



## Sequential batch membrane bio-reactor for wastewater treatment: The effect of increased salinity



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### HIGHLIGHTS

- A SB-MBR pilot plant treating wastewater subject to salinity increase was investigated.
- Salinity increase influenced the biological contribution of COD removal efficiency.
- Salinity increase did not exert a significant stress on heterotrophic bacteria.
- Significant reduction of the respiration rates of autotrophic species due to salt.
- The irreversible cake deposition was the predominant membrane fouling mechanism.

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### ABSTRACT

In this work, a sequential batch membrane bioreactor pilot plant is investigated to analyze the effect of a gradual increase in salinity on carbon and nutrient removal, membrane fouling and biomass kinetic parameters. The salinity was increased by 2 g NaCl L<sup>-1</sup> per week up to 10 g NaCl L<sup>-1</sup>. The total COD removal efficiency was quite high (93%) throughout the experiment. A gradual biomass acclimation to the salinity level was observed during the experiment, highlighting the good recovery capabilities of the system. Nitrification was also influenced by the increase in salinity, with a slight decrease in nitrification efficiency (the lowest value was obtained at 10 g NaCl L<sup>-1</sup> due to lower nitrifier activity). Irreversible cake deposition was the predominant fouling mechanism observed during the experiment. Respirometric tests exhibited a stress effect due to salinity, with a reduction in the respiration rates observed (from 8.85 mgO<sub>2</sub> L<sup>-1</sup> h<sup>-1</sup> to 4 mgO<sub>2</sub> L<sup>-1</sup> h<sup>-1</sup>).

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### 1. Introduction

Industrial saline wastewater is usually rich in organic matter and toxic compounds, meaning that it needs to be treated prior to discharge into the environment to prevent surface and ground water quality decreases (Jang et al., 2013).

Biological processes may represent an alternative and cost-effective solution for treating saline wastewater compared to commonly used physicochemical processes. Therefore, several authors have investigated the impact of high salinity on conventional activated sludge (CAS) processes (Campos et al., 2002; Rene et al., 2008). Researchers found that high salt contents produce negative impacts on the performance of CAS systems (e.g., compromised settling, cell plasmolysis, decrease of biomass respiration rates, etc.) (Kargi and Dincer, 1996; Campos et al., 2002; Mannina

et al., 2016). To overcome the negative effect of salt, researchers have investigated using innovative biological technologies for treating saline wastewater (e.g., membrane bioreactor – MBR). The use of MBR technology provides considerable advantages over CAS systems including: higher effluent quality, lower footprint requirement, high sludge retention time (SRT), an almost complete absence of pathogenic bacteria in the effluent and faster biomass acclimation (Judd and Judd, 2010). Thus, the use of MBRs for treating high strength wastewater containing toxic compounds has been proven effective (Jang et al., 2013). However, it is crucial to investigate how biomass kinetics and activated sludge features affect the biological and physical performance of MBRs used to treat saline wastewater. With this in mind, several studies have been recently carried out on MBRs treating saline wastewater (Jang et al., 2013; Johir et al., 2013; Di Trapani et al., 2014; Luo et al., 2015). Jang et al. (2013) found a reduction of the ammonia removal and an increase in membrane fouling when treating high salinity wastewater was due to particular features of the microbial

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community. [Johir et al. \(2013\)](#) found a decrease in the dissolved organic carbon and ammonia uptake rate due to the inhibitory effect of the salinity. [Di Trapani et al. \(2014\)](#) compared the performance of a moving bed membrane bioreactor (MB-MBR) with a MBR system subjected to a gradual salinity increase (up to  $10 \text{ g NaCl L}^{-1}$ ) and showed an increase of the pore fouling tendency with the increase in salinity in the MBR ([Di Trapani et al., 2014](#)). Very recently, [Luo et al. \(2015\)](#) investigated the effects salinity has on the characteristics of the biomass and membrane fouling in MBR systems. They found that ammonia removal efficiency decreased considerably at high salinities ( $\geq 10 \text{ g NaCl}^{-1}$ ), especially immediately after the salinity shock load. Therefore, the autotrophic biomass suffers significantly when exposed to a high salinity environment. Nevertheless, several aspects still require further investigation to improve the efficiency of MBRs treating high strength saline wastewater.

Sequential Batch Reactors (SBRs), set up to treat industrial and civil wastewater in a single biological reactor and operated according to five discrete periods (fill, react, settle, draw and idle), were also investigated to treat saline wastewater ([Artan and Orhon, 2005](#); [Campos et al., 2002](#); [Rene et al., 2008](#)). [Amin et al. \(2014\)](#) recently found that treating saline wastewater in an SBR resulted in substrate consumption that is better described by a second-order kinetic rather than the Monod first order kinetic. [Ye et al. \(2009\)](#) found that the salinity exerted a significant impact on nitrifying bacteria in a SBR. Moreover, the bacterial wash-out due to salinity may promote an increase in the effluent turbidity ([Amin et al., 2014](#)). During the last decades, new approaches to designing and operating SBRs have been explored to achieve very high effluent qualities when treating non domestic wastewater (e.g., multiple tanks and innovative technologies such as membrane bioreactors – MBR, etc.). The use of multiple tanks enhances nutrient removal from non-domestic wastewater ([Bernet et al., 2000](#); [Ra et al., 2000](#)). However, the adoption of MBRs enables good performance for organic matter removal and sludge separation ([Yang et al., 2010](#)). Specifically, the combined use of SBRs and MBRs might overcome the drawbacks associated with salinity. To explore the possibilities using SBRs and widening their potential applications, this paper investigates for the first time, a pilot plant scheme composed of a combination of anoxic/aerobic/external MBR tanks.

