Forward osmosis treatment of drilling mud and fracturing wastewater from oil and gas operations

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A B S T R A C T
To produce large volumes of newly discovered unconventional gas, hydraulic fracturing of wells is commonly practiced in basins where shale gas and coal bed methane are extracted. Hydraulic fracturing of wells during oil and gas (O&G) exploration consumes large volumes of fresh water and generates larger volumes of contaminated wastewater. In this study, a novel application of forward osmosis (FO) was tested for treatment and reclamation of water from drilling waste to facilitate beneficial water reuse. By using FO, two major benefits were achieved: both the volume of the waste stream and the need for a fresh water source were greatly reduced. Results indicate that FO can achieve high rejection of organic and inorganic contaminants, membrane fouling was reversible, and that the process was able to effectively recover more than 80% of the water from the drilling waste. Osmotic backwashing was demonstrated to be an effective membrane cleaning technique; successfully removing fouling and restoring water flux.

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1. Introduction

Environmental impacts of drilling and hydraulic fracturing of oil and gas (O&G) wells are becoming of greater concern in the United States and around the world. The main challenges include the influence of hydraulic fracturing on groundwater quality and the management of drilling and fracturing wastes and produced water [1–4]. Consequently, state and federal governments are enforcing more stringent regulations and discharge limits on O&G companies [1].

Up to 5 million gallons of water are used during drilling and fracturing of a single well, and after drilling and fracturing, water flows back to the surface and becomes drilling and fracturing waste. Subsequently, produced water flows out of the well together with the oil or gas throughout most of the productive lifetime of the well [1,3]. It is estimated that the water-intensive fracturing process is typically responsible for 25–30% of the cost of drilling a well in the Marcellus Shale play [5]. A fraction (up to 15%) of drilling and fracturing waste may constitute impurities and solids, minerals (including heavy metals) and organic matter from geologic formations, polymers and other chemical additives, and propants, which are sand or high-strength ceramic particles/grains used during fracturing to keep shale fractures open and allow free flow of gas and oil to the well [1,6].

The water used for drilling and fracturing of O&G wells is often sourced from local water supplies and/or trucked to the site, thus creating competition for local water resources and supplies. If available, produced water from nearby production wells is also used for development of new O&G wells. To decrease the consumption of regional fresh water, drilling and fracturing waste can be treated and reused on site. Thus, it is critical to explore and develop new technologies that can efficiently and sustainably treat O&G waste streams and facilitate beneficial water reuse.

Current management techniques for drilling and fracturing waste include treatment and discharge and deep-well injection [1,7]. Treatment methods typically involve settling solids and then decanting the fluid; yet, this method only allows for 50–60% recovery of water, which can be reused, but is still contaminated [8]. Once drilling and fracturing operations are complete, the waste generated is usually injected into Class II deep wells [7]; however, the long-term implications of underground injection of O&G waste are not well understood, and this method permanently removes water from the fresh water cycle [2].

1.1. Forward osmosis for treatment of O&G exploration wastewater

Forward osmosis (FO) is an osmotically driven membrane process in which the driving force for separation is the difference in chemical potential between a concentrated draw solution and a broad range of aqueous solutions, including contaminated wastewater [9]. The process involves the permeation of water from a feed solution of low osmotic
pressure, or high chemical potential, across a semipermeable FO membrane, to a draw solution of high osmotic pressure, or low chemical potential. The FO membrane acts as a tight barrier to contaminant transport, including organic matter and dissolved and suspended solids. FO can be incorporated into and tested for a broad range of applications, including treatment of domestic and industrial wastewater, concentration of liquid foods and beverages, and desalination of brackish water and seawater [9–17]. For treatment of impaired water, FO can be coupled with processes such as reverse osmosis (RO) or distillation to constantly reconcentrate the draw solution and simultaneously produce high-quality product water [18–21]. In applications in which high-quality product water is not required, FO can be operated in an osmotic dilution mode (no draw solution reconcentration) to recover water from waste streams and produce a new source of water for beneficial use [22,23]. Because of the high rejection of inorganic and organic constituents and the low irreversible fouling of FO membranes, osmotic dilution is an ideal process for water recovery from drilling wastewater and for simultaneous concentration and volume reduction of the waste stream to be disposed of [2,23].

