Membrane process treatment for greywater recycling: investigations on direct tubular nanofiltration

F. Hourlier, A. Massé, P. Jaouen, A. Lakel, C. Gérente, C. Faur and P. Le Cloirec

ABSTRACT

On-site greywater recycling and reuse is one of the main ways to reduce potable water requirement in urban areas. Direct membrane filtration is a promising technology to recycle greywater on-site. This study aimed at selecting a tubular nanofiltration (NF) membrane and its operating conditions in order to treat and reuse greywater in buildings. To do so, a synthetic greywater (SGW) was reconstituted in order to conduct experiments on a reproducible effluent. Then, three PCI NF membranes (AFC30, AFC40 and AFC80) having distinct molecular weight cut-offs were tested to recycle this SGW with a constant concentration at 25°C at two different transmembrane pressures (20 and 35 bar). The best results were obtained with AFC80 at 35 bar: the flux was close to 50 L m^{-2} h^{-1}, retentions of 95% for chemical oxygen demand and anionic surfactants were observed, and no *Enterococcus* were detected in the permeate. The performances of AFC80 were also evaluated on a real greywater: fluxes and retentions were similar to those observed on SGW. These results demonstrate the effectiveness of direct nanofiltration to recycle and reuse greywater.

Key words | on-site greywater recycling and reuse, synthetic greywater, tubular nanofiltration membrane

F. Hourlier (corresponding author)
A. Lakel
CSTB,
11 rue Henri Picherit, BP 82341,
44323 Nantes Cedex 03,
France
E-mail: *fanny.hourlier@cstb.fr*; *abdel.lakel@cstb.fr*

F. Hourlier
A. Massé
P. Jaouen
GEPEA UMR CNRS 6144,
CRTT, 37 bvd de l'Université, BP 406,
44602 St-Nazaire Cedex,
France
E-mail: *anthony.masse@univ-nantes.fr*; *pascal.jaouen@univ-nantes.fr*

C. Gérente
EMN, GEPEA UMR CNRS 6144,
4, rue Alfred Kastler, BP 20722,
44307 Nantes Cedex 3,
France
E-mail: *claire.gerente@emn.fr*

C. Faur
Université de Montpellier 2, UMR CIRAD 016 Génie des Procédés Eau - Bioproduits,
2 place Eugène Bataillon,
34095 Montpellier Cedex 5,
France
E-mail: *catherine.faur@univ-montp2.fr*

P. Le Cloirec
Ecole Nationale Supérieure de Chimie de Rennes,
CNRS, UMR 6226,
Avenue du Général Leclerc, CS 50837,
35708 Rennes Cedex 7,
France
and
Université européenne de Bretagne,
35000 Rennes,
France
E-mail: *pierre.le-cloirec@ensc-rennes.fr*

INTRODUCTION

Water scarcity is today a worldwide issue: it concerns both arid zones and very dense urban areas, in which wastewater recycling and reuse is one of the main ways to preserve water resource. Recycling wastewater at a city scale requires supplying buildings with both potable and reused water. To avoid the prohibitive cost of a dual reticulation system, wastewater can be recycled inside buildings. Most of the time, these installations recycle only greywater (GW), which can include wastewater from bathrooms (wash basins, baths and showers), from laundry facilities, and

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may also include wastewater from dishwashers and kitchen sinks, but these latter are often left aside because they are putrescible (Lazarova *et al.* 2003). GW recycling and reuse can provide reclaimed water in sufficient amounts to flush toilets, supply washing-machines, wash cars or water gardens, inducing a reduction by 29 to 47% of potable water requirement within a household (Lazarova *et al.* 2003).

