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Short-term fouling control by cyclic aeration in membrane bioreactors for cosmetic wastewater treatment

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ABSTRACT

Air sparging is extensively used to mitigate membrane fouling in membrane bioreactors (MBRs), which represents an important operation cost in submerged MBR. In this work, the effect of intermittent aeration mode on the membrane fouling has been studied by analysing the influence of the aeration cycle duration in a submerged MBR treating cosmetic wastewater. The long-term filtration at a permeate flux of 8.0 L m^{-2} h^{-1} and a constant specific air demand (SAD) of 1.5 m^3 m^{-2} h^{-1} led to a significant increase in the transmembrane pressure (TMP) after 10 d. The tangential shear showed a limited effect on preventing the deposition of small particles, since carbohydrates and proteins present in the fouling layer were mainly smaller than 1 μm. Flocs showed a low stability under shear stress, which limits the increase in the SAD to avoid undesirable effects over the mixed liquor permeability. The intermittent aeration operation allowed the operation of the MBR under low aeration intensity (low SAD) without compromising the fouling of the membranes. The use of aeration cycles shorter than 1 min led to an efficient control of fouling, which resulted in a negligible increase in the TMP after 2 h, operating at a permeate flux of 12.0 L m^{-2} h^{-1}.

Keywords: Cyclic aeration; Fouling; Membrane; MBR; Specific air demand

1. Introduction

Wastewater from the manufacture of cosmetics is characterized by a variable composition and fluctuations in pollutants concentration, which often make their treatment inefficient in conventional activated sludge systems. The main difficulty in treating cosmetic wastewaters is the presence of detergents, surfactants, personal care products, pharmaceutical compounds, preservatives (normally phenol derivatives), dyes, fragrances and co-solvents, which are often non-biodegradable and/or toxic to micro-organisms in biological systems [1,2]. However, the increasingly stringent requirements, concerning industrial wastewater treatment and reuse, demand the

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implementation of new or improved cost-effective technologies. In this sense, the application of advanced biological systems, such as upflow anaerobic sludge blanket reactors and sequencing batch reactors has been reported recently [3,4]. Membrane bioreactors (MBRs) have been increasingly accepted for industrial wastewater treatment that has been recently installed in a cosmetics factory [5]. The MBR system consists of a biological reactor integrated with membranes that combine clarification and filtration of an activated sludge process into a simplified, single-step process. This system can run at high mixed liquor suspended solids (MLSS) concentration, which reduces the footprint of the reactor system. Additionally, it is possible to reduce the production of excess sludge by working at high solids retention time (SRT), under low F/M ratios. MBRs can produce high-quality effluent potentially suitable for reuse and, over the past decade, have experienced unprecedented growth in municipal and industrial wastewater treatment/reuse due to the robustness of the process.

MBRs are frequently operated at long SRT for the treatment of industrial wastewater, which favours the retention and development of specialist degrading micro-organisms. This fact leads to better removal of refractory organics and makes the system more stable against load variations and toxic shocks. Phylogenetic analysis of the mixed liquor present in a MBR treating cosmetic wastewater indicated that the majority of the micro-organisms were affiliated with α-Proteobacteria, Actinobacteria and Fimicutes. In addition, most of the identified bacterial sequences belonged to either surfactant degrading micro-organisms or biofouling communities in membrane separation systems [6]. However, the fouling of membranes is a major obstacle for the wide application of MBR since the introduction of this technology [7,8]. Air sparging is usually employed to mitigate fouling by scouring the membrane surface. The tangential shear force generated by the air bubbling can prevent the deposition of large particles on the membrane surface. However, the tangential shear has only a limited effect on preventing the deposition of small particles [9,10]. Therefore, fine particles are preferentially deposited on the membrane surface under high aeration intensity operation, resulting in a more compacted sludge cake, with high specific cake resistance which usually cause irreversible fouling. Sludge cakes formed under low aeration intensity are composed of large particles that are loosely attached and belong to reversible fouling [11]. The reversible fouling can be easily removed by physical cleaning, but irreversible fouling can only be removed by chemical cleaning. Under high aeration intensity, the shear force can break up the activated sludge flocs, causing the release of colloidal and soluble particles [12]. High aeration also reduces the cake layer formed on the membrane surface. With less protection from the cake layer, the membrane pore is more likely to be blocked by the soluble and colloidal particles [13]. Due to the high energy cost and the irreversible fouling caused by the high aeration intensity, previous works have reported the development of aeration strategies [7,14,15]. Intermittent aeration in which aeration is switched on and off alternatively has been previously studied, but the contradicting conclusions reported in previous studies make it necessary to evaluate its effect in new cases. To have optimized operation conditions, more insights into the cyclic aeration need to be addressed by investigating different air sparging strategies dealing with the air demand, as well as aeration intensity and frequency. Therefore, the specific objective of this study is to optimize the specific air demand (SAD) and to evaluate the influence of the aeration cycle duration on the fouling behaviour in a submerged MBR for the treatment of cosmetic wastewater.