Recently, Bear Creek Services (Shreveport, LA) and Hydration Technology Innovations (HTI) (Albany, OR) developed a new oilfield wastewater reclamation system. The Green Machine utilizes HTI’s FO technology in osmotic dilution mode for treatment of wastewater generated during O&G exploration [24]. The Green Machine enables drilling operators to reclaim water for reuse and thereby reduce the costly and environmentally disruptive transportation methods currently used to bring water to the field and to dispose of wastewater offsite. The Green Machine is currently being operated in the Haynesville Shale exploration play in North Louisiana and East Texas. For each well, more than 80% of the drilling waste can be treated, providing more than 20% of the water required for hydraulic fracturing of new wells. Although this system is successful in providing reclaimed water for hydraulic fracturing, understanding the solute transport in FO and optimizing system operation is necessary for wider application of the technology.

### 2. Solute transport in FO and its implication in treatment of O&G exploration wastewater

In FO, solutes permeate very slowly through the membrane from the feed to the draw solution (forward diffusion) – very similar to solute permeation during RO or NF. Yet, in FO, solutes from the draw solution simultaneously diffuse through the membrane into the feed stream (reverse diffusion) – a phenomenon known as reverse solute transport [25–27]. This phenomenon occurs because of the high concentration difference between the draw solution and the feed solution. Both forward and reverse solute fluxes in FO have major implications on process performance and on subsequent utilization of the product water (i.e., concentrated feed or diluted draw solution streams). Specifically, in FO and osmotic dilution, diffusion of draw solution solutes into the feed stream can decrease the driving force, and thus the water flux, and diffusion of feed solution solutes into the draw solution (e.g., heavy metals or organic compounds) can limit the use of the diluted draw solution in specific applications (e.g., fracturing of new O&G wells) [25,27].

In this study, the FO process operated in osmotic dilution mode was investigated for concentration of drilling wastewater from shale gas wells. The main objective of this study was to evaluate and optimize process performance using O&G waste streams and different operating conditions. Specifically, water flux, solute transport, and organic and inorganic compounds rejection by the FO membrane were investigated under extreme solution chemistry. In addition we explored the limitations of standard chemical analytical methods used for drinking/wastewater to quantify the concentrations of specific constituents in the feed and draw solution streams.

### 2. Materials and methods

#### 2.1. FO membrane

A semipermeable FO membrane was acquired from HTI. The membrane is made of cellulose triacetate (CTA) polymer cast over a fine polyester support mesh and is approximately 50 μm thick [15]. Many previous studies have utilized this FO membrane, which can efficiently operate in harsh conditions (e.g., high organic matter and total suspended solids concentrations) [9,10,17,28].

#### 2.2. Bench scale test system

Bench-scale tests were performed to investigate water flux, solute transport and rejection, and fouling and cleaning of the FO membrane. A custom-made FO test cell with symmetric flow channels on both sides of the membrane was utilized. The flow channels on each side of the membrane (2 mm deep) were further divided by nitrate rubber gaskets into narrower channels to provide adequate support of the membrane without the use of mesh spacers in the flow channels. The effective surface area of the membrane was 1050 cm² (17.5 cm wide and 60 cm long).

A flow schematic of the test apparatus is illustrated in Fig. 1. Feed and draw solution tanks, each with a capacity of approximately 5 L, were connected to a peristaltic pump (Masterflex L/S Digital Drive, Cole Parmer, Vernon Hills, IL) that circulated the feed and draw solution on opposite sides of the membrane at a rate of 1 L/min (equivalent to a flow velocity of 0.075 m/s). A set of experiments with a higher flow velocity (0.3 m/s) was conducted to explore flow velocity effects on membrane fouling. The draw solution tank was positioned on an analytical balance (Model S-8001, Denver Instruments, Bohemia, NY) connected to a SCADA system (LabVIEW, National Instruments, Austin, TX; and Labjack, Lakewood, CO) and the rate of change of the draw solution weight was recorded and used for calculation of water flux through the membrane.

