Desalination and Water Treatment
www.deswater.com

51 (2013) 1831–1837
February

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doi: 10.1080/19443994.2012.714637

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Pre-treatment of Llobregat River raw water through pressurised inside/out hollow fibre ultrafiltration membranes

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Received 14 March 2012; Accepted 18 July 2012

ABSTRACT

The feasibility of raw river direct ultrafiltration, as an alternative to conventional drinking water treatment plant pre-treatment, was investigated at prototype scale (May–October 2011). A highly variable and challenging water resource was selected, in order to assess different scenarios, covering a broad range of conditions. The prototype was able to deal with conditions ranging from 20 to >800 NTU successfully, without any chemical pre-treatment and consuming low amount of chemical reagents for cleaning purposes. The membranes' performance proved to work better in terms of water production yield and resistance build up stability at medium and high turbidity episodes than at lower ones, probably due to a cake layer formation which prevented small binding organic species and particles reaching the membrane. Permeate quality, both in physico-chemical and microbiological terms, was independent of the feed water characteristics.

Keywords: Direct ultrafiltration; Membrane fouling; Low and variable quality surface water; Drinking water treatment

1. Background and introduction

Drinking water treatment plants (DWTPs) will have to face significant challenges in the following decades. On one hand, they will have to be able to supply an increasing water demand. On the other, it is likely that water resources (DWTPs' feed) will present a lower quality and therefore water treatment units will need to be modified accordingly. Additionally, other aspects will have to be taken into account: legislation, increasing energy cost and social requirements in terms of processes sustainability. In the legislative aspects side it is worth mentioning that it is becoming more stringent both in terms of product water as well as the process itself. For instance, the Spanish Order SAS/1915/2009 bans certain chemicals, such as polyacrylamides, frequently used in coagulation/flocculation pre-treatments. As a result, technologies capable of successfully achieving legislative,

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Presented at the International Conference on Desalination for the Environment, Clean Water and Energy, European Desalination Society, April 23–26, 2012, Barcelona, Spain

environmental and social challenges in a sustainable way are urgently needed.

Membrane technology has significantly evolved in the last decades, becoming a technological solution increasingly applied in DWTPs [1] due to its advantages. In particular, low-pressure membrane systems, including microfiltration (MF) and ultrafiltration (UF), are being increasingly used for drinking water production as final treatment sequence [2] and have gradually gained acceptance as the preferred pre-treatment to reverse osmosis (RO) [3]. This work aims at demonstrating at prototype scale the feasibility of applying direct UF (i.e. direct raw water filtration by UF) instead of the conventional pre-treatment (coagulation-flocculation, settling and sand filtration). The case study selected is the Llobregat River, in Barcelona (Spain) a challenging scenario because of its large water variability in terms of quality and quantity.

Few previous works published have assessed the evaluation of direct MF/UF as alternatives to conventional pre-treatment in DWTPs treating surface water, concluding that they are suitable alternatives in terms of quality [4–6]. Nonetheless, the scale of the experiments reported by these studies (bench level), duration (few months) or feed water qualities (canal or reservoir water) were highly different from the ones covered in this paper. Turbidities of the feed water in these previous studies (around 20 NTU, with a maximum of 150 NTU) were well below the average of the Llobregat River (average of 171 NTU and a maximum value of >800 NTU, for the period considered. As a result, the feasibility of direct UF is not proved yet at these extreme feed water quality conditions.

The objective of this work is to study at prototype scale the feasibility of direct UF with pressurised inside/out hollow fibres of highly variable raw surface water, characterising the optimal operational conditions in different water quality scenarios and quantifying technically its performance.

2. Methodology

The Llobregat River is the main surface water resource of Barcelona metropolitan area (North East of Spain) and it is characterised by its Mediterranean behaviour: large flow fluctuations (severe droughts during summer and flash flood events in spring and autumn) and its associated water quality variations. Moreover, the Llobregat River suffers from historical industrial and urban contamination.

