A clinoptilolite-PDMS mixed-matrix membrane for high temperature water softening
Farzad Yazdanbakhsh, Moien Alizadehgiashi, James A. Sawada and Steven M. Kuznicki

ABSTRACT
A mixed-matrix membrane composed of polydimethylsiloxane (PDMS) as the continuous phase and clinoptilolite, a naturally occurring zeolite, as the active phase has been used to decrease the conductivity of water by more than 80% across the membrane. Testing was carried out using a cross-flow configuration at temperatures as high as 160°C using a constant transmembrane pressure of 8 bar. The simple fabrication method for the membrane, the durability of the system under the test conditions, and a suitable flux rate make such membranes promising candidates for industrial wastewater treatment.

Key words | clinoptilolite, mixed-matrix membrane, PDMS, water softening, zeolite

INTRODUCTION
The restricted amount of fresh water available for energy production makes the recycling of process-affected water a necessity. Like most energy production activities, oil production processes generate large quantities of wastewater containing various organic and inorganic compounds. More than 75% of Alberta’s heavy oil reserve is accessible only through in situ production methods and, among the various methods, Steam Assisted Gravity Drainage (SAGD) introduced by Butler (Butler & Stephens 1981; Al Bahlani & Babadagli 2008) in 1978, uses water extensively. The water used during the process is contaminated with high concentrations of dissolved salts, soluble organics, and dissolved inorganics such as silica; all of which can shorten the life time of the boilers if not reduced to trace levels (Heins et al. 2006; Heins 2009).

To re-use the water several processes are carried out including settling and filtration, lime softening (to reduce the amount of silica and magnesium) and weak acid cation exchange for hardness ion removal (Adamaref et al. 2014). The different process steps require the process water to be cooled from about 160°C to temperatures suitable for the various de-oiling and softening stages. The purified water must then be heated again to 160°C for reinjection down the well. A single filtration system which can directly de-oil and soften the process water without requiring cooling and re-boiling could significantly simplify the water treatment system.

Inorganic ceramic membranes were developed through the 1940s and through the 1980s ceramic microfiltration and ultrafiltration membranes were developed (Bhave 1991). Due to their enhanced mechanical, thermal, and chemical stability compared with polymeric membranes, ceramic materials have been used for challenging water purification processes such as high temperature oil-water separation (Zaidi et al. 1992), industrial wastewater treatment, and special applications like bilge water treatment that contain high levels of oil and suspended solids (Benito et al. 2007). Due to manufacturing complexities, ceramics are generally too expensive to be used in large-scale industrial applications and so have been used in small-scale separation processes that cannot be addressed by other type of membranes (Faibish & Cohen 2001).

Zeolite membranes are an important class of inorganic membranes. Classical zeolites are microporous, crystalline aluminosilicate solids which, depending on the zeolite type and its composition, can have an effective pore size of 0.3 nm to about 1 nm. The use of zeolite membranes for the separation of gas mixtures is well established (Caro et al. 2000) but has not been explored extensively for water purification. The use of zeolite membranes for water purification is a natural extension of the materials’ properties because the size-exclusion properties inherent to the nanoporous structure play a directing role in rejecting...
species much larger than water. Using molecular dynamic simulations Lin & Murad (2001) demonstrated the feasibility of using zeolite membranes as a membrane material for reverse osmosis (RO) water treatment. Their study modeled a dense section of a cation-free analogue of zeolite A and their results suggested that, provided the pore size of the zeolite is small compared with the diameter of the hydrated cation, effective reverse osmosis separation is possible. Recent attempts have been made to employ zeolite membranes for oil-water separation and oilfield-produced water which showed that the membranes meet the removal levels required by the standards (Barbosa & Rodrigues 2013).

Synthetic zeolite membranes suffer from almost unavoidable intercrystalline defects that occur during the network growth of the crystals, which limit their performance (Caro et al. 2005). The preparation of membranes with large surface areas is another challenge facing synthetic zeolite membranes (Noack et al. 2001). To overcome these limitations, work has been directed toward developing zeolite-polymer hybrid membranes. Termined mixed-matrix membranes, such formulations combine the selectivity of the zeolite molecular sieves with the manufacturability of polymer membranes.

