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Influence of relaxation frequency on membrane fouling control in submerged anaerobic membrane bioreactor (SAnMBR)

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ABSTRACT

The effect of different intermittent filtration modes on membrane fouling was investigated in a submerged anaerobic membrane bioreactor treating palm oil mill effluent. The filtration was operated at the range of supra critical conditions (permeate flux equal to $20\,\mathrm{L\,m^{-2}\,h^{-1}}$), and the submersed membranes were continuously cleaned by gas injection and intermittent periods of relaxation. Four conditions of relaxation (S1: 240 s filtration/30 s relaxation, S2: 480 s filtration/30 s relaxation, S3: 720 s filtration/30 s relaxation, and S4: 960 s filtration/30 s relaxation) were analyzed by comparing the trans-membrane pressure evolution rates, the main fouling origins, and the content of membrane cleaning solutions in terms of proteins and carbohydrates. Results showed (i) the dominant effect of the cake deposit (50% of the total hydraulic resistance) whatever the relaxation frequency, (ii) the equivalent importance of pore blocking and adsorption, and (iii) the clear interest of working with the highest relaxation frequencies to limit the specific hydraulic resistance (50 times lower in comparison with low relaxation frequencies). Moreover, the presence of SMP and EPS was found in the different cleaning solutions confirming their determining role in fouling mechanisms.

Keywords: Submerged anaerobic membrane bioreactor; Palm oil mill effluent; Intermittent filtration modes; Fouling

1. Introduction

In recent years, the submerged anaerobic membrane bioreactors (SAnMBRs) have appeared as an increasingly interesting solution for treatment of municipal as well as many kinds of industrial wastewaters, because they present numerous advantages over the conventional anaerobic treatment processes.

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Indeed the use of membranes to separate the biomass from the effluent can maintain a high concentration of microorganisms in SAnMBR resulting in a highly efficient removal treatment (more than 90% of biodegradable organic matter and total removal of suspended solids, SS) including recovery of renewable energy sources (0.371 CH_4/g $COD_{removed}$). Moreover, the solid liquid phase separation on porous membranes avoids any wash out of biomass even under extreme conditions (elevated temperatures, pH, high content of organic pollutants, and salt) as indicated by Jeison et al. [1], Gao et al. [2], and Abdurahman et al. [3]. Thus SAnMBR can maintain a high solid retention time (>100 d) coupled with high loading rates, which results in less sludge production and HRT shortening [4,5].

In addition, the filtration through porous membranes not only produces better effluent quality including disinfection in relation to water reuse applications such as washing, agricultural irrigation, and power plant cooling, but also eliminates the necessity of large clarifying basins to settle out the biomass [6–8]. Then, SAnMBR appears as an intensive process with regard to conventional anaerobic digesters [9].

However, the control of membrane fouling during operation remains the main challenge in SAnMBRs. Because of filtration, a lot of compounds present in suspension that are retained by the membrane barrier interact with the membrane material, then membrane permeability is drastically modified obliging the use of energy to maintain it at a sustainable level (gas bubbling, backwashing, and chemical consumption) and the intensity and frequency of membrane cleaning can significantly shorten membrane life-time [10–12]. Membrane fouling can be attributed to different reversible and irreversible mechanisms such as membrane pore blocking, adsorption of foulants, concentration of polarization, and sludge cake formation [13]. For SAnMBRs application, the cake layer formation has been identified as the predominant fouling origin [14]. According to the filtration conditions, sub or supra critical conditions, the cake layer formation is mainly due to (i) Extracellular polymeric substances (EPS) adsorption and individual bacteria deposition onto the membrane surface, after which the cells multiply and form a cake layer as biofilm [14] and (ii) accumulation of SS until a structured deposit is formed which can be progressively compressed, dewatered, and made denser causing some high hydraulic resistance and rapid flux decline or transmembrane pressure (TMP) increase [14–16].

