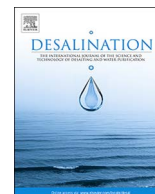




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Comparison of ceramic and polymeric nanofiltration membranes for treatment of abandoned coal mine drainage

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ABSTRACT

Performance of ceramic and polymeric nanofiltration membranes for treatment of abandoned mine drainage from a coal mine was investigated in this study. The increase in permeate recovery improved ionic rejection but reduced the permeability for both membranes. Arsenic was poorly rejected by both membranes with maximum rejection being 33% for the polymeric membrane. Fouling occurred at 75% permeate recovery and was dominated by gypsum scales. Chemical cleaning improved permeability but reduced ionic rejection indicating a slight increase in the effective membrane pore size for both membranes. When feed pH was adjusted to 4, ionic rejection increased for the ceramic membrane and decreased for the polymeric membrane due to impacts on the charge of the active layer. Addition of antiscalant improved ionic rejection for both membranes, especially for arsenic whose rejection improved by at least 141%, but resulted in about 40% decrease in permeability for both membranes and was attributed to the formation of a more complex and gel-like scale. A tighter polymeric nanofiltration membrane achieved > 99% rejection of all multivalent ions to meet all drinking water standards except for arsenic, which has to be removed prior to nanofiltration step.

1. Introduction

Membrane technology has been increasingly applied in wastewater treatment and desalination applications over the last few decades. Use of polymeric microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO) membranes has been growing exponentially owing to their wide set of separation characteristics for different applications [1]. Ceramic membranes have recently been gaining prominence due to better resistance to fouling, easier cleaning, lower maintenance, better thermal resistance and greater mechanical strength [2, 3]. Ceramic NF membranes are commonly made using the sol–gel technique with Al₂O₃, ZrO₂ or TiO₂ as the active layer, with the latter two preferred due to greater stability [4]. Ceramic membranes are typically available with molecular weight cut-off (MWCO) close to ~1000 Da and have been applied to remove organic molecules and natural organic matter (NOM) [5, 6]. Newer manufacturing techniques like atmospheric pressure atomic layer deposition (APALD) [7] and DNA template technology [8] are being studied to manufacture ceramic NF membranes with MWCO below 500 Da that could effectively reject multivalent ions. This study was designed to compare ceramic and polymeric NF membranes for treatment of abandoned or acid mine drainage (AMD) from a coal mine in terms of ionic rejection and fouling behavior.

AMD has been a major environmental concern over the past five decades as it is a highly contaminated stream with high acidity (pH 2–4), high sulfate concentration (0.1–20 g/L) and presence of potentially toxic elements such as Al, As, Ca, Cd, Cu, Fe, Mg, Mn, Ni, Pb and Se [9]. Most of these contaminants occur from the natural oxidation of sulfide minerals like pyrite (FeS₂), chalcocite (Cu₂S) and mackinawite (FeS) when in contact with water and oxygen [10, 11]. Because the conventional AMD treatment with limestone addition cannot achieve requisite effluent standards [12], other techniques for sulfate removal, including sulfate reducing bacteria [13] or cation exchange resin [14] have been investigated; however, these techniques are not widely accepted as they depend on the external carbon source and have high cost, respectively. Polymeric NF membranes have been investigated to achieve effluent standards [9, 10, 15–19] or even drinking water standards [16]. Commercially available NF membranes can reject > 95% sulfate with real AMD [17] and their performance depends on pH, temperature, operational conditions and feed quality [18, 19]. Two recent studies successfully tested polymeric NF membranes at pilot-scale and offered information on operational and maintenance costs [20, 21] and concluded that AMD pretreatment is essential prior to the use of NF membranes to treat AMD.

Ceramic NF membranes have been previously tested with simple

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synthetic solutions of NaCl, KCl and Na₂SO₄ [4, 22]. Chen et al. [22] found that ceramic NF membrane with a MWCO of 900 Da rejected 10% chloride and about 40% sulfate in addition to effectively rejecting various dyes. Gestel et al. [4] found that the rejection of simple monovalent and divalent ions was minimal at the iso-electric point (IEP) or point of zero charge (PZC) of the membrane. 85% rejection of NaCl and 95% rejection of Na₂SO₄ was reported at low and high pH.

Fouling has been a major concern with membrane treatment [23–27] and chemical cleaning is typically applied when the permeability decreases by about 10% or when the pressure drop increases by about 10–15% [26]. Therefore, it is critically important to understand both performance and fouling characteristics of ceramic NF membranes to ensure optimal performance for any application.

This study compared the performance and fouling characteristics of ceramic and polymeric NF membranes for AMD treatment. The performance was characterized by ionic rejection and permeability as a function of permeate recovery rates. Fouling and fouling mitigation strategies were investigated in terms of the type of foulants and the effects of pH adjustment and antiscalant addition on performance of the ceramic and polymeric NF membranes. The efficiency of chemical cleaning procedures for recovery of membrane performance was also evaluated in this study.

