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Assessment of UV Pre-Treatment to Reduce Fouling of NF Membranes

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1. Introduction

Nanofiltration (NF) is an efficient technology to produce safe and biologically stable drinking water from surface water (Cyna et al. 2002). NF can respond to the increased demand of water with higher quality due to the evolution of the legislation and of the customer's expectation. NF treatment allows the reduction of the concentration of organic precursors to disinfection byproducts, and the reduction of the concentration of trace contaminants such as pesticides and pharmaceuticals. The introduction of NF in the drinking water production plant of Méry-sur-Oise, France, conducted to several changes in quality of the distributed water: reduction of total organic carbon (TOC) and Biodegradable dissolved organic carbon (BDOC) by a factor 3 to 5, reduction of THMs by a factor 2, reduction of viable bacteria population by a factor 10, reduction of chlorine demand of the distribution system by a factor 3, amount of pesticides below detection level.

Four membrane properties are important for the efficiency of a water treatment plant: high rejection of dissolved organics, low salt rejection, low energy consumption, and stable performance after repetitive cleanings. Membrane fouling generates flux decline leading to an increase in production cost due to increased energy demand and chemical cleaning. Moreover, fouling induces reduction in membrane life. Different types of NF fouling can be defined on the basis of fouling material: inorganic fouling due to deposition on membrane surface of inorganic scales; organic fouling due to humic acids, proteins and carbohydrates (natural organic material, NOM); biofouling due to biofilm formation at the membrane surface.

Flux decline associated with NOM fouling and with biofouling can be partially restored by chemical cleaning (Al-Amoudi et al. 2005, Di Martino et al. 2007, Houari et al. 2010, Roudman et al. 2000). Biofouling is distinct from NOM fouling caused by contaminated organic matter derived from biological systems (Flemming et al., 1997). Biofouling involves biologically active microorganisms which grow at the membrane surface as complex structures termed biofilms (Lappin-Scott et al. 1989). Biofilm formation precedes biofouling, which becomes an issue only when biofilms reach thickness and surface coverage that cause declined normalized flux and/or increase in normalized pressure drops during NF operation (Vrouwenvelder et al., 1998, Ridgway, et al. 1996).

Fouling and subsequent chemical cleaning of nanofiltration membranes used in water treatment are inevitable but can be decreased by adequate pretreatments (Al-Amoudi and Lovitt 2007, Di Martino et al. 2007, Flemming et al. 1997, Hilal et al., 2004, Houari et al. 2010, Speth et al. 2000). Pre-treatment must achieve a strong removal of microbial cells and growth-promoting compounds from the feed water (Vrouwenvelder et al. 1998, Wend et al. 2003, Vrouwenvelder and Van der Kooij 2001, Marconnet et al. 2009). Several pre-treatments schemes prior to NF have been evaluated including coagulation/precipitation, biofiltration, granular activated carbon (GAC) adsorption, microfiltration/ultrafiltration, ozonation, pre-chlorination, slow sand filtration, and UV irradiation (Cyna et al. 2002, Wend et al. 2003, Vrouwenvelder et al. 1998, 2008, Speth et al. 2000, Koyuncu et al. 2006). UV pre-treatment has several advantages: it has immediate germicidal effect; it is a closed and thus a safe system that requires only a small space for equipment; it does not generate byproducts; it acts only on water and cannot have deleterious effects on membranes. Nevertheless, since UV disinfection has no residual effect, the time between the UV irradiation and the membrane filtration has to be the shortest possible, so as to avoid bacterial regrowth (Salcedo et al. 2007). Few studies have evaluated the benefit of using UV as a pre-treatment upstream from NF and/or RO membranes (Conlon and Jhawar 1993, Koyunku et al. 2006, Munshi et al. 2005, Mofidi et al. 2000).

In the present study, the addition of a UV irradiation process upstream from NF modules was assessed to maintain membrane performances and to limit biofouling.

2. Materials and methods

2.1 Pilot units

Two identical nanofiltration pilot units (2 x 1 m^3/h) containing each a single 4-inch spiral wound NF element harbouring NF200B membranes (DOW Filmtec, Delft, The Netherlands) were used (Figure 1). A UV pilot including a low pressure mercury vapour lamp (monochromatic at 254 nm) was operated at a dose of 400 J/m^2 upstream from one of the pilots. The pilot units were fed by clarified river water pre-treated through sand filtration (sand-filtered water, SFW). Pilot 1 was fed SFW while pilot 2 was fed SFW irradiated by UV (SF + UVW), during a filtration run of 10 weeks (from February to May). Both NF pilots included an internal pre-treatment step made of pH neutralisation, 20 and 6 μm cartridge filtration and antiscalant injection, similar to what can be found in most of NF industrial units. The role of this pre-treatment was to reduce scaling (pH neutralisation and antiscalant addition) and particulate fouling (pre-filtration). A recycling loop enriches the module feed water with concentrate at a ratio concentrate/feed of 70%, in order to accelerate fouling.

