Management of table olive processing wastewater byan osmotic membrane bioreactor process

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Abstract

The management of fermentation brines from the table olive processing is very complex due to its characteristics: high salinity and high organic matter concentration including phenolic compounds, which behave as slow degradable compounds when a biological process is performed. In this work, the management of these effluents by an osmotic membrane bioreactor has been assessed. This technique combines a biological reactor with forward osmosis membranes. For the study, a laboratory plant consisting of 1 L reactor and a forward osmosis module equipped with a membrane of 42 cm^2 of active surface has been used. Fermentation brine from table olive processing was fed to the system both as draw solution to set out the driving force for the membrane process and as a part of the feed to the reactor, mixing it with municipal wastewater. The

experiments were carried out at a constant feed to microorganism ratio of 0.4 g COD·g $SS^{-1}·d^{-1}$. Results indicated that the hypersaline effluent was able to produce the needed driving force by the process. Permeate fluxes ranged between 1 and 1.5 $L·m^{-2}·h^{-1}$ after the flux decay of the first operation days. Concerning the biological reaction, it has to be highlighted that phenols were eliminated after 24 days. Until that day, the biological process was jeopardized due to the quick increase of the conductivity in the reactor (ranging between 30 and 35 $mS·cm^{-1}$), which was caused not only by the salinity of the influent but also by the reverse salt flux phenomenon. Soluble microbial products and extracted extracellular polymeric substances also increased in the reactor during the start-up.

1. Introduction

The treatment of hypersaline wastewaters containing organic matter (such as those from food-processing, leather or petroleum industries) [1] is very complex, requiring further researches and new economic and environmental-friendly solutions. One of these hypersaline effluents is produced inthetable olive processing, which is one of the major industrial activities in Mediterranean countries such as Spain, Italy or Greece [1]. According to the data published [2], the table olive production worldwide during the 2017/2018 season was more than 2900 tonnes. Table olive processing includes several steps: rinsing with fresh water, debittering in an alkaline solution, a second rinsing with water and a final fermentation in a NaClbrine [3]. Around 6 $L·kg^{-1}$ of table olive processing wastewaters are generated in the overall process [4]. Although wastewater generated in the fermentation step only involves the 20% of the total volume generated, the wastewater from fermentation of table olive processing (FTOP) produces the 85% of the total wastewater pollution [5]. The main characteristics of the FTOP are high salinity (conductivity around 80 $mS \cdot cm^{-1}$) and high organic matter concentration due to oil and greases, phenolic compounds and volatile organic acids [6]. However, these characteristics clearly depend on the olive maturity, cultivar type, season and fermentation time [7,8].

Although it could be thought that these wastewaters can be treated effectively by means of several techniques such as evaporation or reverse osmosis, the high content in organic matter make the use of these technologies especially complicated. In this context, the possibility of offering environmental-friendly solutions for this wastewater acquires relevant importance.

In the recent years, the appearance of novel forward osmosis (FO) membranes has increased the potential applications of this technique. Specifically, osmotic membrane bioreactors (OMBR) offer the possibility of a simultaneous degradation of the organic matter in the biological reactor and a dilution of the hypersaline wastewater as a consequence of its use as draw solution. An OMBR is an emerging technology combining FO membranes with a biological reactor [9]. A typical OMBR consists of a FO membrane module, a biological reactor and a draw solution (DS) reservoir. The DS creates a high osmotic pressure difference, which acts as a driving force, between the both membrane sides. As a consequence, the treated water is transported from the biological reactor through the FO membrane to the DS (what implies the dilution of the DS) [10,11]. Compared to conventional membrane bioreactors (MBR), OMBR presents lower membrane fouling degree, higher fouling reversibility and significant lower energy consumption since is not hydraulically driven membrane technology [12]. In addition, OMBRs offer higher water quality due to the high rejection capacity of FO membrane to pathogens [13], ions [14], and organic compounds [15]. One potential application of OMBRs is for eliminating slowly degradable compounds from industrial wastewaters. In this way, Praveen et al., [16] studied the biodegradation of phenol compounds from saline wastewater using an OMBR. The results indicated that the treatment was effective and the membrane fouling was reversible.

However, the main shortcoming in the OMBR operation is the salinity build-up in the biological reactor, which is associated with the reverse salt flux and with the high rejection capacity of the FO membranes [17,18]. Salt accumulation in the bioreactor has negative effects on the overall process efficiency since the osmotic driving force decreases and salinity build-up can also affect the physical and biochemical characteristics of the microorganisms [19]. As a consequence, the membrane water flux is reduced and the activated sludge could increase the production of soluble microbial products and extracellular polymeric substances (EPS), leading to their concentration in the bioreactor, which implies higher membrane fouling [19].

In this research, a laboratory OMBR was operated to study the suitability of the process both for the treatment of FTOP as a part of the bioreactor feed and for its dilution as a DS for the FO process. The process was studied in terms of treated water quality (paying special attention to elimination of phenols), membrane water flux, salinity accumulation in the reactor, mixed liquor characteristics and membrane fouling. Until now, this alternative for the FTOP treatment has not been reported in the bibliography.

