



Understanding the fouling of UF/MF hollow fibres of biologically treated wastewaters using advanced EfOM characterization and statistical tools

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HIGHLIGHTS

- Coupling of EEM and LC-OCD predicts the fouling potential of secondary effluents.
- Identification of the OM characteristics responsible for a high fouling potential.
- Correlation between protein content indicators and a high fouling potential.
- Differentiation of HS from terrestrial origin and HS produced in biological reactors.
- Impact of biological treatment on the EfOM composition by OM characterization.

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ABSTRACT

Five secondary effluents and a river water source were characterized using size exclusion chromatography (LC-OCD-UVD-OND) and emission–excitation matrix (EEM) fluorescence spectroscopy in order to identify the major effluent organic matter (EfOM) fractions responsible for membrane fouling. This study showed the feasibility of coupling fluorescence EEM and LC-OCD-UVD-OND to investigate the fouling potential as well as a means to differentiate natural organic matter (NOM) from EfOM. The secondary effluents and river water showed a significant difference in organic matter characteristics and fouling potential, highlighting the importance of biological processes and the feed water source on EfOM characteristics and fouling potential. On the basis of statistical analysis, protein-like substances were found to be highly correlated to the fouling potential of secondary effluents.

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1. Introduction

Treated sewages have been considered for wastewater reuse and/or reclamation to resolve water shortages. Wastewater

Abbreviations: EfOM, effluent organic matter; EEM, emission–excitation matrix fluorescence spectroscopy; NOM, natural organic matter; SMP, soluble microbial products; BP, biopolymers; HS, humic substances; BB, building blocks; LMW, low molecular-weight; OND, organic nitrogen detection; SUVA, specific UVA; UF, ultrafiltration; MF, microfiltration; CAS, conventional activated sludge processes; MBR, membrane bioreactor; TOC, total organic carbon; DOC, dissolved organic carbon; MW, molecular weight; LC-OCD-UVD-OND, liquid chromatography with organic carbon, UVA and organic nitrogen detections; FRI, fluorescence regional integration; Ex, excitation; Em, emission; UMFI, unified membrane fouling index; PCA, principal component analysis; CTOC, chromatographic organic carbon values; HOC, non-chromatographic organic carbon values.

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reclamation plants mainly use biological processes as secondary treatment for the removal of suspended solids, organics and nutrients followed by membrane filtration in order to produce high quality water suitable for reuse purpose. However, a major issue in wastewater treatment plant is membrane fouling. Membrane fouling results in reduction of membrane performance and the need for cleaning. Effluent organic matter (EfOM) has shown to play an important role in this phenomenon (Amy, 2008; Shon et al., 2006). Therefore, several studies aimed to characterize the composition of the EfOM to better understand the effects on membrane fouling (Jarusutthirak et al., 2002; Shon et al., 2006).

EfOM is described as a combination of natural organic matter (NOM) and soluble microbial products (SMP). NOM in waters is derived from plant or terrestrial (allochthonous) and algal (autochthonous) sources and it is composed of higher molecular weight refractory humic and fulvic substances, lower molecular weight proteins, organic acids, carbohydrates and other possible

anthropogenic compounds (Leenheer and Croué, 2003). SMP are complex organic compounds released through substrate metabolism as well as by biomass decay and mainly consist of proteins, polysaccharides (Rosenberger et al., 2006) and humic-like substances that have both anthropogenic (metabolite of surfactants) and biological origins (Drewes and Croue, 2002).

Due to the complex nature of both NOM and EfOM, advanced analytical tools, such as size exclusion chromatography coupled with organic carbon detection (LC-OCD) and the fluorescence emission–excitation matrix (EEM) are today proposed for their characterization (Drewes, 2006; Nam et al., 2008). An advantage of these methods is that comprehensive information regarding the nature of the organic matter (e.g. size, structure, functionality) can be provided with minimal sample preparation. LC-OCD is commonly applied to subdivide the pool of polar organic matter according to their molecular size and can be divided into five major fractions: biopolymers (BP), humic substances (HS), building blocks (BB), low molecular-weight (LMW) acids and LMW neutrals (Huber et al., 2011). Liquid-chromatography can be coupled with organic carbon detection, UV detection and fluorescence detection, providing both quantitative (DOC content) and qualitative (degree of aromaticity and fluorescence properties) information of fractions of organic moieties separated based on their molecular weight. Organic nitrogen detection (OND) was recently used to characterize the EfOM in terms of nitrogen content and estimate the nitrogen content of the BP fraction in natural water (Huber et al., 2011). Fluorescence spectrometry is used due to the fluorescence properties of dissolved organic matter and high instrument sensitivity (Henderson et al., 2009). Different spectral regions can be distinguished into humic-like and protein-like fluorophores associated with different types of functional groups (Chen et al., 2003).

