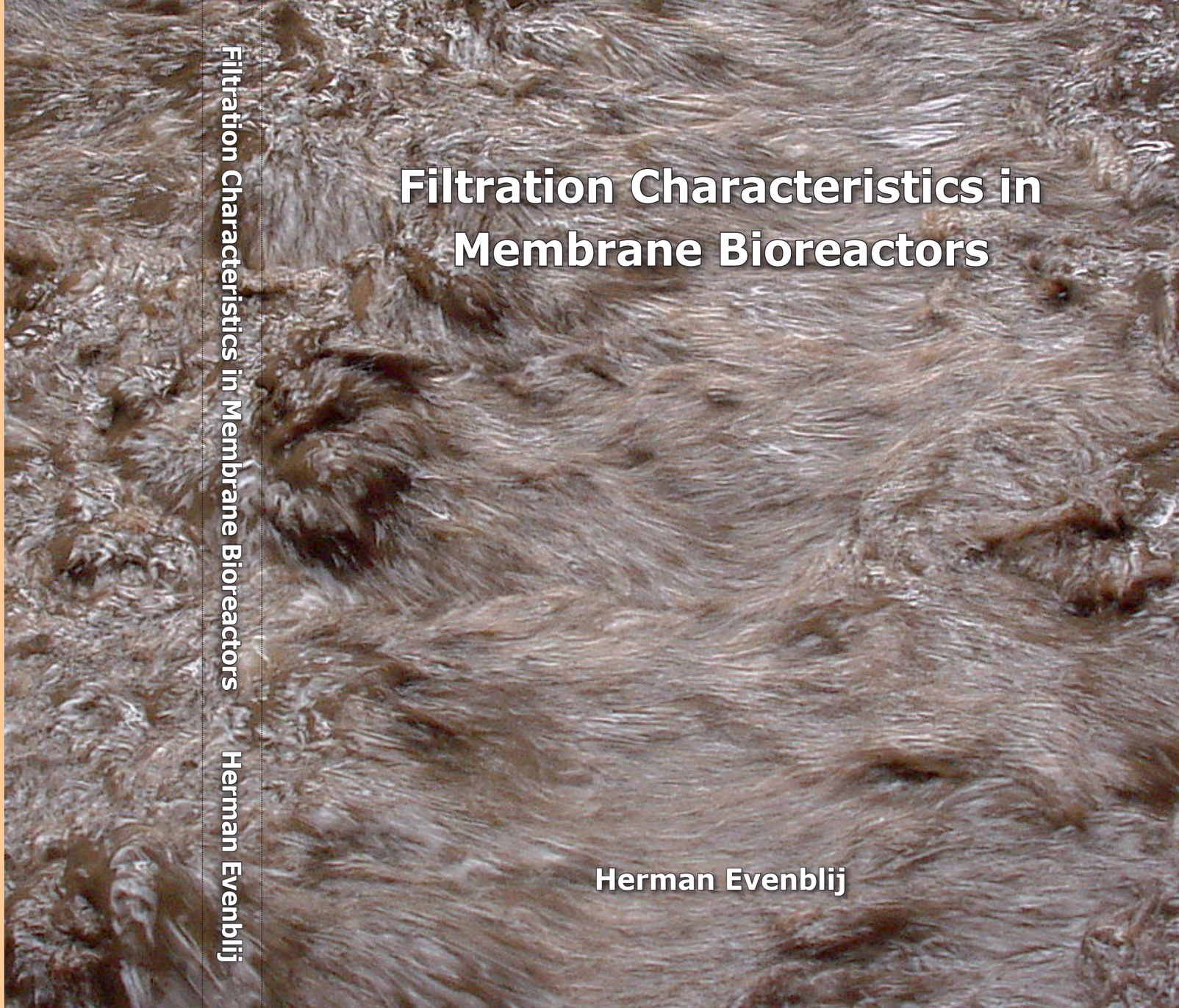
Filtration Characteristics in Membrane Bioreactors
Herman Evenblij
Filtration Characteristics in Membrane Bioreactors
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Propositions

1. Filtration with a single tubular cross flow membrane is a good way to quantify MBR activated sludge filterability. *(This thesis)*
2. Each MBR system for wastewater treatment produces an activated sludge with specific filterability properties. *(This thesis)*
3. (Dead-end) filtration experiments with fractions of activated sludge are not the appropriate tool to identify foulants in MBR activated sludge. *(This thesis)*
4. Fouling behaviour of MBR activated sludge cannot exclusively be correlated to EPS concentrations in the water phase of the activated sludge. *(This thesis)*
5. The character and properties of human language are such that they cannot be the product of evolution. *(To be verified by anyone who is witnessing a 2-year old child learning to talk.)*
6. Simplicity is very often not the hallmark of truth, but the result of over-simplification. (A. van den Beukel, *Geen beter leven dan een goed leven*)
7. A church that determines its course regarding young people by the wishes of 'the youth' is bound to perish.
   (*in reaction to the proposition: The statement* 'If young people leave the church, the church has already left young people' *should be a guiding principle for church policy regarding young people, by S. Dekker*)
8. The MSc-course Civil Engineering and Geosciences is an upgraded Technical School, not a universitarian study. *(prof. ir. J. Wiggers, during the lectures 'Sewerage systems')*
9. The most important work is as a rule unpaid work.
   (A. van den Beukel, *Geen beter leven dan een goed leven*)
10. To state that a human is no more than a result of chemical reactions is the same as stating that the 3rd Symphony of Ludwig van Beethoven is no more than a pile of paper with an irregular ink pattern.

Propositions pertaining to the thesis 'Filtration Characteristics in Membrane Bioreactors'.

These propositions are considered opposable and defendable and as such have been approved by the supervisor,

prof. ir. J.H.J.M. van der Graaf.

Herman Evenblij, Delft, 19 June 2006.

Stellingen

1. Filtratie met een enkelvoudig tubulair membraan is een goede manier om de filtreerbaarheid van actiefslib uit een MBR te kwantificeren. *(Dit proefschrift)*
2. Elke MBR-installatie produceert een actiefslibmengsel met zeer specifieke filtratie-eigenschappen. *(Dit proefschrift)*
3. (Dead-end) filtratie-experimenten met fracties van actiefslib zijn niet het juiste middel om vervuilende componenten in actiefslib uit MBRs te traceren. *(Dit proefschrift)*
4. Vervuilingsgedrag van actiefslib uit een MBR kan niet exclusief gekoppeld worden aan EPS-gehalten in de waterfase van het actiefslib. *(Dit proefschrift)*
5. De aard en eigenschappen van menselijke taal zijn dusdanig dat ze niet geëvolueerd kunnen zijn. *(Iets wat iedereen zal beamen die meemaakt dat een 2-jarig kind gaat praten.)*
6. Eenvoud is maar al te vaak niet het kenmerk van het ware, maar een gevolg van oversimplificatie. (A. van den Beukel, *Geen beter leven dan een goed leven*)
7. Een kerk die zijn beleid laat bepalen door de wensen van 'de jeugd' is ten dode opgeschreven.
   (In reactie op de stelling van S. Dekker: De uitspraak 'Als de jeugd de kerk verlaat, heeft de kerk de jeugd al eerder verlaten' *behoort richtinggevend voor kerkelijk jongerenbeleid te zijn.)*
8. De opleiding Civiele Techniek en Geowetenschappen is een veredelde ambachtschool, geen universitaire studie. *(prof. ir. J. Wiggers, tijdens het college Riolering I)*
9. Het belangrijkste werk is in de regel onbetaald werk.
   (A. van den Beukel, *Geen beter leven dan een goed leven*)
10. Te zeggen dat een mens niet meer is dan het gevolg van chemische reacties is hetzelfde als beweren dat de 3^{e} symfonie van Ludwig van Beethoven niet meer is dan een stapel papier met een onregelmatig patroon van inktvlekken.

Stellingen, behorend bij het proefschrift 'Filtration Characteristics in Membrane Bioreactors'.

Deze stellingen worden opponeerbaar en verdedigbaar geacht en zijn als zodanig goedgekeurd door de promotor, prof. ir. J.H.J.M. van der Graaf.

Herman Evenblij, Delft, 19 juni 2006.

Filtration Characteristics in Membrane Bioreactors

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Published and distributed by: Herman Evenblij
Home : E: Herman.Evenblij@gmail.com
Work : T: +31 570 697 467; E: H.Evenblij@WitteveenBos.nl
copies: www. gezondheidstechniek.tudelft.nl and www.library.tudelft.nl/dissertations

Filtration Characteristics in Membrane Bioreactors

Proefschrift

ter verkrijging van de graad van doctor
aan de Technische Universiteit Delft,
op gezag van de Rector Magnificus prof.dr.ir. J. T. Fokkema,
voorzitter van het College voor Promoties,
in het openbaar te verdedigen

op maandag 19 juni 2006 om 17:30 uur

door
Herman EVENBLIJ

Civiel Technisch ingenieur
geboren te Rotterdam

Dit proefschrift is goedgekeurd door de promotor:
Prof. ir. J. H. J. M. van der Graaf

Samenstelling promotiecommissie:

Rector Magnificus	voorzitter
Prof. ir. J.H.J.M. van der Graaf	Technische Universiteit Delft, promotor
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Acknowledgements

Although attempting to be complete, also aware of being incomplete, the following lines are meant to express my gratitude to those who have contributed to the accomplishment of this dissertation. First of all, I would like to mention Jaap van der Graaf, my promotor. Thank you for your perseverance in starting up the project and your endurance while seeing this book becoming finished; I'll long remember the many discussions about in (my view) useless measurements obtained from utterly failed experiments, invariably ending with the cheering conclusion that the results are 'quite interesting' and 'should be published'; also the study tours and its after dinner discussions had a special inspiring quality. I enjoyed working with and for you.

I would like to mention Helle van der Roest who played a special role in the MBR developments in the Netherlands over the past decade. Your unquenchable enthusiasm kept the 'boat moving' and taught me a new meaning of the expression 'going for something'.

The fine colleagues who introduced me in the world of profs, measurements, results, membranes, writing abstracts, more abstracts, articles and oral presentations: Arjen, Jaap de K, Jasper, Jeroen, Hans K, Hans vD, Luuk, Rob and of course Eefje: it was a pleasure having you around. A special place for you Jelle and Sander: "iron sharpens iron; so one man sharpens another".

Those who joined later: Sandy, Ignaz, Cathelijne, Remy, Viviane, Aldo and Christof, thank you for contributing to the good atmosphere, the under valued coffee breaks and the anti-RSI-sessions. Furthermore I am grateful for the priceless help and cooperation of Cees and Tonny in setting up the measurement tools and constantly improving them; Ria and Van for the analytical work.

The students who had a hard time doing practical work with real sludge: Maaike, Mark (with worse than sludge: raw sewage…), Karim, Roel, Farid, Aldo, Bart, Frank, Stefan: thank you for your efforts and input in this thesis.

The operators at the pilot-plants: Darren Lawrence, Jaap Verkuilen and Robin Kraan (DHV Water), Ron Corsten (Waternet), Dennis Piron (Waterboard Rivierenland), Ferdinand Kiestra (Royal Haskoning), as well as Kees de Korte and Chris Ruiken (Waternet), thank you for your help and support with the research.

More than thanks to you Heidi, for being there; for enduring three births during the birth of this book; for encouraging me and for those many other things you are and do. Thank you, my wonderful children Joris, Lucie and Victor! What a blessing to see you grow up, you are a living proof of the absoluteness of many and the relativity of even more values in life.
Lastly I thank him to whom I owe everything, I am.

Table of Contents

Let no one say that I have said nothing new; the arrangement of the subject is new. When we play tennis, we both play with the same ball, but one of us places it better.
Blaise Pascal, Pensées

1
INTRODUCTION

1.1. Membranes and a Bioreactor: Problems Assured

The Membrane BioReactor process (MBR) for (waste) water treatment forms an elegant combination of the well-known and widely applied activated sludge process with the more sophisticated membrane separation process. This combination should theoretically lead to some advantages:

- smaller footprint because the space consuming sedimentation step is omitted;
- higher effluent quality as a result of the absolute barrier for particles provided by the membrane;
- smaller installation size because the maximum biomass concentration is no longer limited by its settling properties.

The introduction of membranes in the activated sludge process also has some drawbacks, of which membrane fouling is the most significant. The prevention and control of fouling requires a lot of energy, either for sludge circulation or for bubble aeration, to create a shear stress at the membrane surface. Furthermore, the applicable flow through the membranes (the permeate flux) necessitates a large membrane surface. Since membranes are relatively expensive and the energy input to prevent membrane fouling is considerable, both investment cost and operational cost are much higher compared to conventional wastewater treatment.

Despite these issues, MBR has found application in industry, in-building treatment of (grey) wastewater and small-scale municipal wastewater treatment systems.
As a result of decreasing membrane prices, improvements of equipment and more stringent effluent guidelines, a shift towards application in full-scale municipal wastewater treatment can be observed. Many pilot trials were carried out to facilitate this process and at this moment some 15 full-scale MBR plants for the treatment of municipal wastewater are in operation in Europe, see Appendix I. In almost all of these installations, the possible problems have been neutralised by simply avoiding them: large buffer tanks and very low design fluxes are often applied.

As mentioned before, membrane fouling is a bottleneck in the operation of an MBR system. Much effort has to be put into keeping the activated sludge sufficiently filterable and avoiding irreversible fouling. Here lies one of the key problems in attacking this problem, since a complete quantitative overview of the related processes and determining parameters is not yet given.

Furthermore, a standardised method to describe and assess the filtration behaviour of an activated sludge is still missing. Thus, seemingly corresponding results might be different when evaluated in the same way, and contradictory results might nevertheless turn out to be corresponding.

1.2. General Objective of this Thesis

This thesis deals with the problems that arise when trying to pinpoint causes of membrane fouling in membrane bioreactors. Much research is based on a purely scientific approach, often on lab scale, with synthetic wastewater. Although this is necessary to develop sound theory and fundamental knowledge, it is very difficult to translate the results obtained in this way to problems that are encountered in full-scale installations.

To avoid the drawbacks of micro scale research many pilot installations were operated and investigated. Results of this approach show that although real life

circumstances are approximated more accurately in a pilot, real scientific research can hardly be carried out in this way. One reason for this is that by increasing the size of the installation many unknowns are introduced. Measured parameters are often either an average with unknown distribution, or a local value that is not sufficiently representative.

To overcome this dilemma, a compromise is proposed, that combines scientific accuracy with representativity of a pilot installation. A well-defined method was developed for measuring the required parameters needed to increase understanding of the processes that influence filtration behaviour in MBR.

