

## OPTIMUM OPERATION CONDITIONS OF DIRECT CAPILLARY NANOFILTRATION FOR WASTEWATER TREATMENT

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### ABSTRACT

Direct Capillary Nanofiltration (Direct CNF) is a new technique used for surface water and wastewater treatment in one step without pre-treatment. The CNF membrane module combines the favourable cleaning properties of capillary ultrafiltration membranes with the favourable separation properties of nanofiltration membranes in terms of removal of DOC, colour, bacteria, viruses and pesticides.

Direct CNF was applied to Twente Canal surface water to produce high quality permeate in a single step. In this paper, Direct CNF has been applied (for the first time) for wastewater treatment under continuous and stable process conditions and to acceptable cost. An important aspect was to determine the optimum operation conditions of the pilot plant.

The optimum conditions for Direct CNF when applied to domestic wastewater are 15 minutes filtration time, 20 l/m<sup>2</sup>.h flux rate with undiluted domestic wastewater. The filtration can operate continuously under stable process conditions for approximately 8 hours. Subsequently, the pilot plant must be chemically cleaned due to contamination of the membrane fibres.

**Keywords:** Capillary nanofiltration, Wastewater, Optimum operation conditions, Flux rate, Filtration time, Hydraulic and chemical cleaning

### 1- BACKGROUND

Membrane technology is a new treatment method for producing water from contaminated sources. The membrane separates the contaminated feed water in a clean water flow, i.e. permeate and a concentrated waste stream.

The CNF membrane is a pressure-driven membrane with properties which are in between reverse osmosis (RO) and ultrafiltration (UF) membranes. CNF offers several advantages, i.e. low operation pressure, high flux, high retention of multivalent ions and molecular weight cut off for organic matter above 300 Dalton. The investment and operation and maintenance costs are relatively low [1].

Nanofiltration membranes have applications in several areas; one of the main applications has been in the drinking water treatment. NF can either be used to treat different kind of water types including ground water, surface water, and wastewater. Depending on the feed water quality a pre-treatment step will be necessary, e.g. with ultrafiltration. NF membranes have shown to be able to remove turbidity, micro-organisms and hardness, as well as fractions of the dissolved salts [2].

## **2- INTRODUCTION**

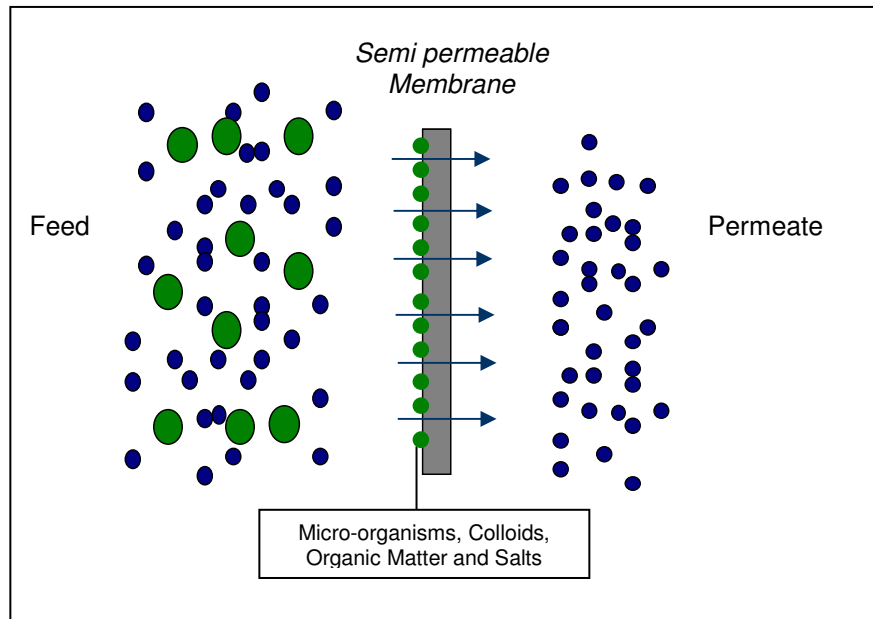
### **2-1. Membrane history**

The history of NF membrane dates back to the seventies, when reverse osmosis membranes with a reasonable water flux operating at relatively low pressures were developed [3,4]. Hence, the high pressures traditionally used in reverse osmosis resulted in considerable energy consumption. On the other hand, the quality of the permeate was very good, and often even too good. Thus, membranes with lower rejections of dissolved components, but with higher water permeability would be a great improvement for separation technology. Such ‘‘low-pressure reverse osmosis membranes’’ became known as nanofiltration membranes. By the second half of the eighties, nanofiltration had become established, and the first applications were reported [5,6]. From the very start, the drinking water industry has been the major application area for nanofiltration. The historical reason for this is that NF membranes were essentially developed for softening, and to this date NF membranes are still sometimes denoted as ‘‘softening’’ membranes [7,8].

### **2-2. Membrane filtration process**

Reverse osmosis (RO), the first membrane-based separation process to be widely commercialised, is a liquid/liquid separation process that uses a dense semi permeable membrane, highly permeable to water and highly impermeable to micro-organisms, colloids, dissolved salts and organic matter. Figure (1) is a schematic representation of the process.

