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# Optical coherence tomography for the *in situ* three-dimensional visualization and quantification of feed spacer channel fouling in reverse osmosis membrane modules

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ABSTRACT

To understand the biofouling in membrane modules in more detail, three-dimensional *in situ* imaging of the feed spacer channel by means of optical coherence tomography (OCT) was applied. The biofilm formation was investigated in membrane fouling simulators (MFS) mimicking the feed spacer channel of spiral wound reverse osmosis membrane modules. To test the capability of OCT for the visualization of feed spacer fouling, river water and effluent of a waste water treatment plant were used as feed solutions. Additionally, the impact of the feed spacer geometry on the biofouling deposit accumulation was tested using a wide-meshed and small-meshed feed spacer, respectively. 3D OCT datasets were quantified and the determined amount of biofilm was correlated to the feed channel pressure drop (FCP). The study validates the application of OCT as imaging tool to time-resolved visualize and quantify the biofilm in feed spacer filled channels. The amount of biofilm correlated with the FCP. However, the feed spacer geometry and feed composition yielded differences in the structure and distribution of grown biofilm. Hence, the FCP as macroscopic measure does not necessarily allow to assume a certain biofilm structure or distribution. A more detailed understanding of the FCP development could be achieved by the high-resolution 3D imaging data provided by OCT. Hence, this study emphasizes the capabilities of OCT to investigate the biofouling development in membrane modules.



## 1. Introduction

Biofilm growth in the feed spacer channel of spiral wound membrane modules causes increased pressure losses resulting in an additional energy demand to maintain permeate production. Hence, with increasing feed inlet pressure the efficiency of membrane systems is subsequently decreasing. Suitable monitoring parameters are required for process control and for the understanding of the mechanisms that govern biofilm growth [1,2]. The feed channel pressure drop (FCP) and transmembrane pressure (TMP) are macroscopic measures related to the fouling with high practical value [3–5]. In addition to these macroscopic parameters, imaging techniques are applied more frequently to understand the biofilm/deposit formation and structure in more detail. A first option is the observation of the biofouling deposits inside feed spacer channels by eye when transparent fouling simulators are used [6]. At the microscale for instance scanning electron microscopy, atomic force microscopy, and confocal laser scanning microscopy are often used techniques to visualize the biofilm structure on fouled membranes and feed spacers [7–9]. Also oxygen sensitive optodes have been used to visualize biofilm development in feed spacer channels [10]. Furthermore, the interaction of fouling formation and deposit accumulation with the moving bulk phase has substantially been investigated using magnetic resonance imaging (MRI) [11]. This technique was also used to evaluate the effectiveness of cleaning protocols as presented by Creber et al. [12]. The advantage of MRI is that in addition to the three-dimensional distribution of the biofilm, the flow field can be measured *in situ* and non-invasively [13]. The major drawbacks are the high administrative demand, the costs and the limited resolution. Another imaging technique which can be used to visualize biofilms at the mesoscale (mm-scale) at high resolution, *in situ* and also non-invasively is optical coherence tomography [14–16]. OCT imaging is increasingly applied in the field of membrane research. Gao et al. proved its potential for visualizing liquid flow patterns in spacer filled feed channels [17]. The effect of (natural) predation on the structure of the fouling layer in gravity driven membrane systems has been shown by

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Derlon et al. [18]. In another study the compaction and detachment of the biofouling layer in a microfiltration unit was investigated [19,20]. Here no spacer was installed in the feed channel. A first study which used OCT to image the biofouling layer in the presence of a feed spacer has recently been published [21]. OCT was therein employed for a detailed study of biofilm removal by two-phase-flows in a nano-filtration unit. The enumerated studies using OCT for the visualization of biofilms have in common, that the image acquisition was performed without opening the cultivation device allowing for the *in situ* and non-invasive determination of the structure of the biofilm/biofouling layer. Although OCT allows a fast and reliable acquisition of three-dimensional (3D) datasets [22], only two-dimensional (2D) cross-sections of the fouling deposits in membrane systems have been presented to date.

The objective of this study was thus to analyze the biofouling in the feed spacer channel of reverse osmosis membrane modules by acquiring *in situ* and non-invasively representative three-dimensional OCT datasets. More precisely, the influence of the feed spacer geometry and organic load were investigated exploring the capabilities of OCT for characterizing the biofilm development and structure as well as its effect on the feed channel pressure drop. For this purpose, biofilm was cultivated in modified, high-pressure stable membrane fouling simulators (MFS) containing optical windows, reverse osmosis membranes, and diamond shaped feed spacer. A quantification method for OCT datasets was developed to correlate the amount of accumulated biofilm with the feed channel pressure drop. Thereby, the biofouling characteristics and MFS operation were linked.

## 2. Materials and methods

### 2.1. Experimental setup

Biofilm was cultivated in two membrane fouling simulators (MFS). A photograph of one MFS prepared for an OCT measurement is displayed in Fig. 1A.

