

# Evaluation of the usefulness of bench-scale studies for the determination of full-scale membrane performances

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## **Keywords**

Reverse osmosis, bench scale, pilot scale, industrial application, scale-up, concentration, saline water

## **Abstract**

In this study, bench-scale reverse osmosis (RO) study results were compared to pilot-scale RO study results in order to evaluate the relevance and usefulness of bench-scale studies in the assessment of full-scale membrane performances. The study was conducted with process wastewater from a novel saline soil remediation technology. The membrane selected for the study was a BW30 RO membrane from Filmtec. It was determined that the membrane's salt rejection capacity as well as its water recovery capacity could be accurately estimated by bench-scale tests. With an operation pressure of 2760 kPa, an average water recovery capacity of 56% with a salt rejection of 92% was obtained with the bench and pilot-scale units. However, transport parameters, fouling, and concentration polarization measurements were found to be different between the bench-scale and pilot-scale tests. The  $\alpha$  factor which represents the effect of concentration polarisation was found to be 30% higher for the pilot-scale unit compared to the bench-scale unit. The main explanation for this disparity is the difference in membrane configuration between the bench-scale and the pilot-scale units used in this study.

## **1. Introduction**

Reverse osmosis (RO) was first introduced in the 1950's as a novel filtration technology capable of separating ions from water. Since then, this technology has been utilized around the world for multiple industrial applications, including drinking water purification[1], water and wastewater purification[2], maple syrup production, groundwater desalination[3], seawater desalination[4, 5] and many more[6, 7].

RO technology is constantly being improved upon, with new market applications being subsequently discovered. For each new application, studies must be conducted in order to evaluate the design parameters of the RO unit used, such as the most suitable membrane, the membrane's permability coefficient, the membrane's rejection capacity of the solutes present in the water to be filtered, the flux decline that could be potentially caused by foulants and the appropriate cleaning procedure. Usually, these studies are first conducted as bench-scale and then as pilot- and industrial-scale. However, the question remains whether the bench-scale studies are useful in the determination of full-scale membrane performances.

In this study, RO was investigated for a new application. During the last few years, the department of Innovation and Development of Englobe Corp. has developed an innovative salt contaminated land remediation technology [8]. With this technology, rather than performing in-situ remediation, saline soils are excavated and transported to a soil treatment facility specifically designed for their treatment. While this soil remediation technology offers more efficient salt removal than conventional technologies, it still produces a large volume of highly saline process wastewater (3 to 40 g/L of total dissolved solids) that needs to be disposed off by deep well injection [9]. The distance between the remediation site, the water supply and the disposal options, and costs associated with this distance (water transportation and supply/disposal costs) set certain geographical limits and thus dictate the economical applicability of the technology. One option to extend such geographical limits, therefore reaching a larger portion of the market, is to combine the new remediation technology with a RO unit, thus minimizing water usage. Since very little data concerning the concentration and reuse of saline soil treatment wastewater was available in the litterature [7], bench-scale, as well as pilot-scale tests were performed in order to evaluate the RO unit design parameters for this particular application.

### 1.1. Objectives and scope of work

The litterature concerning RO contains a wide variety of bench-scale and pilot-scale studies. However, very few of them compare the difference between the bench-scale tests and the pilot-scale tests results. In this study, we evaluated the relevance and usefulness of bench-scale RO studies in the assessment of full-scale membrane performance for a specific application, while subsequently providing a method for assessing membrane performances. The main parameters evaluated in this study were the water recovery capacity, the transport parameters and salt rejection capacity, and the fouling of the membrane.

## 2. **Material and methods**

### 2.1. Membranes

BW-30 reverse osmosis membranes were obtained from Filmtec, a wholly owned subsidiary of the Dow Chemical Company. The 3 membranes were received as 4 inches spiral wound (7 m<sup>2</sup> of membrane surface) with fiberglass outer wrap. To get membrane coupons for lab tests, a spiral wound was cut using a saw and then cut to fit the bench-scale RO cell. The inused membrane

coupons were stored in a bin filled with 1.5% sodium metabisulfite solution, stored at room temperature. The opened spiral wound was placed in a 6 inch PVC pipe filled with 1.5% sodium metabisulfite solution and stored at room temperature.

## 2.2. Bench scale RO system

The laboratory set-up was designed according to the diagram at Fig. 1. The key components were the membrane test cell (1), the high pressure pump (2) and the feed water reservoir (3).

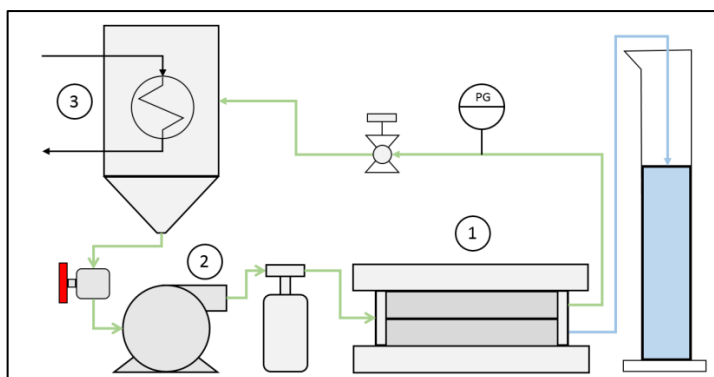


Fig. 1 Bench scale reverse osmosis set-up

Experiments were conducted using a commercially available bench-scale membrane test cell (Sepa CF, GE Osmonics). The stainless steel module is adapted to flat-sheet membranes provided by membrane suppliers. The flow channels on each side of the membrane were filled with a mesh spacer to simulate the hydrodynamics of a spiral-wound membrane element [10]. These mesh spacers were cut from the spiral wound at the same time as the membrane coupons were cut. Feed water was circulated on the active layer side of the membrane through ten round 4.7 mm diameter openings. The effective surface area of the membrane was 149 cm<sup>2</sup> (10 cm wide and 15 cm long with rounded corners). Permeate was then collected through another ten round 4.7 mm diameter openings located in the center of the membrane coupon. Pressure on the stainless steel module was maintained at 4130 kPa (600 psi) to avoid leakage.

