

An innovative treatment concept for future drinking water production: fluidized ion exchange – ultrafiltration – nanofiltration – granular activated carbon filtration

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Received: 18 August 2008 – Published in Drink. Water Eng. Sci. Discuss.: 12 January 2009

Revised: 29 May 2009 – Accepted: 21 July 2009 – Published: 26 August 2009

Abstract. A new treatment concept for drinking water production from surface water has been investigated on a pilot scale. The treatment concept consists of fluidized ion exchange (FIEX), ultrafiltration (UF), nanofiltration (NF), and granular activated carbon filtration (GAC). The FIEX process removed calcium and other divalent cations; the UF membrane removed particles and micro-organisms; and the NF membrane and GAC removed natural organic matter (NOM) and micro-pollutants. This study focused on the prevention of fouling of the UF and scaling of the NF and investigated the overall removal of micro-pollutants by the treatment concept. The results of the experiments showed that in 14 days of continuous operation at a flux of 65 l/h m² the UF performance was stable with the FIEX pre-treated feed water without the aid of a coagulant. The scaling of the NF was also not observed even at 97% recovery. Different micro-pollutants were spiked in the NF feed water and their concentrations in the effluent of NF and GAC were measured. The combination of NF and GAC removed most of the micro-pollutants successfully, except for the very polar substances with a molecular weight lower than 100 Daltons.

1 Introduction

Conventional surface water treatments with coagulation, rapid sand filtration and granular activated carbon filtration are not able to remove all micro-pollutants from surface water. In order to sustain consumer confidence regarding the drinking water quality, the water companies in the Netherlands launched the Q21 project to reach an impeccable quality in the tap water in the 21st century (van Dijk and van der Kooij, 2004). In this study a new treatment concept is introduced: FIEX, UF, NF, and GAC (see Fig. 1). Based on the experience of the existing UF-NF concept, serious NOM fouling of the UF and scaling of the NF are expected. By removing positive divalent ions from the feed water, fouling can be controlled (Hong and Elimelech, 1997; Abrahamse et al., 2008). Scaling is caused by precipitation of over-saturated salts in water, such as calcium carbonate, so it can be theoretically controlled by removing divalent cations in

advance. To avoid clogging of the ion exchange process, a fluidized ion exchange process was used. Calcium and magnesium in drinking water are necessary for health benefit. Reducing them in the FIEX step is to solve the fouling of UF and scaling of NF (which can substantially enhance the lifetime of UF and NF membranes and thereby the investment cost). As far as the health benefit concern, we can simply place a marble/GAC mixed bed filter after NF for the refilling of calcium and magnesium in drinking water (marble filtration will be incorporated in future).

Fouling of the membrane is governed by the following mechanisms: pore blockage, and adsorption and cake layer formation (Makadissy et al., 2003; Katsoufidou et al., 2005). Many researchers have reported that the presence of calcium makes the fouling more serious and irreversible (Hong and Elimelech, 1997; Song and Singh, 2005). Their main hypothesis is that fouling is enhanced by the bridge effect of calcium ions. Because both the membrane and NOM are negatively charged, calcium can form bridges between NOM and the membrane. Calcium can also form bridges between two negatively charged NOM molecules (Kabsch-Korbutowicz et al., 1999; Seidel and Elimelech, 2002; Li and



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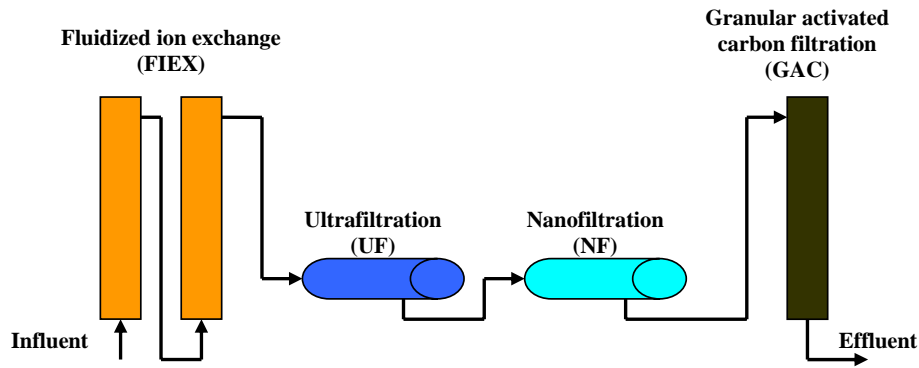


Figure 1. Flow scheme of treatment concept.

