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Biodegradation of olive mill wastewater in a membrane bioreactor: acclimation of the biomass and constraints

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ABSTRACT

In order to overcome the toxic effect of olive mill wastewater (OMWW) on biomass during biological treatment, this work will test OMWW biodegradation in a membrane bioreactor (MBR) using an acclimation procedure and will study its constraints. Special focus will be put on soluble microbial products (SMP) analysis in MBR and their impact on membrane fouling. The study was realized in an external ceramic ultrafiltration MBR which offers more flexibility than the other biological treatments (i.e. independence between both hydraulic and sludge retention time) and a smaller footprint. Fed with a mass ratio of 40% OMWW/60% glucose, MBR biomass showed efficient chemical oxygen demand and polyphenols removal rates of, respectively, 90 and 65% despite a low activity of 3.2 $mg_{O2}\ g_{MLVSS}^{-1}\ h^{-1}$ due to the harsh and toxic environment. Moreover, HPLC analysis has showed a removal from the permeate of the major phenolic compounds including hydroxytyrosol, tyrosol, and caffeic acid. The monitoring of SMP concentrations has contributed to identify the presence of an environmental stress during OMWW input. Polysaccharide and protein are the main SMP fractions released with, respectively, 10 ± 0.1–20 ± 0.5 mg g_{MLVSS}^{-1} and 4 ± 0.01–8 ± 0.01 mg g_{MLVSS}^{-1}. These SMP and higher molecular weight compounds brought by OMWW were found to be partially responsible for the intensive membrane fouling obtained. The feasibility of biomass acclimation directly to OMWW composed of multi-phenolic compounds was proved in MBR and its constraints were discussed. Microfiltration membrane would be suggested to overcome the constraints observed when ultrafiltration membrane was used (150 kDa).

Keywords: Acclimation; External ceramic membrane bioreactor; Soluble microbial products; Fouling; Olive mill wastewater

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1. Introduction

Olive extraction process is mainly carried out by traditional discontinuous press process or by continuous centrifugation of a mixture of milled olives and hot water. Both systems generate liquid waste with 40% coming from the presses and 95% from centrifugation [1]. Olive mill wastewater (OMWW) is considered as a contaminated and toxic effluent due to its high content of both organic matter and noxious phenol compounds [2,3]. The biological oxygen demand (BOD) values are ranging between 12 and 63 g L^{-1} and chemical oxygen demand (COD) values between 80 and 200 g L^{-1} [4]. These concentrations are around 200–400 times higher than those measured in a typical municipal sewage [5]. Usually, the OMWW phytotoxic and antimicrobial properties were related to monomeric phenols [6].

In Morocco, OMWW are pumped and discharged into evaporation ponds, directly dumped in rivers, or spread on the soil, without any reliable detoxification systems. Numerous researchers were interested in OMWW valorization options such as in agriculture, bioenergy production [1,7,8]. Dermeche et al. [9] have reported the possibility of bioconversion or extraction of valuable phytochemical compounds existing in OMWW. However, most of the proposed applications are not effective and has to be adapted to specific needs of local area.

The other option was to treat OMWW residues, reducing its pollutant effect. Various technologies have been applied and could be classified as physicochemical, biological, or combined processes. Physical and chemical methods are based on the principles of precipitation, coagulation, extraction, sedimentation, ion exchange, adsorption on active carbon, chemical oxidation, or advanced oxidation [10,11]. However, these processes suffer from serious drawbacks such as low efficiency. Recently, emerging processes such as membrane separations gained a lot of attention as promising tool for OMWW treatment [12]. These technologies, when combined to other methods of treatment (i.e. physicochemical or biological), lead to a maximal purification of OMWW [13,12]. Several combinations were investigated such as centrifugation/ultrafiltration [14], advanced oxidation process/ultrafiltration [15], and also a combined application of three technologies fungal pretreatment/anaerobic digestion/ultrafiltration [16]. Nevertheless, the high cost associated to these treatments remains the main problem.

Aerobic biological treatment is, in most cases, widely used and environmentally friendly. Aerobic treatment can be conducted by different micro-organisms including fungi [17] or bacteria [18].

At large scale, conventional activated sludge (AS) process remains the most practical for wastewater treatment [19]. However, the application of this kind of system to OMWW biodegradation could be hampered due to the inhibitory effect of phenols on the microbial metabolism. So, the great restriction to biological processes application is related to the acclimation of biomass to phenolic compounds [20,21]. Therefore, a simple and effective method to obtain a specified biomass from AS for phenols treatment is highly desired.

