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The evolution of pore-blocking during the ultrafiltration of anaerobic effluent–like mixtures

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ABSTRACT

The global market for membrane bioreactor technology (MBR) is growing fast. Membrane biofouling hinders the broader application of MBR because it reduces permeate yield. The main goal of this manuscript was to identify the arrangement of fouling mechanisms during the ultrafiltration (UF) of suspensions comprising extracellular polymeric substances (EPS) and inorganic colloids (IC). Standard blocking (SB) was the main operative fouling mechanism in the early stages of filtration, which eventually shifted to cake filtration (CF). Longer SB stages were observed in UF experiments with a slight electric charge density ratio (ECD). CF was the predominant fouling mechanism with a higher ECD and led to the formation of mineralized-EPS-building blocks (MEBB) within the cake. MEBB accumulation in the membrane produced sharp reductions in filtration performance after 0.5 and 1.5 mM CaCO₃ were added. Non-covalent interactions fostered the development of irreversible fouling and biomineral seeds on membranes.

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1. Introduction

The global market for membrane bioreactor technology (MBR) is growing fast (15% per year according to [1]), largely because MBR wastewater treatment plants (WWTP) are relatively easy to deploy and capable of producing clean water that meets stringent regulations for direct water reclamation. In comparison to other technologies for municipal sewage treatment, such as activated sludge, MBR WWTPs are not only smaller but also more efficient at removing pollutants and are capable of providing disinfected water [2]. During the last decade, dozens of full-scale MBR plants were installed in Japan, Korea, China, Germany and the USA. This success was the result of strategic partnerships between governments, enterprises and research centers (i.e., MBR-network, E-water, PERMEANT). The knowledge associated with this technological milestone (i.e., optimal strategies in the design, operation and maintenance of the membrane process) has fostered the potential of anaerobic MBR (AnMBR) as a practical alternative for municipal wastewater treatment [3,4].

Although the AnMBR concept was developed more than 30 years ago, its commercial applications have been limited by

the efficiency of the membrane process due to fouling. Fouling control, especially in long-term operation, seems to be the most critical constraint for the applicability and feasibility of AnMBR in the growing field of municipal wastewater treatment [3]. So far, membrane fouling in AnMBR has not been fully understood due to the complex nature of anaerobic effluent and the diversity of operational conditions and bioreactor configurations. AnMBR systems will likely benefit from the development of efficient technologies to prevent fouling [4]. Non-covalent interactions (hydrogen bonding, metal coordina-

tion, van der Waals forces), between extracellular polymeric substances (EPS) and inorganic colloids (IC) have been identified as potential causes of irreversible fouling during water treatment performed by membranes; Ma et al. [5] suggested that the biomineralization of Fe crystals onto the extracellular matrix was the cause of irreversible scaling during a hybrid (coagulation/UF) desalination plant's pretreatment. Later, Teychene et al. [6] reported that interactions between the acidic chemical groups in EPS (i.e., COO-) and Ca ions were able to enhance the stiffness of fouling layers. Herrera-Robledo et al. argued that metal complexation by EPS fostered the deposition of insoluble deposits in the fouling layers during AnMBR operation [7].

In addition to the abiotic components, several strains of EPS-releasing bacteria have been identified in membrane fouling and could influence the dimensions and structure of the layer [8–11]. In considering biotic/abiotic interactions, other reports concluded that biomineralized deposits are important structural components







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of cake layers and could be the origin of irreversible membrane fouling [12–14].

In accordance with the premise that 'fouling phenomena in AnMBR should be investigated under well-defined combinations of parameters and conditions which individually might even present opposing effects' [3], this research aimed (1) to describe the evolution of pore-blocking during the ultrafiltration of anaerobic effluent-surrogates using available mathematical models and (2) to discuss the links between biologically-induced mineralization, irreversible fouling and pore-blocking.

2. Materials and methods

2.1. Membranes and cross-flow UF apparatus

Single membrane tubes (polyvinylidene fluoride with a diameter of 1.1 cm and 100 kDa as the nominal molecular weight cutoff) were removed from a commercially available UF module (ULTRA-COR© module: 10-HFM-251-UVP by Koch Membrane Systems, MA), cut into smaller ones (5.8 cm length) and then stored at 4 °C in bottles with DI water. The operational surface area of each UF experiment was 2.35×10^{-3} m². Prior to each UF experiment, the membrane tubes were compressed for 120 min at a transmembrane pressure (TMP) of 190 ± 10 kPa.

A closed filtration loop was assembled using a cylindrical recirculation tank (operative volume of 2.5 l; Plexiglas), high-performance tubing (Pliovic © with internal diameter of 3/4'', by Goodyear), two $\frac{1}{2}''$ globe valves and a positive displacement pump with electronic control (Moyno 33201, 1/2 HP, by Baldor, AR). The TMP was measured using a pressure transmitter (0–2100 kPa, Model 68001 by Cole Parmer, IL) and transferred to a personal computer through a data acquisition card (Ni6009 by National Instruments, TX). The permeate volume was measured at fixed time intervals using 100 ml graduated cylinders.

