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### 1 Urban wastewater reuse using a coupling between nanofiltration and

- 2 ozonation: techno-economic assessment.
- 3

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7 Abstract: Combination of nanofiltration and ozonation was investigated for the treatment of urban 8 wastewater. First objective was to demonstrate that nanofiltration can be used instead of reverse 9 osmosis as it enables good rejection rates with reduced cost because of lower operating and 10 maintenance costs. In this way, this paper presents an economic and technical evaluation of the 11 proposed coupling where ozonation is used to treat retentates from nanofiltration. Reverse Osmosis System Analysis (ROSA) software was applied to simulate the filtration design. The effect of 12 membrane choice on specific energy consumption, capital, operation and maintenance costs and 13 14 scaling potential was investigated. It was demonstrated that using nanofiltration instead of reverse 15 osmosis enable cost saving of 35 k\$/year for 125 m<sup>3</sup>/h. Second objective was to evaluate the impact of 16 the treatment of retentates by ozonation on the global cost. It was highlighted that the coupling would 17 be an acceptable solution from an economic point of view for wastewater reuse. The possible reuse of 18 both permeate and concentrate enable an operating cost saving of 15.4 k\$/year for 125 m<sup>3</sup>/h. An 19 optimum recovery rate of 80 % was found for which cost of membrane process is balanced by a 20 decrease in the cost of ozonation.

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22 Keywords: nanofiltration, ozonation, wastewater reuse, economic analysis.

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### 25 Highlights:

26	Nanofiltration is a possible alternative to reverse osmosis for wastewater reuse
27	Nanofiltration combined to ozonation is a competitive process for wastewater reuse
28	Nanofiltration is a barrier for micropollutants with reasonable operating costs
29	Coupling nanofiltration and ozonation results in cost saving due to reuse possibility

30

### 31 **1. Introduction.**

Water resources management is a major challenge in economic development and public health. The 32 demographic growth and associated economic development will further exacerbate the situation in the 33 next twenty years by increasing water demand while discharging more and more polluted effluents. It 34 35 is therefore necessary, in addition to the draft of very strict regulations, to protect the quality of the resource, to challenge the marine or brackish resources desalination by developing cost-effective 36 systems, and also to define new and relevant treatment processes for urban wastewaters allowing 37 38 direct wastewater reuse and improvement of water resource protection. This concept of wastewater 39 treatment allowing direct recycling is an issue already tackled and resolved in part in the industrial 40 environment and in some "early bird" countries (Singapore for drinking water and Tunisia, California and Florida for irrigation water) (Wintgens et al., 2005). However, it is still secondary in densely 41 42 populated urban environments, where the major concern is to comply with standards imposed with 43 regards to the fragility or constraints of the receiving environment. Nevertheless, this reuse becomes 44 one of the essential points in optimal management of a freshwater resource, especially when considering its global cost, less than half the cost of desalination (Cote et al., 2005). 45

After massive investment in energy-intensive desalination processes which increased the global production capacity of desalinated water by 67% to 52 million m<sup>3</sup>.d<sup>-1</sup> (GWI, 2009), governments are now looking at overall water efficiency and start to prioritize water reclamation and reuse as a solution to water scarcity since water reclamation requires only a third to a fourth of the energy needed for

reverse osmosis (RO) desalination. While the combined approach of seawater desalination and water reuse has proven to be successful in a number of coastal sites such as Barcelona, Singapore, land locked cities have to focus completely on water reuse for irrigation or aquifer recharge, sometimes even accepting direct potable reuse as practiced in Windhoek, Namibia (Bunani et al., 2015, Bellona et al;, 2012). With a closed water loop, land locked cities require high-quality water reuse systems mostly based on membrane technology as affordable and efficient barrier.

56 Municipal wastewater is an alternative water source but increasingly contaminated with toxic organic 57 pollutants (Schwarzenbach et al., 2006). The occurrence of organic micropollutants (OMPs) in the 58 aquatic environment, often called 'emerging pollutants' as they became increasingly detectable since 59 the last two decades, has been identified as a global issue. Research has shown the ubiquitous presence of a broad range of pharmaceuticals, personal care products and industrial chemicals in the water 60 61 cycle, particularly in the effluent of wastewater treatment plants (WWTP) and surface waters in low 62 concentrations from ng.L<sup>-1</sup> to µg.L<sup>-1</sup> (Jorgensen and Halling-Sorensen, 2000; Heberer, 2002, Wode et 63 al., 2015, Hermes et al., 2018). Also, important concerns have been raised regarding that continuous discharge of antibiotics and anti-microbial products into aquatic environment may facilitate the 64 development or proliferation of resistant strains of bacteria (Kim and Aga, 2007). Indeed, the 65 emergence and spread of antibiotic resistant bacteria have been classified by the World Health 66 Organization as one of the three biggest threats to public health in the 21st century. Moreover, chronic 67 toxicity effects have been reported for aquatic organisms exposed to OMPs at trace concentrations. 68 Current technologies in existing WWTPs struggle to eliminate OMPs or to limit their concentration 69 and toxicity sufficiently to comply with the thresholds in new regulations. These evolutions are 70 driving the urban wastewater treatment to come up with advanced technologies like RO or activated 71 carbon (AC) are able to effectively remove OMPs without chemicals addition and limiting by-72 products formation (Gomez et al., 2012, Paredes et al., 2018). Nonetheless, RO is expensive because 73 74 of high operating pressure and is very sensitive to fouling thus requires a high level of water pretreatment or works best on groundwater or low solids surface water. AC could be efficient but its 75 76 performances are greatly affected by variations in pH, temperature, and flow rate as it could release or desorb (Benstoem et al., 2017). To recover fresh reusable water from urban wastewater treatment,
efficient additional treatment processes which are economically viable are required and thus must be
developed.