These advanced analytical tools have been used to differentiate EfOM from NOM (Drewes and Croue, 2002; Nam et al., 2008). EfOM is characterized by distinct properties, such as lower specific UVA (SUVA), higher hydrophilic organic matter, increased fluorescence index values, higher polysaccharides peak in LC-OCD chromatogram, and clear protein-like peak in EEM, as compared to NOM (Nam et al., 2008). The same characteristics have been identified as indicators of the presence of potential NOM foulants (Lozier et al., 2008). Several studies demonstrated that the composition of EfOM varies with the origin of effluent which is most likely caused by the differences in treatment processes, influent wastewater composition and operational conditions (Esparza-Soto et al., 2006; Jarusutthirak and Amy, 2007).

LC-OCD and fluorescence EEM have been also used for the identification of EfOM characteristics responsible for membrane fouling in wastewater treatment plants (Haberkamp et al., 2011; Henderson et al., 2011; Zheng et al., 2010). The macromolecules (i.e. proteins and polysaccharides), identified as the hydrophilic fraction of EfOM have been found to exert severe fouling while humic substances, the so called hydrophobic fraction, were found to be of minor importance (Huang et al., 2007; Jarusutthirak et al., 2002; Shen et al., 2010; Zheng et al., 2010).

The objective of this study was to compare the ultrafiltration (UF) and microfiltration (MF) hollow fibres fouling potential of six different water sources characterized using size exclusion chromatography separation coupled with UVA, OCD and OND detectors and fluorescence EEM spectroscopy. OND was used to determine the relative importance of each fraction (polysaccharide versus protein) in the biopolymer fraction. Statistical analysis was used for the interpretation of the data. In this paper, the EfOM from conventional activated sludge (CAS) processes and membrane bioreactor (MBR) is compared to the NOM from natural water source (Marne River) in terms of structural composition and fouling rate. The pertinence of coupling different analytical tools and their reliability are also discussed.

2. Methods

2.1. Water samples

Five secondary effluents and one river water were considered in this study:

- SJA-SE, a biologically treated secondary effluent from an anaerobic and aerobic activated sludge (N removal) (France);
- Bu-SE, a biologically treated secondary effluent from an oxidation ditch process (N and P removal) (Australia);
- STV-SE, a biologically treated secondary effluent from a Bios-tyr®-biofilter (N removal) (France);
- Ban-MBR-SE, a MBR supernatant from a MBR with anoxic zone performing pre-denitrification and biological phosphorous removal (N and P removal) (Australia);
- STV-MBR-SE, a MBR supernatant from a MBR with cyclic aeration (N removal) (France);
- Marne River, surface water (France).

The secondary effluents were collected from either MBR treatment plants or from CAS treatment plants, which differed both in size and type of treatments. MBR supernatant (i.e. the non-settleable fraction of the MBR biomass) was obtained after centrifugation at 4000g for 10 min. All wastewater effluents including the MBR supernatants were filtered through a 10 µm glass-fibre cartridge filter (Millipore, USA) prior UF and MF filtration. The 10 µm filtrate was kept at 4 °C until analysis. All analyses were performed within 3 days after sampling.

2.2. Analytical tools used for organic matter characterization

2.2.1. General characterization (organic parameters)

Total organic carbon-TOC (<10 µm) and dissolved organic carbon-DOC (<0.45 µm) were determined after acidification (2 N HCl) and air-sparging (bubbling for 4 min) using a Shimadzu TOC analyser (TOC-V-CSH, Shimadzu, Japan) that utilized a catalytic oxidation at 720 °C prior to infrared detection of CO₂. Each sample was analyzed three times to produce an average value and a coefficient of variation below 4%. A calibration curve was made with standards prepared from a potassium hydrogen stock solution in a concentration ranging between 0.5 and 20 mgC/L. UV absorbance was measured after 0.45 µm filtration with an UV-VIS spectrophotometer SAFAS DES (Double Energy System) 190 at 254 nm. The SUVA at 254 nm is then calculated by the ratio of UV_{254nm} (<0.45 µm) and DOC (SUVA = UV_{254nm}/DOC) expressed in L/mgC m.

2.2.2. Size exclusion chromatography

The molecular weight (MW) distribution of the dissolved organic matter was determined using liquid chromatography with organic carbon, UVA and organic nitrogen detections (LC-OCD-UVD-OND, Doc-Labor, Germany) as described elsewhere (Huber et al., 2011). Samples were analyzed without 0.45 µm inline-filter. The first detector after chromatographic separation is non-destructive, fixed wavelength UV-detection (UVD 254 nm, type S-200, Knauer, Berlin, Germany) and thereafter the organic carbon detector. The organic carbon content of each fraction was calculated by integrating the areas below the peaks. For nitrogen detection a side stream is diverted after UVD with a restricted flow rate of 0.1 mL/min for nitrogen analysis. The relative proportion of proteins in the biopolymers fraction that incorporates large amount of polysaccharides is expressed by the N/C ratio. To calculate the protein content in BP fraction, it was assumed that all nitrogen in this fraction is assigned to be protein material and that proteins contain 50–52% C (as mass) and 15–18% N (as mass), i.e. an average C/N ratio of 3 as

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