In detail, this paper was aimed at evaluating the short term effect of a gradual increase of the inlet salinity in terms of carbon and nutrient removal, membrane fouling tendencies and biokinetic behavior.

## 2. Methods

### 2.1. The pilot plant and sampling scheme

The SB-MBR pilot plant was built at the Laboratory of Sanitary and Environmental Engineering of Palermo University ([Fig. 1](#)). A 320 L filling tank used for the storage of wastewater taken from the sewer system (the hydraulic retention time of the filling tank was equal to 1 day). This was connected to a 40 L stirring tank, where salt was added to the wastewater prior to being fed to the pilot plant. The SB-MBR pilot plant was designed according to a pre-denitrification scheme and consisted of two reactors in series, one anoxic (volume 45 L) and one aerobic (volume 224 L) and a MBR compartment (50 L). Furthermore, an oxygen depletion reactor (ODR) was installed between the MBR compartment and the anoxic reactor ([Fig. 1](#)).

The membrane was periodically backwashed (every 9 min for 1 min) by pumping a volume of permeate back through the membrane module from the Clean-In-Place (CIP) tank. The extraction flow was equal to  $20 \text{ L h}^{-1}$ . The SB-MBR plant was operated accord-

ing to the sequencing batch approach. In particular, 8 cycles per day were carried out. During each cycle, 40 L ( $Q_{IN}$ ) of urban wastewater with salt addition was added to the pilot plant. The reaction period and the solid–liquid separation (settle + draw of classical SBR) phase were set to 1 h and 2 h, respectively. During the solid–liquid separation phase, a permeate flow of  $20 \text{ L h}^{-1}$  ( $Q_{OUT}$ ) was continuously extracted. During the cycle, an  $80 \text{ L h}^{-1}$  flow ( $Q_{R1}$ ) was continuously pumped from the aerobic tank to the MBR tank. Furthermore, a recycled activated sludge ( $Q_{RAS}$ ) flow equal to  $80 \text{ L h}^{-1}$  during the reaction period and  $60 \text{ L h}^{-1}$  ( $Q_{R1}-Q_{OUT}$ ) during the solid–liquid separation phase, was continuously recycled from the MBR to the anoxic tank through the ODR tank.

The SB-MBR pilot plant was started up with sludge inoculum, withdrawn from a CAS system of Palermo's wastewater treatment plant, to obtain an initial mixed liquor suspended solids concentration of  $4000 \text{ mg L}^{-1}$ . The pilot plant was operated for 3 months without sludge withdrawing (indefinite SRT). [Table 1](#) summarizes the main wastewater characteristics (in terms of the average values and standard deviations – SD) as well as the pilot plant operational parameters. From [Table 1](#), it is worth noting that the HRT and F/M values were set to enable biomass acclimation to the increasing salinity, in particular to the nitrifying species (Ammonia Oxidizing Bacteria-AOB and Nitrite Oxidizing Bacteria-NOB) that are known to be very sensitive to salinity variations.

The experiment was divided into six Phases, with each characterized by a different salt concentration in the feeding wastewater. In detail, the salt concentration was gradually increased from 0 to  $10 \text{ g NaCl L}^{-1}$  (Phase I: no salt addition, Phase II:  $2 \text{ g NaCl L}^{-1}$ , Phase III:  $4 \text{ g NaCl L}^{-1}$ , Phase IV:  $6 \text{ g NaCl L}^{-1}$ , Phase V:  $8 \text{ g NaCl L}^{-1}$  and Phase VI:  $10 \text{ g NaCl L}^{-1}$ ). The NaCl dosing was increased  $2 \text{ g NaCl L}^{-1}$  per week. Phase VI had a longer duration (26 days) to investigate the biomass recovery, as discussed in the following.

During plant operations, the influent wastewater, the mixed liquor inside the anoxic and aerobic tanks and the effluent permeate were sampled and analyzed for total and volatile suspended solids (TSS and VSS), total chemical oxygen demand ( $\text{COD}_{TOT}$ ), supernatant COD ( $\text{COD}_{SUP}$ ), ammonium nitrogen ( $\text{NH}_4\text{-N}$ ), nitrite nitrogen ( $\text{NO}_2\text{-N}$ ), nitrate nitrogen ( $\text{NO}_3\text{-N}$ ), total nitrogen (TN), phosphate ( $\text{PO}_4\text{-P}$ ), total carbon (TC) and inert carbon (IC). All the analyses were carried out according to Standard Methods ([APHA, 2005](#)).

To discriminate between the removal effect that the biological processes had and the filtration provided by the membrane, two different removal efficiencies are calculated ([Di Trapani et al., 2014](#); [Mannina and Di Bella, 2012](#)): the biological removal efficiency and the total removal efficiency.

The biological removal efficiency was calculated from the difference of the  $\text{COD}_{TOT}$  in the influent and the  $\text{COD}_{SUP}$  measured in the supernatant of the mixed liquor samples (filtered using a pore size of  $0.45 \mu\text{m}$ ) withdrawn from the MBR tank. Conversely, the total COD removal efficiency (including the membrane filtration removal effect) was determined from the difference of the influent and the permeate  $\text{COD}_{TOT}$ .

The capillary suction time (CST) measurement ([Veselind, 1988](#)) was carried out in accordance with the EN 14701-1:2006 International Standard to investigate the sludge dewaterability.