![Fig. 1. A schematic drawing of the forward osmosis bench-scale test unit operated in osmotic dilution mode. Initial draw solution concentration was 260 g/L in all experiments and feed and draw solution flow rate was 1 L/min.](image-url)
2.3. Experimental procedures

Batch experiments with different membrane cell orientations were performed (Fig. 2). The first set of experiments was conducted with the membrane cell positioned horizontally with the feed side (active layer) facing up. To reduce settling of particles on the membrane surface, the membrane orientation during the second and third sets of experiments was changed. The second set of experiments was operated with the membrane cell positioned vertically and feed and draw solution flowing vertically upward, and the third set of experiments was operated with the membrane cell positioned horizontally and the feed side facing down.

The initial draw and feed solution volumes were 1 L and 5 L, respectively. The feed solution was a representative drilling wastewater acquired from a shale gas field in Northern Louisiana (Bear Creek Services, Shreveport, LA) and the draw solution was a concentrated sodium chloride solution (industrial un-graded salt) used in the Green Machine systems. The initial draw solution concentration was 260 g/L (similar to the operation of the Green Machine), and it became diluted during the experiments. The first and second sets of experiments proceeded until approximately 80% of the feed volume was recovered and the third set of experiments was conducted until approximately 50% of the feed volume was recovered. Temperature and conductivity of the feed were monitored (PCD 650, Oakton Instruments, Vernon Hills, IL) and recorded throughout the experiments. Water flux and change in feed conductivity with time were used for calculation of water recovery and reverse salt flux through the membrane from the draw solution into the feed.

2.4. Membrane cleaning

Although FO has the capability to treat highly contaminated waste streams such as O&G wastewater, membrane fouling can occur and inhibit process performance by reducing water flux across the membrane. Several studies demonstrated successful restoration of water flux through FO and RO membranes by applying an osmotic backwashing cleaning technique [10,29–31]. In osmotic backwashing the direction of water permeation across the semipermeable membrane is reversed, thus effectively removing foulants that accumulated on the membrane surface. In most cases, this cleaning technique is performed without the use of chemicals.

Solids in the feed solution slowly deposited on the membrane during all experiments. At the end of the first and second experiments, the concentrated feed solution was replaced with deionized water that was recirculated on the membrane and partially removed the cake layer by hydraulic flushing. Osmotic backwashing membrane cleaning was performed after the third set of experiments to enhance cake layer break-up on the feed side of the membrane. In this approach, the diluted draw solution was replaced with deionized water at the end of the experiment and continuously circulated on the support side of the membrane, while the feed solution was kept the same. Employing a reverse osmotic pressure gradient (feed salinity higher than that of the deionized water), osmotic backwashing was performed before flushing of the feed side with clean water.

2.5. Analytical methods

Feed and draw solution samples (250 mL each) were drawn from the feed and draw solution reservoirs at the beginning and end of each experiment. Samples were stored at 4 °C until analyzed and were acidified with nitric acid to a pH of 2 for cation analysis only. Samples were diluted and filtered through a 0.45 µm filter and analyzed for anions using ion chromatography (Model ICS-90, Dionex, Sunnyvale, CA). EPA digestion method 3050B was used to prepare the samples for cation analysis using inductively couple plasma optical emissions spectrometry (Optima 5300 DV, PerkinElmer Inc., Waltham, MA). The digestion method was chosen because it is effective in dissolving almost all elements that would become environmentally available. Chemical oxygen demand (COD) and dissolved organic carbon (DOC) concentrations were quantified with a COD reagent kit (TNT822, Hach Co., Loveland, CO) and a TOC analyzer (TOC 500, Shimadzu Corp., Kyoto, Japan), respectively. Samples were also analyzed for organic compound classification using ultraviolet radiation (DU 800 UV Spectrometer, Beckman Coulter, Brea, CA) and a spectrofluorometer (FluoroMax-4, HORIBA Jobin Yvon, Edison, NJ). All samples (both feed and draw solution) were diluted to levels suitable for analysis for the different analytical instruments.