Nanofiltration (NF) has been proved to be efficient as a tertiary treatment as it enables high rejections 80 of small organic molecules (around 200-300 Da) due to size exclusion and electrostatic or 81 hydrophobic interactions. Moreover, this membrane technology requires operation pressure 82 significantly lower than traditional RO processes and without the need of further permeate 83 84 remineralization. Hence, NF provides an interesting alternative to RO for wastewater reuse with a 85 number of advantages: lower energy consumption due to lower pressure requirement, less chemical 86 additives, lower rejection of monovalent salts and thus less problematic membrane concentrates and 87 finally less heavy post treatment of re-mineralization (Bellona and Drewes, 2007). However, two main 88 limitations of NF application in WWTP are i) membrane fouling which affects directly process 89 performances and costs; and ii) the disposal of retentates which contains a wide range of concentrated 90 organic pollutants. Indeed, NF applied to a polluted water provides a large volume of practically clean 91 water (permeate) that can be reused immediately and a concentrated stream that requires further 92 treatment. The recovery ratio for NF is normally 50-85% which corresponds to a volume reduction 93 factor (VRF) of 2 - 7 and results in a waste stream which could be returned to the biological treatment 94 or could be discharged or treated depending on regulations and possibilities.

95 However, retentate recirculation upstream of the biological treatment is not possible in the long term 96 as salinity greater than 5 g.L<sup>-1</sup> may inhibit biological activity (Reid et al., 2006). In addition, the 97 recirculation of NF concentrates within a MBR significantly increases its clogging (Kappel et al., 98 2014). Finally, direct discharge into surface or marine waters is not easy to encourage given the 99 average composition of the urban retentates i.e. conductivities of 2 to 20 mS.cm<sup>-1</sup>, CODs ranging from 100 20 to 55 mg.L<sup>-1</sup> and levels of organic micropollutants multiplied by 3 to 7 (Benner et al., 2008; Solley 101 et al., 2010). Hence, treatment of membrane concentrates is recommended. 102 With this view in mind, ozonation was found to degrade efficiently most OMPs present in treated wastewater through the strong oxidative properties of ozone (applied dose:  $3-8 \text{ mg } O_3.L^{-1}$ ) and of the 103 104 hydroxyl radicals produced spontaneously during its decomposition. AOP based on ozonation could 105 thus be used to treat membranes rejection streams (retentate) containing high concentration of harmful 106 organics. The general advantages of the combined process NF/AOP or AOP/NF with recirculation of 107 the concentrate over direct treatment would be, depending on the configuration: (i) lower ozonation 108 treatment time, (ii) more efficient reagent consumption, (iii) treatment of lower flow rate, (iv) higher permeate flow (Van Geluwe et al., 2011; Park et al., 2017). Concentration of OMPs can be reduced to 109 a desired level by NF or ozonation separately, but only few researches took advantage of these two 110 wastewater treatment process to produce high quality effluent. 111

112 Over past years, objective of our researches is to prove that the combination of NF and ozonation 113 could be a promising, efficient and affordable solution for OMPs and global toxicity removal from 114 urban wastewater while reducing costs and limitations occurring when the two processes are carried 115 out separately, making wastewater reuse possible and safe for various applications (Azais et al., 2014; Azais et al., 2016; Azaïs et al., 2017). The final objective is to design novel integrated process 116 117 schemes combining NF and ozonation with structural (flow-sheet) and parameters optimization of 118 these processes. One of the design objectives of the integrated processes should be the minimization of 119 liquid wastes, thus reducing the load of the following waste treatment stage. Any new NF membrane plant needs an evaluation of the membrane performance in order to evaluate the number of NF 120 elements and type of membrane, this is essential in the prediction of the initial cost of the plant. In 121 addition, costs of emerging processes have to be evaluated. To our knowledge, this has never been 122 done for such coupling applied to wastewater reuse. In this way, the specific objective of this paper is 123 124 to present an economic and technical evaluation of the proposed coupling between NF and ozonation where ozonation is used to treat retentate from nanofiltration. Reverse Osmosis System Analysis 125 126 (ROSA) software developed by Filmtec (Dow Water and Process Solutions, 2011) was applied in this 127 study to simulate the proposed filtration design. The effect of membrane choice on power consumption in terms of specific energy consumption (kWh.m<sup>-3</sup>), capital, operation and maintenance 128

(O&M) costs and scale potential was investigated. Interest of the use of NF instead of RO was analyzed by comparing both processes. A process analysis and a comparative economic analysis of capital and operating costs for both technologies were performed. Cost of ozonation was estimated based on experimental results concerning ozonation of real wastewater matrice concentrated by nanofiltration (Azais et al., 2016a). Thus, ozone doses are known for this effluent. Finally, impact of the treatment of concentrates by ozonation on the treatment costs was considered.

135

136 **2.** Material and methods.

### 137 2.1. Cost of nanofiltration

ROSA 9.0 software model was applied to predict water quality and flow rate for different membrane system configurations. ROSA software is a model designed by Nisan in 2005, applied to design and manipulate RO and NF systems taking into account effects of concentration polarization factor and hydrodynamic pressure drop in the concentrate-side on the membrane performance (Nisan et al., 2005). This software is often used in research work to simulate designs using reverse osmosis or nanofiltration plants (Yangali-Quintilla et al., 2010 ; Haryati et al., 2017).

The main inputs of the model include the amount of feed water and its chemical characteristics including the SDI range, feed water and concentrate feed pressures, temperature and pH. Then, setting up of the number of membranes, pressure vessels, type of membrane, and if needed booster pumps, is performed by ROSA. Depending upon the system, the goal is to minimize feed pressure and membrane costs whilst maximizing permeate quality and recovery. After performing calculations, ROSA produces a report predicting among others the water quality and flow rate.