The high-pressure feed water pump was a Hydra-Cell positive displacement pump with a diaphragm design. In order to stabilize the pressure, a bladder accumulator was installed at the outlet of the pump. Pump speed was set manually by the operator. Pressure in the system was adjusted manually with a globe valve and measured with an analogic pressure gauge placed between the valve and the outlet of the membrane test cell.

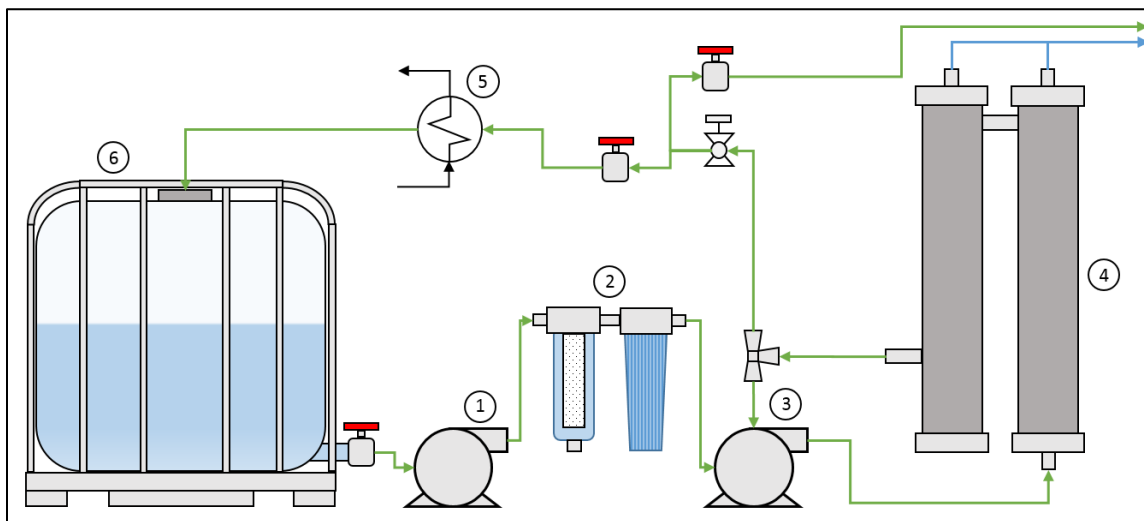
The feed water reservoir was a double shell stainless steel tank with a capacity of approximately 4 L. Temperature control was provided by a heat exchanger which circulated a cooled glycol solution inside the reservoir walls. To ensure adequate cooling, temperature in the tank was recorded manually for the duration of the experiment.

The permeate flow was calculated by collecting a sample at the outlet of the cell every 10 minutes. The volume of the sample was measured with a 50 mL graduated cylinder. The

electrical conductivity of the sample, and the recirculated feed, was measured using a Thermo Scientific Orion 013005MD conductimeter.

### 2.3. Pilot scale RO system

Pilot-scale experiments were conducted using a small-scale commercial RO unit (Turbo Compak, Darveau) designed according to the diagram at Fig. 2. The key components were the feed pump (1), the pre-treatment system (2), the high-pressure pump combined to a recirculation pump (3), the RO membranes (4), the heat exchanger (5) and the feed water reservoir (6).



Water was supplied to the system by a 0.37 kW (1/2 hp) peristaltic pump. The pump operated at a constant speed when the system was switched on. Feed water was pretreated through a two-step filtration system: the first filter was a washable 24  $\mu\text{m}$  mesh filter and the second filter was a 5  $\mu\text{m}$  disposable cartridge filter. Filter fouling was monitored with 2 analog pressure gauges located upstream and downstream of the filters. When the pressure difference was over 70 kPa (10 psi), the mesh filter was washed and the cartridge filter was replaced.

The high-pressure pump installed on the Turbo Compak unit is a Double Turbo Pump. This model combines a centrifugal submersible pump capable of a maximum pressure of 3450 kPa (500 psi) and a centrifugal recirculation pump with a capacity of 110 L/min. The pump was manually activated and powered by a 2.24 kW (3 hp) motor operating at 230 V. The recirculation ratio was controlled manually by a globe valve which also controlled the pressure in the system. This pressure was measured with an analogic pressure gauge placed between the valve and the outlet of the membrane vessels.

The system included 2 high-pressure resistant stainless steel vessels to hold the 4"x40" spiral-wound membranes. The vessels were connected as a series allowing the system to operate with one or two membranes.

Two operation modes were possible with the system: single pass mode, where the permeate and the concentrate are both collected individually at the system output, or recirculation mode,

where the concentrate is returned to the feed tank. To operate the system in recirculation mode, a heat exchanger was installed on the concentrate conduit in order to maintain the feed at a constant temperature. A custom-made shell and tube heat exchanger was assembled. Tap water was used as coolant and the overall heat-transfer coefficient of the heat exchanger was evaluated at  $430 \text{ W/m}^2\text{*}^\circ\text{C}$ .

Temperature in the system was monitored with type T thermocouples. Data acquisition was done by an OM-CP-OCTTEMP-A data logger from OMEGA. The permeate and the concentrate flows were measured with analog in-line flow meters. Electrical conductivity was measured using an OAKTON Con 6 Acorn Series conductimeter.

#### 2.4. Synthetic saline water

Both lab and pilot-scale tests were performed using a synthetic saline wastewater imitating the process water produced during saline soil treatment. The synthetic water was prepared using the following salts bought in bulk: Calcium chloride ( $\text{CaCl}_2$ ), Magnesium chloride ( $\text{MgCl}_2$ ), Sodium chloride ( $\text{NaCl}$ ), Sodium bicarbonate ( $\text{NaHCO}_3$ ), Sodium sulfate ( $\text{Na}_2\text{SO}_4$ ), Potassium carbonate ( $\text{K}_2\text{CO}_3$ ), Barium chloride ( $\text{BaCl}_2$ ), Strontium chloride ( $\text{SrCl}_2$ ) and Manganese chloride ( $\text{MnCl}_2$ ). The analytical results for the characterization of the process water as well as for the synthetic saline wastewater are presented in Table 1.