### 2.2. Respirometric batch test

Respirometric batch tests were conducted using a “flowing gas/static-liquid” type batch respirometer ([Spanjers et al., 1996](#)). The suspended biomass samples were taken from the aerobic bioreactor and diluted with permeate to obtain a mixed liquor concentration in the range of  $2.0\text{--}3.0 \text{ g VSS L}^{-1}$ . Before performing the respirometric test, each sample was aerated until endogenous conditions were reached. In the batch tests aimed at determining the

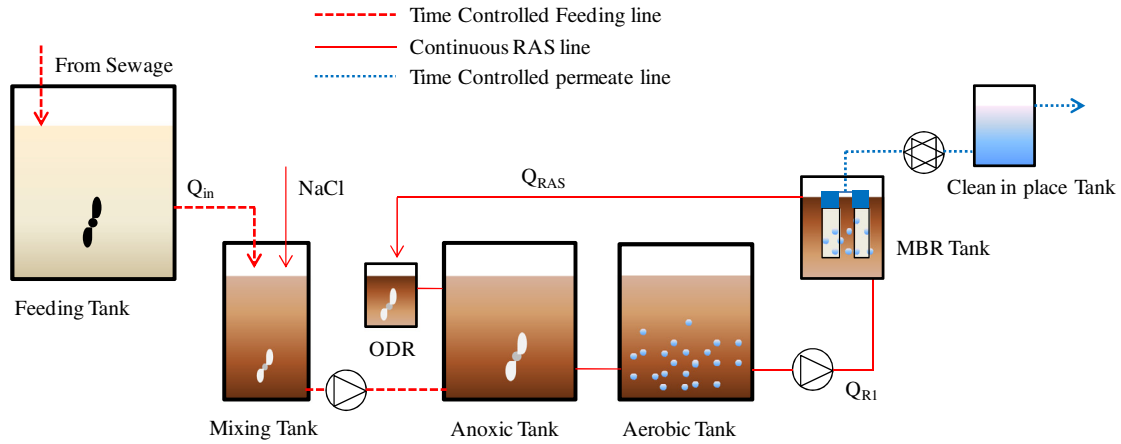


Fig. 1. Layout of the SB-MBR pilot plant.

**Table 1**  
Average influent characteristics, standard deviation and operational conditions.

System parameter	Unit	Value	SD
COD	mg L <sup>-1</sup>	240	100
BOD <sub>5</sub>	mg L <sup>-1</sup>	82	30
NH <sub>4</sub> -N	mg L <sup>-1</sup>	30	15
PO <sub>4</sub> -P	mg L <sup>-1</sup>	4	1
pH	–	7.2	0.5
NaCl	g L <sup>-1</sup>	0–10	–
HRT	h	20	–
F/M	kgBOD kgVSS <sup>-1</sup> day <sup>-1</sup>	0.085	–

HRT = hydraulic retention time; F/M = food microorganisms ratio; SD = standard deviation.

heterotrophic biokinetic parameters, the nitrifying biomass was inhibited by adding 10–15 mg L<sup>-1</sup> of Allylthiourea (ATU), whereas the exogenous oxygen uptake rate (OUR) was enhanced by the addition of a readily biodegradable organic substrate (sodium acetate in this study). The kinetic parameters of the autotrophic species were estimated using the same procedure. Nevertheless, no inhibiting substance such as ATU was added and ammonium chloride (NH<sub>4</sub>Cl) was directly spiked to evaluate the biokinetic parameters. For further details on the procedure used here, the reader is directed to the literature (Di Trapani et al., 2014; Di Trapani et al., 2010; Mannina and Viviani, 2009).

### 2.3. EPS analysis and extraction

Total extracellular polymeric substances (EPS<sub>T</sub>) were measured using the thermal extraction method, as reported by Mannina et al., 2016 and Cosenza et al. (2013a,b). Using this method, the EPS<sub>T</sub> can be partitioned into two fractions: soluble microbial products (SMPs) and bound EPS (EPS<sub>Bound</sub>). Both the SMPs and EPS<sub>Bound</sub> were divided into proteins and carbohydrate compounds. From Eq. (1), EPS<sub>T</sub> was evaluated as the sum of the proteins and carbohydrate compounds of the SMPs and EPS<sub>Bound</sub>:

$$EPS_T = \underbrace{EPS_P + EPS_C}_{EPS_{Bound}} + \underbrace{SMP_P + SMP_C}_{SMP} \quad (1)$$

where the subscripts P and C denote the content of proteins and carbohydrates in the EPS<sub>Bound</sub> and SMP, respectively.

Carbohydrates in the EPS<sub>T</sub> were determined according to the phenol-sulfuric acid method with glucose used as the standard (DuBois et al., 1956). Proteins were determined by the Folin method, as proposed by Lowry et al. (1951). For further details, the reader is directed to the literature (Cosenza et al., 2013a).

### 2.4. Resistances analysis

The total resistance ( $R_T$ ) to membrane filtration was evaluated using Darcy's law, as described in Eq. (2):

$$R_T = \frac{TMP}{\mu J} \quad [m^{-1}], \quad (2)$$

where TMP is the trans-membrane pressure (Pa),  $\mu$  the permeate viscosity (Pa s), and  $J$  the permeation flux (m s<sup>-1</sup>).