Feed water whose characteristics were implemented in ROSA was the one used in previous studies (Azais et al., 2014; Azais et al. 2016 a), 2016 b), Azais et al., 2017). It is a real secondary effluent from a municipal WWTP located in the south of France (Baillargues) with a SDI=2.5 which is inferior to 3 as recommended for a direct use of the effluent as feed water for NF, without pretreatment. The membrane bioreactor (MBR) system incorporates immerged ZeeWeed 500 (GE Zenon) ultrafiltration 155 (UF) membranes in polyvinylidene difluoride (PVDF) which have a pore size of 0.035 microns. The 156 total membrane surface area is about 7 605 m<sup>2</sup> and produces approximately 1 500 m<sup>3</sup> of permeate per 157 day while operating at a mixed liquor total suspended solids (TSS) concentration of between 4 and 7 g 158 L<sup>-1</sup>. Summary of major constituents of the secondary effluent is presented in table 1 with intervals 159 given with 95% confidence.

160 161

Table 1. Water quality of the WWTP effluent.

Parameters	Units	WWTP effluent
pH	-	$7.69\pm0.31$
Conductivity	$mS \ cm^{-1}$	$1.30\pm0.22$
Alkalinity	mg eq $CaCO_3 L^{-1}$	$430\pm80$
COD	$\rm mg \ L^{\cdot 1}$	$18 \pm 3$
DOC	$\rm mg \ L^{\cdot 1}$	$6 \pm 2$
NO <sub>3</sub> <sup>-</sup> -N	$\rm mg \ L^{\cdot 1}$	$2.0\pm0.6$
Total Nitrogen (TN)	$\mathrm{mg}\ \mathrm{L}^{\text{-}1}$	$4.1\pm1.6$
$PO_4^{3}$ -P	$\mathrm{mg}\ \mathrm{L}^{\text{-}1}$	$0.11\pm0.08$
Na <sup>+</sup>	$\mathrm{mg}\ \mathrm{L}^{\text{-}1}$	$152 \pm 14$
K+	$\mathrm{mg}\ \mathrm{L}^{\text{-}1}$	$22 \pm 3$
$Ca^{2+}$	$\rm mg \ L^{\cdot 1}$	$160 \pm 20$
$\mathrm{Mg}^{2+}$	$\rm mg \ L^{\cdot 1}$	$6.6 \pm 0.8$
Cl	$\rm mg \ L^{\cdot 1}$	$267 \pm 35$
$\mathrm{SO}_4{}^{2\text{-}}$	$\rm mg \ L^{\cdot 1}$	$89 \pm 13$
Ionic strength	mM	$22 \pm 5$

162

No nitrites were detected in the effluent so that the value of TN could be due to the presence of ammonia because of incomplete nitrification. Regarding water pH and ionic composition, ROSA estimates Langelier Saturation Index (LSI) which is a measure for the saturation of CaCO<sub>3</sub> in water. The LSI should be used for low Total Dissolved Solids (TDS) ranges (<10 000 ppm). At the pH of saturation (pH<sub>s</sub>), the water is in equilibrium with CaCO<sub>3</sub>. To avoid calcium carbonate scaling by acid addition alone, the LSI in the concentrate stream must be negative. If a high quality scale inhibitor is 169 used, the LSI in the concentrate stream can be increase up to 1.5. This will reduce or eliminate the acid 170 consumption. Many inhibitors allow operation up to an LSI of < 1.8 in the concentrate. In this study 171 the water is hard, so acid (H<sub>2</sub>SO<sub>4</sub>) was added. Finally, the low value of SDI will reduce fouling 172 problems due to particulate matters which tends to accumulate at the membrane surface resulting in a 173 membrane flux decline.

174 Capital cost of a NF plant is directly impacted by the design of the system. This issue should be taken 175 into account into the system design as most companies try to reduce the capital cost. Moreover, 176 maintenance cost of NF system can be reduced because of lower propensity to fouling of elements 177 when correctly chosen. Thus, balance has to be found between design and maintenance costs to meet 178 both economic and technical requirements.

179 A two or three stage configuration with a decreasing number of pressure vessels in parallel allows respecting the minimum concentrate flow rate constraint and other required hydraulic conditions in 180 nanofiltration process. Consequently, this kind of configuration was chosen in this study in order to 181 reach high conversion rates (> 80 %), obtain a reliable permeate quality without a severe fouling risk 182 (figure 1). First stage is composed of N pressure vessels each including M elements in series. Next 183 184 stage has N/2 pressure vessels and last stage N/4 pressure vessels. In the experimental study, three Volume Reduction Factor (VRF) were tested: 2, 5 and 10 which correspond to recovery rate (Y, 185 186 defined as the ratio of permeate flow by feed flow) of about 50, 80 and 90 % respectively (Azais et al., 187 2016 a). To reach these recovery rates, number of stage has to be adapted. Y of 50 % was applied to 188 the first stage and Y of 60% was applied to second stage thus resulting in global Y of 80% for two stages. Y of 50% was applied on last stage resulting in final Y of 90 % close to VRF 10 which was 189 190 obtained at pilot scale (Azais et al., 2016b). To reach these conversion rates, ROSA adjusted pressure 191 at the entrance of each stage according to the number of stage and pressure vessels and type of 192 membrane.

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Figure 1. Configuration of the nanofiltration unit (number of pressure vessels: first stage N=4, second stage N/2, third stage N/4).

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The average flux of the entire system, i.e. the system permeate flow rate related to the total active membrane area of the system, is a characteristic parameter of a given design. The system flux is a useful number to quickly estimate the required number of elements for a new project. In this study, the feed water flow of Baillargues wastewater treatment plant ( $125 \text{ m}^3.\text{h}^{-1}$ ) was chosen as inlet feed flow in ROSA. Tested recovery rates (Y) were chosen as they correspond to VRF reached in a previous study (Azais et al., 2016 a)).