The method comprises the use of a filtration characterisation unit, equipped with a single tubular membrane module, which enables control over all involved parameters. Activated sludge from any MBR installation can be used in the system to assess its filterability. By monitoring filtration data and the significant feed and permeate properties a fingerprint of filtration behaviour is obtained.
Apart from quantifying the filtration performance, the proposed method is a powerful tool for identifying dangerous situations and simulating short-term effects on a small scale. If applied in this sense, the characterisation unit acts as a batch reactor, operated in parallel with, or independent from the pilot or full-scale installation.

1.3. Structure of the Thesis

For a general understanding of the terms and concepts in wastewater treatment and membrane filtration, a concise introduction is presented in *Chapter 2 Fundamentals*.
Subsequently, a literature review is given in *Chapter 3*, treating common ways of characterising filtration in MBR, the state of art in modelling MBR fouling and achievements in the field of identifying substances that cause it.
Chapter 4 and *5* deal with the development of the filtration characterisation method and the results of the first trials.

The filtration characterisation method was applied to 3 different MBR pilot installations operated with three different membrane types. Results of these tests are presented and discussed in *Chapter 7* and *Chapter 8*.

The second application of the proposed method, to the aim of identifying and quantifying dangerous substances and situations, is tested with so called substrate experiments. Activated sludge samples were provided with different types of substrate, and the effect on filterability was assessed with the characterisation unit. Results and discussion of these experiments are presented in *Chapter 6*.

A general evaluation and conclusions are given in *Chapter 9*, ending with defining directions for further research.

...our assumption that everything is provisional and soon to be superseded, that the attainment of goods we have never yet had, rather than the defence and conservation of those we have already, is the cardinal business of life, would most shock and bewilder our ancestors if they could visit our world.
C.S. Lewis, De descriptione temporum

2
FUNDAMENTALS

2.1. Introduction

This chapter provides the background information for this thesis. It consists of three parts, describing firstly the characteristics of the activated sludge process. Secondly, the principles of cross flow membrane filtration are outlined in 2.3, with emphasis on ultrafiltration. Thirdly, the combination of both processes is concisely discussed in the remaining paragraph: Membrane Bioreactors. For the compilation of the first part the 4th edition of the handbook on 'Wastewater Engineering; Treatment and Reuse' (Metcalf&Eddy, 2003) is used. For further reading this textbook is strongly recommended to the interested reader. The third part uses the IWA publication "Membrane Bioreactors in wastewater treatment" by Stephenson *et al.*(2000). The middle part is set up with various sources.

2.2. Activated Sludge Process - Description and Definitions

2.2.1. Activated Sludge

Since its first application in 1913 by Ardern and Lockett (1914) in Manchester, England, the activated sludge process has found wide application all over the world. The concept is founded on the observation that the biomass present in the wastewater could be 'activated' by intensive aeration, stirring and recirculation. Once this biomass was activated it could be used to treat a wastewater.

'For reference purposes and failing a better term, the deposited solids resulting from the complete oxidation of sewage have been designated "activated

sludge".'(Ardern and Lockett, 1914). The same paper reports questions and remarks following the presentation of the work by Ardern and Lockett, e.g. Mr. O'Shaughnessy regarded 'the paper as an epoch-making one, provided that the process experimentally established by the authors could ultimately be applied on the large scale at reasonable cost'.[1]
Eighty years later, the same remarks were heard when MBR was introduced as an option for large scale municipal wastewater treatment…

One of the major features of the activated-sludge process is the formation of floc particles, ranging from 50-200 μm. These floc particles contain bacteria that are held together by extracellular polymeric substances (EPS) (Flemming and Wingender, 2000), and can be removed by gravity settling. What remains is a relatively clear liquid that can be discharged, and sludge that can be returned to the aeration tank to continue biodegradation.

The activated sludge flocs contain a wide range of species of bacteria and protozoa. These organisms are responsible for the conversion of organic material and nutrients. Depending on the type of organism and boundary conditions, different types of conversions can take place. Most important type of conversion is aerobic oxidation, in which oxygen is the electron acceptor and organic compounds act as the electron donor.
Two other reaction types are nitrification and denitrification, processes in which ammonia is converted to nitrite and nitrate (nitrification), which is further converted to nitrogen gas (denitrification). The reactions are all performed as part of the life cycle of the respective bacteria. For each reaction type the bacteria require a carbon source, an electron donor and an electron acceptor, which together yield an end product. Several examples are presented in Table 2-1.

[1] Mr. O'Shaugnessy continues by enumerating the advantages of this process: 'it would reduce the area of the works and would probably go far to eliminate nuisance, and these too were very important considerations from the public point of view. In Germany sewage was often merely precipitated or sedimented and the sewage liquor then passed through open channels for many miles to the nearest river. The Germans seemed to have no objection to this, but in England such a procedure would not be tolerated'. Written April 1914!

The aerobic oxidation is a relatively easy to achieve reaction, since it requires only oxygen, organic compounds and a solids retention time[2] of a few days. The first treatment plants that were built were designed mainly to perform this type of reaction, which required only aeration and mixing. In the past two decades also nutrient removal was incorporated in almost all biological treatment systems (Van der Graaf, 1995). This requires more complicated measures to provide conditions in which the desired bacteria can grow in sufficient numbers. Nitrifying bacteria for example require a solids retention time of 10 to 20 days, to properly perform nitrification (Metcalf&Eddy, 2003).

Table 2-1 *(Incomplete) Classification of micro organisms by electron donor, electron acceptor, sources of cell carbon and end products (from: Metcalf&Eddy, 2003, pp. 563).*

Type of bacteria	Reaction name	Carbon source	Electron donor	Electron acceptor	Products
Aerobic heterotophic	Aerobic oxidation	Organic compounds	Organic compounds	O_2	CO_2, H_2O
Aerobic autotrophic	Nitrification	CO_2	NH_3, NO_2	O_2	NO_2, NO_3
Facultative heterotrophic	Anoxic de-nitrification	Organic compounds	Organic compounds	NO_2, NO_3	N_2, CO_2, H_2O

2.2.2. The Activated Sludge Process

The activated sludge process basically consists of three processes in series in which the mixture of wastewater and biomass is manipulated so as to perform the desired reactions, resulting in a clean effluent. The first step consists of pre-treatment to remove coarse material and other undesired substances. Usually this is followed by primary treatment, like sedimentation, in order to remove particles. Subsequently the influent is mixed with the biomass and treated under aerobic and/or anoxic conditions. The treated water is separated from the biomass in a clarifier, usually a sedimentation tank. The biomass is partially returned to the reactor and partially wasted; a schematic overview of a typical conventional activated sludge process is presented in Figure 2-1.

[2] For an explanation see 2.2.2

For the operation of an activated sludge system the following parameters have to be considered.

Pre-treatment

The amount of pollutants that have to be treated by the activated sludge is usually expressed in terms of substances to be removed, like oxygen consuming substances, total nitrogen, and total phosphorus. As a measure for oxygen consuming substances, (bio)chemical oxygen demand is commonly used (**BOD** and **COD**). This represents the amount of oxygen needed, e.g. by micro organisms, in the (bio)chemical oxidation of organic matter and is expressed as mg O_2/L.

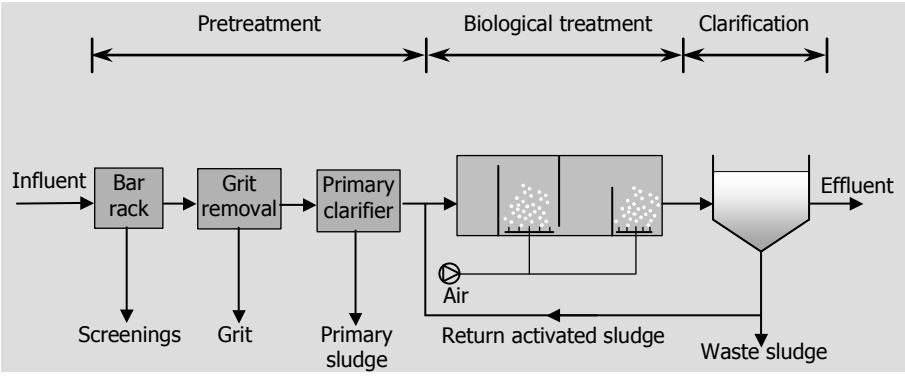


Figure 2-1 *Typical flow scheme of a conventional activated sludge process (adapted from Metcalf&Eddy, 2003)*

The amount of solids in a sample can be determined is different ways. The total of settleable solids is the amount of suspended solids that will settle out of suspension within a specified period of time. The total suspended solids (**TSS**) is that portion of the total solids (residue after evaporation at 103° to 105°) retained on a filter with a specific pore size, measured after being dried at 105°C.

Bioreactor

In the first compartment of the bioreactor the influent is mixed with return sludge from the sedimentation tank, with a biomass concentration, X_R. The resulting mixture has a suspended solids concentration X, which is commonly

called Mixed Liquor Suspended Solids (**MLSS**). MLSS concentrations for wastewater treatment in the Netherlands range from 3.3 to 4.2 g/L (CBS, 2005).

The period of time in which activated sludge remains in the system is the Solids Retention Time (**SRT**), which is an important parameter because it influences treatment process performance, aeration tank volume, sludge production and oxygen requirements (Metcalf&Eddy p. 677). The solids retention time can be calculated as the total amount of solids in the system, divided by the outflow with the waste sludge flow ($Q_w \cdot X_R$) and the effluent ($Q-Q_w) \cdot X_e$, see equation 2-1.

$$SRT = \frac{VX}{(Q - Q_w)X_e + Q_w X_R} = \frac{1}{\mu} \qquad \textbf{(2-1)}$$

where:		
	SRT = solids retention time	[h]
	V = volume	[m^3]
	Q = flow rate	[m^3/h]
	Q_w = waste sludge flow rate	[m^3]
	X = biomass concentration	[g/L]
	X_e =concentration of biomass in effluent	[g/L]
	X_R = concentration of biomass in return flow	[g/L]
	μ = specific growth rate	[h^{-1}]

The specific growth rate can be used to estimate net biomass production rate.
In 2002, wastewater treatment plants in the Netherlands were operated with SRTs ranging from 14 to 36 days, with an average of 23 days (CBS, 2005).
Depending on the chosen SRT the incoming flow rate determines the amount of substrate available for the biomass. This is commonly expressed as the food (F) to micro organisms (M) ratio (**F/M**), which can refer to different types of substrate, i.e. BOD *F/M*. In the Netherlands typical values for BOD *F/M* were around 170 g/kg·MLSS·d in 2001 (CBS, 2005).
Another important parameter is the sludge production. Part of the substrate is used for cell maintenance and part is used for cell duplication. This parameter depends on the sludge production yield (**Y)**, and can be expressed as *g biomass formed/g substrate consumed*. In terms of BOD, typical values are around 425 g TSS / kg BOD (CBS, 2005).

Sedimentation

The last step in the conventional activated sludge process comprises the separation of the effluent from the biomass. This is usually accomplished by gravity in sedimentation tanks. There are two parameters to quantify settling characteristics of activated sludge: the sludge volume index (**SVI**) and the zone settling velocity (**ZSV**). The SVI is defined as the volume of 1 g of sludge after 30 minutes of settling, ml/g. In the Netherlands operating values for SVI ranged from 90 to 110 ml/g, from 1995 until 2001 (CBS, 2005). ZSV is defined as the settling velocity of the sludge/water interface at the beginning of the sludge settleability test; the procedure is described in Standard Methods (WEF, 1998). Based on this parameter the maximum surface overflow rate (OR) of a sedimentation tank can be calculated as (Metcalf&Eddy, p.686):

$$OR = \frac{(V_i)}{SF} \qquad \textbf{(2-2)}$$

where OR = surface overflow rate, $m^3/m^2 \cdot d$
V_i = settling velocity of interface, m/d
SF = safety factor, typically 1.75 to 2.5

Typical values for OR range from 16 to 28 m/d, determining the size of the sedimentation tanks (Ibid. p.687) [3].

With these parameters the solids loading rate of a sedimentation tank can be calculated (Ibid. p. 688)

$$SLR = \frac{(Q + Q_R)X}{A} \qquad \textbf{(2-3)}$$

where		
	SLR = solids loading rate	$[kg/m^2 \cdot h]$
	Q = influent flow rate	$[m^3/h]$
	Q_R = return activated sludge flow rate	$[m^3/h]$
	X = MLSS concentration	$[kg/m^3]$
	A = clarifier cross sectional area	$[m^2]$

SLR represents the amount of solids that can be treated per square metre of sedimentation tank per unit of time. Typical values for SLR range from 4-6 $kg/m^2 \cdot h$ for settling following air-activated sludge excluding extended aeration (Ibid, p.687).

[3] 22 m/d = 0.92 m/h = 917 $L/m^2 \cdot h$

In the Netherlands other guidelines are used for the design of secondary clarifiers (van der Graaf, 1995). The starting point for this calculation is the allowable Sludge Volume Loading rate (SVL). SVL is a function of SVI, MLSS concentration and the allowable surface load, q_A.
The product of SVI and MLSS is the sludge volume (SV) entering the secondary clarifier.

$$SV = SVI \cdot MLSS \quad \textbf{(2-4)}$$

where SV = Sludge Volume [ml/L]

Each value of SV corresponds to an optimum value of allowable surface load. The product of SV and the allowable surface load gives the SVL, which has to be between 0.3 and 0.4 $m^3/m^2 \cdot h$ under all circumstance, especially at maximum flow.

$$SVL = SV \cdot q_A \quad \textbf{(2-5)}$$

where SVL = Sludge volume loading rate [$L/m^2 \cdot h$]
q_A= surface loading rate [$m^3/m^2 \cdot h$]

With these parameters the required sedimentation area can be calculated. The depth of the sedimentation tank is dependent on several parameters, which will not be discussed in this thesis; for further reading see STOWA (2002).

Waste sludge

Apart from the treated effluent the activated sludge process also results in a waste stream: waste sludge. Part of the substrate will be used by the biomass to multiply, which results in an increase of MLSS concentration. Increasing the MLSS concentration will decrease the efficiency of the oxygen transfer and give rise to problems with the sedimentation step. Therefore, periodically or continuously part of the suspended solids are removed from the system. The volume of the waste stream can be reduced by mechanical dewatering, centrifuges, filter presses or belt presses. Subsequently the sludge can be treated by drying, digestion or composting and finally incineration (Metcalf&Eddy, 2003).

