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Applicability of short-term accelerated biofouling studies to predict long-term biofouling in reverse osmosis membrane systems

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ABSTRACT

Biofouling studies addressing biofouling control are mostly executed in short-term studies. It is unclear whether data collected from these experiments are representative for long-term biofouling as occurring in full-scale membrane systems. This study investigated whether short-term biofouling studies accelerated by biodegradable nutrient dosage to feed water were predictive for long-term biofouling development without nutrient dosage. Since the presence of a feed spacer has an strong effect on the degree of biofouling, this study employed six geometrically different feed spacers. Membrane fouling simulators (MFSs) were operated with the same (i) membrane, (ii) feed flow and (iii) feed water, but with feed spacers varying in geometry. For the short-term experiment, biofilm formation was enhanced by nutrient dosage to the MFS feed water, whereas no nutrient dosage was applied in the long-term experiment. Pressure drop development was monitored to characterize the extent of biofouling, while the accumulated viable biomass content at the end of the experimental run was quantified by adenosine triphosphate (ATP) measurements. Impact of feed spacer geometry on biofouling was compared for the short-term and long-term biofouling study. The results of the study revealed that the feed spacers exhibited the same biofouling behavior for (i) the short-term (9-d) study with nutrient dosage and (ii) the long-term (96-d) study without nutrient dosage. For the six different feed spacers, the accumulated viable biomass content (pg ATP.cm^{-2}) was roughly the same, but the biofouling impact in terms of pressure drop increase in time was significantly different. The biofouling impact ranking of the six feed spacers was the same for the short-term and long-term biofouling studies. Therefore, it can be concluded that short-term accelerated biofouling studies in MFSs are a representative and suitable approach for the prediction of biofouling in membrane filtration systems after long-term operation.

Keywords: Biofouling; Reverse osmosis; Membrane fouling simulator; Feed spacers; Modified spacer geometry

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1. Introduction

Membrane-based water treatment processes have been well developed and widely applied in the recent years for the mitigation of fresh water scarcity [1]. Reverse osmosis (RO) and nanofiltration (NF) are amongst the most effective and robust technologies for drinking water production [2]. The use of spiral-wound membrane modules in RO and NF installations is prevalent. However, the effectiveness of the membrane system may be compromised due to fouling, of which biofouling (excessive growth of biomass) is the most problematic [3–6]. Biofouling is an unavoidable problem in RO and NF membrane treatment processes despite advances in improving membrane properties [7,8], process design [9,10] and optimizing operational conditions [3,11,12]. Biofouling contributes to more than 45% of all cases of membrane filtration suffering from membrane fouling [13] and it can be detrimental to membrane performance, ultimately leading to filtration process failure.

Biofouling is largely attributed to feed spacer characteristics [14–18]. In spiral-wound membrane configurations, feed spacers serve to separate the individual membrane sheets and to promote turbulence [19]. Research has demonstrated that feed spacer biofouling effects overall performance more adversely than membrane biofouling [14,16,18]. The modification of feed spacer geometry has shown to have an effect on the hydraulic resistance and biofouling of membrane elements [20].

Short-term biofouling experiments are an efficient approach for the rapid assessment of fouling behavior of a given membrane system under different operating conditions. In literature, several studies have been described applying a short-term experimental approach to compare the effect of modifying operational parameters and filtration materials on biofouling [21–25]. Much work has been carried out using membrane fouling simulators (MFSs) in which laboratory-scale experiments can be conducted, representative for the performance of spiral-wound membrane elements in full-scale installations [26]. Earlier studies [27–30] have shown reproducible development of biofouling in MFS units. However, a point of concern is that short-term experiments may not yield membrane performance data representative for long-term membrane process operation [31].

The objective of this study was to test the applicability of short-term accelerated biofouling studies to predict the biofouling susceptibility of feed spacers during long-term operation. The comparative studies were carried out in MFSs using six geometrically different feed spacers.