2. Materials and methods

2.1. Experimental system

The process flow diagram of the OMBR used for the experiments is shown in Fig. 1. The FO flat sheet membrane module CF042-FO, whichwas supplied by Sterlitech (USA), was coupled with a laboratory reactor. This module has the capacity for testing one membrane with an effective area of 42 cm^2. The FO commercial membrane employed was CTA-NW membrane from Hydration Technology Innovations (HTI, USA). Further details of the laboratory OMBR can be found in earlier works[20].

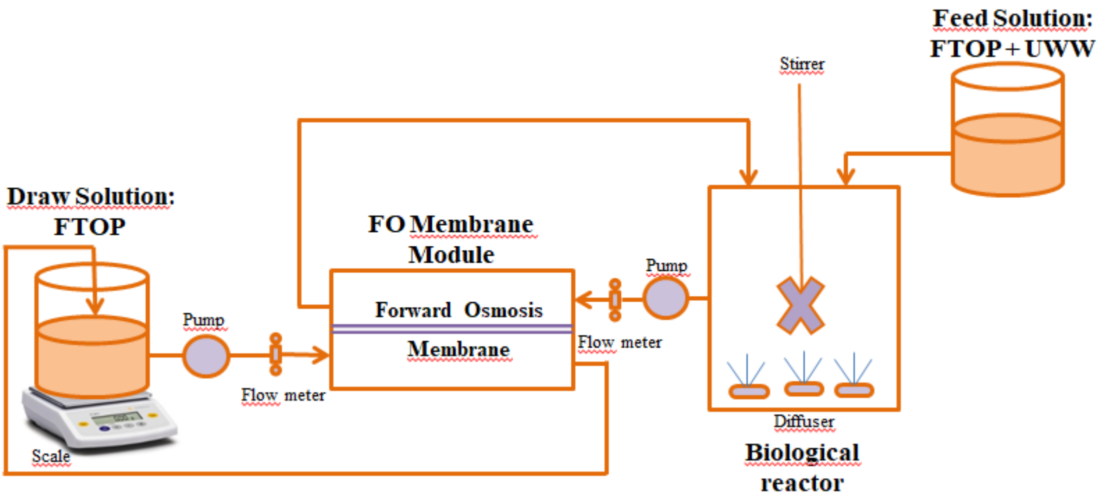


Figure 1: Process flow diagram of the experimental pilot plant.

2.2. Wastewaters characterization

The influent to the OMBR,consisted of FTOP and urban wastewater mixed in different proportions according to the strategy detailed in Section 2.3. FTOP was provided by a company located in Valencia (Spain). Each sample of 5-10 liters was filtered in a 60 µm sieve in order to reduce the suspended solids concentration and was stored at 4ºC before its use. FTOP was characterized in terms of pH, conductivity, chloride (Cl^-), sodium

(Na^+), soluble total nitrogen (TN), soluble total phosphorous (TP), soluble chemical oxygen demand (COD) and total phenolic compounds. pH and conductivity were measured using a pH-Meter GLP 21^+ and EC-Meter GLP 31^+ (Crison, Spain), respectively. $Na^+$was analyzed by means of ion selective digital probe ISENa 381 (Hach, USA). Cl^-, TN, TP and COD were measured using kits from Merck. Finally, the total phenolic compounds concentration wasmeasured following the Folin-Ciocalteu method [21]. In this way, the total phenolic content (expressed as tyrosol concentration, mg $TY\cdot L^{-1}$) was measured spectrophotometrically. Sodium carbonate (20% w/v) from Panreac, Folin&Ciocalteu'sreagent and Tyrosol analytical standards, from Sigma Aldrich, were used in the analytical procedure.

The urban wastewater was characterized measuring soluble COD, TN and TP using kits also from Panreac (Spain). Table 1 and 2 show the characteristics of each wastewater used.As it can be observed in Table 1, FTOP has high conductivity values due to the NaCl used in the fermentation brine process [22]. However, due to its wide variability, sodium chloride addition was required (until reaching a conductivity value of 80 $mS\cdot cm^{-1}$) to the first and third FTOP samples in order not to reduce the osmotic pressure difference in the process.

Table 1: Samples of FTOP used for the experiments.

	Experiment day		
	0-9	10-48	49-62
Conductivity ($mS \cdot cm^{-1}$)	34.4	82.1	16.46
pH	5.52	3.74	4.82
Cl ($mg \cdot L^{-1}$)	13,200	44,700	5,700
COD ($mg \cdot L^{-1}$)	8,050	25,850	4,500
TP ($mg \cdot L^{-1}$)	22.8	70	24.5
TN ($mg \cdot L^{-1}$)	138	220	120
Na ($mg \cdot L^{-1}$)	11,000	35,000	3,975

Table 2: Samples of urban wastewater used for the experiments.