A pressurized feed solution is passed over one surface of the membrane. As long as the applied pressure is higher than the osmotic pressure of the feed solution, ‘‘pure’’ water will flow from the more concentrated solution to the more diluted one through the membrane. If other variables are kept constant, the water flow rate is proportional to the net pressure [9].



**Fig. (1) Schematic representation of reverse osmosis process**

### **2-3. Nanofiltration concept**

Membranes in current nanofiltration installations are usually designed as spiral-wound elements and placed in pressure vessels. In order to use spiral-wound NF membrane elements a pre-treatment process is necessary in general. The pre-treatment depends on the composition of the feed water, i.e. the Membrane Fouling Index (MFI). An exception is the direct treatment of anaerobic ground water experienced by Vitens Water Company, The Netherlands (internal report).

In practice feed water with high suspended solids, membrane filtration will be part of a treatment plant consisting of various treatment steps, such as:

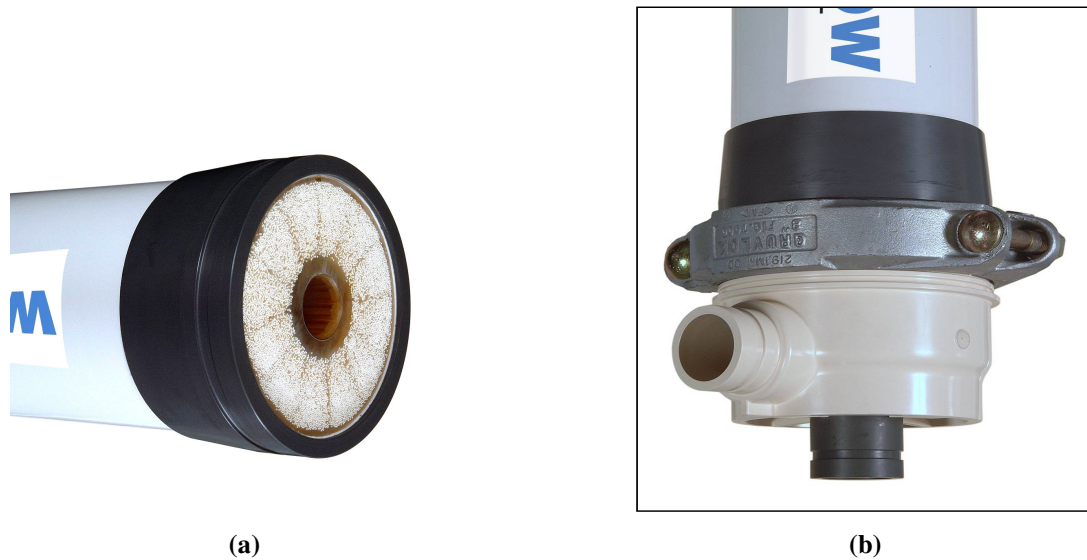
- flocculation/sedimentation + rapid sand/filtration + NF;
- (double) rapid sand-filtration + NF;
- (capillary) ultrafiltration + NF.

## **3- CAPILLARY NANOFILTRATION**

### **3-1. CNF module**

A capillary nanofiltration membrane combines the favourable properties of the capillary UF membranes in terms of ease of cleaning with the favourable properties of the NF membrane in terms of the removal of bacteria, viruses, pesticides and heavy metals. The capillary NF membrane is presently available in the well-known 8 inch modules which are also being used successfully for capillary UF (Fig. 2).

This new process enables the treatment of various water sources in a single step to produce high-quality water. The direct use of the NF process is referred to as "direct capillary NF". In a previous study [10] a new purification concept was introduced: direct capillary NF and its potential for direct treatment of surface water and effluent of a wastewater treatment plant was shown.



**Fig. (2) Capillary 8 inch NF module: (a) front view; (b) mounted with end cap**

### **3-2. Operation of CNF**

The capillary NF module is operated in the same way as semi dead-end UF (Fig. 3). During the production run the concentrate valve is closed and all the feed supplied to the system is withdrawn as permeate. In order to stabilise the flux and rejections at an acceptable level a small cross-flow velocity is applied over the module. If the rejection drops too much, the concentrate valve is opened and the system is flushed by means of air-enhanced forward flushing, a so-called AirFlush®. During this flushing procedure a backflush can also be carried out. Subsequently, the concentrate valve is closed and the next filtration run starts again.

### **3-3. Direct capillary nanofiltration for surface wastewater**

The capillary NF concept is developed further for surface water treatment by Dijkstra [11] was focussing on back flushing and chemical cleaning. Next, the removal rates for various components are studied by modelling and sampling the feed and permeate streams. Attention was paid especially to the membrane performance in comparison with the model. The final objective was to determine the operating conditions for a full scale plant. A part of the research was dedicated to the long term operation experiences of a fully-automated pilot installation with two 8 inch CNF modules.