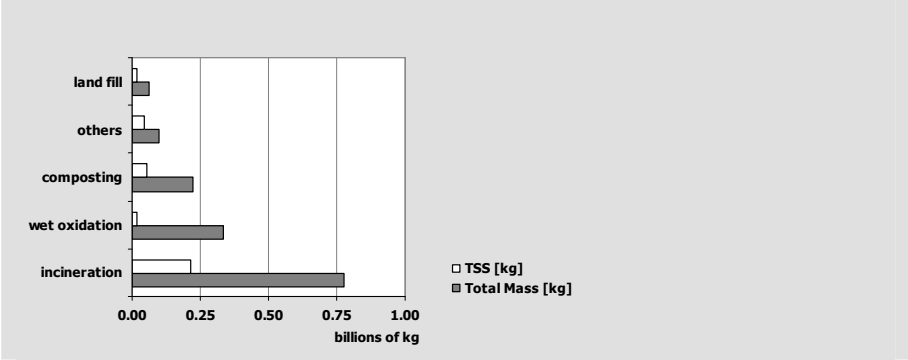


Figure 2-2 *Disposal of waste activated sludge in the Netherlands in 2003 (CBS, 2005)*

Figure 2-2 shows data from 2003 for the different disposal methods applied in the Netherlands. For each method the total mass of waste activated sludge that is treated is presented. The total mass of waste activated sludge amounted to approximately $1.5 \cdot 10^9$ kg containing a total $343 \cdot 10^6$ tons of dry solids, which corresponds to 43 g dry solids/day·p.e. (CBS, 2005).

2.2.3. Application in the Netherlands

Treatment Capacity

In the Netherlands 96% of the municipal wastewater is treated in wastewater treatment plants (wwtp), almost exclusively (99%) by applying the activated sludge process in some form. In 1970 the act on pollution of surface water came into effect. This led to the design and construction of more than 500 wastewater treatment plants in 1981 with a treatment capacity of $1.24 \cdot 10^9$ m^3/year (CBS, 2005).

In 2003 the total treatment capacity of all wastewater treatment plants in the Netherlands amounted to $25.1 \cdot 10^6$ p.e., corresponding to $1.76 \cdot 10^9$ m^3/year (Ibid.).

Treatment Efficiency

The composition of the wastewater may be different for each wwtp, but an average influent quality can be calculated for the Dutch municipal wastewater, see Table 2-2. There are considerable variations in loads and concentrations of

pollutants on different time scales, depending on human behaviour, storm events, type of sewer system, etc. For a further discussion on this topic and consequences for wwtp operation, see (Langeveld, 2004).

Table 2-2 *Typical wastewater and wwtp effluent composition in the Netherlands, together with the current standards for effluent discharge and expected standards*

		Influent[a]	Effluent[b]	Discharge standards[c]	Future Standards[d]
COD	mg O_2/L	477	43	125	-
BOD	mg O_2/L	185	5	20	-
N_{tot}	mgN/L	44	10	10-15	2.2
P_{tot}	mg P/L	7	2	1-2	0.15
SS	mg/L	212	18	30	<5

[a] Data 2002, (CBS, 2005); [b] Average effluent quality of wwtp with capacity > 15, 000 p.e; data 2002 (CBS, 2005); [c] Maximum Tolerable Risk (MTR) according to MinVenW (1998).

The pollutants can be found in different forms, e.g. particulate, bound to colloids and dissolved (Van Nieuwenhuijzen, 2002). Depending on the type of pretreatment a certain amount of pollutants in a certain form will enter the bioreactor.
Of the total of $2.0 \cdot 10^9$ m^3 of wastewater that was treated in 2002, 95% was treated in installations with a capacity of more than 15, 000 p.e (CBS, 2004). The average effluent quality that was produced by these installations is also presented in Table 2-2.

The treatment was originally designed for removal of COD and suspended solids. Due to problems with eutrophication of surface waters further treatment was required which included removal of nutrients like nitrogen and phosphorus.
As an example of the developments in wastewater treatment, total phosphorus is discussed here. The removal efficiency of total phosphorus increased considerably from the mid-eighties. Especially the larger treatment plants were extended to remove nutrients.

This resulted in a P-removal efficiency of 58% in 1990 increasing up to 81% in 2001, whereas influent concentrations did not change so much since 1991. The

effluent of the larger treatment plants nowadays contains around 2 mg P/L, see also Figure 2-3.

However, even these improvements are probably not sufficient to meet future (European) legislations. With regards to the standards for effluent concentrations, P will probably have to be removed down to P_{tot} < 0.15 mg/L and nitrogen down to N_{tot} < 2.2 mg/L (MinVenW, 1998).

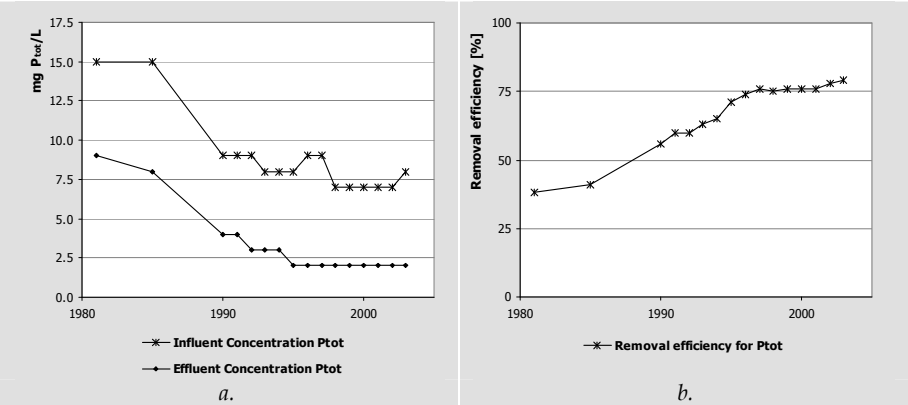


Figure 2-3 *Development of (a.) concentrations and (b.)treatment performance of P_{total} at Dutch wwtp's in the capacity range 15, 000 – 150, 000 p.e. (CBS2005)*

Next to the nutrients there is a long list of micro pollutants that will have to be removed from the wastewater. Special attention will have to be paid to the removal of medicine residues as well as endocrine disruptors, pesticides and heavy metals.

2.3. Membrane Filtration

2.3.1. Process description

Membrane filtration denotes the separation process in which a membrane acts as a barrier between two phases. In water treatment the membrane consists of a finely porous medium facilitating the transport of water and solutes through the membrane, see Figure 2-4.

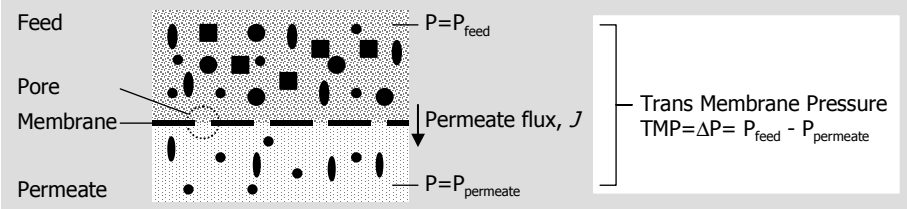


Figure 2-4 *Schematic representation of membrane filtration*

The membrane separates on the basis of molecular (or particle) size; it retains constituents bigger than the pore size. According to the pore size of the membrane, the filtration process can be classified as microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) or reverse osmosis (RO), see also Figure 2-5. In micro- and ultrafiltration the chemistry of the membrane does not play a major role in the separation process itself (Lonsdale, 1981). It should be noticed however that the chemistry plays an important role in the process performance, mainly in the interaction of feed water constituents with the membrane, which may cause a resistance increase.

TMP – viscosity - flux

The driving force for permeation is a trans membrane pressure (TMP) in most water treatment membrane filtration applications, see Figure 2-4.

The rate of permeate flux (J) for a pure solvent feed flowing under laminar conditions in tortuous membrane pores may be described by Darcy's law (Lojkine *et al.*, 1992):

$$J = \frac{\Delta P}{\eta_p R_t} \quad \textbf{(2-6)}$$

where:	J = permeation flux	[L/m² ·h], or [m/s]
	ΔP = trans membrane pressure	[Pa], or [bar]
	η_P= permeate dynamic viscosity	[Pa ·s]
	R_t= total filtration resistance	[m^{-1}]

In the presence of foulants, the total filtration resistance (R_t) is the sum of the clean membrane resistance (R_m) and a fouling resistance, R_f.

$$R_t = R_m + R_f \quad \textbf{(2-7)}$$

Remark the inverse proportionality of permeate flux to permeate viscosity. The permeate viscosity is often close to that of pure water (Manem and Sanderson, 1996). For the calculation of the permeate viscosity, the formula given by Huisman is used in this thesis (Huisman, 1996):

$$\eta_p = \frac{479 \cdot 10^{-3}}{(T + 42.5)^{1.5}} \quad \textbf{(2-8)}$$

where	η_P = permeate viscosity	[Pa ·s]
	T = temperature	[°C]

Selectivity

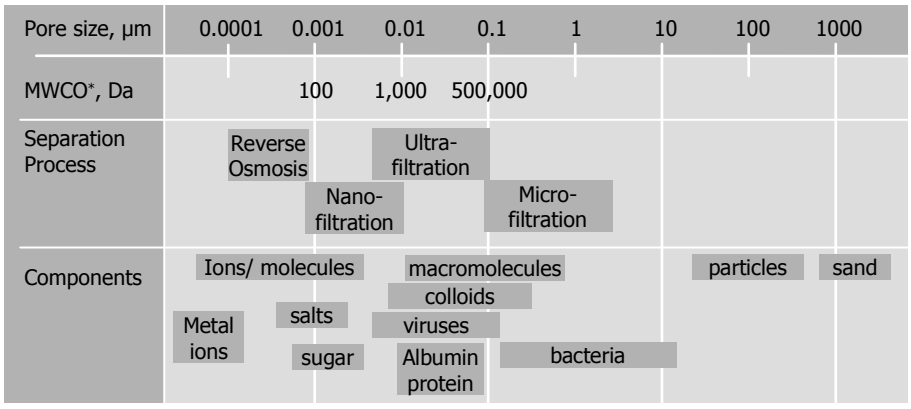
The pore size of the membrane defines its selectivity. Selectivity is sometimes indicated by the molecular weight cut off (MWCO), which is the molecular weight of a solute corresponding to a 90% rejection factor for a given membrane; where the rejection factor (R), is (Koros *et al.*, 1996):

$$R = 1 - \frac{c_{i,\,permeate}}{c_{i,\,feed}} \quad \textbf{(2-9)}$$

where	R = rejection factor	[-]
	$c_{i,\,permeate}$ = concentration of component *i* in permeate	[mg/L]
	$c_{i,\,feed}$ = concentration of component *i* in feed	[mg/L]

MWCO is used for membranes where particle size is not the determining factor, but difference in diffusivity result in selectivity of the membrane.

The driving force causing a trans membrane flux can be a temperature gradient, a concentration gradient, electrical potential difference or a hydraulic pressure gradient. The driving force in water treatment membrane processes is usually a hydraulic trans membrane pressure (TMP) (Mulder, 1996). With decreasing pore size the operating trans membrane pressure increases (Mulder, 1996), because the hydraulic resistance of the membrane increases; see also Table 2-3.



*MWCO = Molecular Weight Cut Off

Figure 2-5 *Classification of Membrane Separation processes (Mulder, 1996; Van Nieuwenhuijzen 2002; Metcalf&Eddy, 2003)*

Table 2-3 *Operating Transmembrane Pressure for different membrane separation processes (Mulder, 1996, Koros et al., 1996)*

Membrane process	Pressure (bar)	Pore size (nm)
Microfiltration	0.1 - 2	100 - 1000
Ultrafiltration	0.1 - 2	10 - 100
Nanofiltration	4 - 20	1 - 10
Reverse osmosis	10 - 30	0.1 - 1

Permeability

A commonly used parameter to represent membrane performance in MBRs is the permeability. Permeability can be calculated as the ratio between the flux and the TMP, see eq. 2-7:

$$Permeability, P = \frac{J}{TMP} \quad [L/m^2 \cdot h \cdot bar] \qquad \textbf{(2-10)}$$

If equation 2-10 is compared with equation 2-6, it can be seen that the permeability multiplied by the permeate viscosity equals the reciproke of the filtration resistance.
The parameter permeability should be used with care, because it is a useless parameter when presented without more data. It can be useful when for example the clean water membrane permeability is known, as well as the mode of operation and which parameter is kept constant, flux or TMP.
See for example the representation of two hypothetical experiments in Figure 2-6.

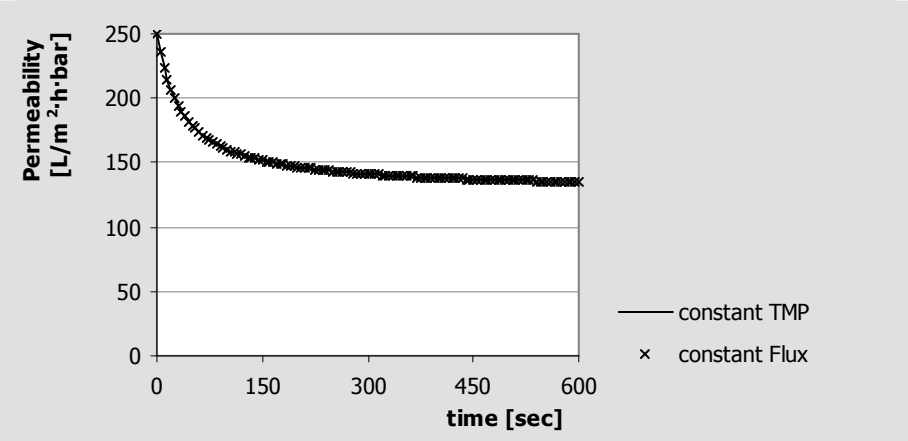


Figure 2-6 *Development of permeability during experiments presented in Figure 2-7*

The first experiment is performed with a constant TMP of 0.5 bar, see Figure 2-7a. Since the clean water permeability of the membrane is 250 L/m²·h·bar, the flux starts at 125 L/m²·h and drops down to a value around 50 L/m²·h. The amount of permeate that is produced during this experiment amounts to 12.4 L/m².

The second experiment is performed under constant flux conditions, with 62.5 L/m²·h. Because the permeability of the clean membrane is again 250 L/m²·h·bar, the TMP starts at 0.25 bar and gradually increases to 0.46 bar, see Figure 2-7b.

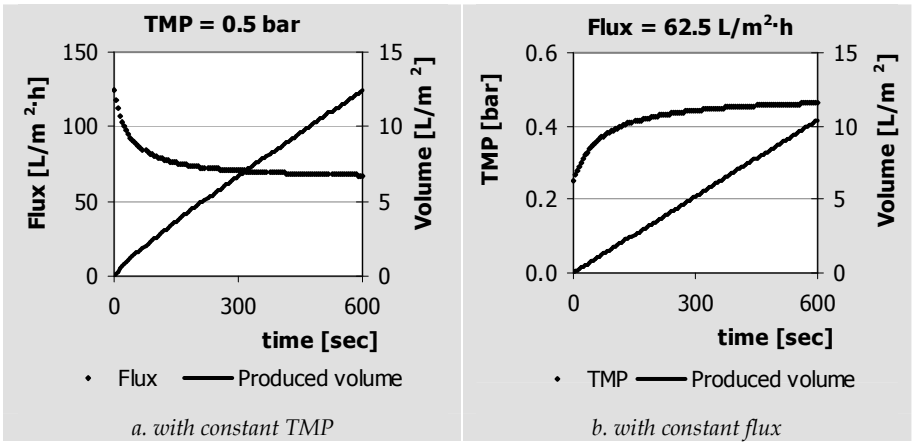


Figure 2-7 *Flux and TMP developments, together with the produced volume during two hypothetical experiments*

The amount of permeate produced during the constant TMP experiment is higher than during the constant flux experiment, 12.4 against 10.4 L/m^2.
If the permeability development during both experiments is considered, these are exactly the same, see again Figure 2-6. Although these curves were never measured, it makes clear that simply presenting permeability, or even a permeability curve is not enough to evaluate filtration performance. The circumstances under which the filtration took place must be taken into account as well.

