CIE4485 Wastewater Treatment
Lab experiments

Ultrafiltration of effluent

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Challenge the future

1 Objective

In the Netherlands interest in advanced wastewater treatment is increasing as almost all treatment plants apply full biological treatment and nutrient removal. Membrane filtration, especially ultrafiltration, is believed to be a very promising method for effluent polishing. After ultrafiltration the water can be used in industrial applications, for agriculture or in the urban water cycle. A better effluent quality has a positive impact on the quality of the receiving water.

2 Theory

Membrane filtration is a separation technology where a component in one phase is separated from the other phase by a membrane. The membrane is a selective barrier between two phases. The membrane may discriminate between two types of components because of differences in size, shape or chemical structure, but it will not be able to separate a mixture completely. A certain amount of energy is required to accomplish the separation.

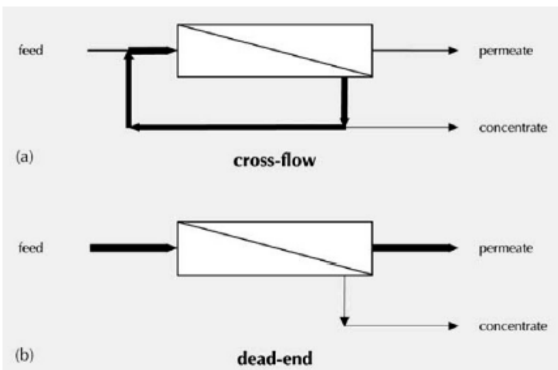

Figure 1. Schematic representation of cross-flow (a) and dead-end filtration (b) (Roorda, 2004)

In membrane filtration processes the phase is liquid, mostly water, and the components can be colloidal, suspended or dissolved. The driving force in this process is a (hydraulic) pressure difference over the membrane. Pressure driven membrane processes can be distinguished into 4 categories, which are dependent on the particle size of the solute and consequently on the membrane structure. The properties of the 4 different processes are shown in table 1.

Table 1 Membrane filtration processes.

Membrane process	Pore diameter (μm)	Applied pressure (bar*)
Microfiltration	0.1 - 1.0	0.05 - 0.1
Ultrafiltration	0.01 - 0.1	0.1 - 1
Nanofiltration	0.001 - 0.01	1 - 10
Reverse Osmosis	< 0.001	10 - 30

* 1 bar = 10^5 Pa

For further explanation about membrane filtration fundamentals, see Van Dijk *et al*.

The total filtration resistance is often expressed as the sum of membrane resistance and the additional resistance from fouling.

Dead-end ultrafiltration operation consists of subsequent filtration, backflush and chemical cleaning steps. During filtration the retained components will foul the membrane and create an increase in filtration resistance over time (or filtrated volume). A good filterability means that the increase of resistance is small. Reversibility is described as the extent to which the filtration resistance is returned to the original value after applying a hydraulic cleaning. If the filtration resistance after backflush is equal to the filtration resistance at the start as the previous filtration period, than the fouling is considered to be completely reversible. Both terms, filterability and reversibility are relates to the properties of the feedwater, membrane material and operational conditions. When the amount of irreversible fouling becomes too high, of after a predetermined amount of backflush cycles, the membrane is chemically cleaned. Ideally, chemical cleaning removes all the irreversible fouling from the membrane.