Wu et al. [17] present research that was applied for the treatment of palm oil mill effluent that contains high concentrations of oil and grease, organic matter, SS, protein, and polysaccharide. Moreover the SAnMBR generally operates with high concentrations of SS and colloid matter, so the biological suspensions appear more viscous than the suspensions in the aerobic MBR. It is then more difficult to induce controlled and homogenous turbulences close to the membrane surface. Moreover when filtering is so complex, suspensions in which it appears has more intensive interactions between colloids and membrane materials and the cake structuring is rapid, more resistant, and becomes the determining step of fouling to be controlled [5]. To avoid or to minimize this cake structuring, it is then important to combine complementary techniques: biogas injection or biogas recirculation as a replacement for air bubbling in the aerobic SMBR with intermittent filtration modes [18] by introducing relaxation periods because (i) continuous filtration generally induces a denser and more compressed cake layer and (ii) intermittent filtrations offer a potential solution to favor membrane fouling reduction and energy saving [19–23].

The proposed document is then focused on (i) the influence of intermittent filtration frequency on filtration step control and (ii) to quantify the main origin of membrane fouling.

2. Materials and methods

2.1. Experimental setup

The laboratory scale SAnMBR reactor used was composed of a 5 L chamber equipped with an immersed fiber module as shown in Fig. 1. The reactor was filled with a biological suspension coming from a fullscale biogas plant. After collecting the biological suspension, it was incubated at 35˚C for, approximately, one week in order to reduce the biodegradable content of the sludge before experiments. The characteristics of the suspension are given in Table 1.

The membrane module equipped with polyvinylidene fluoride (PVDF) hollow fibers was located at the upper part of the reactor. The hydrophilic PVDF membranes had a nominal pore size of 0.1 μm (obtained from Shanghai Jofur Advanced Materials Co. Ltd, China). The effective filtration area was 0.1 m^2. A diffuser providing nitrogen bubbles was setup at the bottom of the membrane module. Permeate was filtrated from the SAnMBR at a constant flux by using a peristaltic pump and it was recycled into the bioreactor to work at constant MLVSS concentration. The TMP was continuously monitored by a pressure transducer setup on the permeate side of the

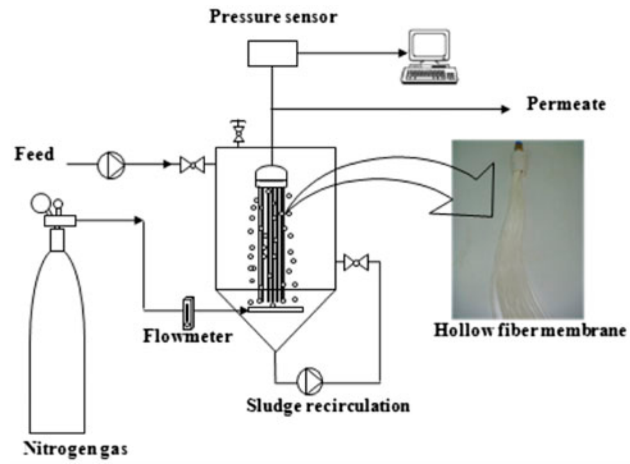

Fig. 1. Schematic diagram of the laboratory scale submerged anaerobic membrane bioreactor.

membrane module. A computer combined with data acquisition was conducted with LabView (National Instruments, Austin, USA) and used to record the TMP data in real time.

2.2. Operation conditions

To highlight the importance of the cake deposit on membrane fouling rates, according to Jeison and van Lier et al. [24] the membrane module was operated in the range of supra-critical conditions (about $20\,L\,m^{-2}\,h^{-1}$). The filtration was shut down when the TMP reached a value close to 250 mbar (25 kPa), and the membrane module was then taken off from the SAnMBR and cleaned according to a specific procedure (see analytical methods) to identify the main origins of membrane fouling.

In order to minimize membrane fouling dynamics, experiments were operated for four short-term intermittent filtration modes including only a relaxation period of 30 s (without any backwashing): S1: 240 s filtration/30 s relaxation, S2: 480 s filtration/30 s relaxation and S3: 720 s filtration/30 s relaxation, and S4: 960 s filtration/30 s relaxation. In each case, the nitrogen gas injection flow rate close to the membrane surface was $1.8\,m^3\,h^{-1}\,m^{-2}$. The filtration and the membrane cleaning were operated at 30℃.