Among elements proposed by ROSA, NF 90-400 was first chosen. NF90-400 element is a high area, high productivity element designed to remove a high percentage of salts, nitrate, iron and organic compounds. The high active area membrane combined with high permeability of the membrane allows the removal of these compounds at low operating pressure. The membrane active area is 400 square feet; the diameter and length are respectively 7.9 and 40 inch. Then, same calculation were performed for NF 270-400 (same dimensions as NF 90-400) and for BW 30-400 (brackish water reverse osmosis membrane, same dimensions as NF 90-400).

Cost of NF 90-400 element and pressure vessel was estimated to respectively 850 and 1 500 \$. Price
of kilowatt-hour was based on current rate in France which is about 0.09\$.kWh<sup>-1</sup> in accordance with
Plumlee et al. (2014) (Plumlee et al., 2014). Moreover, annual membrane replacement rate was taken

equal to 10% as estimated by Yangali-Quintanila et al. (2010) (Yangali-Quintanila et al., 2010). To
calculate the annual capital costs, project lifetime was taken equal to 10 years (life time of membranes)
with an interest rate of 7%.

Finally, two uses of the treated water (permeate) were considered: irrigation according to the FAO recommendations and the aquifer recharge in agreement with the Groundwater Recharge Requirements proposed by the California Department of Public Health (CDPH). The standard values are presented in a previous study (Azais et al. 2014).

222

#### 223 2.2. Cost of ozonation

Economic evaluation of NF retentate ozonation is necessary so as to define an optimal conversion rate which assures a compromise between permeate flux and retentate treatability. This economic evaluation was done on the basis of transferred ozone dose in concentrated real wastewater which guarantees a 90% elimination of three pollutants among the four chosen in our previous studies (see Azais et al., 2016 a)).

229 As only little ozone doses are required to treat retentates of this study, on site oxygen generation using 230 Pressure Swing Absorption (PSA) was chosen. This is ideally suited to supply an ozone generator with medium frequency (60 to 1 000 Hz). Specific energy for ozone production was estimated to 10 231 kWh.kgO<sub>3</sub><sup>-1</sup> (Rosenfeldt et al., 2006). According to Margot et al. (2013), the transfer efficiency of 232 ozone into the dissolved phase in ozonation pilot plant is between 70 to over 90% depending on the 233 gas flow (Margot et al., 2013). In this study, it was fixed to 95%. Investment cost was taken equal to 234 100  $g_{3}^{-1}$ .h<sup>-1</sup> as suggested by ozone water treatment plants manufacturers and suppliers. The 235 236 operational cost was then evaluated in this study. Finally, as cooling water is used in a close loop system, this item of expenditure was neglected. 237

Considering the different input parameters (economic as well as process design and performance data) and their variability in the cost assessment, the maturity level is fixed to 5 (corresponding to 10 % of complete definition) with an accuracy range of -50 % to 50 % according to the AACE (American

Association of Cost Engineering) cost estimate classification matrix. A narrow accuracy range was defined since the approach developed in this study could easily lead to a feasibility study or even to a pre-budget authorization (maturity level 4 or 3) as the membrane and ozonation technologies are mature and controlled.

245

- 246 **3. Results and discussion**
- 247
- 248 *3.1. Optimization of nanofiltration design for NF-90*

249 To verify that the chosen configuration (figure 1) is well adapted to the targeted conversion rates, 250 simulations were done by imposing the three recovery rates for different configuration (one, two or 251 three stages). For this, value of the number of pressure vessel in first stage (N) was modified between 10 and 30. At first approximation, number of elements in a pressure vessel was fixed to 7 as usually 252 253 encountered at industrial scale (Van der Meer et al., 1998). For each simulation, total power to be supplied by the pumps was noticed so as to determine specific energy consumption (kWh/m<sup>3</sup>) of each 254 255 configuration. In addition, the rejection of total dissolved solids (TDS) was estimated as a function of 256 N. Figure 2 presents the results of these simulations.





Figure 2. Influence of pressure vessel number in the first stage (N) (number of elements M=7).

260 Regarding specific energy consumption, it appears that each recovery rate is adapted to a certain number of stages. A single stage system is more energy efficient when a conversion rate of 50% is 261 262 applied. Two stages are more energy efficient to reach Y=80% and three stages to reach Y=90%. 263 Indeed, at low conversion rate, the concentrate flow rate is high thus when using only one stage pressure drop along the unit is limited. On the contrary, for higher conversion rate (lower concentrate 264 flow rate), the decrease of hydraulic pressure due to the increase of stages is more interesting from an 265 266 energetic point of view. In the next part of the article, a conversion rate of 50%, 80% and 90% was thus reached with respectively one, two and three stages. On figure 2, it appears that specific energy 267 268 consumption decreases with increasing number of pressure vessels in the first stage. For example, feed pressure decreases from 4.6 to 2.1 bars when N increases from 10 to 30. Nonetheless, an increasing 269

number of pressure vessels will have a direct consequence on capital and maintenance cost. Moreover,
the decrease of inlet pressure leads to a decrease in TDS rejection because of high diffusive transfer.

Number of pressure vessels influences both TDS rejection and energy consumption. Thus, it has to be 272 optimized so as to minimize operating costs. Annual power cost (\$ year<sup>-1</sup>) was obtained by multiplying 273 specific energy consumption (kWh m<sup>-3</sup>) by the price per kilowatt hour (\$ kWh<sup>-1</sup>) and by annual 274 permeate flow (m<sup>3</sup> year<sup>-1</sup>). Maintenance costs were obtained by multiplying total number of elements 275 (M) by replacement rate of 10 % and by the price of one element. These maintenance costs were 276 calculated for each conversion rate (50, 80 and 90 %) by varying the number of pressure vessels in the 277 278 first stage, N, thus varying the total number of pressure vessels. Figure 3 presents operating costs as a 279 function of N and number of stages. Number of elements (M) in a pressure vessel was fixed to 7.



