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Comparison of two fractionation strategies for characterization of wastewater effluent organic matter and diagnosis of membrane fouling

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ABSTRACT

Two fractionation strategies were compared for characterizing organic components in effluent organic matter (EfOM) and natural organic matter (NOM). The first method is widely used and requires sample acidification and then re-neutralization during sequential organic removals onto resins. The second method uses a different suite of separation methods, does not require pH manipulation, and sequentially removes particles, colloids, organic acids, and hydrophobic neutrals without the need for adjusting pH. The NOM samples were dominantly organic acids while EfOM contained a broader distribution of organic functionalities so further evaluation was focused on EfOM. The new method completely removed colloidal matter from EfOM while the conventional fractionation method resulted in an increase in the percentage of EfOM >100 kDa with each fractionation step after filtration. Organic acids were removed in one fractionation step using the new method instead of three steps with the conventional method. The conventional method resulted in increased fouling after the final separation step apparently caused by production of inorganic colloids. The new fractionation method provided a clearer diagnosis of organic acids were the primary cause of fouling even though they were only 14% of EfOM organic carbon. We suggest that the new fractionation method should be considered for diagnosing the effects of NOM or EfOM on the performance of membrane filtration.

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1. Introduction

Natural organic matter (NOM) and wastewater effluent organic matter (EfOM) are complex mixtures of organic materials. The majority of NOM and EfOM is organic matter for which chemical structure cannot be uniquely identified but which can be grouped according to functional moieties (Croué, 2001; Hatcher et al., 2001; Filella, 2009). Consequently characterization of NOM and diagnostic work involving NOM often requires preliminary separation into component fractions using

operational strategies such as size separation and adsorption onto non-ionic resins or anion exchange resins. Typical but not universally accepted categories of NOM include strong hydrophobic organics (HPO), transphilic organics (TPI), charged anionic species (often denoted Char), and hydrophilic organics (HPI) with further identification as acids (a) and as base/neutral (b/n) compounds (Carroll et al., 2000; Fan et al., 2001; Jarusutthirak et al., 2002; Shon et al., 2006).

Fractionation strategies can be used to diagnose the effects of NOM components on water and wastewater treatment

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processes such as coagulation, membrane fouling, and formation of disinfection byproducts. This paper focuses on membrane fouling because there is disagreement about whether HPI b/n's or organic acids are the most important organic foulants. In prior work we concluded that short-term fouling of polyethersulfone (PES) ultrafiltration (UF) and polyvinylidene fluoride (PVDF) microfiltration (MF) membranes by EfOM was due to organic acids (Kim and Dempsey, 2008, 2010). HPI b/n's did not cause fouling in those experiments but they accumulated on membrane surfaces. Other investigators have also implicated organic acids as important membrane foulants (Crozes et al., 1993; Hong and Elimelech, 1997; Cho et al., 1999; Lin et al., 2000; Aoustin et al., 2001; Liang and Song, 2007) but several researchers have reported that HPI b/n's were the most important foulants in EfOM and NOM (Cho et al., 1998; Carroll et al., 2000; Fan et al., 2001; Lee et al., 2001; Koh et al., 2006; Zularisam et al., 2007). It was also reported in some of these papers that the concentration of colloids was unexpectedly high in the HPI b/n fraction (Carroll et al., 2000; Fan et al., 2001; Gray et al., 2008). Thus there remains controversy about which components of EfOM or NOM cause the most fouling.

The most commonly used fractionation strategy, denoted the conventional strategy in this paper, involves filtration to remove particles, acidification to pH 2 followed by adsorption of HPO and TPI components onto non-ionic resins, and then readjusting to neutral or slightly alkaline pH followed by anion exchange. Some problems have been noted with the conventional fractionation procedure (Leenheer, 1981; Aiken et al., 1992) including incomplete recovery of NOM organic acids by alkaline extraction and high colloid concentration in the final residual that should contain only dissolved HPI b/n compounds (Leenheer et al., 2000; Ma et al., 2001; Jarusutthirak et al., 2002; Croué, 2004). Possibly colloids can pass through the macro-reticular resins in the conventional method or perhaps new colloids are produced by coagulation of humic materials at pH 2 or by formation of inorganic precipitates when the pH is raised from 2 to about 8. These hypotheses are tested in this paper. It has also been noted that ester or ether functionalities in some resins could be hydrolyzed at pH 2, possibly resulting in addition of new organic matter (Abbt-Braun et al., 2004).