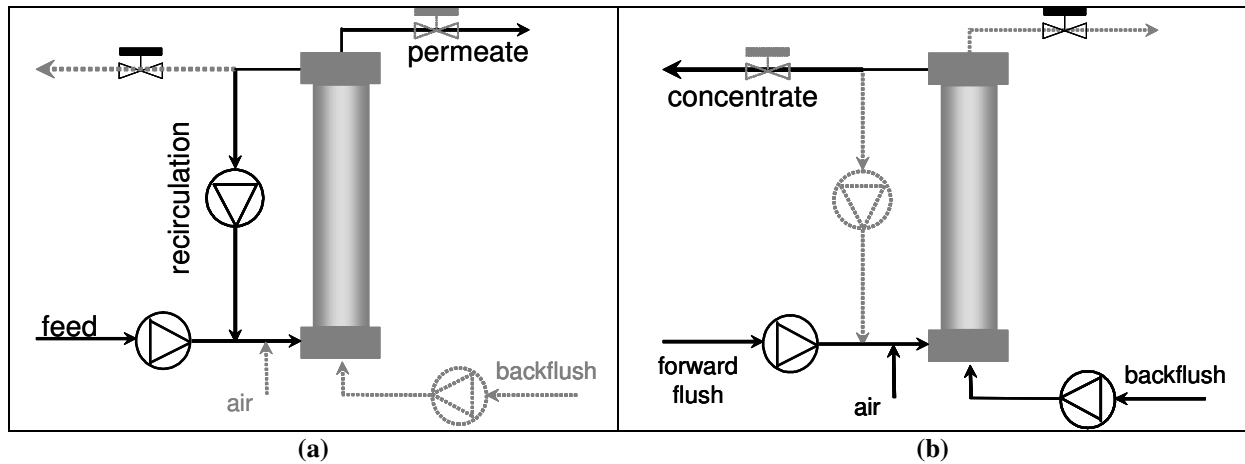


Fig. (3) Operation of capillary nanofiltration: (a) production; (b) cleaning

### 3-4. Direct capillary nanofiltration for domestic wastewater

Membrane technology can also be applied in the treatment of domestic wastewater, e.g. ultrafiltration membranes and membrane bioreactor (MBR). Even high quality water can be achieved by nanofiltration (NF) which membrane removes bacteria and viruses as well as pesticides, organic matter, heavy metals, and to some degree also salts. The permeate is of reliable quality and can be used for various purposes e.g. process water for industry.

NF is an efficient and ecologically suited technology for decontamination and recycling of wastewater generated in many industries [2], e.g. treatment of fish meal wastewaters [12].

NF reduces the organic load in the wastewaters and promotes the partial desalination, making water reuse possible. A major problem of the wastewater treatment is the water recovery rate, which should be as close as possible to 100 % [11].

## 4- EXPERIMENTAL

### 4-1. Continuous stable operation of CNF

This study focuses on continuous and stable operation of CNF. So acceptable rate between feed and permeate, flow and quality. To determine the optimum process conditions several parameters were studied.

### 4-2. Process parameters

During the period of January until July 2005 a study was carried out with a 2×4" CNF pilot plant. The studied process parameters were:

- **Feed concentration**  
Different dilutions of domestic wastewater with tap water were used (percentage of waste water: 25 %, 50 % and 100 %) with a filtration time 5 minutes and a flux of 20 l/m<sup>2</sup>.h. The Mass Transfer Coefficient (MTC) and Trans Membrane Pressure (TMP) were measured for each experiment.
- **Filtration time**  
Different filtration times were examined (5 minutes, 10 minutes, 15 minutes, 20 minutes and 25 minutes) with a flux of 20 l/m<sup>2</sup>.h and undiluted domestic wastewater (100 %). MTC and TMP were also measured for each experiment to determine the optimum filtration time.
- **Flux rate**  
Different flux rates were examined (10 l/m<sup>2</sup>.h, 20 l/m<sup>2</sup>.h and 30 l/m<sup>2</sup>.h) at 15 minutes filtration time with undiluted domestic wastewater (100 %). Again, MTC and TMP were measured for each experiment to set the optimum flux rate.
- **Hydraulic cleaning**  
Hydraulic cleaning takes place for about 4 minutes after every filtration time (e.g. 15 minutes). The hydraulic cleaning is a combination of forward flush, airflush®, back wash and rinsing of the circulation loop.
- **Chemical cleaning**  
The membrane filtration plant is operated continuously for a certain period, e.g. 26 filtration times resulting in a filtration run of approximately 8<sup>1</sup>/<sub>4</sub> hours. At the end of a filtration run the membranes must be cleaned chemically due to persistent (bio)fouling and/or scaling.  
The chemical cleaning is rather extensive and composed of various chemical agents, i.e. hydrogen peroxide, hydrochloric acid and sodium hydroxide in 5 successive steps. The complete chemical cleaning takes place in approximately 8 hours. The objective of chemical cleaning is to achieve the ideal membrane characteristics, i.e. Mass Transfer Coefficient (MTC) and Trans Membrane Pressure (TMP) as mentioned in the membrane specifications of the manufacturers (X-Flow Company).

### 4-3. Pilot installation

The applied 2×4" NF50 M10 capillary nanofiltration membranes were provided by X-Flow Company, the Netherlands. The membrane is a composite one of polyethersulfone coated with a polyamide thin layer. The membrane pilot installation receives the domestic wastewater after a 500 µm strainer to avoid particles damaging the membrane surface and/or clogging the membrane fibers itself. The concentrate will be treated in an anaerobic digestion tank as shown in Fig. (4).