	Experiment day			
	0-6	7-27	28-44	45-62
COD ($mg \cdot L^{-1}$)	167	113	147	184
TP ($mg \cdot L^{-1}$)	2.1	1.8	4	7.1
TN ($mg \cdot L^{-1}$)	26	7	48	41
pH	7.12	6.85	6.70	7.05

On the other hand, the low COD values of the urban wastewater samples were typical for the wastewater treatment plantwhere the samples were taken. It has also to be commented that the ratio C/N/P was checked in the mixed wastewater feeding the OMBR to avoid a lack of nutrients in the reactor. Thus, dipotassium phosphate (from Panreac, Spain) and urea (from Panreac, Spain) were employed to adjust the phosphorous and nitrogen concentrations to ensure a COD/N/P relationship of 100/5/1 $mg \cdot L^{-1}$, since the FTOP had low phosphorous and nitrogen concentrations (as it is shown in Table 1).

2.3. Experimental protocol

The OMBR was inoculatedwith activated sludge from a municipal wastewater treatment plant (located in Valencia, Spain). The food to microorganisms ratio (F/M) was fixed to 0.4 g COD·g SS^{-1}·d^{-1}. To this purpose, FTOP and urban wastewater were mixed in the appropriate proportions to achieve a COD around 2,600 mg·L^{-1} since the flow treated per day was fixed by the membrane water flux at 0.3 L·d^{-1}. The total duration of the OMBR operation was 60 days. The initial pH and conductivity values were 7.13 and 1.65 mS·cm^{-1}, respectively.

The reactor was fed 8 times per day, by adding the same wastewater volume as the permeated water volume through the FO membrane.

The osmotic backwashing was carried outtwice per week to clean the FO membrane. In this process, deionized water was used as DS and a NaCl solution with a concentration of 70 g·L^{-1} as FS. Both solutions were recirculated through the system during 1.5 hours. In addition, every 20 days, the membrane backwashing was followed by a chemical cleaning step. This step was carried out by using a solution of 1% w/w of Alconox (from Alconox, United States) and 0.8% w/w of EDTA (from Alfa Aeser, United States) recirculated during 2 hours. It is important to note that after each membrane cleaning step, a fresh DS was introduced for the OMBR operation.

2.4. Analytical methods

2.4.1. Membrane characterization

On one hand, the pristine FO membrane was characterized measuring the membrane water flux and RSF following the methodology described in a previous study [23].

2.4.2. Mixed liquor characterization

To study the concentration of the biomass, MLSS and mixed liquor volatile suspended solids (MLVSS) concentrations in the biological reactor were measured twice a week following the procedure described in [24]. In addition, pH was measured also twice a week.

To analyze the filterability of sludge, capillary suction time (CST) was measured once a week using the equipment 304M from Triton Electronics Ltd (United Kingdom). The results were normalized against MLSS concentrations and the result was expressed in units of $s \cdot L \cdot gMLSS^{-1}$.

Finally, chemical characteristics of the sludge in terms of Extracellular Polymeric Substances (EPS) and Soluble Microbial Products (SMP) were studied. The extracted EPS were obtained by means of a cation exchanger resin (Dowex Marathon C, Sigma Aldrich, Spain) following the procedure described in [25]. eEPS and SMP in the biological reactor were studied in terms of proteins and carbohydrates. Proteins concentrations were analyzed using Bicinchonic acid (BCA) assay test from Novagen. Carbohydrates were analyzed following the Antrone method [26]. Furthermore, DNA was quantified using Quant-it^{TM} dsDNA HS (0.2-100 ng) kit from Invitrogen (Spain).

2.4.3. Reactor performance

In order to study the OMBR performance in terms of organic matter, phenolic compounds and nutrients removal, the soluble fraction of the biological reactor was analyzed to check the quality of the treated water.

3. Results

3.1. Properties of the CTA NW FO membrane

Membrane water fluxes (J_w) and reverse salt flux (J_s) were measured for different concentrations of NaCl. Thus, the normalized salt reverse flux (J_s/J_w, $g\cdot L^{-1}$) was of 0.58 $g\cdot L^{-1}$ as mean value of the data obtained for NaCl concentrations of 25, 100 and 200 $g\cdot L^{-1}$.These results are in agreement with previous findings [27,28]. For instance, Siddique et al.[29] published a normalized salt reverse flux value of 0.75 using NaCl with a concentration of 14 $g\cdot L^{-1}$ as DS. Moreover, according to Luo et al. [30] who also tested this FO membrane, the contact angle and zeta potential of this membrane were 60.4º and -4.5 mV, respectively.