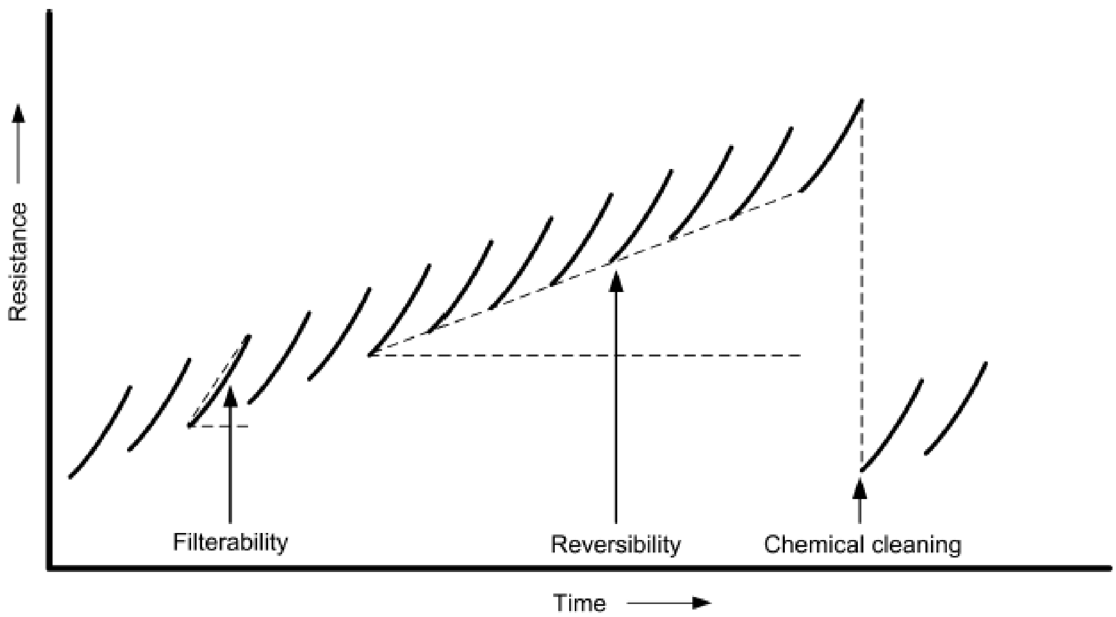

Figure 2. Typical filtration curves illustrating filterability, reversibility and chemical cleaning (Janssen, 2011)

Roorda (2004) developed a parameter for evaluation of filtration characteristics during dead-end ultrafiltration of WWTP effluent and proposed the Specific Ultrafiltration Resistance (SUR) as a parameter. This parameter provides useful information about the filterability of WWTP effluent and can be measured in a short period. The SUR is described as the cake layer resistance per unit of filtered feedwater per m^2 membrane surface and is expressed in m^{-2}. Low SUR values indicate relatively high filterability, whereas high SUR values indicate the opposite (rapid membrane fouling and poor filterability).

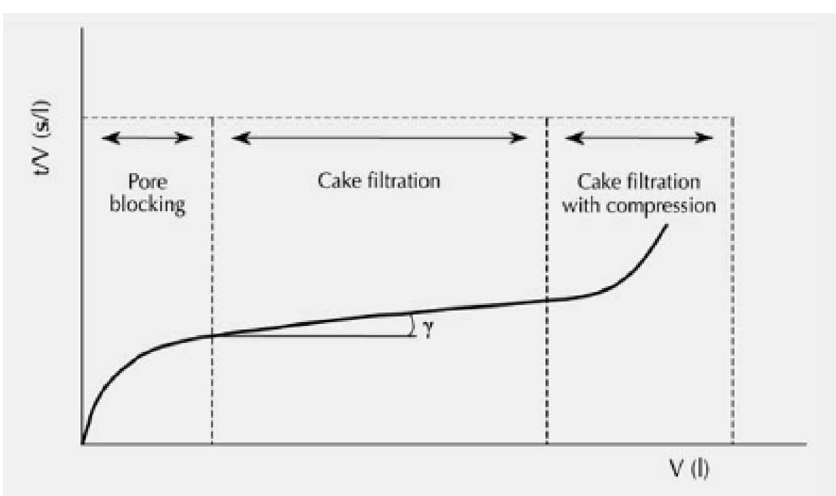

Figure 3. Picture of three theoretically occurring filtration mechanism (pore blocking, cake filtration and cake compression) during dead-end ultrafiltration at constant pressure; tan γ is used to calculate the Specific Ultrafiltration Resistance (Roorda, 2004)

The reversibility of the fouling layer can be defined as the ratio between the change in resistance caused by cleaning of the membrane and the resistance of the fouling layer as a function of the various cleaning procedures.

$$Rev = \frac{\Delta R_{fo} - \Delta R_{fo,x}}{\Delta R_{fo}} . 100\%$$

Rev = Reversibility [%]

ΔR_{fo} = resistance of the fouling layer [m^{-1}]

$\Delta R_{fo,x}$ = resistance of the fouling layer after cleaning with procedure x [m^{-1}]

A cleaning method is sufficient when after filtration and the subsequent cleaning the total membrane resistance is at its initial value.

