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Role of naphthenic acid contaminants in the removal of *p*-xylene from synthetic produced water by MEUF

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ABSTRACT

The high demand for water in oil sands operations in addition to the shortage of fresh water resources mandate continuous search for reliable and cost effective water treatment technologies. Micellar-enhanced ultrafiltration (MEUF) is potentially applicable to produced water treatment. MEUF is, however, susceptible to membrane plugging and back contamination, especially at high surfactant dosages. Recent investigations showed that addition of small amounts of a non-ionic surfactant to an ionic surfactant solution reduces the critical micellar concentration (CMC) of the mixed surfactant solution, and consequently reduces the surfactant dosage required to achieve certain removal of contaminants using MEUF. In the present study a naphthenic acid, octanoic acid, typically existing in produced water is shown to play a similar role as the non-ionic surfactant when added to the cationic surfactant cetylpyridinium chloride (CPC). Cross-flow MEUF using three different molecular weight cutoffs (MWCs) of polyacrylonitrile (PAN) hollow fiber membranes was used to treat synthetic produced water containing *p*-xylene. The mixed CPC/octanoic acid solution removed comparable percentages of *p*-xylene contaminant at much lower concentrations of CPC. Decreasing CPC concentration in the feed resulted in less fouling and higher permeate flux and reduced back contamination. This, in turn, enhanced the performance of MEUF.

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Keywords: Oil sands; Produced water; Octanoic acid; *P*-xylene; Micellar-enhanced ultrafiltration; Cetylpyridinium chloride

1. Introduction

Produced water is the largest waste stream co-produced during oil extraction (Ali et al., 1998; Khatib and Verbeek, 2003). In thermal heavy oil recovery operations, e.g. oil sands processing and steam assisted gravity drainage (SAGD), typical water to oil volume ratio of 3–12 leads to large volumes of produced water (Hum et al., 2006). This high volume ratio imposes a big strain on fresh water resources. The treatment and beneficial recycling of millions of barrels of produced water is essential for the sustainability of oil recovery operations from oil sands. Moreover, organic contaminants in produced water may cause severe environmental and industrial problems. These contaminants have adverse effect on industrial operations; including scaling and corrosion in pipelines (Simms et al., 1992; Cline, 1998; Quagraine et al., 2005). Furthermore, drinking water sources and aquatic life may be negatively affected if such water is disposed of without proper treatment (Cline, 1998;

Veil et al., 2004). Oil and grease is the widely used parameter for characterizing the quality of produced water. Simms et al. (1992) have reported a series of specific components of produced water to be quantified as oil and grease. These components are carboxylic and naphthenic acids, normal aliphatic compounds, aromatic compounds (BTEX: benzene, toluene, ethyl benzene and xylenes) and sulphur and nitrogen containing hydrocarbon derivatives. Octanoic acid, from the series of naphthenic acids, and *p*-xylene, an aromatic organic compound, which were employed as selected model components in this work, are major organic contaminants of produced water from oil sands processing (Janks and Cadena, 1992; Simms et al., 1992).

Many techniques are currently employed in oil sands processing sites in order to achieve produced water recycling (Thomas et al., 1987; Jan and Reed, 1989; Bridle, 2005; Heins and Peterson, 2005; Shpiner et al., 2007). Micellar-enhanced ultrafiltration, MEUF, has been shown to be a highly efficient

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and selective separation process for organic and heavy metal contaminants from industrial wastewaters (Dunn et al., 1985, 1989; Markels et al., 1995; Fillipi et al., 1999; Jadhav et al., 2001; Juang et al., 2003; Purkait et al., 2004, 2005, 2006; Lee et al., 2005; Yang et al., 2005). MEUF is a physicochemical separation process that couples the dissolution of organic contaminants within the micelles with physical treatment using membrane filtration (Dunn et al., 1989). MEUF is a relatively low energy, selective membrane-based separation process that can be scaled up easily to meet industrial operations (Fillipi et al., 1999). However, MEUF suffers from fouling, which reduces the permeate flux, and back contamination, which is the leakage of surfactant monomers to the permeate side of the membrane (Syamal et al., 1997; Kamble and Marathe, 2005; Beolchini et al., 2006).

Recent studies in the literature showed that addition of a non-ionic surfactant to ionic surfactant solutions significantly reduces the amount of surfactant dosage required to remove a given contaminant (Tung et al., 2002; Bielska and Szymanowski, 2004; Holmberg et al., 2004; Lee et al., 2005; Li et al., 2006). This, in turn, significantly improved the treated water flux and lowered back contamination. In the present work, the synergistic effect resulting from adding octanoic acid to cetylpyridinium chloride, CPC, during the removal of *p*-xylene was investigated. In more general terms, the work explores utilizing contaminants already existing in produced water to improve the removal of other organic contaminants while employing MEUF. Octanoic acid belongs to naphthenic acids which are commonly found in produced water (Yen et al., 2004; Quagraine et al., 2005). *P*-xylene is highly toxic and is categorized as one of the constituents of oil and grease in produced water (Simms et al., 1992). The choice of cetylpyridinium chloride as a surfactant for the current application stemmed from the fact that it has high dissolution capacity for organic contaminants and low critical micelle concentration (CMC) (Purkait et al., 2004, 2005). Moreover, electrostatic attraction between the negative electron cloud of the benzene ring in the structure of *p*-xylene and the positive head group, N^+ , in CPC helps attracting *p*-xylene to the surface of the CPC micelles (Kandori and Schechter, 1990).