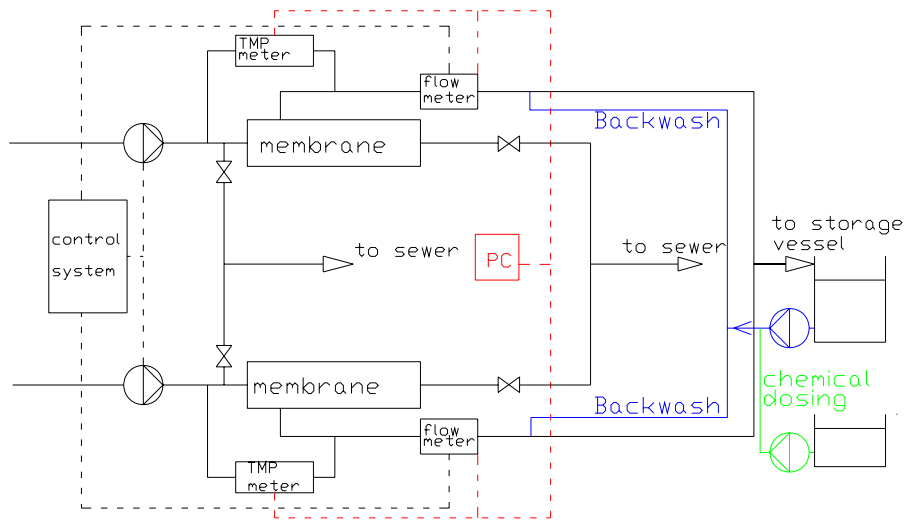


Figure 2. Flowscheme of ultrafiltration.

Elimelech, 2004). Removing calcium from the feed water is essential to avoid fouling of the UF membranes and scaling of the NF membranes.

Kweon et al. proved that sludge softening can significantly reduce organic fouling of UF (Kweon and Lawler, 2004). However, as sludge softening cannot completely remove calcium from the water, the remaining calcium is still a factor in UF fouling and NF scaling. In this study, we used IEX-softening as a pretreatment to obtain almost complete calcium removal in the feed of UF and NF.

This new treatment concept focuses on the drinking water requirement in future. The removal of micro pollutants present in surface water will be necessary. In that case, the NF is required although it is an expensive treatment step, since the traditional concept cannot reach a satisfying removal of micro pollutants. In this concept, a double barrier (UF-NF) for pathogens and particles and a double barrier (NF-GAC) for micro-pollutants were created. Without fouling and scaling problems, the UF and NF membranes can be

operated for a longer time, while the flux and recovery of NF can be increased, which results in a reduction of installation costs (Paassen et al., 2002). As NF removes most of the NOM, the pre-loading, pore blocking and site competition of GAC are reduced and a short contact time of three minutes is sufficient to remove the residual micro-pollutants after NF (Heijman and Hopman, 1999). All these benefits can reduce the total cost of this concept.

2 Materials and methods

2.1 Water source

Raw surface water from the Schie River in Delft, the Netherlands, was used as the water source. The water quality is listed in Table 1. Because this study focused on the effect of divalent ions and NOM on fouling and scaling, analyses of the feed water were mainly conducted on metal concentrations and dissolved organic matter (DOC). With weak acid cation resin, there should not be too much sodium introduced

Table 1. Water quality of Schie water and Schie water treated by FIEX.

Water quality parameter	Raw water	Effluent of FIEX
Ortho phosphate (mg/l)	0.65	0.56
Silicate (mg/l)	7.5	6.0
Sulphate (mg/l)	154	176
Aluminum ($\mu\text{g/l}$)	5.9	10
Barium ($\mu\text{g/l}$)	16	1.3
Calcium (mg/l)	150	<0.5
Potassium (mg/l)	18	9.4
Magnesium (mg/l)	26	<0.01
Manganese ($\mu\text{g/l}$)	0.99	13
Sodium (mg/l)	92	290
Iron (mg/l)	<0.05	0.1
DOC (mg/l)	15	16

in drinking water after FIEX. In Table 1, effluent of FIEX show a very high sodium concentration, but that is probably because the sample was taken at the early stage of this study, the dosage of NaHCO_3 for pH adjustment in FIEX column is not optimized yet (too much NaHCO_3 in the column). When the dosage is optimized, the sodium concentration in FIEX effluent should be about 100 mg/l, based on the theoretical calculation (4 mmol/l NaHCO_3 dosed).