When the filter is operated with repetitive filtration and cleaning steps, the increase in the initial resistance after the cleaning step ($\Delta R/\Delta t$) indicates the reversibility. A low value indicates a good reversibility.

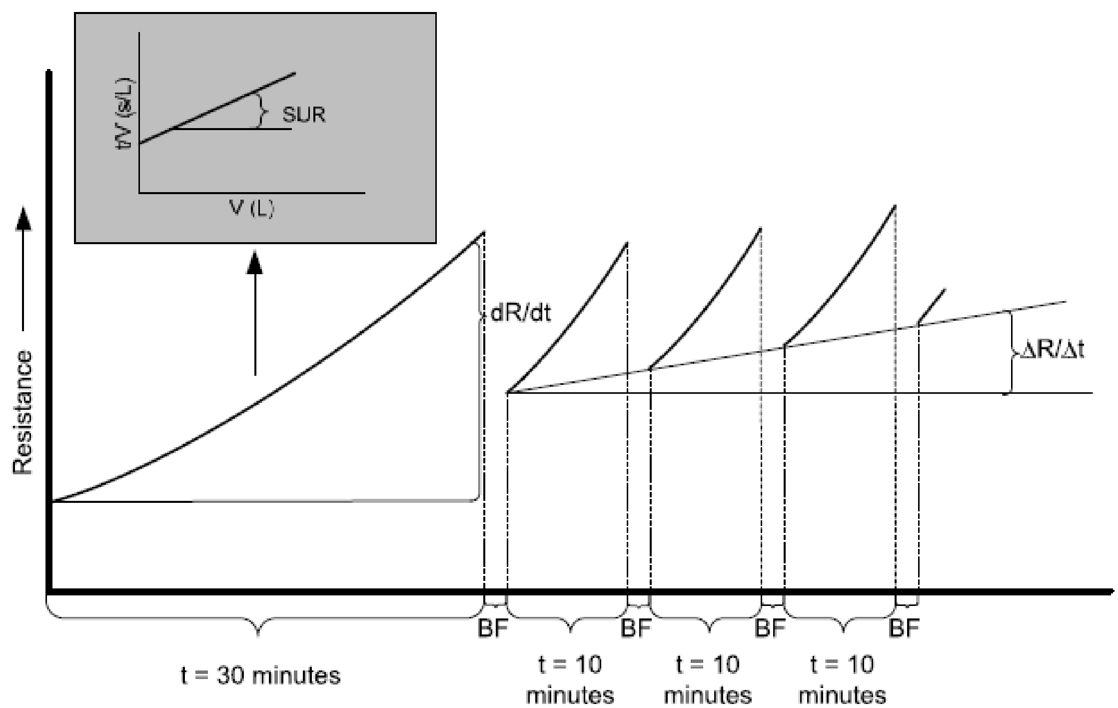

Figure 4. Determination of filterability (SUR and dR/dt) and reversibility by $\Delta R/\Delta t$. (Janssen, 2011)

3 Definitions

Trans Membrane Pressure (TMP)

Pressure difference over the membrane is:

$$TMP = \left(\frac{P_{Feed} + P_{Concentrate}}{2} \right) - \left(P_{Permeate} \right)$$

With: P_{feed} = pressure at feed side
$P_{concentrate}$ = pressure at concentrate side
$P_{permeate}$ = pressure at permeate side

Permeate Flux

Flow per unit membrane area and time:

$$J = \frac{dV}{A_m \cdot dt} = \frac{TMP}{\eta \cdot R} \quad [l/m^2 h]$$

with: J = permeate flux [m^3/m^2.s or L/m^2.h]
V = permeate volume [m^3 of Ll]
A_m = membrane area [m^2]
TMP = Trans membrane pressure [Pa of bar]
η = Viscosity [N.s/m^2 or Pa.s]
R = Resistance [1/m]

$$R_{total} = R_{membrane} + R_{fouling}$$

Retention

The partly or completely retained solute relative to the concentration in the feed water:

$$\text{Retention} = \frac{c_f - c_p}{c_f} \quad [-]$$

with: c_f = solute concentration in the feed
c_p = solute concentration in the permeate