2. Experimental

2.1. Materials

P-xylene (99 wt.% pure, Fisher Scientific, ON) and octanoic acid, $C_8H_{16}O_2$ (99 wt.% pure, Fisher Scientific, AB) were employed as model organic contaminants. Cetylpyridinium chloride (96–100 wt.% pure, Fisher Scientific, ON) was used as the surfactant. Methanol (99% pure, VWR, ON) was used to break the micelles and release *p*-xylene for the high-performance liquid chromatography (HPLC) measurements. Polyacrylonitrile (PAN) hollow fiber ultrafiltration membranes (Pall Corporation, ON) with three different MWCO of 6, 13 and 50 kDa were used. The membranes were identical, except for the MWCO and permeability, and their specifications are listed in Table 1. Hydrophilic membranes are desirable because they allow for easy passage of water molecules through the pores.

2.2. Experimental setup

Fig. 1 shows the cross-flow MEUF experimental apparatus used in this study. The feed stream was pumped from the feed

Table 1 – Hollow fiber membranes specifications

Length (cm)	34.7
Diameter of the membrane module (cm)	4.5
Inner diameter of the hollow fibers (mm)	0.8
Membrane effective surface area (m^2)	0.2
Permeability of 6 kDa membrane (kg/Ns) ^a	1.37×10^{-7}
Permeability of 13 kDa membrane (kg/Ns) ^a	4.79×10^{-7}
Permeability of 50 kDa membrane (kg/Ns) ^a	11.65×10^{-7}

^a Calculated from the manufacturer's data for virgin water flux at 100 kPa.

tank through a pulsation dampener to the membrane module using a peristaltic pump (Masterflex L/S, Cole-Parmer Instrument Co., QC). During the passage of the feed inside the hollow fibers, a permeate stream passes through the membrane pores by virtue of the pressure difference between the inside and the outside of the hollow fibers. The trans-membrane pressure, TMP, is the driving force for the permeate flux and can be calculated from Eq. (1), since the permeate side is open to atmosphere.

$$TMP = \frac{P_{in} + P_{out}}{2} \quad (1)$$

where P_{in} and P_{out} are the pressures across the membrane. The permeate, or treated water, flowrate was determined by collecting permeate samples in a volumetric flask over certain period of time. CPC concentration was measured using a HP 6890 series gas chromatography, GC (Agilent Technologies, ON), having HP-5 GC column (Agilent Technologies, ON). The oven temperature was increased from 80 to 230 °C at 10 °C/min ramp, and was maintained at 230 °C for 5 min. The injector and FID temperatures were 150 and 230 °C, respectively. Helium was used as the carrier gas. Samples of 0.5 μ L from the feed, permeate and retentate were used. The retention time for CPC peaks ranged from 11.7 to 11.9 min. The GC detection limit for CPC was 0.1 mM. A 1100 series high-performance liquid chromatography, HPLC (Agilent Technologies, ON) having Zorbax Eclipse XDB-C18 column (Agilent Technologies, ON) was used to determine the concentrations of *p*-xylene and octanoic acid in the feed, permeate and retentate. A 80:20 volume ratio of methanol/DI water solution was the carrier liquid. The carrier liquid flowrate was fixed at 1 mL/min, and the injection volume was 60 μ L. Peaks specific to octanoic acid and *p*-xylene were detected at 3.6 and 4.8 min, respectively. The detection limit of the HPLC for *p*-xylene and octanoic acid was 1 and 0.1 ppm, respectively. It is worth noting that no *p*-xylene peaks could be detected by HPLC when CPC micellar solution containing *p*-xylene was injected. It is believed that *p*-xylene molecules were encapsulated within the micelles and passed through the HPLC column without being adsorbed in

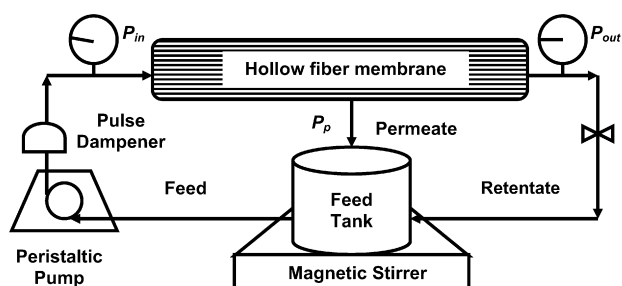


Fig. 1 – Micellar-enhanced ultrafiltration experimental setup.

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