

Reverse osmosis applied to soil remediation wastewater: comparison between bench-scale and pilot-scale results

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1. ABSTRACT

Bench-scale reverse osmosis results were compared to pilot-scale results to evaluate the usefulness of bench-scale studies in assessing full-scale membrane performances in the context of reusing wastewater from a novel saline soil remediation technology. Salt rejection and water recovery capacity were accurately estimated by bench-scale tests, with an average water recovery capacity of 56% and a salt rejection of 92% at 2760 kPa. However, fouling, and concentration polarization were found to differ between scales. Concentration polarisation was 41% higher for the pilot-scale tests,

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19 which might be explained by differences in membrane configuration; coupon versus
20 spiral-wound.

21 Highlights:

- 22 - Bench-scale and pilot-scale tests results were obtained in environmentally
23 realistic conditions
- 24 - Water recovery and rejection capacity are accurately measured by bench-scale
25 tests
- 26 - Concentration polarization is underestimated when using a membrane coupon
27 set-up
- 28 - Flux decline due to fouling is lower in bench-scale tests

29 **Keywords:** Reverse osmosis; soil remediation; wastewater; membrane; polarization

30 2. INTRODUCTION

31 Reverse osmosis (RO) was first introduced in the 1950's as a novel filtration technology
32 capable of separating ions from water[1]. Since then, this technology has been utilized
33 around the world for multiple industrial applications, including drinking water
34 purification[2], water and wastewater purification[3] maple syrup production[4],
35 groundwater desalination[5], seawater desalination[6, 7] and many more[8, 9].

36 RO technology is constantly being improved upon, with new market applications being
37 subsequently discovered. For each new application, studies must be conducted in order
38 to evaluate the design parameters of the RO unit used, such as the most suitable

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membrane, the membrane's permeability coefficient, the membrane's rejection capacity, the flux decline that could be potentially caused by foulants and the appropriate cleaning procedure. Usually, these studies are first conducted as bench-scale (using a membrane coupon), and then as pilot- and industrial-scale (with spiral-wound membranes). While the literature dealing with the use of RO for the concentration of saline wastewater contains a wide variety of bench-scale [10-13] and pilot-scale studies [5-9, 14], information regarding the comparison between the bench and pilot-scale of an RO membrane performance are sparse [15]. As mentioned by Ladner & al., the question remains whether the bench-scale studies are useful in the determination of full-scale membrane performances[10].

In the present study, RO was investigated for a new application consisting in process water reuse for an innovative salt contaminated land remediation technology which has recently been patented[16]. 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 offering more efficient salt removal than conventional technologies, this soil remediation technology still produces a large volume of highly saline process wastewater (3 to 40 g/L of total dissolved solids) that is currently disposed of by deep well injection [17]. 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 geographical limits and thus dictate the economical applicability of the technology. One option to extend such geographical limits, is to minimize water usage by combining the new remediation technology with a

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RO unit. Since very little data concerning the concentration and reuse of saline soil treatment wastewater was available in the literature [9], bench-scale, as well as pilot-scale tests were performed in order to determine the RO unit design parameters for this particular application.

2.1. OBJECTIVES AND SCOPE OF WORK

In the present 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 synthetic saline water used in this study did not contain organic matter. Therefore, organic fouling of the membranes is not discussed and the findings presented in this manuscript only apply to effluents with a mineral charge. The main parameters considered for the comparison were the water recovery capacity, the transport parameters and salt rejection capacity, as well as the mineral fouling of the membrane.

3. MATERIAL AND METHODS

3.1. MEMBRANES

Previous tests led to the selection of the BW-30 membrane for the filtration of saline soil treatment wastewater. BW-30 reverse osmosis membranes were obtained from Filmtec (MI, USA) a wholly owned subsidiary of the Dow Chemical Company. Membranes were received as 4 inch spiral wound (7 m² of membrane surface) with fiberglass outer wrap. To get membrane coupons for lab tests, a spiral wound was cut to

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81 fit the bench-scale RO cell. The BW-30 membrane manufacturer's data are presented at
82 Table 1.

83 *Table 1 BW-30 membrane manufacturer's data*

| <u>Operating limits</u> | |
|---|--------------------------------------|
| Membrane type | Polyamide thin-film composite |
| Maximum operating temperature | 45°C |
| Maximum operating pressure | 4100 kPa |
| pH range (operation) | 2 - 11 |
| pH range (cleaning) | 1 - 12 |
| <u>Membrane specifications¹</u> | |
| Membrane permeation | 50 L m ⁻² h ⁻¹ |
| Salt rejection | 99.5 % |

¹: Based on the following conditions: 2000 ppm NaCl, 1550 kPa, 25°C and 15% recovery

84

85 3.2. BENCH-SCALE RO SYSTEM

86 The laboratory set-up was designed as presented in the diagram in Figure 1. The key
87 components were the membrane test cell (1), the high-pressure pump (2) and the feed
88 water reservoir (3).

