Separation and Purification Technology 119 (2013) 19–27

Contents lists available at ScienceDirect

Separation and Purification Technology

journal homepage: www.elsevier.com/locate/seppur

A continuous nanofiltration + evaporation process for high strength rubber wastewater treatment and water reuse

Gang Xin [a,*], Mafalda P. Lopes [b], João G. Crespo [b], Bjørn Rusten [a]

[a] Aquateam COWI AS, P.O. Box 6875, Rodeløkka, N-0504 Oslo, Norway
[b] CQFB/REQUIMTE, Department of Chemistry, FCT, Universidade Nova de Lisboa, P-2829-516 Caparica, Portugal

ARTICLE INFO

Article history:
Received 2 June 2013
Received in revised form 23 August 2013
Accepted 26 August 2013
Available online 5 September 2013

Keywords:
Rubber demolding wastewater
Nanofiltration
Evaporation
Water reuse
Energy saving

ABSTRACT

In the rubber parts industry, the treatment of wastewater from the demolding process utilising polymeric agents, traditionally poses a challenge. This is due to the high strength (COD > 10,000 mg/L) and the low biodegradability of the main organic content in the wastewater. Typically, a thermal process of boilers followed by evaporators is commonly employed to treat the wastewater. Besides frequent boiler maintenance due to severe fouling caused by the nature of the wastewater, high energy costs are nearly prohibitive. At Cikautxo, a Spanish rubber parts manufacturer, the energy costs are in the region of €4.2/m^3 treated wastewater.

Following an initial lab scale membrane screening, a pilot study was carried out at Cikautxo in 2012. Nanofiltration using a NF270 membrane was tested and the reject stream was collected and used to feed a full size evaporator.

Without pretreatment, the rate of the nanofiltration process was maintained at a permeate flux of 11 L/m^2-h for periods of over 30 h in between flux recovery cleaning. The use of the NF process gave a significantly improved feed water quality, consequently improving the total capacity of the evaporator when compared with the use of the boiler blow down method. The NF + evaporator solution was shown to effectively reduce energy costs by 55% (from €4.2/m^3 to €1.9/m^3 treated wastewater).

1. Introduction

The treatment of wastewater from the demolding process in rubber parts manufacturing presents a challenge. In particular, when utilising polymeric demolding agents such as Getren™, (a polyether and non-ionic surfactant). This is due to its high strength (COD > 10,000 mg/L) and low biodegradability [1] of the main organic content in the wastewater. Membrane filtration was proposed as a preferred treatment technology in two technology screening studies [2,3].

Cikautxo is a major rubber parts manufacturer in the Basque Country in Spain. The factory produces approximately 70 m^3/d of wastewater from the rubber manufacturing process, including molding, autoclaving and demolding. A polymeric release agent, Getren™, is used in the process and ends up in the wastewater. This polymeric release agent contributes to the majority of the chemical oxygen demand (COD) in the wastewater. Currently, a thermal process is employed to treat wastewater at Cikautxo, where boilers followed by evaporators are used for wastewater treatment [4]. Besides frequent boiler maintenance due to severe fouling caused by the wastewater, an energy cost of approximately €4.2/m^3 treated wastewater is nearly prohibitive.

In an initial lab scale study [5] both ultrafiltration (UF) and nanofiltration (NF) were tested for treating the rubber wastewater from Cikautxo. Although UF allowed relatively high water fluxes, the rejection of contaminants in the wastewater in terms of COD and TOC (total organic carbon) was clearly insufficient to comply with the European legislation (the Directive 96/61/EC) for effluent discharges. Permeate generated from nanofiltration using either a NF90 or a NF270 module (Dow, Midland, Michigan, USA) can meet the effluent discharge requirement (<2000 mg COD/l). For the large scale study the NF270 membrane was chosen due to its higher membrane permeability [6–8]. In order to accomplish maximize reuse of the wastewater, an existing evaporator at Cikautxo was tested for further volume reduction of the membrane reject stream.

Two modes of operation with the use of NF technology are used: continuous mode and concentration mode. Continuous mode operation continuously provides a reject stream is typically used in full scale membrane applications [9]. However, in processes where the flow rates are small and discontinuous, the concentration mode is often used [10]. The reject is sent back to the NF feed tank in the concentration mode operation.

* Corresponding author. Tel.: +47 22358100; fax: +47 22358110.
E-mail address: gang.xin@aquateam.no (G. Xin).

1383-5866/$ - see front matter
http://dx.doi.org/10.1016/j.seppur.2013.08.036

A pilot study was carried out in 2012 at Cikautxo, in which the continuous mode operation was used. The treatment results from the concentration mode study are described in another paper [5]. This paper describes the operational and economic challenges of the NF270 process and the integration with the evaporation process. The pilot study had the following objectives:

- Maintaining a competitive permeate flux with a minimal cleaning frequency.
- Meeting the industrial water reuse standards or discharge standards.
- Achieving the desired evaporator performance using a membrane concentrated feed stream.
- Minimizing energy input to the integrated system.
- Achieving at least 99% water recovery.
- Minimizing the amount of waste for off-site treatment.

2. Materials and methods

2.1. The pilot system

The pilot process involved the integration of nanofiltration and evaporation technologies for rubber wastewater treatment. A NF pilot and a full scale evaporator were used in the study. Fig. 1 shows the process diagram. The NF pilot and evaporator were equipped with programmable logic controllers (PLCs) for data acquisition and to facilitate automation and control. From the NF pilot, the following parameters were recorded by the PLC: date, time, pressure after feed pump, pressure after pressure pump, retentate pressure, retentate flow, permeate flow, reject flow, pressure pump frequency and permeate temperature. From the evaporator, the following parameters were recorded by the PLC: date, time, pressure in water drum, temperature in water drum, steam flow and steam pressure. An electricity meter was installed in the electrical cabinet of the NF pilot. The electricity consumption by all of the components in the pilot, including the feed pump, the pressure pump, electrical valves, sensors, touch screen, etc., was logged by the electricity meter.