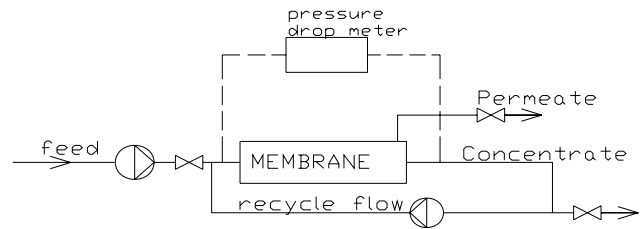
2.2 Fluidized Ion Exchange (FIEX)

The ion exchange process was in a fluidized bed mode. This system was composed of two columns in a series. Each column was 2.5 m high and its inner diameter was 19 cm. A freshly regenerated column was used as the final polishing column. In that way, the breakthrough of the first column was always removed by the second column.

The flow rate was controlled at 3.6 m/h. The pH value of the raw surface water was 8.1. After the ion exchange reaction the pH of the effluent of the first column was lower (around 4) because H^+ -ions were released from the resin. As the weak acid cation resin only works properly at a $\text{pH} > 4.8$, 4 mmol/l NaHCO_3 was dosed in the effluent of the first column to make the second polishing column work properly to increase the buffering capacity.

The resin used in this study was a weak acid cation resin: Amberlite IRC86, produced by ROHM&HAAS Company. The capacity of this resin is 4.1 eq/l. Since the content of the waste stream ($\text{Ca}(\text{NO}_3)_2$, $\text{Mg}(\text{NO}_3)_2$) probably can be used for agriculture, nitric acid was used for regeneration.

After one week of operation, the first FIEX column was saturated with calcium and needed to be regenerated. During the regeneration process only one column was used. Regeneration was carried out by on-line dosing with 60% concentrated nitric acid in demineralized water. The resin bed was brought in contact with the regeneration solution (5% con-

**Figure 3.** Flowscheme of nanofiltration.

centrated nitric acid) from the top of the column. The flow rate of the regeneration solution was 0.8 m/h. The pH of the waste stream was monitored, and the regeneration process was stopped when the pH of the waste stream was low (1–2). Demineralized water was used to rinse the residual nitric acid from the bed. After slow rinsing from top to bottom, an upwards-flow fast rinsing was carried out.

2.3 Ultrafiltration

The UF system, “Quick Scan”, was an automatic pilot-scale apparatus for capillary fiber modules, designed and constructed by Kiwa Water Research, the Netherlands. The system was operated in dead-end mode. Four membrane modules can be used at one time, and the total membrane area depends on the specific module. Quick Scan consists of two parallel centrifugal feed pumps, one centrifugal backwash pump and two chemical dosing systems, making parallel experiments and combined chemical backwash possible. In front of each centrifugal feed pump, there are two sieves (0.5 mm) to retain large suspended solids that can block membrane fibers (Heijman et al., 2007). The permeate of each module was collected in the same storage vessel. The trans membrane pressure (TMP) and flow rates of feed and permeate were measured with precision manometers (Endress & Hauser, Cerabar) and real-time recorded every eight seconds, and the respective graphs were plotted automatically as a function of time.

Capillary polyethersulfone membranes (UFC M5 0.8, X-Flow B.V, Enschede, The Netherlands) charged with polyvinylpyrrolidone and housed in a cylindrical plastic casing were used in the experiment. Two kinds of membrane modules with different surface areas were used, 2.4 m² and 1.3 m². The modules were potted with the same fibers (MWCO=200 kDa). Because of the limitation of capacity of the FIEX process, the module with a small surface area was used when the FIEX was used as a pretreatment; the module with a large surface area was used when in-line coagulation was used as a pretreatment or when there was no pretreatment.

The system worked automatically according to the settings in the computer program. The filtration cycle was: 15 min of filtration at a constant flux of 65 l/h m² followed by a one-minute backwash at a flux of 130 l/h m², and an enhanced

chemical backwash every five hours with NaOH at a pH of 12 (30-s backwash with the chemical, 15-min soaking time, 20-s normal backwash at a pH of 7).