*Table 1 Process water and synthetic saline wastewater characterization*

Parameter	(units)	Process water*	Synthetic water
Barium (Ba)	mg/L	6	2
Calcium (Ca)	mg/L	1430	1303
Magnesium (Mg)	mg/L	250	239
Manganese (Mn)	mg/L	20	17
Potassium (K)	mg/L	60	31
Sodium (Na)	mg/L	3530	3539
Strontium (Sr)	mg/L	30	45
Chloride (Cl)	mg/L	7650	6865
Sulfates ( $\text{SO}_4$ )	mg/L	420	377
Conductivity	mS/cm	23	20
Total Dissolved Solids (TDS)	mg/L	14000	13629
Total Hardness ( $\text{CaCO}_3$ )	mg/L	6600	4200
pH		7,31	7,84

\*: Analysis done by Maxxam Laboratory, an accredited analytical laboratory

The composition profiles of both solutions were similar enough to perform bench and pilot-scale tests with the synthetic saline wastewater.

#### 2.5. Experimental procedures

##### 2.5.1. Membrane characterization

A membrane characterization was performed in order to evaluate the membrane's transport parameters and to assess any changes that could occur due to fouling and/or physical modification (deterioration, compaction, deformation). In this study, membrane

characterization was done in two filtration steps: a clean-water test and a test with a known concentration of charged solute [11].

The clean-water test provided the data used to calculate the intrinsic water permeation coefficient of the membrane ( $k_w$ ) defined as:

$$k_w = \frac{Q_p}{A * (\Delta P - \Delta \pi)} \quad \text{Eq.1}$$

Where  $Q_p$  corresponds to permeate flow,  $A$  is the area of the membrane,  $\Delta P$  is the transmembrane pressure and  $\Delta \pi$  is the difference in osmotic pressure between the feed and the permeate. Since demineralized water is used as feed solution for the clean-water test,  $\Delta \pi$  is equal to 0.

The lab-scale clean-water test was performed with 4 L of demineralized water while the pilot-scale clean-water test was performed with 80 L of tap water. In both cases, the pressure was set manually at 689 kPa (100 psi) and incremented by 344 kPa (50 psi) until 1379 kPa (200 psi). For the lab-scale test, permeate flow was estimated by collecting samples of permeate for each increment at the outlet of the cell over 5 minutes. For the pilot-scale test, permeate flow was directly read on the unit.

The rejection capability of a membrane was evaluated by performing a separation test with a 2g/L NaCl solution. This test provided data to evaluate the rejection capacity of monovalent ions and charged species. Membrane rejection capacity ( $R_i$ ), which corresponds to the percent reduction of each target solute ( $i$ ) concentration, is defined as [12]:

$$R_i = \left(1 - \frac{C_{pi}}{C_{fi}}\right) * 100 \quad \text{Eq.2}$$

Where  $C_i$  is the solute concentration and  $p$  and  $f$  denote permeate and feed respectively.

The method to perform the test with a 2 g/L NaCl solution was the same as the clean-water test with the exception that the feed was 4 L of a 2 g/L NaCl solution for the lab-scale test and 80 L of a 2 g/L NaCl solution for the pilot-scale test. Samples were taken at each pressure increment for the measurement of conductivity. The concentration of solutes was determined via conductivity measurements using a pre-established TDS versus conductivity relationship for NaCl.

### 2.5.2. Determining the effect of CP

Intrinsic membrane permeability ( $J_w$ ) in reverse osmosis is described by the solution diffusion model [13]. According to this model, water flux depends on the hydraulic pressure applied on the feed side ( $\Delta P$ ), the transmembrane osmotic pressure difference ( $\Delta \pi_m$ ), and the water permeation coefficient of the membrane ( $k_w$ ), as shown in Eq. 3.

$$J_w = k_w * [\Delta P - \Delta \pi_m] \quad \text{Eq.3}$$

The transmembrane osmotic pressure is described as the difference in osmotic pressure between the permeate and the concentrate side of the membrane at the membrane surface.  $\Delta\pi_m$  is given by the Eq. 2.

$$\Delta\pi_m = f_{os}(C_b - C_p) \quad \text{Eq.4}$$

Where  $C_b$  and  $C_p$  are the solute concentration on the concentrate and permeate side of the membrane respectively and  $f_{os}$  is the osmotic pressure factor available through first-principle calculations.

Eq.2 is valid only for an ideal situation and does not take concentration polarization (CP) into account. If considering CP when determining transport parameters for a membrane,  $C_b$  in Eq.2 must be replaced by the solute concentration at the membrane surface ( $C_w$ ). To avoid having to measure the solute concentration at the membrane surface during the experiments,  $C_w$  was assumed to be linearly proportional to  $C_b$ . The calculation of  $\Delta\pi_m$  when taking into account CP at the membrane surface is shown in Eq. 3.

$$\Delta\pi_m = f_{os}[\alpha C_b - C_p] \quad \text{Eq.5}$$

Where  $\alpha$  is a constant linking the bulk solute concentration to the membrane surface solute concentration.  $\alpha$  will vary in relation to the CP. Eq. 3 can be rewritten in terms of osmotic pressure, resulting in:

$$\Delta\pi_m = \alpha\pi_b - \pi_p \quad \text{Eq. 6}$$

Where  $\pi_b$  and  $\pi_p$  are the osmotic pressure of the concentrate and the permeate respectively. The permeate osmotic pressure is typically a minimum of two order of magnitude lower than the feed osmotic pressure. Thus, the permeate-side osmotic pressure can be neglected. Inserting Eq. 4 in the flux equation (Eq.1), we obtain the following equation:

$$J_w = k_w * [\Delta P - \alpha\pi_b] \quad \text{Eq.7}$$

According to the film theory, solutes accumulate at the membrane surface, thus increasing the osmotic pressure that need to be compensated for by the hydraulic pressure in order to obtain a water flux across the membrane. The  $\alpha$  factor serves to quantify this accumulation of solutes. In a case where no CP occurs,  $\alpha$  is equal to 1, meaning that the solute concentration at the membrane surface is the same as the solute concentration in the bulk. When CP does occur,  $\alpha$  increases, resulting in a lower difference in pressure across the membrane. Therefore, the greater the effect of CP on the membrane, the higher the value of  $\alpha$ .