These flat channel membrane modules exhibit hydrodynamic conditions equivalent to those in spiral wound membrane modules as described elsewhere [3,4]. The feed channel dimensions were 280 mm × 40 mm × 0.8 mm. The MFS were made from stainless steel and are pressure resistant up to 13 bar. The cover contained three optical windows (sapphire glass, 1 in. diameter, 5 mm thick; Thorlabs, Dachau, Germany) allowing for the *in situ* visualization of biofilm by means of OCT during full operation of the MFS. Feed inlet pressure and concentrate outlet pressure where recorded using pressure gauges (type 528, Huba Control AG, Switzerland). The volumetric flow rate of the concentrate was measured in-line using flowmeters (FCH-m-POM, B.I.O-TECH e. K., Germany). The mean volumetric flow rate of the feed was constant at 30 L/h. Pressure and volumetric flow rate were recorded every 15 min. The permeate flux was adjusted and constant at 80 mL/h (10 L/($m^2$ h)) and determined daily by gravimetry. The feed ($V$=40 L) was stored in the recirculation tank at a constant temperature of 20 °C (thermostat F25-MV, Julabo GmbH, Seelbach, Germany). Each MFS was supplied separately with feed using a magnetic gear pump (Niemzik Pumpen und Anlagentechnik, Haan, Germany). The feed was replaced every second day to maintain almost constant nutrient and substrate levels. Concentrate and permeate were recirculated into the storage tank. The complete setup was protected from light to prevent photoautotrophic growth. For each experiment the MFS were equipped with new pipes, membranes, and feed spacers.

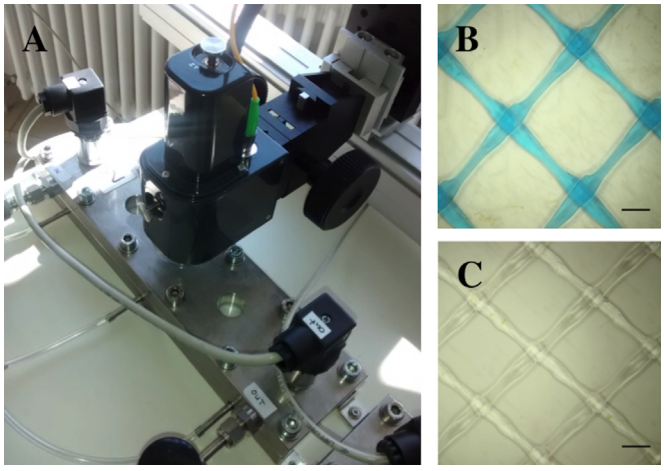


**Fig. 1.** (A) Photograph of the OCT probe positioned above the MFS for visualization of the biofilm. (B) and (C) show stereo micrographs of the wide-meshed (blue) and small-meshed spacer, respectively. Scale bar=1 mm. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

### 2.2. Reverse osmosis membrane, feed spacer, and feed

A Filmtec XLE-2521 reverse osmosis membrane was purchased at Lenntech B.V. (Delft, The Netherlands) and employed in all experiments. Two different diamond shape spacers were tested on their influence on the development of biofouling. The main difference between both spacers is the mesh size defined as the length $L$ of the spacer filament between two filament junctions [23]. For the wide-meshed (WM) spacer, $L$ equals 3.7 mm and equals 2.8 mm for the small-meshed (SM) spacer. Photographs of both spacers are presented in Fig. 1B and C, respectively. The thickness of the spacer was 34 mil (WM) and 31 mil (SM), respectively. The WM spacer was blue colored and contained 0.5% (w/w) Triclosan as anti-fouling component. According to Araújo et al. [24] the spacer containing the anti-fouling agent did not show differences in the development of biofouling. Thus, the fouling behavior of the WM and SM spacer can be related to their geometry.

Both spacers were tested for their biofouling behavior with two different feed solutions. Water from the river Pfinz (near Karlsruhe, Germany, coordinates: 49.085310° 8.415068°) and biologically treated waste water (secondary effluent of the waste water treatment plant Heidelsheim, Germany) were used as feed solutions, respectively. Table 1 summarizes the cultivation conditions. Feeds were supplied without pretreatment and each experiment was performed in duplicate ($n$=2).

### 2.3. Visualization of biofouling deposits

Optical coherence tomography (GANYMEDE system equipped with an LSM03-BB objective lens, Thorlabs, Lübeck, Germany) was used to visualize the biofilm formation directly inside the feed spacer channel without opening the MFS.

Briefly, optical coherence tomography (OCT) is based on the reflection, scattering, and interference of infrared light from a low-coherence light source. Excitation light reflected at surfaces and structures is detected by a spectrometer after interfering with a reference beam. The resulting interference pattern contains information about the location and intensity of the signal [15]. Each OCT measurement provides depth information at the scanned position. A depth profile is named 'A-scan'. By acquiring multiple A-scans a two-dimensional cross section (B-scan, *xz*-plane) is visualized [25]. In a further step, the scanning of multiple B-scans allows the acquisition of 3D datasets. In this communication an