove gases produced by electrode reactions. The major factors affecting the electrodialysis are ion concentrations, applied potential, retention time, membrane characteristics, operational and design parameters [13]. There have been a lot of studies on desalination of seawater for drinking and household purpose. However, there have not been many studies for ions alteration in seawater using electrochemical method.

The ionic composition of seawater used in this study is presented in Table 1 [18]. The monovalent ions,  $Na^+$  and  $Cl^-$ , and divalent ions,  $SO_4^{2-}$ ,  $Ca^{2+}$  and  $Mg^{2+}$  are the dominant ionic species in seawater. Several researchers have investigated ED to fractionate ions from aqueous solutions [19-25]. Although the research verifies that it is possible to separate multivalent ions from monovalent ions to some extent, the membrane selectivity is still very limited for practical applications [26]. Ions competition in migrating through the membrane may impact the ion selectivity in ED process in mixed salt waters [27]. Sata [21] studied the transport properties of anions through a nonselective AEM and a monovalent selective AEM under different pH and current density, and discovered that lowering the current density separated the monovalent anions from the multivalent anions for both membrane types. However, it was difficult to separate anions with similar size and charge. Due to the fact that monovalent selective membranes have a perm selective layer, they show selectivity towards ions with a different valences. This functional layer is argued to work on the principles of steric hindrance and electric repulsion [28]. Lambert et al. [29] reported the successful removal of trivalent chromium from sodium by using a modified cation exchange membrane. Zhang et al. [23] claimed the fractionation of multivalent ions from monovalent ions and were able to separate  $SO_4^{2-}$  from  $Cl^-$  in a mixture. Most of these studies were performed with equal amount of monovalent and divalent ions in the solution which do not represent the real conditions for seawater [24-26].

In this study, the efficiency and selective removal of ions in the nonselective AEM and CEM in the electrodialysis cell are tested at two different current densities,  $10 A m^{-2}$  and  $100 A m^{-2}$ . Galama et al. [30] has performed studies with similar procedures at the current densities range  $10-300 A m^{-2}$ . In this study, the different operational and design parameters, and ternary, synthetic, and real seawater are experimented. Several models based on Nernst-Planck equation for desalination are reported in the literature [30-33]. The widely used Nernst-Planck flux equation containing diffusion and migration term is stated in eq. (1) [30].

$$J_i = - \left[ \frac{D_i dc_i(x)}{dx} + D_i c_i(x) z_i \frac{d\phi}{dx} \right] \quad (1)$$

where,  $J_i$  is the ion flux ( $\text{mol m}^{-2} \text{s}^{-1}$ ),  $D_i$  is the ion diffusion coefficient ( $\text{m}^2 \text{s}^{-1}$ ),  $c_i$  is the concentration of ion ( $\text{mol m}^{-3}$ ),  $z$  is the valence of ionic species  $i$ ,  $\phi$  is the electric potential in the membrane.

## 2. Materials and methods

### 2.1 Desalination cell

The desalination cell (Figure 1a) was built up as a four-chambered cell with two end chambers for the anode and the cathode. The length and width of the cell was 20 cm and 10 cm, respectively, and the spacer was 5 mm. Two cation exchange membranes (Lanxess Sybron, IONAC MC-3470) were placed between the anode and cathode chamber, and an anion exchange membrane (Fumatech, FAS-PET-130) was placed between two cation exchange membranes. The active area of each membranes was 17 cm x 7 cm. Titanium mesh coated with iridium oxide, shown in Figure 1b, was used both as anode and cathode. The area of each electrode was measured to be approximately 165  $\text{cm}^2$ . For convenience, the four chambers were named anode chamber, adsal chamber, desal chamber and cathode chamber (left to right in series in Figure 1a). In the desal and adsal compartments, the concentration of ions was decreased (diluate) and increased (concentrate), respectively.

