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Recovery of detergents in food industry: an industrial approach

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ABSTRACT

A Nanofiltration pilot plant (300 Da cut-off and 50 m^2 membrane surface) has been used *in situ* to recover a spent single-phase detergent (DEPTAL EVP®) in a yogurt industrial factory. The plant worked during 60 h at constant operation conditions previously selected in experiments at laboratory and pilot-plant scales. Membranes retained around 90% of the chemical oxygen demand and permeates were reused in the "cleaning in place" plant. Savings of detergents in the yogurt plant were estimated in 15–20%. The data obtained during the tests were used to do an economic evaluation of the feasibility of the plant under several hypotheses of volume concentration rates. Payback was estimated in 2.6 years.

Keywords: Nanofiltration; Detergents; Dairy; Cleaning-in-place

1. Introduction

The Food and Beverage industry consumes large amounts of water, which is involved in many processes and unit operations, including production and cleaning purposes [1]. In particular, the dairy industry is one of the most polluting food industries considering the generated effluent (0.2–10 L of effluent per litre of processed milk) [2].

The cleaning process plays an important role in this type of industry since it is essential to the maintenance of hygienic production requirements. Cleaning-in-place (CIP) technology is the commonly used system to perform this cleaning in a low cost, rapid and automatic manner. Furthermore, its application minimizes the amounts of water and commercial agents used in comparison with traditional manual cleaning processes.

The cleaning procedure is usually composed of acidic and caustic solutions passes, being required rinse water between them. Acidic solutions are used to remove inorganic soil while caustic ones prevent the deposition of organic compounds. Nowadays, single-phase solutions are being substituted for the aforementioned detergents in order to reduce these two time-consuming steps into a single unique passage through the soiled installation. However, the high prices of these novel cleaning solutions requires their recovery and reuse to avoid an increase in the cleaning process costs [3].

Membrane technology has been used successfully in the dairy industry to achieve three main purposes: the concentration of milk and whey constituents from waste streams [4–13], the production of new dairy products [12,13] and water reuse, including the

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recovery of cleaning solutions from CIP processes [2,3,9–11,13–25]. The membrane technology selected (microfiltration, ultrafiltration, nanofiltration (NF) and/or reverse osmosis) will depend on the required permeate quality. Food industry standards specify that spent process water intended for reuse, even for cleaning purposes, must be at least of drinking quality [26]. In the case of cleaning solution recovery, membranes permit reutilization of the cleaning agents and consequently confer three main advantages: (1) reduction in detergent and water consumption; (2) decrease in disposal cost and (3) smaller amounts of chemical agents discharged into the environment. In our previous work [27], the use of membranes in spent cleaning solution treatment and detailed information about feed, membranes and experimental conditions were reviewed.

The aim of this study was to evaluate the viability of a NF pilot plant installed in a Spanish dairy company that is fed with part of a spent single-phase detergent coming from the yogurt industrial area. The information obtained was used to scale up a future industrial plant.

2. Experimental

2.1. Materials and methods

The fresh commercial single-phase detergent DEPTAL EVP® (HYPREP, France) is diluted to a concentration of 2% w/w before to use in the CIP process of the yogurt plant. This cleaning procedure generates around 6,200 m^3/y of the depleted detergent solution. The automatic "cleaning in place" cycle consists of several steps: (1) a rinse with recycled water; (2) a passage with the single-phase cleaning solution and, (3) a rinse with clean tap water. The solution to be treated with NF is very heterogeneous due to the different varieties of yogurt produced in the industry. The conductivity of the spent detergent varied between 15.3 and 17.3 mS/cm, chemical oxygen demand (COD) between 1,200 and 2,300 mg/L and pH values between 12.2 and 12.6. The high alkalinity was due to the presence of sodium and potassium hydroxides in the commercial formulation. These compounds are also responsible of the high feed conductivity. Another component of the detergent is ethylenediaminetetraacetic sodium salt. However, the rest of ingredients are unknown. The surface tension was between 30 and 40 mN/m and the total dry extract was 1–2%. The same cleaner was also used in the CIP process of the reception facility, producing an annual waste of 13,900 m^3/y of the diluted solution. Table 1 compared the main measured analytical parameters of the commercial fresh detergent (DEPTAL EVP® diluted at 2% w/w) and spent detergent (feed solution of the NF plant).