In this example it is assumed that the temperature is constant and the same during both experiments. In practice this may not be true and the permeability can be corrected for the difference in temperature by incorporating the viscosity, as follows:

$$P_c = \frac{J}{TMP}\frac{\eta_{act}}{\eta_{ref}} \quad [L/m^2 \cdot h \cdot bar] \qquad \textbf{(2-11)}$$

where P_c= corrected permeability
η_{act} = actual viscosity
η_{ref} = viscosity at reference temperature

The temperature correction can be used to filter out the influence of permeate viscosity. It should however be realised that changes in temperature also affect the filtering properties of the activated sludge, for which a correction cannot easily be made (yet).

2.3.2. Membrane material and configuration

Membrane material

Membranes can be manufactured from different materials, such as ceramics, organics and metals (Baker, 2000). Most commonly applied in water treatment are organic membranes, with a wide variety of membrane materials, pore sizes, pore size distributions, membrane configurations and production processes. The main reason to apply organic membranes is connected with the manufacturing costs. Ceramic membranes are about 10 times more expensive than organic membranes (Owen *et al.*, 1995). Other differences can be found in resistivity for cleaning agents, hydrophobicity/hydrophilicity, mechanical strength etc.. Ceramic membranes for example can resist extremely high trans membrane pressure and temperatures. Organic membranes, like cellulose acetate membranes, are usually sensitive for oxidising agents or biological activity of the medium.

The membrane structure can be isotropic or anisotropic. Isotropic membranes have a uniform composition and structure throughout. Anisotropic membranes (or asymmetric) membranes consist of a number of layers, each with different structures and permeability (Baker, 2000).

Table 2-4 *Different membrane materials (Mulder, 1996)*

Organic membranes		*Ceramic membranes*	
Cellulose acetate	CA	TiO_2	
Polyetherimide	Ultem	Zircon oxide	ZrO_2
Polyacrylonitrile	PAN		
Polyethersulphone	PES	*Metal membranes*	
Teflon		Aluminiumoxide	y-Al_2O_3
Polyvinylidenefluoride	PVDF		
Polyethylene	PE		

Membrane configuration

Membranes can be configured into membrane modules in different ways. Depending on the production process the membrane can be in the form of sheets, hollow fibres and tubes (Mulder, 1996). Flat sheet membranes are used to construct spiral wound modules or they can be mounted on a frame, resulting in the plate and frame modules, see Figure 2-8.

Tubular membranes are usually anisotropic membranes with the separating layer at the inside. Hollow fibre membranes are often isotropic membranes that can be operated inside out or outside in. Submerged hollow fibres can be oriented horizontally or vertically; for application in MBR where air scouring is applied, vertical orientation seems favourable (Chang and Fane, 2000)

For the treatment of suspensions flat sheet, tubular and capillary membranes (hollow fibres) are preferred, see also Table 2-5. In recent years, membrane processes have found wide application and nowadays membrane processes exist for most of the fluid separations encountered in industry. (Bowen and Jenner, 1995).

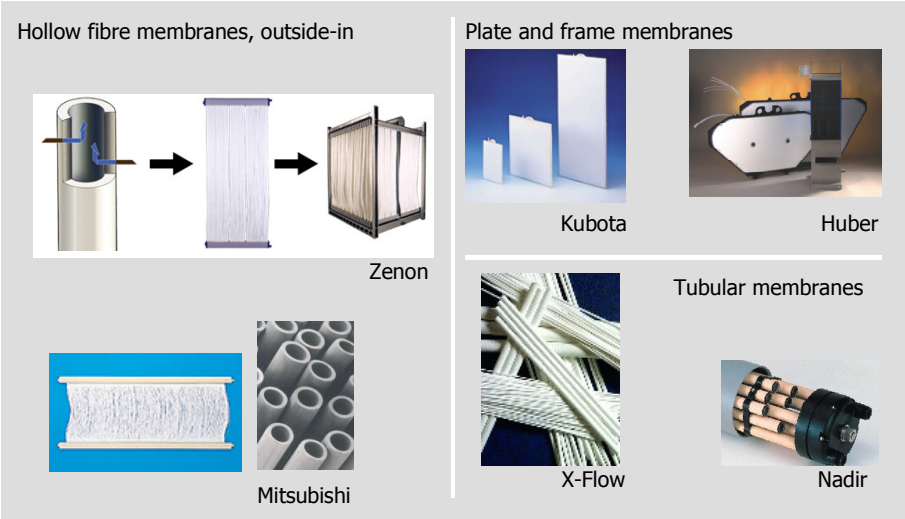


Figure 2-8 *Examples of commercially available membranes, applied in cross flow filtration*

Table 2-5 *Membrane configurations and application in different separation processes (after Baker, 2000)*

Membrane configuration		Applied in:			
		RO	NF	UF	MF
Spiral wound	SW	x	x	x	
Tubular	T		x	x	x
Hollow fibre inside-out	HO-IO	x	x	x	x
Hollow fibre outside-in	HO-OI			x	x
Plate and Frame	PF			x	x

2.3.3. Cross flow and dead-end filtration

Membrane filtration can be operated basically in two modes: dead-end and cross flow. In dead-end mode, all solutes, suspended and dissolved, are transported towards the membrane by the permeate flux. This leads to an increased

concentration of retained material at the membrane and membrane fouling[4] may occur. It was noted very soon that moving the feed flow tangential to the membrane surface results in much higher permeation fluxes ((Bechold, 1907) cited in (Ripperger and Altmann, 2002)). Another approach to avoid excessive accumulation of material at the membrane is the application of air scouring. In this way the fouling is intermittently removed, as further discussed in this thesis. The differences between cross flow filtration and dead-end filtration are illustrated in Figure 2-9.

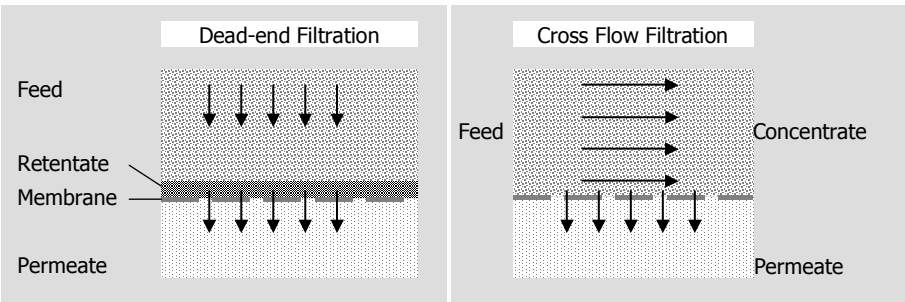


Figure 2-9 *Schematic representation of dead-end filtration and cross flow filtration*

Cross flow membrane filtration originates from the first half of the 20th century. The first patent for micro porous membranes was issued to Zsigmundy in 1922. Early applications were developed during World War I and II mainly for bacteriological assays. Since then microfiltration was applied in many processes (Lonsdale, 1981).

During cross flow filtration the cross flow stream continuously removes retained material. Compared to dead-end filtration, water with higher solids content can be treated and fluxes can be higher. It must be noted however that operating costs of cross flow filtration is high compared to dead-end filtration, because of the energy needed to circulate the feed flow. There are developments to increase energy efficiency by applying the airlift principle, see also §2.5.

[4] For the definition of membrane fouling, see §2.3.4

2.3.4. Cross flow Ultrafiltration

During cross flow ultrafiltration (CFUF) two streams can be distinguished: the feed flow, that is circulated at a certain cross flow velocity, and the permeate flow, see Figure 2-10.

Permeate is forced through the membrane by the hydraulic trans membrane pressure (TMP). In industrial applications, values for TMP in CFUF can go up to 5 bar, with cross flow velocities between 0.5 and 5 m/s (Evenblij, 2001). For municipal wastewater treatment in MBR the ranges are narrower, with TMP not higher than 0.5 bar and representative cross flow velocities up to 1 m/s (Stephenson *et al*, 2000).

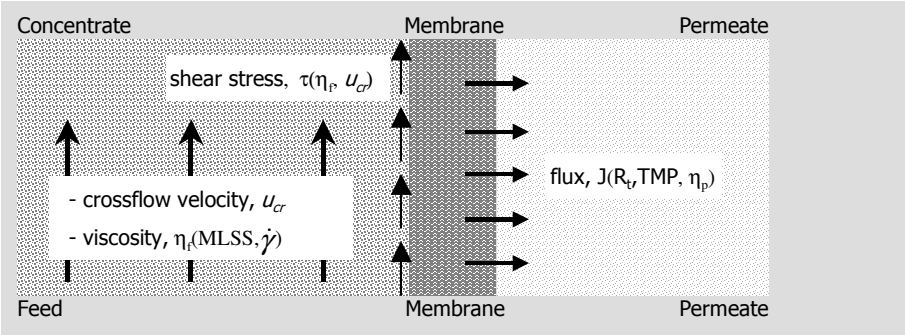


Figure 2-10 Parameters affecting cross flow membrane filtration

All submerged membrane systems for MBR applications apply a coarse bubble aeration from below the membrane unit to provide turbulence and shear forces which prevent excessive membrane fouling, see also §2.5.

2.3.5. Viscosity and Permeation

Equation 2-4 shows the relation between trans membrane pressure, viscosity, filtration resistance and permeate flux. It is important to note that two viscosities play a role in CFUF (see Figure 2-10):

- **Permeate viscosity, η_P**

This is a function mainly of temperature (Metcalf&Eddy, 2003) and in most cases equals clean water viscosity. The permeate viscosity determines the flow behaviour through the membrane pores.

- **Feed viscosity, η_f**

In the case of activated sludge filtration this is a function of MLSS concentration, temperature and shear rate (Rosenberger *et al.*, 2002). The feed viscosity determines the flow pattern around the membrane modules or in the membrane tube.

2.4. Membrane fouling

Application of a cross flow is meant to achieve stable operation, or, avoid a too fast flux decline (or TMP increase). This decrease in process performance is generally indicated with the term fouling. Several definitions of fouling can be found in literature. A broad definition is given by (Cheryan, 1998) 'Fouling manifests itself as a decline in flux with time of operation, and in its strictest sense the flux decline that occurs when all operating parameters (…) are kept constant'. Van den Berg and Smolders (1990) regard it as that part of the flux decline, which is irreversible, and a long-term phenomenon. Lojkine and co-workers also leave out short term phenomena, and define it somewhat different: 'Fouling is a blanket term used to cover the physicochemical causes of flux decline, which are NOT reversed when the transmembrane pressure is relaxed' (Lojkine *et al.*, 1992). Important here is that they do not consider concentration polarisation (or: 'loose' cake layer formation) as fouling.

In this thesis the definition by the International Union of Pure and Applied Chemistry is used:

> '*Process resulting in loss of performance of a membrane due to deposition of suspended or dissolved substances on its external surfaces, at its pore openings, or within its pores.*' (Koros *et al.*, 1996).

This definition is interpreted as including concentration polarisation phenomena. In this way fouling is encountered at two levels: the filterability, which is reflected as the loss of 'process performance', e.g. during a filtration run. The second level is the reversibility, which is a measure of the extent to which the membrane performance can be regained after it was fouled during filtration. Although filterability and reversibility are linked, they must be separated when discussing membrane fouling, see also (Roorda, 2004).

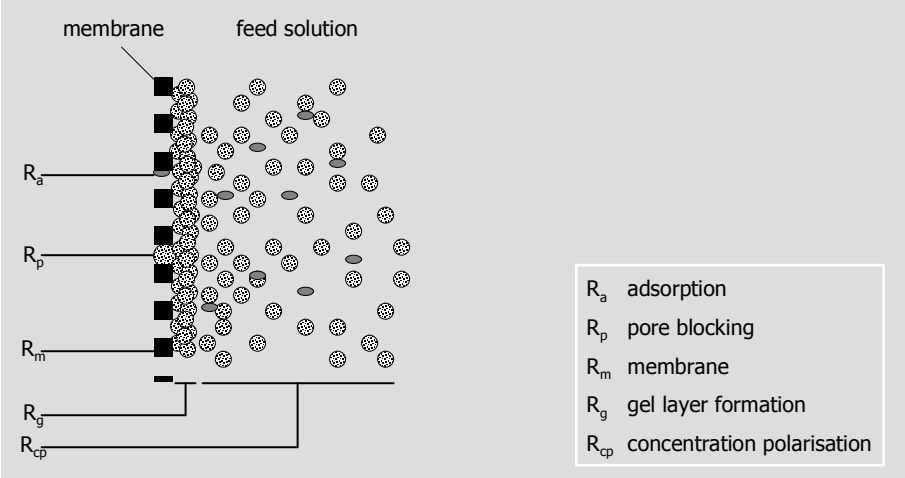


Figure 2-11 Fouling mechanisms in membrane filtration (van den Berg and Smolders, 1990)

Different fouling mechanisms may occur during cross flow membrane filtration, some of which were mentioned before, see also Figure 2-11 (van den Berg and Smolders, 1990):

- **Pore blocking**

Particles enter the pore and get stuck in its opening, reducing the number of pore channels available for permeation.

- **Pore narrowing, e.g. by adsorption**

Substances and/or particles enter the pores and are adsorbed to the pore wall, thus narrowing the pore channel, reducing the permeate flow.

- **Gel or Cake layer formation**

Particles and macromolecules accumulate at the membrane surface, forming a more or less permeable layer. When its constituents are non-interacting, the cake layer may disappear when TMP is released or crossflow in increased.
If there is an interaction the particles may form a cohesive gel layer, which is difficult to remove. In both cases the fouling mechanism will lead to an increase in total filtration resistance.

- **Concentration polarisation**

According to the definition of the IUPAC concentration polarisation (CP) is *a concentration profile that has a higher level of solute nearest to the upstream*

membrane surface compared with the more or less well-mixed bulk fluid far from the membrane surface (Koros *et al*, 1996). The following paragraph will discuss the relevance of concentration polarisation in ultrafiltration and microfiltration.