Filterability: SUR

$$SUR = \alpha_{av} \cdot c_v = \frac{d\left(\frac{t}{V}\right)}{d(V)} \cdot \frac{2 \cdot TMP \cdot A_m^2}{\eta}$$

SUR = Specific Ultrafiltration resistance [m^{-2}]

α_{av} = average specific cake resistance [m/kg]

c_v = solid concentration in feedwater [kg/m^3]

TMP = Trans membrane pressure [N/m^2 or Pa]

A_m = Membrane Area [m^2]

η = dynamic viscosity [N.s/m^2 or Pa.s]

4 Experimental set-up

Figure 1 gives a schematic drawing of the filtration setup. In the centre of the test setup a membrane module (G) is placed vertically. The membranes in this module are 3 capillary ultrafiltration membranes, diameter 0.8 mm. The permeate flow in the membrane module is inside out. For the experiments two types of feed water are used: demineralised water (B) and wwtp effluent (C). The type of feed water is selected with valve E. The permeate is collected in a pressure vessel (M). The flow is measured with a mass flow meter (J) and the TMP is measured with a differential pressure meter (I). Both meters are connected to a computer. The pressure is controlled using pressurized air on the feed and permeate tanks. Reducing valve V-1 is used to set the feed pressure in the pressure tanks B and C. Reducing valve V-2 sets the pressure in the permeate tank.

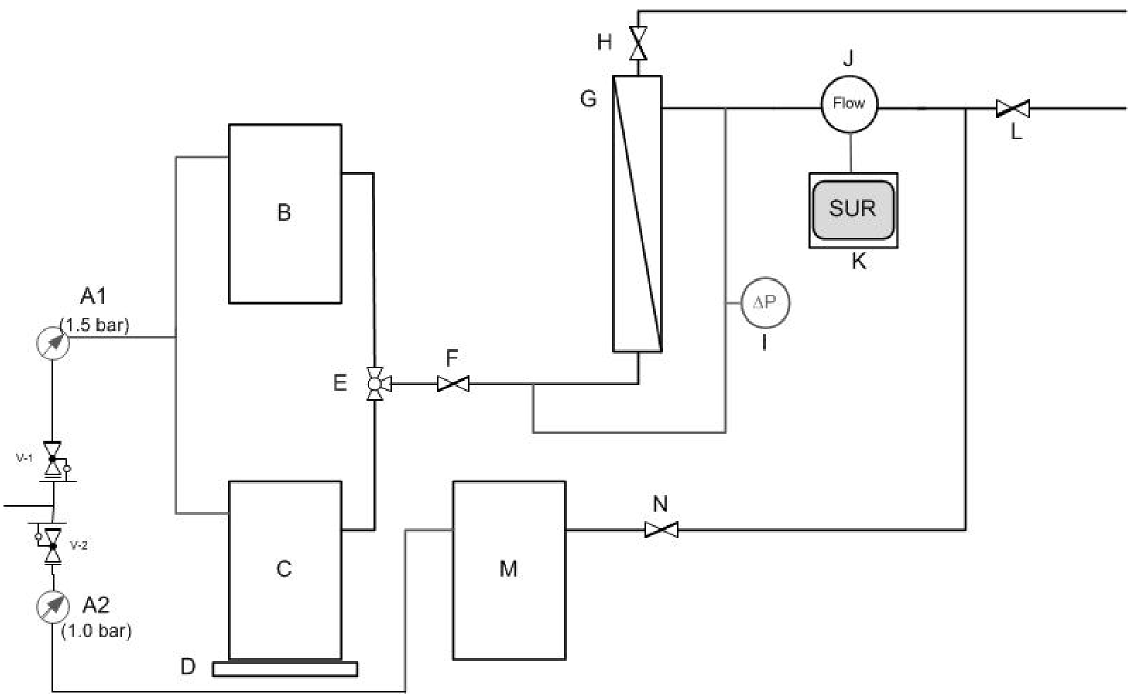

Figure 1 Experimental set-up ultrafiltration of effluent.