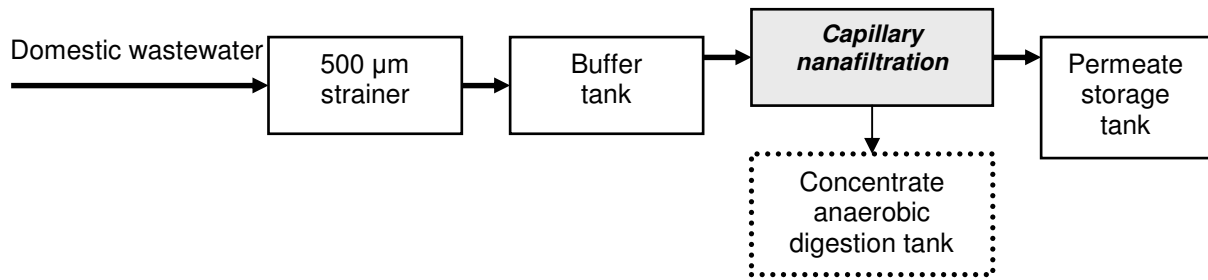


Fig. (4) Schematic diagram CNF pilot installation

## 5- RESULTS AND DISCUSSION

### 5-1. Integrity and retention of membrane modules

This paragraph illustrates the results of the experiment as described above.

In first instance the two new 4-inch membrane modules were subjected to an integrity and retention test, i.e. pressure test and magnesium sulphate test. These tests provide figures about possible broken membrane fibres and the retention for salts. No broken fibres were detected and the retention of  $\text{MgSO}_4$  was higher than 96 % for both membrane modules, which confirms the specifications.

### 5-2. Feed Concentration

To vary the feed concentration the domestic wastewater was diluted with tap water in three different concentrations; which are:

- 25% wastewater
- 50% wastewater
- 100% wastewater

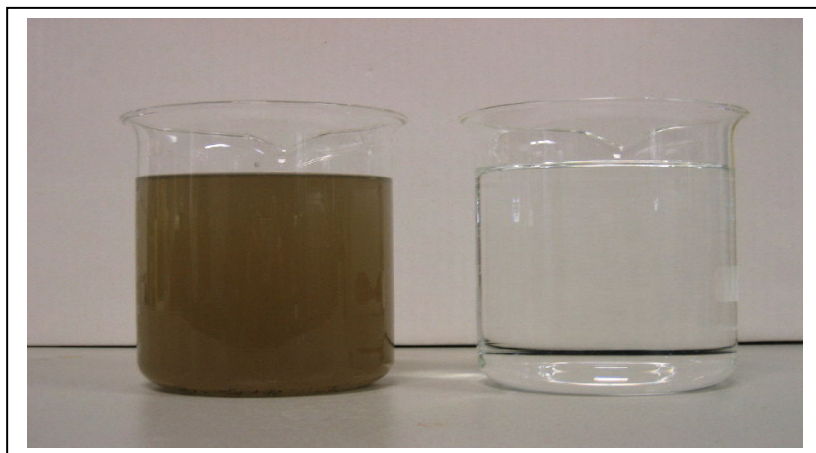


Fig. (5) Photo representing feed water and permeate water

For each concentration the CNF pilot installation was operated during 150 minutes (16 times filtration during 5 minutes) with a flux rate of  $20 \text{ l/m}^2 \cdot \text{h}$ .

Increasing the wastewater concentration, results in a mayor difference in TMP and MTC values at the beginning and at the end of each filtration run.