Two commercial spiral wound SelRO MPS-34 80–40 (KOCH Membrane Systems, USA) NF membranes were used for the experiments. Their main characteristics are shown in Table 2.

A schematic diagram of the NF pilot plant is depicted in Fig. 1. The waste single-phase cleaning solution was pumped to a 10,000 L feed tank (T1). Two pumps (P1, low-pressure pump and P2, high-pressure pump) provided the required pressure and velocity to the feed solution to pass through the 50-μm filters PF1 and PF2 and then through the NF membranes. The latter were installed into the shell 1 (S1) in a serial way. The waste stream was fed at an intermediate flow rate recommended by membrane manufacturer (8 m^3/h) and at a constant inlet pressure of 1,216 kPa and an average temperature of 60°C. The permeate was sent to the CIP process to be reused in the yogurt plant. Membrane cleaning is an important operation of the pilot-plant maintenance. A rinse with tap water was performed alternate days. However, chemical cleaning was also required using caustic (NaOH solution of 0.1% w/w) and acidic (HNO_3 solution of 0.2% w/w) passages at 50°C during 1 h each one and then rinsing with tap water between them during 30 min.

The conductivity and pH of the feed and permeate samples were measured with a CRISON MM40 portable equipment (Crison, Barcelona, Spain). The COD was measured with standard test kits using a Spectroquant NOVA 60 spectrophotometer (Merck, Madrid, Spain).

The recovery rate (R) in the NF plant was defined as the ratios between the volumetric fluxes of permeate and feed. The volume concentration rate (VCR) is related with the recovery rate $VCR = 1/(1-R)$.

2.2. Economic assessment, background

The viability of the project was evaluated in terms of three parameters: net present value (NPV), internal rate of return (IRR) and payback period (n). The best project option requires a positive NPV, a high IRR and the shortest payback period.

The NPV represents the risk in a project and is defined as:

$$NPV = -I + \sum_{n=1}^{N} \frac{CF}{(1+r)^n} \quad (1)$$

Table 1
Analytical characterization of the fresh detergent (2% w/w), spent detergent (feed of NF plant) and permeate stream

Parameters	Commercial detergent	Spent detergent	Permeate stream
pH	13.11	12.2–12.6	12.2–12.6
Conductivity (mS/cm)	17.25	15.3–17.3	13.5–16.5
DQO (mg O_2/L)	2,985	1,200–2,300	100–300
Surface tension (mN/m)	35.6	30–40	30–40

Table 2
Membrane main characteristics

Material	Not available (proprietary composite)
MWCO, (Da)	300
Feed channel spacers, (mil)	30
Membrane area, (m^2)	25
Permeate flow[a], (m^3/h)	1.5
Glucose/sucrose rejection[a], (%)	95/97
NaCl rejection[a], (%)	35
Feed flow rate (m^3/h)	5.7–17.1
Maximum operating pressure, (kPa)	3,546
Maximum operating temperature, (°C)	70
pH operating range	0–14
Maximum pressure drop per element, (kPa)	71

[a]Test conditions: RO water at 3,039 kPa and 30°C. Feed solution for rejection tests: 3% glucose/3% sucrose or 5% NaCl.