2.2. The membrane

Two NF270-4040 modules (active membrane area of 7.6 m^2 per module) were used during the pilot study. Both membrane modules used went through a compaction process with tap water at 12–13 bar for at least 3 h before treating any wastewater. A magnesium sulphate rejection test was performed for one of the modules. The test results showed a 98.2% rejection of magnesium sulphate at 4.8 bar pressure and a 27% water recovery. This indicated adequate membrane performance and module integrity. Polypropylene (PP) cartridge filters (1 µm pore size) were placed up-stream of the NF270 module for removing larger particles from the wastewater.

2.3. NF operation

The NF pilot was operated in continuous mode, with the permeate flux was being an independent variable. This was preset in the PLC by the operators. The TMP (trans-membrane pressure) was a dependent variable, resulting from the preset permeate flux and membrane permeability. Flushing to allow the removal of accumulated material on the membrane surface was employed in the NF operation. During a flushing cycle the pressure pump was turned off for a preset duration with the feed pump on. At the same time, a bypass electrical valve in the reject line was opened by the PLC. Flushing differs from backwashing which typically employs permeate to backwash the membrane from the permeate side. The flushing operation allows to release high pressure in the NF. Consequently, part of the retentate in the system is replaced by feed water and high flow of retentate during the pressure release will create high shear forces that clean the membrane surface periodically.

2.4. The evaporator

The evaporator used in the pilot study is a full size drum type evaporator with a design capacity of 5 m^3 per day with the boiler blowdown. The evaporator is always operated in a batch mode. In each batch the evaporator starts with an empty drum. A feed

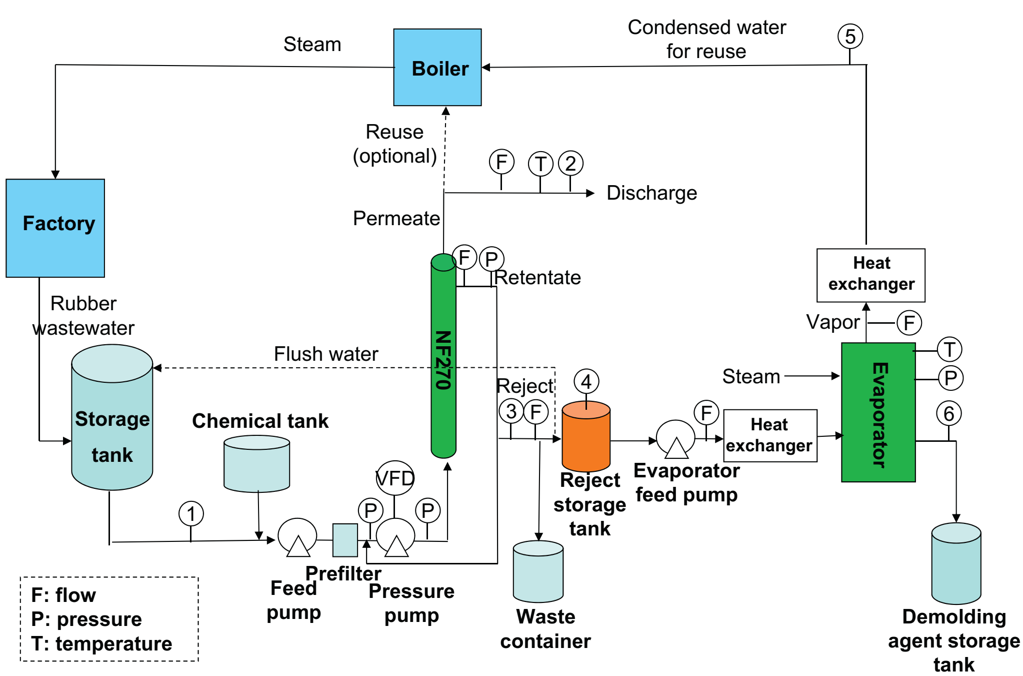

Fig. 1. Schematic process diagram of the pilot system at Cikautxo. Sensors for flow, pressure and temperature are labelled as F, P and T, respectively. Sampling locations are numbered. VFD: variable frequency drive.

pump is used to feed the drum and the pump is controlled by a level switch that is installed inside the drum. It is believed that the evaporator achieves the best performance in terms of distillate flow rates when the water level in the drum is slightly higher than half of the drum height and varies within a small range. The evaporator is periodically emptied for discharging residue (from the NF process) to the demolding agent storage tank when a certain amount of wastewater has been processed by the evaporator. Before discharging the residue a drying step is performed. Feeding does not commence during drying. The absence of distillate from the evaporator indicates the end of the drying step.

2.5. Analytical methods

During the period of operation, feed, membrane permeate and membrane reject were analyzed for total COD, oil and grease, viscosity, total suspended solids (TSS), total volatile solids (VSS), metals, pH, conductivity and turbidity. Hach Lange kits were used for the analyses of TOC (LCK385 and 386) and COD (LCK 014, 414 and 514). The amount of oil and grease was measured by a gravity method. The water samples were passed over a solid phase extraction (SPE) column in which oil and grease was retained by a solid absorbent. This was later eluted with *n*-hexane. The *n*-hexane was allowed to evaporate and the residue corresponded to the concentration of oil and grease in the original sample (Uriker internal test procedure: PEN/COA-024). Viscosity was measured using a viscometer (Brookfield, DV-I Prime). TSS and VSS measurements followed the standard methods with 1.2 μm pore size glassfiber filters. Inductively coupled plasma atomic emission spectrophotometry (ICP–AES) equipped with a radio-frequency (RF) generator of 40.68 MHz, a Czerny–Turner type monochromator with 1.00 m (sequential), an AS500 autosampler and data acquisition software was employed to determine the concentration of metals.