2. Materials and methods

2.1. Experimental set-up for continuous runs

A 15-L MBR was inoculated with 8.6 g VSS L^{-1} (9.1 g MLSS L^{-1}) of flocculent sludge from a full-scale MBR treating cosmetic wastewater. The reactor was operated for 140 d at 20°C, neutral pH, and a constant SRT and HRT of 30 and 2.6 d, respectively. Aerobic conditions in the sludge bulk were ensured by supplying compressed air through porous diffusers, thereby reaching a dissolved oxygen concentration of 6.6 mg L^{-1}. The MBR was fitted with chlorinated polyethylene microfiltration flat sheet membranes (KUBOTA®) of 0.1 m^2 and a nominal pore size of 0.4 μm. Membranes were aerated at a constant SAD of 1.5 $m^3\ m^{-2}\ h^{-1}$. Permeate was discharged by a peristaltic pump (Gilson Minipuls 3), which allows operating at a constant permeate flux. Transmembrane pressure (TMP) was measured in the permeate line by a pressure transducer (XA-300, mPm) connected to a data acquisition system.

The effect of the SAD was analysed in the range between 0.0 and 1.5 $m^3\ m^{-2}\ h^{-1}$ and at a constant permeate flux of 12.0 L $m^{-2}\ h^{-1}$. The effect of the aeration cycle duration (1–30 min) under cyclic aeration operation was also studied, maintaining the SAD and aeration time constant at 0.5 $m^3\ m^{-2}\ h^{-1}$ and 50%, respectively.

The performance of the MBR was evaluated by analysing soluble and total chemical oxygen demand

(SCOD and TCOD), total and volatile suspended solids (TSS and VSS), total organic carbon (TOC) and total nitrogen (TN).

2.2. Wastewater composition

The MBR was fed with wastewater collected from a primary clarifier in a wastewater treatment plant of a personal care products factory. The characteristics of the wastewater are summarized in Table 1. Owing to the low N and P concentration in cosmetic wastewaters, ammonium sulphate and phosphoric acid were externally added in the feed to reach a COD:N:P ratio of 100:5:1.

2.3. Membrane fouling analysis

The foulant layers were removed sequentially via a three-step procedure, whereby an increasing amount of strength was applied to remove the fouling layers from the membrane surface. This protocol was aimed to obtain three fouling layers in a well-controlled environment for further characterization by analytical tools. Although this is not a standardized method, this protocol has been used to provide further insights into fouling layers' properties [16,17]. The membrane modules used were submerged in 1.2 L of milliQ water and were shaken manually for 2 min to remove the weakly adsorbed fraction. The intermediate fraction was removed by backwashing with 1.2 L of milliQ water at 4.0 L m^{-2} h^{-1} and the strongly bound fraction was desorbed by submerging the membrane in 1.2 L of 0.4%wt. sodium hypochlorite solution for 24 h with slow stirring. The resulting solutions upon each cleaning step were analysed in terms of COD, TOC, TN and TSS.

