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In-situ biofilm characterization in membrane systems using Optical Coherence Tomography: Formation, structure, detachment and impact of flux change

C. Dreszer [a,b], A.D. Wexler [a], S. Drusová [a], T. Overdijk [a], A. Zwijnenburg [a], H.-C. Flemming [b], J.C. Kruithof [a], J.S. Vrouwenvelder [a,c,d,*]

[a] Wetsus, Centre of Excellence for Sustainable Water Technology, Agora 1, P.O. Box 1113, 8900 CC Leeuwarden, The Netherlands
[b] Biofilm Centre, University Duisburg-Essen, Universitätsstrasse 5, 45141 Essen, Germany
[c] Department of Biotechnology, Faculty of Applied Sciences, Delft University of Technology, Julianalaan 67, 2628 BC Delft, The Netherlands
[d] King Abdullah University of Science and Technology, Water Desalination and Reuse Center, Thuwal, Saudi Arabia

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ABSTRACT

Biofouling causes performance loss in spiral wound nanofiltration (NF) and reverse osmosis (RO) membrane operation for process and drinking water production. The development of biofilm formation, structure and detachment was studied *in-situ*, non-destructively with Optical Coherence Tomography (OCT) in direct relation with the hydraulic biofilm resistance and membrane performance parameters: transmembrane pressure drop (TMP) and feed-channel pressure drop (FCP). The objective was to evaluate the suitability of OCT for biofouling studies, applying a membrane biofouling test cell operated at constant crossflow velocity (0.1 m s^{-1}) and permeate flux (20 L m^{-2} h^{-1}).

In time, the biofilm thickness on the membrane increased continuously causing a decline in membrane performance. Local biofilm detachment was observed at the biofilm–membrane interface.

A mature biofilm was subjected to permeate flux variation (20 to 60 to 20 L m^{-2} h^{-1}). An increase in permeate flux caused a decrease in biofilm thickness and an increase in biofilm resistance, indicating biofilm compaction. Restoring the original permeate flux did not completely restore the original biofilm parameters: After elevated flux operation the biofilm thickness was reduced to 75% and the hydraulic resistance increased to 116% of the original values. Therefore, after a temporarily permeate flux increase the impact of the biofilm on membrane performance was stronger. OCT imaging of the biofilm with increased permeate flux revealed that the biofilm became compacted, lost internal voids, and became more dense. Therefore, membrane performance losses were not only related to biofilm thickness but also to the internal biofilm structure, e.g. caused by changes in pressure.

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* *Corresponding author*. Department of Biotechnology, Faculty of Applied Sciences, Delft University of Technology, Julianalaan 67, 2628 BC Delft, The Netherlands. Tel.: +31 (0)6 44195396/+31 (0)15 27 84169; fax: +31 (0)15 27 82355.
E-mail addresses: j.s.vrouwenvelder@tudelft.nl, johannes.vrouwenvelder@kaust.edu.sa (J.S. Vrouwenvelder).
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Optical Coherence Tomography proved to be a suitable tool for quantitative *in-situ* biofilm thickness and morphology studies which can be carried out non-destructively and in real-time in transparent membrane biofouling monitors.

1. Introduction

Biofouling is the major problem in membrane operation for process and drinking water production (Winters and Isquith, 1979; Paul, 1991; Tasaka et al., 1994; Ridgway and Flemming, 1996; Flemming et al., 1997; Gamal Khedr, 2000; Saeed et al., 2000; Vrouwenvelder et al., 2008; Flemming and Ridgway, 2009).

Biofouling occurs when the growth of biofilms in the membrane system negatively impacts membrane performance parameters e.g. feed-channel pressure drop (FCP) and transmembrane pressure drop (TMP), reducing the quantity and quality of produced water. Biofouling is defined as the biofilm formation causing a membrane performance decline exceeding 10–15% of the start-up values under the applied operational conditions At variations larger than 10–15%, corrective actions are recommended and guarantees are restricted by the manufacturers of membrane elements (Hydranautics, 2001; GE Water, 2014; Dow, 2014). Biofouling increases the costs of plant operation strongly (Ridgway and Flemming, 1996) and may even be prohibitive for the application of membrane filtration in water treatment.