2.2 Water quality parameters

Total direct counts (TDC) of microbial cells after 4',6-Diamino-2-phenyindole dihydrochloride (DAPI) staining, and active bacteria counts (ABC) of microbial cells after cyano 2-3 dytolyl – tetrazolium chloride (CTC) staining are determined by epifluorescence microscopy. Dissolved Organic Carbon (DOC) is measured with a Total Carbon Analyzer using persulfate oxidation of organic carbon, followed by infra-red non-dispersive detection of the CO_2 produced by the oxidation reaction. Biodegradable dissolved organic carbon (BDOC) is determined by batch incubation with sand-fixed bacteria according to the bioassay procedure developed by Joret and Lévi (1986) and the French standard XP T 90-319.
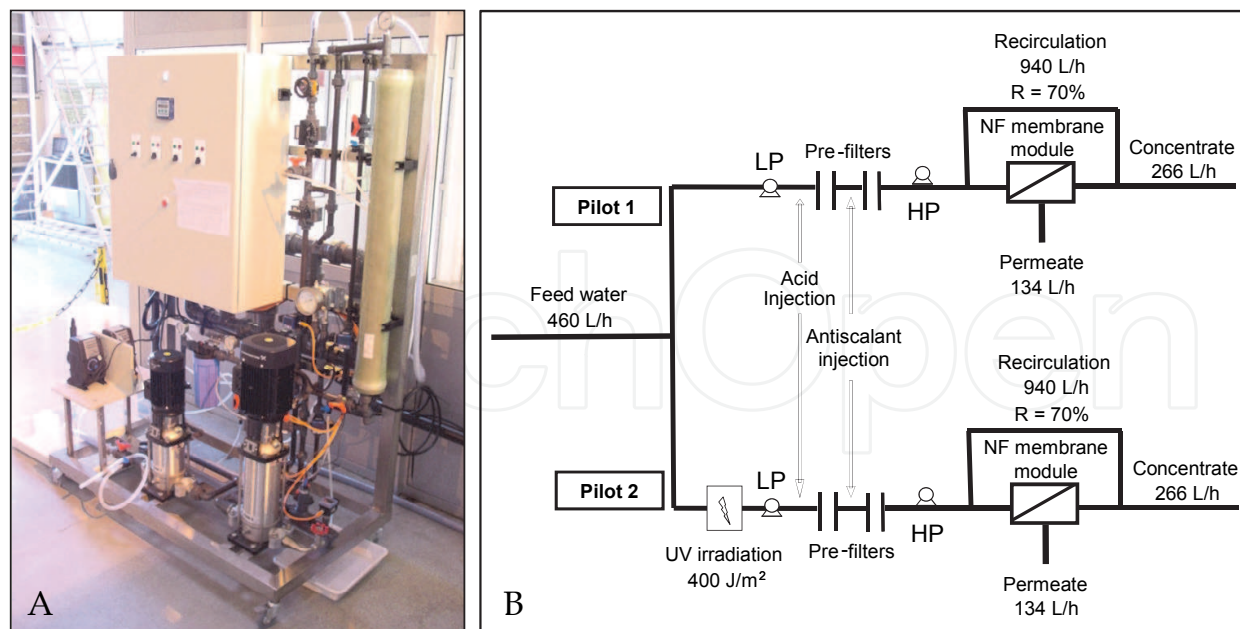
Fig. 1. A, photograph of the nanofiltration pilot showing the membrane module, the pumps and the electronic instrument panel. B, scheme of the experimental setup. Feed water was sand-filtered water. LP, low pressure pump. HP, high pressure pump.

2.3 Modules performance monitoring

The performances of the membrane modules, water permeability (L. h^{-1}. m^{-2}. bar^{-1}) normalized at 25°C and corrected of the osmotic pressure, and longitudinal pressure drop (LPD) also termed feed channel pressure drop (FCP) normalized by temperature and the flow rate along the module, were monitored on-line during both filtration tests.

2.4 Membrane autopsy

NF modules from the two pilot units were autopsied at the end of the filtration test. Membrane samples cut from randomly chosen areas of the module were air-dried and analysed by Attenuated Total Reflection Fourier Transform Infrared (ATR-FTIR) spectroscopy. Other membrane samples were fixed in paraformaldehyde before staining and fluorescence microscopy analysis, as described below.

2.4.1 Dry weight, protein content, microbial activity and wettability of the deposit

A membrane coupon of 50 x 90 cm was gently scraped to collect the wet deposit. The deposit was lyophilized, and the lyophilisate weight was measured. The lyophilisate was then dissolved in ultrapure water, vortexed and sonicated.

The protein concentration was determined after centrifugation by Bradford's colorimetric assay (Biorad protein assay, Biorad laboratories GmbH, München, Germany), bovine serum albumine (BSA, Sigma-Aldrich) being used as a standard (Bradford 1976).

For ATP assays, remnants of NF membrane (9.6 cm^2 for each one) were cut. Each sample was put in 3 mL of ultrapure water and sonicated (Branson sonifier 450). The concentration of ATP released by microbial cells after sonication was measured by the luminescence luciferine/luciferase test (ATP Determination Kit, Invitrogen). The intensity of light produced by the reaction was measured with a luminometer (Sirius Luminometer, Berthold

detection systems GmbH, Pforzheim, Germany). The ATP quantity was calculated by comparison with free ATP standards. The ATP concentration was an indicator of the microbial activity within the deposit.