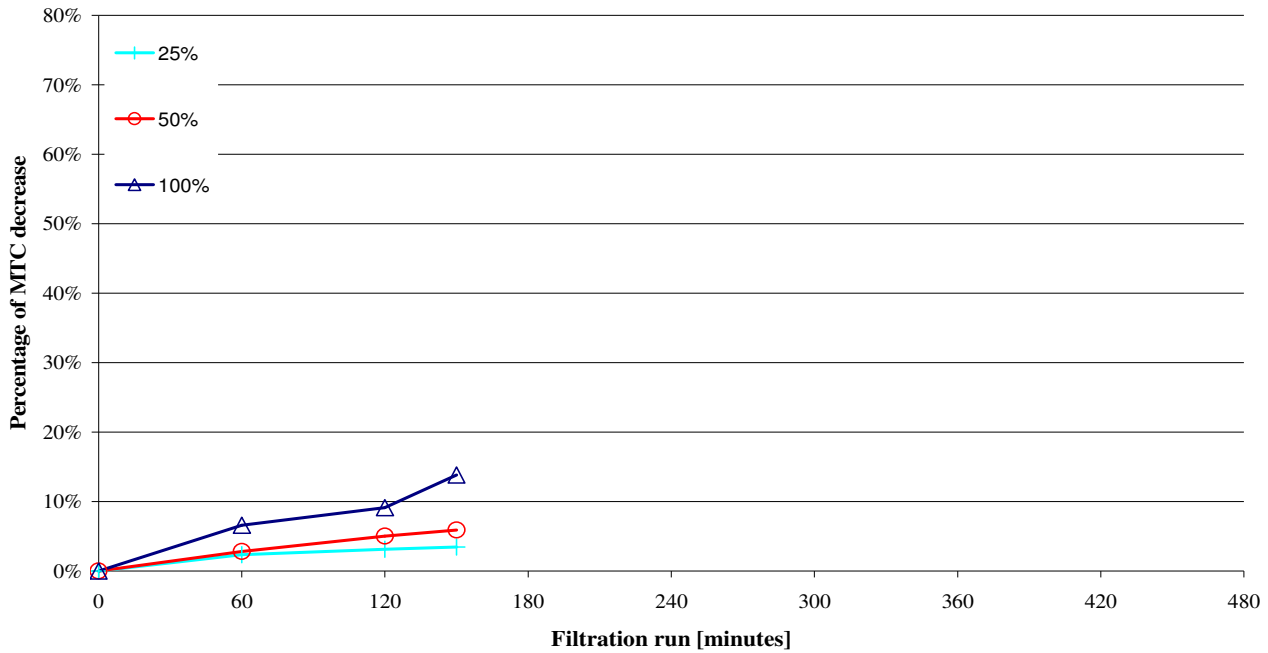
At the beginning of filtration the TMP values were 2.12 bar, 1.75 bar and 1.98 bar at 25 %, 50 % and 100 % wastewater concentrations respectively. The TMP increased at the end of the filtration run to 2.22 bar, 1.98 bar and 2.15 bar, resulting in a difference of 4.7 %, 13.1 % and 13.6 % increasing wastewater concentrations respectively (Table 1). On the other hand, MTC was decreased by  $0.44 \text{ l/m}^2 \cdot \text{h} \cdot \text{bar}$ ,  $0.84 \text{ l/m}^2 \cdot \text{h} \cdot \text{bar}$  and  $1.83 \text{ l/m}^2 \cdot \text{h} \cdot \text{bar}$  with increasing percentages of 3.5 %, 5.9 % and 13.8 % in case of 25 %, 50 % and 100 % wastewater concentration respectively (Fig. 6). This is attributed to increasing contamination of the membrane surface as a consequence of the higher wastewater concentration and also osmotic pressure.

**Table (1) The relation between TMP and MTC at different wastewater concentrations**

Filtration run	TMP (bar)			MTC ( $\text{l/h} \cdot \text{m}^2 \cdot \text{bar}$ ) at 20°C		
	25 %	50 %	100 %	25 %	50 %	100 %
0	2.12	1.75	1.98	12.72	14.15	13.22
15	2.13	1.79	2.02	12.71	14.09	12.65
30	2.13	1.80	2.07	12.63	13.89	12.42
45	2.15	1.81	2.09	12.54	13.79	12.24
60	2.16	1.83	2.10	12.42	13.75	12.35
75	2.17	1.86	2.11	12.37	13.73	12.34
90	2.17	1.89	2.12	12.35	13.65	12.26
105	2.17	1.92	2.13	12.33	13.56	12.16
120	2.18	1.95	2.15	12.32	13.44	12.01
135	2.20	1.97	2.23	12.30	13.38	11.73
150	2.22	1.98	2.25	12.28	13.31	11.39

Filtration time = 5 minutes and flux rate =  $20 \text{ l/m}^2 \cdot \text{h}$





**Fig. (6) Decrease in MTC values with time for each dilution of domestic wastewater**

A higher wastewater concentration results direct in a more pronounced MTC drop, as expected. The decrease in MTC value is more or less linear with the wastewater concentration, which can be expressed as the load rate of the membrane. If CNF modules are applied it will be possible to treat undiluted wastewater (100 %).

### 5-3. Filtration time

Different filtration times of 5, 10, 15, 20 and 25 minutes were examined with a flux of 20 l/m<sup>2</sup>.h using undiluted domestic wastewater (100 %) and a filtration run of 8 hours.

In case of 20 minutes and 25 minutes filtration time experiment, the system stopped at a filtration run less than 8 hours, due to high TMP values (more than 7 bar) (Table 2). Regarding the 25 minutes filtration time, the system stopped after 4 hours, while it stopped after 7 hours in case of 20 minutes filtration time. The extreme high TMP value was attributed to fast fouling of the membrane, with again a clear relation between loading rate\* on the membrane surface and TMP.

TMP values at the end of the filtration time at 5 minutes, 10 minutes and 15 minutes filtration time were less than 7 bar. After a filtration run of 8 hours the TMP increased with 3.91 bar, 4.08 bar and 1.69 bar at 5 minutes, 10 minutes and 15 minutes filtration times respectively (Table 2). The differences between MTC values in the beginning and at the end of filtration run were 7.36 l/h.m<sup>2</sup>.bar, 8.86 l/h.m<sup>2</sup>.bar and 6.44 l/h.m<sup>2</sup>.bar at 5 minutes, 10 minutes and 15 minutes filtration times respectively (Table 2).

At 5 minutes and 10 minutes filtration time the data are not accurate due to limitations of the pilot installation itself. If the filtration time is longer than 150 minutes the error is even more pronounced, so these data will not be taken in account.