2. Materials and methods

2.1. Feed spacer geometry

Feed spacer thickness, distance and angle between spacer filaments, and filament orientation are important properties to characterize an effective membrane performance [32]. In the MFS experiments, six feed spacers were employed with a different geometrical design (Table 1). Two reference feed spacers (CON-1 and CON-3) were obtained from Conwed Plastics (Minneapolis, USA) with the same filament shape and an internal strand angle (β) of 90°. The difference between CON-1 and CON-3 was the spacer thickness, 34 mil (~863 μm) and 31 mil (~787 μm), respectively. Four feed spacers with a modified geometry were provided by DOW, Hydranautics, and Lanxess. The differences between the spacer geometries have been elicited by X-ray computed tomography (CT) scanning of the feed spacers [33]. The internal contact angle of the DOW spacer (DOW, USA) was 70° while the contact angle for the other spacers was 90° (Table 1). DOW had a uniform filament thickness. HYD (Hydranautics, Oceanside, USA) was designed with thinner regions around the filament intersections and a larger average parallel strand distance. This spacer also contained 0.5 wt% triclosan ingredient which acts as a biocide. LXS-ASDi and LXS-ASD (Lanxess, Bitterfeld, Germany) had alternating thick and thin spacer filaments with a larger average parallel strand distance (Table 1). LXS-ASDi also had an irregular filament shape along its fiber length. All feed spacers consisted of

Table 1
Overview of geometric characteristics of the six feed spacers used in this study [20]

Spacer code	CON-1	CON-3	DOW	HYD	LXS-ASDi	LXS-ASD
Thickness according to specifications (mil)	34	31	34	34	34	34
Measurements						
Average spacer thickness (μm)	847 ± 24	717 ± 9	806 ± 14	820 ± 22	837 ± 8	830 ± 15
Average parallel strand distance (mm)	2.26	2.29	2.43	2.85	2.79/2.72	2.95/2.63
Average strand thickness (μm)	50	49	43/48	50	57/45	65/50
Inner strand angle, β (°)	90	90	70	90	90	90
Porosity (ø)	0.856	0.874	0.877	0.893	0.899	0.898
Remarks	Reference spacer	Reference spacer	Modified inner strand angle and thinner strands	Modified larger mesh-size and thinner strands	Modified alternate strand thickness with irregularities in strands	Modified alternate strand thickness

polypropylene with a density (ρ) of 0.91 g/cm^3. The feed channel porosities for the spacers were calculated based on CT-scan measurements of the feed spacers (Table 1) [20,33].

2.2. Experimental setup

The laboratory setup consisted of two cartridge filters in series (10 μm pore size), flow controllers, nutrient dosage pump, MFSs and back-pressure valves [20]. The six MFSs used for all studies had identical flow channel dimensions of 20 cm × 4 cm × 863 μm. Membrane and feed spacer coupons (20 cm × 4 cm) were placed inside each MFS to mimic the structure of spiral-wound membrane elements in terms of materials and dimensions. All six MFSs (each containing a different type of spacer) were operated in parallel, simultaneously for 9 d during the short-term accelerated biofouling study with a constant dosage of biodegradable nutrients to the MFS feed water. In the long-term biofouling study, the MFSs were operated simultaneously for 96 d without nutrient dosage. During operation, the MFS window was covered with a light-tight lid to prevent growth of phototropic organisms. The development of fouling was monitored by measuring the pressure drop increase over the feed spacer channel of the MFS [15] and by quantifying the amount of active biomass accumulated in each MFS [34] at the end of operation (Table 2).

2.3. Operating conditions

The feed water used for this experiment was tap water produced from surface water at the Kralingen treatment plant (Water Supply Company Evides, The Netherlands). The disinfectant (ClO_2) concentration in the reservoir effluent water was below the detection limit and no residual disinfectant concentration was maintained in the distribution network. The colony forming units (CFU) in feed water, measured using bacterial enumeration methods, were 2 × 10^3 CFU/mL after 10 d incubation at 25°C. Feed water was pumped to the MFSs at a flow rate of 17.0 L/h equivalent to a linear flow velocity of 0.16 m/s, representative of practice [14]. The MFSs were operated at a pressure of 1.7 bar to avoid degassing. For the short-term study, biofilm development in the MFS was accelerated by dosing a biodegradable nutrient solution containing acetate, nitrate and phosphate in a mass ratio C:N:P of 100:20:10 to the feed water. The organic carbon concentration (acetate) added to the MFS feed water was 150 μg C/L. No nutrient solution was dosed to the feed water during the long-term MFS operation.

Table 2
Experimental design of the short-term and long-term biofouling studies

	Short-term study	Long-term study
Spacer	+	+
Membrane	+	+
Feed water	+	+
Nutrient dosage	Yes	No
Run time	9 d	96 d

+, Same materials and parameters.