89 Experiments were conducted using a commercially available bench-scale membrane test
90 cell (Sepa CF, GE Osmonics). The flow channels on each side of the membrane were
91 filled with a mesh spacer to simulate the hydrodynamics of a spiral-wound membrane
92 element [11]. These mesh spacers were cut directly from the spiral wound. Feed water
93 was circulated on the active layer side of the membrane through ten round 4.7 mm
94 diameter openings. The effective surface area of the membrane was 149 cm² (10 cm
95 wide and 15 cm long with rounded corners). Permeate was then collected through
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another ten round 4.7 mm diameter openings located in the center of the membrane coupon.

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 analog pressure gauge.

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

The permeate flow was calculated by collecting a new sample at the outlet of the cell every 10 minutes. The volume of the sample was measured with a 50 mL graduated cylinder and recorded. The electrical conductivity of the sample, and the recirculated feed, was measured using a Thermo Scientific Orion 013005MD conductimeter.

3.3. PILOT-SCALE RO SYSTEM

Pilot-scale experiments were conducted using a small-scale commercial RO unit (Turbo Compak, Darveau) designed as presented in the diagram in Figure 2. The key components were the feed pump (1), the pre-treatment system (2), the high-pressure dual pump (3), the RO membrane (4), the heat exchanger (5) and the feed water reservoir (6).

116 Water was supplied to the system by a 0.37 kW (1/2 hp) peristaltic pump. Feed water
117 was pretreated through a two-step filtration system: a washable 24 μm mesh filter and
118 a 5 μm disposable cartridge filter. Filter fouling was monitored with 2 analog pressure
119 gauges located upstream and downstream of the filters. When the pressure difference
120 was over 70 kPa (10 psi), the mesh filter was washed and the cartridge filter was
121 replaced.

122 The high-pressure dual pump combines a multi-stage centrifugal submersible pump
123 capable of a maximum pressure of 3105 kPa (450 psi) to a centrifugal recirculation
124 pump with a capacity of 110 L/min. The pump was manually activated and powered by a
125 2.24 kW (3 hp) motor operating at 230 V. The recirculation ratio was controlled
126 manually by a globe valve, which also controlled the pressure in the system. This
127 pressure was measured with an analog pressure gauge located at the outlet of the
128 membrane.

129 In order to operate the system in recirculation mode as shown at Figure 2, a heat
130 exchanger was installed on the concentrate conduit in order to maintain the feed at a
131 constant temperature. A custom-made shell and tube heat exchanger was assembled.
132 Tap water was used as coolant and the overall heat-transfer coefficient of the heat
133 exchanger was evaluated at $430 \text{ W/m}^2 \cdot ^\circ\text{C}$.

134 Temperature in the system was monitored with type T thermocouples. Data acquisition
135 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.

3.4. SYNTHETIC SALINE WASTEWATER

Due to the limited quantity and the associated procurement cost of saline soil treatment process wastewater at the time of these experiments, both lab- and pilot-scale tests were performed using synthetic saline wastewater imitating the process wastewater. The composition of this synthetic wastewater was based on the composition of the lixiviates produced during the treatment of salt impacted soils from an oil and gas associated facility in Alberta, Canada. The following salts bought in bulk from LabMat were used: Calcium chloride (CaCl_2), Magnesium chloride (MgCl_2), Sodium chloride (NaCl), Sodium bicarbonate (NaHCO_3), Sodium sulfate (Na_2SO_4), Potassium carbonate (K_2CO_3), Barium chloride (BaCl_2), Strontium chloride (SrCl_2) and Manganese chloride (MnCl_2). These salts were of a laboratory grade. The analytical results for the characterization of the process water as well as for the synthetic wastewater are presented in Table 2.

Table 2 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 |

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| | | | |
|-------------------------------------|-------|-------|-------|
| Strontium (Sr) | mg/L | 30 | 45 |
| Chloride (Cl) | mg/L | 7650 | 6865 |
| Sulfates (SO ₄) | mg/L | 420 | 377 |
| Conductivity | mS/cm | 23 | 20 |
| Total Dissolved Solids (TDS) | mg/L | 14000 | 13629 |
| Total Hardness (CaCO ₃) | mg/L | 6600 | 4200 |
| Total Organic Carbon (TOC) | mg/L | 10,1 | 0 |
| pH | | 7,31 | 7,84 |

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

Due to the low concentration and the complex nature of the TOC, no organic matter was added to the synthetic wastewater. Therefore, organic fouling was not assessed in this study. If the real wastewater was used instead, the presence of 10.1 mg/L of organic carbons could have caused organic fouling of the membrane on the long term. However, considering the volumes used for each treatment sequence (approximately 150 L/m² of membrane surface), this potential fouling would have been negligible during this experiment. The composition profiles of both solutions were similar enough to perform bench and pilot-scale tests with the synthetic wastewater.

3.5. EXPERIMENTAL PROCEDURES

3.5.1. 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 the present study, membrane characterization was done using two different conditions: a clean-water test and a test with a known concentration of charged solute [10].

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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, ΔP 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 dechlorinated tap water. In both cases, the tests were performed at 1380, 2070 and 2760 kPa.

The rejection capacity of the 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[18]:

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

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-

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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.