2.3. Analytical methods

2.3.1. Hydraulic resistances

The membrane fouling was identified as being caused by several resistances in a series based on Darcy's law:

$$R_t = R_m + R_{cake\ deposit} + R_{pore\ blocking} + R_{adsorption} = TMP/\mu J$$

where R_t is the total membrane resistance at the time of cleaning (m^{-1}), R_m is the clean membrane resistance (m^{-1}), $R_{cake\ deposit}$ is the cake layer resistance (m^{-1}), $R_{pore\ blocking}$ is the pore blocking resistance (m^{-1}), $R_{adsorption}$ is the resistance due to compounds adsorbed in pores and only removable by chemical cleaning (m^{-1}), J is the membrane flux ($m^3\,m^{-2}\,s$), TMP is the trans-membrane pressure (Pa), and μ is the dynamic viscosity of permeate considered as water ($0.81{\cdot}10^{-3}$ Pa.s at 30℃).

The quantification of each resistance was done step by step. R_m was determined by measuring the water flux when filtering DI water with a new membrane (R_m was found equal to $2.5\times10^{11}\,m^{-1}$ in this study) and R_t was calculated at the final time of operation before membrane cleaning (it corresponded to the maximal value of TMP at the end of each experiment).

To evaluate each fouling resistance, a specific cleaning procedure was carried out. When the TMP reached a value close to 250 mbar, the membrane module was taken off from the reactor and then three successive steps of cleaning were practiced:

(1) Physical cleaning was carried out by scraping off the cake layer from the membrane surface carefully by using a plastic sheet. The membrane surface was then rinsed with DI water to

Table 1
Characteristics of the anaerobic suspension in the SAnMBR.

Parameter	Range and macromolecular types		
1. MLSS (g/L)	16.7 ± 2		
2. MLVSS (g/L)	10.5 ± 2		
3. Macromolecular in Sample	*Protein*	*Carbohydrate*	*Protein/carbohydrate (PN/PC)*
Mix liquor (mg/L)	547.6 ± 55	293.4 ± 65	1.9
SMP (mg/L)	130.2 ± 4.3	63.9 ± 2.5	2.0
EPS (mg/L)	190.4 ± 16.4	92.3 ± 3.7	2.1

remove any sludge entrapped in the module. Then membrane resistance was measured by filtering DI water. It corresponded to R_1.

(2) Backwashing with DI water was then carried out for 2 h to remove any compounds inducing pore blocking. Then membrane resistance was measured by filtering DI water. It corresponded to R_2.

(3) Finally, a chemical cleaning was carried out by soaking the membranes (i) in a 0.5% NaOH solution for 2 h. and then after rinsing (ii) in a 0.5% NaClO solution for 2 h. After rinsing, the membrane resistance was measured by filtering DI water. It corresponded to R_3.

The resistance due to each identified phenomenon could then be deduced as follows:

- $R_{\text{cake deposit}} = R_t — R_1$ (Residual resistance after physical cleaning)
- $R_{\text{pore blocking}} = R_1 — R_2$ (Residual resistance after backwashing with DI)
- $R_{\text{adsorption}} = R_2 — R_3$ (Residual resistance after chemical cleaning)

After the last cleaning step, the residual hydraulic resistance R_3 was equal to R_m.

2.3.2. The soluble microbial products and EPS extraction and analysis

EPS and soluble microbial products (SMP) have been established as an important cause of membrane fouling by structuring cake deposit and favoring pore size decrease. EPS and SMP typically consist of carbohydrates (PC), proteins (PN), nucleic acids, lipids, and other polymeric compounds coming from bacterial growth and decay [25–26]. The PN/PC ratio in EPS and SMP can also appear as a criterion of membrane fouling dynamics [27–28].

SMP and EPS concentrations were normalized as the sum of protein and polysaccharide. Protein concentration was analyzed according to the colorimetric method which suggested the use of the modified Lowry procedure of Peterson [29], which was using bovine serum albumin as a standard protein. Carbohydrate concentration was analyzed by the phenol-sulfuric acid methods using D-glucose as a standard carbohydrate. Carbohydrate samples analyses were measured at 480 nm [30].

It was important to quantify the SMP and EPS not only in the filtered suspension but also in cleaning solutions. The rinsing, backwashing, and desorption solutions were collected and centrifuged at 6,000 rpm for 30 min and the centrifuged supernatant was filtrated through a membrane with a mean pore size of 0.45 μm.