Rearranging Eq. 7 to solve for  $\alpha$ , we obtain the following equation:

$$\frac{1}{\pi_b} \left( \Delta P - \frac{J_w}{k_w} \right) = \alpha \quad \text{Eq.8}$$

In this study, the comparison of the effect of CP between the bench and pilot-scale tests was based on the calculated  $\alpha$  for both RO units.

Eq. 8 is solve through two experimental tests. At first, the water permeation coefficient ( $k_w$ ) is obtained from the clean water test during the membrane characterization. The  $k_w$  value used for the calculations is the average  $k_w$  obtained from each pressure of characterization.

Then,  $\Delta P$ ,  $J_w$  and  $\pi_b$  were obtained from the treatment tests described at section 2.5.4. In order to estimate the permeate flow, samples of permeate were collected every 15 minutes at the outlet of the cell over 5 minutes. In order to measure  $\pi_b$ , conductivity in the feed reservoir was also measured every 15 minutes.

### 2.5.3. Osmotic pressure calculation and temperature correction

The evaluation of the transport parameters requires an accurate measurement of the concentrate and permeate osmotic pressures. Ladner and *al.* collected empirical values for seawater osmotic pressure from the literature and plotted them in order to obtain an equation linking the total dissolved solids (TDS) to the osmotic pressure. Eq. 9 was obtained for TDS concentrations ranging from 10 000 to 80 000 parts per million (ppm) [11].

$$\pi_b = 1.416 * 10^{-7} c_b^2 + 6.913 * 10^{-2} c_b - 80.64 \quad \text{Eq.9}$$

Where  $\pi_b$  is the bulk osmotic pressure in kPa and  $c_b$  is the bulk TDS in ppm.

Since the synthetic water produced for this study was similar to seawater, and its TDS concentration varied between 15 000 to 35 000 ppm, this equation was used to calculate the osmotic pressure.

Since all tests were not performed at the exact same temperature, measurement of water flux across the membrane had to be normalized with a reference temperature. The corrected water flux ( $J_{w,c}$ ) was calculated using Eq. 10.

$$J_{w,c} = J_w * TCF \quad \text{Eq. 10}$$

Where TCF is the correction factor, which is calculated using Eq. 11.

$$TCF = 0.025 T^2 - 0.1457 T + 3.1605 \quad \text{Eq. 11}$$

Where T is the temperature in °C. Eq. 11 was obtained from empirical data used by the industry [14].

### 2.5.4. Lab-scale and pilot-scale treatment tests

The bench-scale RO unit was initially rinsed with 4 L of demineralized water for 10 minutes with no hydraulic pressure applied. 2.5 L of synthetic saline wastewater was then added to the feed water reservoir. The system was initially run with no pressure in order to dilute any dead volume. After recirculating the wastewater, pressure in the system was set to 2068 kPa (300 psi). Permeate was collected in a 2 L graduated cylinder and the concentrate was returned to the feed water reservoir. Pressure was manually increased during the experiments in order to maintain a constant permeate flow across the membrane. The system was shut down when the pressure reached 3447 kPa (500 psi). For each experiment, three 50 mL water samples were



collected for ion analysis. These samples were collected in 1) the feed reservoir after the initial recirculation, 2) the feed reservoir at the end of the filtration experiment, and 3) the graduated cylinder containing the collected permeate.

Two operation modes were tested with the pilot-scale RO unit. To compare the pilot-scale unit to the bench-scale unit, the pilot-scale unit was first operated with the concentrate recirculated following the same protocol as the bench-scale tests with few modifications; the system was rinsed with 80 L of tap water, and 1000 L of synthetic saline wastewater was added to the feed water reservoir. Permeate was collected in a 1 m<sup>3</sup> reservoir. Since the pressure limit of the system was 3100 kPa (450 psi), the system was shut down when pressure reached 2930 kPa (425 psi). Samples were collected following the bench-scale test protocol.

The second operation mode tested was a single-pass mode with no recirculation. The rinsing protocol and the volume of treated water was the same as for the previous operation mode. After recirculating the wastewater, pressure in the system was set at 2760 kPa (400 psi) and kept constant for the duration of the experiment. Permeate and concentrate were collected individually in 1 m<sup>3</sup> reservoirs. The system was shut down when the feed water reservoir was empty. For each experiment, three 50 mL water samples were collected for ion analysis. These samples were collected in 1) the feed reservoir, 2) the permeate reservoir at the end of the filtration experiment, and 3) the concentrate reservoir at the end of the filtration experiment.

#### 2.5.5. Membrane cleaning procedure

Treatment tests were followed by a membrane cleaning procedure, performed in two steps. First, for the bench-scale and the pilot-scale unit respectively, a 2 L and a 100 L HCl solution with a pH of 2, was circulated in the system at a flow of 3 L/min for 30 minutes. Then, a clean-water test was performed following the method described at section 2.5.1. in order to assess any change of the membrane performance.