Membrane bioreactor (MBR) could be an efficient alternative. Indeed, MBR system combines the retention of AS with long sludge retention time (SRT), which allows the development of specific slow growing micro-organisms able to remove low biodegradable pollutants, resulting in improvement of high strength wastewater treatment [22,23]. Furthermore, due to the independence between hydraulic retention time (HRT) and SRT, this technology permits an accurate control of operating parameters.

The main drawback of MBR process, membrane biofouling, has to be managed to guarantee the development of sustainable process. Exopolymeric substances (EPS) in their soluble form called soluble microbial products (SMP) have been considered as a primordial biofouling agent to control because of their easily accumulation in bioreactors [24]. These substances are composed of different organic compounds (e.g. polysaccharides, proteins, humic acids) and are secreted by micro-organisms in their surrounding environment during their growth, decay, and/or in a response to changing environmental conditions [25–28]. The type of substrate used (e.g. urban or industrial) to feed the biomass is one of the multiple parameters influencing the nature and quantities of SMP produced [29]. Unfortunately, there is a lack of information about the type and quantities of SMP released in MBR process when treating OMWW and their influence on membrane fouling.

Some studies were carried out using MBR and reported the acclimation of the biomass to a synthetic solution of phenol [30]. Dhaouadi and Marrot [31] showed the feasibility of OMWW biodegradation in MBR using previously acclimated biomass to synthetic phenol solution during one week. However, no information of acclimation of the biomass directly to OMWW with multi-phenolic compounds composition is available. Actually, it could be very challenging if the acclimation took place on real OMWW, especially to investigate the possibility of high biodegradation of this effluent. In view of above, this work will test the biodegradation of OMWW in an MBR using an acclimation procedure; will verify the process

feasibility and their constraints. Another aim was to identify the biomass limits in terms of phenolic compounds degradation via the acclimation procedure used. Special focus will be put on the SMP analysis in the MBR to evaluate their release during biomass acclimation and their impact on membrane fouling.

2. Materials and methods

2.1. The MBR setup

Experiment was carried out in an external 30 L MBR (Fig. 1) (Polymem, France) supplied by 18 L of mixed liquor. Biological medium aeration was provided by small bubbles and temperature was maintained at approximately 25°C using a cooling coil system in the MBR. Ultrafiltration was operated under crossflow filtration with a centrifugal pump which recycled sludge back to the membrane. The membrane system used was a tubular ceramic membrane (ultrafiltration, Novasep-Orelis, France) composed of ZrO_2–TiO_2 active mineral support. Membrane was characterized by a 150 kDa cut off and a 0.02 m^2 filtration area. Membrane used for the entire experiment had an initial water permeability of 100 $L\,h^{-1}\,m^{-2}\,bar^{-1}$. The transmembrane pressure (TMP) increase (i.e. for maintaining permeate flow constant) was monitored with manometers set at the inlet and outlet of membrane module. Both permeate and feed flows were kept constant at 0.75 $L\,h^{-1}$ which set an HRT of 24 h. Crossflow velocity was maintained around 4 $m\,s^{-1}$ to limit membrane fouling. The monitoring of membrane fouling was done by the usual control of TMP parameter and the check of both pressures (i.e. outlet pressure (Ps) and permeate pressure (Pp)) within the membrane. The TMP was determined according to the following formula:

$$\mathrm{TMP} = (\mathrm{Pe} + \mathrm{Ps})/2 - \mathrm{Pp} \quad (1)$$

where Pe = 1 + Ps, Pe; inlet pressure (bar), Ps; outlet pressure (bar), and Pp; permeate pressure (bar).

2.2. Kinetics of biodegradation study

The kinetics study of OMWW biodegradation was performed in batch experiment. AS were directly taken from WWTP of Le Rousset and put under endogenous conditions during 4 h. After that, specific volumes of OMWW to reach concentrations ranging from 0.005 to 2.5 $g_{COD}\,g_{MLSS}^{-1}$ were prepared and added to AS samples (500 mL) in order to determine the maximum specific rate of the OMWW biodegradation by a non-acclimated biomass. The kinetics were carried out by collecting samples of 30 mL during a total incubation of 24 h (between five and seven samples analyzed at a fixed time of 0, 0.5, 2, 4, 12, and 24 h). Then, samples were centrifuged and filtered to