3. Results and discussion

3.1. Hydraulic filtration performances

The TMP evolutions for the four relaxation conditions are shown in Fig. 2. All curves can be divided in two parts with time. The first is a progressive increase of TMP with time until the maximal allowed value was reached (the second part of the curve where TMP appeared constant, might correspond to permeate pump limitation due to the depression observed in the pipe). Only the first part of TMP evolutions has been discussed, they correspond to constant permeate flux. The highest points in the curves correspond to the TMP measured during filtration, and the lowest correspond to the TMP values at the end of each relaxation period. Results allow the differentiation of S1 and S2 from S3 and S4.

For S1 and S2 (frequent relaxation periods), relaxation allowed a net decrease of TMP when comparing the TMP at the end of each filtration period and its value at the end of the following relaxation period. Even with a progressive TMP evolution when the filtration was operating, relaxation was able to regenerate partially the membrane permeability during more than 5–6 h with an average TMP evolution rate of about 0.6–0.8 mbar/min. After this first period, it was obvious that relaxation was not sufficient to maintain the membrane fouling dynamics.

On the other hand, for S3 and S4, too long a time between two relaxation periods did not allow any control of TMP evolution and the maximal value was reached in hardly 30 min, about 10 times lower than observed in S1 and S2 conditions (the average TMP evolution rate was close to 8 mbar/min). This point was soon noticed by other authors (Bae et al. [31]).

The relaxation frequency appeared as determining to favor the control of TMP evolution. As discussed by Huang et al. and Wang et al. [32–33], the slow increase of TMP in the early stage was related to the formation of a biogel layer due to ESP and SMP adsorption. Biogel layer makes easier the attachment of small flocs and/or bacterial clusters on the membrane surface to induce sludge cake formation. During relaxation, the loosely attached compounds were removed from the deposit. The high relaxation frequency avoided any rapid structuring of the cake deposit but it appeared not to be sufficient in these experiments to limit the TMP increase during filtration. That can be due to