280

Figure 3. Influence of number of pressure vessel in the first stage (N) and number of stages on annual operating costs (number of elements M=7).

For each case, annual operating costs show a minimum for an optimum number of pressure vessel which is equal to 13 for Y=50% and Y=80% and 12 for Y=90%. In the rest of the study, N was taken equal to 12 so has to have 6 and 3 pressure vessels respectively in the second and third stage.

287 Concerning the number of elements per pressure vessel (M), van der Meer et al. (1998) showed that even though the classical system configuration of pressure vessels in RO and NF systems is a 288 289 multistage design with a number of membrane modules per pressure vessel of 6 up to 8, this design 290 trend results in high pressure losses because of high feed velocities and long feed/brine channel lengths (van der Meer et al., 1998). The authors showed that an increase in permeate productivity of 291 292 20% can be achieved by lowering the number of membrane modules per pressure vessel. In contrast to 293 the hydraulic optimum, they found an economic optimum between 3 to 4 elements per pressure vessel. Research of optimal number of elements M was conducted using same approach as for N. The 294 295 pressure vessel number was 12 in the first stage, 6 in the second stage and 3 in the last stage. M was varied between 2 and 8 (maximum authorized by ROSA). Then, operating costs were evaluated for 296 297 each configuration. Results are presented in figure 4.



298

Figure 4. Influence of number of elements (M) and stages on annual operating costs (total number of pressure vessels N=12).

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The economic optimum is M=6 elements for a single stage and M=5 for two and three stages which is in accordance with van der Meer et al. (1998) (van der Meer et al., 1998). At low number of membrane modules, the permeate flow rate decreases as a result of the increasing concentration 305 polarization in this region. This effect becomes more pronounced at higher recoveries, caused by 306 increasing osmotic pressure differences across the membrane surface, as a result of the increasing salt 307 concentrations at the feed/brine side of the membranes at high recoveries. In the rest of the paper, 308 number of elements (M) was taken equal to 6 for one stage and to 5 for two or three stages.

- 309 *3.2. Economic analysis of filtration*
- Table 2 presents the economic analysis of the process in term of final membrane capital cost (pressure
- vessel and membrane module) with one, two or three stages as shown on figure 1.
- 312 Table 2. Economic analysis of membrane capital cost for nanofiltration operation with NF90-400313 elements.

Targeted conversion rate	Y = 50 %	Y = :	80 %		Y = 90 %	
Stage	1	1	2	1	2	3
System details						
Feed flow (m <sup>3</sup> .h <sup>-1</sup> )	125	125	62.5	125	62.5	25
Permeate flow (m <sup>3</sup> .h <sup>-1</sup> )	62.5	62.5	37.5	62.5	37.5	12.5
Retentate flow (m <sup>3</sup> .h <sup>-1</sup> )	62.5	62.5	25	62.5	25	12.5
Recovery rate (%)	50	50	60	50	60	50
Total membrane area (m <sup>2</sup> )	2 676	3 3	344	3 902		
Average permeate flux (L.h <sup>-1</sup> .m <sup>-2</sup> )	23.4	29	9.9	28.8		
Feed pressure (bar)	5.04	5.55	7.52	5.55	7.52	6.75
Economic analysis						
Pressure vessel (quantity)	12	12	6	12	6	3
Pressure vessel cost (\$)	18 000	27	000		31 500	
Membrane module (quantity)	72	60	30	60	30	15
Membrane module cost (\$)	61 200	76	500		89 250	
Membrane capital cost (\$)	79 200	103	500		120 750	
Annual membrane capital cost (\$/year)	11 276	14	736		17 192	
Annual membrane capital cost (\$/m³)	0.020	0.0	016		0.017	

315 The economic analysis of the process shows that an optimum conversion rate of 80% leads to a membrane capital cost of 0.016 \$/m<sup>3</sup>. The increase in recovery rate enables a decrease in operating 316 317 cost per cubic meter of treated water. Nonetheless, this increase in conversion rate would result in an 318 increase in polarization concentration at membrane surface resulting in severe risk of scaling and a lower salt rejection. Indeed, TDS rejection decreases from 94.2% to 89.0% when increasing Y from 50 319 to 90%. However, this strategy avoids a too high demineralization of permeate water. In addition, 320 321 nitrate concentration in permeate for Y=90% is still inferior to the recommendation for groundwater 322 recharge (Bellona et al. 2012).

323 As mean value of LSI of feed water was 1.3 which is superior to the recommended value of 1, use of 324 acid was included in the economic evaluation of the process. ROSA proposes two agents (HCl, 325  $H_2SO_4$ ) which enable the LSI to be lowered close to zero value by modifying pH. In this study,  $H_2SO_4$ 326 was chosen as sulfate ions are retained by more than 90% by NF-90 membrane and thus did not 327 impact the permeate quality. The targeted LSI was 0.2 which required 140.5 mg of  $H_2SO_4$  per liter of 328 inlet effluent. This represented about 84 m<sup>3</sup> of  $H_2SO_4$  (96%) per year. As the cost of a cubic meter of 329 H<sub>2</sub>SO<sub>4</sub> is about 730 \$ thus annual expense for scaling control by acidification was about 61 000 \$ 330 (Table 3). Finally, acid cost was about four times operation and maintenance expenses. Proportion of 331 electrical cost slightly increases when increasing conversion rate (figure 5).

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Table 3. Economic analysis of operating and maintenance cost for nanofiltration operation with NF90400 elements at several conversion rates.