3.2. Water flux and salinity build-up in the bioreactor

Salinity build-up in the bioreactor is a critical issue during the OMBR operation mainly due to the high salt rejection of the FO membrane and the reverse salt flux from the DS [31]. As it can be observed in Fig. 2, salinity increased dramatically in the bioreactor from 3.66 to 29.8 $mS\cdot cm^{-1}$ within the first 15 days of OMBR operation. The severe increase can be attributed mostly to the high salinity of the influent in this period (reactor feed was the mixture of FTOP and urban wastewater, as describe in materials and methods section). In addition, the higher membrane water flux in that period indicated the lower membrane fouling degree, what implied higher reverse solute flux. By contrast, from the 15th day of operation, the conductivity of the influent was lower than the reactor conductivity and the membrane water flux was also lower due to the reduction of the osmotic pressure difference. Furthermore, the sludge withdrawals to maintain the MLSS concentration also contributed to hinder the end of the salinity increase in the reactor. This confirms thatin this type of processes a maximum salinity value in the reactor is being established. For instance, Raghavan et al. [32] treated synthetic wastewater with an OMBR and observed a final value of the biological reactor conductivity of 27.9 mS·cm^{-1}. Nevertheless, [33] Luo et al. also studied the treatment of synthetic wastewater with an OMBR and reached a final mixed liquor conductivity value of 11.5 mS·cm^{-1}.

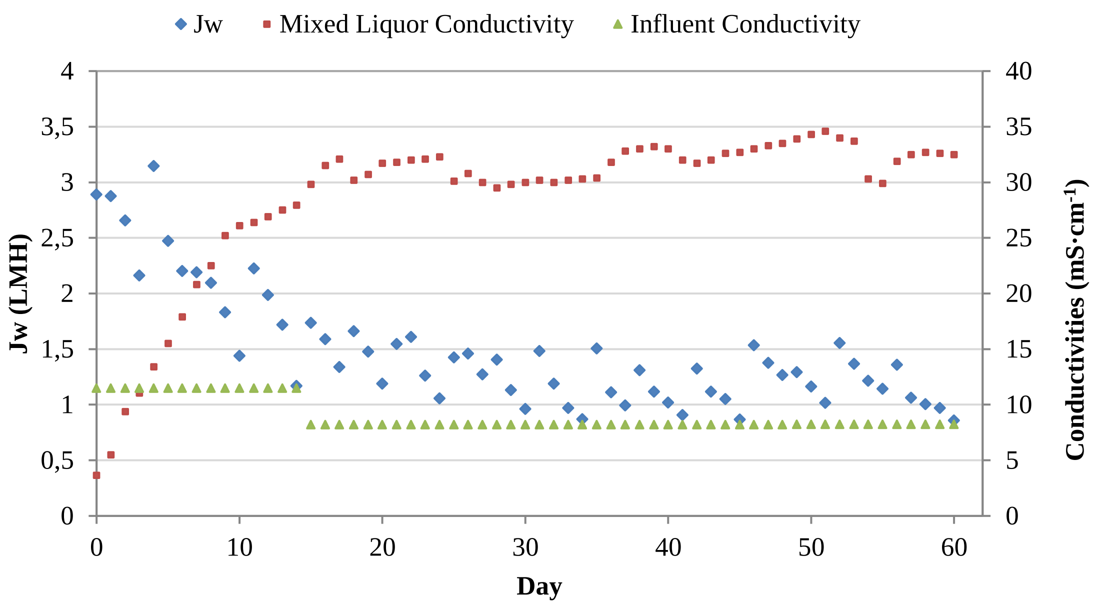


Figure 2: Water flux and mixed liquor conductivity evolution.

FO membrane water flux decreased continuously during the OMBR operation. This trend is due to membrane fouling and salinity build-up in the bioreactor [34,35] and it is in agreement with the results reported by Luo et al. [33]. Membrane water flux evolution can be divided into two stages: the first one is until the10thday of OMBR operation and the second one related to the last part of the experiment. In the first stage, the membrane water flux significantly decreased from 3.1 LMH to 1.4 LMH. This fact could be attributed to the fast decrease in the osmotic pressure difference between both sides of the membrane and, for a less extent, to the deposition of foulants on the membrane surface [36]. In the second stage (from the day 10th of operation time), the fouling layer was already formed and a low membrane water flux, being almost constant, was observed. These results are in concordance with previous studies performed with laboratory OMBRs [23,32,37].

3.3. Pollutants removal performance

3.3.1. Organic matter removal

The high COD value of the FTOP made difficult to check that no organic matter had permeated through the FO membrane from the FS to the DS. Anyway, it was calculated the theoretical COD of the diluted DS considering the volume increment in the DS reservoir; this result was then compared with the analysed COD. Results indicated that COD removal efficiencies in the OMBR were around 100%. Other authors like Raghavan et al.[32] and Morrow et al.[38]also published high total organic carbon removal efficiencies of 98% and 98.4%, respectively, operating an OMBR.

Concerning the soluble COD in the reactor, it was increasing until it reached an approximately constant value of 900 mg·L^{-1}at 35th day. This means that after an adaption period, no accumulation of COD occurred in the reactor. In other words, the increase of the organic matter biodegradation counteracted the concentration effect of the FO membrane.