The direct UF prototype plant used in this work treated raw Llobregat River water, which exhibited fluctuations between 20 and >800 NTU (maximum reading limit) during the tested period (May–October 2011), without any chemical pre-treatment. The prototype was equipped with a strainer (300 μm) as mechanical pre-treatment to prevent excessive clogging of the subsequent inside-out pressurised hollow fibres (Pentair X-Flow Aquaflex—polyethersulphone membranes with a nominal pore size of 0.020 μm and 0.8 mm of internal fibre diameter), and was controlled automatically by a Scada system. The software enabled the modification of several variables, such as filtration time, permeate flow, cleaning conditions (frequency, duration, backwash flow, air flow, reagents' nature and concentration), etc. so that the prototype was adaptable to the changing conditions when necessary.

The prototype initially worked under 30 min of filtration time treating $3.3\,\mathrm{m^3/h}$ ($60\,\mathrm{L/(m^2\,h)}$) at constant permeate flow, followed by a hydraulic cleaning (HC) (backwash [$250\,\mathrm{L/(m^2\,h)}$,15 s] enhanced by airflush [$10\,\mathrm{Nm^3/h}$, 10 s]). Every 50 filtration cycles, a chemically enhanced backwash (CEB) was undertaken, composed of a basic-oxidizing stage (NaOH and NaOCl) and an acid stage (HCl) subsequently. The CEB sequence consisted of HC, dosing [$125\,\mathrm{L/(m^2\,h)}$, 45 s], soaking [10 min] and rinsing [$250\,\mathrm{L/(m^2\,h)}$, 45 s] stages. Alkaline solution concentration was 480 mg/L, oxidiser 200 mg/L and acid 438 mg/L. In August 2011, filtration time was decreased to 15 min and in order to maintain one CEB per day approximately, the CEB was conducted every 100 filtration cycles.

When a pre-set value of recoverable resistance was reached ($1.4 \times 10^{12}\,\mathrm{m^{-1}}$), a HC was automatically performed, shortening the filtration time but restoring membranes' resistance. If high resistance was reached in less than 10 min of filtration time, the prototype plant was automatically stopped, as a safety measure, to avoid situations of continuous HCs. Also, if a higher pre-set value of recoverable resistance ($1.8 \times 10^{12}\,\mathrm{m^{-1}}$) was achieved almost immediately after starting filtration, the prototype stopped.

Specific cake resistance (α), which represents the increase of the cake layer resistance build up with filtered volume, was calculated as shown in Eq. (1). Membrane resistance (R) during a filtration cycle was approximated to a first order polynomial and then it was derived respective to the specific volume (v) (i.e. filtered volume per unit area).

$$\alpha = \frac{\mathrm{d}R}{\mathrm{d}v} = \frac{\mathrm{d}R}{\mathrm{d}(v/A)} \tag{1}$$

Both Llobregat River water and membrane permeate stream were characterised by different analyses in a periodical basis. The parameters monitored and the methods used were: temperature by resistive temperature device (Endress & Hausser TR10-ABG1HD-SAG2000), conductivity by electrometry (Endress & Hausser CLS21D-C1 + CM42-KAA000EAN00), pH by potenciometry (Hach-Lange DPD1P.99), turbidity by nefelometry (Hach-Lange Ultraturb SC), total suspended solids (TSS) by ESS 340.2, absorbance at 254 nm by spectrophotometry (Hach-Lange DR 5000), silt density index (SDI) by ASTM D4189 (Simple SDI Meter 9C-281-0157), dissolved organic carbon (DOC) by combustion-infrared method using a DOC analyser (non-purgeable organic carbon, UNE-EN 1484), after filtration with a 1.2 μm glass fibre filter for the raw water samples (TOC-V CSH Shimadzu), total coliforms, faecal coliforms and *E.Coli* quantification by the defined substract method (most probable number), *Clostridium Perfringens* and aerobic bacteria at 22°C by plaque counting, and algae count by counting chamber.