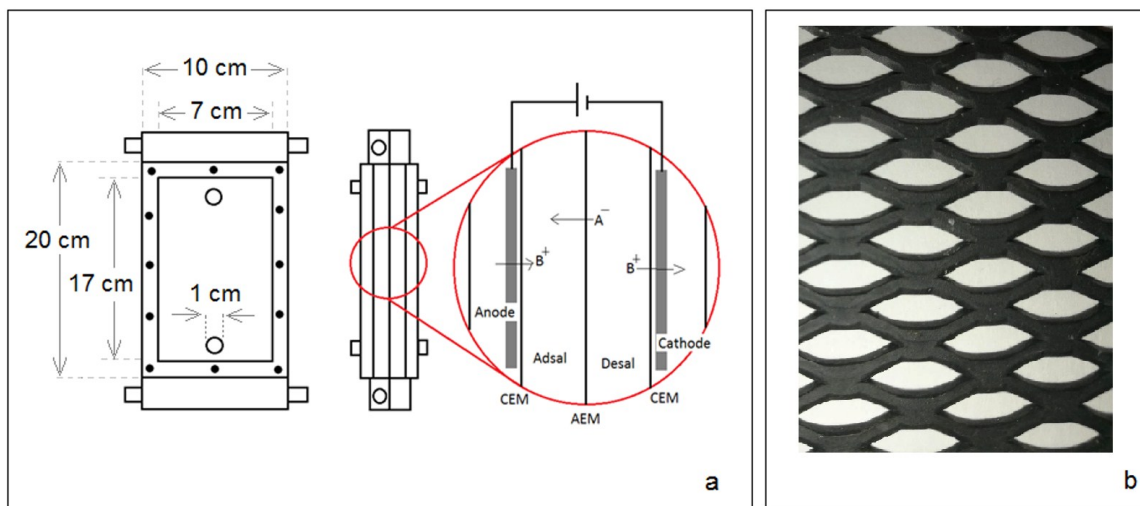


Figure 1. (a) Desalination cell; (b) Anode/cathode material.

### 2.2 Test set-up and procedure

Four different composition solutions were used for the experiments as shown in Table 1. Composition 1 was a 0.05 M sodium sulphate solution which was used both for the anode and cathode chamber in all experiments. Sulphate solution was used instead of chloride solution in order to avoid the chlorine evolution in the anode chamber. Composition 2 was a ternary solution of sodium chloride and sodium sulphate, used for both adsal and desal chamber. Composition 3 consisted of sodium chloride, sodium sulphate, potassium chloride, calcium chloride, and magnesium chloride, and was also used for both the adsal and desal chamber. Composition 3 was prepared as a synthetic seawater. Real seawater was tested as Composition 4 in both the adsal and desal chambers to simulate the field conditions.

All the tests were performed in a recirculation mode at a flow rate of 30  $\text{mL min}^{-1}$ . A volume of 300 mL of solution was used both for the adsal and the desal chamber, and 10 L solution was used for the anode and cathode chamber. For compositions 2 and 3, the tests were carried out at a current density of 10  $\text{A m}^{-2}$  for 8 hours or 100  $\text{A m}^{-2}$  for 4 hours. For composition 4, the test duration was for 8 hours at 10  $\text{A m}^{-2}$  and for 1.5 hours at 100  $\text{A m}^{-2}$ . The initial and final conductivity of the solution was measured. The experiments were carried out at room temperature. Deionized water was pumped through the cell followed by the test electrolyte for at least 1 hour before starting an experiment. The cell was also flushed with deionized water after each experiment to remove residual salt. After all the experiments with one composition, the cell was opened and observed for the possible scale formation, and 0.1 M citric acid was used for dissolving the

scales. The experiments were done under the controlled environment in the laboratory and the reproducibility of the results was ensured by comparing the results obtained for different water qualities at different current densities.