3.3.2. Removal of phenolic compounds

Fig. 3 illustrates the OMBR and influent concentrations of total phenolic compounds during the total duration of the experiment. Phenolic compounds increased rapidly during the first 20 days of operation independently of the influent concentration, being the maximum value detected 149.41 mg·L^{-1}at the day 24th of operation.

The variation of the total phenols concentration with time can be divided into 4 periods. The first period coincides with the feeding of the first sample to the reactor. Due to its high total phenols concentration the accumulation of these compounds in the reactor was very quick. The second period starts with the feeding of the second sample, whose concentration of total phenols was considerably lower than the first sample, up to the 24th day. Until that day, the total phenols concentration in the reactor increased gradually at a rate lower than in the first period until reaching the concentration of 149.41 mg·L^{-1}. From 24th to 45th day (third period), total phenolic compounds concentration was approximately constant (around 140 mg/L), which means that biomass has been adapted to the salinity and the high phenols concentration and the biodegradation of these compounds began to occur. Finally, in the fourth period (last 10 days of operation) the total phenols degradation rate increased in the reactor, and the concentration decreased until 109.8 mg·L^{-1}.

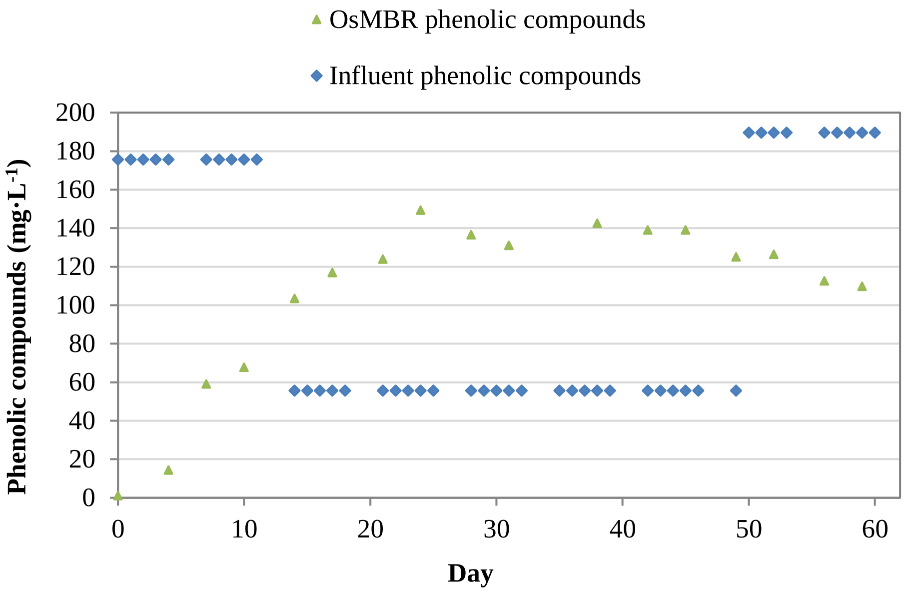


Figure 3: OMBR and influent phenolic compounds evolution.

These results suggest that phenolic removal was performed through biological degradation [39]. Ferrer-Polonio et al., [40] published a total phenolic compounds biodegradation in a sequencing batch reactor (SBR) of around 76%, what implies that phenolic compounds biodegradation is really viable. However, these authors had to operate the reactor at very high hydraulic retention time (40 days).

3.4. Nutrients concentration in the reactor

Total phosphorous was monitored by means of measuring the TP content in the supernatant and the TP concentration in the influent. As it can be observed in Fig. 4, the TP removal percentage varied between 69.23% and 93.33%, reaching a final value of 82.92%. TP removal percentage slightly decreased until the day 35th of operation. Since the TP removal was due to assimilation by microorganisms for growth purposes, this trend was probably due to a mild deterioration of biomass.

TN concentrations were clearly affected by the nitrogen concentration in the wastewater used as FS. The evolution of the different nitrogen forms during the OMBR operation can be observed in Fig. 5.The TN content in the supernatant was maintained at around 40 mg·L^{-1} most of the operating days, excepting from day 28th until 35th during which an increase in the NH_4-N concentration was observed. After that period, the NH_4-Nconsumption raised again, which corresponded to the increase of the COD biological degradation as reported in Section 3.3.1. The average removal percentage of total nitrogen was around 70% during the whole duration of the OMBR experiment. However, this nitrogen elimination was only due to biomass assimilation since the low TN concentrations in the influent required the addition of an external N source.