2. Experimental

2.1. Membranes and AMD

Ceramic nanofiltration membrane prototype (MWCO of ~500 Da) comprised of fused alumina and active surface layer of amorphous titania (TiO₂) and was provided by Cerahelix (Orono, ME). DNA template technology was used to make linear and identical pores with typical size of 1 nm [28]. Polypiperazine amide membrane (NF270) with MWCO of ~200–400 Da (Dow Filmtech, Edina, MN) and polyamide membrane (TS80) with MWCO of ~150 Da (Trisep, Goleta, CA) were also used in this study. Pore radius of 0.87 ± 0.02 nm and 0.71 ± 0.02 nm for NF270 and TS80 membranes, respectively have been measured using the membrane potential technique [29]. Water permeability tests were conducted with deionized (DI) water obtained from MilliQ water system (Millipore, Billerica, MA). Dilute NaOH, HCl and Na₂SO₄·10H₂O were purchased from Fisher Scientific (Pittsburgh, PA). Antiscalant RL9004 used for fouling mitigation was purchased from ChemTreat (Glen Allen, VA). Dilute NaOH and HCl were used for pH adjustment.

AMD was collected from a site in southwestern Pennsylvania. The actual AMD had about 60 mg/L of total dissolved iron, which had to be removed before nanofiltration to prevent severe fouling. Hence, 20–24 h of aeration followed by filtration through 0.22 μm membrane preceded all NF tests. The composition of AMD post aeration and microfiltration is given in Table 1.

2.2. Module and experiments

All NF experiments with polymeric membranes were carried out in the laboratory-scale test cell SEPA-CFII (GE Osmonics, Minnetonka, MN) with a usable membrane area of 140 cm² [30]. Pristine polymeric membrane used for each experiment was immersed in DI water for at least 24 h to ensure complete wetting of membrane pores. Each polymeric membrane was first exposed to DI feed pressure of 50 bar for 1 h to ensure no compaction effects during testing and DI water permeability was measured at experimental pressures for membrane integrity testing. Ceramic membrane tests were conducted in the same system except that the SEPA-CFII module was replaced with the housing designed for a single channel tubular membrane with diameter of 6 mm and length of 500 mm (total membrane area of 100 cm²). Unlike polymeric membranes, no compaction of ceramic membranes was necessary prior to testing with AMD.

Assessment of recovery rate: The first step in this study included the

Table 1
Characteristics of AMD post aeration and microfiltration.

Ions	Concentration
Sulfate (mg/L)	645.9 ± 2.5
Chloride (mg/L)	97.8 ± 1.9
Sodium (mg/L)	108.9 ± 4.2
Calcium (mg/L)	151.8 ± 2.1
Magnesium (mg/L)	29.7 ± 1.1
Potassium (mg/L)	4.3 ± 1.6
Manganese (mg/L)	1.2 ± 0.6
Strontium (mg/L)	1.7 ± 0.3
Barium (μg/L)	76.7 ± 4.1
Aluminum (μg/L)	50.5 ± 1.2
Nickel (μg/L)	38.5 ± 4.9
Arsenic (μg/L)	70.0 ± 6.4
Selenium (μg/L)	55.2 ± 3.9
Total iron (mg/L)	< 0.02
pH	7.8 ± 0.2

assessment of the effect of permeate recovery rate (i.e., 0%, 50% and 75%) on membrane performance. The feed tank was filled with 20 L of AMD and was allowed to stabilize for 2 h with total recirculation when samples corresponding to 0% recovery were collected. The system was monitored for the next 24 h to collect transient permeability and conductivity rejection data at 0% recovery. After that, 10 L of permeate was collected to achieve a 50% recovery rate. The ceramic membrane was then chemically cleaned and the polymeric membranes were replaced. Permeate samples were collected after 2 h of stabilization in total recirculation mode and transient permeability and conductivity rejection data were monitored for the next 24 h. After that, 5 L of additional permeate (i.e., a total of 15 L permeate) was removed from the system and membranes were either cleaned (ceramic) or replaced (polymeric) prior to collecting samples for 75% recovery after 2 h of stabilization period with total recirculation. Transient permeability and conductivity rejection data was also collected over the next 24 h.

Fouling analysis: Only one ceramic membrane was available and hence it had to be chemically cleaned prior to testing new process parameters. Unlike the ceramic membrane, a new polymeric membrane was employed each time since the used membrane underwent destructive analysis by scanning electron microscopy (SEM, JEOL JSM6510, Peabody, MA) and energy dispersive X-ray spectroscopy (EDS) to characterize the fouling layer.

Assessment of chemical cleaning: The effect of chemical cleaning on membrane performance was evaluated using chemical cleaning procedures shown in Table 2. The performance data were compared at 75% AMD recovery to evaluate the efficiency of chemical cleaning.

Fouling mitigation strategies: Two fouling mitigation strategies, namely pH adjustment and antiscalant addition, were evaluated in this study. Membrane performance with no antiscalant addition or pH adjustment at 75% AMD recovery was compared with that when feed pH was adjusted to 4 or when 15 mg/L of antiscalant was added to the feed. In each case, the membrane was stabilized for 2 h followed by permeate sample collection to determine ionic rejections and then measurement of transient permeability and conductivity data over the next 24 h period.

All experiments were conducted at a constant pressure of 35 bar, temperature of 25 ± 1 °C and feed flow rate of 5.68 LPM unless otherwise indicated. This feed flow rate corresponds to a crossflow velocity of 1.16 m/s with the flat sheet polymeric membranes and 3.35 m/s with the tubular ceramic membrane. The rejection of various ions was calculated as:

$$R(\%) = \frac{c_i^{feed} - c_i^{permeate}}{c_i^{feed}} \times 100$$

where, R is the observed rejection and c_i^{feed} and $c_i^{permeate}$ are the concentrations of ion 'i' in the bulk feed and the bulk permeate,

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