2.6. Membrane autopsy

Membrane autopsy was conducted with the first NF270 module after it was removed from the pilot unit in April 2012. The membrane module was opened and unrolled for visual inspection. A sample of fouled membrane was removed and a cleaning protocol was performed with a bench scale cross flow filtration unit (GE Infrastructure Water & Process Technologies). Membrane surface and membrane cross sections were examined using Field Emission Scanning Electronic Microscopy (Jeol JSM – 7001F).

3. Results and discussion

3.1. Wastewater characteristics

The rubber wastewater is a type of high strength wastewater with relatively low conductivity. Temperatures of the wastewater were between 35 and 45 °C at the end of the pipe to the wastewater storage tank. Table 1 summarises the main characteristics of the raw wastewater.

The strength of the wastewater varied significantly, from <5000 mg/L COD to >22,000 mg/L COD, during the pilot period. The COD:TOC ratio was relatively stable, indicating no change in the main COD contributor during the test period.

3.2. Results from continuous mode membrane study

Selected test results are shown in Fig. 2.

3.2.1. Permeate flux

A wide range of permeate fluxes were tested during this study. At relatively high permeate fluxes TMP rose quickly and the membrane permeability dropped quickly. An example is shown in Fig. 2A where a permeate flux of 23.6 L/m^2-h was maintained. When membrane permeability was below 2 L/m^2-h-bar, recovery cleanings needed to be executed to recover membrane permeability. However, more sustainable membrane performance was observed at lower permeate fluxes imposed (Fig. 2B). Table 2 summarizes the results of three continuous tests. At an average flux of 11.3 L/m^2-h an operation time of 33.3 h was maintained before the membrane permeability dropped below 2 L/m^2-h-bar during the 20th–22nd of April. A similar performance was also observed on the 23rd and the 27th of April. This can be explained by the "critical flux" phenomenon. At high permeate fluxes strong concentration polarization and high compaction of the deposit layer (cake layer) lead to rapid membrane fouling and subsequently rapid increases in TMP [11]. Therefore, a permeate flux below 12 L/m^2-h was recommended to obtain a sustainable membrane performance when treating the rubber wastewater at Cikautxo.

3.2.2. Water recovery

A wide range of water recovery, namely 70–93%, was tested during this study. Due to the limitation of the flow meter measurement range, the reject stream flow rates were recorded as 0 L/min when the actual flow rates were lower than 0.25 L/min. This meant that the highest water recovery that could be correctly recorded was 85% when the permeate flow was at 1.42 L/min (11.2 L/m^2-h) or 90% when permeate flow was at 2.25 L/min (17.8 L/m^2-h). At the beginning of the study high water recoveries (>85%) were used when high fluxes were tested. Later, only moderate water recoveries (78–83%) were used due to lower permeate flux imposed. It was found that a low water recovery of 70% did not aid in the maintenance of a stable membrane performance when the permeate flux was high (23.5 L/m^2-h). In addition a high water recovery (>85%) did not induce rapid membrane fouling when a low flux was imposed (27th of April, Fig. 2B).

3.2.3. COD rejection

The first NF270 membrane had over 90% COD rejection on the rubber wastewater in the first two weeks (the 7th–22nd of March), but the COD rejection went down to below 80% and the permeate COD went up to over 2000 mg/L after the 11th of April (Fig. 3A). The reduced COD rejection was probably caused by intensive chemical cleanings employed in this study. Details about the chemical cleaning protocols are discussed in Section 3.2.7. The strength of chemical cleaning was reduced for the second membrane. The COD rejection for the second membrane was over 95% during the entire testing period (Fig. 3B). It has been concluded

Table 1
Characteristics of wastewater at Cikautxo.

Parameter	COD	TOC	COD/TOC ratio	TSS	VSS	VSS/TSS ratio	O&G	pH	Cond.	Turb.
Unit	mg/L	mg/L		mg/L	mg/L		mg/L		μS/cm	NTU
Average	14,829	4420	3.6	54	49	0.92	15.1	6.4	766	44
Standard deviation	6269	1520	0.2	17	15	0.05	11.7	0.8	192	15
Number of Samples	42	17	17	20	20	20	2	34	35	26