Membrane separations are today considered as a valuable solution for greywater recycling, whereas they were previously considered unsuitable, mainly because of their high costs. A great asset of membrane technologies is their ability to ensure health safety: ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO) membranes are physical barriers to solutes and to harmful micro-organisms (Pearce 2007). More, membrane technologies are compact systems, so they are easy to install in the basement of a building. The quality of reclaimed water barely depends on the polluting charge of greywater, so a variation in contaminants concentration has negligible influence (Madaeni 1999). Last, in membrane processes, waste generation is reduced, the main waste being the retentate, which is often sent to the sewage collection system. Various studies have compared greywater recycling efficiency by different technologies, including membrane bioreactors (MBR) and other biological processes, such as biological aerated biofiltres (Jefferson *et al.* 2000; Hasar & Kinaci 2004; Pauwels *et al.* 2006). But there still is a lack of knowledge concerning greywater treatment by direct membrane filtration, while it is known to be less constraining than MBR (Ramon *et al.* 2004), whereas both technologies can achieve high effluent quality. Thus, several membrane structures, configurations, materials and pore size ranges could be used for direct filtration of GW. Compared to UF membranes, NF membranes have a tighter structure and are therefore able to reject small organic molecules having molecular weights as low as 200–300 Da (Trébouet *et al.* 2001).

Ramon *et al.* (2004) studied direct UF and NF of wastewaters from public showers in a sports centre in Israel. At a transmembrane pressure (TMP) of 1 to 2 bar, organic UF flat-sheet membranes with molecular weight cut-off (MWCO) of 400 kDa and 30 kDa, achieved respectively 45 and 69% retention in chemical oxygen demand (COD), and 92 and 96% retention in turbidity. A NF polyamide tubular membrane (MWCO of about 200 Da), at a pressure between 6 to 10 bar, allowed retentions of 93% in COD, 83% in total organic carbon and 98% in turbidity. These results are promising, and NF seems to be the process that meets the best compromise between solutes retention (better than UF) and energy consumption (lower than RO) (Trébouet *et al.* 1999). Moreover, NF tubular membranes can operate on a high load effluent without any pre-treatment, which is synonym of reduced maintenance. The absence of pre-treatment also means the effluent is a complex mixture where some interactions between the different components can occur and possibly increase the removal yield (Pignon *et al.* 2000).

This study was focused on greywater filtration with NF tubular membranes. A synthetic greywater (SGW) was elaborated and used to test the recycling efficiency of three tubular membranes with different molecular weight cut-offs at 25°C at two different TMP (20 and 35 bar). Finally, the membrane achieving the best compromise between flux and retentions was then evaluated on a real greywater, in order to validate the results obtained on SGW. The novelty of this experimental work consists in several points, including: i) the characterisation of the SGW and the permeate using numerous key parameters, including biochemical oxygen demand, concentration of anionic surfactants and enumeration of indicators of faecal pollution ii) the precise measure of the permeate flux during each experiment, iii) the comparison of the performances of the process on synthetic and real greywaters.

MATERIALS AND METHODS

Pilot plant with constant concentration configuration

NF experiments were performed on tubular membranes fitted onto a Microlab 40 plant provided by VMA Industries (Figure 1). This pilot plant operated between 10 and 40 bar by means of a triplex plunger pump. The flux was measured by a Mettler PM4600 balance linked to a computer, which recorded the mass (±0.1 g) of permeate produced. Experimental error on flux measurement was lower than 2%. Permeate and concentrate were recirculated into the feed tank so that the effluent concentration was kept constant.

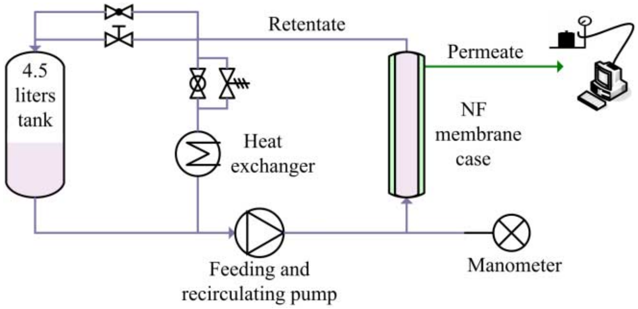

Figure 1 | Simple flow sheet of the NF pilot plant Microlab 40.

The temperature was maintained at 25.0 ± 0.3°C thanks to a heat exchanger connected to a cooling group. The tangential velocity of the solution over the membrane surface was 2.5 $m\,s^{-1}$.