Targeted conversion rate	Y = 50 %	Y = 80 %	Y = 90 %
Stage	1	2	3
Specific consumption (kWh.m <sup>-3</sup> )	0.35	0.316	0.295
Electric cost (\$.year <sup>-1</sup> )	17 246	24 913	26 165
Membrane replacement (\$.year <sup>-1</sup> )	6 120	7 650	8 925
the second se			
Acid (\$.year <sup>-1</sup> )	61 000	61 000	61 000
	94.266	02 562	07.000
O&W cost (\$/year)	84 366	93 563	96 090
<b>O</b> & M aget (\$/m <sup>3</sup> nonmageta)	0.15	0.11	0.10
(\$/m°permeate)	0.15	0.11	0.10



341



Figure 5. Part of operating and maintenance costs for several conversion rates.

#### 343 *3.3. Comparison with loose-nanofiltration and reverse osmosis process*

To highlight the interest of using nanofiltration instead of reverse osmosis, same simulations were performed with NF-270, a loose NF membrane, and BW-30 a RO membrane (Dow Filmtec). Price of NF-270 and BW-30 elements were respectively 900 \$ and 680 \$ (Yangali-Quintanilla et al., 2010). Table 4 presents the comparison of these processes for Y=80%. As expected, use of RO enables capital cost saving of about 2-3 k\$ per year because of lower prices of RO equipment.

Table 4. Comparison of the membrane capital cost of nanofiltration and reverse osmosis for Y=80%.

Mombrana	Mombrane capital cast (\$)	Annual membrane capital	Annual membrane capital
Wendrane	Membrane capital cost (\$)	cost (\$/year)	cost (\$/m³)
NF90 - 400	103 500	14 736	0.016
NF 270 - 400	108 000	15 377	0.017
BW30 - 400	88 200	12 558	0.014

350

351 Table 5 presents the comparison of operating and maintenance cost of nanofiltration and reverse 352 osmosis processes. For high conversion rates, booster pumping may be required between stages. Operating and maintenance cost varies between 0.09 and 0.19 \$/m<sup>3</sup> depending on the conversion rate 353 354 and membrane type. These costs determined by ROSA are in accordance with literature concerning NF/RO systems (Cote et al., 2005; Englehardt et al., 2013). Globally the cost decreases when 355 356 increasing membrane permeability ( $L_p$ ) which is as follow in l/h/m<sup>2</sup>/bar (LMHB): Lp BW30-400 = 3.2 357 LMHB < NF90-400 = 10.8 LMHB < NF270-400 = 13.6 LMHB. For Y=80% and an inlet flow rate of 125 m<sup>3</sup>/h, 29.6 k<sup>\$</sup>/year and 35.2 k<sup>\$</sup>/year could be saved using respectively NF90-400 and NF270-400 358 instead of BW30-400. This is in accordance with other authors (Yangali-Quintanilla et al., 2011; 359 360 Bellona et al., 2012). Indeed, even if RO elements are less expensive than NF elements, energy requirement of the pumps was twice or third the one needed with NF processes which make operating 361 and maintenance costs of RO process higher. Finally, type of NF (loose or tight NF) does not seem to 362 363 influence considerably the process cost. Nonetheless, this influences performances and rejection rates 364 of the process as it can be seen in table 6.

# Table 5. Comparison of operating and maintenance cost of nanofiltration and reverse osmosis processes.

Targeted conversion rate	Y = 50 %	Y = 80 %			Y = 90 %	
NF270-4	00					
Feed pressure (bar)	3.57	3.91 4	4.77	3.91	4.77	3.89
Pumping power (kW)	16.97	23.87			24.41	
Specific consumption (kWh.m <sup>-3</sup> )	0.27	0.24			0.22	
Electric cost (\$.year-1)	13 379	18 819	)		19 245	
Membrane replacement (\$.year-1)	6 480	8 100			9 450	
Acid (\$.year <sup>-1</sup> )	61 000	61 000	)		61 000	
O&M cost (\$/year)	80 859	87 919	)		89 695	
O&M cost (\$.m <sup>-3</sup> )	0.12	0.10	I		0.09	
NF90-40	00					
Feed pressure (bar)	5.04	5.55 7	7.52	5.55	7.52	6.75
Specific consumption (kWh.m <sup>-3</sup> )	0.35	0.32			0.29	
Electric cost (\$.year-1)	17 246	24 913		26 165		
Membrane replacement (\$.year-1)	6 120	7 650			8 925	
Acid (\$.year <sup>-1</sup> )	61 000	61 000	)		61 000	
O&M cost (\$/year)	84 366	93 563	3 96 090		96 090	
O&M cost (\$.m <sup>-3</sup> )	0.15	0.11			0.10	<u>ا</u>
BW30-4	00					
Feed pressure (bar)	11.18	13.2 1	7.22	13.2	17.22	15.68
Specific consumption (kWh.m <sup>-3</sup> )	0.80	0.71			0.63	
Electric cost (\$.year-1)	39 428	28 56 071 56 0		56 071		
Membrane replacement (\$.year <sup>-1</sup> )	4 896	6 120 7 14		7 140		
Acid (\$.year-1)	61 000	000 61 000 61 000		61 000		
O&M cost (\$/year)	105 324	324         123 191         124 211				
O&M cost (\$.m <sup>-3</sup> )	0.19	0.14			0.13	<u> </u>