The permeability of the different fouling layers was determined in terms of hydraulic resistances using TMP and flux based on Darcy's Law. At the end of every fouling experiment, the total resistance (R_T) was determined. The fouling resistance (R_F) was then calculated by subtracting the intrinsic membrane resistance (R_M) from the total resistance (R_T). Upon removing each layer, a clean water test was applied working at a permeate flux of 10 L m^{-2} h^{-1} at 20°C for 20 min in a thermostatized bath to measure the resistance associated with each fouling layer removed. These experiments were carried out in triplicate. The hydraulic resistance associated with each fouling layer (R_{Rinsed}, $R_{Backwashed}$ and $R_{Desorbed}$, respectively) was calculated using Eqs. (1)–(3). Hence, the permeability resistance associated with each fouling layer, namely unstable (R_U), easily reversible (R_{ER}) and hardly reversible (R_{HR}), was calculated as follows:

Table 1
Characteristics of the pre-treated cosmetic wastewater

Parameter	Value
pH	7.1 ± 0.1
Conductivity (mS cm^{-1})	1.6 ± 0.8
TCOD (g L^{-1})	5.2 ± 0.7
SCOD (g L^{-1})	4.5 ± 0.9
TSS (g L^{-1})	1.3 ± 0.3
VSS (g L^{-1})	0.9 ± 0.2
Fats, oils and grease (g L^{-1})	0.2 ± 0.1

$$R_U = R_T - R_{Rinsed} \tag{1}$$

$$R_{ER} = R_{Rinsed} - R_{Backwashed} \tag{2}$$

$$R_{HR} = R_{Backwashed} - R_{Desorbed} \tag{3}$$

In all cases, $R_{Desorbed}$ was approximately equal to R_M indicating the presence of minimal irrecoverable fouling. Thus, R_T corresponds with the sum of fouling layers and membrane resistance:

$$R_T = R_M + R_U + R_{ER} + R_{HR} \tag{4}$$

The critical flux was determined using the flux-step method proposed by Le-Clech et al. [18]. A step height of 2.0 L m^{-2} h^{-1} and a holding time of 20 min were chosen and a VSS concentration of 9.3 g L^{-1} was kept constant for the sake of comparison. Membranes were cleaned before every critical flux test by soaking in 0.4%wt. sodium hypochlorite for 2 h to achieve a complete permeability recovery. These tests were performed in duplicate.

2.4. Shear experiment

The mixed liquor was stirred with a guarded triple-bladed propeller at a rotation speed of 900 rpm for 10 h. This corresponds to 800 s^{-1} of shear, considered as a standard for shear sensitivity determination [19]. The critical flux was determined to assess the effect of shear on sludge filterability using the aforementioned method purposed by Le-Clech et al. [18].

2.5. Chemical and physical analysis

Analysis of SCOD, TCOD, TSS and VSS was carried out according to the APHA Standard Methods [20], TOC was measured by a TOC analyser

(TOC-VCPN, Shimadzu) and TN was measured by an analyser (TNM-1, Shimadzu). Serial molecular weight fractionation of the supernatant was conducted using a 400 mL Amicon 8400 dead-end stirred cell (Millipore, USA) with 76 mm diameter and polyethersulphone UF membranes with nominal molecular weights of 10 kDa, 300 kDa, and pore sizes of 1 μm and 8 μm (Millipore, USA). Soluble microbial products' (SMPs) fractionations were conducted at a constant shear (through magnetic stirring) under an applied pressure of 2 bar and a filtrate/retentate ratio of 0.4, using laboratory grade nitrogen gas (>99%). Concentrations of proteins and carbohydrates were determined according to the adapted methods of Lowry et al. [21] and Dubois et al. [22], respectively. Bovine serum albumin and D-glucose were used as standards for the determination of proteins and carbohydrates, respectively [21,22]. Scanning electron microscopy images of the mixed liquor and fouled membranes were obtained with a Hitachi S-3000N apparatus. Previously, the samples were fixated and sputter coated with gold/palladium. The image processing and analysis procedure comprise the image pre-treatment, segmentation and debris elimination, whereas the image analysis programme is oriented to the aggregates determination [23]. The image processing and analysis programme for flocs and aggregates was developed in Matlab 7.6 language (The MathWorks, Inc.).