Polymeric materials span a range of properties from amorphous and rubbery to crystalline. Rubbery polymers, such as polydimethylsiloxane (PDMS) have a glass transition temperature (Tg) well below ambient, which allows the polymer chains to pack together efficiently. The ability for the chains to easily conform largely eliminates microscopic voids between poorly packed polymer chains. It also allows the polymer to wrap tightly around solid particles and seal the inter-particle spaces (Lötters et al. 1997; Chung et al. 2007). A PDMS-cellulose acetate mixed-matrix membrane has been studied for ethanol/water separation and the results indicate that the separation performance is enhanced compared with a pure PDMS membrane (Lingyun et al. 2015). Adnadjevic et al. (1997) studied PDMS mixed-matrix membranes containing various hydrophobic zeolites. Pervaporation of an ethanol/water mixture at 50 °C demonstrated that the permeability of the membrane scaled with its zeolite content. Another study by Hussain & König (2012) developed a zeolite-PDMS mixed matrix membrane for the separation of N2 and CO2 in the gas phase. The study showed that at 30 °C and 20 bar pressure the presence of zeolite improves the selectivity and permeability. These previous studies establish that PDMS is a suitable matrix for zeolite mixed-matrix membranes though the application of this matrix at high temperature and high pressure has not yet been established for water purification.

In this study, finely ground, naturally occurring clinoptilolite was mixed with PDMS to form a mixed-matrix membrane. Clinoptilolite, a hydrophilic zeolite, is particularly well suited for water purification because it has the smallest pore size amongst all of the zeolites while simultaneously having a thermal and hydrothermal stability. A membrane containing 66 wt% clinoptilolite and having a diameter of 25 mm and thickness of ~150 μm was tested for its ability to reject hardness ions from municipal water at temperatures as high as 160 °C using a transmembrane pressure of 8 bar. Tap water, rather than synthetic SAGD water, was used as a feed fluid to simplify the testing and to demonstrate, under clean water conditions, the thermomechanical stability of the PDMS-clinoptilolite system under the target process temperature and pressure. Both the polymer and zeolite used in the membranes are available in commercial quantities. The polymer matrix enables continuous casting processes to be used and if the PDMS-zeolite membrane can be prepared in a roll configuration (equivalent to RO membranes) then the technology can be scaled to address very large volume water treatment applications.

METHODS

Materials

Clinoptilolite was received from the St. Cloud Mining Company’s Ash Meadows deposit (Winston, NM, USA) as crushed and sieved powder having maximum particle size of 44 microns (~325 mesh). The phase purity of the material was stated as 99%+ which was confirmed by X-ray diffraction (XRD) and energy dispersive X-ray (EDX) analysis. Elastosil® RT 601 A/B was selected as the matrix phase and was provided by Wacker Chemie AG (Munich, Germany). The silicone rubber contains two components (A and B), which must be mixed at ratio of 9:1, respectively, followed by a curing process at 75 °C for 20 min. Toluene (ACS Fisher Scientific) was used as a diluent for the mixed-matrix membranes.

Membrane preparation

A pure PDMS membrane was fabricated using 1.8 g of component A and 0.2 g of component B. The components were weighed into a glass beaker and mixed for 2 minutes. The solution was then poured onto a metal casting plate having a circular plenum 50 mm in diameter and 300 μm deep machined into its surface. A doctor blade was drawn.
across the surface of the plate to remove the excess solution and to ensure a consistent quantity of fluid filled the plenum. The mixture was outgassed in a vacuum desiccator to remove air from the mixture, which was entrained during mixing. In accordance with the manufacturers recommendations the casting plate was then placed in an oven at 75 °C for 20 min to cure the polymer.

To prepare a mixed-matrix zeolite membrane film the procedure outlined by Hussain & König (2012) was followed. The same amount of each polymer component was weighed into a glass beaker and thoroughly mixed for the same period of time. 3 g of clinoptilolite (as-received) was then added to the polymer mixture and 2 mL of toluene was then added to reduce the viscosity of the mixture. The final mixture was then poured onto the casting plate and the excess was similarly removed using a doctor blade drawn across the surface of the plate. The mixed-matrix membrane was outgassed and cured under the same conditions as the pure PDMS mixture. The pure PDMS membranes had an average thickness of ~250 μm while the zeolite mixed-matrix membranes were ~150 μm. The difference between the thickness of pure PDMS and the mixed-matrix membrane is due to the addition of toluene to the mixture during the mixed-matrix membrane, which evaporates during polymer curing in the oven. The resulting rubbery sheets were trimmed to discs having an outer diameter of ~25 mm which were subsequently mounted in the permeation test system fixture.