# Table 6. Comparison of performances and rejection rates of nanofiltration and reverse osmosis process.

Targeted conversion rate	Y = 50 %	Y =80 %	,		Y = 90 %	
Stage	1	1	2	1	2	3
	NF-270					
Feed pressure (bar)	3.57	3.91	4.77	3.91	4.77	3.89
Osmotic pressure (bar)	0.66	0.95			1.27	
Average permeate flux (L.h <sup>-1</sup> .m <sup>-2</sup> )	23.4	29.92			28.84	
TDS rejection (%)	62.5 ± 2.0	64.0 ± 1.5	70.0 ± 1.2	64.0 ± 1.5	70.0 ± 1.2	70.2 ± 0.1
Average permeate conductivity ( $\mu$ S.cm <sup>-1</sup> )	868.48	927.77			1037.4	
Permeate nitrate concentration (mg.L <sup>-1</sup> )	4.93	4.78	5.2	4.78	5.2	6.36
pH of permeate	6.15	6.2			6.28	
		NF-90				į
Feed pressure (bar)	5.04	5.55	7.52	5.55	7.52	6.75
Osmotic pressure (bar)	0.78	1.51			2.52	
Average permeate flux (L.h <sup>-1</sup> .m <sup>-2</sup> )	23.4	29.9			22.44	
TDS rejection (%)	91.8 ± 1.9	95.5 ± 0.4	96.3 ± 0.1	91.8 ± 1.8	92.0 ± 2.3	90.3 ± 6.6
Average permeate conductivity ( $\mu$ S.cm <sup>-1</sup> )	98.2	113			152.5	
Permeate nitrate concentration (mg.L <sup>-1</sup> )	3.40	3.23	3.52	3.13	3.52	4.01
pH of permeate	5.22	5.28			5.41	
		BW30				
Feed pressure (bar)	11.18	13.2	17.22	13.2	17.22	15.68
Osmotic pressure (bar)	0.8	1.53			3.02	
Average permeate flux (L.h <sup>-1</sup> .m <sup>-2</sup> )	23.37	29.9			29.21	
TDS rejection (%)	> 99	> 99			> 99	
Average permeate conductivity ( $\mu$ S.cm <sup>-1</sup> )	12.94	15.97			22.93	
Permeate nitrate concentration (mg.L <sup>-1</sup> )	0.76	0.66	1.38	0.66	1.38	3.94
pH of permeate	4.71	4.77			4.8	

Inlet pressure required to ensure the permeate flux strongly increases when using RO elements. BW30elements enable near-total rejection of ionic species except nitrate ion whose rejection is 91%. These

375 high rejection rates imply high hydraulic pressure almost twice the hydraulic pressure in NF. Thus, 376 excepted for direct potable reuse, this type of membrane is not recommended for water reuse for 377 groundwater recharge of other use that direct consumption. Rejection of monovalent ionic species with 378 NF-270 does not exceed 70 % which makes it unsuitable for water reuse projects. In fact, as demonstrated in a previous paper (Azais et al. 2014), the NF-270 membrane could produce a good 379 water quality according to the FAO irrigation standards for all of the analyzed parameters except for 380 the potassium. Concerning the aquifer recharge, the CDPH specifies that for replenishing a 381 groundwater basin, the effluent must not exceed 5 mg.L<sup>-1</sup> of total nitrogen and 0.5 mg.L<sup>-1</sup> of TOC for 382 projects where no dilution in the subsurface is occurring (reclaimed water contribution of 100%). As 383 presented in the Table 6, the major limitation of the NF-270 for water reuse is the poor rejection of 384 385 nitrogenous species for groundwater recharge. The nitrate concentration in the NF-270 permeate is too 386 close of the accepted limit to preconize this membrane for indirect potable water production. Besides, from previous laboratory tests, the NF-270 TOC permeate concentration marginally lower than the 387 standard of the CDPH could limit its application for water reclamation. 388

NF-90 permits high TDS rejection which is not impacted per recovery rates. Moreover, it was shown that nitrate concentration in the NF-90 permeate would enable its reuse in direct consumption or ground recharge. Thus, tight NF membranes seem to be well-suited for reuse water projects with strict usages which could be an alternative to the current RO schemes.

### 393 *3.4 Sensivity analysis of the cost of nanofiltration*

A sensibility analysis was performed in order to evaluate the impact on the total cost of slight cost variations of membrane equipment. Basic case was defined as the one previously presented with NF-90 and Y=80%. Several cases were tested: a 10% decrease of both elements and pressure vessels, a 10% increase of both elements and pressure vessels, a membrane replacement rate of 5% instead of 10%, a membrane replacement rate of 15% instead of 10%, and a mix of these conditions (table 7).

399

 

 Table 7. Effect of slight variations of membrane cost and replacement rates on the total cost of membrane operation.

Membrane capital cost (\$)	Annual membrane capital cost (\$/year)	Annual membrane replacement (\$/year)	∆\$/year
Basic case NF-90 Y=80%	14 736	7 650	0
Pressure vessels and elements - 10%	13 262	6 885	-2 239
Pressure vessels and elements + 10%	16 210	8 415	2 239
Annual membrane replacement rate 5%	14 736	3 825	-3 825
Annual membrane replacement rate 15%	14 736	11 475	3 825
Pressure vessels and elements + 10% and Annual membrane replacement rate 15%	16 210	12 622	6 446
Pressure vessels and elements - 10% and Annual membrane replacement rate 5%	13 262	3 443	-5 682

As it can be seen in table 7, variations of membrane equipment and/or membrane replacement rate can
result in a modification of annual cost from -5 682 to 6 446 \$/year in comparison with the basic case.
Thus, considering the project lifetime of ten years, total amount of the project can be modified by
around 60 000 \$ depending of current costs which is not negligible.

### *3.5. Economic evaluation of ozonation of NF retentates*

Economic evaluation of ozonation step is necessary so as to define the optimal conversion rate which ensures a compromise between permeate flux and treatability of the NF retentate. Cost of retentate ozonation was estimated for the three conversion rates of the study and compared to direct ozonation of the effluent. Results are presented in table 8.