A peristaltic pump (Watson Marlow 520S) was used for circulating the electrolytes, and a power supply (Manson HCS 3300) was used for supplying the current to the electrodes in the cell. A conductivity meter (WTW 350i) was used for measuring the conductivity of the solution. The initial and final ions composition in the solution was measured by the ion chromatography (Dionex<sup>TM</sup> ICS-5000).

Table 1. Ion composition (M) of the electrolytes in the compartments.

Ions	Electrolyte 1 Anode/Cathode compartment	Electrolyte 2 Adsal/Desal compartment	Electrolyte 3 Adsal/Desal compartment	Electrolyte 4 Adsal/Desal compartment
Na <sup>+</sup>	0.100	0.465	0.465	0.373
K <sup>+</sup>	-	-	0.011	0.0032
Ca <sup>2+</sup>	-	-	0.010	0.007
Mg <sup>2+</sup>	-	-	0.051	0.045
Cl <sup>-</sup>	-	0.409	0.542	0.438
SO <sub>4</sub> <sup>2-</sup>	0.050	0.028	0.028	0.043

### 3. Results and discussion

The reduction in concentration of Na<sup>+</sup>, Cl<sup>-</sup> and SO<sub>4</sub><sup>2-</sup> ions in ternary or synthetic seawater solutions at applied current density of 10 A m<sup>-2</sup> for 8 hours and 100 A m<sup>-2</sup> for 4 hours experiments is shown in Figure 2. In the ternary solution, applying a current density of 10 A m<sup>-2</sup>, the concentration of ions was reduced noticeably. The conductivity was decreased from 42.2 mS cm<sup>-1</sup> to 31.1 mS cm<sup>-1</sup>, and Na<sup>+</sup>, Cl<sup>-</sup> and SO<sub>4</sub><sup>2-</sup> concentrations decreased by 26%, 31% and 8%, respectively, as determined by ion chromatographic analyses. Since conductivity is influenced by H<sup>+</sup> ions formed in the process these conductivity data are only reported as supporting results. In synthetic seawater, at 10 A m<sup>-2</sup>, the conductivity decreased from 50.6 mS cm<sup>-1</sup> to 40.0 mS cm<sup>-1</sup>, and Na<sup>+</sup>, Cl<sup>-</sup> and SO<sub>4</sub><sup>2-</sup> concentrations decreased by 19%, 19% and 0.05%, respectively. The concentration of SO<sub>4</sub><sup>2-</sup> in simulated seawater was not altered much at low current density, while the Cl<sup>-</sup> concentration was reduced noticeably even at a lower current density.

When an applied current density of 100 A m<sup>-2</sup> was used in ternary solution or synthetic seawater, the concentrations of ions were lowered significantly. The conductivity decreased from 42.2 mS cm<sup>-1</sup> to 3.0 mS cm<sup>-1</sup> in ternary solution, and from 50.6 mS cm<sup>-1</sup> to 15.0 mS cm<sup>-1</sup> in synthetic seawater. The concentration of Na<sup>+</sup>, Cl<sup>-</sup> and SO<sub>4</sub><sup>2-</sup> ions decreased by 99%, 90%, and 80% in ternary solution, and 72%, 77%, and 31% in seawater, respectively. The sulphate concentration decreased significantly in ternary solution at 100 A m<sup>-2</sup> from 0.409 M to 0.006 M.

The percentwise reduction of monovalent and divalent ions from synthetic seawater at 10 A m<sup>-2</sup> and 100 A m<sup>-2</sup> is shown in Figure 3. At 10 A m<sup>-2</sup>, the ions reduction trend (maximum to minimum) is Ca<sup>2+</sup>, K<sup>+</sup>, Na<sup>+</sup>, Cl<sup>-</sup>, Mg<sup>2+</sup>, SO<sub>4</sub><sup>2-</sup>, and at 100 A m<sup>-2</sup>, the trend is K<sup>+</sup>, Ca<sup>2+</sup>, Cl<sup>-</sup>, Mg<sup>2+</sup>, Na<sup>+</sup>, SO<sub>4</sub><sup>2-</sup>. At 100 A m<sup>-2</sup>, K<sup>+</sup>, Mg<sup>2+</sup>, and Ca<sup>2+</sup> decreased by 92%, 76%, and 84%, and at 10 A m<sup>-2</sup>, by 43%, 16%, and 58%, respectively. Sulphate was most difficult to remove at both current densities. However, at a current density of 100 A m<sup>-2</sup>, 31% of the sulphate ions were removed. The low removal rate of sulphate is most likely due to the ionic radii with sulfate being the largest anion in the tested electrolytes, see Table 1, and also being a divalent ion. However, this effect was not observed among the cations, and the CEM was found to be selective for most of the cations.

