#### Chemosphere 117 (2014) 144-150

Contents lists available at ScienceDirect

### Chemosphere

journal homepage: www.elsevier.com/locate/chemosphere

# Fouling of enhanced biological phosphorus removal–membrane bioreactors by humic-like substances



<sup>a</sup> Department of Biotechnology, Chemistry, and Environmental Engineering, Aalborg University, Sohngaardsholmsvej 57, DK-9000 Aalborg, Denmark <sup>b</sup> Department of Physics and Nanotechnology, Aalborg University, Skjernvej 4, DK-9220 Aalborg, Denmark

#### HIGHLIGHTS

• Free EPS of an EBPR-MBR system consisted mainly of humic-like substances.

• The humic-like substances adsorbed to the membrane.

• Filtration of the free EPS suspension formed a gel layer.

• The gel formation caused a significant irreversible flux decline.

#### ARTICLE INFO

Article history: Received 19 February 2014 Received in revised form 2 June 2014 Accepted 3 June 2014 Available online 9 July 2014

Handling Editor: O. Hao

Keywords: Free EPS Humic-like substances Adsorption Gel layer

#### ABSTRACT

Fouling by free extracellular polymeric substances was studied in an enhanced biological phosphorus removal-membrane bioreactor. It was demonstrated that the free extracellular polymeric substances, primarily consisting of humic-like substances, were adsorbed to the membrane used in the enhanced biological phosphorus removal-membrane bioreactor plant. Infrared analyses indicated the presence of the humic-like substances on the membrane's active surface after filtration of the free extracellular polymeric substances suspension. Scanning electron microscopy showed the presence of a gel layer on the membrane surface after filtration of the free extracellular polymeric substances as supprised as ignificant decline in water flux. This layer was not entirely removed by a backwashing, and the membrane's water flux could not be re-established. The membrane used in the enhanced biological phosphorus removal-membrane bioreactor plant showed infrared spectra similar to that fouled by the free extracellular polymeric substances suspension in the laboratory. Thus, the results of this study show the importance of humic-like substances in irreversible fouling of enhanced biological phosphorus removal-membrane bioreactor systems.

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#### 1. Introduction

Membrane bio-reactors (MBRs) are widely used in wastewater treatment. They have excellent solid–liquid separation, and have a smaller foot-print and produce less sludge than conventional activated sludge systems (Judd, 2006). However, they suffer from the general problem of fouling. Part of the flux decline in MBR systems can be predicted by typical theories of cake filtration as modelled by Bugge et al. (2012) by considering cake build-up during filtration and cake fall during relaxation. However, the fouling that occurs during extended operation cannot be explained by the

\* Corresponding author. *E-mail address:* mlc@bio.aau.dk (M.L. Christensen).

http://dx.doi.org/10.1016/j.chemosphere.2014.06.006 0045-6535/© 2014 Elsevier Ltd. All rights reserved. model, and may be due to accumulation of extracellular polymeric substances (EPS) between the membrane and the cake layer (Wang et al., 2011). Studies have highlighted a correlation between the concentration of EPS in MBR sludge and membrane fouling (Liu et al., 2012).

EPS encompass several types of biopolymers, including proteins, humic-like substances, and carbohydrates. EPS are often classified as extractable and non-extractable. The extractable EPS include both bound and free EPS. The free EPS fraction is sometimes called soluble microbial products (SMP) or soluble EPS (Nielsen and Jahn, 1999). Because not all of the EPS components of the mentioned fraction may be completely soluble, but may exist as colloidal components, we avoid calling them SMP or soluble EPS in this work. Several papers have documented the importance of the liquid phase of MBR sludge, and that the free







EPS play a key role in fouling (Rosenberger et al., 2005; Ji et al., 2008; Wang et al., 2011).

#### 2005; Ji et al., 2.2. EPS extraction

A common practice in using MBR systems is combination with anaerobic (AN) hydrolysis and denitrification (DN) units to eliminate phosphorus and nitrate, respectively (Zhang et al., 2009). Such systems are called enhanced biological phosphorus removal–membrane bioreactor (EBPR–MBR). In the anaerobic hydrolysis unit, the phosphorus-removing microorganisms consume carbohydrates (Kristiansen et al., 2013). In addition, Zhou et al. (2013) showed that the denitrification process reduces carbohydrate and protein concentrations of the free EPS. In contrast to carbohydrate and protein, humic-like substances are highly resistant to biodegradation (Kästner and Hofrichter, 2001). Thus, in the EBPR–MBR systems, the free EPS of the MBR sludge are expected to consist mainly of humic-like substances.

The membranes used in MBRs are often polymeric and can adsorb biopolymers; for example, the adsorption of humic acid to polyethersulfone membranes has been shown to cause irreversible fouling (Jermann et al., 2007). Apart from adsorption, a gel layer has been reported to exist on the MBR membranes, which is composed of macromolecules (Ji et al., 2008). The gel layer itself reportedly has a much higher specific resistance than the cake layer (Wang et al., 2011). The humic-like substances are present in the gel layer formed on the MBR membranes (Aryal et al., 2009); therefore, adsorption of and gel formation by the humic-like substances from free EPS are hypothesised to contribute to membrane fouling of EBPR–MBR systems.