Several investigators have shown that weak base anion exchange resins efficiently remove organic acids and that the organic acids can be recovered with an alkaline eluent (Miles et al., 1983). Tuschall et al. (1985) reported that benzoic acid, resorcylic acid, and cinnamic acid were retained by diethylaminoethylcellulose (DEAE cellulose) at neutral pH and quantitatively released with a NaOH solution, while a variety of phenols were not retained. The organic acids were not released from the DEAE resin by eluting with up to 0.5 M NaCl, NaNO₃, or Na₂SO₄, demonstrating that DEAE cellulose is effective for separation of organic acids from other NOM and EfOM components.

Therefore the main objective was to compare the widely-used conventional fractionation method for separating organic constituents in NOM and EfOM with a new method that does not require manipulation of the pH and therefore might result in less chemical and physical changes in organic matter. Comparisons of the fractionation methods were based

on (1) characteristics of residual and recovered organics from the fractionation steps, (2) removal of metals during fractionation, and (3) effects of sequential removal of EfOM organics on fouling of membrane. PES UF and PVDF MF membranes were used. Three sources of organic materials were evaluated: a northern bog, bio-treated landfill leachate, and EfOM, with emphasis on EfOM.

2. Materials and methods

2.1. Sample collection and characteristics

Wastewater mixed liquor was collected from the University Area Joint Authority (UAJA) wastewater treatment facility in Centre County, PA. UAJA uses an anoxic-anaerobic-oxic process for biological nutrient removal and adds alum (downstream from the sampling location) for enhanced removal of phosphorus. Samples were settled for 1 h and then 60 L of supernatant (EfOM) was siphoned and immediately transported to the laboratory for fractionation. Surface water from a northern bog (Bear Meadows, Centre County, PA) and municipal landfill leachate that had been treated using extended aeration (Waste Management, Antrim, PA) were sampled, settled, and decanted in a similar fashion.

Some characteristics of the waters used in this study are shown in Table 1. The pH, conductivity, and total dissolved solids (TDS) concentrations were typical for waters from the three sources. Apparent molecular size distributions were determined by parallel sample processing using regenerated cellulose UF membranes with MW cutoffs between 100 and 1 kDa (YM100, YM30, YM10, YM3, and YM1, Millipore Corp., USA) and a 500 mL stirred cell (Cole Parmer, Model H-02910-42) pressurized by nitrogen gas at room temperature. Less than 50% of the initial volume of settled samples were filtered at $<50 \text{ L m}^{-2} \text{ h}^{-1}$ with constant stirring (Aiken, 1984; Kilduff and Weber, 1992). These procedures minimize concentration polarization effects, which at any rate are usually negligible in UF and MF (Yuan and Zydney, 2000). The total organic carbon (TOC) in each size category was calculated by difference, thus TOC in the 3–10 kDa range was the difference between permeate TOC's in the 10 and 3 kDa filters.

2.2. NOM fractionation procedures

The fractionation methods are described in Fig. 1a and b. For the new fractionation strategy, 20 L of sample was filtered using 1.0 μm glass-fiber filters. The filtered sample was sequentially applied to four columns.

The first column (colloid collector) was designed for removal of colloids $>20 \text{ nm}$ and contained a 2.5 cm depth of DEAE cellulose (Whatman diethylaminoethylcellulose) that was pre-saturated with commercial humic acid (Aldrich Chemical, Milwaukee, WI), washed with deionized water until no organic carbon passed, and acclimated by passing 20 L of sample at a surface loading rate of $1.0 \text{ L min}^{-1} \text{ m}^{-2}$ (0.1 cm min^{-1}) before conducting the fractionation. Less than 0.3% of the loaded humic acid was eluted when the column was eluted with 20 L of 50 mg L^{-1} sulfate solution. Subsequent

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