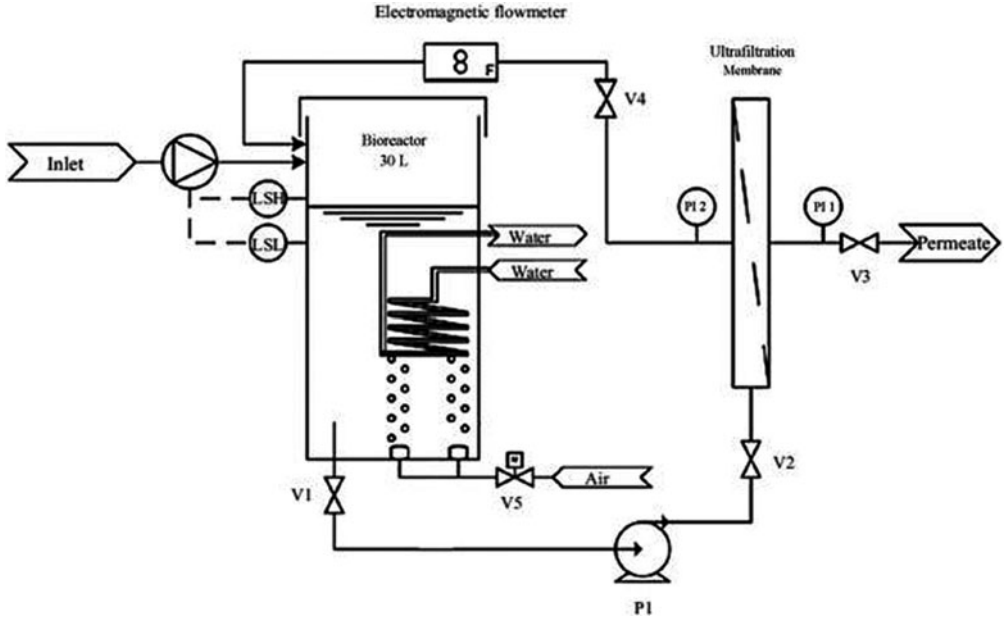

Fig. 1. Experimental set up of MBR. P: pump, V: valve, LSH: level security high, LSL: level security low, PI: pressure indicator.

separate mixed liquor and recover filtrate for subsequent COD concentration analysis.

2.3. Biomass acclimation

Biomass came from municipal wastewater treatment plant which is a submerged MBR (Le Rousset), France, 12,000 inhabitant equivalent, 1,800 $m^3\ d^{-1}$, organic load 0.1 $kg_{BOD5}\ kg_{MLVSS}^{-1}\ d^{-1}$). Biomass samples (AS) were taken from the submerged MBR and then transported to the lab scale MBR without aeration (1 h) for the experiments. The initial mixed liquor suspended solids (MLSS) concentration was around 6 $g\ L^{-1}$ and then was concentrated at around 8 $g_{MLSS}\ L^{-1}$ in MBR process. A balanced synthetic sewage influent was prepared (Table 1). The food-to-micro-organisms ratio (F/M) was changed successively during this experiment according to biomass growth (i.e. steps I/II/III). Eq. (2) was used for the expression of F/M ratio.

$$F/M\,\text{ratio} = (Q \times S \times 24)/(X \times V) \quad (2)$$

where Q; daily flow of effluent to be treated (L h^{-1}), S; the COD concentration of the effluent (g L^{-1}), X; the concentration of the biomass in the aeration tank ($g_{MLVSS}\ L^{-1}$), and V; volume of the aeration tank (L).

In step I, the F/M ratio was fixed around 0.2 $kg_{COD}\ kg_{MLVSS}^{-1}\ d^{-1}$ with the adjustment of the nutrient content (COD) in the feed to the mixed liquor volatile suspended solids (MLVSS) content. Then, the F/M ratio was increased to around 0.3 $kg_{COD}\ kg_{MLVSS}^{-1}\ d^{-1}$ to ensure the biomass growth (step II). In the two first steps, the glucose was used as carbon source in synthetic effluent. In step III, the glucose content in synthetic effluent was progressively changed by OMWW collected from a discontinuous extraction unit from Marrakech, southern Morocco (Table 1). The F/M ratio was kept at 0.3 $kg_{COD}\ kg_{MLVSS}^{-1}\ d^{-1}$ and progressive addition of OMWW volume was realized as biomass growth was observed for several days. The biomass was maintained at an infinite SRT.