2.4.1. UF and MF: no concentration polarisation

It should be remarked that the term concentration polarisation is originating from RO applications. This phenomenon results in a back transport of solvent from the permeate side to the feed side due to an increase in osmotic pressure, requiring an increased transmembrane pressure to maintain permeation. In membrane filtration processes with higher molecular weight cut off, like UF and MF, the retained material does not have the properties to induce an osmotic pressure difference over the membrane. Although material is accumulating at the membrane surface, the only effect is the build up of a filter cake. This also causes an increase in required TMP, but this pressure is needed to overcome the cake layer resistance. These two processes must therefore be regarded separately.
Since it is assumed that concentration polarisation is not occurring in MF and UF, it is left out of consideration in the further study of fouling, except for the discussion of some studies that model fouling by including a CP-term.

2.4.2. Variables that influence fouling

All of the mentioned fouling mechanisms will result in a performance decrease of the separation step.
Operational performance of membrane filtration is a function of time and many other variables. These can be placed in three groups that will be discussed in the following paragraph (Lojkine *et al*., 1992):
1. operating conditions;
2. nature of the membrane and
3. nature of the feed solution.

Ad 1. Operating conditions

TMP and flux

In practice there are three ways of operating a membrane separation process: with constant TMP, with constant permeate flux or with a combination of these.

With constant TMP, flux will decrease over time; with constant J, TMP will increase over time. In the case of a non-fouling feed, there is no difference between these two.
Some systems are operated with a combination of constant flow/constant TMP, which seems favourable to minimise fouling and optimise process performance (Vyas *et al.*, 2002). After a period of working with constant TMP, the system is changed to constant flux to avoid excessive membrane fouling.

The permeate flux can be regarded as a measure for the fouling load on the membrane, because it determines how much foulants are transported towards the membrane. This is of course influenced by the flow profile near the membrane, which is primarily determined by the cross flow velocity.

Cross flow
The cross flow is applied to create turbulence preventing the accumulation of material at the membrane and to promote back transport mechanisms. In this way, steady state can be reached for those applications in which the foulant transport towards the membrane is equalled by the back transport caused by the cross flow. The permeate flux that is obtained in this way is called steady state flux, J_{ss}. In general the influence of cross flow on steady state flux can be written as: (Lojkine *et al.*, 1992)

$$J_{ss} \propto (u_{cr})^n \qquad \textbf{(2-12)}$$

where u_{cr} is cross flow velocity, m/s
n is a flow coefficient, which may vary between 0.5 and 1.1, depending on module design and feed properties.

The exact mechanism that makes the cross flow effective is difficult to explain, which is reflected by the numerous modelling approaches to describe observed behaviour (Romero and Davis, 1988; Gekas and Hallström, 1990; Belfort *et al.*, 1994; Bowen and Jenner, 1995). Different ideas exist as to which mechanism or mechanisms are responsible for balancing the effect of the drag force caused by the permeate flux, see also Table 2-6.

Table 2-6 *Cross flow microfiltration transport models (Gekas and Hallström, 1990)*

Model	Drag force is balanced by	Reference
Concentration polarisation	Particle diffusion (Brownian)	
Deposition theory	Lift force (inertial)	
Combination	Lift force and particle diffusion additive	Madsen (1977)
Improved lift-force models		Green and Belfort (1980)
Convective model	Convection parallel to membrane due to shear stress forces; lift forces and diffusivity ignored	Vassilief *et al.*, (1985)
Improved CP	Shear-stress enhanced particle diffusivity	Zydney and Colton, (1986)

Almost all modelling work is carried out with mono disperse solutions containing spherical (latex) particles. Furthermore the models leave out membrane structure and morphology as well as interaction between particles.
In the case of MBR, particle interaction can be expected to occur, since activated sludge is in itself a product of particle interaction.

The immediate effect of a cross flow velocity is the shear stress on the membrane wall, which may be a measure of its effectiveness. With activated sludge however it is difficult to calculate shear stress *a priori*. The calculation requires the relation between shear rate and viscosity to be known, as well as the flow profile near the membrane. *A posteriori* it can be calculated, with the known pressure drop along the membrane in the direction of the feed flow, with equation 2-9 (in case of a tubular membrane element) (Cheryan, 1998):

$$\tau_m = \frac{d_m \cdot \Delta P_{tube}}{4 \cdot L} \qquad \textbf{(2-13)}$$

where
τ_m = shear stress [Pa]
ΔP_{tube} = pressure drop along membrane tube [Pa]
d_m= channel diameter of membrane tube [m]
L = length of membrane tube [m]

In submerged systems it is more difficult to calculate shear stress because of the discontinuous flow pattern around the membranes, caused by the air bubbles.
With the shear stress the ratio between permeate flux and shear rate can be calculated. Chen *et al.*, (1996) point out the importance of this value with regard to the type of fouling that occurs. Gésan-Guiziou and co-workers (1999) calculate an effective shear rate for steady state filtration and conclude that there is a critical value for each suspension, reflecting the properties of the formed cake layer. Operating above this critical value leads to irreversible fouling.

Ad 2. Membrane properties
The membrane pore size distribution will determine to a considerable extent the operation of a membrane separation step. The preferred membrane pore size should be as large as possible to achieve the desired separation, and small enough to prevent constituents from entering the pores (Lojkine *et al.*, 1992). For wastewater treatment and especially membrane bioreactor applications the feed solution (activated sludge) contains a wide variety of components, which makes it impossible to choose a minimum pore size. To prevent constituents entering the pores would require a pore size in the nano-filtration range, which leads to high energy cost. With respect to the particles or substances that have to be removed, the maximum pore size can be chosen, for example with the aim of disinfecting the effluent.

Membrane material also plays a role, since it may interact with species in the feed flow. For applications in water treatment hydrophilic membranes will be preferred (Ibid.). This is for example described by Chang and Lee (1998), who tested both hydrophilic and hydrophobic membranes on the same activated sludge broth.

Ad 3. Suspension characteristics
Steady state flux generally decreases with increasing foulant concentration. Flux versus concentration shows a sharp initial decline, followed by a plateau, which may be followed by a second sharp decline, see Figure 2-12 (Lojkine *et al.*, 1992).

In MBR applications, the concentrations seem to be in the plateau phase, i.e. a filterability seemingly independent on TSS concentration.

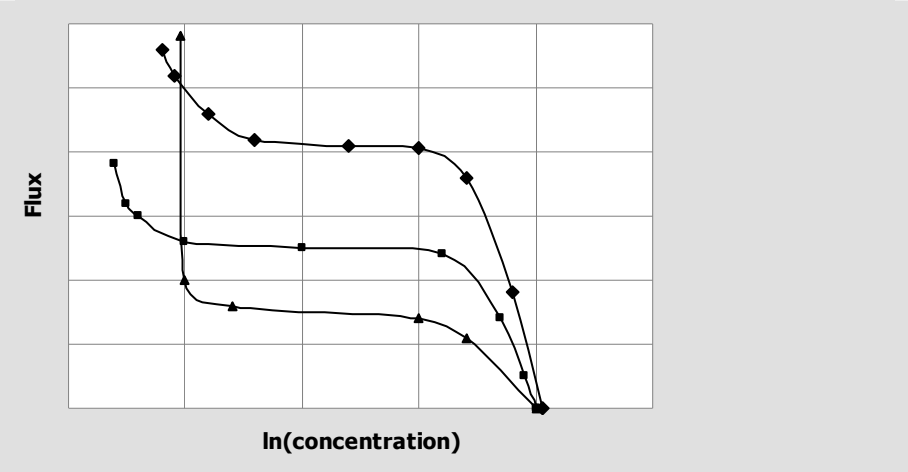


Figure 2-12 *Typical sigmoidal plots for cell concentration (from Lojkine* et al.*, 1992)*

Referring to eq. 1.3 and Figure 2-10 it is worth mentioning that increase in feed concentration, together with a viscosity increase will cause the shear stress to increase as well, leading to higher steady state fluxes. On the other hand, increasing viscosity will decrease the Reynolds number, reflecting the shift towards laminar conditions which is quite detrimental for cross flow filtration.
The particle size distribution shows its influence in the properties of the cake layer that will be formed. With non-interacting particles, smaller particles will lead to a less permeable cake layer (Lahoussine-Turcaud, 1990; Kwon and Vigneswaran, 1998; Vyas, 2002). Particles smaller than about 0.5 μm influence fouling to a great extent, whereas particles larger than several μm had little effect on flux. In the case of tubular crossflow filtration it was observed that larger particles were concentrated in the centre of the tube (Cheryan, 1998) leading to a higher concentration of small particles in the cake layer (Ould-Dris *et al.*, 2000). Furthermore, Al-Malack and Anderson (1997) found out that after coagulating wwtp effluent its filterability was better than without coagulating. This was explained by supposing that aggregated particles were more easily swept away

by the cross flow and could not form a gel layer. In other words, by aggregating the small particles, they are excluded from entering the boundary layer.

From the previous paragraphs at least one conclusion can be drawn: an accurate characterisation of experimental set up, operational conditions and influent properties is needed to compare results from different filtration experiments. Usually this is done by mentioning cross flow velocity and operational mode (i.e. constant flux or constant pressure). Sometimes this is extended with the dimensions of the membrane, information on the membrane material and properties, etc.
In the case of activated sludge filtration this is even more complicated since the determining properties of the activated sludge are at the moment not known *a priori*. The particle size distribution of activated sludge is bi-modal, containing two particle classes, i.e. flocs, sized ca. 25 – 100 μm and free colloids sizes ca. 0.5-5 μm (Mikkelsen and Keiding, 2002).

2.5. Membrane Bioreactors

2.5.1. Introduction

The Membrane Bioreactor combines the biological activated sludge process with a membrane filtration step for sludge water separation. The membranes can be incorporated in the process in two ways:

- **Internal**

The membranes are submerged in an aerated tank and permeation takes place under a vacuum, to the inside of the membrane (see Figure 2-13a). Commonly used membrane configurations are hollow-fibre and plate and frame modules.

- **External (Side stream)**

The membranes are placed external to the reactor and sludge is recirculated through the (tubular) membrane elements, where permeation takes place inside-out (see Figure 2-13c).

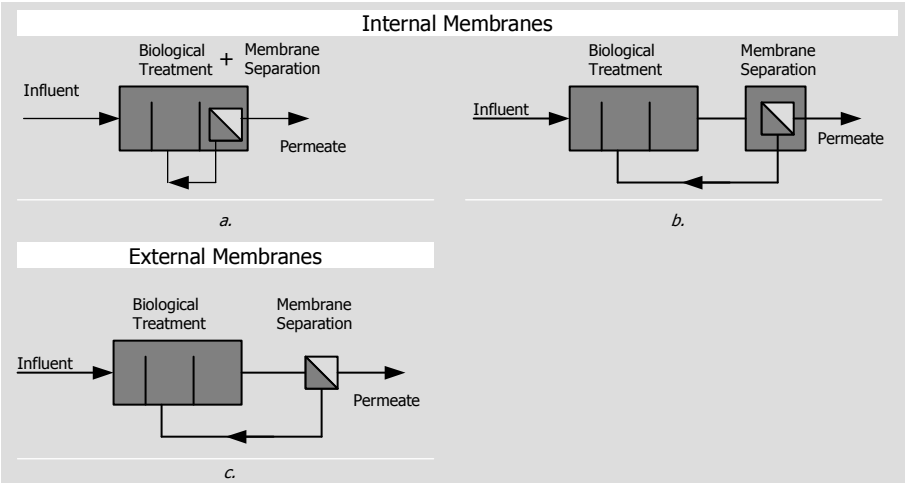


Figure 2-13 Different configurations of the MBR process: internal and external membranes

The strict distinction between internal and external MBR is not maintained in practice, because in many applications there is a separate membrane compartment, with its own aeration and a circulation flow, as illustrated in

Figure 2-13b (van der Roest *et al.*, 2002; Meraviglia *et al*, 2003). A circulation through the membrane compartment, together with aeration is more effective for filtration performance than aeration alone (Chang and Fane, 2000).

2.5.2. Historical development

The first descriptions of this technique date from the late sixties. In the 1970s the technology entered the Japanese market and by 1993, 39 external MBR systems had been reported for use in sanitary and industrial applications. At this moment MBR systems are applied widely throughout Japan for domestic wastewater treatment and reuse and some industrial applications. In the 1980s, the Japanese government invested in the development of a low footprint, high product quality process that would be suitable for water recycling. Within this programme the Kubota plate and frame membrane was developed. Many small-scale applications were realised in Japan during the 80's and 90's (Stephenson *et al.*, 2000).

At the American continent the developments in the MBR field led to the development of the hollow fibre submerged membrane, by Zenon. In the nineties, the application of the process was extended to larger scale wastewater treatment and much of these developments took place in the USA and Europe, see for example Côté *et al.* (1997), Krauth and Staab, (1988). Due to decreasing membrane prices and improvements of membrane modules and materials, the number of MBR installations in Europe increased considerably in the past ten years.

At this moment some 45 municipal MBRs are in operation throughout Europe, see Appendix I *Municipal MBR in Europe* and Appendix II *Industrial MBR in the Netherlands*. Many manufacturers of membranes have developed membrane configurations and membrane materials especially for application in MBR, see Table 2-7.

Table 2-7 *Characteristics of 10 commercially available MBR configurations (Van Houten, 2003) PES Polyethersulphone; PE Polyethylene; PVDF Polyvinylidenefluoride.*

Supplier	Bio-reactor	MBR config., membrane proc.	Membrane configuration	membrane material and structure
Huber VRM®	aerobic	submerged, UF	plate and frame	PES composite, assymetric
Hydranautics	aerobic	external, UF	Capillary	PES, asymmetric
Kubota	aerobic	submerged, MF	plate and frame	
Mitsubishi	aerobic	submerged, MF	Capillary	PE, symmetric
X-Flow/NoritMT	aerobic	external, UF	Tubular	PVDF, asymmetric
Puron	aerobic	submerged, UF	capillary	PES, symmetric
Rhodia/Orelis	aerobic	external, UF	plate and frame	PES, asymmetric
Seghers/Keppel	aerobic	submerged, MF	plate and frame	PVDF, asymmetric
USFilter/ Memcor	aerobic	Submerged	capillary	PVDF, asymmetric
Zenon	aerobic	submerged, UF	capillary	PVDF, asymmetric

2.5.3. Cost comparison MBR-Conventional AS

High cost connected with MBR is often mentioned in discussions about applicability of MBR. It is interesting to evaluate the development in cost estimates over the past 7 years.

Davies *et al.*(1998) made a cost comparison for two wwtps, with capacities of 2, 350 and 37,500 p.e.. With the assumptions they made (for example a membrane lifetime of 7 years) they conclude that depending on the design capacity (i.e. 2 times DWF to be treated) MBR is competitive with conventional treatment up to a treatment capacity of 12,000 m^3/day, see Table 2-8.