Previous research indicated that fouling is higher if the pH is low (Hong and Elimelech, 1997). Because the carboxyl functional groups of the NOM molecules can be protonated with hydrogen ions in water, if the pH of water is low, there are more hydrogen ions available for protonation and consequently the NOM molecules become less negatively charged so that they are easier to deposit on the negatively charged membrane. The effluent of FIEX has a low pH (around 4), so 1.8 mmol/l NaOH was dosed in the feed tank to increase the pH of the UF feed to 7, which is close to the normal pH of natural water.

2.4 Nanofiltration

A pilot-scale apparatus, operated in cross-flow mode for the spiral wound membrane (Kiwa Water Research, The Netherlands) was used in this study.

A spiral-wound membrane with a surface area of 2.6 m² and a molecular weight cut off (MWCO) of 200 Da (2540-TS80-TSF, Trisep), housed in a cylindrical steel casing, was used in this study. The feed water was pressurized by a multi-impeller centrifugal pump (Grundfos CRNE-1), and part of the concentrate was circulated by a Verder-Iwaki MDH 25 magnet pump to maintain a high cross-flow velocity (0.12 m/s) in the membrane. Temperature was maintained at 20 Celsius degrees by a cooler during the experiment. Condumax W CLS21 sensors with a Liquisys M CLM253 transmitter (Endress and Hauser) were used for on-line monitoring of the conductivities of feed, concentrate and permeate. The pressure of the feed, concentrate and permeate was measured by precision manometers (Endress and Hauser).

The mass transfer coefficient (MTC), which defines the amount of produced permeate under unit time, area and pressure, was used to evaluate the scaling of NF. The calculation of MTC is normalized to temperature and osmotic pressure, as the following equation:

$$MTC = \frac{Q_p \cdot TCF_{MTC}}{A_{mem} \cdot TMP}$$

Where:

Q_p = discharge of permeate [m³/s]

A_{mem} = membrane area [m²]

TMP = trans membrane pressure [kPa]

TCFMTC = temperature correction factor for MTC [-]

2.5 Granular Activated Carbon (GAC)

The GAC system was a pilot-scale column (TU Delft, the Netherlands) with a bed height of 40 cm operated in filtration mode. ROW 0.8 supra GAC, from the Norit Company, was used in this experiment. The empty bed contact time was controlled at three minutes, corresponding to a flow rate of 8 m/h in the column.

2.6 Experimental procedure

The TMP graphs over time of two UF pretreatments (in-line coagulation and FIEX) were first compared at a constant flux (65 l/h m²) in short-term experiments (6 h). Four mmol/l Fe³⁺ was dosed for the in-line coagulation, whereas NaOH was dosed to increase the pH of the FIEX effluent. Next, after the short-term comparison, only FIEX-UF was tested over a long-term period (14 days) to check its stability at a constant flux (65 l/h m²), incorporating an enhanced chemical backwash with NaOH at pH of 12. Demineralized water was used for backwash in the long-term FIEX-UF experiment. After that long-term experiment, NF and a GAC filter were added to the process train. The effectiveness of calcium removal on NF scaling prevention was investigated at a flow of 100 l/h. The MTC of NF as a function of time was plotted at three different recoveries: 80%, 87% and 97%, taking into account the measured temperature, flow rate, conductivity and pressure.

In order to investigate the effectiveness of the NF-GAC double barrier for the removal of micro-pollutants, some micro-pollutants (pharmaceuticals and volatile solutes) were continuously spiked in the feed of the NF. Table 2 shows the spiked micro-pollutants and their concentrations. All pharmaceuticals were obtained from Sigma-Aldrich (St Louis MO, USA), except phenazon, ibuprofen, aminopyrine, carbamazepine and cyclophosphamide, which were provided by Acros Organics (Geel, Belgium). Samples of the NF-permeate and the effluent of the GAC filter were taken after certain time intervals (2 h, 4 h, a half day, 1 day, 2 days, 3 days) and sent to DVGW-Technologiezentrum Wasser (TZW) in Germany for measurements. The concentrations of pharmaceuticals were analyzed by high performance liquid chromatography with tandem mass spectrometric detection. The analysis of volatile solutes was performed with gas chromatography/mass spectrometry.