#### 2.6. Analytical methods

For the characterization of the process water and the analysis of the barium and strontium ions, samples were sent to Maxxam Laboratory, an accredited analytical laboratory. All the samples collected during the experiments were analyzed at *Centre des Technologies de l'Eau* (CTE) according to the following methods: concentrations of calcium, sodium, manganese and potassium ions were measured by flame atomic absorption spectroscopy, Standard Method #3111, using a Shimadzu AA-7000 instrument. Total dissolved solids were measured following the Standard Method #2540C (total dissolved solids dried at 180°C). Sulfate concentrations were measured by turbidimetry following the Standard Method #4500E. The apparatus used was a DR-2700 in single signal mode set at 420 nm. Chloride concentrations were measured by titrimetry following the Standard Method #4500 B, pages 4-70, 4-71. Total hardness was also measured by titrimetry following the Standard Method 2320B, pages 2-27, 2-28. For all conductivity measurements performed in the analytical laboratory, an Accumet xl500 conductimeter with an Accumet 13-620-100 probe was used. Total organic carbon (TOC) was

measured by high-temperature combustion following the Standard Method #5310B. The apparatus used was a Shimadzu TOC-L model CPN.[15]

### **3. Results and discussion**

#### **3.1. Comparison between bench and pilot-scale process saline water concentration test results**

In order to compare the bench to the pilot-scale tests, 3 treatment tests with 2.5 L of synthetic saline wastewater and 4 treatment tests with 1000 L of synthetic saline wastewater were performed with the bench-scale and the pilot-scale RO units respectively. For the bench-scale unit, the ratio of water treated to membrane area was 154 L/m<sup>2</sup> while for the pilot-scale unit this ratio was 143 L/m<sup>2</sup>. For both units, each treatment test was done in recirculation mode where the concentrate is returned to the feed tank. Pressure was adjusted manually during the treatment tests in order to maintain a constant permeate flow. The same membrane coupon and spiral-wound membrane was used for all treatment tests.

The comparison was done on 4 key elements: the water recovery capacity, the transport parameters, the salt rejection and the fouling of the membrane. Each of these elements are discussed in the following sections.

##### **3.1.1. Water recovery capacity**

The RO technology was studied in order to assess its potential application for the concentration and reuse of process saline wastewater. This technology was selected among others, because of its capacity to achieve a high concentration factor thereby reducing the concentrate volume. According to previous case studies, in which RO was used to concentrate saline water with similar characteristics to this study's process saline wastewater, RO technology could achieve water recovery capacities ranging from 24% to 75% with salt rejection rates between 89.9% and 99.3% [3-5, 16, 17]. Water recovery capacities obtained for each treatment experiment as well as the average recovery for both bench and pilot-scale tests are presented at Table 4.

*Table 2 Water recovery capacity the of bench-scale and the pilot-scale RO unit*

	<u>Bench-scale test</u>	<u>Pilot-scale test</u>
Treatment #1	69%	56%
Treatment #2	72%	55%
Treatment #3	72%	57%
Treatment #4	-	55%
Average	71%	56%

The average water recovery capacity for the pilot-scale tests was 56%, 15% lower than the 71% recovery capacity of the bench-scale tests. Recovery rates obtained in the pilot-scale tests were comparable to what was observed by Kelkar and *al.*, who obtained recovery capacities of 50% and 55% using RO units with a 3.8 m<sup>3</sup>/day and 5.7 m<sup>3</sup>/day capacity respectively [18]. The difference observed between the bench and field-scale tests is mostly due to the difference of

applied pressure on both systems since the flow conditions were similar with both units. During bench-scale tests, the final applied pressure was 18% higher than during the pilot-scale tests. During the bench-scale tests, a higher pressure difference between the feed osmotic pressure and the applied pressure was maintained, resulting in a higher permeate flux and a higher water recovery rate.

### 3.1.2. Transport parameters

Experiments were performed with a constant permeate flux by varying the pressure to measure the  $\alpha$  factor using Eq. 8. Representative data from a constant flux experiment with both the bench and pilot-scale unit are shown in Fig. 2. Permeate was collected and the concentrate was returned to the feed tank to allow the feed side's osmotic pressure to increase over time. Pressure was adjusted manually after each flow measurement in order to maintain a constant permeate flux.

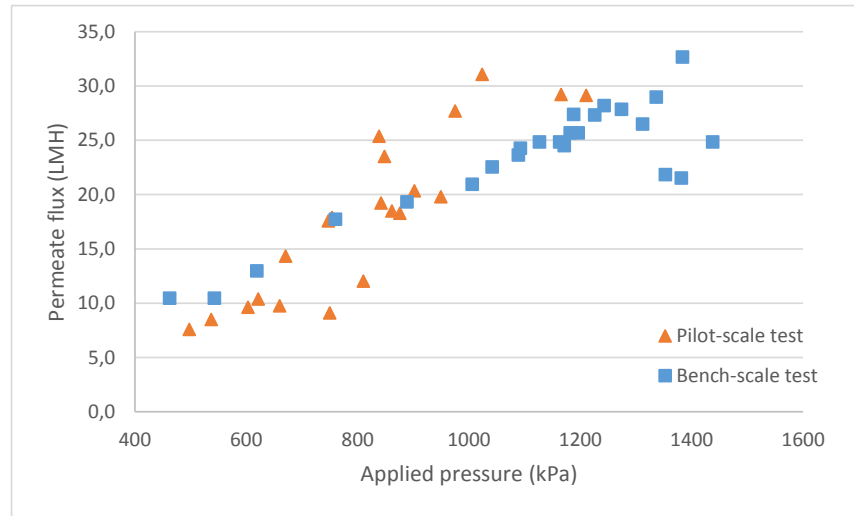


Fig. 2 Representative data showing the results from a bench-scale and a pilot scale experiment for a constant-flux experiment

Synthesized data for the 3 bench-scale experiments and the 4 pilot-scale experiments are summarized in Table 3. Tests were all performed with a BW30 membrane from Filmtec.

Table 3 Synthesized data for the bench-scale and the pilot-scale constant flux experiments. Experiments were done with BW30 membrane from Filmtec

	Bench-scale test		Pilot-scale test	
	Water permeation coefficient ( $k_w$ ) (LMH/kPa)	$\alpha$ factor	Water permeation coefficient ( $k_w$ ) (LMH/kPa)	$\alpha$ factor
Clean-water test	0,031	1	0,042	1
Treatment #1	0,021	1,26	0,018	1,29
Treatment #2	0,022	1,20	0,015	1,26
Treatment #3	0,025	1,14	0,014	1,27
Treatment #4	-		0,019	1,30
Average (treatment tests)	0,023	1,20	0,017	1,28

For both units, the flow condition at the membrane surface was similar with an estimated tangential flow of 0.5 m/s. However, differences in transport parameters were observed.