Table 1
Characteristics of the OMWW and synthetic influent

Characteristics of the OMWW	
pH	5
Conductivity (ms cm^{-1})	9.3
COD (g L^{-1})	96
Total phenols (g L^{-1})	3.23
Dry matter (g L^{-1})	104
Volatile matter (g L^{-1})	88
Ash (g L^{-1})	16
Mass composition of synthetic influent (g g_{MLVSS}^{-1})	
$C_6H_{12}O_6$	2.1
$NaHCO_3$	0.4
$MgSO_4$	0.1
KH_2PO_4	0.2
$CaCl_2$	0.02

2.4. Analytical methods

AS was daily sampled from the biomass tank (30 mL) and centrifuged at 16,000 g for 15 min to isolate suspended solids from the supernatant. COD and phenolic compounds concentrations were measured biweekly in the inlet (Substrate), bioreactor supernatant, and the outlet (Permeate) to assess their removal rates. COD concentrations were evaluated by spectrophotometer (605 nm) with reagent kits purchased from Aqua Lytic (Germany). Phenolic compounds were quantified by means of the Folin–Ciocalteu colorimetric method according to Macheix et al. [32]. Phenolic extracts were also analyzed by HPLC performed using Eurospher II 100-5 C-18 reversed phase column (Knauer-HPLC) equipped with a photodiode array detector and a software analysis. An efficient gradient of acetonitrile-o-phosphoric acidified bidistilled water (pH 2.6) was used and the elution consisted of linear gradient program of the acenotrile/water mixture over a detection time about 60 min. The separation inside the chromatographic system was realized at a temperature of 25°C under a pressure of 109 bar and 1 mL min^{-1} as a flow rate. The identification of phenolic compounds was executed on the basis of their spectra in comparison with nine phenolic standards (caffeic acid, ferulic acid, syringic acid, p-coumaric acid, tyrosol, quercetin, 2-hydroxycinnamic, 2-4 hydroxyphenylethanol, hydroxytyrosol). To measure oxygen uptake rate (OUR) ($mg_{O2}\ L^{-1}\ h^{-1}$), an oxygen probe was directly implemented in the bioreactor. During this measurement, biomass was still fed (i.e. exogenous conditions). The aeration was stopped and a series of dissolved oxygen concentration measurements ($mg_{O2}\ L^{-1}$) were realized within the bioreactor. Then, the exogenous activity was obtained, dividing the OUR by biomass concentration ($g_{MLVSS}\ L^{-1}$). Polysaccharides [33], proteins, and humic substances [34] as main SMP constituents, were quantified by colorimetric methods in the bioreactor supernatant and in the permeate to evaluate their relation with the membrane fouling.

3. Results and discussion

3.1. OMWW biodegradation kinetic

Several experiments have been already performed to investigate the biodegradation kinetic using synthetic wastewater including phenol as unique inhibitory substrate [21,30]. In order to investigate the biodegradation kinetic of real OMWW effluent, our experiment was performed in aerated batch reactor to obtain information about the inhibitory effect of OMWW on biological growth and COD removal. The specific growth rate (μ) vs. OMWW concentrations was determined as shown in Fig. 2. Eq. (3) was used for the calculation of μ at each given OMWW concentrations analyzed.

$$\mu = \alpha / [\text{MLSS}] \quad (3)$$

where μ: specific growth rate (d^{-1}), α: the slope of the curve ($g_{COD}\,L^{-1}\,h^{-1}$) relating COD removal during time at a given OMWW concentration, and [MLSS]: concentration of MLSSs ($g\,L^{-1}$).

The results have shown that the OMWW biodegradation rate increased first with the increase of OMWW concentration. A linear specific growth rate was detected at the same time of OMWW addition to reach a maximum of $1.8\,d^{-1}$ in the presence of $2\,g_{COD}\,L^{-1}$, and then tended to decline after further augmentation of COD concentration (superior to $2\,g_{COD}\,L^{-1}$). The substrate inhibition effect was apparent at this very low concentration of OMWW.

After biomass adaptation to the new process and hydrodynamic conditions of the pilot, the acclimation experiment to OMWW was started using a new substrate composed of a mass ratio 20% OMWW/80% glucose. With this ratio, a COD concentration from OMWW equal to $0.33\,g_{COD}\,L^{-1}$ was added to the substrate. This concentration lower than the one of $2\,g_{COD}\,L^{-1}$ obtained for maximal specific growth rate was considered as a good one to acclimate the biomass softly. Furthermore, this low concentration had no inhibitory effect on biomass and it allowed, in the same time, to limit the membrane fouling, easily obtained by a too fast biomass growth at higher OMWW concentration.