The selected preparation conditions were the result of a number of trials that demonstrated that entrained air bubbles produce large defects that circumvent the zeolite particles. The thickness of the membranes demands that all of the zeolite particles be in fluid communication with each other to allow water to permeate through the film. It was determined that membranes having zeolite compositions between 50 and 66 wt% were impermeable due to the zeolite particles being fully encapsulated in the matrix. Membrane compositions having in excess of about 66 wt% lacked mechanical integrity and would break apart. Hence the upper limit of zeolite content was chosen at 66 wt%, which concurs with the findings of Hussain & König (2012). This result was unexpected as the particle size and density of clinoptilolite and ZSM-5 powders are not identical.

Characterization

XRD analysis

The crystal structure of the powders was analyzed by powder XRD using a BRUKER D4 Endeavor diffractometer (Bruker-AXS, Karlsruhe, Germany). The measurements were carried out with Cu Kα radiation, a step size of 0.01° and a step time of 0.5 s. The acceleration voltage was 40 kV and the emission current was 40 mA.

Scanning electron microscopy and energy dispersive X-ray (SEM/EDX) analysis

Scanning electron micrographs and EDX map were collected using a Zeiss Sigma Field Emission Electron Microscope equipped with an Oxford 150 mm² X-MaxN Silicon Drift Detector.

Thermal gravimetry-mass spectrometry

In order to investigate the thermal-oxidative stability of the membrane, thermal gravimetry-mass spectrometry (TGMS) scans were performed on the mixed-matrix membrane as well as the cured PDMS rubber. All analyses were done using air to facilitate the complete combustion of the organic fraction of the polymer. The TGA plots were collected using TA Instruments Q500 thermogravimetric analysis. Mass spectroscopy analysis was performed using Pfeiffer OmniStar GSD 220. Samples were loaded on platinum pans and heated at a rate of 10 °C/ min from ambient to 750 °C. The mass spectrometer was configured with a quartz capillary which was positioned at the center of the exhaust stream to sample the evolved gas. To start data collection, the two instruments were manually synchronized and so an uncertainty of a few tenths of a degree for the starting temperature can be expected between the TG and MS plots. Experiments were run in MID mode with fragments at m/z = 15 (CH₃), 18 (H₂O) and 44 (CO₂) tracked over time. These fragments are believed to be the most diagnostic signals associated with thermal decomposition of the PDMS polymer.

Surface area measurements

Surface area data were collected using Micromeritics ASAP 2020C Surface Area and Porosity Analyzer. Prior to analysis samples were outgassed at 350 °C for at least 12 h under vacuum (5 × 10⁻⁵ Pa).

Permeation testing

A bench-scale test system was constructed and used to test the membranes under a range of temperatures and
transmembrane pressures. Figure 1 shows the schematic diagram of this system.

Water was pumped from the 20 L tank to a bypass loop whose pressure was controlled by a backpressure regulator. Excess flow was returned to the tank and since the permeate flow rate was negligible compared with the feed flow rate the concentration of salts in the reservoir was considered constant. A rotameter installed on the bypass loop controlled the flow into a stainless steel pipe, which was centered in the furnace and was equipped with a temperature controller. The furnace output was controlled from an external thermocouple (TI2) placed in contact with the fluid leaving the stainless steel pipe at the outlet of the furnace. In this way the water temperature could be rigorously controlled using the furnace temperature controller. Two thermocouples were used to monitor the water temperature before and after the membrane and confirmed that the temperature drop across the membrane was not greater than 5 °C.

The membranes were placed on a porous, stainless steel disc having an outer diameter of 33 mm, a thickness of 2.5 mm, and average pore size of 2 μm to provide mechanical support to the membrane. The lower surface of the support disc and the upper surface of the membrane were sealed by compressing the assembly in a stainless steel assembly between using #214 Buna-N O-rings. The membrane films, as tested, had an effective surface area of 5 cm².

A back-pressure regulator was used on the outlet of the membrane feed line to control the transmembrane pressure. The feed water flow rate was maintained at 50 mL/min for all experiments. The liquid permeate, was collected into a scintillation vial at temperatures below 100 °C. Above 100 °C a glass cold finger that was immersed in ice water was used as the permeate collection system in order to condense all of the permeate vapor. By avoiding uncontrolled evaporation of steam from the permeate port, the dissolved solids in permeate water were not artificially concentrated.