The water permeation coefficient measured during the clean-water test with the pilot-scale unit was 26% higher compared to the bench-scale unit. Conversely, the water permeation coefficient measured during treatment tests was 26% lower in average with the pilot-scale unit compared to the bench-scale unit. The difference observed between the treatment tests can be explained by the significant variation in the permeate flux measured at different pressures with the pilot-scale unit at Fig. 2. In addition, membrane suppliers also warn membrane users that performances can vary up to 30% between different membrane batches. However, the difference observed between the results from the clean-water tests and the treatment tests suggest that CP occurs with both units. With the bench-scale unit, the water permeation coefficient decreased from 0.031 to 0.023 LMH/kPa while for the pilot-scale unit, it decreased from 0.042 to 0.017 LMH/kPa. These differences suggest that concentration polarization was more important for the pilot-scale unit.

As seen in Eq. 8, the  $\alpha$  factor is an indicator of the importance of concentration polarization on the membrane performance by linking the bulk solute concentration to the membrane surface solute concentration. The higher the  $\alpha$  value, the greater the effect of CP. The  $\alpha$  factors calculated for the pilot-scale unit are higher than the bench-scale unit, confirming that CP had more effect with the spiral-wound module. Since both units had the same operating conditions, this difference is probably due to membrane's different configuration and normal difference in performances caused by the fabrication process.

### 3.1.3. Membrane rejection capacity

Membrane rejection capacities for the different ions present in the solution were assessed for both bench and pilot-scale experiments. The lowest, highest and average calculated rejection capacities are presented in Table 4. The average was calculated over the 3 treatment tests for the bench-scale experiments and over the 4 treatment tests for the pilot-scale experiments.

Table 4 Membrane rejection capacity for ions in solution during the bench-scale and pilot-scale experiments

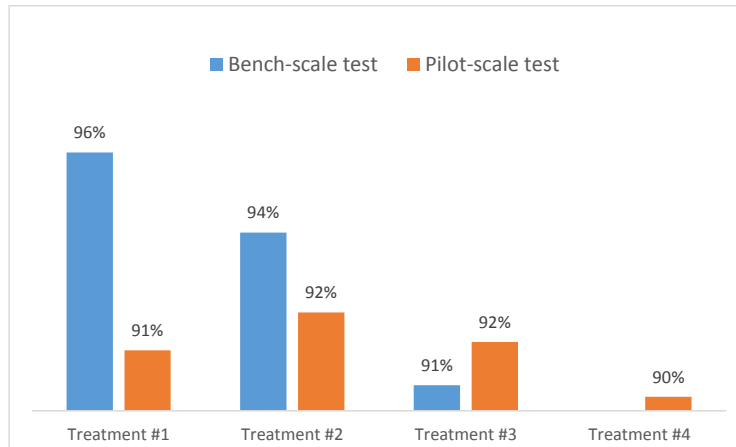
	Bench-scale test			Pilot-scale test		
	Rejection (R <sub>i</sub> )			Rejection (R <sub>i</sub> )		
	Lowest	highest	average	Lowest	highest	average
Sulfates (SO <sub>4</sub> )	94,3	99,9	98,2	96,6	97,6	97,2
Sodium (Na)	87,7	94,3	91,0	87,3	94,8	91,1
Calcium (Ca)	94,5	99,7	97,4	95,6	96,1	95,9
Magnesium (Mg)	94,1	99,7	97,2	96,6	97,1	96,8
Potassium (K)	ND	ND	ND	79,3	87,3	85,0
Manganese (Mn)	NA	NA	NA	97,4	97,7	97,6
Chloride (Cl)	90,0	96,3	93,5	90,8	94,3	93,2
Barium (Ba)	NA	NA	NA	94,3	98,3	96,9
Strontium (Sr)	NA	NA	NA	97,0	97,1	97,0
Total solids	90,6	96,4	93,8	90,3	92,4	91,5

NA: Not analyzed

ND: Not detected

The average salt rejection (total solids rejection) for all bench-scale tests was 93.8% with the lowest being 90.6% and the highest being 96.4%. The average salt rejection for all pilot-scale tests was slightly lower at 91.5%. However, the pilot-scale tests' lowest value was almost equal to that of the bench-scale test, at 90.3%. When comparing the salt rejection for each ion individually, no significant difference was observed. The largest difference observed was for the calcium, for which the pilot-scale tests had a rejection rate of 95.9%, 1.5% lower than what was observed during the bench-scale tests. As expected with membrane filtration systems, rejection capacity was higher for divalent ions than for monovalent ions. The lowest average rejection capacity observed was for sodium, with values of 91% and 91.1% for the bench and pilot-scale experiments respectively.

In order to evaluate the consistency of the rejection capacity of the membrane, the total solids rejection rates were plotted for each of the treatment tests performed. The data is presented on Fig. 3.



*Fig. 3 Membrane rejection capacity for the total solids measured for each treatment test during bench-scale and pilot-scale tests*

Compared to rejection capacities calculated during the bench-scale tests, those obtained during the pilot-scale tests are more constant, with values varying between 90.3% and 92.4% over 4 treatment tests. This difference could be due to several factors, such as the membrane conservation and the membrane compaction. Membrane coupons were initially cut with a saw from a spiral-wound membrane and then stored directly in a 1.5% sodium metabisulfite solution for a few weeks before the experiments. A study reported that flux and rejection can be affected by skin shrinkage due to the contact of the membrane with a salt solution [19]. Since the spiral-wound membrane used during the pilot-scale experiment was used immediately upon reception, skin shrinkage would not be an issue. However, since the membrane coupon used in the bench-scale unit was conserved in a sodium metabisulfite solution, this could explain the difference in salt rejection between the two. The difference could also be due to a greater compaction of the membrane coupon with the bench-scale set-up compared to with the spiral-wound membrane, particularly since the pressure applied on the membrane coupon was 689 kPa higher than the pressure applied on the spiral-wound membrane.

