A comparative study of the influence of salt concentration on the performance of an osmotic membrane bioreactor and a sequencing batch reactor

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Abstract

An osmotic membrane bioreactor (OMBR) is a wastewater treatment technique that presents low energy requirements, low membrane fouling and high removal of nutrients and organic matter. However, reverse salt flux (RSF) is the main disadvantage because causes conductivity increase in the bioreactor. This study compares the performance of a sequencing batch reactor (SBR) and an OMBR in terms of chemical oxygen demand

(COD) removal, soluble microbial products (SMP) and extracellular polymeric substances (EPS) production. For that, the influent conductivity in the SBR was increased as increases conductivity in the osmotic membrane bioreactor. Comparing the results obtained at two mixed liquor suspended solids (MLSS) concentrations in terms of membrane fouling, a concentration of 5 $g \cdot L^{-1}$ of MLSS was chosen for the comparison with the SBR. The SBR achieved slightly higher COD removal efficiencies than OMBR is spite of the accumulation of cellular debris in the membrane bioreactor. The accumulation of SMP and EPS in the OMBR was also higher than in the SBR due to the cellular debris and organic matter accumulation. In both reactors the microbial activity measured in terms of standard oxygen uptake rate decreased due to the increase of the salt concentration in the bioreactor.

1. Introduction

Osmotic membrane bioreactor is a novel process that differs from MBRs in the membrane process used for the separation of the treated water from the mixed liquor [1]. Whereas ultrafiltration (UF) or microfiltration (MF) is used in MBRs, FO is used in OMBR. In this process, water permeates through the membrane from the mixed liquor to a stream with high osmotic pressure named draw solution (DS) [2,3] .

Among the advantages of the OMBR the following ones are found: only water is withdrawn from the system, the high retention membranes used are a barrier for the organic persistent pollutants, some of which are retained and degraded [4] and membrane fouling is lower than in MBRs. Nevertheless, on the one hand cellular debris accumulates in the mixed liquor and the RSF phenomenon leads to an increase of the conductivity in the mixed liquor. RSF phenomena in FO processes have been studied by different authors [5–7].

The use of DS with divalent ions would imply lower RSF. However, it is known that DS containing divalent ions yield lower water fluxes than DS containing monovalent ions at similar osmotic pressure. This is due to the lower diffusivity coefficients of the divalent ions in comparison with those of the monovalent ions [8]. In addition to it, industrial brines, which could be used as DS, are normally rich in sodium chloride (the usual preservative for hides, food…). Thus, inhibition of biomass due to the increase of the salt concentration has to be studied. Ansari et al. [9] observed inhibition in the anaerobic treatment of wastewater pre-concentrated by FO.

The increase of salt in conventional reactors has been widely studied. Different authors have summarized the problems of treating saline effluents in review papers [10–12]. Concentrations above 10 $g \cdot L^{-1}$ may cause cellular lysis. At the same time the separation of the mixed liquor will be detrimental for its separation both by membranes (loss of hydrophobicity) and by settling (the sludge volume index increases) [13,14]. Mannina et al. [15] concluded that it is crucial to maintain a constant salinity in the operation of a MBR. A salinity increase between 2 and 10 $g \cdot L^{-1}$ led to an increase in the EPS, enhancing membrane fouling [15].

However, the effect of salt and non-degraded organic pollutants build-up in the OMBRs has not been yet deeply studied. Luo et al. [16] compared the operation of an MBR and an OMBR. These authors used synthetic wastewater as feed and in the case of the OMBR, no diminution in the TOC removal efficiency was observed in spite of the salt accumulation and of the increment of the SMP concentration from 48.4 to 237 $mg \cdot L^{-1}$ in 15 days. Wang et al. [17] reported that the operation at a low sludge retention time is necessary for alleviating the salt build-up and, consequently to avoid a high flux reduction and high concentrations of SMP. On the contrary, Aftab et al. [18] did not found differences in the membrane fouling at three different organic load rates (OLR), though the highest tested value of the OLR enhanced EPS formation. The duration of each test was 14 days. The salinity increase in the SBR will also imply changes in the microbial community dynamics [19].

In this work the effect of the salinity on the process performance of an OMBR has been evaluated in terms of flux reduction, COD removal efficiency and SMP production. In parallel, a SBR has been operated at the same food to microorganisms (F/M) ratio, increasing the influent wastewater conductivity as conductivity in the OMBR increased by RSF. The behavior of both reactors has been compared. As reported by Holloway et al. [20], one of the future challenges to be reached in order to achieve that the technology may be commercially feasible is determining the effect of the salinity build-up on the mixed liquor. This is studied on this work.