A cleaning can be provided by

- A backflush; Permeate flows in opposite direction through the membrane and leaves the system at the concentrate outlet.
- Chemical cleaning; the feed side of the membrane is soaked in a NaOCl solution of 500 ppm active chlorine.

The measurements during the experiments will all be done under dead-end conditions: the feed water enters the membrane module and can only leave as permeate. Impurities in the feed stream will accumulate in the module.

5 Measurements

5.1 TMP measurement

The differential pressure meter is connected to the feed and permeate side of the membrane. The reading is used as trans membrane pressure.

5.2 Flux measurement

The mass flow meter measures the flow of permeate in kg/h. Assuming the density of the permeate is 1 kg/l, the flow can be expressed as l/h. The area of the membrane can be calculated using the total length of the membrane and the diameter of the tubes.

5.3 Viscosity correction

To eliminate the temperature effect on the viscosity, the flux can be corrected for the difference in viscosity by using a reference viscosity at 20°C and measuring the temperature.

5.4 Data aquisition

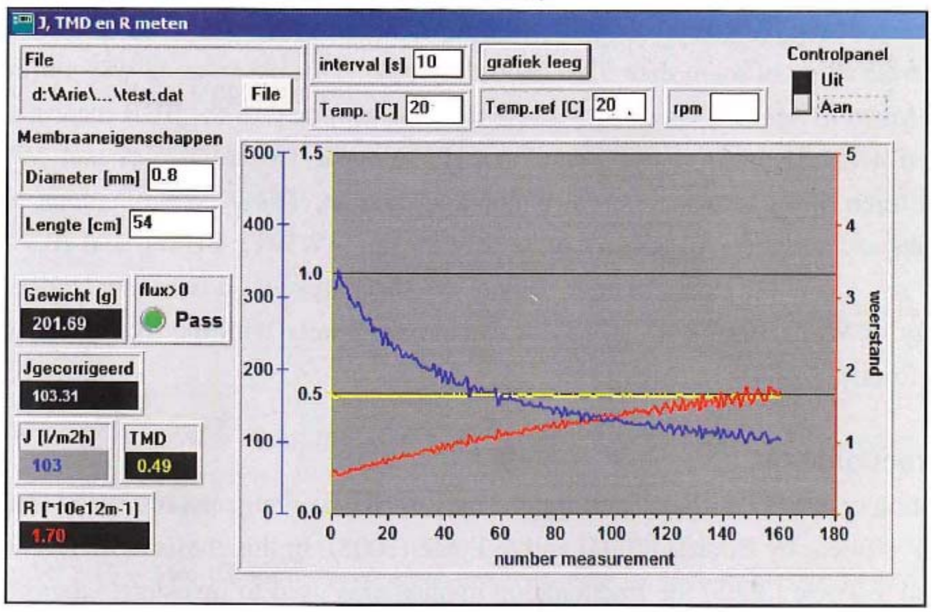

Figure 2. Screendump of data acquisition program.

A data acquisition program is used to store and display the measured and calculated parameters. In figure 2 a screendump is presented. To start the data acquisition, first the communication with the flow meter and differential pressure meter should be opened. Start the 'Flow DDE' program on the desktop and click on 'open communication' on the toolbar at the top of the screen. When the program indicates "Server ready for any client", the communication is open and the data acquisition program 'SUR+ measurement' on the desktop can be started. Select the file button in the program and enter the filename for your datafile. Check the membrane properties (diameter and total length). Switch 'Controlpanel' to 'Aan' and measurements will start.

All data are stored in a .DAT-file (TAB separated ASCII-file). The heading of the file contains the general info like membrane surface and other settings of the measurement. The columns contain the time, flow, TMP, Flux, generalized Flux and Resistance. The .DAT file can be opened in Excel and used for further processing.

6 Removal efficiencies

The performance of a filtration step can be evaluated qualitatively by comparing the influent and permeate quality with respect to different parameters. In this experiment three parameters will be measured:

1. **Turbidity** is measured with a WTW Photoflex Turb turbidimeter in NTU units. The measurement of turbidity is based on comparison of the intensity of light scattered by a sample to the light scattered by a reference suspension under the same conditions (Metcalf & Eddy). For this measurement a standard glass cell is filled with a sample and placed in the turbidimeter; after 10 seconds the display can be read.
2. **Conductivity** is expressed in μS/cm. Put the electrode in the sample and read the value of conductivity.
3. **Colour** is measured as light absorption. A sample is put in a glass cell, and put in the spectrophotometer (see appendix for measuring procedure), the extinction at 455nm can be read from the display. Before testing the effluent, the sample has to be filtrated over a 0.45 µm membrane to avoid interference with particulate matter.