#### 3.1.4. Membrane fouling

Since fouling is one of the major drawbacks of filtration technologies, the fouling of the BW30 membrane was assessed during this study. Fig. 4a and Fig. 4b illustrate the permeate flows in the clean-water tests for the bench and pilot-scale experiments respectively. For the bench-scale tests, the clean-water test was performed with deionised water at a pressure of 2760 kPa (400 psi) after each treatment and cleaning. For the pilot-scale tests, the clean-water test was performed with tap water at a pressure of 2415 kPa (350 psi) after each treatment and cleaning. Since the synthetic solutions prepared for the treatment tests only contained mineral ions, HCl was selected for membrane cleaning. The acid cleaning was done after the last treatment test with an HCl solution at a pH of 2. The initial permeate flows measured for the bench and the pilot-scale experiments were 80 LMH and 49 LMH respectively.

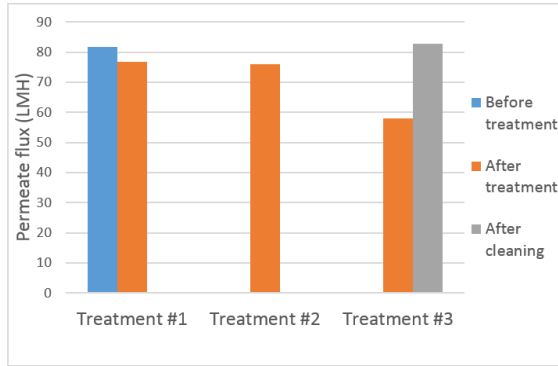


Fig. 4a

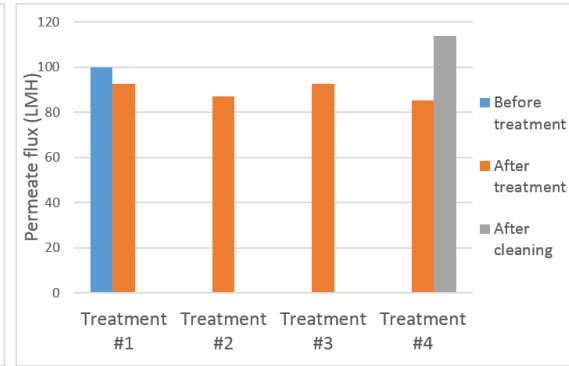


Fig. 4b

Fig. 4a) Permeate flux measurements for bench-scale clean-water tests, 4b) Permeate flux measurements for pilot-scale clean-water tests

For the bench-scale experiments, the permeate flow decreased to 58 LMH after 3 treatments, corresponding to an overall reduction of 29% in permeate flow. For the pilot-scale experiments, the permeate flow decreased to 85 LMH after 4 treatments, corresponding to an overall reduction of 15% in permeate flow. In addition to a smaller decline in flux, the flow reduction pattern during the pilot-scale experiment was different from the bench-scale experiment. A significant flow reduction of 7% was observed after the first treatment followed by a reduction of 8% for the 3 following treatments.

In both cases, the HCl membrane cleaning allowed the membrane to recover a permeate flow higher than its initial value. In order to evaluate the effect of cleaning on membrane performance, the membrane used for the treatment tests was characterized before and after the cleaning. Results of the clean-water permeability measurements as well as for the rejection tests for both bench and pilot-scale tests are presented at Table 5.

Table 5 NaCl rejection tests results for the BW-30 membrane before and after cleaning using a HCl solution for bench-scale and pilot-scale tests.

<u>Operating pressure</u>	<u>Before treatments</u>	<u>After treatments</u>	<u>Difference</u>
kPa (psi)			
	Bench-scale tests - Rejection (%)		
689 (100)	98,2%	94,7%	-3,5%
1033 (150)	98,7%	95,9%	-2,8%
1379 (200)	99,1%	97,1%	-2,0%
	Pilot-scale tests - Rejection (%)		
689 (100)	94,3%	94,1%	-0,2%
1033 (150)	96,9%	96,4%	-0,5%
1379 (200)	97,1%	97,0%	-0,1%

In both cases, rejection capacities were higher when hydraulic pressure was increased since more water flowed through the membrane with approximately the same amount of salt [10]. For the bench-scale tests, a significant difference in rejection capacities was noted after the cleaning of the membrane. At Fig. 3, we observe a reduction of 7% in rejection capacities

between the first and the third treatment test. However, this is not due to the cleaning procedure since a similar loss in rejection capacity was observed during the treatment tests. For the pilot-scale tests, the loss in rejection capacities was almost negligible. The results in Table 5 are then consistent with what was observed on Fig. 1.

The assessment of the membrane fouling shows that mineral fouling that occurs during the treatment of the synthetic saline wastewater is reversible. The cleaning method using an HCl solution returned the membrane's clean-water permeability to its initial value for both bench and pilot-scale tests. In terms of salt permeability, a loss of rejection capacity of 2% to 3.5% for the membrane coupon suggests that it was affected by the cleaning. However, this loss was also observed during the treatment tests, which means that it could also be due to membrane shrinkage as described in section 3.1.3. For the spiral-wound membrane, no important loss in rejection capacity was observed. These results suggest that the cleaning method using an HCl solution is efficient and has no negative impact on the spiral-wound membrane performances.

### 3.2. Evaluation of the optimal operation mode

The pilot-scale RO unit was operated in two different modes in order to find which could achieve the optimal results. The two modes were the single-pass mode and the recirculation mode. With the single-pass mode, the applied pressure was initially set at 2769 kPa (400 psi) and maintained constant for the duration of the treatment tests. The particularity of this operation mode is that the feed water's osmotic pressure is constant and the system operates at maximum capacity for the duration of the treatment. It is important to note that water was still recycled in the system through the Double Turbo Pump. With the recirculation mode, pressure was initially set at 2068 kPa (300 psi) and then increased until 2769 kPa (400 psi) in order to maintain a constant permeate flow rate. With the recirculation mode, the feed water's osmotic pressure increased during the treatment test.