Fluorescence spectroscopy analyses were also performed on draw solution samples before and after the experiments. Fluorescence spectroscopy produces an excitation–emission matrix (EEM) that is visualized on a 3-dimensional plot, and is a physical method to qualitatively analyze for organic matter. A common water sample EEM is shown in Fig. 3 (adapted from [32]). Previous studies identified peak A as humic acid- and fulvic acid-like material occurring in natural organic matter derived from plant material, peak C as humic acid-like, and peak T as tryptophan and protein-like material related to biological activity [32]. All EEMs show strong linear features that bound the optical area of the sample at the upper left and lower right corners. These linear features are the result of the Rayleigh–Tyndall effect that occurs when source excitation energy is reflected off the cuvette walls [33].

3. Results and discussion

3.1. Process performance

Baseline osmotic dilution experiments were conducted with deionized water as the feed and 260 g/L NaCl as the draw solution. Water flux, feed conductivity, and draw solution concentration as a function of time are illustrated in Fig. 4. The draw solution was diluted to approximately 50 g/L NaCl during the first and second experiments.
and to approximately 75 g/L NaCl during the third set of experiments. Water flux declined rapidly due to decline of the osmotic pressure driving force—it can be seen in Fig. 4 that the decrease in water flux corresponds to the decrease in draw solution concentration. The feed water conductivity increased to 4.4 mS/cm (2.5 g/L NaCl) over the duration of the experiment, which lowered the osmotic driving force and slightly contributed to flux decline. In a previous study, using the same HTI membrane and flow velocities, but with a draw solution of 50 g/L NaCl and a feed solution of deionized water, the water flux through the membrane was approximately 7 L/m²·h [25].

Initial water flux during the first experiment with drilling wastewater feed (operated until approximately 80% feed volume recovery) was approximately 14 L/m²·h and it decreased to a final water flux of approximately 2 L/m²·h as the draw solution concentration declined (Fig. 5a). The initial water flux for the second experiment was 12 L/m²·h, which is slightly lower than the initial water flux for the other two experiments, and the final water flux (~2.5 L/m²·h) was slightly higher compared to that in the first experiment (Fig. 5b). The water flux for the third set of experiments (operated until 50% feed volume recovery) followed the same trends as in the first two experiments, and the final water flux declined to approximately 4 L/m²·hr (Fig. 5c). The temperature of the feed and draw solutions gradually increased during the experiments because of heat that was generated and dissipated from the pump; however, because the temperature difference was low (less than 4 °C), water flux was not corrected for temperature.

For all three experiments the initial water flux was substantially lower than the initial flux in the baseline experiment (Fig. 4). This was due to the combined effects of high salinity and viscosity of the feed solution, and the high concentration of suspended solids in the feed stream. Water flux decline during the experiments with drilling wastewater feed was faster due to the combined effects of membrane fouling, increase of feed salinity, and decrease of draw solution concentration.

In an attempt to reduce membrane fouling, the membrane cell was oriented horizontally with the feed side of the membrane facing down during the third set of experiments. As illustrated in Fig. 5d, the flux at the end of the third experiment was very similar to the flux of the first and second experiments after 3 h of operation (approximately 45% water recovery). These results indicate that membrane cell orientation plays a minimal role in controlling water flux in this treatment application, and that solution chemistry and hydrodynamics most likely have a larger role in process performance.

Experiments were conducted at higher (0.3 m/s) and lower (0.075 m/s) feed and draw solution flow velocities; two of the experiments were performed with deionized water feed while the other two were performed with drilling wastewater feed. Results in Fig. 6 illustrate that flux decline was slightly slower due to increased membrane surface scouring and reduced concentration polarization at the feed-membrane interface when flow velocities were higher for experiments with deionized feed water. When drilling mud was used as the feed, flux was substantially lower, and the rate of flux decline was faster when feed flow velocity was slower. These observations are further supported by a recent study published by Boo et al. [34], which concluded that increasing cross-flow velocities can reverse colloidal fouling on FO membranes.