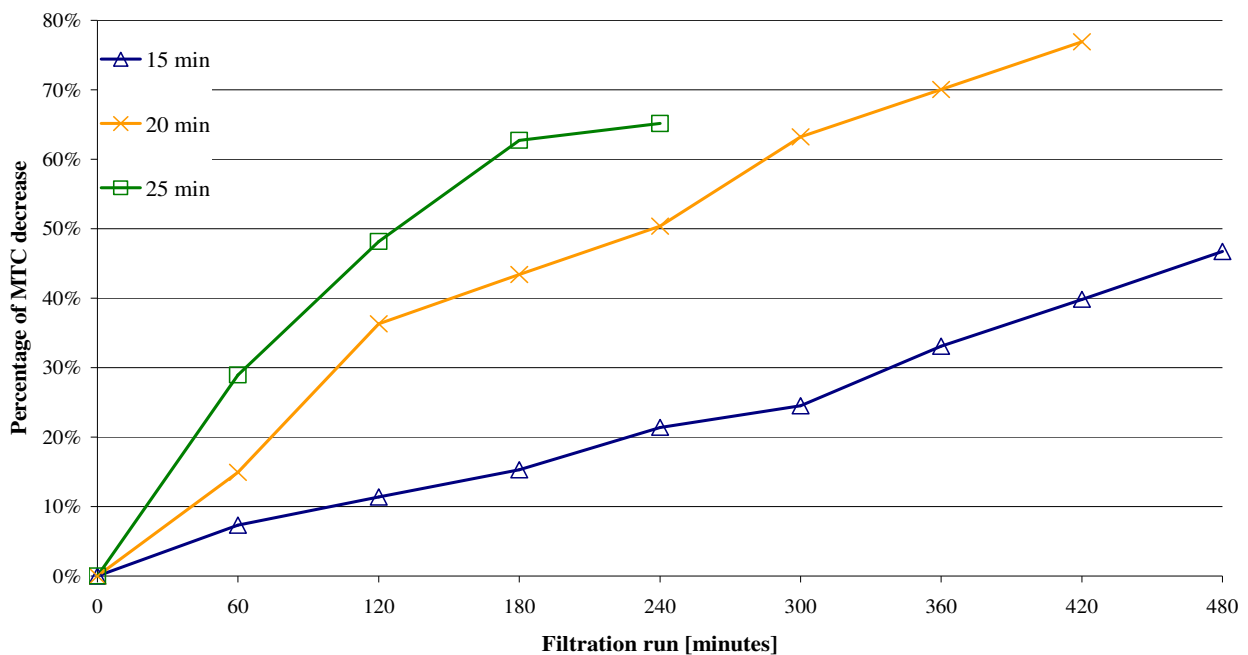
As shown in Fig. (7) 15 minutes filtration time had the lowest percent of MTC decrease and most stable operation during the filtration run.

Accordingly, 15 minutes filtration time seems to be the optimum between the pilot installation restrictions and the maximum load of wastewater on the membrane surface before chemical cleaning.

**Table (2) The relation between TMP, MTC and filtration run at different filtration times\***

Filtration run	TMP (bar)					MTC (l/h.m <sup>2</sup> .bar) at 20°C				
	5 min	10 min	15 min	20 min	25 min	5 min	10 min	15 min	20 min	25 min
0	1.98	1.88	1.82	1.98	1.90	13.22	13.12	13.78	13.38	12.71
60	2.10	2.41	1.99	2.44	2.92	12.35	10.75	12.77	11.38	9.03
120	2.15	2.73	2.11	3.17	4.02	12.01	9.95	12.21	8.52	6.59
180	2.44	3.11	2.35	3.56	6.09	11.09	9.05	11.67	7.57	4.74
240	2.76	3.52	2.48	4.08	7.01	10.60	8.18	10.83	6.64	4.43
300	3.48	3.92	2.55	5.25	-	9.48	6.86	10.40	4.92	-
360	4.44	4.50	2.66	6.10	-	8.22	5.99	9.22	4.01	-
420	5.17	5.21	3.00	7.10	-	6.92	5.11	8.29	3.09	-
480	5.89	5.96	3.51	-	-	5.86	4.26	7.34	-	-

Flux rate = 20 l/m<sup>2</sup>.h



**Fig. (7) Decrease in MTC values with time for each filtration time**

#### 5-4. Flux rate

It is important to determine the optimum flux rate. Different flux rates were examined, i.e. 10 l/m<sup>2</sup>.h, 20 l/m<sup>2</sup>.h and 30 l/m<sup>2</sup>.h with undiluted waste water (100 %), a filtration time of 15 minutes and a filtration run of 8 hours.

With respect to flux of 30 l/m<sup>2</sup>.h the system stopped after 3 hours and 20 minutes because of a too high TMP value (6.89 bar).

With respect to 10 l/m<sup>2</sup>.h and 20 l/m<sup>2</sup>.h flux rates, after a 8 hours filtration run the TMP value increased with 1.13 bar and 1.79 bar respectively (Table 3). While, MTC value decreased with 3.19 l/h.m<sup>2</sup>.bar and 6.44 l/h.m<sup>2</sup>.bar respectively (Table 3).