The membranes were tested over a range of temperatures from ambient up to 160 °C using a constant transmembrane pressure of 8 bar. The operating pressure was chosen to exceed the vapor pressure of saturated steam at all temperatures to ensure the water in the feed lines was kept in the liquid phase. All permeate samples were allowed to cool to ambient temperature before measurement. Total conductivity measurements were performed in triplicate using an Accumet XL 20 conductivity meter and the average values reported.

RESULTS AND DISCUSSION

The phase purity of the as-received clinoptilolite was stated by the supplier to be 99% and was confirmed by XRD. The diffraction pattern for clinoptilolite in Figure 2 did not have any unassigned reflections and no measurable amount of quartz was detected. As expected, the intensities of the zeolite reflections were reduced by ~40% in the samples containing PDMS due to the dilution from the amorphous, polymer phase. The XRD patterns were otherwise identical.

The nitrogen physisorption results (not shown) measured a surface area of 14 m²/g for the pure clinoptilolite powder. The results demonstrated that the pore size for clinoptilolite prevented the N₂ molecules from diffusing into the pores under the cryogenic conditions. This effect has been previously reported for ultramicroporous materials (Kaneko & Ishii 1992). Different clinoptilolite samples from various resources have been reported in the literature to have different surface areas in the range of 11–17 m²/g (Korkuna et al. 2006) which agree with the results for clinoptilolite used in this study. The physisorption results confirm that the ultramicroporous characteristics of
clinoptilolite allowed it to physically exclude any molecules larger than about 0.4 nm.

Figure 3 shows the TGMS plots for the PDMS silicone rubber. A period of time was necessary for atmospheric CO$_2$ and water to be purged from the furnace but by $\sim 150 \, ^\circ C$ all baseline MS signals were stable. Pure PDMS did not show any measurable weight loss until 250 $^\circ C$; a temperature well in excess of the target process temperature of 160 $^\circ C$. According to Camino et al. (2002), at lower temperatures PDMS decomposed to cyclic oligomers. The observation of both CO$_2$ and water between 250 and 400 $^\circ C$ suggested that these oligomers were reactive toward oxygen as they combusted at relatively low temperatures. As the temperature increased, scission began on the Si-C bonds (78 kcal/mol) until 650 $^\circ C$ after which the polymer was completely degraded.

The TGMS trace for the mixed-matrix membrane shows a different water profile due to the intracrystalline water contained within the zeolite pores. The combustion behavior, as expected, remains largely unchanged and the residual mass fraction at the end of the scan is representative of the weight fraction of zeolite combined with the fraction of silica left over from the PDMS.

Figure 4 shows SEM images of the surfaces of a pure PDMS and the mixed-matrix zeolite membrane. Figure 4(a)
demonstrates that PDMS has a uniform surface without any obvious defects in the film. The occlusions on the surface were small impurities stuck to the soft rubber surface. Once clinoptilolite was added to the polymer mixture (Figure 4(b)), the surface texture changed to reflect the random packing of zeolite particles. The clinoptilolite was supplied as a ∼325 mesh powder which ensures particles larger than 44 microns are removed from the blend; it does not provide a Gaussian particle size distribution. The wide distribution of particle sizes is evident in Figure 4(b) and this distribution is beneficial, as the smaller particles will occupy the voids created between larger particles thereby increasing the packing density of the particles.

The homogeneity of the membrane was assessed by examining samples in cross-section. Cross-section images of a pure PDMS film and the zeolite mixed-matrix membrane are presented in Figure 5. The PDMS film displayed a glassy texture typical of polymer rubbers and the absence of pinhole defects or blind pores indicated that the degassing procedure was effective in removing entrained air. The texture of the mixed-matrix membrane (Figure 5(b)) was significantly different from the pure polymer and suggested that zeolite particles were evenly distributed within the polymer matrix. The image of the membrane also established that a continuous skin of polymer, which would significantly hamper permeation, had not formed on the surface of the membrane.