2.4. Quantification of viable biomass content

At the end of the biofouling studies, autopsy of the membrane and spacer sheets was carried out in order to quantify the accumulated viable biomass content on the inlet side of the MFS. Adenosine triphosphate (ATP) is present in all metabolically active microorganisms, thus ATP analysis can be used to measure the viable biomass content. It is a generally accepted parameter for diagnosis of biofouling [28,34–37]. Membrane and feed spacer coupons (4 cm × 4 cm) were removed from the MFS and placed in centrifuge tubes containing 30 mL of autoclaved tap water. The tubes with the coupons were placed in an ultrasonic water bath (Bransonic, model 5510E-DTH, output 135 W, 42 kHz) for 2 min followed by mixing on a Vortex for 1 min to remove biomass from the membrane and spacer surface. The procedure was repeated three times and the solution after removing the coupons was used to determine the viable biomass content by means of ATP analysis using the ATP Celsis Luminometer according to the suppliers' protocol [20,38,39].

3. Results

3.1. Short-term biofouling study with nutrient dosage

Six MFSs containing a different feed spacer each were operated in parallel for 9 d with a constant supply of nutrients to accelerate biofilm formation. Aside from the blank MFS control, the study employed a commonly used reference feed spacer (CON-1) and four modified feed spacers (DOW, HYD, LXS-ASDi and LXS-ASD). Biofilm development for each feed spacer was studied by monitoring the feed channel pressure drop increase in the MFSs in time, followed by a quantitative analysis of the accumulated viable biomass content at the end of operation (Table 3).

There was no significant difference between the viable biomass accumulation, measured as ATP which amounted to 10^5 pg ATP cm^{-2}, in each MFS irrespective of the type of feed spacer (Fig. 1(B)). On the contrary, the feed channel pressure drop increase was significantly different for reference and modified feed spacers at the end of the study (Fig. 1(A)), showing that feed spacer geometry had an impact on the hydraulic resistance, which was lower for the modified feed spacers (DOW, HYD, LXS-ASDi and LXS-ASD) than for the

Table 3
Definitions of important parameters in this study

Parameter	Definition
Biomass	Accumulation of biological materials on a surface (biofilm formation)
Biofouling	Impact of excessive biomass accumulation on membrane performance, or a biofilm leading to operational problems
Pressure drop	Differential pressure measured between the inlet and outlet of membrane module/MFS
Pressure drop increase	Change in pressure drop with time
Operational problem	An increase of normalized pressure drop (NPD) by 15% of the start-up values