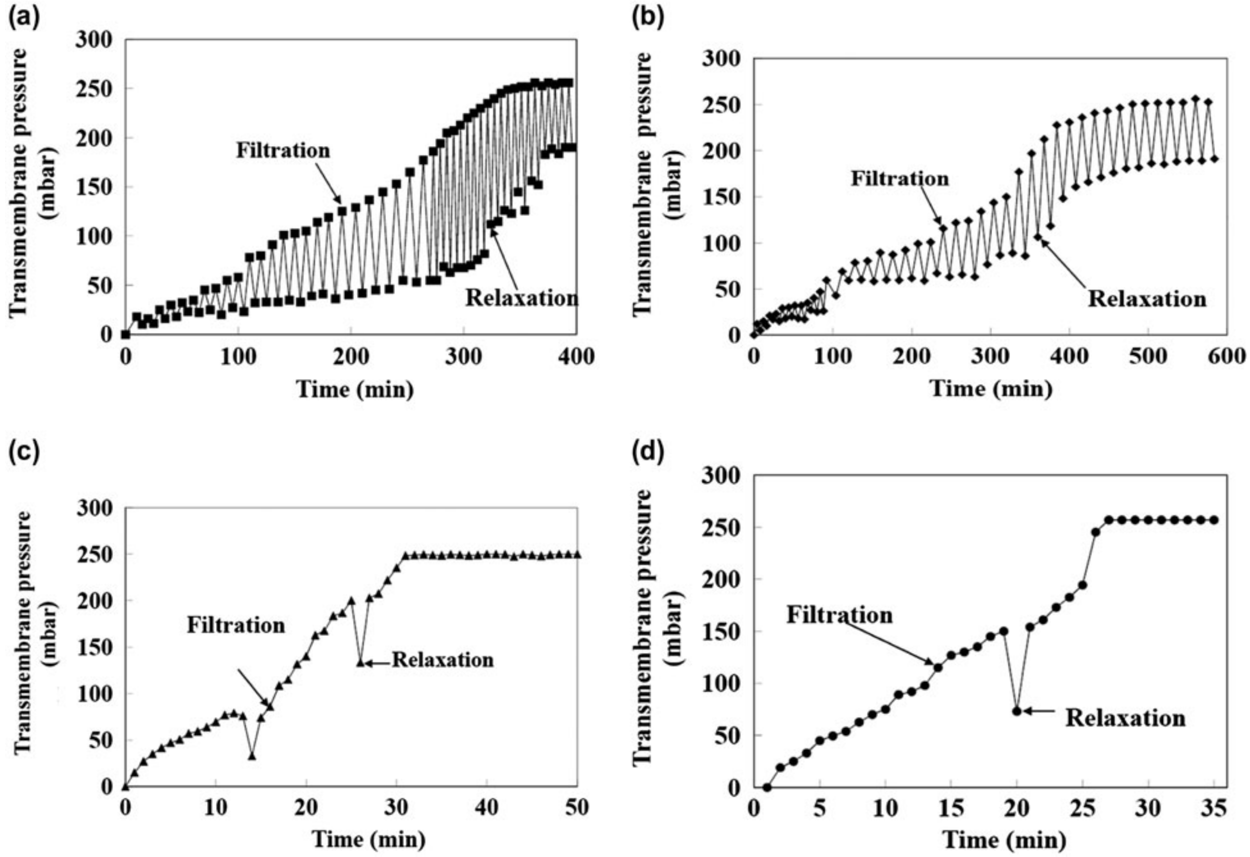

Fig. 2. Variations of TMP under different intermittent permeate filtration modes (a) 240 s filtration/30 s relaxation (S1); (b) 480 s filtration/30 s relaxation (S2); (c) 720 s filtration/30 s relaxation (S3); and 960 s filtration/30 s relaxation (S4).

- A progressive compression of the deposit becoming less breakable. Moreover, with time, the deposit structure became comparable to a dense biofilm composed by mucilaginous biomass presenting low water permeability.
- The other origins of fouling. If relaxation can limit the deposit development it is inefficient for pore blocking and adsorption of solutes in pores. According to the intensity of these phenomena, relaxation can then appear as insufficient with time.

Cleaning procedure confirmed this analysis. Compounds stored on the membrane surface were collected after the initial cleaning of the membranes for each condition. The intermittent filtration 960/30 s relaxation (S4) showed the highest foulant content of 1,173 mg/m^2, while the lowest biofoulant content was observed in the intermittent filtration 480/30 s relaxation (S2) (593 mg/m^2). This indicated that membrane fouling could be dominated by cake layer formation while operating at a low relaxation period, whereas high relaxation frequency may also be influenced by pore-blocking and adsorption.

As expected, the optimal relaxation frequency was dependent on the suspension characteristics and the filtration conditions (permeate flux in regard with local shear stresses). In this experiment, the permeate flux value chosen was very high (for SAnMBRs) to highlight the role of relaxation as a simple means of membrane fouling control (relaxation being an economic cleaning procedure: no loss of filtered water and energy necessary with backwashing, for example).

3.2. *Hydraulic resistances of the different intermittent filtration modes*

Fig. 3 presents the hydraulic resistances due to the main causes of fouling as defined in "Materials and methods". Whatever the relaxation frequency maybe, the external deposit appeared as the more influent phenomenon on global hydraulic resistance (about 50%). The two other origins of fouling were of the same order of magnitude and even the pore blocking seemed slightly higher. Thus the role of deposit remained important though the external cleaning by

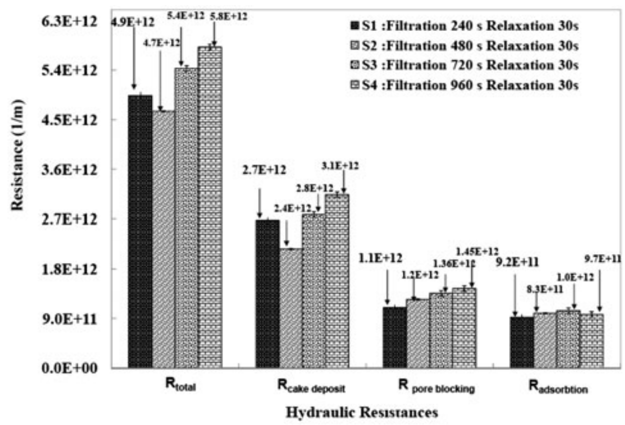

Fig. 3. Hydraulic resistances versus intermittent permeate filtration modes.