3 Results and discussion

3.1 Effect of pretreatment on UF fouling

In this section, the effect of calcium on the ultrafiltration fouling is evaluated. The raw Schie canal water was pretreated by FIEX. Table 1 shows the water quality of the raw water and the effluent of FIEX. After FIEX, the calcium concentration was around zero, while DOC was almost the same as the raw water, so there was no DOC removal in FIEX. Sodium concentration increased as a result of sodium bicarbonate and sodium hydroxide dosing. Most of the parameters were measured after a filtration with a 0.45 micron filter. The total concentration was only measured for silicate. There is no clear explanation for the increase in concentrations of sulphate, aluminum manganese and DOC. That was probably due to the residues after ion exchange regeneration. Because the ion exchange resin used in this study was regenerated

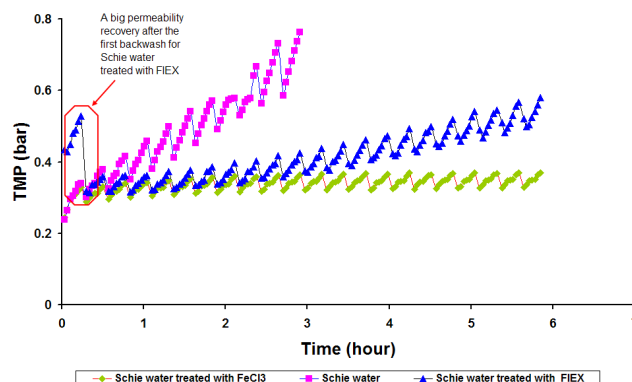
Table 2. Micro-pollutants used in this study and their spiked concentrations.

Micro-pollutants	Concentration _{feed} ($\mu\text{g/l}$)
Terbutaline	40
Salbutamol	3
Pindolol	50
Propranolol	100
Atenolol	50
Metoprolol	80
Sotalol	2.5
Clenbuterol	2.5
Phenazon	100
Aminopyrine	100
Carbamazepine	25
Cyclophosphamide	100
Pentoxifylline	100
Ibuprofen	30
Clofibric acid	100
Fenoprofen	100
Gemfibrozil	100
Diclofenac	5
Bezafibrate	50
Ketoprofen	50
Tert butyl alcohol (TBA)	100
Methy tert butyl ether (MTBE)	20
Ethyl tert butyl ether (ETBE)	20
Tert amyl methyl ether (TAME)	20

resin other than brand new resin, the residues from previous experiments, which were not completely flushed out during the rinsing step of regeneration, could get into the effluent of ion exchange columns.

The TMP as a function of time for three UF experiments with different pretreatments are plotted in Fig. 4. The exact square, triangle and rhombus lines correspond to direct ultrafiltration, FIEX pretreatment and Fe inline-coagulation, respectively. Since backwash recovered a certain amount of permeability in the membrane, the TMP of all lines increased during filtration and decreased after the backwash.

The difference in the three types of pretreatments becomes clear from Fig. 4. It was observed that the TMP at a constant flux ultrafiltration (65 l/h m^2) was stable when feed water was pretreated by Fe coagulation. The untreated surface water fouled the membranes rapidly while the fouling of the FIEX-pretreated water fell between the coagulated and the untreated waters. Because the removal of calcium from surface water reduced fouling in the UF membrane, calcium is probably a significant fouling factor, as mentioned in other publications (Hong and Elimelech, 1997; Wei and Zydney, 1999, 2000; Cho et al., 2000; Seidel and Elimelech, 2002; Kweon and Lawler, 2004; Li and Elimelech, 2004). The removal of calcium reduced the possibility of binding between the negatively charged NOM and the negatively charged membrane.

**Figure 4.** Short-term comparison of direct ultrafiltration, FIEX and in-line Fe coagulation at a flux of 65 l/h m^2 . 15 min filtration, 1 min backwash.

There was a large membrane permeability recovery after the first backwash for the UF with the FIEX pretreatment, which is probably due to air in the membrane module.

In-line Fe coagulation as a UF pretreatment is better than FIEX according to the short-term experiments. However, as was mentioned, in-line Fe coagulation will produce a large amount of Fe sludge during backwash. Furthermore, after a chemical cleaning of the UF membrane with HCl, dissolved Fe can enter the membrane pores, possibly leading to serious pore blockages in the long term.