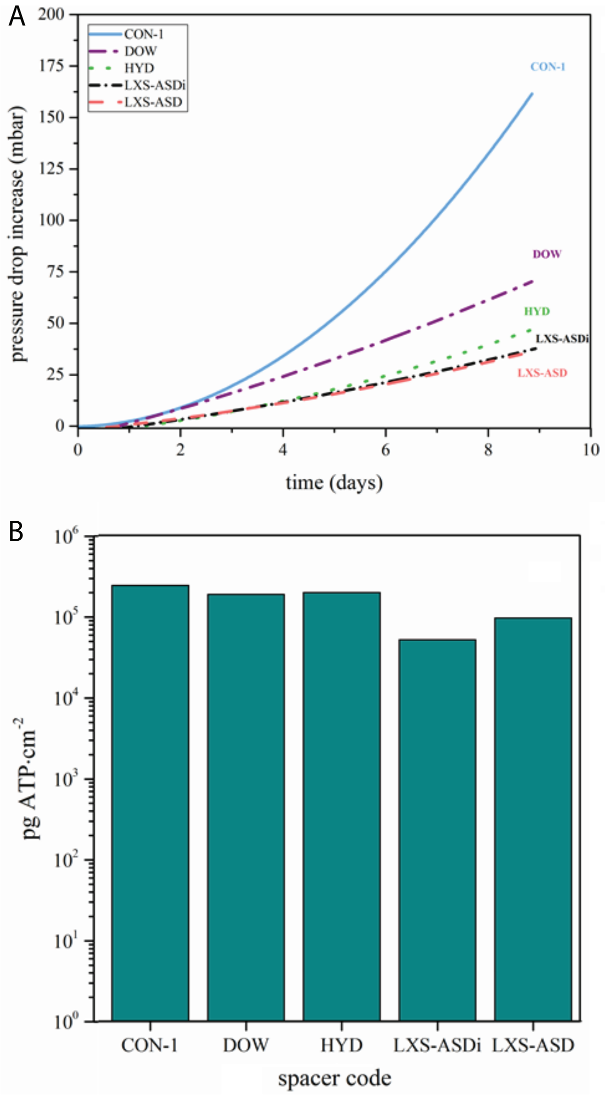

Fig. 1. (A) Pressure drop increase in time in MFSs containing one reference and four modified feed spacers at a constant feed flow rate and (B) accumulated viable biomass content (pg ATP.cm^{-2}) for the short-term (9-d) biofouling study accelerated with nutrient dosage [20].

reference feed spacer (CON-1). Amongst the modified feed spacers, the LXS-ASD/ASDi spacers had the lowest feed channel pressure drop; probably due to the larger mesh size and alternating thick and thin strand arrangement, providing less resistance to the feed flow, resulting in a lower pressure drop increase. It is also interesting to note that until day 6 the pressure drop for the HYD spacer was in close proximity to the LXS-ASD/ASDi spacers, after which the triclosan in the HYD spacer may have started to wear off, diminishing the biocidal effect and resulting in a higher pressure drop increase thereafter.

3.2. *Long-term biofouling study without nutrient dosage*

This experiment was conducted over a period of 96 d without nutrient dosage. It involved two reference feed

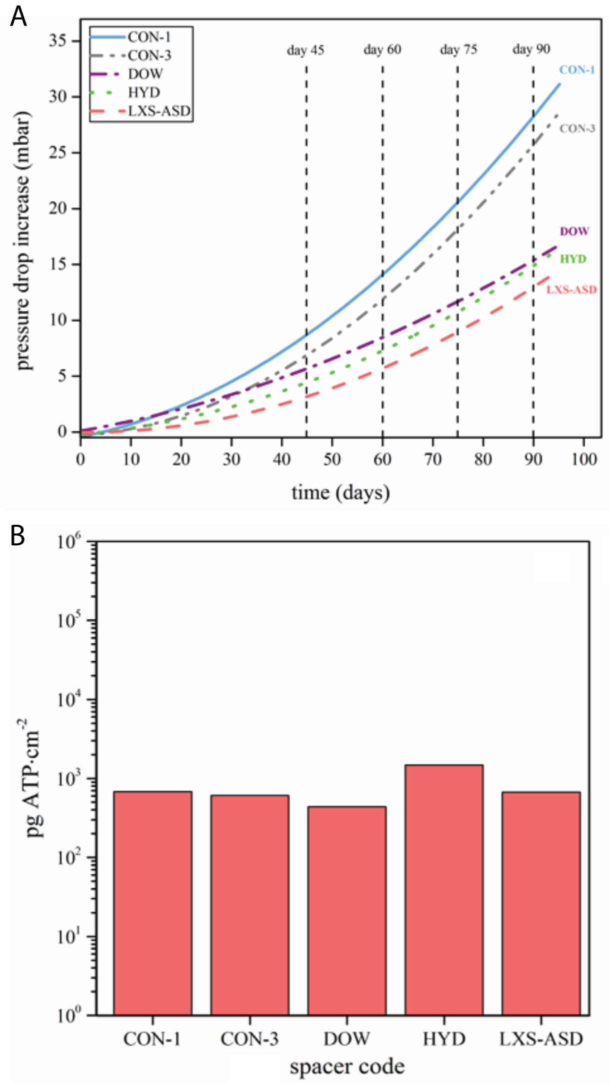

Fig. 2. (A) Pressure drop increase in time in MFSs containing two reference and three modified feed spacers at a constant feed flow rate and (B) accumulated viable biomass content (pg ATP.cm^{-2}) during the long-term (96-d) biofouling study without nutrient dosage [20].

spacers (CON-1 and CON-3) and three modified feed spacers (DOW, HYD and LXS-ASD). Similar to the short-term study (section 3.1), the feed channel pressure drop increase in each MFS was measured in time and the accumulated viable biomass content was quantified at the end of the study (Table 3).