Because the experiments were operated in osmotic dilution mode, the osmotic pressure difference between the two streams decreased and thus the driving force for water permeation also decreased. Fouling on the membrane surface was also observed and contributed to flux decline because of the hydraulic resistance created on the membrane surface. Similar effects of solids precipitation on the membrane surface and their effects on water flux were observed in past studies [10,17,22].

### 3.2 Solute transport during osmotic dilution

While FO membranes are capable of rejecting all particulate matter and almost all dissolved contaminants from impaired streams, some low molecular weight organic and inorganic solutes may diffuse through the FO membrane and may affect upstream or downstream processes or environments [25]. Results from the experiments indicate that the feed conductivity increased over time due to the simultaneous concentration of the feed and reverse diffusion of the salts from the draw solution into the feed. Results from the third experiment are summarized in Table 1 and show that although 50% of the water was recovered, and an insignificant amount of minor ions diffused from the feed into the draw solution, the concentration of these minor ions in the feed solution did not double as expected. This is most likely the result of partial precipitation of many constituents on the membrane surface and in the feed stream.

Results from analysis of the two streams indicate that the most prevalent ions in the draw solution (Na⁺, Cl⁻, SO₄²⁻) became diluted and the most prevalent ions in the feed (Al³⁺, Ba²⁺, Ca²⁺, Fe³⁺, K⁺, Mg²⁺, and Si) did not contaminate the draw solution. Additionally, all EPA primary drinking water regulated constituents were below the maximum contamination level [35]. Therefore, the diluted draw solution can be safely and beneficially used for well fracturing or further desalinated to produce purified water.

When a mass balance is conducted on the feed side, results from the ionic composition of the samples indicate that approximately

![Fig. 3. EEM of a water sample showing common peaks. Previous studies identified peak A as humic acid-like and fulvic acid-like material occurring in natural organic matter derived from plant material, peak C as humic acid-like, and peak T₁ as tryptophan and protein-like material related to biological activity. Adapted from Zepp et al. [33].](image3)

![Fig. 4. Water flux, feed conductivity, and draw solution concentration as a function of time during baseline osmotic dilution experiment conducted with deionized water feed and 260 g/L NaCl draw solution.](image4)
1300 mg TDS were added to the feed stream, whereas results from conductivity measurements indicate that only 1150 mg TDS were added to the feed stream. Considering that 2.5 L of water permeated through the membrane, the specific reverse salt flux based on feed mass balance is approximately 500 mg/L, which signifies the mass of TDS lost from the draw solution to the feed (in milligram) for every liter of water recovered from the feed (Table 1). This value is close to what is expected from an HTI membrane [22]; however, it is likely that due to precipitation of minerals in the feed stream, the mass balance (based on dissolved solids) on the feed is underestimated. Evaluation of the feed mass balance based on the conductivity measurement yields a specific reverse salt flux of approximately 460 mg/L. Based on the two TDS measurements, and considering the extreme operating conditions in the current tests, the difference in reverse TDS flux through the FO membrane is very small and can be considered negligible. This claim is further supported by past experience [22], where the specific reverse TDS flux ranged from 400 to 500 mg/L for experiments performed with draw solution concentrations of NaCl ranging from 0 to 70 g/L and feed streams of various wastewater effluents.

Table 1

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<tr>
<th>Inorganic composition of the feed and draw solutions at the beginning and end of the third experiment.</th>
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<td><strong>Initial</strong></td>
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<td>Boric acid</td>
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Fig. 5. Water flux, feed conductivity, and feed temperature as a function of time for (a) the first experiment with membrane feed side facing up, (b) the second experiment with membrane cell oriented vertically, and (c) the third experiment with the membrane feed side facing down (see Fig. 2). Water flux as a function of water recovery for the three experiments is summarized in (d).

Fig. 6. Water flux as a function of water recovery for experiments conducted with deionized water (DI) feed or drilling wastewater (DW) feed, with higher (0.3 m/s) and lower (0.075 m/s) feed and draw solution flow velocities.
Furthermore, when considering the high initial and final concentrations of the feed stream, the effect of reverse salt flux on process performance (especially water flux) is very minimal.