The wettability of the membrane surface was determined by measuring the water contact angle of the foulant matter. A membrane coupon of 1 cm^2 was cut. An ultrapure water drop was deposited on the surface of the membrane, and the tangent angle between the drop and the surface was measured with a drop shape analysis-profile device (DSA-P, Kruss, Germany).

2.4.2 Analysis of membrane foulants by ATR-FTIR

Samples of air-dried fouled membranes were analyzed by ATR-FTIR spectroscopy. IR spectra were recorded using a Tensor 27 IR spectrophotometer with a 45° diamond/ZeSe flat plate crystal and an average depth penetration of 2 μm. Each spectrum presented was the result of 32 accumulations obtained with a resolution of 2 cm^{-1} with air as the background. IR bands indicative of biomass were detected near 1650 cm^{-1} (amide I, corresponding mainly to proteins), 1550 cm^{-1} (amide II, corresponding mainly to proteins), 1090 and 1040 cm^{-1} (corresponding mainly to polysaccharides) as previously defined (Doumèche et al. 2007). The IR signal near 700 cm^{-1} was used to calculate ratios corresponding to relative IR signals of biomass (amide I / membrane signal, amide II / membrane signal, and band near 1040 cm^{-1} / membrane signal) (Houari et al. 2009). Mean values ± standard deviation of relative IR signals of biomass are shown here.

2.4.3 DAPI and lectin staining of foulants

DAPI and lectin staining of foulants were done as previously described (Doumèche et al. 2007). Samples of the fouled membranes were treated with paraformaldehyde (4 %, v/v) prior to lectin application in order to fix the foulant matter. Double staining with a mixture of TRITC-labelled peanut agglutinin (PNA) and FITC-labelled *Lycopersicon esculentum* agglutinin (LEA) (Sigma, Saint Quentin Fallavier, France) was done. 100 μL of a mixture of two lectins (final concentration of 100 μg/mL each) were carefully applied directly on top of the membrane. After incubation during 30 min in the dark at room temperature, unbound lectins were removed by washing with filter-sterilized water. Following the lectin staining step, the fouled membranes were treated with a solution of DAPI (1 mg/L) (Sigma, Saint Quentin Fallavier, France) in filter-sterilized water. After incubation during 30 min in the dark at room temperature, unbound DAPI was removed by washing the membrane surface with filter-sterilized water. The stained preparations were then mounted with Mowiol (Calbiochem, Meudon, France) and stored at 4°C in the dark. For the negative control, the lectins and DAPI were replaced by filter-sterilized water.

2.4.4 Microscopy and image analysis

The fouled membranes stained with DAPI and fluorescently labelled lectins were examined with a Leica epifluorescence microscope (MPS 60) and with a Leica SP2 upright confocal laser scanning microscope (DM RAX-UV) equipped with the Acousto-Optical Beam Splitter (AOBS) system and using 63X, N.A. 1.32, oil immersion objective (Leica microsystems, Rueil-Malmaison, France). For epifluorescence images, DAPI was excited at 364 nm, CTC was excited at 450 nm, FITC was excited at 494 nm, and TRITC was excited at 550 nm. For confocal images, DAPI was excited at 405 nm and observed from 410 to 600 nm, FITC was excited at 488 nm and observed from 505 to 540 nm, TRITC was excited at 543 nm and

observed from 560 to 600 nm. The selection of spectral emission window for each fluorophore has been determined through λ scan analysis on single stained membrane fragments. The gain and offset for each photomultiplier have been adjusted to optimize nucleic acid and lectins detection. Images of the Confocal Laser Scanning Microscopy (CLSM) observations (1024×1024 pixels) have been acquired through a sequential mode, between stacks, to exclude spectral crosstalk from the data. 400 Hz scan speed was used and signal/noise ratio has been increased through frame average. Overlay and maximum projection of the z-stacks files have been performed with post acquisition Leica confocal software (LCS) functions to obtain the presenting snapshots. Original z-stack Leica files have then been imported into Imaris 4.0 software (Bitplane AG, Zürich) to obtain snapshots illustrating xz and yz representations.

2.5 Statistical analysis

ATP concentrations, contact angles measurements and values of relative IR signals of biomass obtained with or without UV irradiation were compared by the equal-variance Student's *t* test, following the variance test with Fisher *F* statistics. *P* values below 0.05 are considered significant.