2. Materials and methods

2.1. OMBR plant

Fig. 1 shows a scheme of the plant. The configuration of the membrane in the OMBR was external to the reactor. The plant consisted basically of a 1 L reactor and a CF042-FO module from "Sterlitech" with capacity for a 0.0042 m^2 membrane. The membrane used was CTA-NW from Hydration Technology Innovation (USA). Two peristaltic pumps (Pumpdrive 5006, Heidolph, Germany) pumped the feed solution (FS), i.e. the mixed liquor, and the DS, continuously. Permeate flux was measured by weighing the mass of the DS by a digital scale model PKP from KERN (Germany). Conductivity of draw and feed solutions were monitored with two “CDH-SD1” conductivity meters from Omega Engineering (USA). A magnetic stirrer agitated the mixed liquor and a compressor EHEIM 100 (Spain) provided air in order to maintain an oxygen concentration in the mixed liquor of around 2 mg·L^{-1}.

Membrane cleaning was carried out when membrane water flux decreased below 1 LMH or every 7 days instead. It consisted of a backflushing with deionized water as draw solution and a sodium chloride solution of 100 g·L^{-1} as feed solution. After each cleaning step, new DS was prepared to continue the OMBR operation.

2.2. Measurement of membrane water flux and reverse salt flux

Membrane characteristics were evaluated using water flux and RSF. The membrane water flux (J_w) was determined from the change in the permeated water volume from the feed to the draw solutions per unit membrane area per time (LMH). To carry out the characterization, deionized water was used as FS and different sodium chloride concentrations (25, 50, 100, 150 and 200 g·L^{-1}) were used as DS. Additionally, RSF (J_s, g·m^{-2}·h^{-1}) was calculated following Eq. 1.

$$J_s = \frac{V_t \cdot C_t - V_{t-1} \cdot C_{t-1}}{A \cdot \Delta t} \quad (1)$$

Where, V_t and C_t are the volume and the concentration of sodium chloride in the feed solution measured at time t. Both parameters were measured on pristine membranes before beginning the experiment to ensure that membranes were not defectives.

2.3. SBR plant

SBR was operated in parallel to OMBR. The reaction volume of the SBR was 6 L. Two peristaltic pumps (D-25 V from Dinko) carried out the filling and the drawing of the reactor at the beginning and at the end of every cycle, respectively. A compressor EHEIM 400 provided the needed air to maintain an oxygen concentration of 2 $mg \cdot L^{-1}$ in the reactor in the reaction phase. Mixing was carried out by a Heidolph mechanical stirrer and was connected during all the reaction phase. Timers were used to switch on and switch off the equipments according to the phase to be carried out.

2.4. Operation strategy

Three experiments were performed: two preliminary experiments with different MLSS concentration (test preliminary 1 at 14 $g \cdot L^{-1}$ and test preliminary 2 at 5 $g \cdot L^{-1}$) and a longer experiment with the chosen conditions with a mixed liquor suspended solids (MLSS) concentration of 5 $g \cdot L^{-1}$.

The SBR reactor was seeded with activated sludge taken from a municipal wastewater treatment plant (WWTP). Then, the OMBR was seeded with acclimated sludge of the SBR. The acclimation period to the simulated wastewater used in the tests was very short (3 days). The simulated wastewater consisted of bacteriological peptone, meat extract (both from Cultimed, Panreac) and tri-sodium phosphate 12-hydrate (Panreac) to keep the relation COD:N:P in 500:25:5. Chemicals were mixed and dissolved in tap water.

The food to microorganisms (F/M) ratio was calculated according to Eq. 2, where V_F is the daily wastewater volume fed to the reactor and COD_0 is the influent wastewater COD. The F/M ratio employed for the two preliminary experiments was 0.12 g COD·g $MLSS^{-1}·d^{-1}$ and for the longest experiment was 0.12 and 0.33 g COD·g $MLSS^{-1}·d^{-1}$ for the OMBR and SBR, respectively.

$$\frac{F}{M}(g\,COD \cdot g\,SS^{-1} \cdot d^{-1}) = \frac{Q \cdot COD_0}{MLSS \cdot V_F} \quad (2)$$

Due to the concentration of the mixed liquor in the OMBR and to the RSF phenomenon, there was salt accumulation in the OMBR. In order to compare both systems, sodium chloride (supplied by VWR) was added to the simulated wastewater feeding the SBR to the same extent as the salt concentration in the OMBR increased.