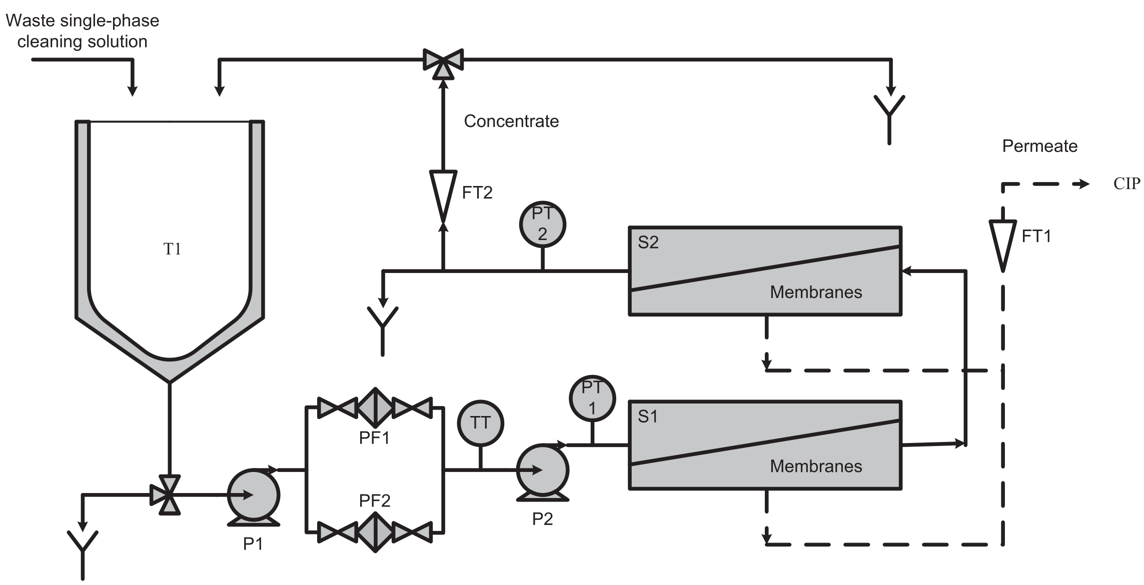

Fig. 1. NF plant scheme: T1, feed tank; P1, low-pressure pump; PF1 and PF2, pre-filters (50 μm); TT, temperature transmitter; P2, high-pressure pump; PT1 and PT2, pressure transmitters; S1 and S2, membranes shells; FT1 and FT2, flow meter for permeate and concentrate streams, respectively.

where *I* is inversion (including the cost of the plant and its set-up), *CF* is cash flow (€/y), *r* is the interest rate which the project is compared with and *N* is the amortization period of the inversion (*y*). The *IRR* is defined as the r value that converts the NPV to 0. The payback period indicates the time required to recover the investment and is calculated as the ratio between the inversion and the CF. This latter factor is determined as the difference between the total operating costs (€/m^3) and the savings obtained with the application of the NF plant for the recovery of the waste cleaning agents. The operation costs include the amortization (A), membrane replacement (MR), energy (E), maintenance, chemicals for membrane cleaning and concentrate disposal costs. The equations used to calculate the terms A, MR and E were based on the model proposed by Sethi and Wiesner [28]:

$$A = \frac{r \cdot (1+r)^N}{(1+r)^N - 1} \cdot \frac{I}{C \cdot \frac{\text{Work days}}{y} \cdot \frac{\text{Work hours}}{d}} \tag{2}$$

where A is the amortization (€/m^3) and C is the plant capacity (m^3/h).

The "cleaning in place" process in the industrial plant was carried out daily (excluding weekends). Thus, the treatment of spent cleaning solution was on the basis of 240 d per year. According to this operating scenario, the membrane replacement cost (MR, €/m^3) was estimated as:

$$MR = \frac{A_m \cdot MC \cdot \frac{i}{(i+1)^{ML} - 1}}{C \cdot \frac{\text{Work days}}{y} \cdot \frac{\text{Work hours}}{d}} \tag{3}$$

where A_m is the membrane area (m^2), MC is the membrane cost (€/m^2), i is the discount rate for MR and ML is the membrane lifetime (y).

The power demand (kWh/m^3) was calculated from Eq. (4):

$$\text{Power demand} = \frac{\frac{(Q_f P_f) + (Q_R P_R)}{\eta}}{C} \tag{4}$$

where Q_f and Q_R are feed and recirculate flux, respectively. P_f and P_R are the corresponding pressures of these streams and η is the pump efficiency. Energy costs (E) were calculated with the data provided by the company (0.1018 €/kWh).

The maintenance costs were assumed to be 1.5% of the initial non-membrane capital cost stated by Owen et al. [29].

The chemical costs related to the membrane cleaning were calculated considering the required volume of the cleaning solution (0.4 m^3), the concentration of the caustic (0.1% w/w) and acidic (0.2% w/w) solutions and the periodicity of the membrane cleaning procedure.

The concentrate disposal costs were estimated from the unitary price of the disposal (€/m^3) and the amount of concentrate generated per year. On the other hand, the savings were calculated as the sum of the fresh single-phase detergent, tap water and concentrate disposal cost savings.