3.2. Biomass development and treatment performances

The evolution of the MLVSS and the F/M ratio are represented and divided in three characteristic steps of the process development (Fig. 3). The F/M ratio was initially fixed at $0.2\,kg_{COD}\,kg_{MLVSS}^{-1}\,d^{-1}$ using synthetic substrate to simulate the WWTP conditions. The MLVSS concentration decreased during the first step (I) followed by a lag phase that was due to micro-organisms adaptation period to the MBR process and to the synthetic influent. The F/M during this period of adaptation (step I) has been maintained around 0.22–$0.25\,kg_{COD}\,kg_{MLVSS}^{-1}\,d^{-1}$ by daily adjusting the substrate content to biomass concentration. Then, the F/M ratio was increased around $0.3\,kg_{COD}\,kg_{MLVSS}^{-1}\,d^{-1}$ and the nutrient concentration of synthetic effluent was progressively increased to obtain the exponential growth of the biomass occurred in the second step (II).

The biomass growth was obtained first in the presence of glucose as a synthetic effluent sole carbon source (step II). At the end of period II, biomass was characterized by an exogenous activity of $22\,mg_{O2}\,g_{MLVSS}^{-1}\,h^{-1}$ and an excellent COD removal efficiency of 95% (Fig. 4(a)). The high exogenous activity was in accordance with the values found in literature on heterotrophic exogenous activity operating with same synthetic substrate and F/M ratio [35].

Afterwards, the glucose substitution by OMWW effluent had progressively changed during the MBR feeding (step III). The F/M ratio was maintained at $0.3\,kg_{COD}\,kg_{MLVSS}^{-1}\,d^{-1}$ and the OMWW/glucose ratio was increased as soon as the biomass growth was observed. At the first contact with OMWW effluent

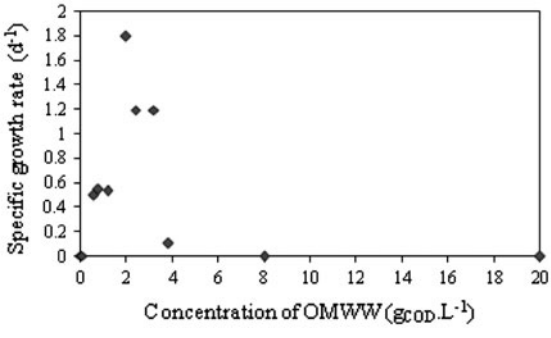

Fig. 2. Specific growth rates as a function of the OMWW concentration.

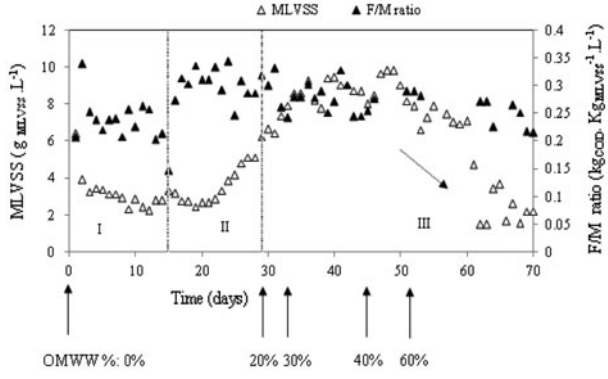

Fig. 3. Evolution of MLVSS and F/M ratio vs. time.

(20% OMWW/80% glucose as mass ratio), biomass dropped to about 3% (day 29–31). Simultaneously, the biomass exogenous activity measure reached 3.2 $mg_{O2}\ g_{MLVSS}^{-1}\ h^{-1}$. This value is typical of biomass activity during endogenous conditions or during stress period [35]. The brutal change of substrate provoked stress conditions on biomass previously fed with synthetic substrate, easily biodegradable. However, biomass degraded COD at 95% rate (Fig. 4(a)) and phenolic compounds were removed at a 65% rate (Fig. 4(b)). Furthermore, HPLC analysis performed on the feed and on the permeate has showed a removal of caffeic acid, hydroxytyrosol, and tyrosol, the major phenolic compounds present in the inlet. The polyphenols elimination rate was acceptable as this was the biomass' first contact with OMWW.

After that, MLVSS concentration continues to increase until 8 g L^{-1} (Fig. 3). This result showed that a portion of energy resources is still assigned to biomass growth and development, even if the major part seems to be diverted for the stress management and the protection of micro-organisms against the OMWW toxicity. Then, in the two successive periods (day 36–38) and (day 40–44), biomass losses of 15 and 11% were detected due to technical problems in the bioreactor.