2.5. Analytical methods

To study the performance of both reactors, the effluent from the SBR and the supernatant from the OMBR were analyzed twice per week. OMBR samples were centrifuged at 10,000 rpm for 15 min, and the supernatant was then analyzed for COD, TN and TP. Regarding mixed liquor analysis, standard methods [21] were used for the measurement of MLSS and mixed liquor volatile suspended solids (MLVSS). In addition, pH and conductivity were also evaluated. SMPs were obtained from the mixed liquor after centrifugation and filtration of the centrate through a filter of 0.45 µm. On the other hand, EPS were extracted by means of a cation exchanger resin (Dowex Marathon C, Sigma Aldrich, Spain) according to the procedure described by Zuriaga et al. [22]. The total protein, carbohydrate and DNA (deoxyribonucleic acid) concentrations of the SMP and extracted EPS (eEPS) were determined using Bicinchoninic acid (BCA) assay test from Novagen, Antrone method [23] and Quant-it™ dsDNA HS (0,2–100 ng) from Invitrogen assay kits, respectively. The three procedures were described in [22].

2.6. Respirometry test

Respirometry test represents the oxygen consumption by the microorganism from the activated sludge. This experiment was carried out in a BM-Advance analyser from SURCIS (Spain). The SOUR (Specific Oxygen Uptake Rate), OUR (Oxygen Uptake Rate) and Y_H (heterotrophic biomass yield coefficient) parameters were determined as previously was described by Ferrer et al. [24] and Zuriaga et al. [25]. The equipment consists of a vessel of 2 L of capacity (which was filled with 1 L of activated sludge previously aerated during 24 h to achieve endogenous conditions in the biomass) a peristaltic pump, a Peltier cooler module and software to acquire the results. A static analysis (without oxygen addition) was carried out to calculate OUR/SOUR. In addition, to obtain Y_H, a dynamic experiment was performed by continuous stirring, aeration and activated sludge recirculation between both sides of the vessel. The dissolved oxygen was continuously measured and temperature was kept constant at 22ºC.

3. Results

3.1. FO membrane characterization

For each preliminary test and for the longest experiment a new CTA-NW membrane was used. Fig. 2 shows the J_w and J_s of the CTA-NW virgin membrane employed for the longest experiment. The results obtained for the other pristine membranes were practically equal. Similar J_w and J_s results were published by Yang et al. [26], who tested different FO membranes and reported that CTA-NW membrane had a flux around 4.8 LMH using a NaCl solution of 58.44 $g \cdot L^{-1}$ as DS. Other characteristics of this membrane can be found in the bibliography. For example, Wei et al. [27] reported that FO CTA-NW membranes had a thickness of 144 µm and a contact angle of 74º. By contrast, Nguyen et al. [28] published a contact angle of 53.5º, zeta potential of -25 mV (at pH 7) and a arithmetic average roughness of 0.821 nm.

3.2. Comparison between the tests with two different MLSS concentrations

3.1.1. Water flux and feed conductivity evolution

Fig. 3 illustrates the evolution of the FO membrane water flux and the conductivity of the mixed liquor over the testing period for each preliminary test (Fig. 3.a. for preliminary test 1 and Fig. 3.b. for preliminary test 2). The represented membrane water fluxes correspond with the mass data registered every 60 minutes. Each vertical line represents a cleaning step. It seems clear that the preliminary test 1 showed higher membrane fouling than the preliminary test 2, which was due to the fact that the MLSS concentration was much higher. The initial J_w value of the membrane used in the preliminary test 1 decreased sharply (from around 6 to 1 LMH in 20 hours). The initial flux could not be restored after the cleaning. By contrast, the membrane used in the preliminary test 2 was cleaned successfully recovering almost 100% of the initial flux after each cleaning. The flux decay in test 2 was less sharp than in tests 1 (around 1 LMH in the first 24 hours). In Fig. 3, it can be also observed that the membrane water flux decreased over the experimental period under relatively stable mixed liquor salt concentrations. This trend was also observed by Qiu et al. [29].

As commented in the introduction section, salinity build-up in the bioreactor is an inherent problem to the OMBR operation due to the presence of solutes from the influent wastewater and the reverse salt flux of NaCl from the DS. In this way, the conductivity in the OMBR increased for the preliminary test 2 sharper than for the preliminary test 1. This fact was probably due to higher resistance to the salt passage due to the high MLSS concentration in the preliminary test 1. The cake layer formation was an additional barrier for the RSF.