In the current study, we have investigated adsorption of and gel formation by the humic-like substances present in the free EPS fraction of an EBPR–MBR system. Effects of the adsorption and gel formation on membrane permeability and, reversibility or irreversibility of the effects have been studied.

#### 2. Materials and methods

#### 2.1. Sludge samples

The sludge was sampled from an MBR unit of a pilot EBPR–MBR plant. The MBR unit was 1.6 m<sup>3</sup> in volume with a trans-membrane pressure (TMP) of approximately 3 kPa being run in constant TMP mode, with 10 min filtration and 2 min relaxation periods. The other operational parameters were as follows: solid retention time: 15 d; hydraulic retention time: 12 h; MLVSS: 6 g L<sup>-1</sup>; food to microorganism ratio: 0.03 BOD<sub>5</sub>·SS<sup>-1</sup> d<sup>-1</sup>; aeration rate: 200 L min<sup>-1</sup>; COD removal: 90%; phosphorus removal: 50–75%; nitrogen removal: 75%. The feed of the MBR unit was municipal wastewater passed through a side stream anaerobic hydrolysis and an anoxic tank as illustrated in Fig. 1.

Flat sheet microfiltration (MF) membrane was used in the pilot plant. The active surface of the membrane was composed of polyvinylidene difluoride (PVDF) hydrophilized with polyvinylpyrrolidone, and the nominal pore diameter was 0.2  $\mu$ m (Alfalaval A/S, Copenhagen, Denmark).



Fig. 1. Process configuration of the pilot EBPR-MBR plant.

The free EPS were extracted from the sludge of the MBR unit. Sludge sampling and EPS extraction were performed on the same day. Free EPS were obtained by means of centrifugation of MBR sludge at 9000g for 1 h and followed by two subsequent filtrations of the supernatant through glass–fiber filters of 1.6  $\mu$ m and mixed cellulose ester filters of 0.45  $\mu$ m nominal pore diameter. Similar procedures have been used as reported by Rosenberger et al.

#### 2.3. EPS analyses

(2005).

Concentrations of proteins, carbohydrates, and humic-like substances were measured in the free EPS suspension and MBR permeate within 48 h of extraction. Concentrations of proteins and humic-like substances were measured by the modified Lowry method using bovine serum albumin and Aldrich humic acid as standards, respectively (Frølund et al., 1995). Concentration of carbohydrates was measured by the Anthrone method using glucose as a standard (Frølund et al., 1995). The measurements were triplicated. A UV-Vis spectrophotometer (Model 50; Varian, Palo Alto, CA, USA) was used for the measurements; Milli-Q water was used as the zero baseline. The same spectrophotometer was used for obtaining UV-Vis spectra of the free EPS suspension and MBR permeate. Size-exclusion chromatography (SEC) was performed to compare molecular mass distribution of the EPS components of the free EPS suspension and the MBR permeate. The specifications of the size exclusion column (Agilent SEC) were 3 µm, 300 Å,  $7.8 \times 300$  mm. Detection at a wavelength of 280 nm was accomplished with a UV detector (DIONEX, Sunnyvale, CA, USA).

#### 2.4. Filtration and adsorption experiments

The membrane type was the same as that used in the pilot plant. The as-received membrane sheets were immersed and shaken first in ethanol and then in deionized water for 3–4 h to remove the protective glycerol coating. Milli-Q water was then filtered through the membrane sheets at 200 kPa for 24 h to compact the membrane and obtain a constant permeability. After that, 47-mm-diameter discs were cut from the prepared membrane sheets for the experiments.

The membrane discs were used for the permeability and adsorption experiments using Milli-Q water and free EPS suspension, respectively. In this study, the adsorption experiments were divided into static and dynamic adsorption experiments. Static adsorption refers to adsorption during contact of the free EPS suspension with the active surface of the membrane at no applied pressure, whereas dynamic adsorption refers to adsorption during filtration of the free EPS suspension through the membrane. The dynamic adsorption has been subdivided into dynamic adsorption with and without recirculation of the free EPS suspension to the feed container.

As illustrated in Fig. 2, four different setups were used for (a) (Milli-Q) water flux measurement, (b) static adsorption, (c) dynamic adsorption with recirculation, and (d) dynamic adsorption without recirculation and backwashing. The water flux was measured before adsorption, after adsorption, and after backwashing at 50 kPa and room temperature. The water flux values were corrected to 20 °C by applying a viscosity correction function (EPA, 2005). The normalized water flux of each membrane disc was obtained by dividing the corrected flux after adsorption and backwashing by the corrected water flux before adsorption.

As the adsorption capacity of the membrane was expected to be low in the static adsorption, a small volume (10 mL) of the free EPS suspension was chosen for the static adsorption experiments Download English Version:

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