3. Results and discussion

3.1. Membrane characterization

The characterization of the membrane was carried out with tap water from the dairy industry. This water shows a range of conductivity between 0.05 and 0.5 mS/cm. The membrane water flux vs. transmembrane pressure (ΔP) is shown in Fig. 2. The water flux measured with the new membranes was, as expected, linearly pressure dependent. The permeate flow rate was lower when the membrane was fed with the spent cleaning solution due to the presence of dissolved soil after the cleaning of the yogurt plant. However, the relationship between the permeate flux and the applied transmembrane pressure maintained linearity. Thus, no polarization concentration effect was observed, at least at the maximum transmembrane pressure used (1,216 kPa). The permeate flux at the highest pressure applied in the experiments with the spent cleaning solution at 1,216 kPa was 59.6 L/hm^2, which was 24.4% less than the values obtained with clean water. The presence of solutes in this feed justifies the lower slope in comparison with the pure water slope.

3.2. Pilot-plant operation

The operation in the plant was carried out during 60 h. Some representative data obtained during the

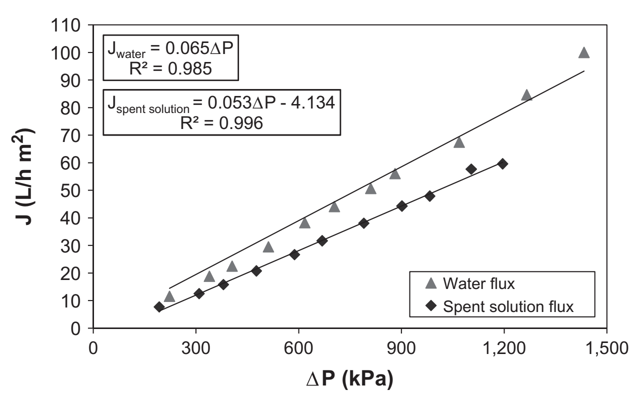

Fig. 2. Permeate flux (J) vs. transmembrane pressure (ΔP). Flow rate = 8 m^3/h, T = 60°C, total recycle mode.

recovery of the waste solution from the yogurt cleaning process during one month are shown in Fig. 3. The variation in the permeate flux (J) with the feed COD and conductivity is presented in Fig. 3(a), respectively. The membrane retention expressed in terms of COD and conductivity is plotted against the COD and conductivity measurements of the feed streams and is shown in Fig. 3(b), respectively. During the experiments, the whole permeate stream was cycled back to the yogurt CIP while the concentrate was recirculated to the NF plant.

The spent cleaning solution that fed the NF plant consisted of a mixture of soil compounds from the cleaning step (lactose, fats…) as well as chelating and surfactant agents and caustic compounds that come from the fresh detergent itself. Thus, the variation in the feed parameters (COD and conductivity values) that can be observed in Fig. 3(a) is attributed to the different yogurt flavours (chocolate, fruits, natural, etc.) manufactured. The membrane permeate flux did not show a clear trend with the feed COD or conductivity, being always between 60 and 100 L/hm^2 (Fig. 3(a)). The COD retention was always around 90% for the COD feed range of 1,400–1,900 mg/L. On the other hand, the conductivity rejection was below 25% (Fig. 3(b)) due to the poor membrane retention of small ions (OH^- and H^+). These results reveal that the components that provide conductivity to the cleaning solution passed preferentially through the membrane, since the components with high COD values were retained in the concentrate stream. The measured parameters of permeate stream are presented in Table 1 to compare with fresh and spent detergent. On the other hand, previous experiments have shown high rejection of ethylenediaminetetraacetic sodium salt at concentrations similar to those found in this commercial detergent due to the membrane/solute interaction mechanism [30]. The rejection of other low molecular weight charged solutes could be explained in the same way, considering the screening of membrane charge and electrostatic repulsions at different solute concentrations [8,31,32]. Finally, negligible differences were found in the pH values measured in the feed and permeate samples (12.2–12.6). This result means that the cleaner caustic ingredients (NaOH and KOH) freely passed through the membrane, which is desirable due to their effectiveness in removing inorganic soils from surfaces and equipment. It is also remarkable that several soil compounds show cleaning effects, since alkaline hydrolysis of milk protein and fats has surfactant properties [17,19–21]. Thus, their presence in the permeate stream could improve the cleaning process compared with the same process with fresh DEPTAL EVP® solution.