3.1.2. Performance of the reactor and mixed liquors characteristics

MLSS concentration slightly decreased during both preliminary tests. This trend is in concordance with others authors such as [30,31] and could be explained by the inhibition of biomass growth and activity due to salinity increase and the low operating F/M ratio. Fig. 4 shows the evolution of the MLVSS percentage and the COD removal efficiencies during both tests. It can be seen that MLVSS percentages were between 93.73% and 86.80% for test 1 and between 97.4% and 81.92% for test 2. Especially for test 2, the MLVSS percentage decreased over the experimental time. This could indicates accumulation of inorganic compounds inside the microbial flocs due to salinity build-up [32], since the salt increase in the reactor in the preliminary test 2 was the highest Regarding to COD removal percentages, a decrease of the reactor performance with the time was observed in both rectors. However, the decrease was sharper in the preliminary test 2. This could be due to the higher increase of the salinity in the bioreactor as commented above. The reverse sodium chloride flux was enhanced for the lowest MLSS concentration. In this way, the control of the salinity increase rate, in addition to the concentration increase itself, in the reactor seems to be a key factor for the reactor performance.

Table 1 shows the proteins, carbohydrate and DNA concentrations in SMP and eEPS for both experiments. It has been published [33,34] that higher salt levels could increase the endogenous respiration of microorganisms in the mixed liquor and thus increase the secretion of organic cellular substances, which would imply an enhancement of SMP and eEPS production. It explains the increasing values with the operation time in the OMBR.

As it can be expected, the biomass of the test 1 presented higher protein, carbohydrate and DNA concentrations in SMP than biomass of the test 2 since the MLSS concentration in test 1 was much higher than for test 2. However, the same trend was obtained for the eEPS in spite of being expressed per mass unit of VSS. It has to be commented that the high SMP and eEPS concentrations in the OMBR in test 1 also contribute to explain the more severe membrane fouling in comparison with the test 2 (as described in section 3.1.1).

The DNA concentrations in the reactor are related with the cellular lysis. As expected, the cellular lysis occurred at a higher extent in the test 1, since the MLSS concentration was considerable higher than in test 1.

3.2. Comparison of OMBR and SBR

3.2.1. Mixed liquor salinity and water flux of the OMBR

Fig. 5 illustrates the water flux and the feed conductivity evolution for the long OMBR experiment. The water flux gradually declined from 6.5 LMH to 1 LMH, which is explained both by membrane fouling and by diminution of the driving force, i.e. the salt concentration difference between draw and feed solutions. The RSF was responsible for the sharp salinity increment in the bioreactor. In this way, within the first 200 hours (8 days) of operation, conductivity in the bioreactor gradually increased from 10 to 18 $mS\cdot cm^{-1}$. This rapid salt increase was due to a combination of salt rejection by the FO membrane and the above explained RSF [35]. However, in the following days the feed solution conductivity became relatively stable at approximately 20 $mS\cdot cm^{-1}$ due to the daily sampling from the bioreactor for analysis (sludge withdrawals) and the increasing resistance of the membrane to salt passage due to the membrane fouling.

3.2.2. Mixed liquor characteristics of both reactors (OMBR and SBR)

Fig. 6 shows the MLVSS percentage and the COD removal efficiencies for both reactors. It has to be highlighted that the organic matter removal efficiency in OMBR is actually calculated considering the COD that passes through the membrane to the DS side. However, this percentage was maintained in practically 100%, since the FO membrane rejected the organic matter in the reactor. This is the reason why the comparison is carried out on the basis of the soluble COD in the reactors and it cannot be stated that the best process is the process with the least COD in the reactor, i.e. with the highest COD removal efficiency, since there will be a certain accumulation of non degradable COD in the OMBR.

The evolution of the MLVSS percentage was similar in both reactors, decreasing during the experiment. The decrease was sharper in the OMBR, which was due to the lower operation F/M ratio. In Fig. 6 it is also observed that slightly low soluble COD concentrations were achieved in the SBR compared with those measured in the OMBR. As stated above, this can be explained by the accumulation of non-biodegradable organic matter in the biological reactor due to the FO membrane rejection [34,36].