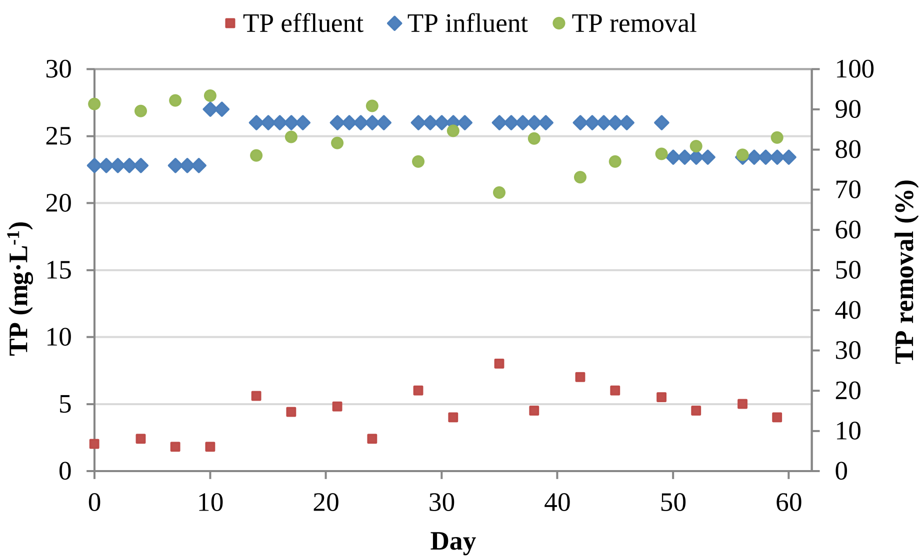


Figure 4: Total phosphorous removal.

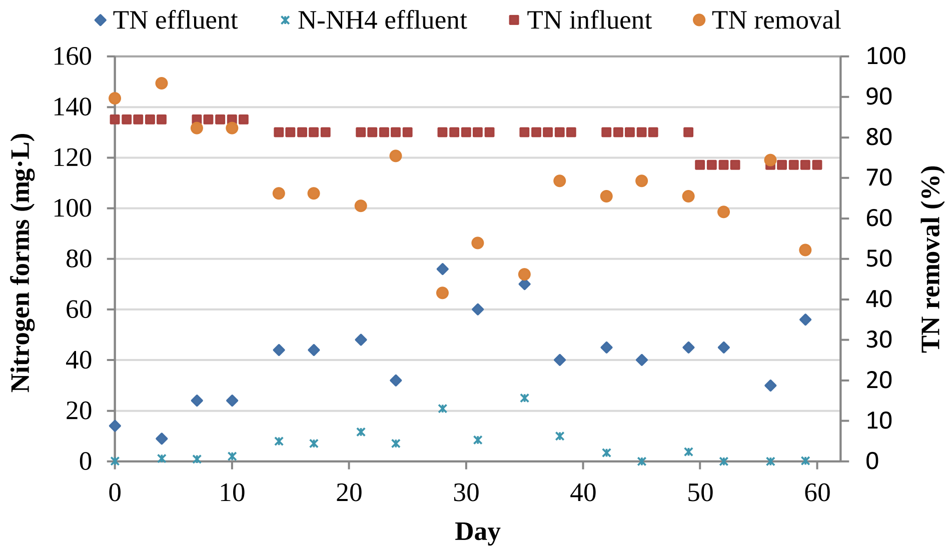


Figure 5: Nitrogen forms in the OMBR and TN removal.

3.5. Mixed liquor characteristics

3.5.1. Mixed liquor pH, MLVSS and normalized CST

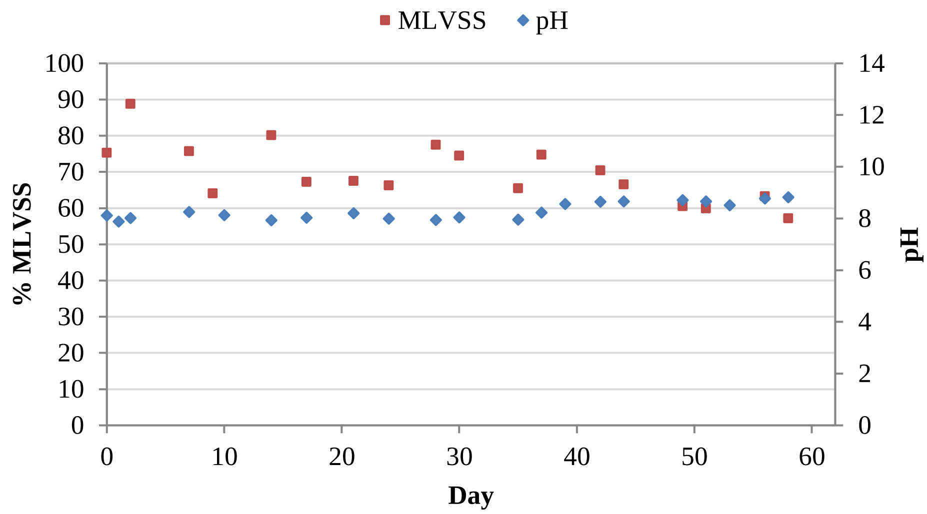
Fig. 6 illustrated the mixed liquor pH, the percentage of MLVSS and the CST in the mixed liquor during the OMBR experiment. Reverse draw solute flux can alter the mixed liquor pH during the OMBR operation. Although both DS and FS used had a pH lower than 7 (Tables 1 and 2), the pH slightly increased with the operating time of the experiment reaching a final value of 8.8 (Fig. 6a).The reason of this pH increase can be ascribed to the volatile acids (mainly lactic, malic, acetic and formic) contained in the FTOP wastewater [41], which were oxidized to CO_2 and H_2O by the biomass, thus producing the pH increase [5].