3.5.2. DETERMINING THE EFFECT OF CONCENTRATION POLARIZATION

Membrane permeability (J_w) in reverse osmosis is described by the solution diffusion model [19]. According to this model, water flux depends on the hydraulic pressure applied on the feed side (ΔP), 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. 4.

$$\Delta\pi_m = \pi_b - \pi_p \quad \text{Eq. 4.}$$

Where π_b and π_p are the osmotic pressure of the concentrate and the permeate respectively

.

Eq. 4. 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, π_b in Eq. 4. must be replaced by the osmotic pressure at the membrane surface (π_w). To avoid having to measure the solute concentration at the membrane

surface during the experiments, π_w was assumed to be linearly proportional to π_b . The calculation of $\Delta\pi_m$ when taking into account CP at the membrane surface is shown in Eq. 5.

$$\Delta\pi_m = \alpha\pi_b - \pi_p \quad \text{Eq. 5.}$$

Where α is a constant linking the bulk solute concentration to the membrane surface solute concentration. α will vary in relation to the CP. The permeate osmotic pressure is typically a minimum of two orders of magnitude lower than the feed osmotic pressure. Thus, the permeate-side osmotic pressure can be neglected. Inserting Eq. 5. in the flux equation (Eq. 1.), we obtain the following equation:

$$J_w = k_w[\Delta P - \alpha\pi_b] \quad \text{Eq. 6.}$$

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 α factor serves to quantify this accumulation of solutes. In a case where no CP occurs, α 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, α increases, resulting in a lower difference in pressure across the membrane. Therefore, for the same applied pressure, the greater the effect of CP on the membrane, the higher the value of α and the lower the value of J_w .

Rearranging Eq. 6. to solve for α , we obtain the following equation:

$$\frac{1}{\pi_b} \left(\Delta P - \frac{J_w}{k_w} \right) = \alpha \quad \text{Eq. 7.}$$

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

Eq. 7. is solved through two experimental tests. At first, the intrinsic 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 obtained during the clean-water test performed at 1380 kPa (300 psi) before each treatment test. Values of K_w are presented in Table 4.

Then, ΔP , J_w and π_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 in a graduated cylinder every 15 minutes at the outlet of the cell over a period of 5 minutes. In order to measure π_b , conductivity in the feed reservoir was also measured every 15 minutes.

3.5.3. 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.* [10] 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. 8. was obtained for TDS concentrations ranging from 10 000 to 80 000 parts per million (ppm) at 25°C [10].

248
$$\pi_b = 1.416 * 10^{-7} c_b^2 + 6.913 * 10^{-2} c_b - 80.64$$
 Eq. 8.

249 Where π_b is the bulk osmotic pressure in kPa and c_b is the bulk TDS in ppm.

250 Since the synthetic water produced for this study was similar to seawater, and its TDS
251 concentration varied between 13 000 to 48 000 ppm in the feed because the RO units
252 were operated in recirculation, we assumed this equation was valid to calculate the
253 osmotic pressure of the synthetic wastewater.

254 In order to compensate for temperature variation that cannot be controlled by the heat
255 exchangers, water fluxes were corrected with a temperature correction factor and
256 standardized at 25 °C using Eq.9.

257
$$J_{w,c} = J_w * TCF$$
 Eq. 9.

258 Where the temperature correction factor (TCF) is calculated using Eq. 10.

259
$$TCF = 0.00101 T^2 - 0.0819 T + 2.4279$$
 Eq. 10.

260 Where T is the temperature in °C. Eq. 10. was obtained from empirical data used by the
261 industry [20].

262 3.5.4. BENCH AND PILOT-SCALE TREATMENT TESTS

263 The bench-scale RO unit was initially rinsed with 4 L of demineralized water for 10
264 minutes with no hydraulic pressure applied. 2.5 L of synthetic saline wastewater was
265 then added to the feed water reservoir. After recirculating the wastewater in order to
266 dilute any dead volume, pressure in the system was set to 2068 kPa (300 psi). Permeate
267 was collected in a 2 L graduated cylinder and the concentrate was returned to the feed

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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.

To compare the pilot-scale unit to the bench-scale unit, the pilot-scale unit was 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 dechlorinated tap water prior to adding 1000 L of synthetic wastewater to the feed water reservoir. Permeate was collected in a 1 m³ 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.

Based on Ladner & al. work, the tangential velocity was set at 0.5 m/s at the membrane surface for all the experiments [10]. Calculations for the velocity in the membrane coupon cell were based on the cell and the mesh geometry. For the spiral-wound membrane (SWM), calculations were based on the equation provided by the supplier.

The water recovery capacities were also compared between both units. This parameter indicates the amount of permeate that can be recovered from the feed. The water recovery capacity is calculated using Eq. 11.

$$RC = \frac{V_{f,permeate}}{V_{i,feed}} \quad \text{Eq. 10}$$

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289 Where $V_{f,permeate}$ is the final volume of permeate and $V_{i,feed}$ is the initial volume of feed.