Membranes properties and cleaning procedure

The pilot plant was equipped with tubular membranes made of a thin-film composite polyamide/polyethersulfone provided by PCI Membrane Systems (Basingstoke, UK). The effective membrane surface was 0.031 m^2. Some properties of the membranes and their operational parameters given by the manufacturer are shown in Table 1. These membranes differed by their MWCO, their $CaCl_2$ retention and their maximal authorised pressures: while AFC30 is limited to 35 bar, AFC40 and AFC80 can be operated at 40 bar. Before and after each experimental work, membranes underwent a standard cleaning procedure, which consisted in an alkaline cleaning (pH 9.4) at 45°C for 60 min with 10 $g\,L^{-1}$ of Ultrasil 53 (Henkel Ecolab), followed by a rinse with deionised water, an acid cleaning (pH = 1.4) at 35°C for 30 min with 3 $g\,L^{-1}$ of nitric acid and a final rinse with deionised water.

Experimental procedures

All experiments were conducted in duplicates, with the membranes being cleaned following the standard cleaning procedure between each repetition. The data presented are the average of both trials made in similar conditions. The results were reproducible: the error between the duplicates was lower than 9%, except for one DOC analysis (17% error) and one *Enteroccocus* enumeration (42% error). The SGW was nanofiltered at constant concentration until a steady state was reached (three hours) before permeate and concentrate were analysed. Two working pressures have been chosen: 20 bar and 35 bar. The lowest pressure has the advantage of being less energy-consuming, but as shown in Table 1, it is lower than the recommended pressure for both AFC30 and AFC80. Trials are then performed at 35 bar which corresponds to the maximum recommended pressure for AFC30 and AFC40. The water flux was measured by filtrating deionised water for 20 min at the same pressure than the concerned trial. The initial water flux was measured after a chemical cleaning of the membrane, while the final water flux took place after 3 hours of SGW filtration and simple rinsing with 4 L of deionised water.

Physico-chemical and microbiological parameters

For all pollutants and bacteria, the retention (R) wa calculated by means of the following equation: $R = 1 - (C_P/C_C)$ where C_P and C_C are the concentrations of the pollutant considered in the permeate and in the concentrate respectively, analysed at steady state. pH was determined with ±5% of uncertainty by means of a Consort C862 apparatus following ISO 10523:199. Turbidity (given in Nephelometric Turbidity Units, NTU)

Table 1 | Characteristics and operating conditions of studied membranes

	AFC30	AFC40	AFC80
Pure water flux* ($L\,m^{-2}\,h^{-1}$): at 20 bar/at 35 bar	88.4/150.7	68.8/155.6	47.3/54.8
MWCO (Da)	200	300	<200
$CaCl_2$ (5 $g\,L^{-1}$) retention at 25 bar	53%	40%	NA
Pressure (bar): recommended/maximal	30–35/60	15–35/60	40–45/60

*Experimental values measured in this study at 25°C.
NA: data not available.

was evaluated by a Lovibond apparatus with ±2% of uncertainty in accordance with ISO 7027:1999. Suspended solids (SS) were measured by filtration through glass fibre filters (0.45 μm porosity) following ISO 11923:1997. Experimental error was evaluated to ±8%. Anionic surfactants (A-surfactants) and chemical oxygen demand (COD) were analysed with Spectroquant kits and Multy spectrophotometer provided by Merck. The methods were respectively similar to ISO 7875-1:1996 and ISO 6060:1989 and they both resulted in experimental errors of ±6%. Biochemical oxygen demand for 5 days (BOD_5) was evaluated by respirometry with an OxiTop system from WTW incubated at 20 ± 0.1°C during 5 days. Dissolved organic carbon (DOC) was analysed by a Shimadzu TOC 5000A apparatus following ISO 8245:1999. The experimental errors on BOD_5 and DOC were respectively worth 2% and 7%. Faecal coliforms and *Enterococcus* were isolated using two different techniques: i) dilution and seeding method for raw greywater and membrane filtration concentrates, and ii) filtration on 0.2 μm pore-size sterile cellulose nitrate filters for permeates (ISO 93081:2000 and ISO 78992:2000). In both methods, the same agars and incubation durations were used. Faecal coliforms were incubated for 21 ± 3 hours at 44 ± 2°C on lactose agar with Tergitol 7 (100 mg L^{-1}) and TTC (25 mg L^{-1}). The error on faecal coliforms count was ±0.8 log. The incubation of *Enterococcus* takes place on Slanetz and Bartley agar for 44 ± 4 hours at 36 ± 1°C. The error on this method was ±0.3 log.