During membrane filtration,  $R_T$  can be defined as the sum of the intrinsic resistance of the membrane ( $R_m$ ) and the resistance due to membrane fouling ( $R_F$ ) (Eq. (3)):

$$R_T = R_m + R_F. \quad (3)$$

The membrane fouling ( $R_F$ ) can be divided into different components, according to Eq. (4):

$$R_F = R_{PB} + R_{C,irr} + R_{C,rev} = R_T - R_m, \quad (4)$$

where  $R_{PB}$  is the irreversible resistance due to colloids and particles deposition into the membrane pores,  $R_{C,irr}$  is the fouling resistance related to superficial cake deposition that can only be removed physically (hydraulic/sponge scrubbing) and  $R_{C,rev}$  is the fouling resistance related to superficial cake deposition that can be removed by ordinary backwashing.

The specific fouling mechanisms were studied by applying the resistance-in-series model described by Di Trapani et al. (2014).

To limit the total resistance with respect to a fixed threshold value, physical membrane cleaning was performed using the procedure described in the literature (Chang et al., 2001). Specifically, during physical cleaning, the membrane module was removed from the reactor and washed with ultrapure water to remove the cake layer on the membrane surface. Furthermore, the washed membrane module was placed into a tank with ultrapure water, and the TMP and  $J$  were monitored so that the total resistance ( $R_{T1}$ ) could be evaluated. Then, the washed module was immersed in the mixed liquor. The TMP and  $J$  were monitored so that the resistance after physical cleaning ( $R_{T2}$ ) could be evaluated.

Consequently,  $R_{T1}$  and  $R_{T2}$  are defined as:

$$R_{T1} = R_m + R_{PB}, \quad (5)$$

$$R_{T2} = R_m + R_{PB} + R_{C,rev}, \quad (6)$$

Thus, each fouling resistance portion was evaluated as:

$$\begin{cases} R_{PB} = R_{T1} - R_m \\ R_{C,rev} = R_{T2} - R_{T1} \\ R_{C,irr} = R_T - R_{T1} - R_{C,rev} \end{cases} \quad (7)$$

### 3. Results and discussion

#### 3.1. Organic and nutrient removal

Fig. 2a shows the total and biological COD removal efficiencies and Fig. 2b shows the removal ammonium nitrogen efficiency. For the COD removal, the total removal efficiency was quite high (the average value was equal to 93%) throughout the entire experiment, confirming the robustness of the MBR systems.

However, during Phases III and VI (which were characterized by salt concentrations equal to 4 and 10 g NaCl<sup>-1</sup>, respectively) the total COD removal efficiency decreased to 74% and 78%, respectively. Such a result may be related to a stress effect on the biomass activity due to the high salt rate (Mannina et al., 2016; Jang et al., 2013).

Fig. 2a shows the biological COD removal efficiency. Some interesting conclusions can be drawn from a subsequent analysis. Indeed, during Phase I (0 g NaCl L<sup>-1</sup>) the biological COD removal efficiency was close to 75% (on average). When the salinity increased to 2 g NaCl L<sup>-1</sup>, we noticed a significant decrease in the removal efficiency, reaching the lowest value (39%) in Phase III (4 g NaCl L<sup>-1</sup>). This decrease was probably due to the initial inhibitory effect of the salinity on the biomass, resulting in cell plasmolysis and/or the loss of metabolic activity (Yogalakshmi and Joseph, 2010; Johir et al., 2013).

Afterwards, we observed an increase in the COD removal efficiency until Phase V (8 g NaCl L<sup>-1</sup>). This was despite the increase to the feeding salt rate, suggesting that the biomass started to recover from the salt stress and began to acclimate to the saline environment (Hong et al., 2013). Therefore, a feeding salt rate equal to 4 g NaCl L<sup>-1</sup> might represent a threshold over which biomass acclimation occurs.

It is worth noting that at the beginning of the Phase VI (10 g NaCl L<sup>-1</sup>), the biological COD removal decreased again due to the high salt concentration. Therefore, a feeding salt rate of 10 g NaCl L<sup>-1</sup> might represent another threshold corresponding to a significant stress effect, despite the biomass being already acclimated to a moderately saline environment. Nevertheless, the biological COD removal efficiency recovered rapidly, because no further increase to the feeding salt rate occurred in the pilot plant. Such results demonstrate that the biomass can guarantee good carbon removal for high concentrations and long durations of salinity (recover biomass activity).

For ammonia removal efficiency, the observed results exhibited a slight decrease during the experiment (Fig. 2b). The lowest ammonia removal efficiency (63%) was obtained during Phase VI at the highest salinity (10 g NaCl L<sup>-1</sup>) (Fig. 2b). This result is consistent with previous studies where salinity values higher than 10 g NaCl L<sup>-1</sup> have been demonstrated to have adverse effects on the nitrification process through the inhibition of the metabolic activity and growth rate of nitrifying bacteria (Moussa et al., 2006; Yogalakshmi and Joseph, 2010). Indeed, the respirometric results confirmed the lower activity of nitrifiers during the first days of Phase VI compared to the results during Phase I.

For the denitrification efficiency, a very low average value was obtained (20%). This result was due to a problem in maintaining the anoxic conditions inside the anoxic tank. Indeed, during the solid–liquid separation phase, the dissolved oxygen concentration inside the anoxic tank increased to 0.8 mg L<sup>-1</sup>, limiting the anoxic growth of the heterotrophic biomass. Moreover, due to the F/M ratio, the readily biodegradable fraction of the influent COD was rapidly consumed, leading to denitrification process limitations. Therefore, denitrification occurred too slowly to guarantee sufficient removal efficiency.