290 3.5.5. MEMBRANE CLEANING PROCEDURE

291 Treatment tests were followed by a membrane cleaning procedure, performed in two
292 steps. First, for the bench-scale and the pilot-scale unit respectively, a 2 L and a 100 L
293 HCl solution with a pH of 2, was circulated in the system at a flow of 3 L/min for 30
294 minutes. Then, a clean-water test was performed following the method described at
295 section 2.5.1. in order to assess any change of the membrane performance. Since no
296 organic matter was present in the synthetic wastewater, no alkaline cleaning was
297 performed.

298 3.6. ANALYTICAL METHODS

299 For the characterization of the process water and the analysis of the barium and
300 strontium ions, samples were sent to Maxxam Laboratory, an accredited analytical
301 laboratory. All the samples collected during the experiments were analyzed at Centre
302 des Technologies de l'Eau (CTE) according to the following methods: concentrations of
303 calcium, sodium, manganese and potassium ions were measured by flame atomic
304 absorption spectroscopy, Standard Method #3111, using a Shimadzu AA-7000
305 instrument. Total dissolved solids were measured following the Standard Method
306 #2540C (total dissolved solids dried at 180°C). Sulfate concentrations were measured by
307 turbidimetry following the Standard Method #4500E. The apparatus used was a DR-
308 2700 in single signal mode set at 420 nm. Chloride concentrations were measured by
309 titrimetry following the Standard Method #4500 B, pages 4-70, 4-71. Total hardness was

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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[21].

4. RESULTS AND DISCUSSION

4.1. COMPARISON BETWEEN BENCH AND PILOT-SCALE PROCESS WASTEWATER CONCENTRATION TEST RESULTS

The flux and the transmembrane pressure in a membrane coupon are essentially constant due the small area. This is in contrast to SWM where there is a significant spatial variation of these parameters. However, even if these differences are known, the industry commonly performs coupon tests prior to the SWM tests in order to determine full-scale design parameters.

In order to compare the bench to the pilot-scale tests results, 3 treatment tests with 2.5 L of synthetic wastewater and 4 treatment tests with 1000 L of synthetic 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/m² while for the pilot-scale unit this ratio was 143 L/m². For both units, each treatment test was done in recirculation mode where the concentrate is returned to the feed tank and the

permeate is collected in a separate tank. The same membrane coupon and spiral-wound membrane were used for all treatment tests.

The comparison was based 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.

4.1.1. WATER RECOVERY CAPACITY

RO was selected because of the technology's 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 wastewater, RO technology could achieve water recovery capacities ranging from 24% to 75% with salt rejection rates between 89.9% and 99.3% [5-7, 14, 22, 23]. Water recovery obtained for each treatment experiment as well as the average recovery for both bench and pilot-scale tests are presented at Table 3.

Table 3 Water recovery capacity the of bench-scale and the pilot-scale RO unit

| | <u>Bench-scale tests</u> | <u>Pilot-scale tests</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-

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scale tests were comparable to what was observed by Kelkar and *al.* (2003), who obtained recovery capacities of 50% and 55% using RO units with a 3.8 m³/day and 5.7 m³/day capacity, respectively [23]. 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 3790 kPa, which is 18% higher than during the pilot-scale tests where the final applied pressure was 3100 kPa. This difference in applied pressure resulted in a higher permeate flux and a higher water recovery rate. The water recovery as a function of the applied pressure for the bench-scale and the pilot-scale units are presented in Figure 3.

The results show that, for both units, the water recovery follows a constant linear trend for the first 2750 kPa of applied pressure after which the slope start to decrease. When water recovery has reached 50%, membrane polarization becomes more important and reduces significantly the effective pressure applied on the membrane. The results also show that the relation between the water recovery and the applied pressure is similar with both configurations and sizes of RO units. Therefore, for the same applied pressure, there is no significant difference in recovery capacity between the two systems.

4.1.2. TRANSPORT PARAMETERS

Experiments were performed with a constant permeate flux to measure the α factor using Eq. 7. Representative data from a constant flux experiment with both the bench

and pilot-scale unit are shown in Figure 4. 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.

Synthesized data for the 3 bench-scale experiments and the 4 pilot-scale experiments are summarized in Table 4.

Table 4 Characteristics of the bench-scale and the pilot-scale constant flux experiments using a BW30 membrane from Filmtec

| | Bench-scale test | | Pilot-scale test | |
|---------------------|--|-----------------|--|-----------------|
| | Intrinsic water permeation coefficient (k_w) (LMH/kPa) | α factor | Intrinsic water permeation coefficient (k_w) (LMH/kPa) | α factor |
| Specification sheet | 0,035 \pm 0,007 | | 0,035 \pm 0,007 | |
| Treatment #1 | 0,031 | 1,20 | 0,035 | 1,26 |
| Treatment #2 | 0,031 | 1,18 | 0,034 | 1,22 |
| Treatment #3 | 0,030 | 1,12 | 0,034 | 1,23 |
| Treatment #4 | - | - | 0,032 | 1,23 |
| Average | 0,031 | 1,17 | 0,034 | 1,24 |