Table 2-8 *Comparing capital and operational costs of MBR and conventional AS, assuming capacity of 2*DWF (Davies et al., 1998)*

Parameter		**MBR (Kubota)**	**Conventional**	**MBR/ Conv.**
Capital Costs				
2, 350 p.e.	*	613, 000	980, 204	0.63
37, 500 p.e.	*	7, 292, 524	3, 642, 259	2.00
Operating Costs	*/yr	75, 373	56, 200	1.34
2, 350 p.e.	*/yr	602, 101	264, 730	2.27
37, 500 p.e.				

* Currency not specified

Engelhardt *et al.*(1998) after carrying out pilot experiments also made a cost calculation for an MBR with a capacity of 3,000 p.e., designed for nitrification/denitrification and treatment of 2*DWF. Investment costs were estimated at €3,104,000 (including pretreatment) and operational cost at €194,000/year.

Adham and co-workers made a cost comparison for conventional activated sludge and MBR (Adham *et al.*, 2001). A comparison was made between MBR, oxidation ditch followed by membrane filtration and conventional activated sludge followed by membrane filtration. The exact assumptions and details of the cost estimate are not available, it can be concluded that MBR is competitive with the other treatment systems, see Table 2-9.

Table 2-9 *Comparing capital and operational costs of MBR and conventional AS+membrane filtration(Adham et al, 2001)*

Alternative	Capital Costs $	MBR/ Conv.	Total Cost $/yr	MBR/ Conv.
Zenon MBR	5,068, 627	-	783,000	-
Oxidation ditch+MF	5,587, 800	0.91	876,000	0.89
Conventional activated sludge+MF	5,933, 520	0.85	867,000	0.90

STOWA (2004) describes a cost comparison between an MBR installation and a conventional activated sludge system with tertiary sand filtration. The calculations are carried out for two new wastewater treatment plants with the aim of producing effluent with low concentrations of nitrogen and phosphorus.
Investment costs are almost the same, and operating costs are 10-20% higher for MBR, depending on the capacity of the plant, see Table 2-10.
The calculation does not take into account those parts that are the same for both treatment trains, like bar racks, waste sludge treatment, etc.

Table 2-10 *Comparing capital and operational costs of MBR and conventional AS+sand filtration, assuming a capacity of 4*DWF (STOWA, 2004)*

Parameter capacity		**MBR**	**Conventional + sand filtration**	**MBR/ Conv.**
Capital Costs				
10, 000 p.e.	€	6,407,000	6,979,000	0.92
50, 000 p.e.	€	23,091,000	22,901,000	1.01
Operating Costs				
10, 000 p.e.	€/yr	748,000	686,000	1.09
50, 000 p.e.	€/yr	2,821,000	2,335,000	1.21

DeWilde *et al.*(2003) conclude that MBR will be more expensive in terms of capital and operational costs and that space availability is more likely to be a driving force for MBR application.

Chang *et al.*(2001) report experiments with low cost membranes. The effect on investment cost is considerable, but operational problems hinder further application of low cost membranes. A drawback of the applied membranes is its limited disinfecting capacity.

Thus, it is clear that it is not easy to make a general economical comparison between MBR and conventional activated sludge systems. First of all, the reference system should not simply be an activated sludge system, but a system that produces an effluent of the same quality. Secondly, investment costs seem to converge for the two; operational costs are still higher in the Dutch situation where 4 times the dry weather (DWF) flow must be treated. If the difference between dry weather flow and storm weather flow (SWF) is small, MBR is more competitive.

Lastly, MBR is a modular system, i.e. easily expandable, which is often mentioned as an advantage of the system. However, this makes the system less competitive with conventional systems, since these become relatively less expensive per p.e. at larger scale.

2.5.4. Differences between MBR and AS

Activated sludge properties

The presence of a membrane for sludge separation has consequences in many ways. Defrance and Jaffrin (1999b) found out that filtering activated sludge from an MBR resulted in totally reversible fouling, whereas filtration of 'conventional' activated sludge led to irreversible fouling.

MLSS concentrations are usually higher in MBR than in conventional activated sludge treatment. Adham *et al.*(2001) describe the results of a questionnaire sent around under four manufacturers (Kubota, Zenon, Mitsubishi and Suez-Lyonnaise des Eaux/Infilco Degremont Inc.) Twenty were returned, and the results show that applied MLSS concentration is around 10 mg/L. This influences rheological properties of the sludge. Defrance *et al.*(2000) compared conventional activated sludge with MBR activated sludge and found that MBR sludge was less viscous than conventional sludge. The same was observed by Rosenberger *et al.*(2002). Furthermore, with increasing shear rate, viscosity of the sludge decreases (Rosenberger *et al.*, 2002), although in some cases the activated sludge behaves as a Newtonian fluid (Xing *et al.*, 2001). Lastly, with increasing MLSS concentration, the viscosity increases (Ibid.).

Another effect of the increased MLSS concentration is a decrease in α-factor[5] Günder and Krauth, 1999. Cornel *et al.*(2003) measured α-values of activated sludge from full scale installations and found that with increasing MLSS concentrations the α-value decreased. With a MLSS concentration of 12 g/L, the average α-value was 0.6, whereas at conventional stabilisation plants, operating at 3-5 g/L MLSS concentration, values of about 0.8 are measured, see also Krampe and Krauth (2003).

The maximum value enabling energy efficient operation of an MBR system is mentioned as 15 g/L (Günder and Krauth, 1999) and 10 mg/L (Itonaga *et al.*, 2004).

[5] α-factor is the oxygen transfer efficiency, defined as the ratio of the volumetric oxygen transfer coefficient under process conditions to the clean water transfer coefficient [-]

Activated sludge composition

It is quite difficult to generalise results from any installation, since each installation promotes different types of activated sludge. This has its effect on the microbial community that can be found in an activated sludge system. Nevertheless, it seems obvious that the presence of the membrane in an MBR system influences the biomass composition. Since no suspended solids are washed out with the effluent, the only sink is surplus sludge. From a secondary clarifier lighter species will be washed out, whereas in an MBR they will be kept in the system by the membrane. Furthermore, changes in SRT and higher MLSS concentrations might lead to changes in the microbial community. Witzig *et al.*, 2000) describe results of measurements on the microbial characteristics of MBR sludge. Although significant changes were observed, compared to conventional activated sludge, the treatment efficiency with respect to COD-removal was the same. When applying Fluorescence in Situ Hybridization (FISH) to an MBR with extremely high sludge ages it was found that, compared to conventional activated sludge, a significantly lower fraction of all living bacterial cells (identified by DAPI) were detectable with EUB probes (Witzig *et al.*, 2002*)*. The majority of the cells were found to be in a non-growing state, reason why it is supposed that these cells are participating in the degradation process just to satisfy their maintenance energy requirements without further cell division, thus producing low amounts of excess sludge, see also Wagner and Rosenwinkel (2000).

Treatment efficiency/Removal capacity

Because the biological treatment in MBR is performed according to the principles known from activated sludge treatment, the types of conversion that can be achieved in MBR do not differ substantially from conventional activated sludge. COD, BOD and SS removals are high throughout all studies described in literature, which is mainly ascribed to the fact that the effluent is particle free (Côté *et al.*, 1997, Engelhardt *et al.*, 1998), DeWilde *et al.*, 2003*)*.

Mansell *et al.* (2004) performed measurements in which MS2 coliphage were seeded tot the influent of a Kubota MBR (characteristic pore size 0.4 μm) and a Zenon MBR (characteristic pore size 0.04 μm). Permeate concentrations showed

a log removal range of 3.2 to 7.4 for the Kubota installation and 5.32 to 7.5 for the Zenon installation. All of the heavy metals detected in the influent were removed to levels below detection limit, as well as the VOC's and BNA's that were measured. Ahn *et al.*, 2003) describe experiments in which phosphorus removal could be achieved in a lab-scale MBR treating household wastewater. Average influent concentration for P_{tot} was 3.7 mg/L and effluent concentrations were averagely 0.26 mg/L. Effluent BOD was smaller than 10 mg/L, TSS < 1 mg/L.

Cicek *et al.*(2000) describe experiments with an MBR equipped with ceramic membranes, and treating a synthetic feed. With varying SRTs (5, 10, 20 and 30 days) COD was removed for more than 98%. Also Kj-N was removed with efficiencies of more than 98%. Kubin *et al.*(2002) measured ammonium removal of more than 97%.

Biological P-removal in a MBR is described by Adam *et al.*(2002). P_{tot} was always lower than 0.2 mg/L with sludge ages between 15 and 25 days. Investigations with P-spiking showed higher bio-P-potential in MBR compared to conventional activated sludge systems.

Lesjean *et al.*(2002) describe experiments in which very low total nitrogen and phosphorus concentration were obtained with an MBR, with sludge ages of 16 and 25 days. Both pre-denitrification and post-denitrification were tested, without additional carbon source, where post-denitrification seemed favourable.

Innocenti *et al.*(2002) investigated the removal of heavy metals and nutrients by an MBR treating a mixture of industrial and municipal wastewater. The removal of Ag, Al, Ba, Cd, Co, Fe, Hg, Mn, Ni, V and Zn increased when SRT was increased from 10 to 190 days. As, B, Cu, Pb and Se removal decreased with longer sludge ages.

Sludge production and treatment

From small-scale lab studies a great advantage of MBR was the observed lower or even zero excess sludge production, caused by low loading rates and high SRT (Benitez *et al.*, 1995*)*.

The amount of excess secondary sludge produced in larger MBR installations is somewhat lower than or equal to conventional systems (Günder and Krauth,

2000). When long SRT's are applied, sludge production of course decreases (Wagner and Rosenwinkel, 2000).

The primary sludge production is higher, because of the higher degree of pre-treatment. Sludge treatment is almost the same compared to conventional activated sludge systems. Recent developments in the USA show a trend towards lower MLSS concentration (<10 g/L) while the plant sizes are increasing (> 40,000 m^3/day). SRT is selected based on the biological process requirements (Daigger *et al*, 2004).

The dewaterability of waste activated sludge from MBR seems to be no problem, compared to aerobic stabilised waste sludge from conventional activated sludge systems (Kraume and Bracklow, 2003).

Space requirements

One of the advantages of MBR is its compactnes, because large sedimentation tanks are not needed. An interesting parameter in this respect is the surface overflow rates for the two systems. The overflow rate of a secondary clarifier is defined as the ratio of its flow and footprint i.e. the volume of water that can be treated per square metre of tank, see eq. 2-2. In practice, values around 22 m/d are used.

For an MBR filtration tank, also an 'overflow rate' can be calculated.
To this aim the packing density of the membranes has to be known, i.e. the amount of square metre of membrane area per square metre of tank.
The overflow rate of a membrane tank can then be calculated as the product of the permeate flux and the packing density, see eq. 2-10.

$$OR_{MT} = packing\ density \cdot J \qquad \textbf{(2-14)}$$

where

OR_{MT}	= overflow rate of the membrane tank	[m/d]
packing density	= membrane surface/tank surface	[-]
J	= permeate flux	[m/d]

From several membrane suppliers the membrane packing density of the respective membrane tank is presented in Table 2-10. The overflow rate depends on the applied permeate flux, therefore results from calcualtions with two values of permeate flux are presented. With an average permeate flux of 15 L/m^2·h, the overflow rates of the membrane tanks are in the range 25-62 m/d which is up to three times higher than the overflow rate of a conventional secondary clarifier.
Compared to an average overflow rate of 22 m/d with a secondary clarifier the space consumption for sludge water separation in an MBR is 10 to 60% lower when J=15 L/m^{2}h, and 50 to 80% lower when J=25 L/m^2·h.

Table 2-11 Comparison of overflow rates of membrane tanks in MBR and clarifiers in CAS

Membrane supplier	Packing density [m^2 membr./ m^2 membr. tank]	'Overflow rate' [m/d]	
		J=15 L/m^{2}h	J=25 L/m^{2}h
Zenon	148	53	89
Mitsubishi	171	62	103
Kubota	68	25	41
Toray	137	49	82

A further reduction in foot print is caused by the higher MLSS concentration that can be applied in an MBR. Remark that the calculations do not take into account backflushings or relaxation periods which will reduce the 'overflow rate'.

2.5.5. Membrane operation and maintenance

The most eye-catching difference between a conventional wastewater treatment system and a MBR is of course the application of membranes. Since most of the commonly applied membranes are quite sensitive, special attention should be paid to operate them properly and assure the required life time.
With respect to the operation of membranes, there is a tendency towards non-fouling operation. This means that the membranes are operated with low flux rates, in order to prevent excessive fouling and penetration of the membrane with foulants. Flux rates between 10 and 35 L/m^{2}h are commonly applied values nowadays. Furthermore, intermittent permeate extraction is applied to enhance air scouring effectiveness (Hong *et al.*, 2002). To save energy, the aearion rate of the membranes may be related to the actual permeate flux (Howell *et al.*, 2004)

With respect to cleaning, common practice in submerged hollow fibre systems is the application of periodic maintenance cleanings. With intervals ranging from 7 days to about one month the membranes are backflushed or soaked with a solution containing cleaning agents, like oxidising agents (see e.g. Frechen *et al.*, 2003).

For plate and frame systems, which are not backflushable and are usually operated at lower fluxes, the intervals between chemical cleanings are even larger, up to months and a few times per year (see e.g. Gander *et al.*, 2000).

References Chapter 2

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It is a most extraordinary thing, but I never read a patent medicine advertisement without being impelled to the conclusion that I am suffering from the particular disease therein dealt with in its most virulent form.
J. K. Jerome, Three men in a boat (to say nothing of the dog)

3
LITERATURE REVIEW ON MEMBRANE FOULING IN MBR

In the year 2002, prof. Roger Ben-Aim wrote a review article on membrane bioreactors in which the following statement is made:

> *"…at this time system optimisation is not possible since we lack a fundamental understanding of the different interactions between the membrane performance and the process operating conditions."* (Ben-Aim and Semmens, 2002).

This chapter gives an overview of the achievements in the efforts to discover the 'different interactions' as described in scientific literature.

3.1.1. Scope of literature review

The focus of this chapter is membrane fouling in aerobic MBRs, the underlying mechanisms and measuring methods. Literature on anaerobic MBRs is mentioned only few times, because:

- fouling mechanisms in anaerobic MBRs are different from those in aerobic MBRs.
- the scope of this thesis comprises large scale municipal MBRs, which are almost exlusively aerobic systems

The majority of studies of membrane fouling in MBR can be divided in two categories:

- **Applied Research.** Given a certain MBR application, the causes of malfunctioning are investigated in order to optimise system performance. This approach is handicapped by the fact that the whole system (with changing inflow conditions, large(r) scale, difficult and often inaccurate measurements) has to be considered.

- **Fundamental Research.** Phenomena typical for any MBR system are measured and described. To this aim the problem is simplified by e.g. using a precisely defined synthetic wastewater. In this way the experiments are well defined and single aspects can be isolated and thoroughly investigated.