#### 3.2. Biomass properties

As previously discussed, the SB-MBR pilot plant was operated without sludge withdrawals throughout the experiment. From the inoculum value (4 g TSS L<sup>-1</sup>), the TSS concentration at the end of Phase VI was equal to 5 g TSS L<sup>-1</sup>. Moreover, the CST measured from the mixed liquor at the end of Phase VI was equal to 24 s, which represents sludge with a significant amounts of bound water and relatively poor dewaterability (Jin et al., 2004).

Table 2 summarizes the results of the specific EPS concentration for each fraction (SMP<sub>p</sub>, SMP<sub>c</sub>, EPS<sub>p</sub> and EPS<sub>c</sub>) measured in the aerobic and anoxic mixed liquor.

From Table 2 we observe that the EPS<sub>T</sub> decreased with the increase in the feeding salt rate until Phase IV (6 g NaCl L<sup>-1</sup>). Conversely, a rapid increase in the EPS<sub>T</sub> occurred during Phase V (8 g NaCl L<sup>-1</sup>). It is worth noting that the EPS<sub>T</sub> decrease that occurred during Phase I did not influence the SMP concentration. Indeed, the SMP fractions were quite negligible throughout the experiment, with the exception of the carbohydrate fraction (SMP<sub>c</sub>) (Table 2). Such a result is likely due to a low F/M ratio, which led to the microorganisms using the released SMP as a substrate. Moreover, the aforementioned difficulties in maintaining the anoxic metabolisms in the reactors affected the SMP productions. This is in line with previous literature results. Indeed, Capodici et al. (2015) found that SMP production is enhanced when anoxic conditions are present in the reactor.

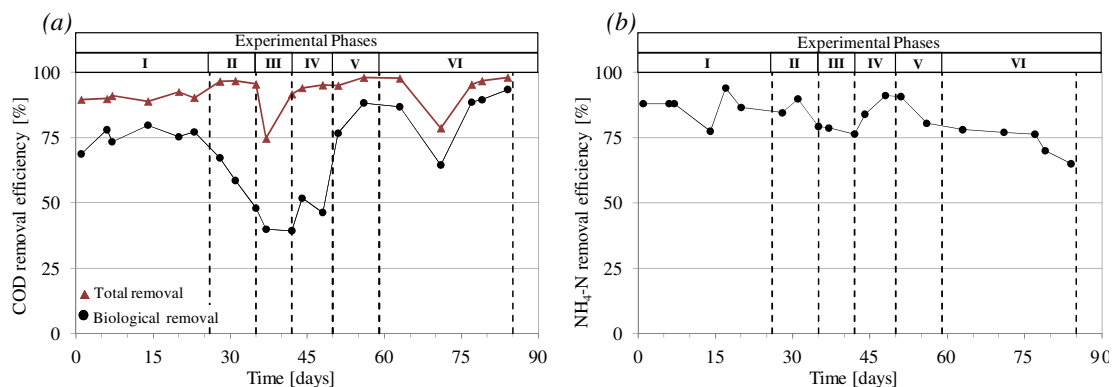
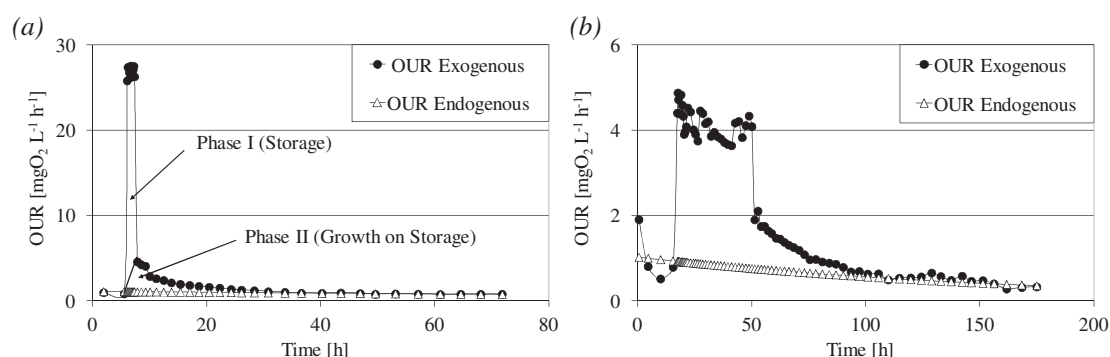


Fig. 2. COD removal efficiency (a) and NH<sub>4</sub>-N removal efficiency (b).

**Table 2**  
EPS<sub>T</sub> and specific concentration for each fraction (SMP<sub>p</sub>, SMP<sub>c</sub>, EPS<sub>p</sub> and EPS<sub>c</sub>) in the aerobic and anoxic mixed liquor.