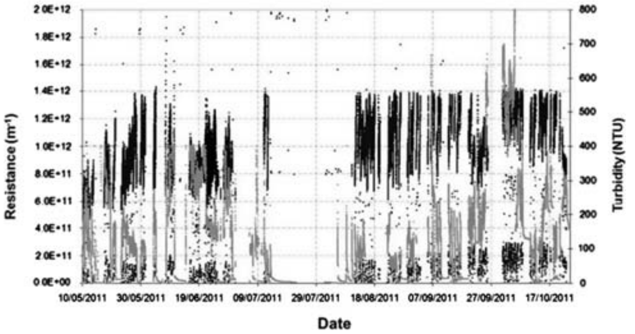

Fig. 1. Membranes' resistance (temperature corrected) and raw river water turbidity evolution along time (May–October 2011). Black symbols correspond to membrane resistance whereas grey symbols turbidity values. Until July, turbidity maximum reading scale was set on 400 NTU, afterwards on 800 NTU.

3. Results and discussion

3.1. Hydraulic response

Fig. 1 shows the membrane resistance evolution (temperature corrected) along time (from May 2011 to October 2011) (black symbols), as well as the raw water turbidity fluctuation (grey symbols). Despite the large variability of the latter (from 20 to >800 NTU), the prototype proved to be able to treat the raw river water without any chemical pre-treatment. Nevertheless, its hydraulic performance varied during the different turbidity periods faced. Long term prototype shutdown, mainly due to external factors such as feed pumping problems, electrical power failure, etc. led to periods where no data was generated and hence, resistance and turbidity data is not available in Fig. 1.

Generally, at greater turbidity (150–250 NTU approximately) the prototype suffered less interruptions caused by exceeding the pre-set value for recoverable resistance than at lower turbidity. Filtration time was sometimes reduced automatically by the control system when reaching the pre-set resistance value ($1.4 \times 10^{12}\,\mathrm{m}^{-1}$), but not being less than 10 min, which would have led to the prototype stop. This can be seen in Fig. 2, where filtration time (dark symbols) is plotted vs. raw river water turbidity (grey symbols). As stated, when raw river turbidity ranged between 150 and 250 NTU, filtration time was normally equal to the filtration time set point (30 min), so that the membrane resistance was below the pre-set resistance value and the production water yield was maintained (HC were not conducted before the filtration time fixed). On the contrary, periods with lower raw river turbidity, filtration time was automatically reduced because high resistance pre-set value was achieved, so that the prototype conducted HCs more often, to such an extent that when it was less than 10 min, the prototype stopped, as programmed. In August 2011, when turbidity was lower than 150 NTU, to minimise prototype stops, filtration time was reduced to 15 min, minimum filtration time to 4 min and CEB frequency increased to 100 filtration (to keep chemical cleaning conditions constant), but filtration flow was not modified (to maintain hydraulic conditions). However, at that point turbidity increased significantly, leading to very high turbidity episodes (>250 NTU). In this case, filtration time also decreased significantly (and hence HC were performed more frequently) by reaching high resistance threshold, but the prototype did not

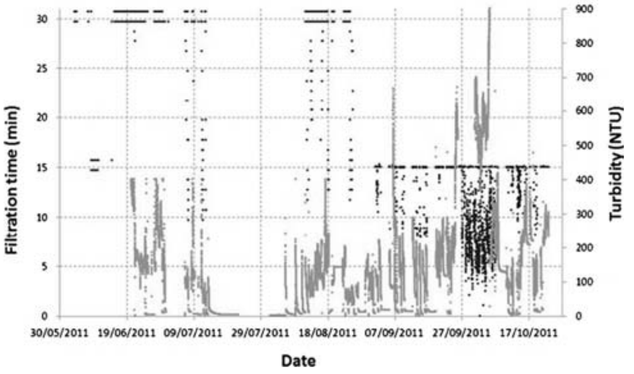

Fig. 2. Filtration time (dark symbols) and turbidity (grey symbols) along time (May–October 2011). Until July, the turbidity maximum reading scale was set on 400 NTU, afterwards on 800 NTU.

stop, since the time needed to achieve this value was greater than 4 min.