The removal of ions in the ternary solution with respect to the recirculation time at the current density of 100 A m<sup>-2</sup> is shown in Figure 4. Na<sup>+</sup>, Cl<sup>-</sup>, and SO<sub>4</sub><sup>2-</sup> concentrations are decreasing significantly over the course of 3.5 hours. After 3.5 hours, there is a decrease in the removal rate of Na<sup>+</sup> and Cl<sup>-</sup>, however, the removal rate of SO<sub>4</sub><sup>2-</sup> increases drastically. The decrement in the concentration of sodium and chlorine ions causes the maximum availability of SO<sub>4</sub><sup>2-</sup> which starts to pass easily across the membrane. After 4 hours, the applied current density in the simulated seawater was increased from 100 A m<sup>-2</sup> to 170 A m<sup>-2</sup>, which resulted in a significant reduction of sulphate in the solution (Figure 4b). The availability of large amount of ions during the initial phase hinders the mobilization of SO<sub>4</sub><sup>2-</sup> through the membrane. Higher current density combined with lower concentration of other ions in the water facilitated the removal of SO<sub>4</sub><sup>2-</sup> ions from the synthetic seawater.

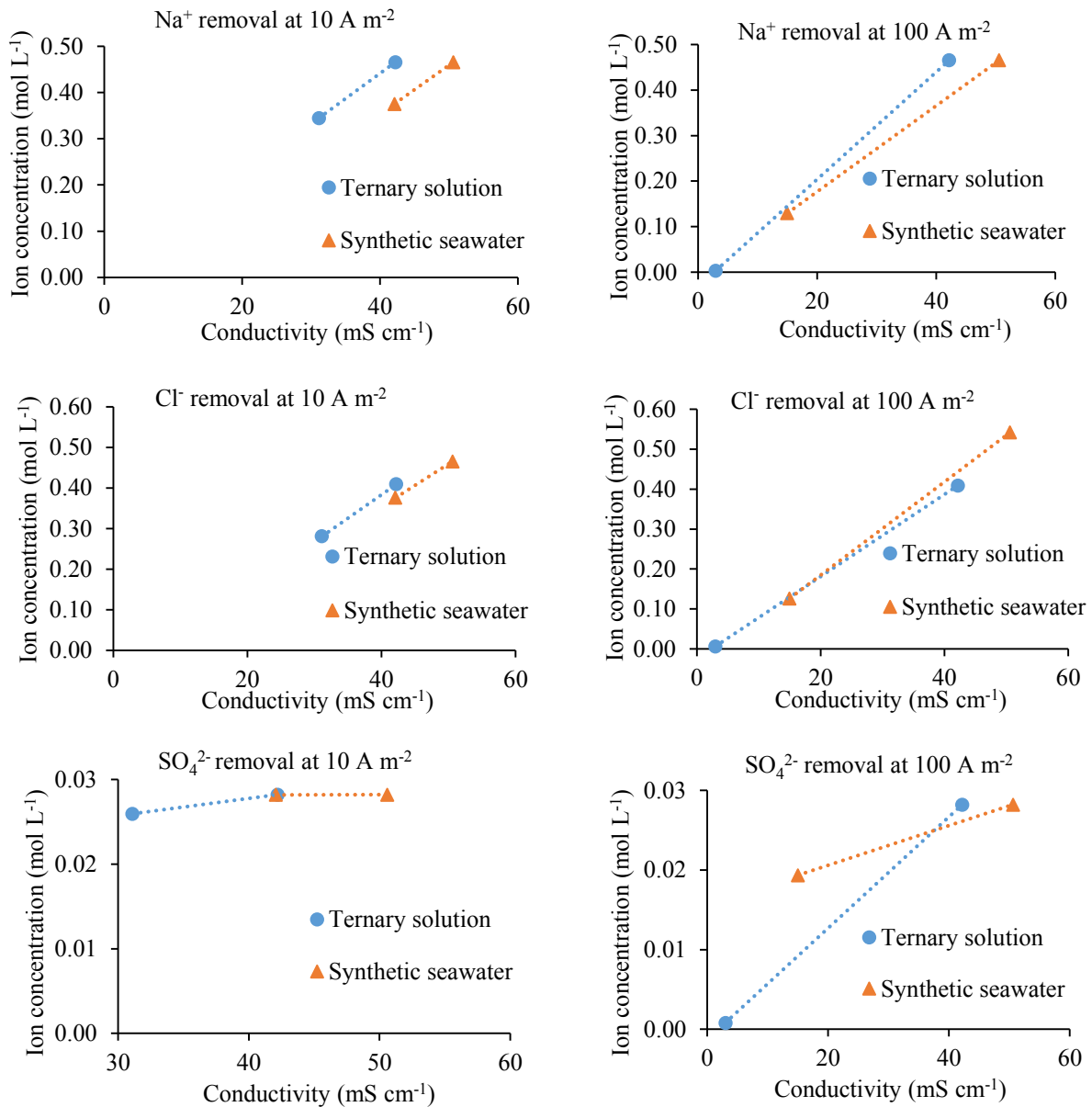


Figure 2. Reduction in concentrations of Na<sup>+</sup>, Cl<sup>-</sup>, and SO<sub>4</sub><sup>2-</sup> in ternary solution and synthetic seawater at a current density of 10 A m<sup>-2</sup> for 8 hours and 100 A m<sup>-2</sup> for 4 hours.

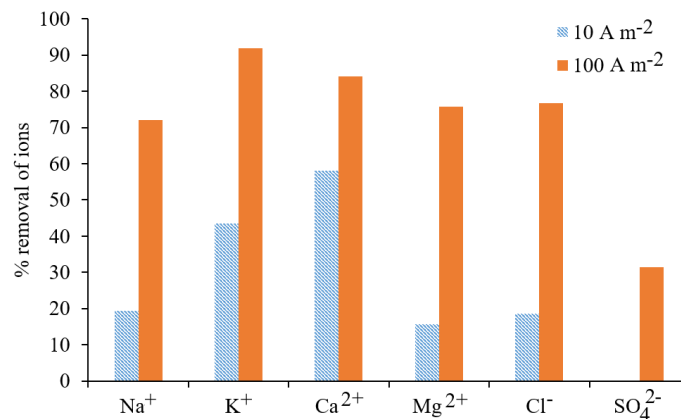


Figure 3. Removal of ions (in %) in synthetic seawater at 10 A m<sup>-2</sup> for 8 hours and 100 A m<sup>-2</sup> for 4 hours.