3.3. Rejection of organic constituents by FO membrane

Several methods were used to evaluate the rejection of organic carbon, including measurement of COD, DOC, and fluorescence spectroscopy in conjunction with ultraviolet absorption. COD measurements of draw solution samples show a decrease in concentration from 7.6 mg/L in the concentrated draw solution to 1.8 mg/L in the diluted draw solution, indicating that the COD concentration in the draw solution declined during the process because of dilution and rejection by the FO membrane of small organic matter (that is easily oxidized by the COD method).

DOC analysis of draw solution samples showed an increase in concentration from 2.49 mg/L in the concentrated draw solution to 3.66 mg/L in the diluted draw solution, indicating that the DOC mass in the draw solution increased during the experiment from 2.49 mg to more than 12 mg (equivalent to 4.82 mg/L in the permeating water). DOC analysis was conducted using a combustion method, and therefore it is likely that the organic compounds that diffused through the membrane are more complex and are not easily digested/oxidized (during COD analysis) but are easily combusted. Still, when considering the feed DOC concentration (ranging from 219.7 mg/L at the beginning of the experiment to 552.4 mg/L at the end of the experiment), the average rejection of DOC by the membrane used in this study was higher than 99%; this value is further supported by previous studies [10,29]. EEM results of draw solution from the first and second sets of experiments (Fig. 7a–d) reveal that large molecular weight protein-like constituents are initially present in the draw solution and are almost completely absent in the diluted draw solution at the end of the experiments. It is also likely that no additional loading from the feed solution occurred. However, throughout the experiment, the draw solution absorbs humic acid-like and fulvic acid-like compounds, which are most likely small molecular weight organic compounds that diffused from the feed into the draw solution. The humic and fulvic acid-like compounds are more pronounced in the second experiment, likely because of the increase in diffusion during the slightly prolonged experiment.

3.4. Membrane cleaning

Solids in the feed solution slowly deposited on the membrane during all experiments. At the end of the experiments the concentrated feed solution was replaced with deionized water that was recirculated on the feed side of the membrane. After approximately 30 min, all the deposited solids were removed from the membrane surface (Fig. 8). A modified osmotic backwash was performed after the third experiment in attempt to more efficiently remove the cake layer that accumulated on the membrane surface. Modified osmotic backwashing was achieved by recirculating deionized water on the draw solution side and the concentrated drilling mud on the feed side. Throughout the cleaning process, water permeated from the support side of the membrane to the

![Fig. 7. EEM of the draw solution (a) at the beginning of the first experiment, (b) at the end of the first experiment, (c) at the beginning of the second experiment, and (d) at the end of the second experiment.](image-url)
active side of the membrane, removing the cake layer that formed on the active side of the membrane. Water flux was restored using both methods of membrane cleaning; however, the modified osmotic backwashing was more efficient in breaking up the fouling layer that formed on the membrane than simply recirculating water on the feed side of the membrane. This was reflected by the shorter time (~30%) of water recirculation on the feed side to achieve complete removal of the fouling layer from the membrane surface.

4. Conclusions

Results from experiments and water analysis show that FO operated in osmotic dilution mode can effectively concentrate feed solids more than three times their initial concentration and recover more than 80% of the volume from drilling wastewater. Water flux declined throughout the experiments because of the reduced driving force and due to membrane fouling. Reverse salt flux had minimal influence on process performance.

The process was able to recover water from the drilling wastewater with minimal irreversible membrane fouling. Osmotic backwashing membrane cleaning was an effective technique to remove the fouling layer and restore the initial water flux without need of chemicals. The FO membrane demonstrated very high rejection of both inorganic ions and organic compounds; therefore, the process can produce water and protect receiving environments from drilling/fracturing waste contamination. Overall, it was demonstrated that FO is an effective technology for concentration of drilling waste-water, facilitating water reuse for fracturing operation and reducing the need for an additional water source.

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