As expected, biofilm formation was slower in the long-term biofouling study without nutrient dosage compared with the short-term biofouling study accelerated with nutrient dosage. In the long-term study without substrate dosage, the ATP concentration was 10^3 pg ATP cm^{-2} (Fig. 2(B)), approximately 10^2 folds lower than the ATP concentration of 10^5 pg ATP cm^{-2} for the short-term study with substrate dosage (Fig. 1(B)). Once again there was no significant difference between the quantities of viable biomass accumulated on the different feed spacers (Fig. 2(B)). The feed channel pressure drop increase differed significantly for reference and modified feed spacers after 96 d of operation (Fig. 2(A)). The ranking of performance for the six feed spacers in terms of pressure drop increase remained consistent at 45, 60, 75 and 90 d, demonstrating that experiments lasting a shorter duration than 96 d are also suitable. The spacer geometry influenced the hydrodynamics of the spacers in the long-term study in the same ranking as in the short-term biofouling study.

3.3. Biofouling impact: short term vs. long term

The short-term and long-term biofouling impact of feed spacers was compared based on the hydraulic resistance in terms of feed channel pressure drop increase. The spacer with the lowest pressure drop increase was ranked as having the best performance (Fig. 3).

At the end of the 9-d study, spacer LXS-ASD had the lowest pressure drop increase (48% of the initial feed channel pressure drop of 25 mbar). Reference spacer CON-1 had the highest pressure drop increase (300% of the initial feed channel pressure drop of 50 mbar). All four modified feed spacers performed better than reference spacer CON-1. Based on pressure drop increase in time, the spacers were ranked as follows: LXS-ASD/ASDi > HYD > DOW > CON-1 (Fig. 3).

The results of the 96-d long-term study corresponded with the 9-d short-term study in terms of biofouling impact. Spacer LXS-ASD had again the best performance with the lowest pressure drop increase of approximately 37% (from an initial feed channel pressure drop of 38 mbar), while reference spacer CON-1 had the highest pressure drop increase (above 50% of the initial feed channel pressure drop of 53 mbar). All feed spacers with modified geometry had less impact on biofouling than the two reference feed spacers. Based on pressure drop increase in time, the spacers were ranked as follows: LXS-ASD > HYD > DOW > CON-3 > CON-1 (Fig. 3).

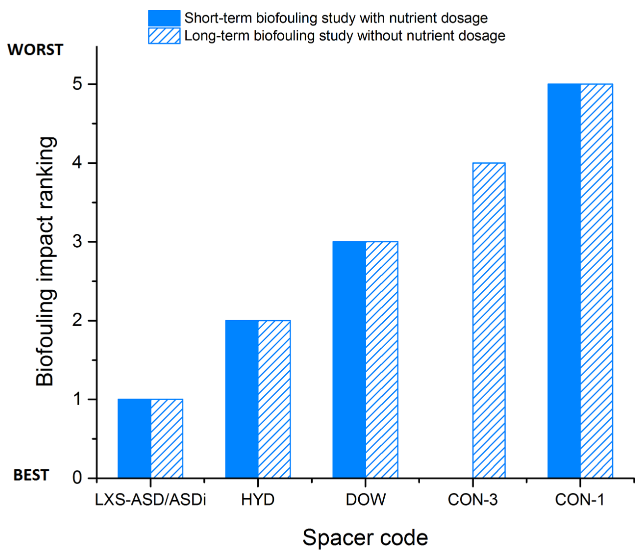

Fig. 3: Biofouling impact ranking of two reference and four modified feed spacers based on the 9-d short-term (with nutrient dosage) and 96-day long-term (without nutrient dosage) biofouling studies [20].
Note: The *Y*-axis of Fig. 3 represents a scale 1–5 from best to worst performing spacer, with the best spacer allotted number 1 and the worst spacer allotted number 5.

The low pressure drop increase of LXS-ASD spacer may be attributed to the large mesh size and the alternating thick and thin strand arrangement. Another spacer with alternating strand thickness, LXS-ASDi, also had a lower pressure drop increase than other spacers.

4. Discussion

Lab-scale experiments investigating filtration materials, biological mechanisms and cleaning strategies are vital for understanding the effects of various parameters of interest on the fouling behavior of membrane filtration systems. The representativeness of short-term lab-scale studies for full-scale operation is scrutinized due to (i) the differences in time scales, (ii) fluctuations in feed water parameters and (iii) dissimilar hydrodynamic conditions in the laboratory set-ups vs. full-scale membrane modules [31,40]. Kraume et al. [31] suggested that for the interpretation of lab-scale results and their application to full-scale plant operation it is critical to conduct lab trials under conditions mimicking those at full-scale. With regards to the duration of biofouling experiments, Miller et al. [41] concluded that short-term (<24 h in duration) experiments are not representative of full-scale biofouling. Therefore, experiments lasting 5–10 d in duration should be carried out under conditions representative for practice. The timescales used in this study were therefore suitably allotted for the short-term (9 d) and long-term (96 d) experiments.