The means in both approaches can essentially be the same. The filtration behaviour of the suspension under consideration is measured, analysed and characterised. This last aspect is the main subject of this chapter: how cross flow filtration of activated sludge is characterised and what information can be obtained by it.
Subsequently a concise overview on the current quantitative knowledge on factors influencing filtration will be given.

3.1.2. Overview of factors influencing fouling in MBR

As a start a general overview of the processes and factors that affect filtration performance in MBR is presented in Table 3-1. Three processes are distinguished (columns) that have a consecutive effect on filtration performance, in the order of the subsequent steps in an MBR:

- *pre-treatment* (2nd column);
- the *activated sludge process* (3rd column) and
- the *membrane separation process* itself (4th column).

Within each of these processes, a distinction is made for the type of influence, presented in the three main rows. The first row, designated as 'boundary conditions', shows the influencing factors that cannot or only very difficult be manipulated.
The second row shows a gradual transition from 'design choice' to 'parameters that can be manipulated'. In the design of an MBR installation choices are made that affect filterability, like pre-treatment, choice of pumping devices, etc. Other parameters, like HRT, SRT and dissolved oxygen content in the bioreactor, can be changed within certain boundaries. These can than probably be used to

improve filterability, given a situation where the filterability of the activated sludge (suddenly) deteriorates.

The bottom row shows the physical 'results' of both boundary conditions and operation of the three processes inasmuch as these influence filterability. The pre-treatment produces the influent for the activated sludge system. The combination of these two results in a mixed liquor entering the membrane separation step. The design and operation of the membrane separation step, combined with the entering mixed liquor yields a certain filtration behaviour, observed as development of filterability in time.
Of course there is also a feed back relation between membrane filtration and activated sludge process, because of the return flow from the membrane tank to the activated sludge process.

Table 3-1 Factors Influencing Filtration Performance in MBR, adapted from Chang et al. *(2002)*

	Pre-treatment		**AS Process**		**Membrane Separation**	
Boundary conditions	Temperature pH COD, N, P, etc. Varying Q		Temperature		Temperature Complete retention of SS	
	+		**+**		**+**	
Choice in design/operation	Pre-treatment		Pump type (shear) C-source type of Chemicals Stages (Anox/Aer) Substrate history pH F/M Mixing devices (shear) C-source addition Chemicals addition Return flows internally		Membrane Type (pore size, configuration, etc.) Membrane Aeration/u_{cr} Constant J or Constant TMP J/τ Relaxation/BF/FF Chemical Cleanings	
Parameters that can be manipulated			HRT SRT DO		Concentration factor in MT	
	↓		↓		↓	
Results in terms of filterability influencing Factors	COD, N, P, etc. Fractions No hair, fat etc.	→	η_{sludge} Floc type MLSS Composition of water phase PSD pH influent MT	↔	PH Floc type Composition of water phase PSD $\eta_{permate}$	→ **Filtration Performance**

Paragraph 3.2 will discuss the results of research focusing on the last column (Membrane Separation) in Table 3-1, with emphasis on filtration characterisation. Paragraph 3.3 will go deeper into identifying and quantifying filterability influencing factors determined by the activated sludge process.

3.2. Modelling Activated Sludge Filtration

Each researcher generates some model in his mind to understand and explain his (or her) results. In many cases, the second step is the translation to a computational model in order to reproduce the measured data. Since the knowledge on the exactly occurring mechanisms in MBR membrane filtration is still lacking, no computational model exists which accurately describes occurring phenomena. There are however many useful models because each model contains part of the researcher's model and thus explains (at least part of) the results. This paragraph describes some of the models that were developed for computational modelling of membrane fouling in MBR.

First some general aspects of cross flow filtration are presented. This is because filtration in an MBR is always engineered as a cross flow filtration step. Either the membranes are submerged in an aerated basin, or the activated sludge is circulated through tubular membranes. In both cases there is a shear stress caused by the cross flowing sludge, possibly enhanced by air bubbles, Stephenson *et al.* (2000).

Several modelling approaches are proposed, among which the resistance in series, empirical models and mass transport models must be mentioned. Each of these models has its own advantages, accuracy and practical value as will be discussed further on.

3.2.1. Cross flow filtration

Cross flow filtration is generally characterised by a sharp initial decline in flux, followed by a (semi-) steady state situation. The first initial performance drop is ascribed to adsorption or concentration polarisation (Belfort *et al.,* 1994; Cheryan, 1998; Song, 1998)). This effect can partially be explained by the fact that many CFUF processes are operated with constant pressure. In that way the starting fluxes are very high and cause a strong initial fouling (Hong *et al.*, 2002) . If

constant flux is applied the amount of fouling could be less (Field *et al.*, 1995; Defrance and Jaffrin, 1999).

Belfort and Nagata (1985) studied hydraulic phenomena during cross flow filtration. They observed an increase in friction coefficient of a flowing fluid in a porous tube with wall permeation. Also the onset of turbulence shifted to Reynolds values of about 4,000 instead of 2,100.

3.2.2. Resistance in series model

In *Chapter 2* the basis of the resistance in series model was described in eq. 2-6, where the obtained flux is shown to be a result of the applied TMP divided by the permeate viscosity and the total filtration resistance.

The fouling resistance can be subdivided according to the different fouling mechanisms, like concentration polarisation, cake layer formation etc., see also chapter 2.2. If for example fouling inside the membrane is negligible eq. 2-4 can be rewritten as:

$$J = \frac{\Delta P}{\eta_p (R_m + \alpha m)} \qquad \textbf{(3-1)}$$

where: R_m= membrane resistance [m^{-1}]
α = specific cake resistance [m/kg]
m= cake mass per unit membrane area [kg/m^2]

When starting a filtration run, only the membrane resistance has to be overcome. Foulants will accumulate at the membrane surface due to convection with the flux; this can be measured as an additional filtration resistance. A further distinction can be made, by removing the foulants with different cleaning procedures, and in between measuring the clean water resistance (Choo and Lee, 1996, Al-Malack and Anderson, 1997).

Another approach is to fractionate the feed flow, and determine the filtration resistance that each fraction causes, which is also applied in dead-end ultrafiltration (Roorda and van der Graaf, 2003). Bouhabila *et al*. (1998) for example centrifuged activated sludge and filtered the supernatant in a filtration cell; the same procedure is followed by Ognier *et al.* (2002). Defrance *et al.*(2000) filtered three fractions: 1. the complete activated sludge; 2. supernatant after settling and 3. flocculated supernatant (with $FeCl_3$ at 0.4 g/L) from the former

procedure. Afterwards, the obtained resistance values for each fraction can be summed to calculate total resistance, e.g. (Wisniewski and Grasmick, 1998).

This approach does not always lead to strong conclusions, e.g. the total of the different resistances is often not equal to the resistance caused by the original sample. This may be explained by cross flow filtration theory, which predicts a classification at the membrane, as a result of the different transport mechanisms, see par. 3.2.1. Furthermore, the filtration experiments are not carried out under circumstances, representative for an MBR. Some researchers perform filtration experiments in dead-end mode (Ognier *et al.* 2002; Itonaga *et al.*(2004)), others in stirred cell tests (e.g. Chang *et al.* 2001, Lee *et al.*, 2001).
Wintgens *et al.*(2002, 2003) succesfully incorporated the resistance in series model in an integrated model to describe an MBR with submerged hollow fibre membranes. The resistance increase due to adsorption to the membrane was made dependent of the total amount of produced permeate.

3.2.3. Mass transfer models

Theoretical considerations about the process of cross flow filtration have led to the development of mass transfer models, or back transport models. They predict the behaviour of particles exposed to cross flow filtration, starting from concentration polarisation and Brownian diffusion, also including inertial lift (tubular pinch effect) (Green and Belfort, 1980), shear induced diffusion (Zydney and Colton, 1986), and surface transport (Belfort *et al.,* 1994). All of these models predict that with decreasing shear forces, bigger particles will be involved in cake layer formation. This is experimentally verified with mono disperse solutions, and also partially for polydisperse solutions, e.g. by Lu and Ju (1989) and Chellam and Wiesner (1998). Much work is done with non-interacting particles, which seems not representative for MBR, but may all the same increase fundamental knowledge.

Tardieu *et al.*(1998) measured the particle size distribution of activated sludge, and calculated for the hydraulic conditions in their installation the magnitude of the different back transport models. There was a fairly good agreement between model predictions and measurements. The observed differences were ascribed to

the influence of particle interactions, presumably caused by their biological origin.

Vyas *et al.*(2002) measured particle size distribution of the formed cake layer. They found that with constant flux experiments particles in the cake were smaller than with constant TMP, which was also observed by Chellam and Wiesner (1998). Filtering a lactalbumin suspension they were able to predict which particle size would be predominant in the cake layer (Vyas *et al.*, 2001).

Ould-Dris *et al.*(2000) tried to model the behaviour of highly concentrated $CaCO_3$ suspension (>20 g/L). They found out that they had to calculate a mean cake particle diameter, to fit the model with the measured data. The calculated mean cake particle diameter was in the range 1-2 μm, which was later confirmed by measuring the particle size distribution of the formed cake layer, after resuspending it. Kromkamp *et al.* (2002) describe modelling efforts and measurements of cross flow filtration of bidisperse solutions. One conclusion is that the steady state flux that can be reached with a bidisperse solution is comparable with the steady state flux of a monodisperse solution containing only the smallest particles from the bidisperse solution. Chang *et al.*(1996) studied the filtration behaviour of bi- and trimodal suspensions of latex particles, and found that introducing bigger particles leads to higher flux, i.e. more permeable cake layer.

Colloids may interact and thus influence filtration behaviour of suspensions, as investigated by Bacchin *et al.* (1996). Cake layer permeability showed a minimum with increasing salinity while filtering bentonite suspensions, which was explained as follows. At low salt concentrations there is balance between surface interaction forces and drag force by the permeate flow. With increasing salt concentrations the repulsive forces become smaller, which leads to cake packing. Further increasing salinity will lead to coagulation, which results in bigger particles and lower cake permeability. A high stability (no further coagulation takes place) of the suspension is favourable for filtration.

Bacchin *et al.* (2002) go further by developing a model that describes fouling during ultrafiltration of colloidal suspensions. The model combines interaction of the colloids, concentration polarisation and deposition phenomena. Figure 3-1

is taken from this article and illustrates the modelling approach. Cross flow filtering of a colloidal suspension with small colloids will lead to concentration polarisation that is balanced by osmotic (back transport) effects. Increasing the driving force will lead to intensive concentration polarisation that can not be checked by the osmotic pressure, leading to particle interaction and eventually gel layer formation. If the colloids are larger, the osmotic back transport mechanism is not relevant anymore and increasing driving force will lead to deposition. This transition is more definite than in the case of small colloids, indicated by the grey gradation. It must be remarked that calculations were made for suspensions containing only particles with one particle size.

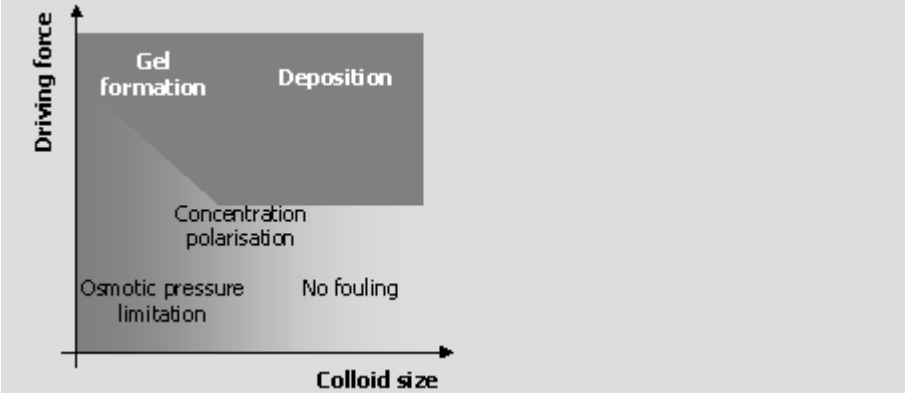

Figure 3-1 *Diagram showing fouling mechanisms as a function of colloid size (or surface repulsion) and driving force (TMP or flux). From: Bacchin et al. (2002)*

The model shows its capacity to describe continuous transition from concentration polarisation to cake formation by seeing cake formation in a feed-back relationship with the concentration polarisation: the cake layer is formed when the volume fraction at the membrane exceeds a critical value corresponding to a maximum osmotic pressure and the cake formation causes a decline in flux until concentration returns to the critical value. The model seems to explain findings by Chan and Chen (2001), who found while filtering a protein solution with stepwise increasing flux, the increase of the wall concentration and onset of apparent critical flux precede an increase in rejection. Comparable experiments were performed by Chen *et al.*(1997) with colloidal

silica, that seem to be in the 'large colloid size range', reflected as a sharp transition between critical flux and cake filtration.
Application of this type of model is restricted to cases where the exact relation between viscosity and shear rate is known, which requires amongst others knowledge of the flow profile (Rosenberger, 2002).

3.2.4. Empirical models

Empirical models are as numerous as there are researchers. Many of the models are based on models for dead-end filtration. Benitez *et al.* (1995) applied a model for unstirred dead-end filtration to submerged activated sludge filtration. This model states that the cumulative permeate volume is proportional to the square root of the elapsed time (Van den Berg and Smolders, 1990). The data fitted the model quite well. They also applied the power law for compressibility of the cake layer (Ramalho, 1983):

$$\alpha_b = \alpha_0 \cdot \Delta P^{\gamma} \quad \textbf{(3-2)}$$

where α_b = specific resistance of boundary layer [m/kg]
α_0= specific resistance for a unit value of P
γ = compressibility coefficient
ΔP = applied TMP [Pa]

and found γ-values of 0.661. Remark that submerged filtration is considered as dead-end filtration here, as do Engelhardt *et al.*(1998). Mallubothla and Belfort (1996) describe a model in which flux decline is a function of time as follows:

$$\frac{J(t)}{J_0} = e^{-\frac{t}{A+Bt}} \quad \textbf{(3-3)}$$

where J_0 = initial flux [m/s]
t = time [s]
A = time constant for cake growth [s]
B = constant for cake growth [-]

The constants *A* and *B* must be determined experimentally. The effectiveness of a backflush is modelled in the same way. Although it is relatively easy to determine the constants and reproduce measured filtration curves, it is questionnable what the physical meaning of the constants is, see also Mores *et al.*(2000) for a further development of this model.