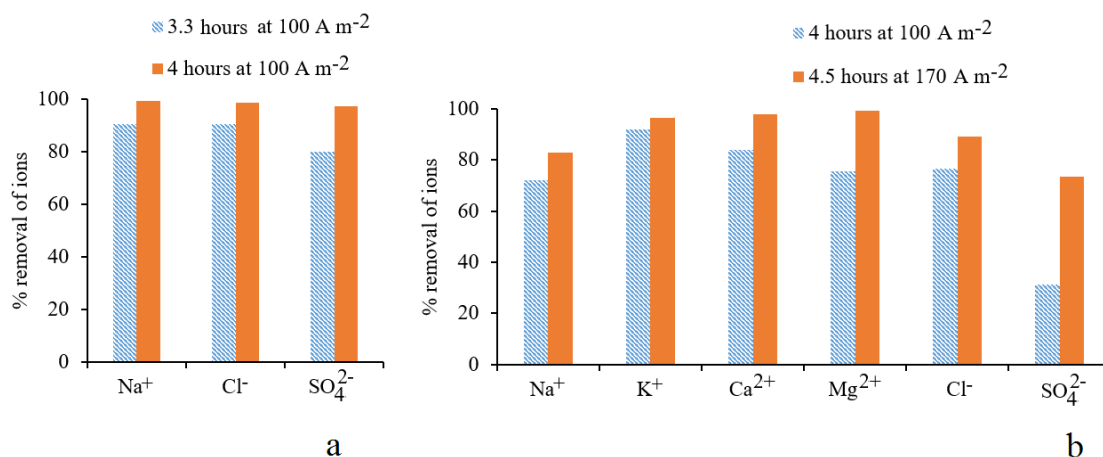


Figure 4. Percentage of ions removed in (a) Ternary solution and (b) Synthetic seawater.

Figure 5 shows the ions removed from real seawater at 10 A m<sup>-2</sup> over 8 hours and at 100 A m<sup>-2</sup> over 1.5 hours. The concentration of Ca<sup>2+</sup> ions was found to be reduced the most at both current densities. The pattern for ion removal followed a quite similar pattern to what was found for synthetic seawater, with highest removal of Ca<sup>2+</sup> ions and lowest removal of SO<sub>4</sub><sup>2-</sup> ions. There were some indication of scale formation at 100 A m<sup>-2</sup> which prompted us to stop the test with synthetic seawater after 1.5 hours. The reduction in concentration of ions was significantly higher in the experiment conducted for 1.5 hours at 100 A m<sup>-2</sup> compared to the experiment conducted at 10 A m<sup>-2</sup> for 8 hours. The Cl<sup>-</sup> ion concentration was found to be decreased more at 10 A m<sup>-2</sup> compared to 100 A m<sup>-2</sup>, and no specific conclusion was drawn from this result. However, sulfate was found to be reduced more at 100 A m<sup>-2</sup> and the competition among anions might have decreased the Cl<sup>-</sup> mobilization. A detail study on the anions mobilization across AEM at higher current density is required for further explanation.

The removal rate of monovalent and divalent ions are dependent on several factors. The size of the ions is anticipated to play some role in the transport of ions across the membrane. Among the ions used in the study, SO<sub>4</sub><sup>2-</sup> has the largest ionic radii of 2.30 Å, see Table 2. The hydrated radius of SO<sub>4</sub><sup>2-</sup> is 3.79 Å whereas for Cl<sup>-</sup>, it is 3.32 Å. The larger anions are more hindered from passing across membranes compared to smaller anions [24, 27, 35].

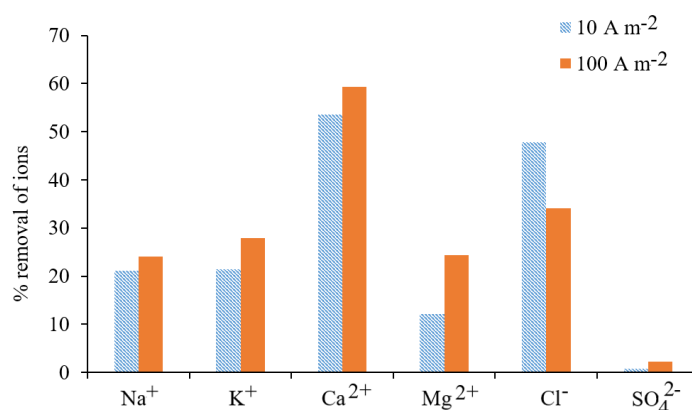


Figure 5. Removal of ions (in %) in seawater at 10 A m<sup>-2</sup> for 8 hours and 100 A m<sup>-2</sup> for 1.5 hours.