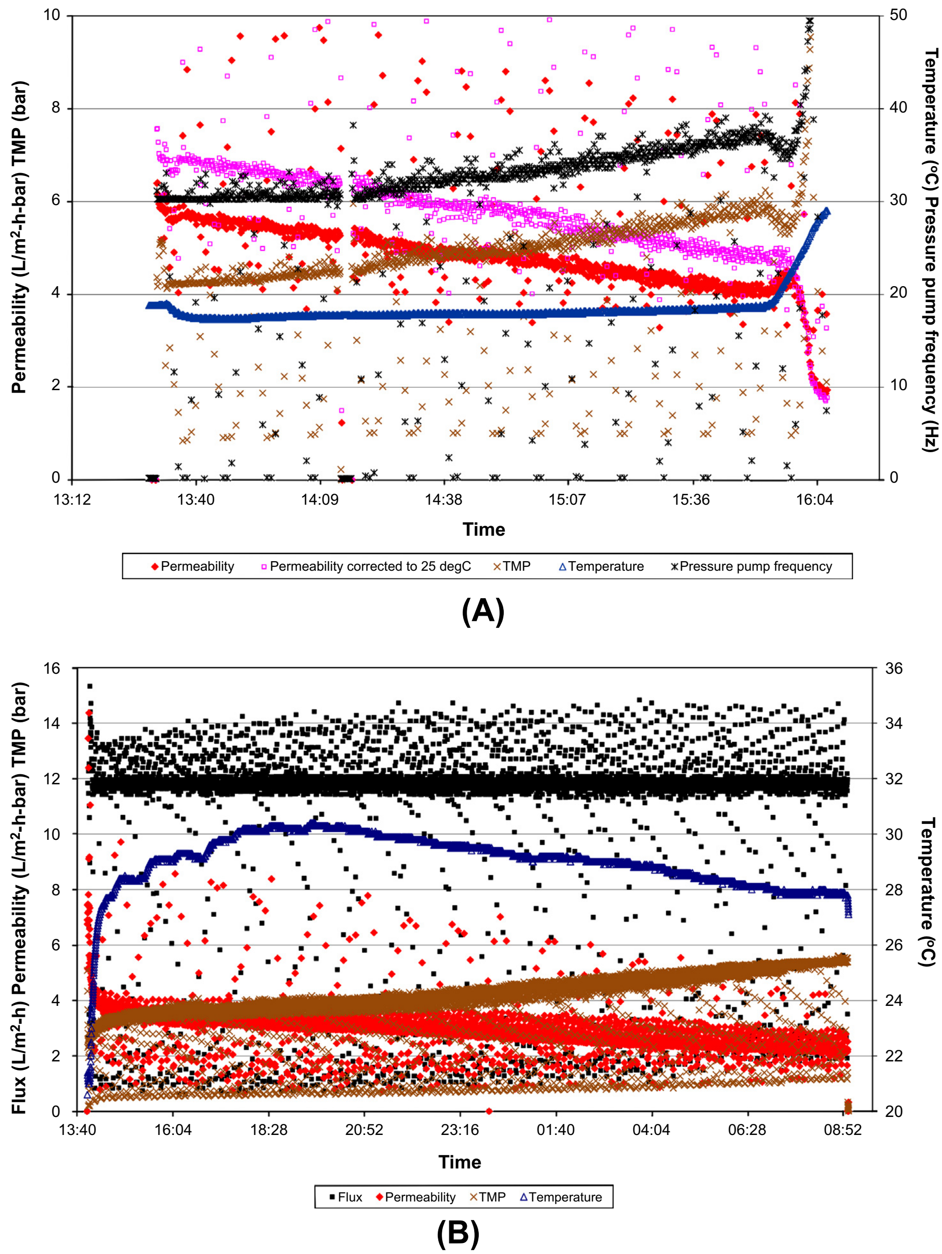

Fig. 2. Flux, TMP, permeability and temperature for the study performed on the 22nd of March where flux was set at 23.6 L/m^2-h (lmh) (A) and the 27th–28th of April (B).

that high COD rejection can be achieved with the NF 270 membrane, however, this can be significantly reduced by frequent use of high strength chemical cleanings.

3.2.4. Can NF permeate be reused as boiler feed water?

Table 3 summarizes the quality of the NF permeate collected in this study and compares it with the boiler feed water requirements. With the exception of total hardness, the characteristics of the NF permeate meet the boiler feed water requirements. Therefore, it can be concluded that the NF270 permeate can be used as boiler feed water if the total hardness in the permeate is further reduced. Cikautxo is considering using a softener to reduce total hardness in NF permeate and to reuse permeate as process water in future full scale applications.

3.2.5. Flushing

Flushing is an important operation to maintain membrane permeability [10]. Cleaning effects of the flushing were not significant with long time intervals. With short time intervals it was found that flushing for 5 seconds in every minute (5 s/min) was the optimal setting for maintaining stable membrane permeability without reducing much of the membrane filtration time. The flushing procedure did not lower the water recovery since the flush water was sent back to the feed tank. It is believed that the flushing had two major benefits for sustaining the membrane performance: (1) cleaning of the membrane surface at high flush water velocity and (2) relaxing membrane by temporarily lowering TMP.

Subsequently, all tests employed a 5 s/min flushing. Due to the membrane operation down times (shutdown of the pressure pump) introduced by flushing and frequent (every 5–10 seconds)

Table 2
Summary of the NF pilot results, including calculated energy consumption data, for the tests with rubber wastewater as the feed water.

Test	Date	Operating hours	Average TMP (bar)	Average flux (L/m^2-h)	Permeate produced (m^3)	Water recovery (%)	Energy consumption (kWh)	Hourly consumption (kW)	Consumption per m^3 permeate produced (kWh)
1	20–22 April	33.3	2.4	11.3	2.88	82.4	29	0.85	9.8
2	23–24 April	27.2	2.7	11.0	2.28	79.2	22	0.82	9.8
3	27–28 April	19.2	3.9	11.1	1.61	>85[a]	17	0.88	10.5

[a] Due to the limitation of the flow meter measurement range, no data were recorded when the flow rates of the reject stream were lower than 0.25 L/min.