The MFS is representative of spiral-wound membrane elements used in practice with regards to the materials used (membranes and spacers), spatial dimensions (height of feed and product spacer channels) and hydraulics (pressure drop, flow rate and flow distribution) [26]. A comparative study of the MFS and membrane modules showed the same development of pressure drop in time and the same viable biomass accumulation [30]. This research effort has further validated that the MFS is a suitable tool for conducting lab-scale biofouling studies which can, within a short time frame, accurately predict long-term biofouling behavior. The results demonstrate that the MFS studies can facilitate the evaluation and optimization of newly developed spacers, avoiding expensive, time-consuming, chemically wasteful and destructive analysis of full-scale membrane elements. For instance, in terms of spacer geometry, our findings showed that the combination of a larger mesh size and alternating strand thickness (as of spacer LXS-ASD) is a promising modification of the standard geometry of feed spacers used in practice. The findings correspond with the flow profiles of feed spacers predicted by numerical modelling, revealing that LXS-ASD was amongst the best of the evaluated spacers, while the highest pressure drop increase was measured for the reference spacer CON-1 [33].

Although the nutrient load is the key parameter for biofilm formation, the impact of a certain amount of biomass on membrane performance also depends on the design (e.g. feed spacer geometry) and operational aspects of the membrane system [42]. For both the short-term and the long-term studies, a similar amount of viable biomass accumulation was obtained for all feed spacers evaluated. Both studies showed significantly different pressure drop increase for reference and modified feed spacers. Feed channel pressure drop measurements are based on the resistance that water experiences when flowing in the feed channel and the location where the biofilm develops determines the resistance per biofilm volume. The spacer region has the largest pressure drop along the feed channel [43] and therefore it is expected that even with the same amount of active biomass present the spatial variation in the biomass development locations due to the different feed spacer geometries will have a different impact on pressure drop. The ranking of the biofouling impact of the feed spacers based on pressure drop increase was the same for short-term and long-term studies. The correlation between short-term and long-term studies is established by the biofouling susceptibility of reference and modified feed spacers. Regardless of the extent of biofouling and the concentration of nutrients, the same biofouling impact ranking based on pressure drop increase was obtained for the six feed spacers. In other words, the short-term accelerated biofouling study predicted the ranking for feed spacer performance for the long-term study without nutrient dosage. For the long-term study, the biofouling impact ranking of feed spacers consistently remained the same after 45, 60, 75 and 90 d.

Microbial biofilm formation has been described as a successional process in structure and composition [44]. Typically, fouling may begin with cake formation within a few minutes of operation, leading to residual fouling after 1–2 weeks and progressing to irreversible fouling after 6–12 months of operation [31]. Experiments that do not last longer than a few hours or some days may only provide data for cake formation and early residual fouling rates. Characterization of the biofilm during the short-term and long-term biofouling studies can provide information on biofilm morphology and composition at the early and late stages of biofilm maturation. In summary, short-term biofouling studies are predictive for the long-term performance evaluation of membrane systems. Short-term studies can be used for rapidly assessing the effects of key operational parameters related to biofouling prior to implementing the methods of operation in practice, leading to the development of membrane systems less susceptible to fouling.

5. Conclusions

Short-term and long-term biofouling potential for different geometry feed spacers was evaluated by performing biofouling studies in MFSs with nutrient dosage (9-d study) and without nutrient dosage (96-d study). Based on the results of this study it can be concluded that:

- Short-term, lab-scale biofouling studies carried out using MFSs are predictive for long-term impact of biofouling.
- The accumulated viable biomass content is independent of the type of feed spacer.
- The impact of biofouling on the hydrodynamic behavior characterized by pressure drop increase of feed spacers is influenced by the spacer geometry.
- The application of a high or a low nutrient dosage did not change the ranking of modified feed spacers based on biofouling development.
- Pressure drop measurements and quantification of biomass must be performed complementary to each other in order to gain a full understanding of the extent and impact of biofouling under different operating conditions.

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