Microorganisms growth can be altered by the salinity increase in the biological reactor thus affecting the MLSS and MLVSS concentration [29]. As it can be observed in Fig.

6.a, the percentage of MLVSS decreased with time due to the increased osmotic stress on the feed side.

Sludge filterability in terms of CST was measured during the OMBR experiment (Fig. 6.b). As capillary suction time increases, the filterability of the sludge decreases [29]. Deteriorated sludge filterability can be observed around the day 46th of operation, what coincided with the maximum mixed liquor conductivity. At the end of the experiment, CST values reached lower values probably due to the diminution of the volatile solids percentage as explained above. These results are in concordance with previous studies [42], which refer normalized CST values between 20.5 and 39.1 s·L·gTSS^{-1} in the sludge of an OMBR for heavy metals removal[42].

a)



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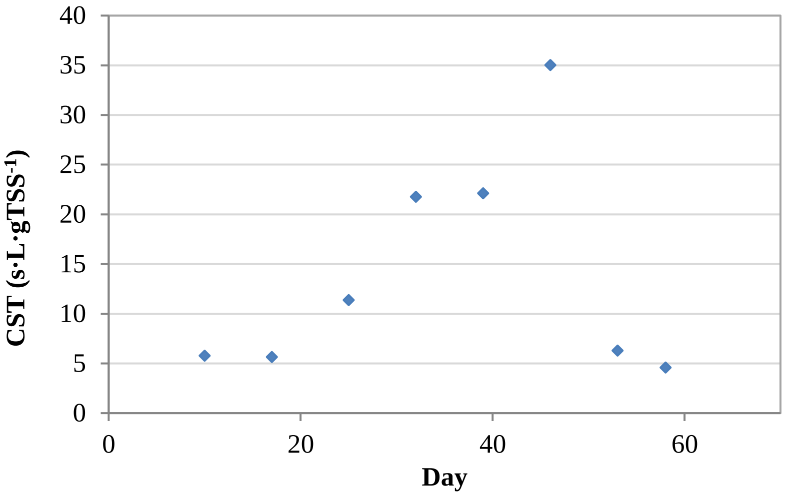


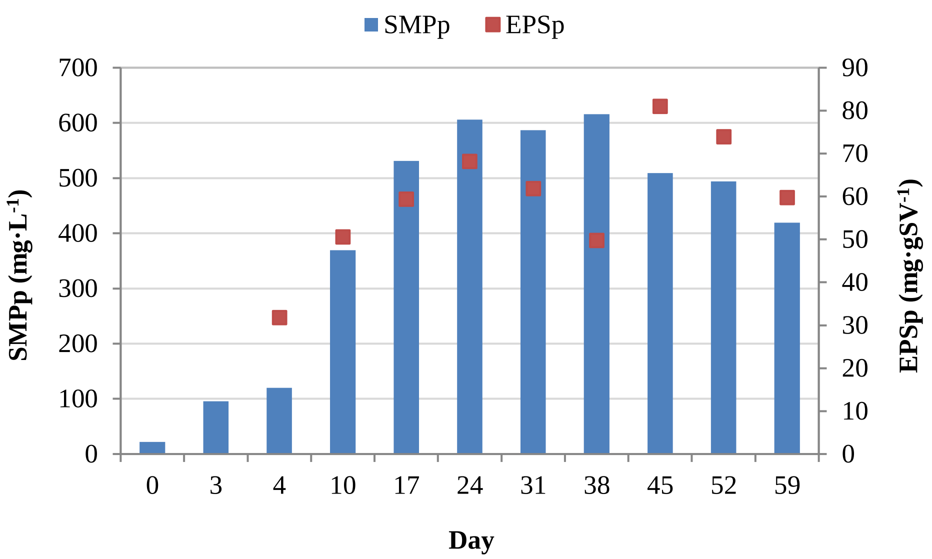
Figure 6: Mixed liquor characteristics a) MLVSS and b) CST.

3.5.2. SMP and eEPS analysis

SMP and eEPS content in an OMBR operation has a paramount importance. Salinity build-up in the biological reactor can increase the SMP and eEPS in the mixed liquor mainly due to cell lysis and secretion of organic cellular substances[43]. Proteins and carbohydrates are the main components found inherently in SMP and EPS [29]. SMP and eEPS content play also an important role in the membrane fouling [44]. Fig. 7 shows SMP and EPS concentrations in terms of proteins (Figs. 7.a), carbohydrates (Fig. 7.b) and DNA (Fig. 7.c) during the experimental period.In Fig. 7.a and 7.b, it can be observed that the SMP and eEPS concentrations increased as salt concentration increased in the biological reactor. However, from the day 38th of operation on, SMP and EPS concentrations decreased with the operation time. This denotes that biomass adapted to salinity environment observed above. These results are consistent with previously reported results, where salt accumulation in the mixed liquor resulted in an increased SMP and EPS content in an OMBR due to salinity accumulation [23,29,44].