For each operation mode, 4 treatment tests were performed with 1000L of synthetic saline wastewater. As presented in Table 4, the average water recovery for the recirculation mode was 56% while an average recovery of 60% was obtained in the single-pass mode. The salt rejection capacity of the membrane as well as the permeate flux obtained for both operation modes for each treatment test are presented at Fig. 5a and 5b respectively.



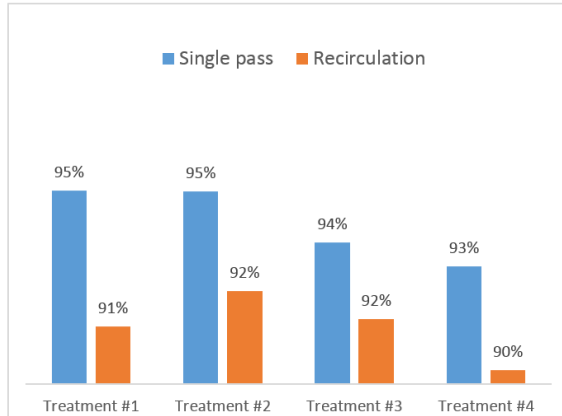


Fig. 5a

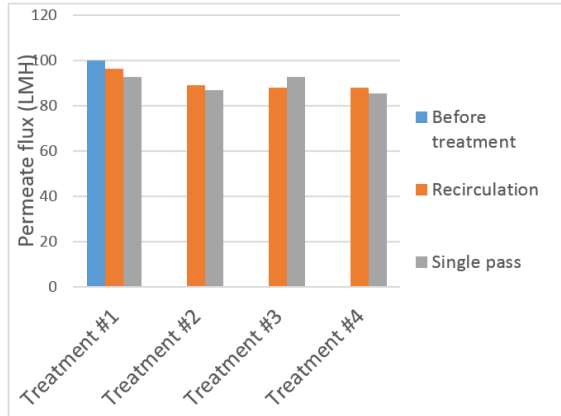


Fig. 5b

Fig. 5a) Salt rejection capacities measured during each treatment tests for both single pass and recirculation operation mode of the pilot-scale RO unit, b) Permeate flux measurements after each treatment tests for both single pass and recirculation operation mode of the pilot-scale RO unit

Fig. 5a presents the total solids rejection for each of the treatment tests. For both operation modes, the rejection capacities follow the same trend. However, the salt rejection capacity of the membrane in single-pass mode was an average of 3% higher than in recirculation mode for each treatment tests. These results suggest that the higher feed concentrations in the recirculation mode have a negative impact on the rejection capacity of the membrane.

Fig. 5b presents the permeate flux obtained from the clean-water test performed with tap water with a pressure of 2415 kPa (350 psi) for both operation modes. As with the rejection capacity, the trend is similar for both modes. However, the permeate flux after the fourth treatment is 4% higher in the single-pass mode with a value of 88 LMH compared to the recirculation mode's 84 LMH, suggesting that the single-pass mode is less prone to fouling.

Even if there is no major difference between the two operation modes, the single-pass mode has a higher water recovery capacity by 4%, a higher average salt rejection by 3% and results from the clean-water test suggest that it can maintain a permeate flux 8% higher after 4 treatments than the recirculation mode.

#### 4. Conclusion

The bench-scale tests demonstrated that reverse osmosis is a suitable and effective technology for the concentration and reuse of saline wastewater produced by the saline soil treatment process. Data collected during the bench-scale tests were utilized in order to perform pilot-scale tests with the same parameters. In this study, data obtained from the pilot-scale test were compared to those from the bench-scale tests in order to evaluate the relevance and usefulness of bench-scale RO studies in the determination of full-scale membrane performance. The comparison between the bench and pilot-scale test was based on the water recovery capacity, the transport parameters, the salt rejection and the fouling of the membrane.

The bench-scale tests exhibited an average recovery of 71% with the BW-30 membrane from Filtmtec while the pilot-scale tests exhibited an average recovery of 56% with the same membrane. Since the pressure limit of the pilot-scale system was lower, 2930 kPa compared to the bench-scale unit's 3447 kPa, it was concluded that this difference in operating pressure was responsible for the 15% difference in water recovery. The average rejection capacities calculated for each element in the solutions with either unit were similar. After the third treatment, the overall rejection capacities for the dissolved solids were the same for the bench and pilot-scale unit.

In terms of the transport parameters, it was found that the membrane coupon could have a water permeation coefficient difference as high as 26% compared to the spiral-wound membrane. Results also suggest that spiral-wound membranes are more influenced by CP than membrane coupons. The  $\alpha$  factor calculated for the bench and pilots-unit were 1.20 and 1.26 respectively.

Finally, the fouling tests showed that bench and pilot-scale tests present different fouling behaviours. During bench-scale tests, fouling occurred at a constant rate for each treatment test. However, fouling occurred primarily during the first treatment test during pilot-scale tests. Nevertheless, they both showed a similar response to the HCl cleaning procedure by recovering their full performance after the cleaning.

This study leads to the conclusion that bench-scale RO tests has to be performed before a pilot-scale test in order to accurately evaluate certain parameters. We found that results from the salt-rejection tests as well as water recovery capacities were the same for both units. Bench-scale studies can also be used to characterize the nature of foulant accumulating at the membrane surface in order to identify the proper cleaning method. However, bench-scale studies neither accurately predict the membrane transport parameters such as the water permeation coefficient nor determine the flux decline that could be potentially caused by foulants. In order to have a better understanding of the factors that cause these differences between the bench and pilot-scale tests, foulant layer should be characterized and a flow simulation comparison should be made for both units used in this study.

In parallel to the bench-scale vs pilot-scale comparison, a comparison between two pilot-scale operation modes was done. The operation with the concentrate returned to the feed tank (recirculation operation) was compared to the operation with no recirculation (single-pass). Results from this comparison lead to the conclusion that the single-pass operation mode offers higher water recovery and salt rejection capacities.

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