2.2. Feed water

Low molecular weight chitosan (LC) and bovine serum albumin (BSA) were selected as EPS surrogates. The carbohydrate to protein ratio in feed waters (Table 1) was set considering the outcomes of a long-term anaerobic bioreactor operation [14]. Two liters of feed water with 116.5 mg L⁻¹ of EPS were used in each filtration experiment, and the pH of suspensions ranged from 6.24 to 7.18 (more acidic as BSA increased).

The electric charge density (ECD), an empirical relationship between a polymer's molecular weight and a monomer's electric charge, was estimated for each EPS component in accordance with De la Torre et al. [15]; the ECD in chitosan was estimated with the consideration that each of its monomers (glucosamine) contributes a positive charge because the amino group has a pKa of ~6.5, which leads to a protonation in acidic and neutral solutions. In the case of the BSA, the ECD estimation considered that each mol of BSA contributed 18 negative charges because the BSA primary structure is acidic (-18) in a neutral pH. Table 1 illustrates several physicochemical properties of the EPS suspensions. A total

Tuble							
Main	phy	vsico	chemical	features	of	feed	water.

Table 1

rejection of the EPS in the membrane was expected, considering that the DLS particle size ranged from 0.22 to 0.41 μ m (data not show). CaCO₃ was added to EPS suspensions in three different concentrations: 0.5, 1.5 and 3 mM.

2.3. Inoculation of metabolic end-products and fermentation

An aliquot of 10 ml of suspended solids-free anaerobic effluent [16] was released into each EPS-CaCO₃ suspension to foster biomineralization [17]. To promote the development of mineralized-EPS-building blocks, each feed was adjusted to a pH 7 \pm 0.05 using HCl and NaOH and incubated at room temperature (25 °C) overnight before UF tests.

2.4. Constant-TMP filtration tests

In a separate set of tests, each incubated feed water was filtered through UF membranes for 6 h at a constant TMP of 180 ± 10 kPa. The permeate volume was measured and periodically re-circulated to the feed tank. The operative mechanisms of membrane fouling (standard, intermediate and complete blocking, as well as cake filtration) were determined using available fouling models for EPS [18] and inorganic colloids [19]. Those mathematical models utilized correlations between experimental data [such as the permeate volume (*V*), initial and temporal flux (J_0 and J_t), flow rate (*Q*) and filtration time (*t*)] to identify the predominant fouling mechanism (Table 2).

2.5. Post-utilization examination on membranes

To estimate the amount of solid deposition on fouled membranes, each of the membranes was cut into small coupons (\sim 0.025 m²) and their dry mass was measured. To obtain a membrane weight reference, randomly selected membrane coupons were the subject of chemical cleaning operations; coupons were shaken for 72 h in a 50 ml-tube containing abrasive glass particles and diluted NaOH (pH 10) and then transferred to other tube containing glass particles and diluted HCl (pH 2) to complete another 72-h abrasive cycle. The resultant chemically cleaned membranes (CCM) were dried at 105 °C for 6 h prior to mass and surface measurements. The specific membrane mass (SMM: mg cm⁻²) of the CCM was then calculated and used as the basis to estimate the specific biofouling mass (SBM).

SEM imaging was performed on fouling-bearing samples in a Jeol JSM 5310 LV SEM without any previous pretreatment.

3. Results and discussion

3.1. Pore-blocking evolution and the relationship to the decrease in flux

Standard blocking (SB) by inorganic colloids was the main fouling mechanism in early stages of filtration, which, sooner or later, shifted to cake filtration (CF) (Fig. 1; the supplementary information provides deeper information about this issue in Table SM-1).

LC/ BSA	[LC] mol l ⁻¹	[BSA] mol l ⁻¹	Number of (+) charges in LC	Number of (–) charges in BSA	ECD _{LC} charges mol g ⁻¹	ECD _{BSA} charges mol g ⁻¹	ECD _{LC} to ECD _{BSA} ratio
0.25 0.42	1.19E–05 1.78E–05	1.41E-06 1.23E-06	2.32E-02 3.48E-02	2.95E-05 2.57E-05	1.28E-04 1.92E-04	2.61E-07 2.27E-07	490 846
0.75	2.56E-05	1.00E-06	4.99E-02	2.10E-05	2.76E-04	1.85E-07	1492
1.49	3.58E-05	7.00E-07	6.99E-02	1.46E-05	3.86E-04	1.29E-07	2992
1.85	3.86E-05	6.10E-07	7.54E-02	1.29E-05	4.16E-04	1.14E-07	3649

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