Fig. 3 presents the specific cake resistance (calculated by Eq. (1)) along time (dark symbols) as well as turbidity evolution (grey symbols). Low turbidity episodes (<150 NTU) induced a greater resistance build-up than medium turbidity schemes (150–250 NTU), leading to a faster achievement of the resistance pre-set value and hence, a reduction in filtration time.

Several parameters affect specific cake resistance, among them particle shape, size distribution, porosity, particle density and TSS [7–9]. As remarked, the episodes where greater specific cake resistance was faced by the membrane in this work were when raw water presented low turbidity and extremely high turbidity (Fig. 3). Based on Carman-Kozeny equation [10], the first scenario may be explained either by low particle density, low cake porosity and/or small particles' diameter. In the case of very high turbidity events, a similar behaviour was described by Sripui et al. [9], who found that specific cake resistance increased with TSS concentration for a certain particle size range (1–20 μm).

Natural organic matter (NOM) is generally recognised as the main UF organic foulant, and recently it has also been suggested to play a detrimental role in inorganic particle fouling [11]. Nonetheless, the effects of NOM on inorganic particles' stabilization reported in the literature are different according to the NOM fraction considered (humic substances and polysaccharides, especially) and sometimes contrary results are reported [12–15]. Extracellular polymeric substances (EPSs), which are mainly polysaccharides, proteins, glycoproteins and glycolipids [16], and in particular, transparent expolymer particles (TEPs) have been recently identified as important fouling agents in UF [17,18]. It has been stated that they may not only be responsible for biological or organic fouling but may also enhance colloidal/particulate fouling [18]. Therefore, the differences in behaviour observed in this study may be due to an increase in TEPs content in raw water. Villacorte et al. [19] analysed TEPs evolution in seawater and the greater concentration was found during spring and early summer (March, April) rather than in August, as found here. Nevertheless, this hypothesis cannot be discarded since the North Sea seawater and Llobregat River composition as well as climate characteristics from the Netherlands and Spain are significantly different.

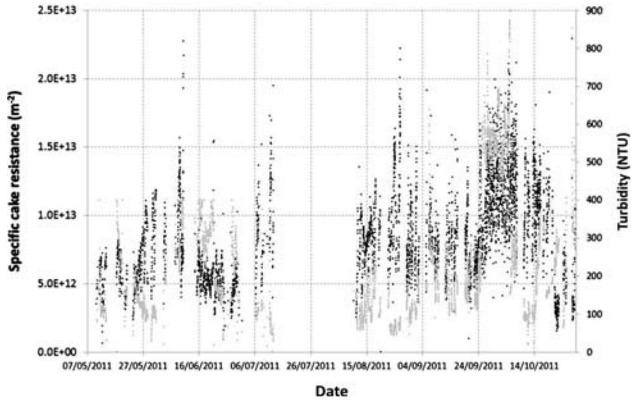

Fig. 3. Specific cake resistance (calculated by Eq. (1)) (dark symbols) and turbidity (grey symbols) along time (May–October 2011). Until July, the turbidity maximum reading scale was set on 400 NTU, afterwards on 800 NTU.

The interactions between NOM and particles are not sufficiently known and they may be one of the causes of the differences in the membranes' behaviour identified during low, medium and high turbidity episodes.

During high turbidity episodes, the higher particles' content or the shifts in particle size distribution may lead to a cake layer formation, preventing small organic molecules reaching the membrane and thus, minimising pore constriction and/or blockage. The latter have been identified in the literature as the most detrimental fouling mechanism in terms of flux decline [20–23], so results found in this work are in accordance to previous experiences. As stated, greater particles' content may lead cake formation as the main fouling mechanism and thus, act as an additional filtration barrier preventing small adsorptive particles, susceptible of binding in the membranes' surface and/or porous structure, reaching the membrane. Particulate cake formed may exhibit a greater average porosity, inducing lower membranes' resistance according to Carman-Kozeny equation [10,11]. Moreover, a cake layer formed by bigger particles presents a lower resistance than one of smaller particles, assuming equal weights of small and big particles, equal rejection and interactions among them and the membrane [24,25]. Additionally, NOM has been identified by several researchers as glue, binding inorganic particles to one another and to the membrane surface. In water matrixes containing high inorganic particles concentration, NOM may not be able to link to all the particulate matter among it and/or to the membrane, leading to a less compact cake and/or less tightly binded and hence, resistance.