The frequency of the membranes cleaning in the economic evaluation was established according to the flux tendency observed in Fig. 4. The fouling effect is attributed to the deposition of solutes onto the

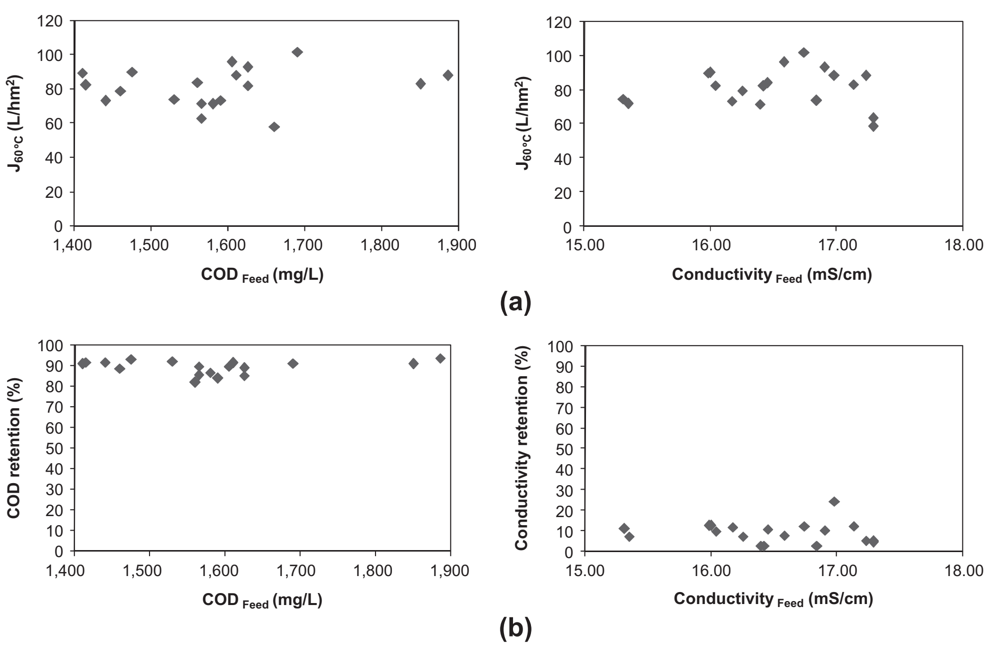

Fig. 3. (a) Permeate flux (J) vs. feed COD and conductivity measurements, (b) COD and conductivity retention vs. feed COD and conductivity measurements. Flow rate = 8 m^3/h, ΔP = 1,216 kPa, T = 60°C.

membrane surface, promoting the increase of filtration resistance and thus, a decrease of permeate flux with time.

In spite of the fluctuation of flux, due to the variety of the yogurt production and the different soil composition, a 30% of decrease is observed after 15 h of operation. Then, a rinse with tap water every two days and a chemical cleaning with NaOH and HNO_3 solutions twice a month was performed to avoid membranes fouling.

Additional experiments have been carried out to study the effect of the recovery rate on the membrane permeability. A pilot-scale plant provided with a spiral wound SelRO MPS-34 25-40 (KOCH Membrane Systems, USA) NF membrane of 300 Da and area of 1.4 m^2 was employed for these experiments. As can be seen in Fig. 5, the decrease in the permeate flux with the increase in the recovery value was not very marked. The correlation obtained permitted the estimation of the permeate flux at different recovery values in order to be used in the economic evaluation.

When permeates obtained in the pilot plant were sent to the yogurt CIP, a reduction in the fresh detergent consumption was observed, as can be seen in Fig. 6. The average value of fresh detergent consumption was around 11,000 L/month and this value decreased to 9,000 L/month due to the permeate reuse. The efficiency of the "cleaning in place" rig was not negatively affected by the mix fresh/reused DEPTAL EVP® and the cleaning step conditions (time, temperature and detergent concentration) were maintained constant. Thus, the savings of fresh DEPTAL EVP® were estimated around 18%.