3. Results

3.1 Feed water characterizations

Mean values of total direct bacteria counts (TDC), active bacteria counts (ABC), Dissolved Organic Carbon (DOC) concentration and Biodegradable Dissolved Organic Carbon (BDOC) concentration of the different feed waters are presented on Figure 2.
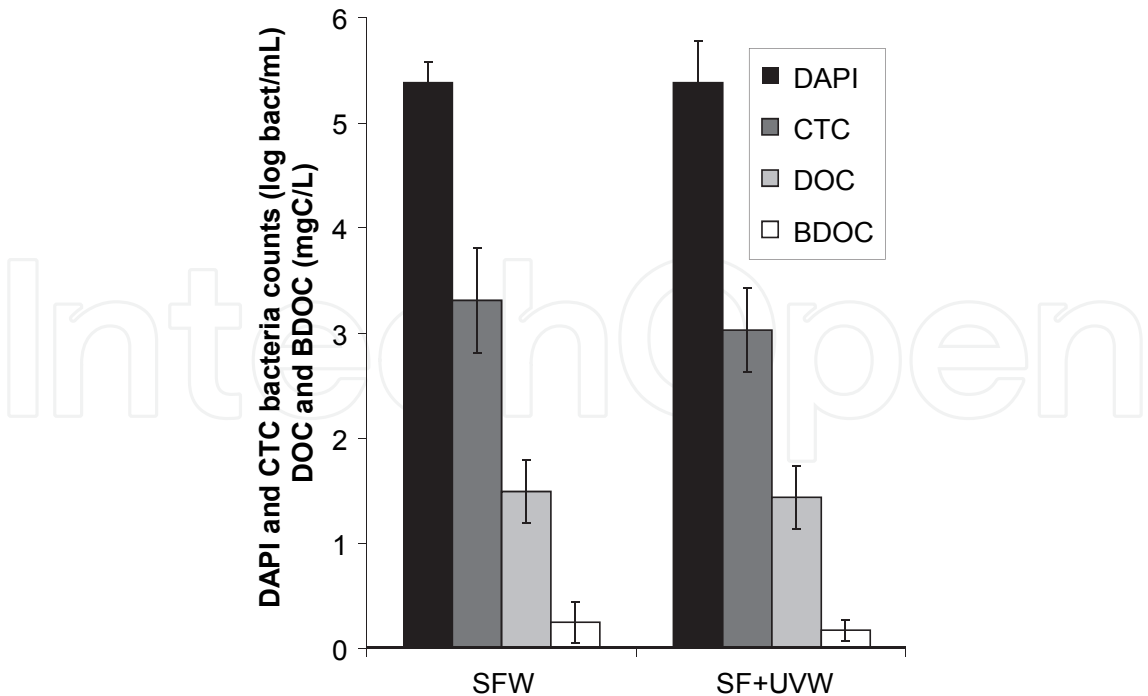

Fig. 2. Total direct Counts (DAPI) and active counts (CTC) of bacteria in feed waters, concentration in of dissolved organic carbon (DOC) and biodegradable dissolved organic carbon (BDOC) in the feed water with (SF + UVW) or without (SFW) UV irradiation.

UV pre-treatment did not affect the organic carbon concentration, as it was applied with a low pressure lamp at a reasonable dose of 400 J/m². The germicide effect of UV pre-treatment can be seen on the active bacteria counts (about 0.3 log removals).

3.2 Membrane performances

Water permeability and Longitudinal Pressure Drop (LPD) varied slightly all along the filtration period (Fig. 3). At the end of the test, water permeability decreased by 2.5 % with SFW as feed water and by 3.6 % with SF + UVW as feed water and the LPD value was 1.08 for SFW and 1.02 for SF+UVW. Both permeability and LPD evolutions were very low and the differences between the two conditions were not significant.
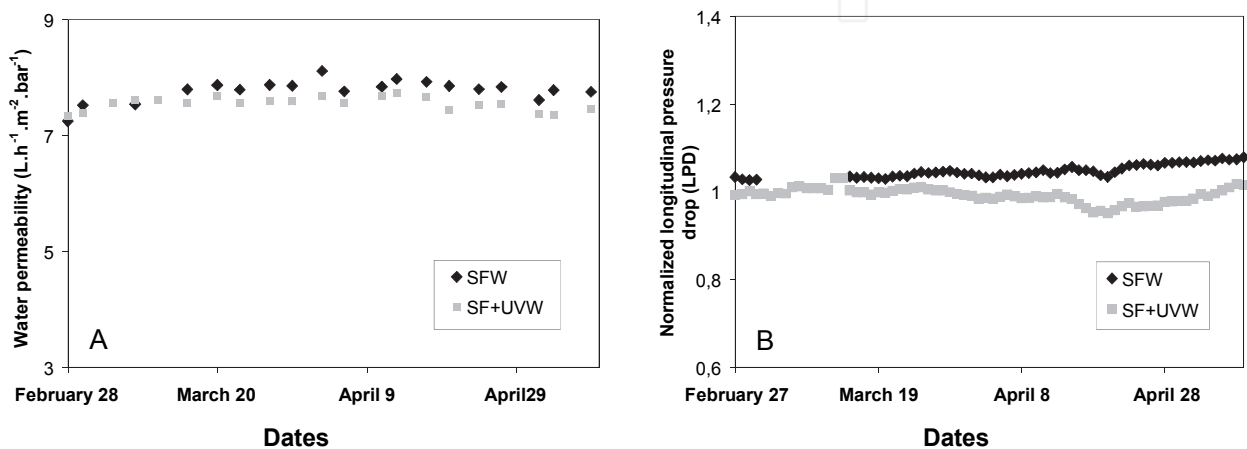

Fig. 3. NF module water permeability (A) and longitudinal pressure drop (B) evolution during filtration. Sand-filtered water with (SF + UVW) and without (SFW) UV pre-treatment.

3.3 Autopsy results: Characterization of the biofilm
3.3.1 Dry weight, protein content, microbial activity and wettability of the deposit

Table 1 gives mean values and standard deviations for the dry weight, protein content, microbial activity (ATP concentration) and wettability of the deposit. Dry weight, protein concentration and microbial activity of the membrane deposit were lowered after UV pre-treatment indicating that UV pre-treatment limited noticeably the formation of the deposit on NF membranes. The difference between the water contact angles of the module fed SFW and the module fed SF + UVW was in concordance with the other data: UV pre-treatment lowered the water contact angle; the membrane surface was more wettable. This may be linked to the decrease of the quantity of exopolymeric substances covering the surface of the membrane; therefore the water contact angle came closer to its initial low value (between 15 and 30°).