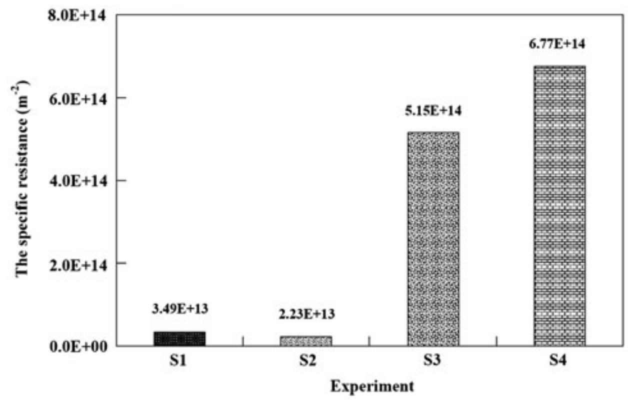

Fig. 4. Specific resistance R^*.

relaxation and gas bubbles under working in supracritical conditions. Pore blocking appeared also as an important phenomenon of fouling. A combination of relaxation and backwashing could then offer a better control of fouling dynamics by letting only molecule adsorption in the membrane pores as the limiting step.

Even if the permeate flux was the same for each intermittent condition, the time of operation was different till reaching the maximal trans-membrane pressure for the different intermittent conditions. Then to compare the hydraulic resistance, it can be interesting to define a specific hydraulic resistance expressed for the same permeate volume whatever the conditions maybe. A specific hydraulic resistance R^* (m^{-2}) was then calculated as the ratio between the total hydraulic resistance and the cumulated permeate volume, per unit of filtration area, obtained when the maximal allowed trans-membrane pressure was reached (just before cleaning). Fig. 4 presents this hydraulic resistance for each intermittent filtration mode. The result clearly confirms the interest of working in conditions S1 and S2. The specific resistance R^* of S4 was about 50 times higher than that of S2, respectively. The results indicated that the specific resistance drastically increased with low relaxation frequency.

3.3. *Role of SMP and EPS in fouling*

At the end of each experiment, the first step of membrane cleaning allowed the recovery of sludge flocs, biogel, and mixed liquor entrapped on the membrane surface and in the capillary fiber network. Then for these first rinsing solutions, it was possible to quantify the concentration of linked EPS and soluble biopolymers as indicated in materials and methods.

Regarding the soluble compounds (SMP) more specifically, the three cleaning solutions (rinsing, backwashing, and desorbing solutions) were characterized in terms of protein and carbohydrate concentrations (as shown in Figs. 5 and 6). The presence of soluble protein SMPp appeared dominant in the cake deposit, about two times higher than in pore blocking and adsorption. The lower concentrations of SMPp and SMPc observed for S1 and S2 conditions (Fig. 5(a) and (b)) confirmed the interest of these working conditions. The decrease of relaxation frequency increased significantly the retention of SMP onto the membrane surface and in pores.

Table 2 shows the PN/PC ratio in the three cleaning solutions. This ratio appeared higher in the first cleaning solution (cake deposit removal) corresponding to the higher fouling effect. It can be due to the cake deposit capacity to retain/adsorb a part of SMP [3]. Nevertheless no actual influence of the relaxation frequency could be noticed. Fig. 6 presents the linked EPS content (protein (PN) and carbohydrate (PC) and PN/PC ratio in the first rinsing solution (cake deposit recovery) for the four studied filtration modes.

Protein was found to be the major component in the EPS rather than carbohydrate. The interest of the S2 condition (480 s of filtration vs. 30 s of relaxation) can be confirmed by other literature data:

- Wu et al. [21] mentioned the interest of time of filtration higher than 220 s (optimal at 440 s) between relaxation periods to obtain a more efficient effect on TMP control.
- In addition, the relaxation time has a strong effect on fouling characteristics. Braak et al. [18] reported that the relaxation time should be moderate. If this time is too long, foulant material (protein and carbohydrate) was induced to attach and build a

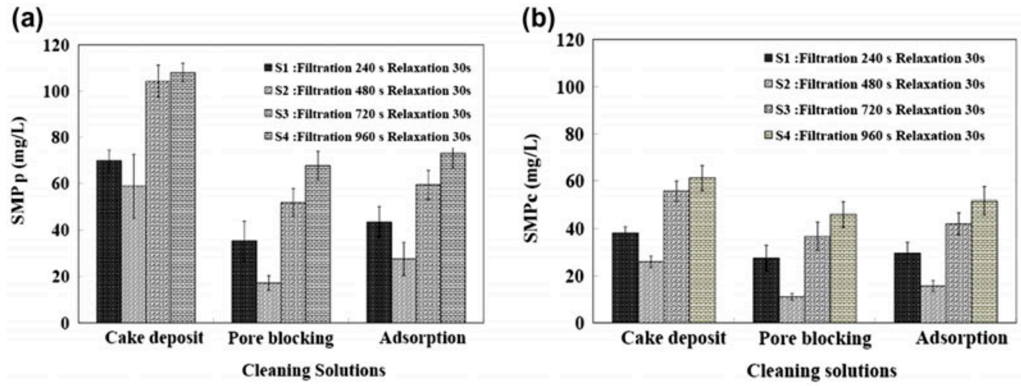

Fig. 5. SMP content (protein + carbohydrate) in different cleaning solutions.