Fig. 7 shows the protein, carbohydrate and DNA concentrations in SMP and the protein and carbohydrate concentration in eEPS from both reactors. The highest SMP and eEPS concentrations in all the samples were measured in the OMBR. This fact could be attributed to the higher stress of the biomass in OMBR, caused both by the accumulation in the reactor of non-biodregradable organic matter [37], including SMP, and by the mechanical stress produced by the biomass pumping to the membrane module [38], though the shear stress is considerably lower in an OMBR than in a MBR. It has to be commented that the salt concentration was the same in both reactors as explained in materials and method section; therefore this parameter had no influence on the SMP and eEPS differences in both reactors. However, the salt concentration increase in both reactors influenced the evolution of the SMP and eEPS concentrations with the time as it was explained in section 3.1.2. As it can be observed in Fig. 7, for

OMBR reactor SMP and eEPS protein and carbohydrate concentrations increased considerably with the operation time. The same trend was published by Whang et al. [39] who obtained a SMP concentration in polysaccharide and proteins the day 15 of operation time three times higher than the initial value and a eEPS concentration (also in terms of polysaccharide and protein concentrations) twice higher than the initial value. By contrast, the increase of the total EPS was very slight. In fact, the increase of the protein concentration (both in SMP and eEPS) in sample 2 can be observed; meanwhile the carbohydrate concentration was slightly lower in sample 2. This is explained by the lack of accumulation of the EPS in the SBR unlike OMBR. This implies a worse effluent quality in the SBR if it is compared with the water passing to the DS in the OMBR, since the rejection of organic matter of the FO membrane is practically 100%.

The analysis of DNA was studied to observe whether cell lyses occurred [40]. In this case, for SBR, DNA concentrations did not vary significantly with the operation time. However, for OMBR, DNA concentration decreased with the operation time, which indicates that biomass was adapted progressively to the operating conditions of the OMBR.

3.2.3. Respirometric characterization of the mixed liquors

Table 2 shows SOUR and Y_H parameters obtained from the respirometric analysis carried out with the initial sludge (from the WWTP at 1.35 $mS \cdot cm^{-1}$) and at the end of the long experiment with each reactor. Mixed liquor from SBR reactor had higher SOUR values than OMBR, what implies that biomass from SBR had higher microbial activity than OMBR. Other reason could be related with the different HRT of both reactors (the HRT were 1 d and 3.33 d for SBR and OMBR, respectively). In this way, Wang et al. [41] published that SOUR values increased when hydraulic retention time decreases. As it can be observed in Table 2, SBR had higher SOUR value (3.77 $mg \cdot g^{-1} \cdot h^{-1}$). However, according to other authors [13,33,42], the high salinity presented in both bioreactors could lead to the inhibition on the biological growth and activity and decrease the SOUR parameter. For this reason, initial sludge SOUR was the highest one.

Concerning Y_H parameter, this coefficient was related with the biomass growth [24]. In addition, high salinity implies high amounts of energy consumed by the microorganism in order to maintain their activity and integrity, what leads to a lower energy consumption for growth [37]. Although there were not significant differences between both reactors, the mixed liquor of the SBR achieved a slightly higher value than the mixed liquor of the OMBR.

Conclusions

Results reported here show that MLSS concentration clearly affects to the FO membrane fouling. Test preliminary 1 (MLSS concentration of 14 $g \cdot L^{-1}$) had a considerable higher membrane fouling than test preliminary 2 (MLSS concentration of 5 $g \cdot L^{-1}$). Regarding COD removal efficiencies (considering the dissolved COD in the reactor as the measured for the calculation of the eliminated COD), the less reverse salt flux in the test with 14 $g \cdot L^{-1}$ led to a slightly better removal efficiencies.

With respect to the comparison between SBR and OMBR reactors, higher SMP and eEPS concentrations were found in the OMBR due to the higher stress of the biomass and the cellular debris and organic matter accumulation. This also influenced on the microbial activity measured by means of the SOUR, since the SOUR of biomass in the OMBR was the lowest.

However, taking into account that only water permeates to the draw solution (the actual efficiency is almost 100%), the soluble COD in the reactor was maintained in concentrations that implied COD removal efficiencies (considering feed and soluble in the rector COD concentrations) near 80%. In this way, controlling appropriate the sludge withdrawal and the operating conditions, OMBR could be a treatment that could be considered as alternative for some types of wastewaters (low flow rate, trend to originate bulking in activated sludge, slowly biodegradable organic matter…). Anyway, further research on OMBR has to be carried out in order to assess the process feasibility for potential wastewater treatment applications.

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