Parameter	Unit	SFW	SF + UVW
Dry weight	µg/cm²	102.3	40.7
Protein concentration	µg/cm²	1.15 ± 0.09	0.34 ± 0.03
ATP concentration	pmol/cm²	0.40 ± 0.18	0.16 ± 0.12**
Water contact angle	degrees	83.4 ± 7.6	74.7 ± 4.3*

Table 1. Characterization of the foulant matter deposited on NF modules during filtration. Sand-filtered water with (SF + UVW) and without (SFW) UV pre-treatment. *, $P < 0.05$. **, $P < 0.01$.

3.3.2 Analysis of membrane foulants by ATR-FTIR

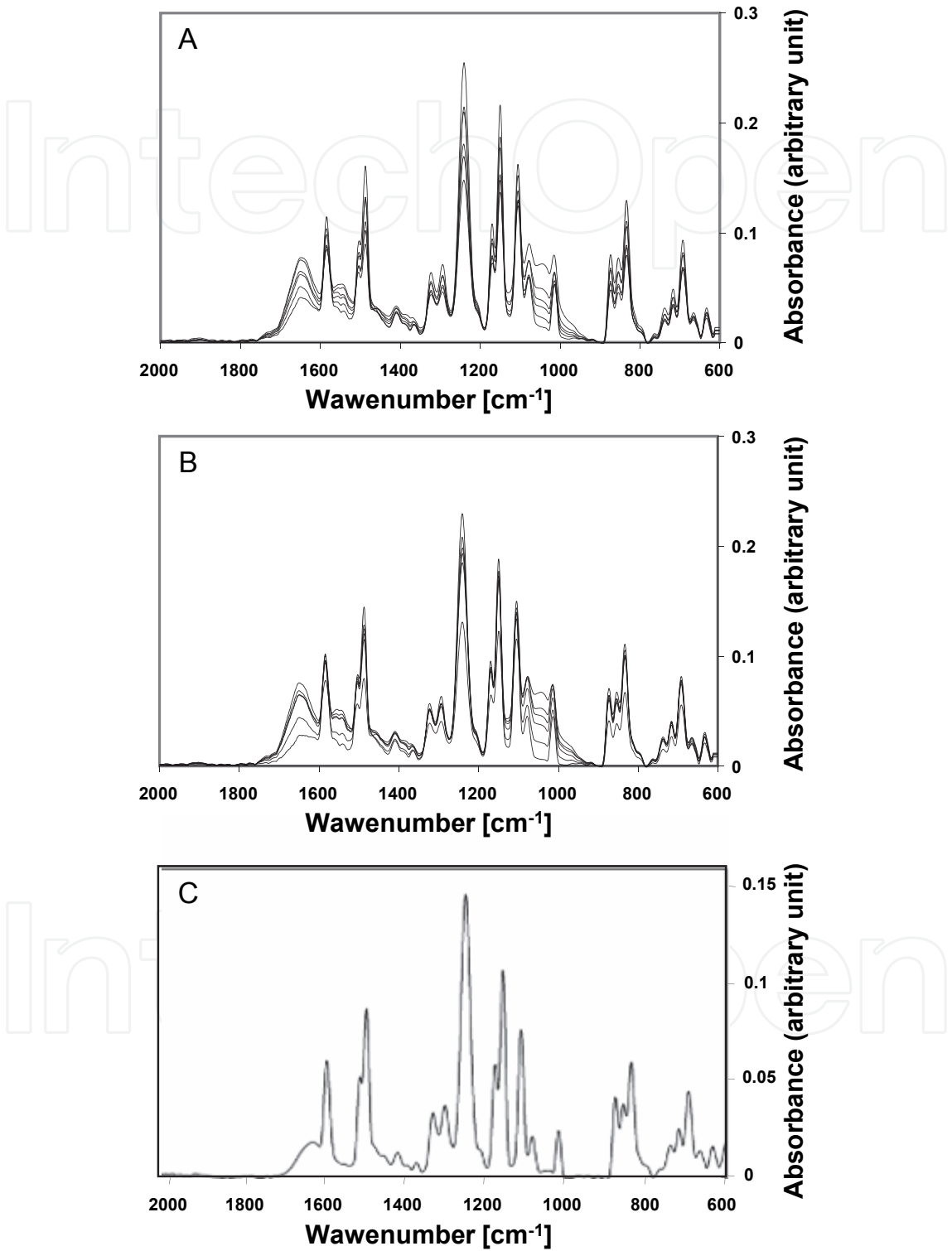IR spectra acquired on the surface of NF modules are presented on Figure 4.

Fig. 4. IR spectra of the foulant matter deposited on NF modules. A, Sand-Filtered water (SFW) without UV pre-treatment. B, Sand-Filtered water with UV pre-treatment (SF + UVW). C, virgin membrane.