Historically, biofouling research on spiral wound membrane systems is typically problem solving oriented (Van Loosdrecht et al., 2012). Biofouling is a complicated process in which many factors can influence each other. Fouling is diagnosed on the basis of parameters such as pressure drop and on membrane autopsies in hindsight. To solve or minimize the negative impacts of biofouling, a clear understanding of biofilm development in relation to e.g. membrane module design and operational aspects is needed. *In-situ* non-destructive visualization methods in small-scale representative test units should be developed to characterize biofouling, enabling the development of strategies to prevent or minimize the impact of biofilm formation on membrane performance. In other words, suitable tools applied under conditions representative for practice are needed to increase the understanding of biofouling processes, which may lead to effective biofouling control strategies.

A suite of *in-situ* non-destructive imaging tools has been recently proposed for biofouling studies of membrane systems such as e.g. magnetic resonance imaging (Graf von der Schulenburg et al., 2008; Vrouwenvelder et al., 2009a; Creber et al., 2010a,b) and oxygen concentration imaging by use of optodes (Staal et al., 2011; Prest et al., 2012). Ultrasonic time-domain reflectometry can provide information on biofilm thickness but requires silica dosage to enhance the acoustic signal (Sim et al., 2013). Confocal laser scanning microscopy (Neu and Lawrence, 1997; Schramm et al., 2000) is another suitable technique to obtain information on the biofilm thickness and structure but requires use of fluorescent dyes, possibly affecting the biofilm formation. Besides the application of confocal laser scanning microscopy is limited by the penetration depth. Therefore, with that method the internal structure of thick biofilms cannot be observed.

Non-invasive *in-situ* tools/methods offer the possibilities to investigate fouling layer thickness and structure without stopping the operation of the membrane filtration unit. Optical Coherence Tomography (OCT) has the ability to provide volumetric imaging of the biofilm at micron resolution in a non-destructive way (Derlon et al., 2012, 2013; Haisch and Niessner, 2007; Janjaroen et al., 2013; Wagner et al., 2010; Xi et al., 2006). OCT imaging provides a quantitative, high-resolution, spatially-resolved means to characterize biofilm growth and detachment, biofilm thickness, and structural heterogeneity. It was developed by Huang et al. (1991) as a tool for medical imaging in 1991. Since 2006, OCT has been used to investigate biofilm structures in water and membrane filtration systems, in capillary flow cells (Xi et al., 2006), and in crossflow filtration systems under laminar, transient, and turbulent flow conditions (Haisch and Niessner, 2007; Wagner et al., 2010). Attachment mechanisms of *Escherichia coli* (Janjoeran et al., 2013), and predation of eukaryotes and metazoan organisms in gravity driven membrane filtration systems has been studied as well (Derlon et al., 2012, 2013). So far, no OCT investigations were performed in a filtration system representative for spiral wound membrane filtration in which the thickness and compaction of biofilms was related to performance losses of the membrane system.

In a study on the intrinsic hydraulic resistance of biofilms Dreszer et al. (2013) subjected a mature biofilm to changes in permeate flux. This experiment raised the assumption that biofilms react to an increase in permeate flux by compaction. The assumption could not be confirmed due to the lack of *in-situ* biofilm thickness measurements. The application of OCT offers the possibility to provide visual evidence for the compressibility of biofilms. OCT is an optical signal acquisition and processing method able to capture, during contact-free and non-invasive operation, micrometer-resolution images from within optical scattering media (Fercher et al., 2003; Wikipedia, 2014). OCT is an interferometric technique, typically employing near-infrared light. The use of relatively long wavelength light allows, compared to confocal microscopy, deeper penetration into the biofilm (Fercher et al., 2003; Wikipedia, 2014). To the authors knowledge this is the first paper showing *in-situ* biofilm thickness data in a crossflow operated permeate producing membrane system.

The objective of the study was to assess and evaluate the suitability of Optical Coherence Tomography for *in-situ* measurements of biofilm thickness and morphology during (i) biofilm development, (ii) biofilm detachment, and (iii)