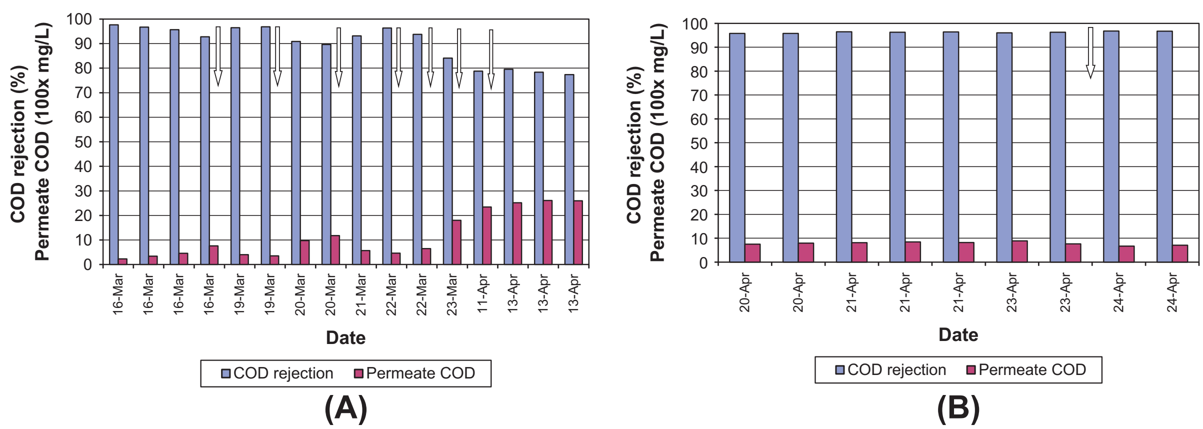

Fig. 3. COD rejection and permeate COD values for the first membrane (A) and the second membrane (B) with the rubber wastewater as feed water. White arrows indicate when chemical cleanings were carried out.

Table 3
Quality of the NF permeate vs. the boiler feed water requirements.

Elements	Unit	Permeate 11 March	Permeate 16 March	Permeate 16 March	Boiler feed water requirements [12]
Conductivity	µs/cm	562	352	375	<6000
Total hardness	mg/L as $CaCO_3$	24	40	37	<0.5
Si	mg/L as SiO_2	3.88	4.06	3.93	<150

recording intervals by the PLC, the PLC data for flux and TMP appear scattered, which can be seen in Fig. 2.

3.2.6. Temperature effects

In surface water treatment and most wastewater treatment applications, higher temperatures improve membrane permeability due to lower water viscosity [13] and lower degrees of concentration polarization at the membrane surface associated with higher temperatures [14]. Similar temperature effects were also observed in this study when feed water temperatures rose from 19 °C to 23 °C during the test on the 22nd of March as shown in Fig. 2A. It was also shown that membrane permeability quickly dropped once the feed water temperature was 24 °C or higher in several tests (one of the test results is shown in Fig. 2A, where the permeate flux was 23.6 L/m^2-h). When the water level in the feed tank was low, the level switch opened the feed valve and warm wastewater started flowing into the feed tank at 15:53, which raised the feed water temperature from 19 °C to 28 °C within 14 min. In the first 5 min the membrane permeability improved due to lower water viscosity and lower degrees of concentration polarization at higher water temperatures. However, the membrane permeability quickly dropped when the feed water temperature rose from 24 °C to 28 °C. This suggests that the feed water temperatures, when higher than 24 °C, play an important role in the drop of the membrane permeability. However, the temperature effect becomes insignificant when the permeate fluxes were set at 11–12 L/m^2-h (Fig. 2B). It is not fully understood as to why the high temperatures caused the catastrophic drop of the membrane permeability at high membrane fluxes.

3.2.7. Chemical cleaning

Two commercial cleaning chemicals, Ultrasil 11 and Ultrasil 75 (both from EcoLab) were used in this study. The alkali cleaning with Ultrasil 11 mainly targets the organic foulants on the membrane and the acid cleaning with Ultrasil 75 mainly targets the inorganic foulants on the membrane.

The two new membranes had membrane permeability of 5.89 and 6.26 L/m^2-h-bar, respectively. With the first membrane 0.5% Ultrasil 11 was used for alkali cleaning. A 60 min long cleaning recovered over 90% of membrane permeability, while a 30 min long cleaning only recovered approximately 50% of membrane permeability. Occasionally, higher than new membrane permeability was observed for the membrane after a 60 min long Ultrasil 11 cleaning, which indicates that the 0.5% Ultrasil solution may be damaging the membrane. The decreasing COD rejection as shown in Fig. 3A also suggests a damaged membrane. Therefore, 0.2% Ultrasil 11 was used for cleaning the second membrane, which was found to be able to recover approximately 60% of the membrane permeability. Therefore, it was recommended to use 0.2% Ultrasil 11 cleaning on a routine basis and 0.5% Ultrasil 11 cleaning

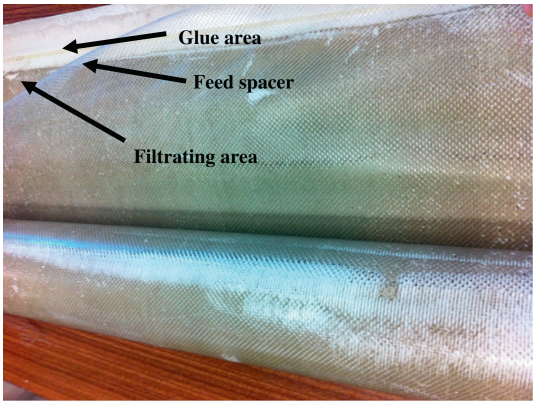

Fig. 4. Inside image of the 1st NF270 module.

when an in depth membrane recovery cleaning was required. The cleaning protocol, mainly in terms of chemical composition, needs to be improved to enhance the membrane permeability recovery and prolong the membrane lifetime.