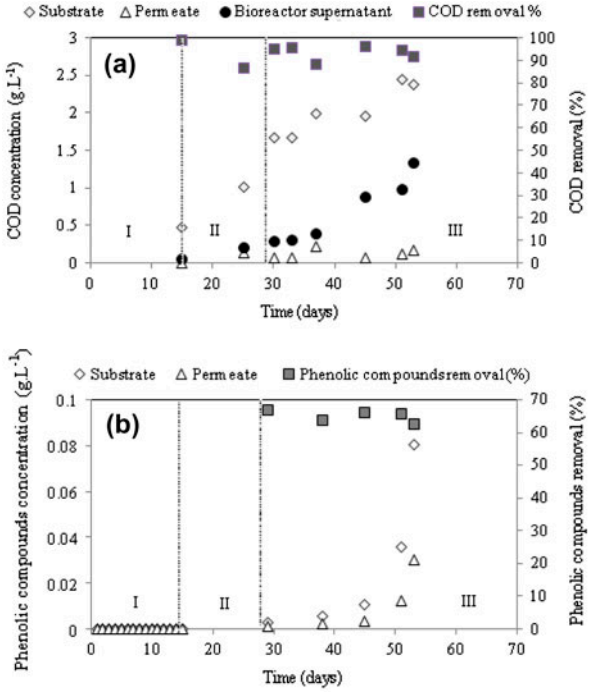

Fig. 4. (a) COD concentration and removal efficiency as a function of time and (b) phenolic compounds concentration and removal efficiency as function of time.

After increasing the mass ratio to 40% OMWW/60% glucose, a slight growth of biomass occurred over six days until day 51 (Fig. 3), showing the biomass survival capacity to the effluent toxicity. Biomass activity was maintained at 3.2 $mg_{O2}\ g_{MLVSS}^{-1}\ h^{-1}$ and both COD and polyphenols removal rates were maintained at, respectively, 90 and 65%. MBR gave interesting results in terms of COD removal against a low one (60%) obtained in a lab-scale sequencing batch reactor (SBR) [36] treating an OMWW with phenolic compounds and COD concentrations in the same range (i.e. 70 g L^{-1} of COD and 3 g L^{-1} of total phenolic compounds). This higher COD removal rate came from two main reasons. The first one was the presence of UF membrane which allowed to obtain a permeate with high quality compared to the released effluent of SBR. Then, the influent used was a mix of glucose easily biodegradable and COD from OMWW slowly biodegradable. In the mentioned study, SBR biomass was only fed with COD supplied by diluted OMWW. Both elements contribute to explain the higher COD removal rate obtained in the present study.

As soon as OMWW/glucose mass ratio was increased at 60% OMWW/40% glucose (day 51), a brutal decline of the biomass was revealed (Fig. 3) with an accumulation of COD in the bioreactor (Fig. 4(a)). The biomass activity decreased to 2.4 $mg_{O2}\ g_{MLVSS}^{-1}\ h^{-1}$ showing that this high load of OMWW was less favorable to biomass growth during step III. This mass ratio of 60% OMWW in the alimentation seems to be too toxic to biomass acclimation. However, another explanation can be given to this rapid biomass drop. In Fig. 4(a), it can be seen that even if high COD removal rate was assessed, an accumulation of COD was showed from day 35 to 55 in the bioreactor supernatant and increased from 0.4 to 1.4 g L^{-1}. This COD increase is clearly due to the accumulation of OMWW compounds with molecular weights higher than the membrane cut off of 150 kDa and/or due to a rearrangement of organic matter from OMWW, in particular phenolic compounds with bacterial flocs and SMP (Fig. 4(b)). As a result of this OMWW accumulation, the global toxicity in the bioreactor may have rapidly increased, provoking an environmental stress and the biological death. The use of membrane with higher cut off as it is the case of microfiltration membrane could an eventual solution to avoid or at least minimize these constraints.

3.3. Evolution of membrane fouling and SMP release

The regulation of TMP was necessary to maintain a permeate flow at 0.75 L h^{-1}. The evolution of TMP over the time (Fig. 5(a)) was also divided on three distinct phases.

During the first 20 days of step (I), the TMP was observed regularly constant at a value of 0.5 bar. This low TMP was maintained during this period of time where the bioreactor showed low biomass growth. In step (II), the biomass evolution was characterized by exponential growth. Thus, with the increase of MLVSS concentration, the TMP increased to approximately 0.75 bar (Fig. 5). The step (III) was characterized by the progressive introduction of OMWW effluent in the MBR. A frequent fouling of the membrane was observed during this step. The initial membrane fouling was noticed directly after the first biomass contact with a mass ratio of 20% OMWW. An immediate increase on TMP to a value of 1 bar was showed at day 31 followed by a consecutive raise on TMP, respectively, during the 41st, 45th, and 57th days. According to these results, the membrane clogging seems to be influenced principally by the introduction of OMWW in a new higher mass ratio that increase progressively the load of molecules with high molecular weight in the tank, and then contribute to the membrane fouling. Recent study carried out by Cassano et al. [37] has reported during the fractionation of OMWW by an integrated membrane process (2 UF + NF), a retention of molecules of high molecular weight during ultrafiltration step. Moreover,