For both units, an estimated tangential velocity of 0.5 m/s was applied on the membrane. For the calculation of the tangential velocity, a perfectly distributed flow was assumed in both the SEPA cell and the spiral-wound membrane. The intrinsic water permeation coefficients measured with both units prior to the first treatment test are consistent with the values provided by the membrane supplier. The values then measured before each treatment test decreases slightly with both units. The values go from 0,031 to 0,030 LMH/kPa and 0,035 to 0,032 LMH/kPa for the bench-scale and the Roy, D., Gherrou, A., Pierre, P., Landry, D., Yargeau, V., **Reverse osmosis applied to soil remediation wastewater: comparison between bench-scale and pilot-scale results**, *Journal of Water Process Engineering*, Volume 16, 115-122 (April 2017)

pilot-scale units, respectively. This variation is attributed to the compaction and the fouling of the membranes and is discussed more thoroughly in section 3.1.4.

As seen in Eq. 7., the α 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 α value, the greater the effect of CP, since more solutes accumulate on the membrane surface. The average α value obtained from the bench-scale test ($\alpha = 1.17$) indicates that the concentration of solutes at the membrane surface is 41% lower with a membrane coupon compared to the SWM from the pilot-scale unit ($\alpha = 1.24$).

These results confirm that CP is more important with the spiral-wound module. As previously mentioned, there is spatial variation of flow in a SWM, and the estimated tangential flow in both units represents the average. These variations are suspected to cause dead zones in the SWM, thus increasing the observable effect of the CP on the membrane surface. Another parameter to take into account is the variability in performance caused by the fabrication process. This variation is negligible with coupons, since only a small part of the membrane is used (which can be considered a punctual sampling of the membrane). However, the 7.6 m² SWM is subject to these variations, which may have caused the variation of CP.

4.1.3. 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

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calculated rejection capacities are presented in Table 5. 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 5 Membrane rejection capacity for ions in solution during the bench-scale and pilot-scale experiments

| | Bench-scale test | | | Pilot-scale test | | |
|----------------------------|---------------------|---------|---------|---------------------|---------|---------|
| | Rejection (R_i) | | | Rejection (R_i) | | |
| | Lowest | Highest | Average | Lowest | Highest | Average |
| Sulfates (SO_4) | 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) | <0,1 | <0,1 | <0,1 | 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

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

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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 in Figure 5. The same membrane was used for each test and a clean-water permeability test was performed between each treatment test.

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, membrane compaction and configuration. A study conducted on dyeing factory wastewater reported that salt rejection is configuration dependant[15]. Another study reported that flux and rejection can be affected by skin shrinkage due to the contact of the membrane with a salt solution [13]. 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. 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 Roy, D., Gherrou, A., Pierre, P., Landry, D., Yargeau, V., **Reverse osmosis applied to soil remediation wastewater: comparison between bench-scale and pilot-scale results**, *Journal of Water Process Engineering*, Volume 16, 115-122 (April 2017)

since the pressure applied on the membrane coupon was 689 kPa higher than the pressure applied on the spiral-wound membrane due to the bench-scale unit's pump's higher pressure limit.

4.1.4. MEMBRANE FOULING

Since fouling is one of the major drawbacks of filtration technologies, the mineral fouling of the BW30 membrane was assessed. Figure 6a and Figure 6b 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 dechlorinated 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 77 LMH and 82 LMH respectively.

For the bench-scale experiments, the permeate flow decreased to 55 LMH after 3 treatments, corresponding to an overall reduction of 28% in permeate flow. For the pilot-scale experiments, the permeate flow decreased to 68 LMH after 4 treatments, corresponding to an overall reduction of 17% 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 10% was

observed after the first treatment followed by a reduction of 7% for the 3 following treatments.

In both cases, the HCl membrane cleaning allowed the membrane to recover a permeate flow similar or higher than its initial value. The permeate flows measured for the bench and the pilot-scale after cleaning were 79 LMH and 85 LMH respectively (Fig. 5). The fact that a higher value of permeate flow was obtained than initially calculated can be explained by a membrane's skin shrinkage when in contact with a concentrated salt solution such as the metabisulfite solution used for membrane storage. This phenomenon was observed to affect the membrane performances elsewhere [13]. 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 6.

Table 6 NaCl rejection tests results for the BW-30 membrane before and after cleaning using a HCl solution for bench-scale and pilot-scale tests

| Operating pressure | Before treatments | After treatments | Difference |
|-----------------------------------|--------------------------|-------------------------|-------------------|
| 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 [11]. For the bench-scale tests, a significant difference in rejection capacities was noted after the cleaning of the membrane. At Figure 5, a reduction of 5% in rejection capacities between the first and the third treatment test was observed. 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 6 are then consistent with what was observed on Figure 5.

The assessment of the membrane fouling shows that mineral fouling that occurs during the treatment of the synthetic wastewater is reversible. The cleaning method using an HCl solution recovered the initial membrane's performances for both bench and pilot-scale tests. In terms of salt permeability, a loss of rejection capacity of 2% to 3.5% suggests that the membrane coupon structure was affected by the cleaning. However, this loss was also observed during the treatment tests, which means that this observation could also be due to membrane shrinkage as described in section 3.1.3. For the spiral-wound membrane (pilot-scale unit), 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 was observed.