The acid cleaning with Ultrasil 75 did not show much permeability improvement over the tested membrane, indicating insignificant inorganic fouling.

3.3. Membrane autopsy and fouling mechanism

A membrane autopsy was conducted with the first NF270 module after it was removed from the pilot unit. The module was cut open and samples were taken for scanning electronic microscopy. Fig. 4 shows an image of the open NF270 module, where a cake layer made of organics and solids had formed on the surface of the membrane.

Fig. 5 shows a cross section and surface SEM images of the NF270. A dense cake layer can be seen in both images. Portions of the cake layer were scrubbed off of the membrane and used for total solids (TS) and volatile solids (VS) analyses. A ratio of VS/TS of 82% was obtained, indicating that organic materials make up the majority of the cake layer on the membrane. The major inorganic elements in the cake layer were silica, iron and copper indicated by the ICP results shown in Table 4.

Aggregation and deposition of rejected hydrophilic polymers, a type of colloid, together with rejected salts, oil droplets and fine solids at the membrane surface forms a thin cake layer. On the top of the cake layer there is a polymer polarization layer where concentration polarization occurs. The solute (in this case the polymers) back diffusion may be hindered in the presence of the cake layer, thus elevating the membrane surface solute concentrations and resulting in higher TMP [15]. that the authors conclude that synergistic effects of the concentration polarization and the cake layer, the so called cake-enhanced concentration polarization by Hoek and Elimelech [16], accelerated the fouling of the NF270 membrane during the treatment of the rubber wastewater.

Table 4
ICP analytical results for inorganic contents in the layer scraped off from membrane surface.

Element	Al	Ba	Ca	Cu	Fe	K	Mg	Mn	Na	Si
Mass (mg/m^2)	0.1	0.8	8	68.2	102	39	6	0.2	0.6	193

Table 5
Summary of three evaporator test results.

Test	Date	Feed water	Amount of feed water (L)	Test duration (h)	Amount of residue (L)	COD of residue (g/L)
1	26 April	NF reject	600	3.4	13	1550
2	27 April	Boiler blowdown	600	4.0	40	1126
3	04 May	NF reject	1000	6.3	28	1683

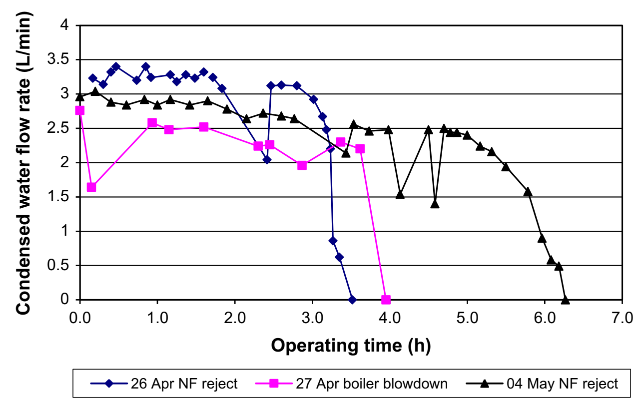

Fig. 6. Condensed distillate flow rate profiles for the three evaporator tests.

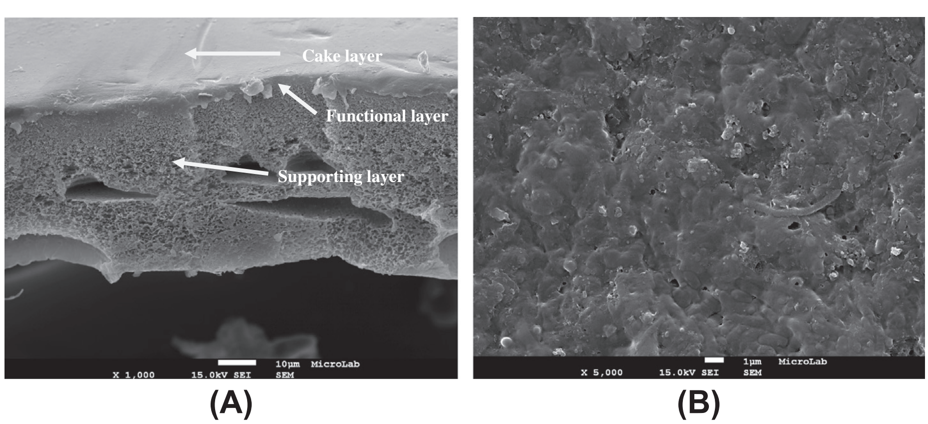

Fig. 5. SEM image of the first NF270. SEM image of cross-section (A) and membrane surface (B).