Phase	Day	Aerobic					Anoxic				
		mgEPS gTSS <sup>-1</sup>									
		SMP <sub>p</sub>	SMP <sub>c</sub>	EPS <sub>p</sub>	EPS <sub>c</sub>	EPS <sub>T</sub>	SMP <sub>p</sub>	SMP <sub>c</sub>	EPS <sub>p</sub>	EPS <sub>c</sub>	EPS <sub>T</sub>
Phase I	28	0.00	15.39	127.30	14.30	156.98	2.26	11.11	129.66	17.44	160.47
Phase II	35	1.04	9.87	84.39	9.58	104.89	10.27	10.80	123.59	14.39	159.05
Phase III	42	0.00	0.00	76.70	28.32	105.02	0.00	0.00	75.46	68.45	143.90
Phase IV	48	0.00	6.08	60.45	16.84	83.37	0.00	1.36	68.05	19.36	88.78
Phase V	56	0.00	8.02	91.55	91.55	191.12	0.00	5.74	95.99	44.31	146.04
Phase VI	63	0.00	0.00	116.65	57.21	173.86	0.00	0.00	122.94	63.24	186.18
	71	0.00	0.00	115.99	42.51	158.49	0.00	0.00	126.23	40.11	166.34
	77	0.00	20.59	79.69	55.68	155.96	0.00	16.93	90.12	57.96	165.01
	84	0.00	0.00	83.16	29.69	112.85	0.00	0.00	81.91	28.26	110.16



**Fig. 3.** Typical respirometers for the heterotrophic (a) and autotrophic (b) bacterial species, respectively.

Therefore, the low F/M values likely masked the influence of the salinity on the EPS<sub>T</sub> production. Indeed, when increasing the feeding salt rate, an increase in the EPS, mostly in terms of SMP, takes place due to the cell autolysis and the secretion of organic cellular constituents, as reported in the previous literature (Jang et al., 2013).

### 3.3. Effect of salinity on biomass respiratory activity and biokinetic parameters

Respirometric batch tests were carried out for measuring the biomass activity during the experiment by evaluating the main kinetic and stoichiometric parameters of both the heterotrophic and autotrophic species.

The obtained respirogram charts featured the typical exogenous and endogenous behavior as a consequence of the readily biodegradable substrate addition (Fig. 3). Table 3 reports the averages obtained for the stoichiometric/kinetic parameters during the

experiment. Referring to the heterotrophic biomass, it is worth noting that the salinity increase did not cause a significant stress effect, with the main kinetic/stoichiometric parameters being in good agreement with the data available in the literature (Karahan-Gül et al., 2002; Hauduc et al., 2011; Mannina et al., 2011; Mannina and Viviani, 2010). Only a slight decrease in the heterotrophic yield coefficient ( $Y_H$ ) and the maximum heterotrophic growth rate ( $\mu_{H,max}$ ) were observed during the experiment. This result might suggest that variations in the biokinetic coefficients may be related to the existence of biochemical mechanisms within the bacterial cell for adaptation to a saline environment, which is in agreement with what was found in previous studies (Amin et al., 2014). Nevertheless, the similarity of the biokinetic parameters determined for the present study with the typical ones observed in CAS processes (Table 3) highlights the fact that a good acclimation level was reached at the end of the experiment. The respiration rates, expressed in terms of the specific oxygen uptake rate (SOUR), showed an almost constant value close to

**Table 3**  
Achieved values of kinetic/stoichiometric parameters during the experiments.

Heterotrophic	Phase II	Phase III	Phase IV	Phase V	Phase VI	Average	Typical	References
$Y_H$ [mgCOD mg <sup>-1</sup> COD]	0.70	0.58	0.59	0.58	0.59	0.61 (±0.05)	0.67	Hauduc et al. (2011)
$Y_{STO}$ [mgCOD mg <sup>-1</sup> COD]	0.78	0.74	0.77	0.78	0.75	0.76 (±0.02)	0.78	Karahan-Gül et al. (2002)
$\mu_{H,max}$ [d <sup>-1</sup> ]	5.37	4.05	3.98	3.11	4.07	4.15 (±0.93)	6	Hauduc et al. (2011)
$K_S$ [mgCOD L <sup>-1</sup> ]	18.13	5.15	6.45	5.37	40.7	17.34 (±16.72)	20	Hauduc et al. (2011)
SOUR <sub>max</sub> [mgO <sub>2</sub> g <sup>-1</sup> TSS h <sup>-1</sup> ]	12.00	13.66	11.77	11.03	10.44	11.78 (±1.41)	-	-
<b>Autotrophic</b>								
$Y_A$ [mgCOD mg <sup>-1</sup> N]	0.24	0.27	0.23	0.3	0.17	0.25 (±0.06)	0.24	Henze et al. (1987)
$\mu_{A,max}$ [d <sup>-1</sup> ]	0.16	0.1	0.14	0.12	0.21	0.15 (±0.05)	0.8	Hauduc et al. (2011)
$K_{NH}$ [mgNH <sub>4</sub> -N L <sup>-1</sup> ]	0.15	0.76	0.85	1.44	1	0.84 (±0.54)	1.00	Hauduc et al. (2011)
OUR <sub>max</sub> [mgO <sub>2</sub> L <sup>-1</sup> h <sup>-1</sup> ]	8.84	4.00	3.67	4.04	9.3	5.97 (±2.84)	-	-
Nitrif. rate [mgNH <sub>4</sub> -L <sup>-1</sup> h <sup>-1</sup> ]	1.74	0.71	0.77	0.74	1.87	1.17 (±0.63)	2.30	Di Trapani et al. (2011)

$Y_H$  = heterotrophic yield coefficient,  $Y_{STO}$  = storage yield coefficient,  $\mu_{H,max}$  = maximum heterotrophic growth rate,  $K_S$  = heterotrophic half-saturation coefficient, SOUR<sub>max</sub> = specific respiration rate,  $Y_A$  = autotrophic yield coefficient,  $\mu_{A,max}$  = maximum autotrophic growth rate,  $K_{NH}$  = autotrophic half-saturation coefficient.