7 Procedures

Both filterability and reversibility is determined in this experiment.

1. First, the condition of the membrane is determined with a clean water flux measurement (CWF). Clean water is filtered through the module for 10 minutes at 0.5 bar TMP. The permeability of the clean membrane is determined.
2. Filterability is determined with the filtration of wwtp effluent for 30 minutes at TMP of 0.5 bar
3. Backwash with permeate at TMP of 1 bar for 30 sec.
4. Filtration of wwtp effluent for 10 minutes at TMP of 0.5 bar.
5. Step 3 and 4 are repeated 4 more times.
6. From step 3 until 5 the reversibility of the foulants in the wwtp effluent can be determined.
7. Take a sample of permeate. Close valve N and disconnect tank M.
8. Chemical cleaning. The membrane is soaked in 400 mg/l NaOCl solution for 10 minutes. The chemicals are removed by forward flushing.
9. Clean water flux measurement as described in step 1.
10. The retention for conductivity, turbidity and colour can be calculated from the measured values in the samples of wwtp effluent and permeate (step 7).

8 Elaboration

- Collect the data stored by the data acquisition program on a USB-stick or disk.
- Plot the flux, resistance and TMP against time for the filtration of effluent for 30 minutes (step 2).
- Calculate the filterability expressed as SUR value using the data from the filtration of effluent (step 2).
- Calculate the reversibility of the foulants in respect to backwash, using the data of the repetitive process of filtration-backwash (step 4-6).

- Calculate the reversibility of the foulants in respect to the chemical cleaning using the data from the two clean water flux measurements (step 1 and 9)
- Compare the different applied cleaning methods.
- Compare the removal efficiencies for conductivity, colour and turbidity.
- Formulate the conclusions of these experiments.

9 Design

With the results of the experiments, a preliminary design can be made of an installation for the ultrafiltration of wwtp effluent. A treatment plant will supply the effluent with a design capacity of 20.000 p.e. The recovery of the installation is 85%.

In order to make a design, certain choices have to be made. Explain these choices.

1. **Flux**. Which flux do you apply for dead end filtration?
2. **Flow**. For which flow do you design the installation? How many membrane modules do you apply? Choose one of the modules from table 2.
3. **Cleaning**. What type of cleaning strategy is applied? (chemical, hydraulic, time intervals, etc). If a backflush is applied, you can assume a flux of 250 l/m^2h, for 90 s.
4. **Pumps.** How many pumps are applied, for which processes? Give for each pump an indication of the required flow(range) and pressure heads.
5. **Technical drawings.** Make a schematic drawing of the installation, with pipes, valves, pumps, etc.

Table 2 Two types of membrane modules.

Membrane module		A	B
Membrane surface	m^2	40	25
Membrane diameter	mm	1.5	5.2
Module diameter	mm	250	205
Length	mm	1500	2000
Initial clean water flux	l/m^2h bar	650	1000

10 Literature

Van Dijk, Rietveld, Verberk: Drinking water technology, September 2001, Chapter 6.

Metcalf & Eddy, Inc., 4th Edition 2003: Wastewater Engineering – Treatment and Reuse, Chapter 11-6.

Janssen, (2011) The applicability of the SUR measurement for ultrafiltration of WWTP effluent, PhD thesis.

Roorda, J.H.,(2004) Filtration characteristics in dead-end ultrafiltration of wwtp-effluent. PhD thesis.

Ultrafiltration of effluent — Data form

Group number: Date:

Datafile name:

Membrane properties:

Diameter : mm

Length module : cm

Number of capillaries :

Settings

Interval : s

Temperature Demi : °C

Temperature Effluent : °C

Temperature reference : °C

Experiment : TMP = bar

Removal efficiency	Turbidity NTU	Conductivity µS/cm	Colour 1/m
Feed			
Permeate			