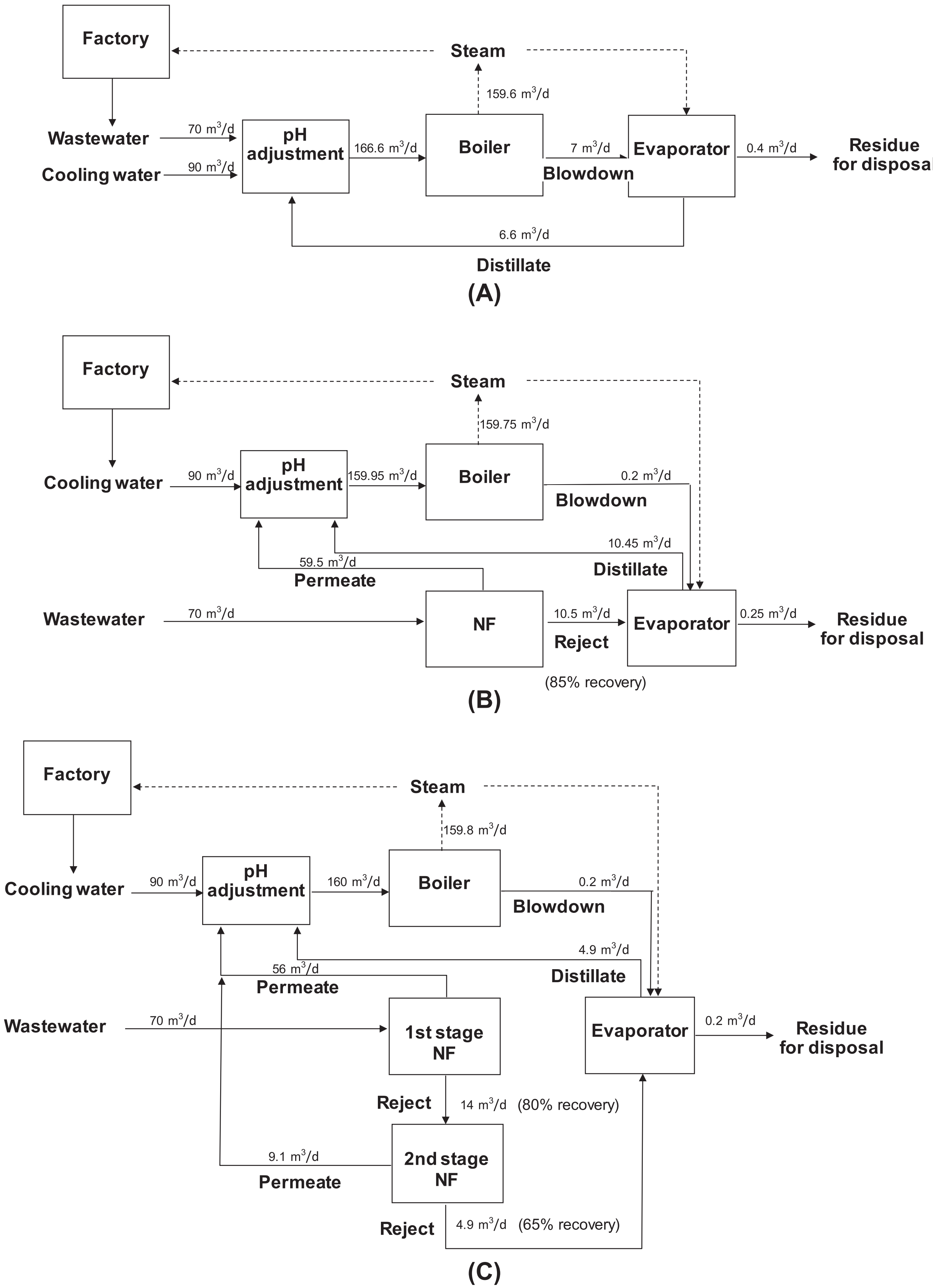

Fig. 7. Mass balance of the existing process (A), the single stage NF + evaporator (B) and the two-stage NF + evaporator design (C).

3.4. Evaporator testing results

Several tests were carried out in the full scale evaporator at Cikautxo with the NF270 reject as the feed water. One test with boiler blowdown was also conducted to draw the baseline for evaluating the evaporator performance with the NF270 reject. Results from three evaporator tests are summarized in Table 5 and Fig. 6. The two tests with NF reject showed higher distillate flow rates than for the test with boiler blowdown (Fig. 6). Distillate conductivity was lower for NF reject than for blower blowdown and the residue concentration was higher. These results indicate that the evaporator performance was slightly improved with the NF reject. But it should be noticed that the boiler feed water was pH adjusted and contained sodium hydroxide which is used for pH adjustment at Cikautxo. Addition of sodium hydroxide raised the salt content in the boilder blowdown, which may deteriorate the evaporator performance.

3.5. Economic analysis for preliminary full-scale plant design

3.5.1. Energy consumption

Table 2 summarizes all the key parameters in three tests with controlled permeate flux. The energy consumption rates in the three tests were similar because the NF was operated at similar permeate fluxes and TMPs during the tests.

At Cikautxo the volume of wastewater produced daily is around 70 m^3, which is reduced to a volume of 7 m^3 in the boiler with the simultaneous production of 63 m^3 of treated water. The 7 m^3 of concentrate are further processed in the evaporator to a final concentrate with a volume of 300 L. The expenditure of energy in the boiler has been determined to be 627.5 kWh per m^3 of blowdown produced. Considering a cost of 0.0303 €/kWh at Cikautxo this results in a boiler energy cost of €19/m^3 of blowdown produced. The energy consumption of the evaporator was determined to be €23/m^3 of feed.

Compared to the high evaporator energy consumption, the energy consumption of NF270 was a very small fraction (<2%) of the total energy consumption in a NF + evaporator plant. It would further reduce the overall energy costs of the treatment plant if the NF produces less reject water for the evaporator. Therefore, a second stage of NF270 is recommended for treating the reject stream from the first stage NF270. If a permeate flux of 7.5 L/m^2-h and a water recovery of 65% for the second stage NF are assumed, the two-stage design, with a 23% larger membrane area, could further reduce the overall reject flow by 65% compared to the single stage design.