3. Results and discussion

3.1. Performance of the MBRs

COD and TOC removal efficiencies of about 96 and 93% were achieved at steady state. Average permeate quality was characterized by a TOC, COD and TN concentration of 44, 75 and 15 mg L^{-1}, respectively. Therefore, the resulting effluent from the MBR reached the highest quality reported treating cosmetic wastewater by means of a biological system. The application of an upflow anaerobic sludge blanket reactor led to a TSS removal efficiency of 85%, and a maximum COD removal efficiency of 75% was reached by a sequencing batch reactor [3,4].

3.2. Membranes performance

Critical flux experiment was carried out *in situ* after the start-up period. The most common criteria to define the value of critical flux is the flux above which the TMP increase rate exceeds 0.6 kPa h^{-1} [24]. Fig. 1 shows the permeate flux and fouling rate for each flux. Fouling did not occur at low fluxes, but TMP increased significantly for fluxes higher than 8.0 L m^{-2} h^{-1}.

Fig. 2 shows the TMP evolution monitored during the operation of the MBR at the critical permeate flux of 8.0 L m^{-2} h^{-1} for 10 d. This fouling profile follows the typical trend reported for constant flux operation of submerged porous membranes [24]. As can be seen in Fig. 2, TMP remained constant during 1.5 h and then increased gradually until 2.5 h at a fouling rate of 2.3 kPa h^{-1}. Similar trend of the TMP evolution for the initial filtration stage has been reported and related to the adsorptive condition in transient biofloc interactions and pore-blocking closure [25]. Then, an exponential increase in the TMP of 23.8 kPa h^{-1} was observed until the end of the test, reaching a TMP of 53 kPa. This TMP jump is believed to be the consequence of severe membrane fouling, which has been attributed to changes in the local flux and sudden changes of the biofilm or cake layer structure [25]. This phenomenon has been reported to be partially caused by the presence of proteins and carbohydrates in the mixed liquor. The average concentration of carbohydrates and proteins in the mixed liquor was around 50 and 45 mg L^{-1}, and 13 and 12 mg L^{-1} in the permeate. Assuming an empirical composition for carbohydrates ($C_6H_{10}O_5$) and proteins ($C_{16}H_{24}O_5N_4$) and an equivalent COD of 1.2 mg COD mg^{-1} polysaccharides and 1.5 mg COD mg^{-1} proteins [26,27], the MBR showed a relative COD fraction of SMPs in the permeate of 31%.

3.3. SMPs concentration and composition

Previous works have showed that SMPs have an impact on membrane fouling, which contributes significantly to the TMP increase in the MBR [24]. The

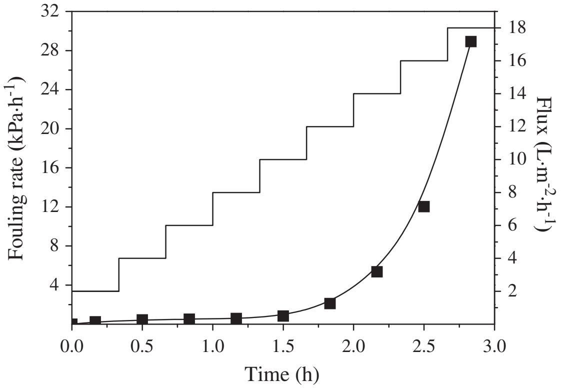

Fig. 1. Fouling rate (squares) observed at different permeate fluxes (line) during the flux-step method.

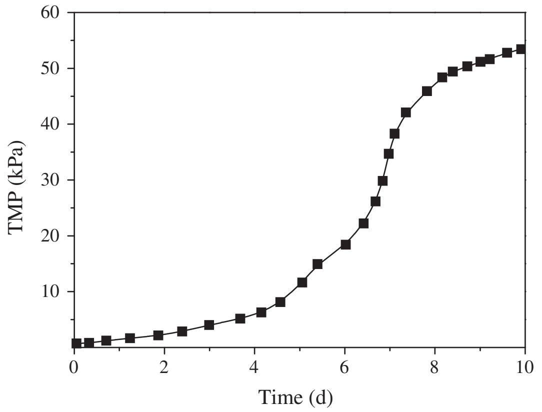

Fig. 2. Time course of the TMP during the long-term filtration experiment at a permeate flux of 8.0 L m^{-2} h^{-1}.