497 5. CONCLUSION

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

507 The bench-scale tests exhibited an average water recovery of 71% with the BW-30
508 membrane from Filtmtec while the pilot-scale tests exhibited an average water recovery
509 of 56% with the same membrane. Since the pressure limit of the pilot-scale system was
510 lower, 2930 kPa compared to the bench-scale unit's 3447 kPa, this difference in
511 operating pressure was assumed to be responsible for the 15% difference in water
512 recovery. The average rejection capacities calculated for each element in the solutions
513 with either unit were similar. The overall rejection capacities after the treatment
514 experiments for the dissolved solids were 90.6% and 96.3% for the bench-scale and
515 pilot-scale units respectively.

516 In terms of the transport parameters, it was found that spiral-wound membranes are
517 more influenced by CP than membrane coupons. The α factor, which expresses the

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518 amount of solutes accumulated on the membrane surface, calculated for the bench and
519 pilots-unit were 1.17 and 1.24, respectively. These values indicate that the
520 concentration of solutes on the membrane surface is 17% and 24% higher than the bulk
521 concentration for the bench-scale and the pilot-scale unit, respectively.

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

527 The results presented lead to the conclusion that bench-scale RO tests do provide
528 valuable information prior to performing pilot-scale testing. The salt-rejection capacity
529 as well as water recovery capacity were found to be adequately evaluated at bench-
530 scale. Bench-scale studies can also be used to characterize the nature of foulant
531 accumulating at the membrane surface in order to identify the proper cleaning method.
532 However, bench-scale studies neither accurately predicted the membrane transport
533 parameters such as the water permeation coefficient nor determine the flux decline
534 that could be potentially caused by foulant. In order to have a better understanding of
535 the factors that cause these differences between the bench and pilot-scale tests, foulant
536 layer should be characterized and a flow simulation comparison should be made for
537 both units.

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Figure 1 : Bench scale reverse osmosis set-up

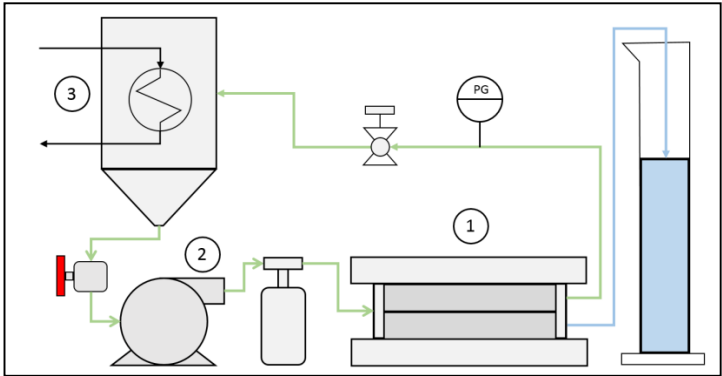
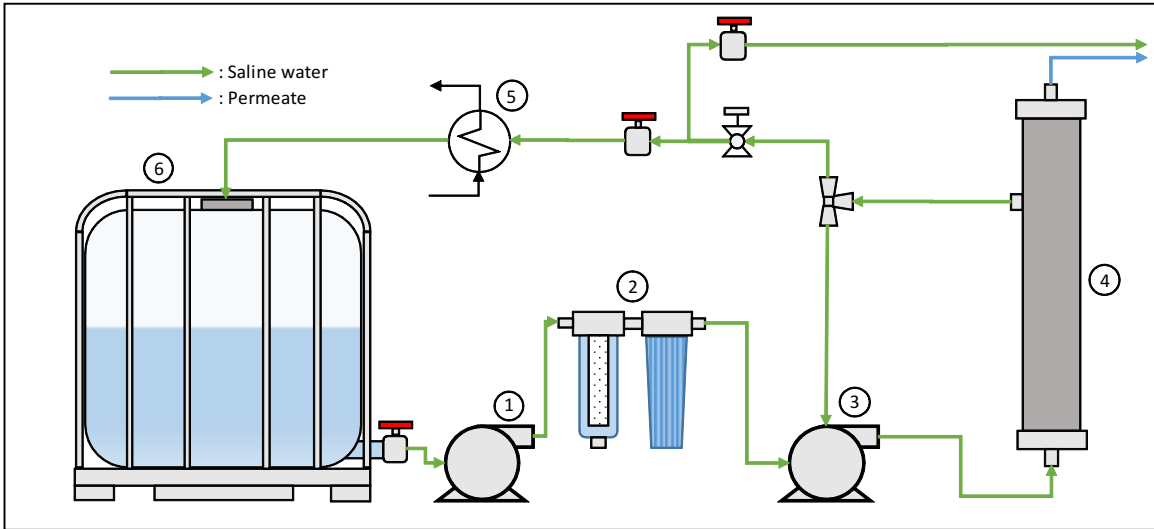
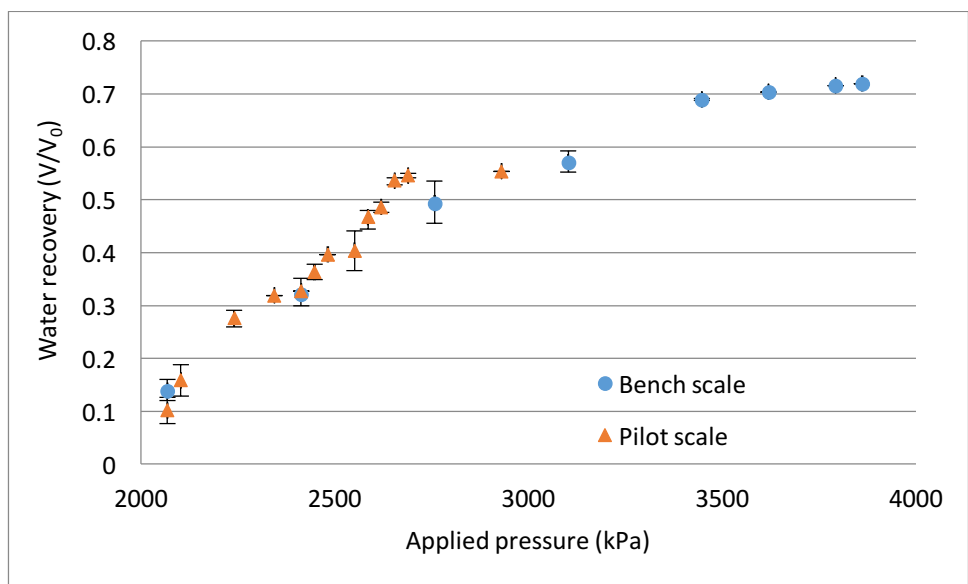