During low turbidity periods, since less particulate matter is contained in raw water, cake formation may not be so important and either more low molecular weight organics may reach the membrane and cause pore constriction/blocking or the cake formed may be more compact because there is a greater NOM/particulate ratio (assuming NOM content is constant, since the DOC does not change significantly in the tested water) and, as a result, NOM binding to particles and/or the membrane is probably more prominent.

Both cake formation and its porosity may explain why the membrane performance is more stable and hence, presents fewer shutdowns due to high resistance, at greater turbidity periods than lower ones.

According to Carroll et al. [25] and Lin et al. [22], coagulation reduces fouling rate, since it transforms dispersed particles and other organic and/or inorganic pollutants into more retainable forms [26] by charge neutralization and flocculants sweep [27]. Microflocs formed progressively grow in size turning into macroflocs, leading to more permeable fouling layer and/or preventing pore blockage [26]. Coagulation prior to the membranes is not necessary in all cases [28], but when beneficial, commonly the doses needed are clearly lower than those needed for conventional pre-treatment [29,30,3], typically 40% or less [3]. As a result, microcoagulation could be implemented in this work in order to improve membrane performance during low turbidity events. A study based on TEPs' effect on membrane fouling found out that that a significant improvement in fouling control was experimented when using in-line coagulation [19].

Regardless raw water turbidity, membrane resistance fluctuated along time. After a CEB, resistance increased over time and, despite the HC performed, it reached a maximum value and then decreased, not necessarily coinciding with a CEB (data not shown). The dependency between resistance oscillation and other raw water physico-chemical parameters apart from turbidity (pH, conductivity, DOC, absorbance at 254 nm, etc.) was analysed but a clear relationship could not been established due to the large number of parameters changing simultaneously in natural waters. This fluctuation of resistance' values may be explained by the synergisms of different feed water components' interactions as claimed by Hong et al. [31] and Seidel and Elimelech [32] for nanofiltration (NF) membranes, as well as membranes and raw water components interactions.

The membrane recovery was 78–87% at 15 and 30 min of filtration time respectively, and the chemicals' consumption (used in CEBs) per cubic meter of feed water was 0.7 mL NaOH/m^3, 2.8 mL NaOCl/m^3 and 1.4 mL HCl/m^3.

3.2. Permeate quality

Despite the fluctuations of the feed water, the permeate produced presented a relatively constant quality in all the conditions tested, both at high and low turbidity events. Table 1 shows the results obtained until October 2011, highlighting the minimum, average and maximum values of each water quality parameter monitored.

As can be seen, turbidity and TSS were highly removed by the UF membrane (greater than 99% removal in both cases, as average), resulting into very low values independently of the raw water fluctuations. DOC was slightly removed by UF membrane (29% removal, as average), but not significantly, as expected, as well as absorbance at 254 nm (33% removal, as average). Nevertheless, these were not goals to be achieved by a membrane-based pre-treatment.

Microbiological parameters were completely removed by the considered UF membrane, except aerobic bacteria at 22˚C and filamentous algae. In the first case, despite the considerable reduction obtained, some colonies could be found in the permeate. This may be explained by their environmental presence, so that samples might have been infected by bacteria present in the atmosphere. In the case of filamentous algae, since their size are considerable larger than the membrane pore size (and membrane integrity was monitored monthly through pressure decay tests, not detecting any fibre broken, the most provable explanation for their presence is that some colonies might be present in the permeate pipes or sampling point, so positive results were obtained and the permeate may be free of them.

The fouling indicator (SDI) of permeate water was always within RO membrane manufactures' specifications (<3 in the case of SDI). Therefore, despite the reported limitations of the SDI method [33–35], a subsequent RO could be successfully connected and thus, lead to a much more compact treatment for DWTPs. In order to prove these results, in the subsequent stages of this work, a RO unit will be installed aiming at determining the real effects of direct raw water UF in a RO unit.