FIEX is not as good as Fe coagulation in the short term, but it is still an effective pretreatment for ultrafiltration in the long term if combined with an enhanced chemical backwash. Figure 5 shows the results of the long-term experiment. The operational settings of this long-term experiment were the usual settings of UF treatment plants in the Netherlands. A TMP increase from 0.22 bar to 0.45 bar was observed in the first 50 h of operation. Afterwards, the TMP stabilized until the end of the experiment, except for the period between 80–100 h. A sharp increase in TMP in that period was observed, which was probably due to the insufficient enhanced chemical backwash. The pH of the backwash solution ($\text{pH}=10.4$) during that period was lower than the desired pH ($\text{pH}=12$) due to a problem with the sodium hydroxide dosing in the backwash water. At the end of this period this operational problem was fixed. The stable TMP during the long-term experiment indicated that fouling layer was still formed during filtration, but it can be controlled by the combination of FIEX pretreatment and usual cleaning. Seen in Fig. 5, the effect of calcium removal on UF fouling is proven again. Because both membrane and NOM are negatively charged, calcium (positively charged) can bridge them (Cohen-Stuart et al., 1991; Schäfer et al., 1998; Wei and Zydney, 1999; Cho et al., 2000; Wei and Zydney, 2000; Aoustin et al., 2001; Seidel and Elimelech, 2002; Kweon and Lawler, 2004; Li and Elimelech, 2004; Verliefe et al., 2006). Another possible explanation is that the repulsion force between the membrane and

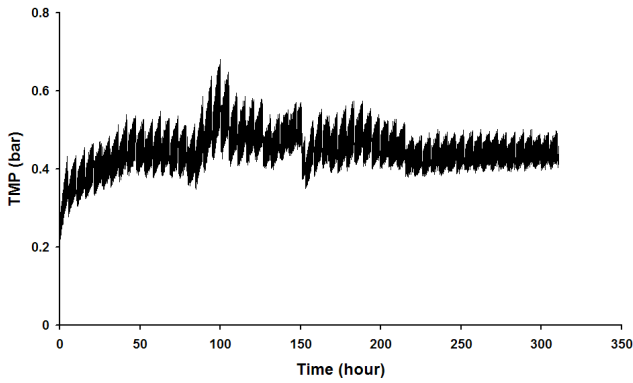


Figure 5. TMP variation tendency of 14 days of continuous ultrafiltration at 65 l/h flux, feed pretreated with Fluidized Ion Exchange.

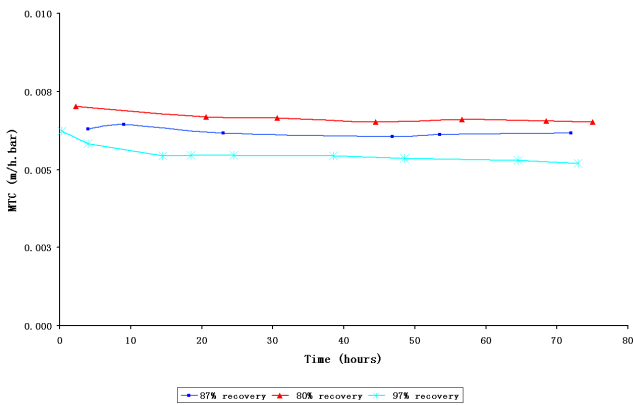


Figure 6. MTC as a function of time at different recoveries (80%, 87%, 97%). Feed was pretreated with FIEX and UF.

NOM was reduced and, therefore, decreased the efficiency of the hydraulic backwash (Hong and Elimelech, 1997).

3.2 Effect of calcium on NF scaling

Scaling occurs due to exceeding the solubility at high concentrations of dissolved ions. In this experiment the removal of calcium by FIEX was used to investigate whether high recoveries are possible. A UF pretreatment eliminates the suspended solid fouling of the NF membrane. Figure 6 shows the MTC of NF as a function of time at three different recoveries: 80%, 87% and 97%. As shown in the figure, the MTC of the NF membrane decreased with the increase of recovery. That is probably because of the NOM fouling at the beginning of each experiment which is influenced by the initial permeate flux. Some researchers reported that NOM fouling of nanofiltration increases with the initial permeate flux (Seidel and Elimelech 2002). However, the NOM fouling in this case did not develop further due to the high cross flow velocity, limiting the further formation of cake layer. After that, scaling was the dominating mechanism of the NF. Since the scalant (calcium) has been removed in the FIEX step, there

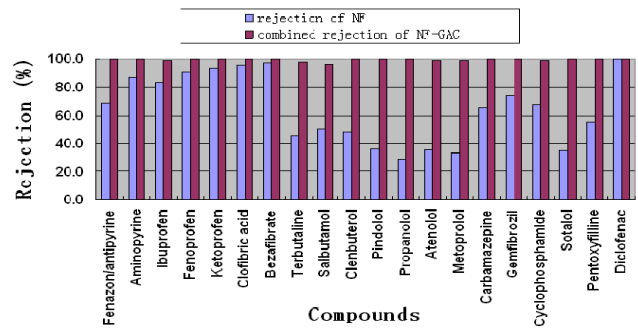


Figure 7. Pharmaceuticals' rejection of NF and the NF-GAC combination.