concentration of SMP determined in the supernatant of the MBR was 102 ± 21 mg L^{-1}. Fig. 3 shows the size distribution of SMPs retained by the membranes, which is calculated by the difference between the SMPs concentration in the mixed liquor and permeate for each molecular size upon molecular fractionation. Carbohydrates and proteins smaller than 1 μm counted 85 and 94%, respectively, among the SMPs retained by the membranes. The tangential shear force generated by the air bubbling can prevent the deposition of large particles on the membrane surface. However, the tangential shear has only a limited effect on preventing the particle deposition of small particles [9]. It is known that irremovable fouling is predominated by relatively high concentration of small bound SMP [28], which is of great importance for long-term and sustainable operation of MBRs. Owing to the small size of these biomolecules (below 1 μm), they can be deposited on the membranes more readily due to the permeate flow, but they have lower back transport velocity due to weak lift forces in comparison to large particles (e.g. colloids and flocs).

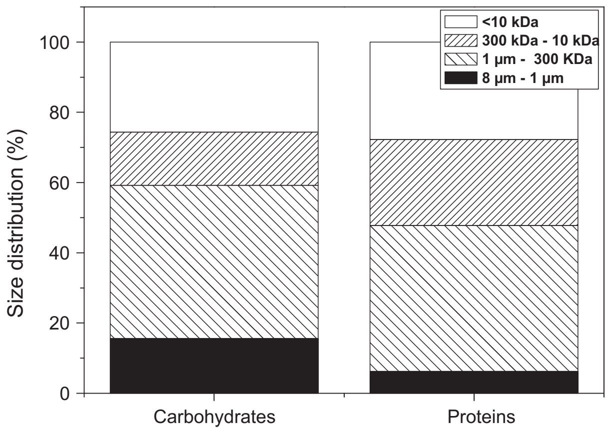

Fig. 3. Size distribution of carbohydrates and proteins retained on the membranes.

An aliquot of mixed liquor was submitted to an extra shear force to analyse the effect on the membrane permeability. The critical flux decreased by 25% upon the shear stress, which has been related to the release of SMPs and multivalent cations because of the cellular disruption since both of them are important for the structure of flocs. Flocs are exposed to shear forces from mixing and aeration operations in the MBR. This fact causes the detachment of foulants from flocs, which has been reported to be one of the causes of membrane fouling [29]. In addition, the mean floc size was reduced from 45 to 28 μm upon the shear stress experiment, which may also reduce the permeability of the mixed liquor. Nevertheless, it has been reported that cyclic aeration operation can preserve the microflocs (10–15 μm) within activated sludge flocs, which reduces their disruption and the release of submicron particles into the supernatant [14].

3.4. *Resistance of fouling layers*

Membranes were operated in the MBR for different operation times to analyse the relative resistance associated with different stages of fouling. Thus, membranes were operated, until reaching different TMP values, for the analysis of the start of fouling (2 kPa), the gel layer appearance (9 kPa) and the cake layer formation (20 kPa). Then, membranes were cleaned by following the three-step protocol and the clean water permeability test to obtain four resistance fractions: intrinsic, unstable, easily reversible and hardly reversible. As can be seen in Table 2, the relative filtration resistance distribution changed with the fouling extension. Intrinsic membrane resistance and the unstable layer were the major sources of filtration resistance when fouling started. The formation of the gel layer, at TMP values in the vicinity of 9 kPa, caused the appearance of an easily reversible layer which was the main filtration resistance at this stage. The unstable cake layer observed at TMP values above 20 kPa led to a sharp increase in the filtration resistance up to 1.9×10^{13} m^{-1}. Hereby, a large percentage of fouling is an unstable layer that can be easily removed by aeration under short-term operation. This cake layer can be transformed into irreversible fouling after a long-term filtration due to the cake consolidation [30]. Easily and hardly reversible layers cannot be removed by aeration, which also make necessary backwashing and

Table 2
Relative resistance (%) of each foulant fraction present in the membrane at different fouling stages

	Resistance			
Fouling stage	Intrinsic	Unstable	Easily reversible	Hardly reversible
Start fouling	43.5	56.5	nd	nd
Gel layer	22.1	29.1	46.1	2.7
Cake layer	9.3	71.1	17.1	2.5

Note: nd, not detected.

chemical cleaning to remove the aforementioned layers, especially in those membranes' configurations that make this necessary for their correct operation, such as hollow fibres or tubular membranes.