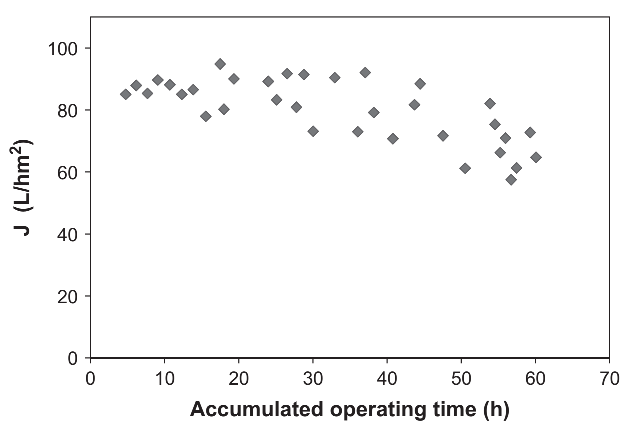

Fig. 4. Permeate flux (J) vs. operating time. Flow rate = 8 m^3/h, ΔP = 1,216 kPa, T = 60˚C.

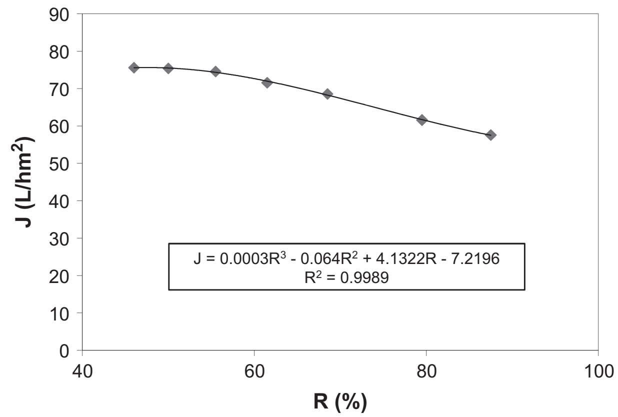

Fig. 5. Permeate flux (J) vs. recovery (R), ΔP = 1,216 kPa, T = 60˚C.

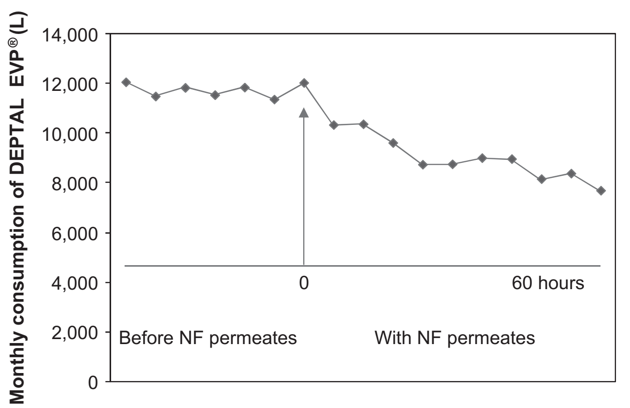

Fig. 6. Evolution of fresh DEPTAL EVP® consumption.

3.3. Economic assessment

The basis of the economic assessment was established to treat 20,000 m^3/y of the depleted detergent solution enough to treat all the DEPTAL EVP® actually used in the company and the experimental device was adapted to supply the pressure drops through the membrane elements and feed rate to 16.1 m^3/h. The economic evaluation was done using nine different *VCRs* as hypothesis:

(1) A *VCR* value was set (Table 3).
(2) The recovery was calculated as $R = 1-(1/VCR)$.
(3) Permeate flux was estimated with the correlation presented in Fig. 5 (Table 3).
(4) The required membrane area was determined as $A_m = R{\cdot}C/J$ (Table 3).
(5) The rest of the parameters were calculated using Eqs. (1)–(4) (Table 4).

Table 3
Basic assumptions and calculations for the economic evaluation of DEPTAL EVP® recovery

