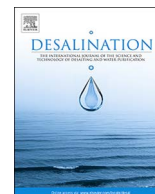




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Produced water impact on membrane integrity during extended pilot testing of forward osmosis – reverse osmosis treatment

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ABSTRACT

Forward osmosis (FO) has proven to be a robust, low-pressure membrane separation process capable of rejecting a broad range of contaminants; thus, providing a high quality diluted brine suitable for further desalination by reverse osmosis (RO). In this study, a pilot-scale FO-RO system treated > 10,000 L of raw produced water from the Denver-Julesburg basin (Colorado) over a four-week period using commercially available FO and RO membranes. Overall, the FO-RO pilot system maintained > 99% rejection of nearly all measured ions and > 95% rejection of hydrocarbons such as semi-volatile linear aliphatic hydrocarbons and polycyclic aromatic hydrocarbons. Although the FO-RO system was able to treat raw produced water, high concentrations of organic compounds severely fouled the FO membrane and substantially reduced water flux by 68% within 21 days. Membrane degradation due to interaction between organic constituents such as aliphatic and aromatic hydrocarbons and the membrane polymer may have compromised the FO membranes, resulting in substantial increase ($\times 15$) in reverse salt flux within 21 days. Further investigations of membrane cleaning and pretreatment will be required in order to better understand the overall economic feasibility of treating raw produced water using FO.

1. Introduction

The quality of treated water is one of the main factors dictating the cost and complexity of water treatment, especially in the oil and gas (O&G) industry. The location where treatment occurs and the duration of operation are additional factors that limit the ability to employ certain technologies and the cost of their utilization [1]. O&G wells are often in rural areas and may be far from existing water treatment and disposal facilities. Off-site treatment of upstream O&G wastewater may involve large-scale operations—it takes hundreds of truckloads to haul wastewater from one well during exploration and production (E&P) activities [2,3]. Truck-traffic related CO₂ emissions were estimated to be in the range of 26–40 tons of CO₂ for the transportation of water and 12–18 tons of CO₂ for wastewater hauling per well, with their total accounting for approximately 14% CO₂ emission for a single hydraulically fractured well [4,5]. Hydraulic fracturing typically uses 1500–19,000 m³ (9500–120,000 bbl) of water and many chemicals that serve as biocides, corrosion inhibitors, cross-linkers, and surfactant. 5–50% of the injected water returns to the surface as flowback water [6,7]. After

several weeks of flowback, water from the geological formation begins flowing out with the oil and/or gas, and is known as produced water. This study focuses solely on the treatment of produced water. Produced water is typically difficult to treat due to the presence of high concentrations of dissolved organic carbon, metals, and salinity in these streams [8–10]; thus, combinations of different processes are required to achieve the treatment objectives [1]. Many technologies used for treatment of O&G waste streams are energy and chemical intensive, and most membrane processes have operational limitations (e.g., membrane fouling, scaling, and low throughput) [8,11]. Thus, technologies are sought that can sustainably reclaim O&G waste streams and enable their reuse on-site at low capital, operation, and maintenance costs.

Engineered osmosis, and specifically forward osmosis (FO), is an emerging desalination technique that can provide robust and modular treatment, reject contaminants found in O&G waste streams, and avoid the drawbacks of pressure driven membrane processes. Engineered osmosis might be a promising alternative for difficult to treat waste streams such as flowback water, produced water, and drilling muds [12–15]. In some cases, FO can be used as a standalone desalination

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process or considered an advanced pretreatment process for reverse osmosis (RO) or other desalination processes [16–18].

Several studies have explored the fouling propensity and contaminant rejection of FO membranes during treatment of produced waters [13,19–21]; however, only few have evaluated changes in polymeric membrane transport and physicochemical properties after exposure to hydrocarbon-laden feed waters over an extended period of time [12,22,23]. Inorganic and organic constituents commonly present in produced water may compromise membrane integrity during extended periods of exposure. Hence, quantitative analysis of individual organic compounds or compound groups is important to assess the water quality of produced water after membrane treatment [24]. Furthermore, the performance of membrane treatment processes must be thoroughly evaluated, because different organic chemicals possess different physicochemical properties and therefore result in different rejection by the membranes.

In this pilot study, commercial spiral-wound FO membranes made of cellulose triacetate (CTA) were used to pre-treat raw produced water upstream from a high-pressure RO system, replicating previous bench-scale experiments [12,13,22]. The transport of organic and inorganic constituents and the changes to active layer properties as a function of produced water exposure time and water recovery rates were studied for both the FO and RO membranes. Pilot testing of the FO-RO system with produced water from the Denver-Julesburg (D-J) basin (Colorado, United States) was conducted in a preliminary investigation over a period of eight weeks in 2015 [24], and a second investigation was conducted over a period of four weeks in 2016. Here we focus on the results from the 2016 investigation, which evaluated membrane performance and integrity under extended produced water exposure time (> 600 h of continuous operation). Membrane performance parameters included water flux, reverse salt flux (RSF, i.e., mass of draw solution (DS) solutes leaking into the feed stream per unit membrane area per unit time), and rejection of inorganic and organic compound (e.g., semi-volatile linear aliphatic hydrocarbons, polycyclic aromatic hydrocarbons (PAHs)) by the FO and RO membranes. Membrane autopsy was performed after completion of the experiments to investigate physicochemical changes to FO and RO membrane surfaces.