Table 2. Ionic radii for ions present in seawater [34].

Ions	Ionic Radii (Å)	Hydrated ionic radii (Å)
Mg <sup>2+</sup>	0.72	4.28
Ca <sup>2+</sup>	1.00	4.12
Na <sup>+</sup>	1.02	3.58
K <sup>+</sup>	1.38	3.31
Cl <sup>-</sup>	1.81	3.32
SO <sub>4</sub> <sup>2-</sup>	2.30	3.79

The competition among the ions in the electrolyte is another important factor for mobilization of ions across the membrane. In the ternary solution,  $\text{SO}_4^{2-}$  were found to be removed in higher amounts at both applied current densities compared to the situation in synthetic and real seawater. The result indicated that the competition is not only among the anions, but the presence of large concentration of cations can also affect the movement of anions. Similarly, the removal of  $\text{SO}_4^{2-}$  was found to be more efficient at lower concentrations of other ions in the electrolyte. This is in agreement with the findings of Kabay et al. [27] who presented a competition experiment among  $\text{Cl}^-$ ,  $\text{F}^-$  and  $\text{SO}_4^{2-}$ , and showed that when 90% of  $\text{Cl}^-$  is removed, the  $\text{F}^-$  removal rate increased and after removal of 98% of  $\text{F}^-$ , the  $\text{SO}_4^{2-}$  removal rate increased.

Thus, the size and charge of the ions, and the ionic composition of monovalent and divalent ions in a solution play a significant role for their mobilization across the membrane. It is expected that the combined effect of these properties regulate the selective removal of ions on the membrane. It is also found from this study that the use of high current density can remove the monovalent ions and divalent cations in a significant amount. Followed by focusing on only removing sulphate can be an efficient method for desalination of seawater. Only removal of monovalent ions and divalent cations can yield sulphate rich water, in cases where that is wanted. The current density plays a significant role for effective use of electro dialysis techniques for the removal of ions. In seawater, a higher current density can be used for more efficient removal of divalent ions. Another method such as nanofiltration could be used prior to electro dialysis in order to remove the sulphate to make the method more efficient, or eventually after dialyses at lower applied current for removing the last traces of sulphate. In addition to the membrane and cell properties, further research should focus on the collective effect of charge, size, ionic composition, and applied current density in order to develop methods for more selective ion removal. Development of selective removal of ions by the membrane would have significant impact for applications such as EOR by water injection into reservoir. At present, the integration of preferential removal of ions removal using electro dialysis with other techniques such as nanofiltration can help in achieving a desired concentration of certain ions in water [26]. Thus, the use electrochemical desalination can achieve in alteration of ionic composition which can benefits different industrial applications. In the adsal chamber, precipitation of calcium sulphate was observed at  $100 \text{ A m}^{-2}$  both for synthetic and real seawater. The formed scale could be dissolved in 0.1 M citric acid. Thus, the use of citric acid in rinsing the cell can be a good option in order to solve the scale problem in the adsal chamber. The future work will be focused on the durability of the cell and problems associated with scales and biofilms in selective desalination cell.

#### 4. Conclusion

The experiments showed that the concentration of ions can be significantly lowered in ternary solution, synthetic seawater, and real seawater at  $10 \text{ A m}^{-2}$  and  $100 \text{ A m}^{-2}$  applied current densities. Monovalent ions and divalent cations were easier to remove than divalent anion sulphate. Sulphate in ternary, synthetic, and real seawater was more difficult to remove compared to other ions. However, at higher applied current densities of  $100 \text{ A m}^{-2}$  and with less competing monovalent ions, the sulphate concentration could be significantly reduced. In addition to the membrane property, the size and the charge of the ions, applied current density, and availability of ions should be considered for the development of more selective electro dialysis cell.

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Corresponding author: Utsav Raj Dotel, utsav.dotel@uis.no