The distribution of the zeolite and matrix components was difficult to assess visually so EDX mapping was carried out to confirm the homogeneous distribution of the zeolite within the membrane film. The zeolite contains silicon and aluminum as well as a range of alkali and alkaline earth ions. Since the polymer also contained silicon, it was excluded from the elemental map. Figure 6(a) shows the SEM of a typical section of the surface of the membrane together with the EDX map of the same area (Figure 6(b)). The image revealed that the distribution of aluminum was uniform across the surface of the sample. The observation that the cations were equally well distributed provided further
evidence that the zeolite particles are well dispersed within the film.

There were regions of the membrane that showed as dark spots in the SEM image, which also manifested as aluminum-free regions in the EDX map. Such an area is circled in the image and is likely a small, polymer-rich region of the film formed, perhaps, by the formation and rupture of a small air bubble during the outgassing process.

The fabrication processes specified earlier were selected to minimize the structural defects in the membrane and the processing used was suitable to fabricate defect-free, pure PDMS sheets. The addition of zeolite to the polymer, however, complicated the fabrication of the membrane film. Figure 7 shows a defect that was visible on the surface of the mixed-matrix membrane surface. This type of defect was likely due to an air bubble that was entrained in the polymer-zeolite mixtures during blending and which was not successfully removed during the outgassing step. It could not be established from the image whether the defect penetrated the full thickness of the membrane although even if the defect penetrated only partially through the membrane, the effectiveness of that portion of the membrane would expected to be compromised. These defects were considered to be the primary reason for not achieving 100% reduction in water hardness and conductivity. The elimination of these defects through changes to the fabrication process was felt to be critical to improvements in the membrane performance.

Permeation testing

The permeate composition for the mixed-matrix membranes was compared against the parent silicone rubber to confirm that the changes in performance seen in the mixed-matrix membranes were due, in fact, to the zeolite content in the membrane. Pure PDMS films having a thickness of ~250 μm were tested for 3 hours at both 25 and 160 °C using a constant transmembrane pressure of 8 bars. Since PDMS is a highly hydrophobic polymer (contact angle 90–120°), no permeation was expected for this membrane. The observation that no permeation was observed for the pure PDMS membranes demonstrated that the polymer itself is suitably defect-free and thermally stable to survive exposure for 3 h to the aggressive test conditions used.

The pores of clinoptilolite have an effective size of less than 0.4 nm which are small enough to allow only the transmission of water molecules (Lin & Murad 2001). Molecules larger than water as well as hydrated cations are rejected. The presence of any ions, hardness or otherwise, in the permeate water should, therefore, be due to defects in the continuity of the polymer phase in the mixed-matrix membrane. As a result, the decrease in total water conductivity of the permeate water was chosen to be the
primary performance indicator. Edmonton (Alberta, Canada) municipal tap water with an average conductivity of 343 μS/cm was used in the permeation experiments. The composition of the municipal tap water is listed in Table 1.

Table 2 shows the measured conductivities for the feed and permeate streams for the mixed-matrix membrane containing 66 wt% clinoptilolite. The conductivities reported were an average of three replicates at each temperature and, as mentioned previously, the permeate water samples were cooled to room temperature prior to measurement to avoid the confounding influence of temperature on conductivity. The data in Table 2 demonstrate that the reduction in conductivity did not change significantly as the water temperature increased, which implies the separation mechanism was consistent across the range of conditions. This type of behavior is reasonable because the water was maintained in its liquid state at the feed side of the membrane.

The change in the permeate conductivity between 100 and 160 °C was not sufficient to indicate widespread failure of the film but at higher temperatures the membrane is expected to be more easily deformed. It is possible that the combination of high temperature and high transmembrane pressure caused the small gaps and defects in the membrane to expand which could have allowed more feed water to be transferred directly to the permeate side, resulting in a marginally lower rejection fraction.

The permeate flux was measured as a function of temperature and the results are presented in Figure 8. The trend in the flux is linear up to 100 °C and then inflects upward beyond that temperature. The linear trend in the flux at and below 100 °C is independent of permeate conductivity (Figure 8) which demonstrates that the permeation through the membrane does not come at the expense of product purity. The inflection seen in the trend above 160 °C can be explained using a combined permeation/pervaporation mechanism.

As mentioned previously, at temperatures above 100 °C the permeate samples were collected in a condenser. At 160 °C the permeate liquid vaporized as it crossed the membrane and was subjected to atmospheric pressure. The change in state of the water creates an additional, pervaporative, driving force across the membrane that did not exist at lower temperatures. The presence of a pervaporation mechanism is likely the cause of the increase in permeate flux seen at this temperature.