Different spectra illustrating heterogeneity among the foulant layer are presented for each fouled membrane. Signals corresponding to the presence of proteins (signal at 1650 cm^{-1}) and exopolysaccharides (signal at 1040 cm^{-1}) were observed on the two modules. Many IR bands corresponding to the membrane were detected for all the spectra, indicating only partial coverage of the membranes by the fouling layer. The membrane IR signal near 700 cm^{-1} was used to calculate relative ratios for the proteins and polysaccharides signals to compare semi-quantitatively the abundance of exopolymers on the different NF elements. Table 2 indicates the mean values and standard deviations of these ratios.

Relative IR signal ratios	SFW	SF + UVW
Amide (I) / membrane[a]	0.87 ± 0.22	0.74 ± 0.25**
Polysaccharides / membrane[b]	0.52 ± 0.22	0.44 ± 0.29*

Table 2. Relative IR signals measured at the surface of NF modules. Sand-filtered (SF)-water, with (SF + UVW) and without (SFW) UV pre-treatment. a, Amide (I) / membrane is the ratio of signal intensities at 1650 cm^{-1} and 700 cm^{-1}. b, Polysaccharides / membrane is the ratio of signal intensities at 1040 cm^{-1} and 700 cm^{-1}. *, $P < 0.05$. **, $P < 0.01$.

The relative signal of amide (I), characterizing the amount of proteins, and the signal near 1040 cm^{-1}, revealing the amount of polysaccharides, were significantly lowered for UV pre-treated waters. FTIR-ATR spectroscopy showed that the exopolymers amount on the membrane surface was decreased when the feed water was UV-irradiated.

3.3.3 Microscopy

Microscopic observations made on NF membrane surfaces are illustrated on Figures 5 and 6. Each micrograph presented corresponds to one area of the corresponding membrane after the observation of at least 5 distinct areas at a magnification of x400 and 10 distinct areas at a magnification of x630 randomly distributed at the membrane surface. The snapshots presented on Figure 6 illustrations are showing the tendency of the fouling extent.

The analysis of the foulant matter after DAPI staining showed that the membrane surface was colonized by many microorganisms organized as microcolonies (Figure 5). Lectin staining revealed the presence of exopolysaccharides on the surface, with a highly heterogeneous repartition. Some areas of the membrane surface were covered by important amounts of microorganisms and polysaccharides, while others were still virgin and did not present any microbial cells (Figure 5 and 6). Microorganisms and exopolysaccharides were organized as a biofilm highly structured in three dimensions clusters. The most intense signal for membranes fed SFW or SF + UVW was obtained with the LEA-FITC lectin.