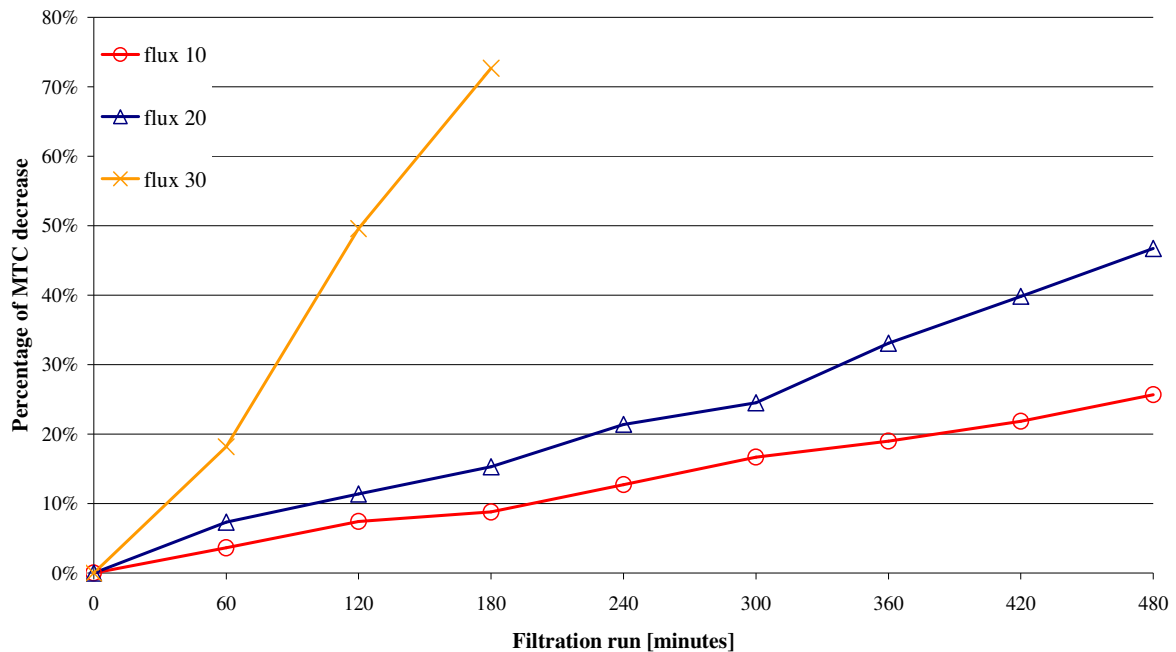
Also, flux rate of 10 l/m<sup>2</sup>.h had lower percentage of MTC decrease than 20 l/m<sup>2</sup>.h (Fig. 8).

A flux of 20 l/m<sup>2</sup>.h is acceptable; experiments during a longer period of operation will indicate the long term effect.

**Table (3) The relation between MTC, TMP and filtration run at different flux rates [l/m<sup>2</sup>.h]**

Filtration run	TMP (bar)			MTC (l/h.m <sup>2</sup> .bar)		
	flux 10	flux 20	flux 30	flux 10	flux 20	flux 30
0	1.83	1.82	1.88	12.41	13.78	14.24
60	1.97	1.99	3.36	11.96	12.77	11.65
120	2.08	2.11	5.09	11.49	12.21	7.18
180	2.17	2.35	6.89	11.32	11.67	3.89
240	2.24	2.48		10.83	10.83	
300	2.36	2.55		10.34	10.40	
360	2.56	2.66		10.05	9.22	
420	2.74	3.00		9.70	8.29	
480	2.96	3.51		9.22	7.34	

Filtration time = 15 minutes



**Fig. (8) Decrease in MTC values with time for each flux rate**

## 5-5. Cleaning of the membrane

To obtain continues and stable operation it is necessary to clean the membrane within certain intervals. The cleaning of the membrane will be based on the following methods:

- a-** Physical cleaning: It depends on mechanical forces to dislodge and remove contaminants from membrane surface. It includes forward flushing, reverse flushing, back washing, vibration, and air spurge [14].
- b-** Chemical cleaning: It is assigned to weaken cohesive forces between the contaminants and the membrane surface area. It is useful in biofouling and scaling of silica [15]. Either gases or liquid containing chemical agents can be used in chemical cleaning [16]. Combination of chemical and physical cleaning also can be applied.

### 5-5-1. Hydraulic cleaning

Every 15 minutes the membrane is cleaned hydraulically for about 4 minutes. There are three hydraulic cleaning modes.

- a-** Forward flush: It is based on rinsing the feed along the membrane surface area with high velocities. This applied for removing the particles on membrane sides.
- b-** Airflush: It is based on injection of air to the instillation and to provoke friction with the membrane surface area. It is used in combination with forward flush and only applied in vertically placed modules (as in this study).

- c- Backwash: It is based on treated water that flows from the permeate side to the feed side [17]. Therefore membrane must have high-pressure durability in both directions.  
The purpose of the backwash process is minimizing the overall hydraulic cleaning time [15].