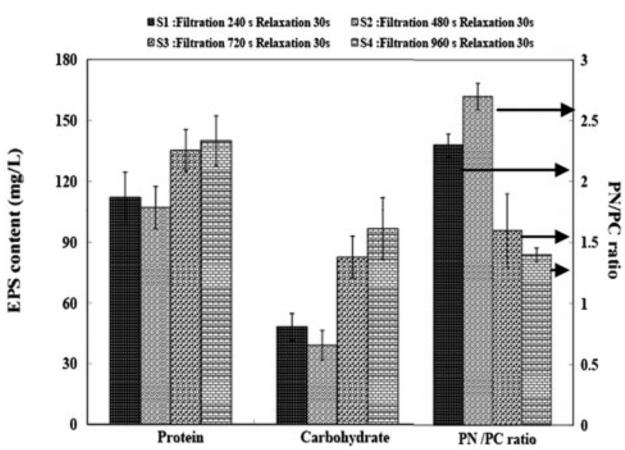

Fig. 6. EPS content (protein and carbohydrate) and PN/PC ratio in intermittent permeate filtration modes.

Table 2
The PN/PC ratio of SMP in different cleaning solutions after practicing cleaning steps.

Experiment	Solution samples		
	Cake deposit	Pore blocking	Adsorption
S1	1.8 ± 0.1	1.3 ± 0.5	1.5 ± 0.3
S2	2.3 ± 0.2	1.6 ± 0.3	1.8 ± 0.4
S3	1.9 ± 0.2	1.4 ± 0.4	1.4 ± 0.4
S4	1.8 ± 0.2	1.5 ± 0.3	1.4 ± 0.3

structured deposit on the membrane surface [23]. If it is too short, it is favorable to their entrance inside the pores to intensify irreversible fouling [18].

- Metzger et al. [16] reported that proteins have higher potential than carbohydrate to deposit/adsorb directly on the membrane surface and appear to be more strongly attached to the membrane. The values of PN/PC ratios observed in our study confirm this observation. S2 condition also induced the lower fixation of carbohydrates (more easily remove during relaxation time).

Liao et al. and Yao et al. [34,35], demonstrated that a decrease in PN/PC ratio (⩽2) could induce a decrease in floc hydrophobicity. The result was a higher resistance from cake formation rather than pore adsorption. Furthermore, Yamato et al. [36] reported that most irreversible fouling on PVDF membrane was caused by dissolved EPS and SMP detected when the PN/PC ratio was close to 1.4. The filtration modes S3 and S4 showed the PN/PC ratio in a range of 1.4–1.6 then it was higher than two for S1 and S2. Then for linked EPS, the ratio PN/PC can be a relevant criterion to qualify the fouling potential of a cake deposit.

4. Conclusion

This study investigated the impacts of different intermittent filtration modes based on different relaxation times on the hydraulic resistance evolutions. The experiments were carried out to analyze the filtering steps of a submersed anaerobic membrane bioreactor SAnMBR. When filtering in supra-critical conditions, the results pointed out the following elements:

- The dominant effect of fouling was the cake deposit (as it is currently observed when filtering in supra-critical conditions), representing about 50% of the total hydraulic resistance. The other origins of fouling were pore blocking and adsorption.
- The highest relaxation frequencies, conditions S1 and S2 allowed an operating time 10 times longer than when working with low relaxation frequencies. The specific resistance of the fouling phenomena

then appeared 50 times lower than when working with the lowest relaxation frequencies.

- Proteins and carbohydrate appeared as the main organic components present in cleaning solutions. They were observed both in soluble and particular fractions. The ratio protein/carbohydrate in linked EPS present in the cake deposit appeared lower when operating at a lower relaxation frequency, and it also corresponded to a higher fouling property of the cake deposit.

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