Synthetic and real greywaters

A SGW was reconstituted in order to conduct experiments on a reproducible effluent. Its composition is given in Table 2. This SGW was composed of chemical products of technical quality and of septic effluent to bring faecal contamination indicators. The SGW was formulated so that the values of its parameters were comprised between the extrema found in literature, given in Table 3. In this table, parameters of the SGW are compared with that of the real greywater used for the validation of the results obtained with the SGW. This real greywater was a mix of samples from five families, composed of adults (80%), children under 15 years old (10%) and babies under 2 (10%). The samples were collected in the five households, directly in the bath tubes, showers or wash basins.

Table 2 | Composition of the synthetic greywater

Chemical product	Supplier	Purity	Concentration (mg L^{-1})
Lactic acid	Panreac	>85%	100
Cellulose	Serva	>90%	100
Sodium dodecyl sulfate	Merck	>85%	50
Glycerol	Panreac	99%	200
Sodium hydrogen carbonate	Panreac	>99%	70
Sodium sulfate	Panreac	99%	50
Septic effluent			10

RESULTS AND DISCUSSION

The flux obtained during trials on the AFC30, AFC40 and AFC80 are shown in Figure 2. Irrespective of the pressure applied, the permeate flux on SGW was higher than 50 L m^{-2} h^{-1}, except for AFC80 operated at 20 bar, where the flux achieved was 25.2 L m^{-2} h^{-1}. The filtration of SGW induced very few fouling, and this fouling was almost only reversible: the final water flux, was very close to the initial water, meaning that the cake layer was completely removed by a simple water rinsing. It is interesting to note that in some cases, the flux on SGW was higher than the initial water flux: this could be observed for AFC30 at 20 and 35 bar, and for AFC40 at 20 bar. This observation could not be explained by the pH or the conductivity of the SGW or by any of the parameters measured in this study, and the phenomenon would have to be explained by further investigation. Cleaning procedure systematically allowed the recovery of the initial water flux.

It comes without surprise that when pressure rose, fluxes increased, but when moving up from 20 bar to 35 bar (representing a 75% raise on pressure), flux on AFC30 increased of more than 70% and it was multiplied by more than two on AFC40, whereas it only increased by 16% on AFC80. Thus, as all membranes showed an

Table 3 | Average composition of studied synthetic and real greywaters

				Real, literature review			
				Eriksson *et al.* (2002)		Ottoson & Stenström (2003)	
		SGW	Real, present study	Min	Max	Min	Max
pH		6.8	7.3	6.40	8.10	–	–
Turbidity	NTU	24.1	62	28.0	240.0	–	–
SS	$mg\,L^{-1}$	71.8	42.2	48	200	–	–
COD	$mg\,O_2\,L^{-1}$	454.1	252	100	424	–	–
BOD_5	$mg\,O_2\,L^{-1}$	65.1	89	76	300	–	–
DOC	$mg\,C\,L^{-1}$	132.4	101.5	30.0	104.0	–	–
A-surfactants	$mg\,MBAS\,L^{-1}$	49.1	27	–	–	–	–
Faecal coliforms	CFU/100 mL	9.6×10^3	1.5×10^5	–	–	1.6×10^2	1.0×10^7
Enterococcus	CFU/100 mL	2.7×10^3	$>6.0 \times 10^4$	–	–	1.0×10^1	2.5×10^5

acceptable permeate flux at the two operating pressures, the selection of an adequate membrane with an adapted pressure was be based on the retentions obtained. The analyses of the effluents produced during SGW filtration on AFC30, AFC40 and AFC80 at 20 and 35 bar are given in Table 4.