Figure 2 : Pilot-scale reverse osmosis set-up



616 Figure 3: Water recovery results for the bench-scale and the pilot-scale RO units



619 Figure 4 : Representative data showing the results from a bench-scale and a pilot scale
620 experiment for a constant-flux experiment

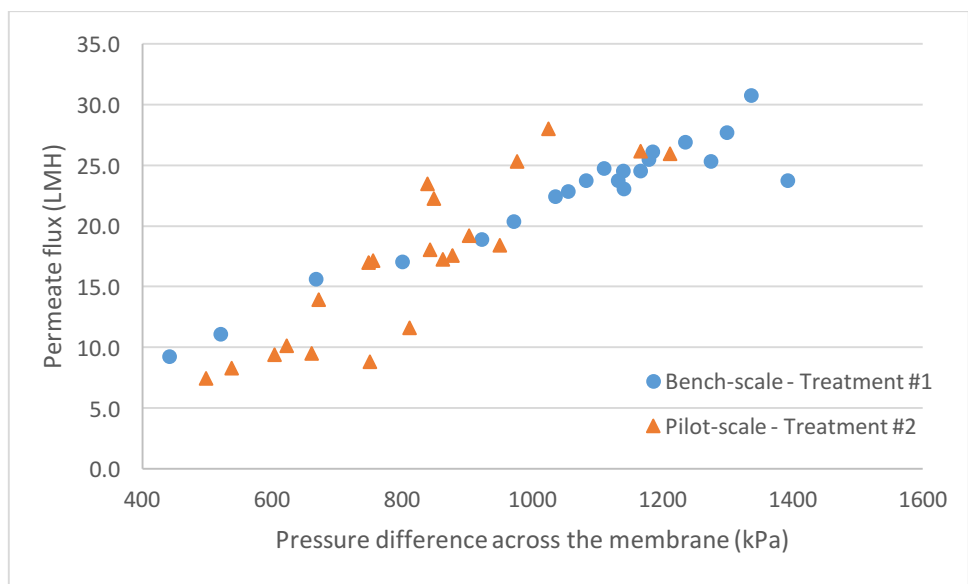
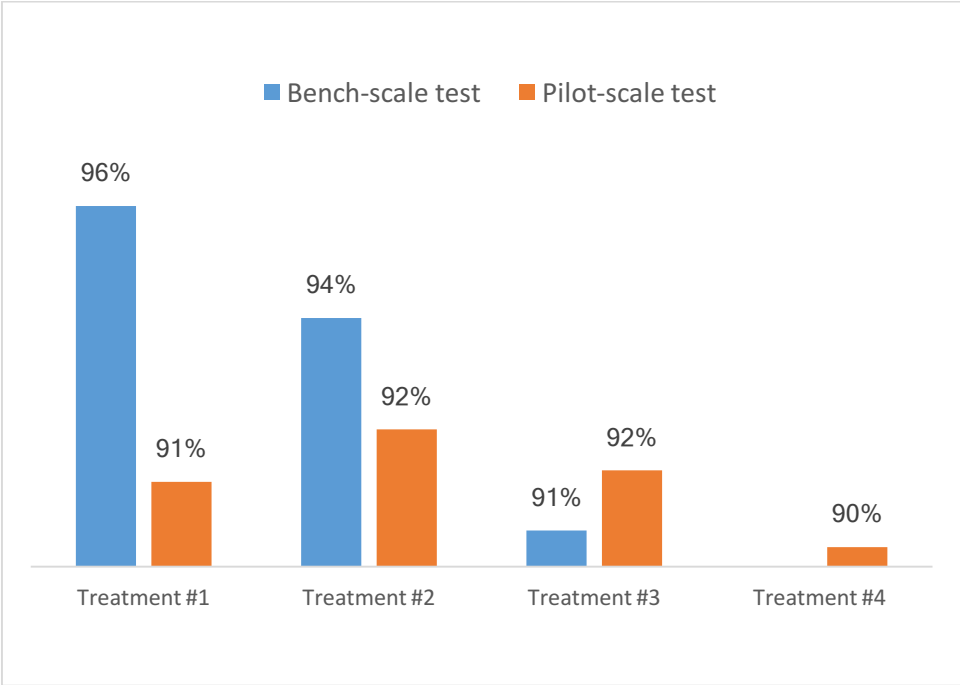
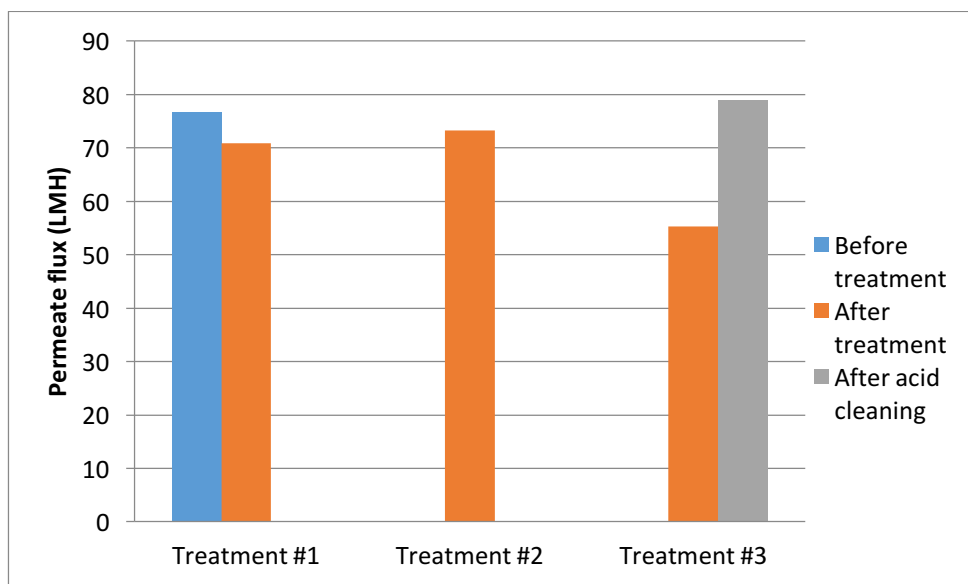