3.5. Influence of the SAD

Fig. 4 shows the relationship between the critical flux and the SAD. A high fouling rate was observed in the absence of air, which was partially mitigated by increasing the SAD. SAD values higher than 0.5 $m^3 m^{-2} h^{-1}$ did not lead to a significant improvement of the permeability and critical fluxes about 12.0 $L m^{-2} h^{-1}$ were obtained. Thereby, the operation of the MBR at a SAD of 0.5 $m^3 m^{-2} h^{-1}$ is recommended for a cost-effective operation of the membranes in the experimental set-up.

As can be seen in Fig. 5, the length of aeration cycles exerts an important effect over the TMP evolution. Whilst aeration cycles longer than 1 min led to a partial control of the TMP increase, cycle length of 1 min gave rise to an efficient control of TMP increase, which resulted in a negligible increase in the TMP after 2 h. Therefore, short aeration cycles allow operation under low aeration intensity (low SAD), which reduce the operation costs of the MBR. The cyclic aeration can also be effective for the removal of the reversible cake layer before its consolidation, preventing the formation of irreversible fouling. Fluctuations in the aeration intensity have been suggested to cause a positive effect on fouling control [31] but, in some cases, a strong fouling has been observed once the aeration was stopped [32]. Previous works have reported that fouling can be reasonably controlled using an aeration cycle duration of 10 min, which also enhance the nitrogen removal as a consequence of the aerobic–anoxic conditions created by the intermittent aeration [33]. In the present study, the use of short aeration cycles avoided the formation of a strong fouling, and the formation of the cake layer was only detected in the absence of air, which led to a filtration resistance of 2.2 × 10^{13} m^{-1}.

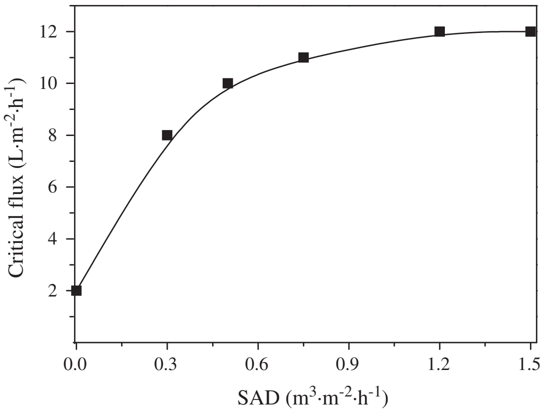

Fig. 4. Critical flux values obtained at different SADs.

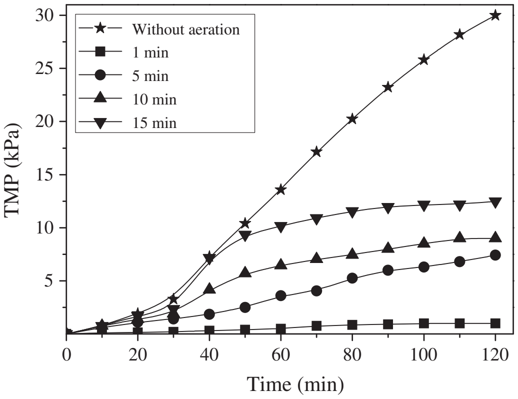

Fig. 5. Time course of TMP using different aeration cycle durations at an aeration intensity of 50%.

4. Conclusions

The extended filtration in MBR for the treatment of cosmetic wastewater under moderate flux is insufficient to prevent the fouling of membranes, whose formation is not avoided by intensifying the membrane scouring at high SAD values. The analysis of the fouling layers deposited on the membrane shows a significant effect of the hardly removable layers by air sparging, which causes almost 20% of the filtration resistance at the last stage of fouling. Cyclic aeration mode is an effective strategy, especially with short aeration cycles, to enhance the membranes' performance at the initial stage of fouling.

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