4. Conclusions

This study shows that direct UF is a suitable alternative in technical terms to conventional DWTP pre-treatment (coagulation-flocculation-settling-sand filtration), especially for periods with high and extremely-high water turbidity. In this case, cake formation appears to be the main fouling mechanism, so that larger particles accumulate on the membrane surface and prevent small organic compounds, which are responsible for not physically removable fouling, reaching the membrane. Also cake layer may be more porous and less tightly binded to the membrane. On the contrary, during low turbidity events, the cake layer formed may not

Table 1
Raw and membrane permeate water quality limits (June–October 2011)

Parameter	Raw water			Permeate water		
	Min	Average	Max	Min	Average	Max
Turbidity (NTU)	20	171	>800	0.03	0.05	0.40
Total suspended solids (mg/L)	48.5	91.6	272.5	LOD[a]	0.6	3.3
DOC (mg/L)	2.9	4.8	45.3	2.7	3.4	6.1
Absorbance at 254 nm	0.069	0.112	0.471	0.064	0.075	0.097
SDI	4.4	>5.0	>5.0	0.1	0.7	2.7
E. Coli [log (MPN/100 mL)][b]	2.82	3.30	4.66	Absence	Absence	Absence
Aerobic bacteria at 22°C [log(CFC/mL)][c]	3.76	4.45	5.43	1.49	2.44	4.64
Clostridium perfringens[log (CFU/100 mL)]	2.60	3.37	4.32	Absence	Absence	Absence
Total coliforms[log (MPN/100 mL)]	3.99	4.50	5.86	Absence	Absence	Absence
Faecal coliforms[log (MPN/100 mL)]	2.94	3.49	4.11	Absence	Absence	Absence
Filamentous algae[log (cells/mL)]	3.39	3.70	4.03	0.48	0.82	1.20

[a] LOD: Below limit of detection.

[b] MPN: Most-probable number.

[c] CFU: Colony forming units.

be so significant, so that molecules susceptible of being adsorbed and/or deposited in/into the membrane may reach it and cause greater resistance build up. Additionally, the fouling accumulated may be binded more strongly than in higher turbidity periods, becoming more difficult to remove. This turns into the need to increase cleaning operations, especially chemically enhanced ones.

Water quality produced along the testing period remained nearly constant, so that UF proved to provide treated water independently of feed water characteristics, regardless of its large fluctuations. General physico-chemical parameters were equal or superior in terms of quality than conventional DWTP pre-treatment. Most of the microbiological indicators assessed (total coliforms, faecal coliforms, *E.Coli* and *Clostridium Perfringens*) were removed from water by direct UF in all the sampling campaigns. The fouling index SDI was below three all the time (with average values of 0.7), which is the recommended value for a subsequent RO process.

As a result, direct UF proved to be an efficient pre-treatment, offering a high water yield (78–87%) and a low chemical consumption, only associated to membranes' cleaning and thus, avoiding chemical pre-treatment for highly variable raw river water.

Acknowledgements

This work has been conducted under the financial support of the LIFE+Programme of the European Commission within the framework of the UFTEC Project (LIFE09 ENV/ES/000467 UFTEC). The authors gratefully acknowledge Carmen Galvañ (CETaqua) and Bastiaan Blankert (Pentair X-Flow) for their technical contribution in this work.

List of symbols and acronyms

A — membrane area (m^2)
CEB — chemically enhanced backwash
DOC — dissolved organic carbon
DWTP — drinking water treatment plants
EPS — extracellular polymeric substances
HC — hydraulic cleaning
MF — microfiltration
NF — nanofiltration
NOM — natural organic matter
R — membrane resistance (m^{-1})
RO — reverse osmosis
SDI — silt density index
TEP — transparent expolymer particles
TSS — Total suspended solids
UF — Ultrafiltration
V — filtered volume (m^3)
v — specific volume (m)
α — specific cake resistance (m^{-2})

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