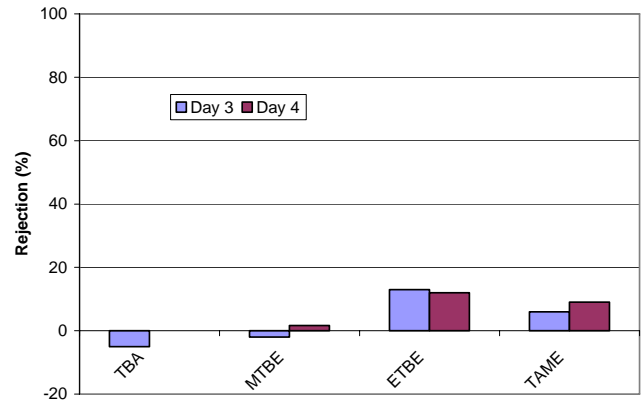


Figure 8. Rejection of micro pollutants after 3-day and 4-day nanofiltration.

was no obvious scaling in these three NF experiments, corresponding to the stable MTC, except for a short period of decrease at the beginning due to the NOM fouling.

3.3 Removal of micro-pollutants

In Fig. 7 the rejection of pharmaceuticals by NF alone as well as by the combination of NF and GAC is shown. Some pharmaceuticals are successfully removed by NF, while some of them are poorly removed by it. However, the combination of NF and GAC removes more than 99% of all tested pharmaceuticals. Therefore, this double-barrier treatment concept is effective for the pharmaceuticals' removal.

The removal of some other micro-pollutants (ETBE, TAME, MTBE and TBA), was also investigated. As shown in Fig. 8, ETBE and TAME were partly removed by NF, but there is almost no rejection of MTBE and TBA. That is probably because the molecular weight of MTBE and TBA is very small (<100 Da). However, GAC showed a good adsorption of ETBE and TAME, medium adsorption of MTBE, and no TBA adsorption at all. The reason for non-removal of TBA is that it is the most polar substance in this group. Although the MWCO of NF is 200 Da, the rejection also depends on the $\log K_{ow}$ of particles (Verliefde et al., 2006). At

high $\log K_{ow}$ (i.e., non-polar substances) substances with an MW smaller than 200 Da would pass through the NF; while at low $\log K_{ow}$ (i.e., polar substances) only substances with an MW smaller than 100 Da would pass through. Normally, substances with high $\log K_{ow}$ are non-polar, so even though these kinds of substances can pass the NF, they will be adsorbed by GAC. According to experimental results reported by Heijman and Hopman (1999), since NF removed most of NOM, the NOM pre-loading of GAC was reduced and consequently the running time of GAC could be considerably prolonged even up to a factor of 100 (Heijman and Hopman, 1999).

4 Conclusions

FIEX is a good pretreatment for UF with several advantages. By incorporating the enhanced chemical backwash, UF can maintain 14-days of stable operation at a constant 65 l/h m² flux. Therefore, it is a feasible option to replace in-line coagulation with iron or alumina coagulant. In this concept, no iron or alumina waste stream was produced during the backwash of the UF. What's more, FIEX pretreatment is important for scaling control in the NF and recovery can be increased to about 97%. This treatment concept successfully removed most of the target compounds: particles, natural organic matter, pathogens and micro-pollutants. Therefore, this new treatment concept is a very good concept for the surface water treatment as it provides two double barriers for the pathogens and micro-pollutants. However, there are still some substances that cannot be removed by this concept: the very polar substances with a molecular weight smaller than 100 Da, because these substances are neither rejected by the NF nor adsorbed by the GAC. Without calcium, there is no scaling in the NF; both flux and recovery can be increased and the NF cost can be reduced. Since the NF removes all NOM, the pre-loading of GAC is reduced. As a result, GAC can have a short empty bed contact time (EBCT) and a long running time. In that case, the regeneration cost and the investment cost for the GAC will be low.

Edited by: H. Y. Ng

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