Figure 5 : Membrane rejection capacity for the total solids measured for each treatment test during bench-scale and pilot-scale tests



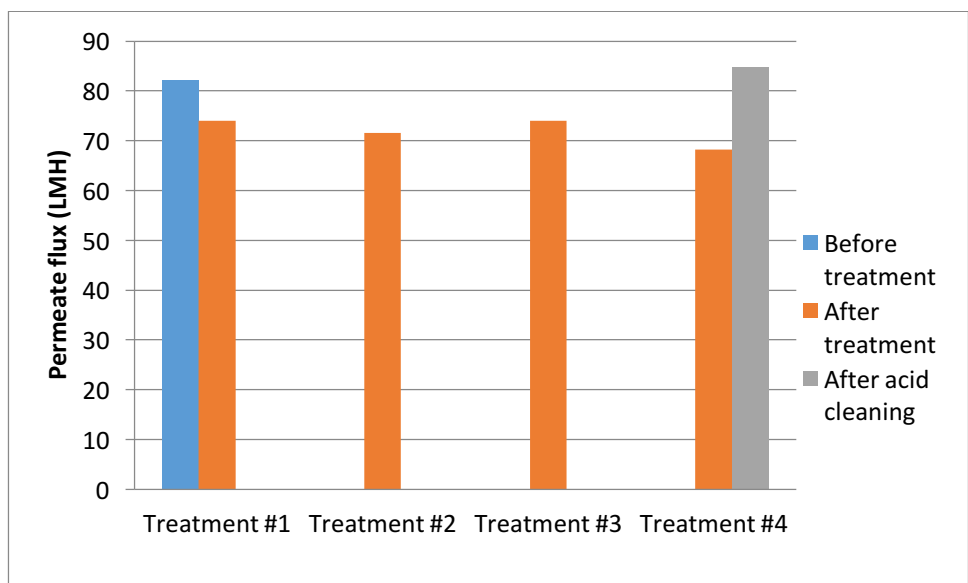
628 Figure 6a : Permeate flux measurements for bench-scale clean-water tests



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631 Figure 6b : Permeate flux measurements for pilot-scale clean-water tests



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