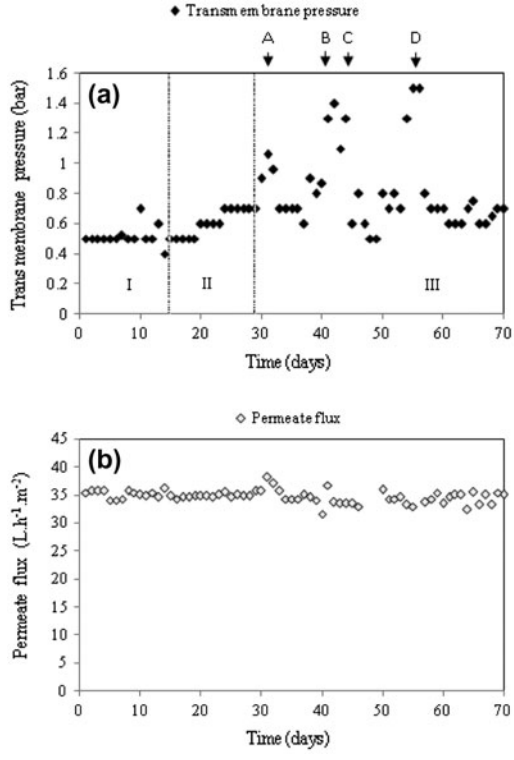

Fig. 5. Evolutions of (a) TMP and (b) permeate flux with time (Wash out times: A, B, C, D).

Sayadi et al. [38] showed that OMWW contain a fraction of high molecular weight >60 kDa which could be involved in membrane fouling.

Moreover, active biodegradation by the biomass or cell lysis could result in the release of a mixture of complex molecular weight polymers, known as SMP, in the surrounding environment [25,39,40]. The concentration of these SMP could vary significantly under stressful conditions (i.e. external supply of toxicity: OMWW). Thus, in the presence of toxic compounds, microbial cells present in AS produced more SMP to protect themselves from the harsh environment [41,42]. Furthermore, the variation on SMP biosynthesis could greatly depend on the availability of carbon and on the balance between carbon and the other nutrients [43–45]. SMP play an important role on maintaining the bacterial cell integrity, however, their deposition and accumulation on membranes surface potentially induce fouling [24,46,47].

Fig. 6 shows the evolution of the SMP release during the period of OMWW input. Polysaccharides, proteins, and humic substances represent the principal components of SMP. The relationship between the presence of these substances in the same period of the increase of OMWW percentage, the increase of TMP, and their respective correlation with membrane biofouling were observed. An increase in the concentrations of polysaccharide and protein substances (Fig. 6(a) and (b)) were detected, respectively, at days 31, also from day 38 to day 45 and at day 53 which are the same periods of the TMP raise and the decline of the membrane permeability (Fig. 5(a)). The release of these compounds at these special intervals of time can be a result of bacterial metabolism or cell lysis which both of them report to the presence of an environmental stress or toxicity inside the bioreactor. However, the membrane fouling can be influenced by the presence of these substances which form a gel layer into the membrane surface ideal for further bacterial attachment, increasing the effect of membrane clogging potential [48,49]. In this study, it was noticed that the amount of polysaccharides and proteins released during the evoked periods were quite high with, respectively, 10 ± 0.1–20 ± 0.5 mg g_{MLVSS}^{-1} for polysaccharides and 4 ± 0.01–8 ± 0.01 mg g_{MLVSS}^{-1} for proteins. Sponza [50] has reported similar observations about EPS composition in the case of AS reactor treating pulp paper and petrochemical effluent. According to the same author, when the micro-organisms were under stress, in the presence of inert and toxic substances, there is an active secretion of polysaccharides in these reactors.