Column	a	b	c	d	e	f	g	h	i
VCR	2	3	4	5	6	8	12	16	20
Plant capacity (*C*), m^3/h					8				
Permeate flux (*J*), L/hm^2	76.9	72.7	69.3	67.4	66.3	65.3	64.9	64.9	64.9
Membrane area (A_m), m^2	52	74	87	96	101	108	114	117	118
Recovery (*R*), %	50	66.7	75	80	83.3	87.5	91.7	93.8	95
Feed pressure (P_f), kPa					1,216				
Feed temperature, ℃					60				
Membranes lifetime (*ML*)[b], y					3				
Pump efficiency (η), %					90				
Power demand, kWh/m^3	0.46	0.50	0.52	0.53	0.54	0.56	0.57	0.57	0.57
Energy cost [a], €/kWh					0.1018				
Amortization period (*N*), y					15				
Interest rate (*r*), %					7				
Membrane replacement cost (*MC*), €/m^2					120				
Discount rate for *MR* (*i*), %					7				
Work days (d/y)					240				
Work hours (h/d)					10				
DEPTAL EVP®costs[a], €/m^3					912				
Tap water costs[a], €/m^3					0.2859				
Disposal costs[a], €/m^3					1.591				
Capital costs[a] (*I*), €					150,000				

[a]Data supplied by the company.
[b]KOCH membrane systems recommendation for this type of application.

Table 3 resumes the basic assumptions and calculations and *NPV*, *IRR* and payback period values estimated from the operating costs and savings are presented in Table 4. The results in both tables are shown from column *a* to column *i* for the different *VCR* values.

Some of the parameters used for the economical estimation were based on the characteristics of the plant (such as pump efficiency, power demand and plant capacity) and the experimental conditions studied (such as feed pressure and temperature). Others values were fixed according to information found in specialized literature (interest rate and maintenance costs) and other were obtained from membrane companies (the membrane's lifetime for this particular application and chemical agents for membrane cleaning).

The parameter studied in Tables 3 and 4 was the *VCR*. It was varied between 2 (50% of spent detergent recovery) and 20 (95% of spent detergent recovery). The permeate flux, the required membrane area, the recovery and the power demand parameters showed different values depending on the selected *VCR*, as can be seen in Table 3. The costs that remained invariant with the *VCR* in Table 4 were those that correspond to amortization, maintenance and chemicals for membrane cleaning (same cleaning frequency was assumed for all *VCRs*).

As can be seen from Tables 3 and 4, the increase in membrane area reduces the total operating cost and increases the total savings. This effect leads to the desired higher values of *NPV* and *IRR* and lower values for the payback periods. The evolution of the payback period, the membrane area and the permeate flux vs. the *VCR* is shown in Fig. 7. As expected, higher *VCR* values require larger membrane areas. However, the required membrane area does not increase proportionally with the *VCR*, following an asymptotic behaviour: the membrane area increases 33.3% when the *VCR* goes from 3 to 5; meanwhile, an increase in the *VCR* from 6 to 8 only requires a 10.6% increase in membrane area. On the other hand, as the permeate flux decreases as the *VCR* increases, the optimal point should be a compromise between the two effects. The variation in permeate flux also follows a negative asymptotic trend, due to the low pollutant charge of the feed. Therefore, the payback period should be considered in selecting the appropriate membrane area. The asymptotic declining tendency in these data is also observed. An increase in the *VCR* from 3 to 5 produces a 14.6% decrease in the payback period. However, an increase from 6 to 8 only leads to a decrease in the payback period of 2.7%.

According to the Fig. 7 and Tables 3 and 4, optimum estimated recovery value was 82% that gives a

Table 4
Operating costs, savings and economic parameters for the economic evaluation of DEPTAL EVP® recovery

Column	*a*	*b*	*c*	*d*	*e*	*f*	*g*	*h*	*i*
Operating costs									
Amortization (*A*), €/m³					0.850				
Membrane replacement (*MR*), €/m³	0.101	0.143	0.168	0.185	0.195	0.208	0.220	0.225	0.227
Energy (*E*), €/m³	0.047	0.050	0.053	0.054	0.055	0.057	0.058	0.058	0.058
Maintenance, €/m³					0.112				
Chemicals[a], €/m³ (membrane cleaning, twice a month)					0.0012186				
Concentrate disposal, €/m³	0.796	0.530	0.398	0.318	0.265	0.159	0.133	0.099	0.080
Total operating costs, €/m³	1.907	1.687	1.583	1.521	1.480	1.388	1.374	1.346	1.329
Savings									
DEPTAL EVP® saved, m³/y	38	50	57	61	63	66	69	71	72
DEPTAL EVP® costs saved, €	34,499	45,999	51,749	55,199	57,499	60,374	63,249	64,686	65,549
Tap water costs saved, €	2,769	3,692	4,153	4,430	4,614	4,845	5,076	5,191	5,260
Disposal costs saved, €	15,407	20,543	23,111	24,652	25,679	26,963	28,247	28,889	29,274
Total savings, €/m³	2.720	3.626	4.080	4.352	4.533	4.759	4.986	5.099	5.167
Net present value (*NPV*), €	−6,604	192,099	290,473	349,341	388,604	444,822	487,269	512,145	527,128
Internal rate of return (*IRR*), %	6.30	24.05	31.72	36.19	39.15	43.34	46.49	48.34	49.44
Payback period (*n*), y	9.53	3.99	3.10	2.74	2.54	2.30	2.14	2.06	2.02