At a pressure of 20 bar, the three membranes performed a good retention of micro-organisms, with more than 1.7 log retention on faecal coliforms (Table 4). The most contaminated permeate contained 55 CFU/100 mL *Enterococcus*. Turbidity and suspended solids were also well retained by the three membranes at 20 bar: maximal turbidity in permeate was 0.6 NTU, and SS remain undetected in all permeates. COD and DOC retentions were similar to data collected in previous works (Ramon *et al.* 2004): all membranes removed more than 60% of COD and more than 48% of DOC. As for BOD_5 and anionic surfactants, while AFC30 and AFC40 allowed a good retention (more than 94% of BOD_5 and more than 83% of surfactants), AFC80 only removed 75% of BOD_5 and 54% of surfactants. So, at a pressure of 20 bar, AFC40 and AFC30 achieved high but perfectible retentions with high permeate flux, whereas AFC80 set poorer results in both retention and flux. For all membranes, retention of organic matters seemed to be the main issue. As the presence of organic matter in recycled water is a source of concerns, such as possible bacterial re-growth and chlorination by-products generation, it is better to reduce the organic content of permeates.

At a pressure of 35 bar, results were noticeably different (Table 4). As noted previously, fluxes were higher than at 20 bar. Disinfection was ensured by the three membranes: no *Enterococcus* was detected in any permeates. But the retentions of other pollutants behaved differently. AFC30 has poorer retention at 35 bar: COD, BOD_5, DOC and anionic surfactants were less retained than at 20 bar. AFC40 showed approximately same retentions at both pressures, with a slightly lower permeate quality at 35 bar. As for AFC80, the retentions increased considerably with pressure: the quality of permeate with the objectives, with 95% retention of COD and anionic surfactants and 98% of BOD_5. Thus, AFC40 behaved like an ultrafiltration membrane, while AFC80 seemed to filtrate by solubilisation-diffusion mechanisms, like reverse osmosis membranes do (Maurel 2006). For this reason, among the three

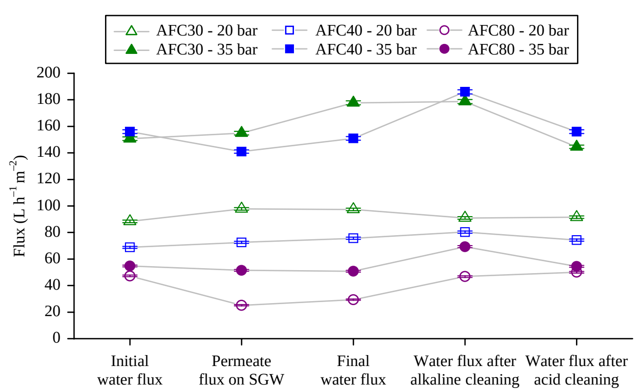

Figure 2 | Flux evolution during filtration of SGW on AFC30, AFC40 and AFC80.

Table 4 | Permeates analyses and retentions during filtration of SGW on AFC30, AFC40 and AFC80 membranes

Greywater		**Synthetic GW**												**Real GW**	
Membrane		**AFC30**				**AFC40**				**AFC80**				**AFC80**	
Pressure (bar)		**20**		**35**		**20**		**35**		**20**		**35**		**35**	
P analysis /retention		***P***	***R***	***P***	***R***	***P***	***R***	***P***	***R***	***P***	***R***	***P***	***R***	***P***	***R***
pH		7.0		7.0	95%	7.1		7.6		7.4		6.8		7.8	
Turbidity	NTU	<1.0	100%	<1.0	98%	<1.0	99%	2.5	94%	NA		<1.0	97%	<1.0	>98%
SS	$mg\,L^{-1}$	NA		1.3	44%	UD	100%	UD	100%	NA		NA		UD	>95%
COD	$mg\,O_2\,L^{-1}$	160	64%	261	63%	138	69%	151	66%	170	60%	26	95%	<25	>90%
BOD_5	$mg\,O_2\,L^{-1}$	3	96%	25	24%	1	>95%	4	94%	18	75%	1	98%	1	99%
DOC	$mg\,C\,L^{-1}$	61	48%	103	<67%	44	59%	64	56%	60	54%	12	92%	38	62%
A-surfactants	*	8	83%	>20		4	89%	10	83%	16	54%	3	95%	3	90%
Faecal coliforms	**	UD	2.53	NA		UD	3.37	UD		UD	1.71	NA		UD	5.28
Enterococcus	**	UD	1.72	UD	3.78	UD	2.21	UD	3.32	1.74	0.53	UD	3.91	UD	4.78

P: permeate, R: retention, UD: undetected, NA: not analysed, *mg MBAS L^{-1}, ** log CFU/100 mL.

membranes tested, AFC80 was the most adequate one to recycle greywater when operated at 35 bar: even if its flux was lower than that of the other membranes, its retentions were very high. Increasing the pressure would certainly have enhanced the flux, but the permeate quality improvement would have been minor, whereas the energy consumption would have risen considerably. As it would have affected the environmental and financial sustainability of an inside-building recycling scheme, and given that it was not desirable to work with high pressures inside a residential building, the maximal operating pressure has been maintained at 35 bar.