	Secondary effluent	Y = 50 %	Y = 80 %	Y = 90 %
Inlet flowrate (m <sup>3</sup> .h <sup>-1</sup> )	125	62.5	25	12.5
Required transfered ozone dose (mgO <sub>3</sub> .L <sup>-1</sup> )	5.5 - 12.5	5.5 - 22.5	10.0 - 30.0	20.0 - 40.0
Required ozone production (kgO <sub>3</sub> .h <sup>-1</sup> )	0.7 – 1.6	0.4 - 1.5	0.3 - 0.8	0.3 - 0.5
<b>Required power (kW)</b>	11 - 16	5 - 15	4 - 8	3.9 - 5.3
Specific consumption (kWh.m <sup>-3</sup> )	0.09 - 0.13	0.09 - 0.24	0.16 - 0.32	0.32 - 0.42
O&M cost (\$.year <sup>-1</sup> )	8 558 - 12 967	4 273 – 11 670	3 112 - 9 336	3 112 - 4 150
Average O&M cost (\$.m <sup>-3</sup> )	0.010	0.014	0.028	0.033
Average capital cost (\$)	115 000	95 000	55 000	40 000
Average capital cost (\$.year <sup>-1</sup> )	19 600	16 200	9 400	6 800
Average capital cost (\$.m <sup>-3</sup> )	0.018	0.029	0.043	0.062

419 Table 8. Economic evaluation of ozonation step on secondary effluent for several conversion rates.

420

421 Cost of ozonation of secondary effluent in this study is in accordance with literature (Margot et al., 422 2013; Ni et al., 2013). Ozonation treatment would result in an increase of 30 % of the WWTP cost if 423 considering that mean cost of WWTP with MBR is 0.49 \$/ m<sup>3</sup> (Guo et al., 2014). Increase in 424 conversion rate lead to a decrease in operating and maintenance cost because of the reduction of 425 treated volume and thus of the power of ozone generator. Finally, Table 9 presents a cost analysis of 426 the coupling between ozonation and nanofiltration (NF90-400) for several conversion rates and 427 considering a reuse of both permeate and retentate.

428

429

Table 9. Cost analysis of coupling between ozonation and nanofiltration using NF90 - 400 with reuse
 of permeate and retentate.

Membrane	Y=50%	Y=80%	Y=90%	
Membrane CAPEX	0.010	0.013	0.016	
(\$/m <sup>3</sup> permeate)	0.010	0.015	0.010	
Ozonation CAPEA	0.014	0.008	0.006	
(\$/m <sup>3</sup> permeate)				
Total CAPEX (\$/m <sup>3</sup> permeate)	0.024	0.021	0.022	
Membrane OPEX (\$/m <sup>3</sup> permeate)	0.077	0.085	0.088	
$O_{-} \cdots (f_{-} O DEV (f_{-} 3 \cdots f_{-} f_{-}))$	0.007	0.007	0.007	
Ozonation OPEA (\$/m <sup>*</sup> permeate)	0.007	0.000	0.005	
Total OPEX (\$/m <sup>3</sup> permeate)	0.084	0.091	0.092	

433

NF/O<sub>3</sub> treatment of the effluent (without considering the benefit of the reuse) resulted in an increase of
almost 80% of the ozone process cost as demonstrated by Plumlee et al. (2014) (Plumlee et al., 2014).
Nonetheless, quality of the treated water is much more satisfying than for ozonation alone and enables
its reuse (Azais et al., 2016 b)). Indeed, ozonation alone does not enable elimination of ionic species
and a complete mineralization and thus does not satisfy actual water reuse standards.

In comparison with NF alone, coupling of NF and ozonation results in approximately same CAPEX (table 3). If considering the optimum conversion rate of 80%, which is usually encountered at industrial scale (Van der bruggen et al., 2013), in comparison with NF alone the coupling results in an increase capital costs of 0.005 \$/m<sup>3</sup>/year and a decrease of operating cost of 0.019 \$/m<sup>3</sup>/year. The cost saving due to the coupling between NF and ozonation is thus 0.014 \$/m<sup>3</sup>/year which means 15 330 \$/year for a flow rate of 125 m<sup>3</sup>/h. 445 If considering annual treated volumes with possible retentate recovery (thus increase of the annual treated volume), NF/O<sub>3</sub> process would thus be more economical than nanofiltration alone. It has 446 447 indeed the potential to be cost-competitive in some situations, especially when the recovery of 448 otherwise wasted water is considered. This is in accordance with other authors who concluded that costs associated with zero liquid discharge (concentrate treatment) may be partially offset by the 449 recovery (Khan et al., 2009; Umar et al., 2015). Finally, by taking into account economy of scale, 450 451 which is found for both capital, operating and maintenance costs (Guo et al., 2014), cost predicted in 452 the present study could be considerably decreased which contributed to make this process attractive 453 for wastewater reuse.

### 454 **4.** Conclusion

455 This study has put in evidence that ozonation of nanofiltration retentates would be an acceptable alternative solution from an economic point of view for wastewater reuse. It was shown that NF could 456 be used in lieu of RO as advanced treatment. NF is an effective barrier for organic contaminants which 457 is less expensive than RO because of lower O&M costs. Cost saving for a flow rate of 125 and Y=8 % 458 459 could be 35 k£/year when using nanofiltration instead of reverse osmosis. This may be enhanced in the 460 future if more demand of NF develops the market, meaning less capital cost when selecting NF 461 membranes instead of RO. Concerning the filtration step of the proposed advanced process, a three 462 stages configuration with a decreasing number of pressure vessels (N) in parallel - 12, 6 and 3 pressures vessels with 6 elements (M) on first stage and 5 elements on second and third stages - is 463 464 well-adapted. An optimum conversion rate of 80 % was found. Ozonation of nanofiltration retentates 465 enable a possible reuse of both permeate and retentate and thus can result in lower production costs. 466 For instance, an economy of 15.4 k\$/year of operating costs was obtained for a flow rate of 125 m<sup>3</sup>/h. 467 One can expect considerable cost saving for higher flow rates as usually encountered. Considering such a process will result in a reduction of greenhouse gas emission compared to conventional high-468 469 energy reuse processes based on the use of reverse osmosis.

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## Zero-liquid discharge process





Process optimization

Economic analysis