[a]Membrane cleaning carried out twice a month with the aqueous solutions: 0.4 m³ of caustic solution at 0.1% w/w (0.369 €/kg_{NaOH}), 0.4 m³ of acidic solution at 0.2% w/w (0.33 €/kg_{HNO3}). Rinse with tap water was established alternate days with 0.4 m³.

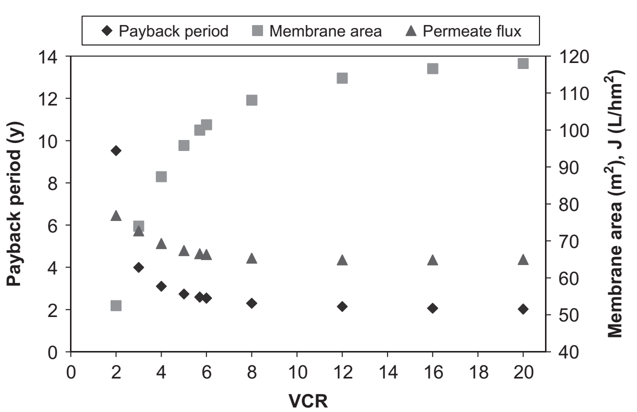

Fig. 7. Payback period, membrane area and permeate flux vs. *VCR*.

payback period of 2.6 years. For these conditions, the calculated membrane area was 100 m² (four spiral wound membranes of 25 m² each).

In the present work, an environmental impact study was not considered. However, a decrease in detergent consumption was demonstrated (Fig. 6) with a consequent reduced discharge of chemical products into the environment. Furthermore, the reuse of the permeate stream led to a large reduction in water consumption with important savings (15,970 m³/y for the recovery value 82%).

The total operating costs for the designed NF plant with the aforementioned membrane area was 1.490 €/m³. This value is higher than the operating cost previously reported in other studies (between 0.100 and 0.970 €/m³) [23,33–37]. However, it is important to remark that the comparison between the economic data is difficult due to the variety in water quality treated and different plant sizes and membranes. In general, operating costs decrease as plant capacity increases; If the sterilizers facilities of the industry were cleaned with DEPTAL EVP®, expanding the working time to 16 h per day and 330 d per year, the operating cost would be 0.845 €/m³. The payback period would also decrease to 1.7 years and a large increase in the *IRR* value would be observed (57.8%).

4. Conclusions

Feasibility of reusing a real single-phase commercial detergent (DEPTAL EVP®) has been demonstrated using a NF spiral wound membrane (SelRO MPS-34, 300 Da cut-off, 50 m² membrane area). Transmembrane pressure (1,216 kPa), temperature (60˚C) and feed flow (8 m³/h) were maintained constant during the pilot-plant tests. Permeates reused in the CIP of industrial plant did not reduce the cleaning efficiency and the fresh detergent savings were around 18%.

An economic approach was made to treat all the depleted DEPTAL EVP® detergent actually generated in the industry (20,000 m^3/y) under different *VCR* hypothesis (from 2 to 20). The total operating cost and total savings were strongly affected by this value, with the costs tending to decrease and the savings tending to increase as the *VCR* increased. The final selected *VCR* (5.7), based on the asymptotic decrease in the return of investment period, led to satisfactory results for the proposed NF plant with a total membrane area of 100 m^2 and a permeate flux of 66.6 L/hm^2. The total operating cost (1.490 €/m^3) and the total savings (4.485 €/m^3) allowed a payback period less than 2.6 years. Furthermore, a great important reduction in water consumption was achieved (15,970 m^3/y).

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