**Table (4) Hydraulic cleaning procedure for CNF used in wastewater treatment**

<b>Program</b>	<b>Time (sec)</b>	<b>Feed flow rate (l/sec)</b>	<b>Permeate flow rate (l/sec)</b>
Forward flush	30	0.33	-
Flush dead piping	15	0.40	-
Forward flush	60	0.33	-
Backwash	75	0.40	0.02 – 0.04
End of Backwash	9	0.00	-
Airflush	12	0.30	-
Forward flush	30	0.33	-
Forward flush end	9	0.00	-
<b>Total</b>	<b>4 minutes</b>		

Application of CNF at real scales can be optimised to less than two minutes only, e.g. Elsbeekweg [11].

**5-5-2. Chemical cleaning**

The membrane cleaning efficiency is a function of multiple parameters such as hydrodynamic conditions, concentration and temperature of chemical cleaning solutions as well as sequence of cleaning. Some of the parameters such as pH and temperature have a strongly non-linear effect on cleaning effectiveness [14].

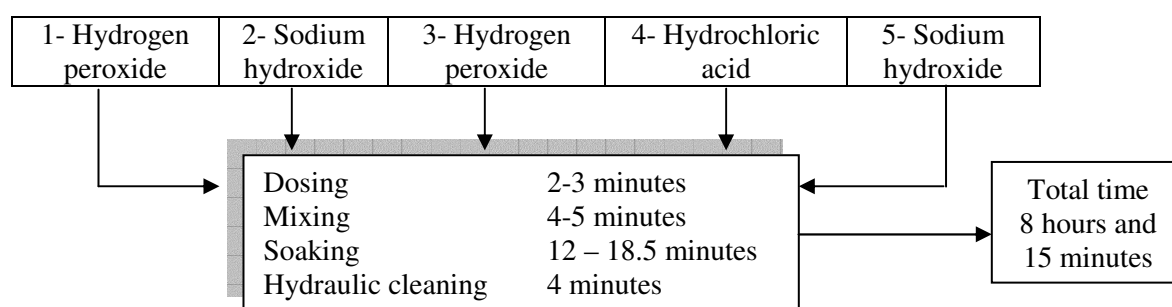
Soft cleaning agents, such as diluted hydrochloric acid, diluted sodium hydroxide and low concentration of hydrogen peroxide, must be used in the chemical cleaning regime of CNF membrane because usage of strong chemical agents may lead to damage of the membrane fibers. These soft chemical cleaning agents must have the ability to overcome the clogging of the membrane, which occurred mostly due to high loading rate of grease, fats and proteins in wastewater composition. Accordingly, chemical agents must have high efficiency in removal of such compounds to tackle (bio) fouling of the membrane.

Chemical cleaning is used from time to time at the end of the filtration run to remove the fouling and/or biofouling. The initial chemical cleaning regime was designed in cooperation between Vitens Drinking Water Company and X-Flow Company, applied in Elsbeekweg surface water treatment plant. The chemical cleaning regime consisted of hydrogen peroxide; hydrochloric acid and sodium hydroxide in 5 successive steps

with total duration of about 8 hours. The sequence of the chemical cleaning procedure is shown in Table 5.

Hydrogen peroxide is used as disinfectant as well as oxidizing agent. Hydrochloric acid removes possible scaling and causes shrinking of the membrane fibres. Sodium hydroxide is the most effective cleaning agent. It removes organic matter from the surface of the membrane on the other hand; it relaxes the fibers of the membrane.

**Table (5) Time table of chemical cleaning steps**



## 5-6. Optimized cleaning procedure

The aim is to optimise this chemical cleaning to increase the filtration time and accordingly, increasing the recovery. Theoretically, optimization of the chemical cleaning regime can be reached either by removing a chemical agent used or by shortening mixing and soaking times. Also, addition of other chemical agents can increase the efficiency of the chemical cleaning regime like enzymatic surfactant free agents, e.g. ultrasil derivatives.

Removing one of the chemical agents will shorten the chemical cleaning regime procedure and accordingly, increase the filtration time / chemical cleaning time ratio.

Mixing and soaking times can be shortened to 1 hour totally. Accordingly, this decrease the chemical cleaning time to 5 hours and this leads also to increasing the filtration time / chemical cleaning time ratio.

New chemical agents such as ultrasil can be used to optimize the chemical cleaning procedure to reach the same MTC value after every chemical cleaning.

## 6- CONCLUSIONS

The conclusions from this paper are:

- 1- It is technically feasible that raw domestic wastewater can be treated by CNF with 15 minutes filtration time and 20 l/m<sup>2</sup>.h flux rate to produce high purified water.

- 2- Apparently, from figures 6, 7 and 8, it is possible to repeat this process with certain range.
- 3- There is fluctuation between MTC start points of filtration runs between 12.41 l/m<sup>2</sup>.h.bar and 14.24 l/m<sup>2</sup>.h.bar after chemical cleaning on short term.
- 4- The initial cleaning procedure from surface water treatment in Elsbeekweg is not economically applicable for domestic wastewater treatment in this case.
- 5- The economical feasibility should be improved by optimizing the chemical cleaning procedure which will result in high (filtration time / chemical cleaning time) ratio.
- 6- The characteristics of domestic wastewater and biofouling of the membrane can be related not only by quantity but also by quality.
- 7- Recovery can reach 62.5 % maximally.

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