3.5.2. Mass balances

Overall water recovery is defined in Eq. (1): Overall water recovery

$$= \frac{\text{cooling water flow} + \text{wastewater flow} - \text{residue flow}}{\text{cooling waterflow} + \text{wastewater flow}} \times 100\% \quad (1)$$

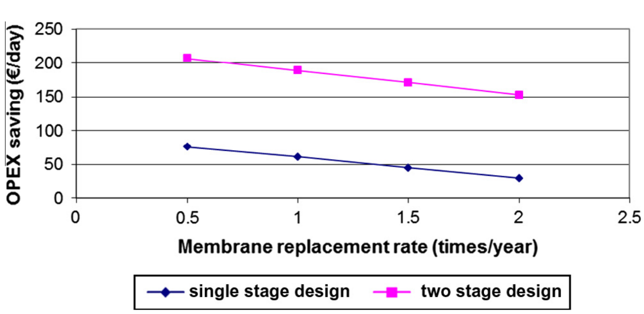

Fig. 8. OPEX savings at different membrane replacement rates (85% water recovery is assumed for the single stage design; 80% and 65% water recoveries are assumed for the first and second stages in the two-stage design).

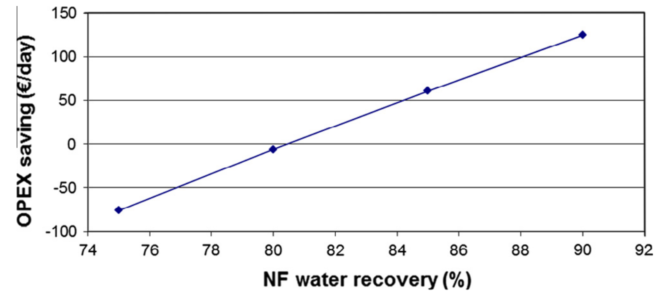

Fig. 9. OPEX savings at different water recoveries for the single stage NF design (the membrane replacement rate is assumed once per year).

Table 6
OPEX savings at different water recoveries for the two-stage NF design (the membrane replacement rate is assumed once per year).

First stage water recovery (%)	Second stage water recovery (%)	OPEX saving (€/day)
75	60	161
75	65	170
75	70	173
75	75	165
80	60	188
80	65	189
80	70	183
80	75	162
85	60	212
85	65	205
85	70	187
85	75	151
90	60	233
90	65	217
90	70	185
90	75	130

Mass balances shown in Fig. 7 indicates over 99% overall water recoveries for all the existing process, the single stage NF + evaporator design, and the two-stage NF + evaporator design, with the two-stage NF + evaporator on the top (99.9%).

3.5.3. Cost comparisons

In order to compare economics between the existing process, the single stage and the two-stage designs, a simple model was developed in this study. The existing process and the two alternative processes were compared in terms of capital investment, energy consumption, membrane replacement cost, chemical cost and off-site disposal cost. Total operational expenditure (OPEX) savings were estimated for the two alternatives compared to the existing process, and simple payback periods were also calculated. Results from two sensitivity analyses with varied membrane replacement rates or membrane water recoveries are shown in Figs. 8 and 9 and Table 6.

The cost model indicates that the two stage NF design would lead to more operational savings for the integrated solution than the single stage NF design. Periods of return on investment for the single stage NF design and the two stage NF design are expected to be 1641 days and 648 days, respectively, if a membrane replacement rate of once per year is assumed. Currently the electricity cost of 0.0303 €/kWh at Cikautxo is relatively low and will probably rise in the future. This also means that the operational savings will increase with higher electricity prices. Consequently this will shorten the simple payback period. Therefore, the cost data favors the two stage NF design for full scale applications.

4. Conclusions

A solution combining membrane filtration and evaporation was developed in the PROMETHEUS project aiming to treat high strength, polymer containing rubber part manufacturing wastewater. A pilot study was carried out in early 2012 at a rubber factory in Spain. The following conclusions have been drawn from the pilot study:

- The rubber wastewater is featured with high COD, low conductivity, moderate solids concentration and moderate oil and grease concentration.
- The NF270 membrane can reject over 95% of COD and the NF270 permeate can be either discharged or reused as boiler feed water, if the total hardness in the permeate is further reduced.
- The membrane can be operated continuously at a controlled permeate flux for over 24 h if the permeate flux is maintained at no more than 12 L/m^2-h.
- A water recovery of 80–90% can be achieved by the NF270 as long as the permeate flux is set below 12 L/m^2-h.
- Frequent flushing, typically flushing for 5 seconds in every minute, can significantly help to sustain membrane performance.
- The concentration polarization enhanced by a cake layer built up with the polymeric demolding agents is believed to be the main mechanism for membrane fouling.
- Alkali cleaning with Ultrasil 11 is more effective than acid cleaning with Ultrasil 75. It is recommended to use 0.2% Ultrasil 11 for daily cleaning and use 0.5% Ultrasil cleaning for in-depth periodical cleaning.
- A slightly higher treating capacity was observed for the evaporator when treating the NF reject compared to treating the boiler blowdown, probably due to lower salt concentrations in the NF reject.
- A two stage NF design can significantly reduce the overall energy costs of the integrated solution. The two stage NF design will lead to higher operational savings for the integrated solution when compared to the single stage NF design. The two stage NF design is recommended for future full scale applications.

Acknowledgements

This work was accomplished within project PROMETHEUS, a research project partly funded by the European Commission under the 7th Framework Programme – Contract Number: 262184-1. The authors acknowledge Cikautxo S Coop (Basque Country, Spain) and Ibon Ormaetxea for their assistance during pilot studies at Cikautxo. The authors also thank Ocelie Kjønnø and Carsten Ulrich Schwermer, both from Aquateam, for their support on sample analysis, and Dr. Anthony J. Dinning, also from Aquateam, for his review on the manuscript. The authors also would like to thank Tecnologías Avanzadas Inspiralia S.L. (Madrid, Spain) and Carlos González for the development of the data acquisition system and automation and control system of the pilot plant.

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