DNA has also been measured in the SMP in order to study the eventual cell lysis. As it can be observed in Fig. 7.c, DNA concentrations increased dramatically until 17th day due to cellular lysis caused by the increase of the salinity in the reactor. From that day on, the increase was very slight. That means that cellular lysis diminished and the sludge withdrawals could stop the DNA increase in the reactor.

a)



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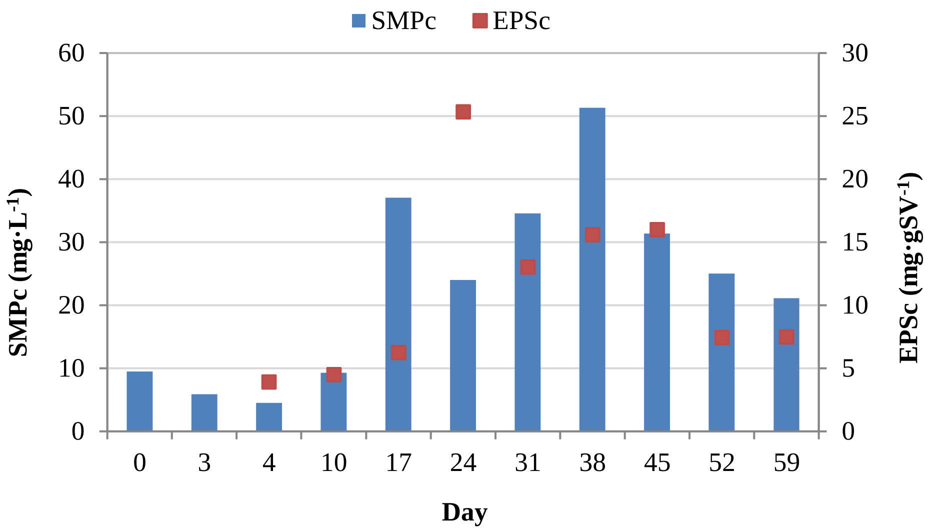

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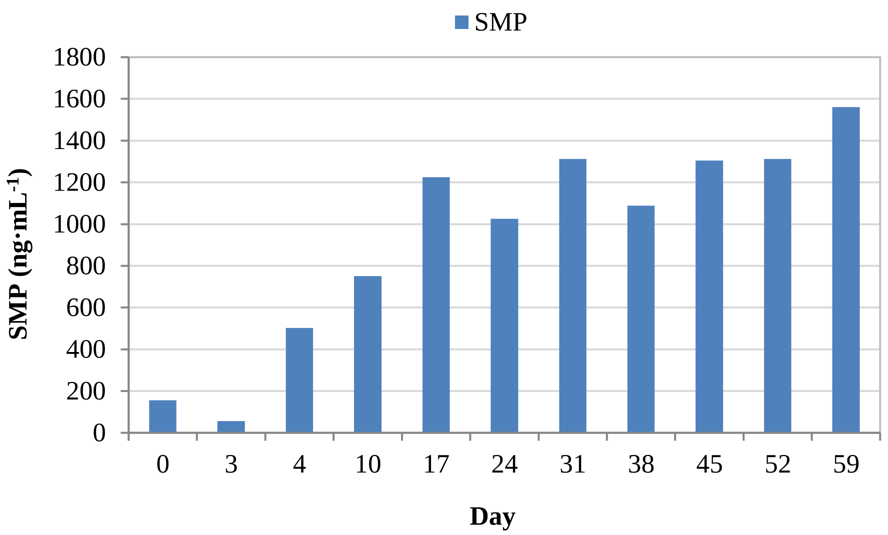

Figure 7: SMP and EPS concentrations in terms of a) proteins, b) carbohydrates and c) DNA.

4. Conclusions

The treatment of mixed municipal and industrial wastewaters has been successfully carried out with a laboratory OMBR. The residual fermentation brine from the table olive processing has produced the needed osmotic pressure difference between both sides of the membrane for the treated water permeation to the DS side. In this way, no costs for the regeneration of the DS have to be considered since the FTOP has been also used for this purpose. In addition, its feeding to the reactor mixed with municipal wastewater treatment has entailed the degradation of the phenols contained in the FTOP after an adaptation period.

On the other side, the salinity-build up, the generated SMP and the non-biodegradable organic matter accumulation have to be controlled in the process in order not to affect the biomass and consequently the organic matter removal efficiency.

The large-scale application of the OMBR to industrial wastewaters with slowly degradable organic matter will depend on the implementation of techniques for controlling the accumulation of the above mentioned substances.

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