The cell concentration on the surface of the membrane was decreased when the feed water was UV-irradiated (SF + UVW). DAPI total direct counts indicate a bacteria concentration of $1.07 ± 0.4 \times 10^6$ cells/cm^2 with SF + UVW as feed water and $2.20 ± 1.17 \times 10^6$ cells/cm^2 with SFW as feed water. As illustrated on Figures 5 and 6, the amount of polysaccharides on the surface appeared to be lower after UV irradiation. Moreover, UV irradiation seemed to increase the heterogeneity of the deposit: the distinction between colonized areas and virgin areas, already visible with SFW as feed water, is yet stronger with SF + UVW.
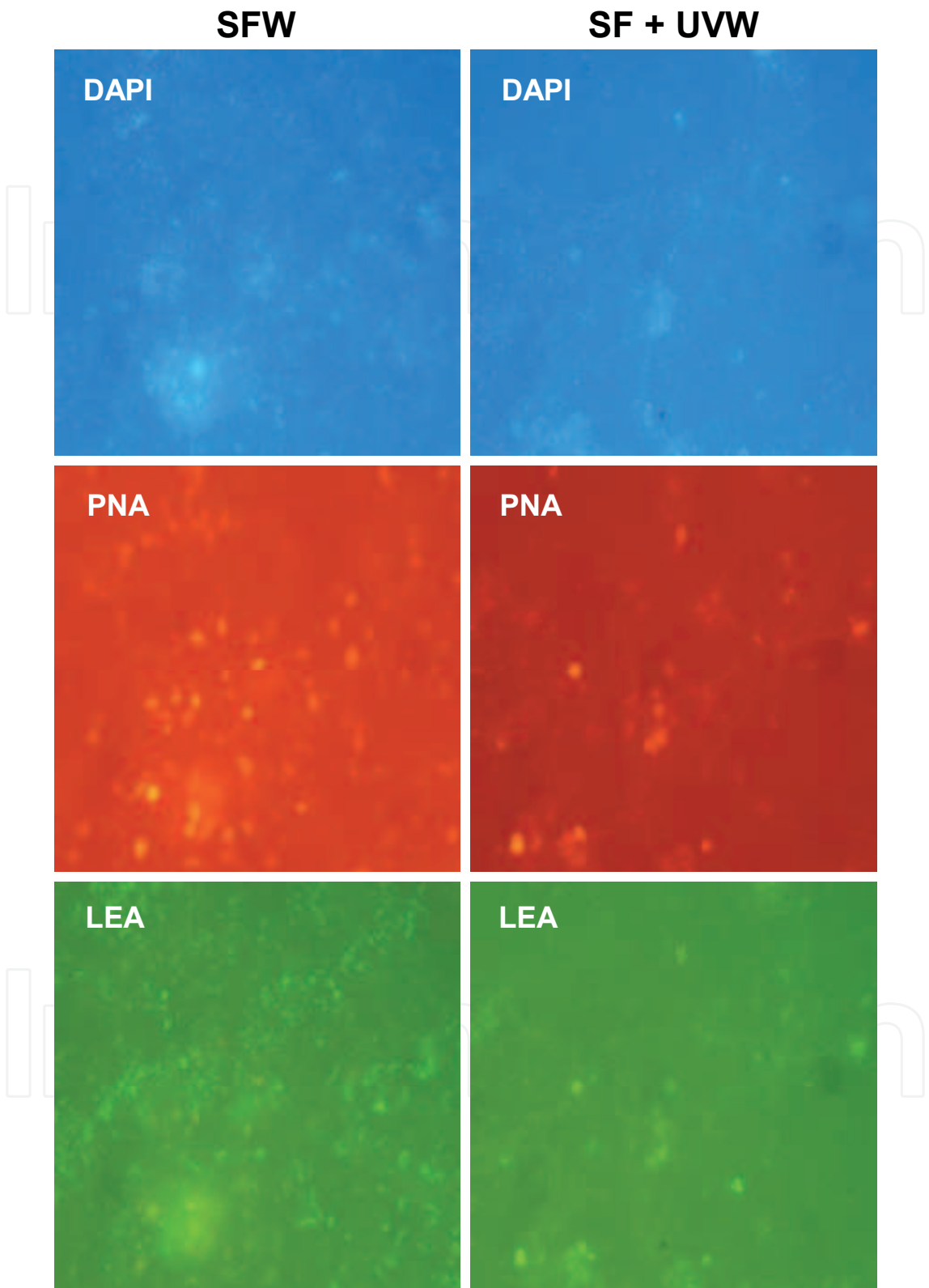
Fig. 5. Interactions between fluorescently-labelled lectins and fouled membranes, as visualized by epifluorescence microscopy. Sand-filtered (SF)- water, with (SF + UVW) and without (SFW) UV pre-treatment. PNA, TRITC-labelled peanut agglutinin. LEA, FITC-labelled *Lycopersicon esculentum* agglutinin. Magnification, x 630.
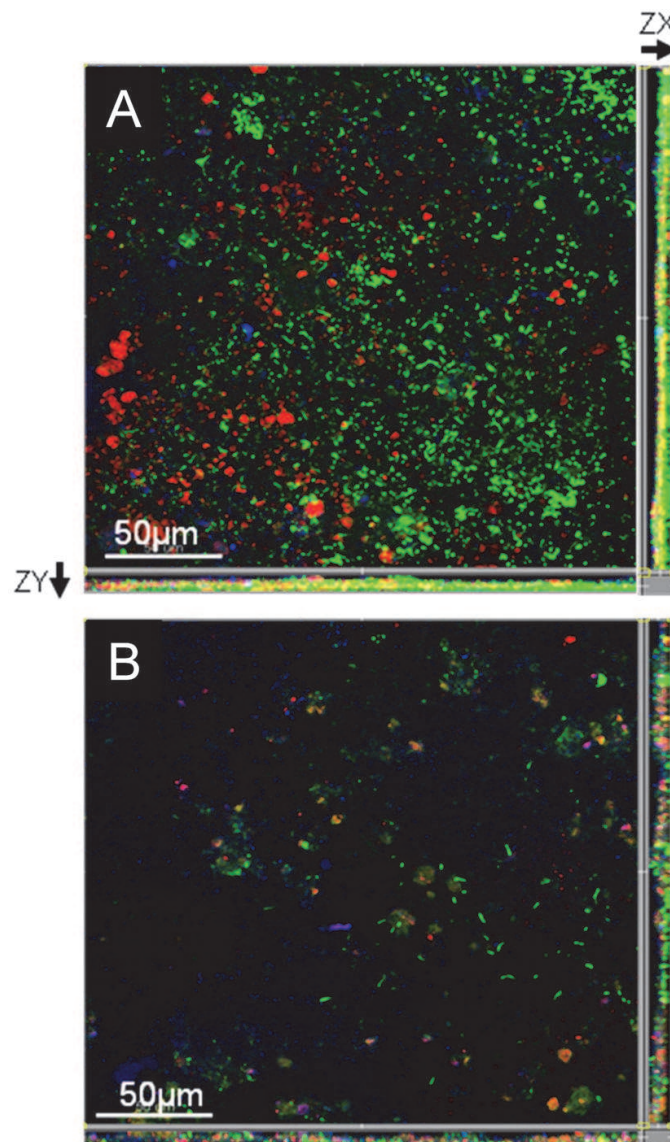
Fig. 6. Interactions between fluorescently-labelled lectins and fouled membranes, as visualized by CLSM. A, sand-filtered water without UV pre-treatment (SFW). B, sand-filtered water with UV pre-treatment (SF + UVW). Colour allocation: blue - DAPI, green - FITC-labelled *Lycopersicon esculentum* agglutinin (LEA), and red - TRITC-labelled peanut agglutinin (PNA). Magnification, x 630. Black arrows indicate the orientation of the foulant matter from the top to the bottom.

4. Discussion

The goal of this work was to study the influence of a UV irradiation pre-treatment located upstream from nanofiltration modules, on the biofouling of NF membranes at a pilot scale. The efficiency of UV pre-treatment to control biofouling was assessed with feed water obtained after coagulation/sedimentation/sand filtration.