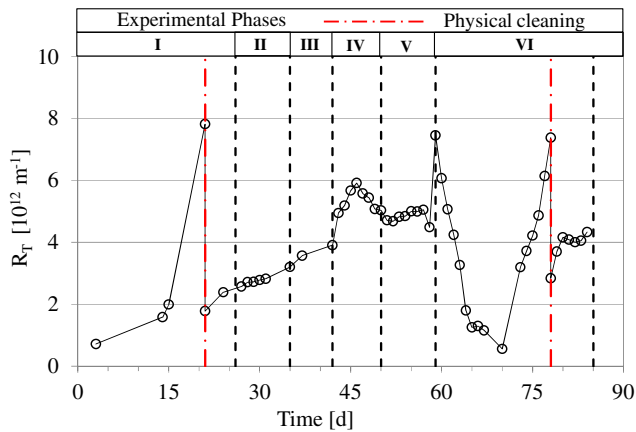


Fig. 4. Total membrane resistance ( $R_T$ ) over the time for each salinity phase.

$12 \text{ mgO}_2 \text{ g}^{-1} \text{VSS h}^{-1}$  (on average). Furthermore, the heterotrophic biomass exhibited a “storage” phenomenon, which is typical of systems subjected to dynamic conditions, such as SBR processes, which characterized by alternating feast/famine conditions. This condition most likely enhanced the growth of the bacterial groups that were able to rapidly convert the organic substrate into storage products. Such behavior is clearly depicted in Fig. 3a, which shows a typical respirogram obtained for the heterotrophic species. The storage yield coefficient  $Y_{\text{STO}}$  was evaluated from the procedure proposed by Karahan-Gül et al. (2002). For the autotrophic species, we observed a higher stress effect due to the salinity, with a significant reduction in the respiration rates (from  $8.85 \text{ mgO}_2 \text{ L}^{-1} \text{ h}^{-1}$  to  $4 \text{ mgO}_2 \text{ L}^{-1} \text{ h}^{-1}$ ). This result is consistent with previous experience that highlights the higher sensitivity of nitrifiers to salinity

variations (Di Trapani et al., 2014). However, this is likely because the salinity gradually increased with a moderate salt shock. At the end of the experiment (Phase VI), we noticed a sort of acclimation of the autotrophic species, with respiration rates that increased up to  $9.3 \text{ mgO}_2 \text{ L}^{-1} \text{ h}^{-1}$  during the batch tests. However, an estimation of the autotrophic biomass fraction would be necessary to properly evaluate the overall nitrification ability of the system that seemed to decrease with the highest salinity level, as reported in Section 3.1.

#### 3.4. Membrane filtration properties and fouling tendencies

Fig. 4 shows the  $R_T$  values during the experiment for each salt feeding rate. To limit the TMP to a threshold value of 0.7 bar, two membrane physical cleanings (days 21 and 78) were performed. During the experiment, we found that the salinity increase promoted an increase in membrane fouling. Indeed, the  $R_T$  value progressively increased from  $1.9 \times 10^{12} \text{ m}^{-1}$  (Phase I – day 21) to  $7.8 \times 10^{12} \text{ m}^{-1}$  (end of Phase V, see Fig. 4) with the salinity. The fouling rate ranged between  $0.02 \text{ bar day}^{-1}$  (during the Phase I) and  $0.06 \text{ bar day}^{-1}$  during Phase V. During Phase VI, the  $R_T$  decreased due to some technical problems (Fig. 4). However, when the technical problems were resolved, the  $R_T$  value rapidly increased to  $7.78 \times 10^{12} \text{ m}^{-1}$ . Indeed, the fouling rate increased at  $0.1 \text{ bar day}^{-1}$  at the end of Phase VI, indicating severe membrane fouling. This result is likely because the salinity increase can lead to an increase in the mixed liquor viscosity and to a reduction of the oxygen solubility, exacerbating membrane fouling (Lay et al., 2010).

Fig. 5 shows the results of the resistance fractions ( $R_{\text{PB}}$ ,  $R_{\text{C,irr}}$  and  $R_{\text{C,rev}}$ ) measured during the two membrane physical cleanings (day 21 and 78) derived from Eq. (7). Furthermore, the percentages of