2. Materials and methods

2.1. System design and operation

The FO-RO pilot system consists of two membrane treatment skids, an FO system and an RO system (Fig. 1). The DS induces an osmotic pressure difference across semipermeable FO membranes to recover water from and concentrate a produced water feed stream. Two 4040 spiral wound CTA FO membrane elements (Hydration Technology Innovations (HTI), now known as Fluid Technologies Systems (FTS-H₂O), Albany, OR) with approximately 4.9 m² total membrane active area were employed; the membrane elements were manufactured with corrugated chevron feed spacers and triple layer tricot spacers in the DS channels. During operation, raw produced water from the D-J basin was circulated in series through the feed channels in the FO membrane elements at 68 L/min, while sodium chloride (NaCl) brine (inlet concentration of 1 M = 58.4 g/L) was recirculated through the DS channels in parallel at an inlet flow rate of 1.25 L/min. The diluted DS from the FO membranes was continually re-concentrated by the downstream RO system and returned in a closed loop to the FO system. The RO system employed three RO membrane elements (SW30–2540, Dow Filmtec) operated in series. The fully automated RO system was operated at an average feed pressure of 5.9 MPa (850 psi) with a feed flow rate of 5.7 L/min. The FO-RO pilot system was controlled by a programmable logic controller (UE9-Pro, LabJack Corp., Lakewood, CO) and a data acquisition and instrument control software (LabVIEW, National Instruments Corp., Austin, TX).

All experiments were conducted in batch mode—760 L (200 gal) of

raw produced water recirculated in the FO system. In contrast to the 2015 testing [24], the concentrated produced water was not drained from the system and no fresh raw produced water was added to the feed tank. Instead, RO permeate generated by RO stage 1 (Fig. 1a) was returned to the FO feed tank. The RO permeate was collected in a 100 L tank, and once the water in the tank reached a predetermined level, the RO permeate was drained into the FO feed tank. The overall time span of the experiment was 28 days and included 5 phases as summarized in Table 1.

Following the increase of pH during the first phase, and the need to keep the pH below 7 in order to protect the CTA FO membranes, a 0.1 M hydrochloric acid (HCl) solution was automatically dosed into the DS to keep pH at 6.5. The RO system was designed to automatically produce DS of 1 M NaCl. Therefore, due to the unavoidable RSF through the FO membranes and salt leakage through the RO membranes into the RO permeate, the RO feed tank lost (over-produced) 10–20 L of water every day in order to maintain reject concentration of 1 M NaCl. Therefore, 10–20 L of 1 M NaCl solution was added to the RO feed tank once a day. At the end of each experimental phase (except first phase), the RO membranes were rinsed with deionized water until water leaving the membranes had an electric conductivity below 1 mS/cm. Then a sodium hydroxide solution at a pH of 11 was recirculated through the membranes followed by an acid cleaning with HCl solution at a pH of 2. The cleaning steps lasted 1 h each. Lastly, the DS was replaced with a fresh solution before beginning a new experimental phase. The FO membranes were cleaned only once between phases 4 and 5 of the study. During cleaning, an ethylene di-amine tetra acetic acid (EDTA) solution (100 g/L) was circulated through the feed channels of the FO membranes for 3 h. Citric acid was added to the EDTA solution as needed to ensure that the pH was below 6.5.

2.2. Membrane integrity testing

The integrity of the FO membranes (via pure water flux testing) was determined at the beginning of the study, at the end of phase 4 (i.e., before membrane cleaning), at the beginning of phase 5 (i.e., after membrane cleaning), and at the end of phase 5. Deionized water was recirculated on the feed side of the FO membranes and a 1 M NaCl solution was recirculated through the DS channels. Water flux through the FO membranes was calculated from the change in volume of water in the feed tank divided by the FO membrane area and the experimental time. A conductivity probe was installed in the feed tank and the electric conductivity of the feed was converted to total dissolved solids (TDS) concentration using a calibration function in the SCADA system. The TDS data was used to calculate RSF.

2.3. Sampling and chemical analyses

2.3.1. Solution chemistry

A single, large batch of produced water from the D-J basin was used for all experiments in this study. The produced water was gravity separated in the field and no further pretreatment was conducted before its use in the FO system. Results from chemical analyses of the produced water are summarized in Table 2.

2.3.2. Pilot system water sampling

Multiple operating parameters of the FO and RO systems were monitored and recorded continuously by the SCADA system throughout the study. These include volumetric flowrates, pressures, temperatures, conductivity, and pH. The locations of the different probes are illustrated in Fig. 1a. During the experiments, water samples were collected for analysis from the FO feed, DS (RO reject), RO feed, and RO permeate from stage 1 of the RO system and stored at 4 °C until preparation and analysis.

Similar to the previous pilot testing [24], samples for gas chromatography–mass spectrometry (GC–MS) analysis were collected from the

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