We first measured the effects of UV irradiation on feed water parameters. UV pretreatment had no effect on the concentration of available dissolved carbonic microbial nutrients, since the DOC and BDOC concentrations were not modified by irradiation. UV treatment with a

low pressure mercury lamp (UV intensity varying from 10 mWs cm^{-2} to 240 mWs cm^{-2}) has been shown to be able to induce the decomposition of humic substances (Li et al. 1996, Lund et al. 1994). These effects of UV irradiation on humic acids have been observed for DOC concentrations of at least 15 mg C/L. The apparent discrepancy between the present results and the literature may be related to the relatively low concentrations of DOC and BDOC in the feed water in our experiments (less than 4 mg C/L). In the present study, UV irradiation decreased bacterial viable counts by 0.3 log. This may be related to the well-established mechanism of UV disinfection, i.e. bactericidal and bacteriostatic effect of UV mainly through genetic damages induction (Gaid et al. 2006, Al-Adhami et al. 2007). This effect of UV irradiation can be potentialized by other pretreatments decreasing BDOC like ozonation/GAC adsorption since dissolved organic matters are necessary to repair cell damages (Alkan et al. 2007, Camper 2004).

The second part of this work measured the effects of UV irradiation on the fouling of NF membranes, i.e. evolution of the filtration performances and formation of the deposit at the membrane surface. The analysis of the foulant matter by complementary technical tools showed that UV pre-treatment decreased the deposit formation. The global quantity of foulant matter and all the biofilm parameters measured were decreased when UV pre-treatment was applied: the total direct bacteria counts, viable bacteria counts, microbial activity (ATP concentration), and the concentration of exopolymeric substances (proteins and polysaccharides) were reduced at the membrane surface after UV irradiation. Nevertheless, UV pre-treatment did not modify the evolution of the permeability and of the longitudinal pressure drop. This may be linked to the short duration (10 weeks) of the test. Biofouling of nanofiltration membranes has been associated with a LPD-increase (Characklis and Marshall 1990, Vrouwenvelder et al., 2000, 2008) and/or a permeability decrease (Speth et al., 2000, Marconnet et al. 2009; Vrouwenvelder et al., 1998, 2009). Water permeability is a physical parameter which is related mainly to the material structure, to the porosity of the deposit, to its wettability, and to a lesser extent to its global quantity (Herzberg and Elimelech 2007). We did not observe any relation between membrane surface wettability and membrane permeability. Despite different evolutions of the membrane surface wettability of the two modules, the water permeability was the same.

On the whole, this work shows that controlling the concentration of active bacteria in the feed water is efficient to reduce deposit, biomass and biopolymer accumulation on the membrane surface. In this study, the 0.3 log removal of planktonic bacteria induced by UV irradiation is strong enough to decrease significantly the biofilm growth on the surface of the membrane but not fouling, i.e. filtration performances decrease. A longer duration test may be necessary to obtain higher biofilm development at the membrane surface with effects on membrane performances, i.e. permeability decrease and longitudinal pressure drop increase.

5. Conclusion

UV irradiation used as a pre-treatment upstream from nanofiltration is able to:
- lower the concentration of viable planktonic bacteria in the feed water.
- reduce the global quantity of deposit, the sessile bacteria concentration and the amount of extracellular polymeric substances present on the surface of the membrane.

UV pre-treatment, by limiting biofilm development at the membrane surface during nanofiltration of surface water, may be able to control the membrane performances decrease

observed during long term operation. New experiments with longer filtration duration tests are needed to demonstrate that UV irradiation can help to maintain membrane performances as well as to limit biofilm growth at the NF membrane surface.
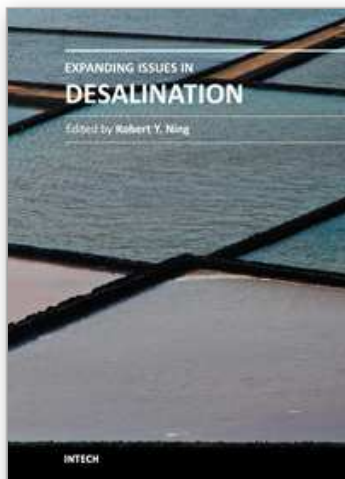
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Expanding Issues in Desalination
Edited by Prof. Robert Y. Ning

For this book, the term "desalination" is used in the broadest sense of the removal of dissolved, suspended, visible and invisible impurities in seawater, brackish water and wastewater, to make them drinkable, or pure enough for industrial applications like in the processes for the production of steam, power, pharmaceuticals and microelectronics, or simply for discharge back into the environment. This book is a companion volume to "Desalination, Trends and Technologies", INTECH, 2011, expanding on the extension of seawater desalination to brackish and wastewater desalination applications, and associated technical issues. For students and workers in the field of desalination, this book provides a summary of key concepts and keywords with which detailed information may be gathered through internet search engines. Papers and reviews collected in this volume covers the spectrum of topics on the desalination of water, too broad to delve into in depth. The literature citations in these papers serve to fill in gaps in the coverage of this book. Contributions to the knowledge-base of desalination is expected to continue to grow exponentially in the coming years.

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