The simultaneous presence of SMP and the compounds coming from OMWW (e.g. mineral matter and

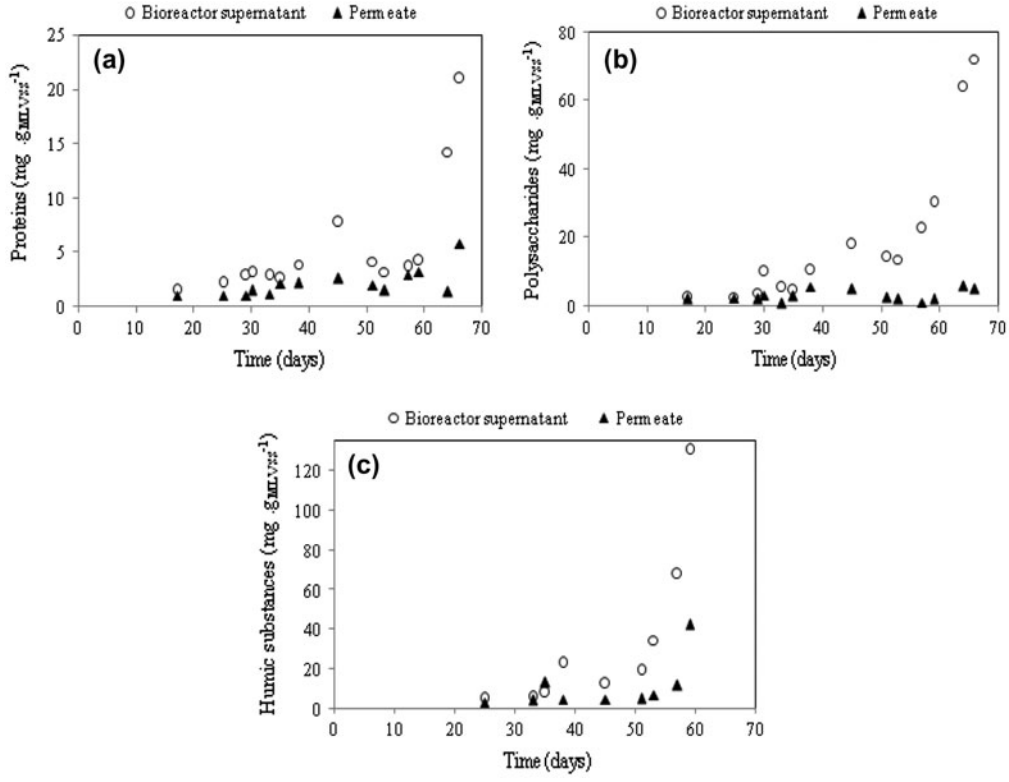

Fig. 6. Evolution of Exopolymeric substances (SMP) release during time: (a) Proteins, (b) Polysaccharides, and (c) Humic substances.

molecules with high molecular weight) during the filtration has no doubt stimulated and intensified the membrane fouling shown in Fig. 5(a). On the other hand, it was found in the analysis of the SMP that the humic fraction (Fig. 6(c)) found in the bioreactor came mostly from OMWW (20 ± 0.01–25 ± 0.01 mg g_{MLVSS}^{-1}). Their measure at low concentration in the permeate revealed their retention by the membrane contributing to its clogging.

A regular cleaning was done when the clogging was set up. Two types of fractions (organic or mineral) can be responsible for the fouling [23,46]. The application of a cleaning procedure (with NaOH 40 g L^{-1} followed by a diluted HNO_3 22 g L^{-1}) after each clogging, allowed the membrane cleaning and the recuperation of its initial permeability.

4. Conclusion

The biodegradation of OMWW in an external ceramic MBR using an acclimation procedure was performed. Biomass characterized by a low activity of 3.2 mg_{O2} g_{MLVSS}^{-1} h^{-1} due to harsh environment showed efficient treatment performances until effluent composition of 40% OMWW/60% glucose. Removal rates of COD and polyphenols reached, respectively, 90 and 65%.

The analysis of the SMP during OMWW input has contributed to finding a part of explanation of the membrane fouling showed in this study. The feasibility of biomass acclimation directly to OMMW was proved. However, microfiltration membrane may be suggested rather than an ultrafiltration membrane (150 kDa) to avoid the accumulation of high molecular weight compounds of OMWW, also responsible for membrane fouling and for the important biomass lysis occurred.

Acknowledgements

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List of symbols

Pe — inlet pressure (bar)
Ps — outlet pressure (bar)
Pp — permeate pressure (bar)
μ — specific growth rate (d^{-1})
α — the slope of the curve ($g_{COD}\ L^{-1}\ h^{-1}$) relating COD removal during time at a given OMWW concentration
MLSS — concentration of MLSSs ($g\ L^{-1}$)
Q — daily flow of effluent to be treated ($L\ h^{-1}$)
S — the COD concentration of the effluent ($g\ L^{-1}$)
X — the concentration of the biomass in the aeration tank ($g_{MLVSS}\ L^{-1}$)
V — volume of the aeration tank (L)

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