CONCLUSIONS

Zeolite mixed-matrix membranes that pair a naturally occurring zeolite with a high-temperature silicone rubber offer an alternative to brittle, synthetic zeolite membranes for water purification and add the advantage of being suitable

Table 1 | Composition of Edmonton municipal tap water (September 2013, EPCOR)

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Unit</th>
<th>Monthly average</th>
<th>YTD* min</th>
<th>YTD* max</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bromate, dissolved</td>
<td>mg/L</td>
<td>&lt;0.005</td>
<td>&lt;0.005</td>
<td>&lt;0.005</td>
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<tr>
<td>Chlorate, dissolved</td>
<td>mg/L</td>
<td>0.12</td>
<td>0.01</td>
<td>0.21</td>
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<tr>
<td>Chloride, dissolved</td>
<td>mg/L</td>
<td>3.78</td>
<td>2.40</td>
<td>11.00</td>
</tr>
<tr>
<td>Conductivity</td>
<td>μS/cm</td>
<td>343</td>
<td>301</td>
<td>485</td>
</tr>
<tr>
<td>Hardness, calcium</td>
<td>mg CaCO₃/L</td>
<td>110</td>
<td>84</td>
<td>258</td>
</tr>
<tr>
<td>Hardness, total</td>
<td>mg CaCO₃/L</td>
<td>161</td>
<td>130</td>
<td>303</td>
</tr>
<tr>
<td>Nitrate (as N), dissolved</td>
<td>mg/L</td>
<td>0.01</td>
<td>&lt;0.1</td>
<td>0.08</td>
</tr>
<tr>
<td>Nitrite (as N), dissolved</td>
<td>mg/L</td>
<td>&lt;0.1</td>
<td>&lt;0.1</td>
<td>&lt;0.1</td>
</tr>
<tr>
<td>pH</td>
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<tr>
<td>Potassium</td>
<td>mg/L</td>
<td>0.83</td>
<td>0.60</td>
<td>2.82</td>
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<tr>
<td>Sodium</td>
<td>mg/L</td>
<td>7.6</td>
<td>3.2</td>
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<tr>
<td>Sulphate, dissolved</td>
<td>mg/L</td>
<td>47</td>
<td>39</td>
<td>131</td>
</tr>
<tr>
<td>Total dissolved solids</td>
<td>mg/L</td>
<td>209</td>
<td>191</td>
<td>263</td>
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*YTD: Year-to-date.

Table 2 | Conductivity of the feed and permeate streams between 25 and 160 °C

<table>
<thead>
<tr>
<th>Temp °C</th>
<th>Feed μS/cm</th>
<th>Permeate μS/cm</th>
<th>Rejection %</th>
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<tr>
<td>25</td>
<td>335</td>
<td>45</td>
<td>87</td>
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<td>60</td>
<td>350</td>
<td>37</td>
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<tr>
<td>100</td>
<td>342</td>
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<td>88</td>
</tr>
<tr>
<td>160</td>
<td>364</td>
<td>62</td>
<td>83</td>
</tr>
</tbody>
</table>

Figure 8 | Permeation flux in the mixed-matrix membrane as a function of temperature.
for high-volume, polymer membrane fabrication techniques. Flexible membranes fabricated from PDMS having 66 wt% high purity natural clinoptilolite have the mechanical and chemical durability to withstand water at high temperature and pressure. The membrane has demonstrated an ability to reject at least 80% of the water conductivity from high pressure tap water at temperatures between 25 and 160 °C. Between ambient and 100 °C the flux of the membrane rose as a function of temperature but the permeate purity remained constant. Above 100 °C, pervaporation is believed to create an additional driving force across the membrane which enhances product permeation. Permeate purity is expected to increase with further optimization of the processing conditions to ensure defects due to entrained air bubbles are eliminated from the cast films.

ACKNOWLEDGEMENTS

The authors thank Peng Li and Gayle Hatchard from the Nano-fabrication and Characterization Facility (nanoFab) of the University of Alberta for collecting SEM and EDX data and Shiraz Merali for measuring the XRD patterns. Financial support from the Canada Research Chair in Molecular Sieve Nanomaterials and Helmholtz-Alberta Initiative is kindly acknowledged.

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