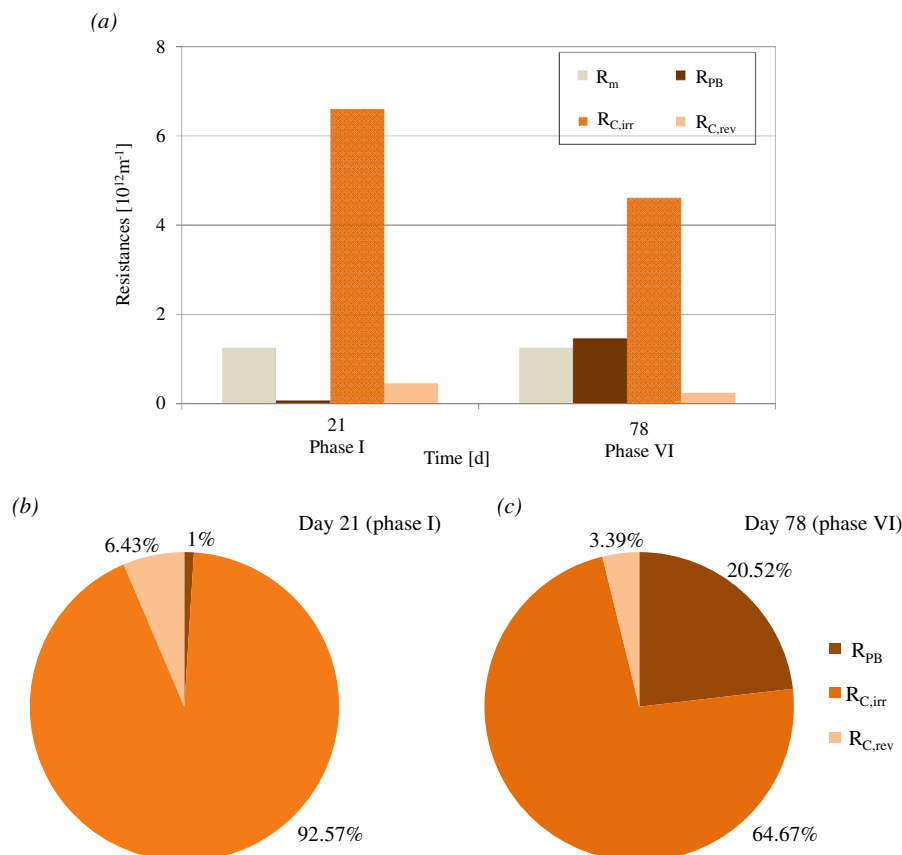


Fig. 5. Resistance fractions ( $R_{\text{PB}}$ ,  $R_{\text{C,irr}}$ , and  $R_{\text{C,rev}}$ ) (a) and percentages of each resistance fraction with respect to  $R_f$  (b and c).

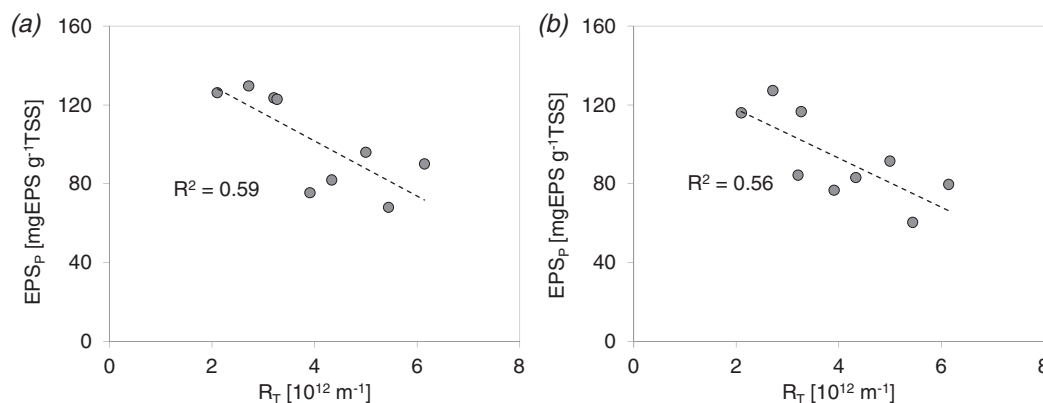


Fig. 6. Correlation between the total resistance to filtration  $R_T$  and the  $EPS_P$ , referring to the anoxic (a) and aerobic (b) compartments.

each resistance fraction with respect to  $R_F$  are reported (Fig. 5b and c). As shown in Fig. 5, a decrease in  $R_{C,irr}$  was observed with the increase in the salt concentration. Furthermore, an increase of the pore fouling resistance ( $R_{PB}$ ) was also found with the increase in the salt concentration. Indeed,  $R_{PB}$  was equal to 1% of  $R_F$  during Phase I and increased to 20.52% during Phase VI. However, this result agrees with other results for using MBR treating conventional wastewater. Indeed, the increase of the 20% of  $R_{PB}$  is likely exclusively due to the progressively increasing of the pore blocking mechanisms, which causes a loss in membrane permeability (Cosenza et al., 2013a).

Irreversible cake deposition ( $R_{C,irr}$ ) was the predominant fouling mechanism during both Phases I and VI (no salt addition and 10 g NaCl L<sup>-1</sup>, respectively). Such a result is in agreement with previous studies that have demonstrated that foulants are slightly transferred from the membrane surface (irreversible cake layer deposition) to the pores (pore blocking) in cases of gradual salinity variations (Di Trapani et al., 2014).

Additionally, the membrane fouling tendency may have been influenced by the EPS concentration during the experiment. In particular, Fig. 6 shows the relationship between the total resistance to filtration  $R_T$  and the specific  $EPS_P$ , referring to the anoxic (Fig. 6a) and aerobic (Fig. 6b) compartments, respectively. Fig. 6 shows that a particular and interesting behavior was observed: the  $R_T$  decreased when the bound  $EPS_P$  increased, which is different than what is generally highlighted by the technical literature (Judd and Judd, 2010). These results could be related to the fact that the salinity caused a decrease in the activated sludge properties (the activated sludge appeared “bloated” and highly hydrophobic, similarly to the bulking phenomenon in CAS systems), thus decreasing the “pre-filtering” effect of the cake layer (dynamic membrane), which was characterized by a lower resistance. As a consequence, the foulant agents, including the EPS excreted by the bacterial consortium, could reach more easily into the internal pores of the membrane, promoting an increase to  $R_{PB}$  (Fig. 5), as described previously.

#### 4. Conclusions

The findings of this study highlight that it is crucial to maintain a constant salinity when operating a SB-MBR in a saline environment. Such conditions could be obtained by proper equalization/homogenization of the inlet wastewater. Indeed, significant variations in the feeding salt rate can worsen the physical and biological performances. Nevertheless, the step-wise operation of the SB-MBR, as well as the possibility of optimizing the cycle duration with respect to the salinity level suggests that a system such as this one has great potential for saline wastewater treatment.

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