The quality of the permeate produced by filtrating SGW at 35 bar with AFC80 (Table 4) met stringent regulations on recycled water, such as Japanese regulation (less than 10 CFU/100 mL of total and faecal coliforms, less than 10 mg $O_2\,L^{-1}$ BOD_5, turbidity lower than 5 NTU and pH between 6 and 9 (Surendran & Wheatley 1998). Thus, the permeate produced by AFC80 at 35 bar seems to be suitable for inside-buildings use, such as toilet flushing and even clothes washing. To comply with regulations, a disinfection step might be necessary. Considering the low organic content of the permeate (in the four samples analysed, BOD_5 is below the detection limit of 1 mg $O_2\,L^{-1}$), low concentration of chemical disinfectant will be sufficient to prevent biofilm growth inside the permeate storage vessel.

The comparison between NF treatment of real and synthetic greywaters took place on AFC80 at 35 bar. Fluxes on real and synthetic greywaters were very similar: the initial water fluxes were comparable (54.8 vs. 55.2 $L\,m^{-2}\,h^{-1}$ for real and synthetic respectively) and the ratios between the fluxes measured and the initial water fluxes corresponded pairwise. Retentions were also similar (Table 4): more than 98% of turbidity and BOD_5 were retained in both real and synthetic greywaters; COD and A-surfactants retentions reached 95% on the SGW and 90% on the real greywater. From a sanitary point of view, the results on real greywater complied with the objectives: no faecal coliforms or *Enterococcus* were to be found in the real greywater permeate.

These trials lead to the conclusion that AFC80 at 35 bar was able to remove pollutants from bathroom greywater with acceptable fluxes. As fluxes and retentions performed on real and synthetic greywater were similar, it post-validates the conclusions made about data obtained with the SGW on the three membranes.

CONCLUSIONS

Greywater recycling by direct nanofiltration was investigated. Three membranes (AFC30, AFC40, AFC80) were tested to nanofiltrate synthetic greywater. The permeate showing the best quality was produced by filtrating bathroom greywaters on the AFC80 membrane at 35 bar: faecal coliforms, *Enterococcus* and majority of organic matter were removed. The permeate flux was then close to $50\,L\,m^{-2}\,h^{-1}$. These performances are encouraging regarding to flux and retentions. The nanofiltrated water meets stringent regulations, such as Greek guidelines, which recommend less than 2 CFU/100 mL of faecal coliforms in 90% of samples, less than $10\,mg\,O_2\,L^{-1}$ BOD_5 and TSS concentration below $10\,mg\,L^{-1}$ (Gkikas 2009).

Nevertheless, the high pressure needed to obtain high retentions implies considerable energy consumption. According to methodology and assumptions adopted by Denis *et al.* (2009), recycling $3\,m^3$ of greywaters issued from a small collective residential building of 50 inhabitants would require about $4.5\,m^2$ of membrane surface. To operate this module in the same conditions than in the present experimental work, the energy consumption would be almost 11.7 kWh per cubic meter of recycled water. But, as previously demonstrated by Trébouet *et al.* (1999), NF membranes would allow a good compromise between solutes retention and energy consumption.

More research studies are needed to manage all the parameters that are necessary to evaluate the financial and environmental sustainability of the process. Additional experiments on nanofiltration technology are necessary to study the long term performance of the process, including cake layer formation and membrane cleaning. Cleaning procedure can have strong environmental and financial impacts: the chemical cleaning of $4.5\,m^2$ of tubular membranes produces $0.8\,m^3$ of alkaline waste solution. For process sustainability, the frequency of membrane cleaning and the conversion rates, which is the ratio between permeate and inlet flow rate, will have to be optimized in order to avoid high fouling intensity and low process productivity.

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