Tardieu *et al*. (1999) performed interesting experiments in which the filtration behaviour of different suspension was measured. Four different activated sludges under steady state biological conditions were tested. The filterability of the different suspension differed according to hydrodynamic and biological conditions. Two models were developed to describe the change of filtration resistance in time, see eq. 3-3. One is based on an energy comparison between the shear forces and the kinetic energy linked with permeation, see eq. 3-4; the second is based on a description of the mass transfer close to the membrane, see eq. 3-4. The general form of the model is given as:

$$\ln\left(\frac{\partial R}{\partial t}\right) = p - q \cdot \chi \qquad \textbf{(3-4)}$$

where

$$\chi = \frac{\mathrm{Re}^{0.8} \cdot \tau_0}{\rho \cdot J^2} \qquad \textbf{(3-5)}$$

or

$$\chi = \frac{\mathrm{Re}^{1.3}}{J \cdot d_h} \qquad \textbf{(3-6)}$$

and

Re	= Reynolds number	[-]
τ_0	= wall shear stress	[Pa]
d_h	= membrane channel diameter	[m]
ρ	= mixed liquor density	[kg/m^3]

The model gives a useful description of the hydrodynamic influence on the rate of fouling, which depends on the biological conditions.
In order to make the model describe the measured data a term is introduced to account for 'the varying "local" composition of the suspension on the surface of the membrane'. The same type of approach is followed by DeWilde *et al.* (2005) who introduce a term describing a representative viscosity of the boundary layer.

The mentioned models try to describe the transient filtration behaviour, i.e. permeate flux in time during one filtration run.

Other researchers try to characterise the situation after reaching steady state filtration. Elmaleh and co-workers (1998) did much work on characterising cross flow filtration by dimensionless numbers. They suggest two dimensionless parameters, the shear stress number, N_s:

$$N_s = \frac{\rho \cdot u_c^2}{\Delta P} = \frac{\tau}{\frac{f}{2}\Delta P} \quad \textbf{(3-7)}$$

where		
	ρ = density	[kg/m^3]
	u_{cr} = cross flow velocity	[m/s]
	ΔP = Trans Membrane Pressure	[Pa]
	τ = shear stress	[Pa]
	f/2 = friction factor	[-]

N_s compares shear stress to the driving force of the filtration process. The second number is the resistance number, N_f:

$$N_f = \frac{\mu \cdot R_f \cdot u_c}{\Delta P} \quad \textbf{(3-8)}$$

where		
	μ = dynamic viscosity	[Pa·s]
	R_f = filtration resistance - R_m	[m^{-1}]

N_f compares the convective transport through the tubular element with a hypothetical flux through the resistance layer. N_f and N_s can be calculated for each case where filtration has reached steady state. If N_f and N_s are plotted in one figure, straight lines are obtained, giving indications as to which type of fouling occurs. The obtained lines can be described with

$$N_f = a + b \cdot N_s \quad \textbf{(3-9)}$$

A negative slope and an intersection with the N_s axis mean that the cross flow can completely eliminate the cake layer formation or polarisation. A negative slope without intersection with N_s axis mean that mass transport-limiting process can only partially be eliminated. A positive slope means irreversible fouling.

3.2.5. Discussion of Fouling models

The Resistance in Series Model can be applied by determining different contributions to fouling by different cleaning steps, or by feed fractionation. Neither of these methods takes into account interdependencies between the different fouling mechanisms on the one hand and the different fractions on the other. In the former case it only gives information about the reversibility of the different fouling types. It is a practical way however to obtain an idea of the predominant type of fouling that occurs.

The Resistance in Series Model also does not take into account the influence of cross flow velocity. The application of this model in cross flow filtration requires standardisation of quantifying the influence of cross flow, e.g. by keeping it constant.

With Mass Transfer Models this problem is overcome, since it predicts attainable flux given particle size distribution and shear rate at the membrane. This is a problem, since shear rate in activated sludge flow with wall permeation is not known *a priori*. Another shortcoming of the Mass Transport Model is that it supposes particles to be spherical and non-interacting, which is known to be a not completely correct assumption.

Empirical models have the drawback that the physical meaning of the introduced constants is rather vague. The models can be used to optimise filtration processes, but are most of the time not very useful for scientific research.

3.3. Critical Flux

3.3.1. Definition of critical flux

In the efforts to determine, describe and quantify filtration characteristics the concept of critical flux has found wide acceptation. Several definitions of critical flux exist, of which the most cited, given by (Field, 1995), is mentioned here: "The critical flux hypothesis for MF is that on start-up there exists a flux below

which a decline of flux with time does not occur; above it fouling is observed. This flux is the critical flux and its value depends on the hydrodynamics and probably other variables." Remark that in this definition the permeability of the membrane equals the clean membrane permeability. When operating under sub-critical conditions constant flux operation equals constant TMP operation.
The idea behind this concept is that any cross flow membrane separation process can be operated without fouling, if operated below the critical flux. It has been shown however that for some activated sludge suspensions it is impossible to define a critical flux, since fouling occurred at any flux (Le-Clech *et al*, 2003). In case of a strong interaction between the feed suspension and the membrane, a critical flux might be non-existing since even at zero flux some species might adsorb to the membrane, causing a resistance increase.

3.3.2. Critical flux determination

The determination of critical flux is done by a stepwise increase of flux while monitoring the required TMP (Kwon *et al.*, 1997 and Madaeni *et al.*, 1996, Le-Clech *et al.*, 2003)). The highest flux at which TMP can be kept constant is regarded as the critical flux. It is also possible to do the determination with constant TMP, registrating the delivered permeate flux (Defrance and Jaffrin, 1999a) which yields a critical TMP at a certain cross flow velocity. With increasing cross flow velocity critical flux also increases. Submerged hollow fibre membranes for example, showed a linear relationship between aeration rate and critical flux while filtering clay suspensions (Madec *et al.*, 2000).

3.3.3. Secondary and weak critical flux

For application in MBR slightly different definitions of critical flux were developed. An interesting approach is the determination of the highest flux, which enables stable operation, although below clean water permeability. It is observed that in many applications flux is linearly dependent on TMP, until the critical value; however with a lower permeability than the clean water permeability, see Figure 3-2.

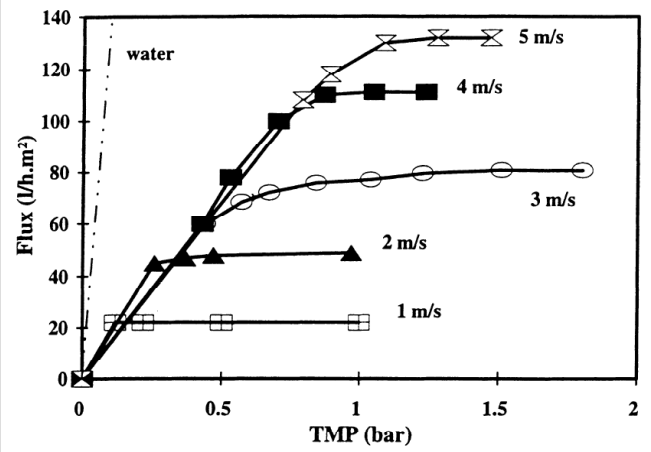

Figure 3-2 *Variation of permeate flux in the MBR with TMP at various velocities (m/s; T=20°C; MLSS = 10 g/L) from: Defrance* et al.*(2000) MBR with tubular ceramic membranes treating municipal wastewater.*

Defrance and Jaffrin (1999a) describe this value as 'the critical flux is the limit below which, when the flux is set by a pump, a stable filtration can be sustained for a long period with constant TMP'. In their experiments TMP stabilised within 15 minutes. The obtained 'weak critical flux' signifies the transition from TMP-dependent flux to TMP-independent flux (Chen *et al.*, 1997). Below critical flux the cross flow velocity has little influence on TMP (Defrance and Jaffrin, 1999b). Causserand *et al.*(1997) studied the influence on filtration behaviour of interaction between different types of suspension constituents. Interactions between the different constituents (clay particles and proteins) under different ionic strengths and pH values resulted in filtration behaviour totally different from solutions with only one of the constituents.

With regard to the hydraulic circumstances it can be said that the higher the imposed shear stress, the higher the critical flux, which confirms the results obtained with mass transfer models (Defrance and Jaffrin, 1999; Bouhabila *et al*, 1998). It must be noted that imposing a high shear stress is only possible by a high energy input, which makes it a matter of optimisation: high permeate flux versus energy costs.

3.3.4. Local critical flux

Submerged hollow fibre membranes show a non-uniform axial flux profile, which leads to non-uniform pattern of foulants deposition (Carroll and Booker, 2000). This phenomenon can lead to a local flux which is higher than the critical flux (Ognier *et al.*, 2001 and 2004; Cho and Fane, 2003).

The 'other variables' mentioned in the definition by Field *et al.*(1995) have been the subject of many studies, some of which are presented in 3.4.

3.4. Identifying Foulants and Fouling Situations in MBR

The microbial population present in the activated sludge is different from activated sludge from a conventional system (Zhang and Yamamoto, 1996; Witzig *et al.*, 2002). This can be caused by the differences in hydraulic regimes, for example expressed as shear stress, see Xing *et al.*(2001). Furthermore, there may be strong (seasonal) fluctuations in composition, without effects on the biological treatment efficiency.

Filterability may change from system to system. In come cases a better filterability is reported for conventional activated sludge, in other cases the opposite. For example, Zhang *et al.*(2004) compared an SBR with membrane filtration for sludge separation with an SBR with gravitational sedimentation. The activated sludge from the system with gravitational sedimentation produced less irreversible fouling, when filtered at a cross flow membranes. This was ascribed to the selective pressure, induced by the sedimentation step, removing those parts of the population causing the irreversible fouling.

For the operation of an MBR process it is desirable to know which circumstances should be avoided and whether it is possible to identify specific substances that cause fouling. These causes can emerge from unexpected side, like the case described by Cicek *et al.*(1999). They treated a synthetic wastewater with MBR equipped with ceramic membranes. Due to high influent phosphorus concentrations struvite crystals were formed that caused abrasion of the top layer of the membrane, leading to the replacement of the membrane unit.

The presence of particles larger than 3 mm and hairs should be avoided at any time in MBR. They tend to accumulate and stick together, disturbing the flow pattern around the membranes, thus causing serious problems (DeWilde *et al* (2003), Van der Roest (2003), Engelhardt (2003)).
The choice of pumping device is of major importance for the filterability of the mixed liquor. Kim *et al.*(2001) showed that applying a pump which causes high shear rates results in a less filterable activated sludge.

3.4.1. MLSS and Particle Size Distribution

MLSS Concentration

Since one of the advantages of MBR is the possibility to increase MLSS concentration, this was one of the first parameters to be investigated. There seems to be no direct relation between fouling and MLSS concentration (Lubbecke *et al.*, 1995; Günder and Krauth (1999), Rosenberger *et al.,* 2002). Bouhabila *et al.*(1998) studied apparent critical flux with MLSS concentrations of 4, 8.2 and 15.1 g/L. They found out that a 'secondary critical' flux could be observed, where permeability was lower than clean water permeability but a stable operation was possible (see also: 3.3.3 Secondary and weak critical flux). Defrance and Jaffrin (2000) did the same experiments with MLSS concentrations from 2-6 g/L and the obtainable flux did not decrease much. Fan *et al.* (2000) made the same observations with MLSS concentrations between 3 and 7.8 g/L., Hong *et al.*(2002) with MLSS concentrations between 3.6 and 8.4 g/L as well as Krauth and Staab (1999).
MLSS concentration nevertheless influences filtration behaviour through viscosity, which is among others a function of MLSS.

Particle Size

A further investigation of the constituents of the mixed liquor and their respective contribution to fouling was undertaken by Wisniewski and Grasmick, (1998), Defrance and Jaffrin (2000), Bouhabila *et al.* (2001), and others. Bouhabila *et al.* (2001) fractionated by centrifugation of the activated sludge and flocculation of the activated sludge followed by centrifugation. The data obtained in this way state that colloids are responsible for 50% of the fouling. All studies apply different means of fractionation, resulting in different and

contradictory results. Furthermore, the filtration cells used to determine filterability or specific resistance of the different fractions are operated under totally different circumstances than the actual conditions in an MBR. However, the general observed trend is that soluble constituents are involved in membrane fouling for at least 50%.

A closer investigation of this fraction made clear that the so-called extracellular polymeric substance (EPS) form the most 'dangerous' part of the soluble fraction (Rosenberger and Kraume, 2002).

Measuring the particle size distribution of an activated sludge broth demonstrates that activated sludge with a higher mean particle size is better filterable than activated sludge with a small mean particle size (see e.g. Lim and Bai, 2003).

3.4.2. EPS in Activated Sludge

Extracellular polymeric substances (EPS) are biological polymers of microbial origin, in most cases polysaccharides and proteins, but also nucleic acids, lipids, etc. (Flemming and Wingender, 2000). EPS form an essential part of activated sludge. Among the many functions of EPS are: formation of flocs and a protective layer around the cell(floc). EPS also facilitate interactions between cells and their environment. These interactions are often essential for microbial survival. Apart from EPS that is bound in microbial flocs, EPS can be found in the water phase as free EPS. Substances in this category originate from break up of flocs and cell lysis or can be introduced by the influent (Wingender *et al.*, 1999). Another group of substances overlapping the EPS is called Soluble Microbial Products (SMP). This group contains a wider range of substances that can also be defined in different ways. Boero *et al.*(1991) for example state that SMP result from intermediates or end products of substrate degradation and endogenous cell decomposition.

Little is known about the circumstances that influence EPS production and their possible release to the water phase. Some authors report an influence of SRT and F/M. Others suggest that ionic strength and substrate conditions, such as influent C:N:P-ratio and lack of O_2, are involved in this process (Flemming and

Wingender, 2000). Kuo (1993) lists a range of factors that cause SMP production, cited in Barker and Stuckey (1999):

- Concentration equilibrium: organisms excrete soluble organic material to establish concentration equilibrium across the cell membrane.
- During starvation bacteria excrete organic material
- Increased presence of energy source
- Sudden addition of carbon source and energy source to bacteria starved for carbon and energy may accelerate death to some bacteria, which may result in production of SMP
- If essential nutrients are available in low concentrations, SMP may be produced to scavenge the required nutrients
- To relieve environmental stress, such as temperature changes, osmotic shocks and maybe in response to toxic substances
- During normal bacterial growth and metabolism SMP are produced.

There seems to be an optimum SRT for which SMP production is minimum.
The same holds for organic loading rate; minimum SMP production was observed at 0.3-1.2 g COD/g MLSS·d. Increasing MLSS concentration leads to higher SMP concentrations (Barker and Stuckey, 1999).
EPS in activated sludge is mainly composed of proteins and polysaccharides (Flemming and Wingender, 2000). Despite the knowledge about the importance of the water phase for membrane fouling, many researchers prefer to analyse the total amount of EPS in both microbial flocs and the water phase (Trussell *et al.*, 2004). This analysis requires the breaking up of the microbial flocs, which can be done in many ways, leading to different results (Frølund *et al,* 1995). Probably this is the reason for contradictory results. Another complicating factor is the wide range of methods to analyse the EPS content, which makes it difficult to compare results from different researches (Huber, 1999).
Laspidou and Rittmann (2002a; 2002b) present a theory and a model in which the EPS, SMP and biomass are incorporated, see Figure 3-3.

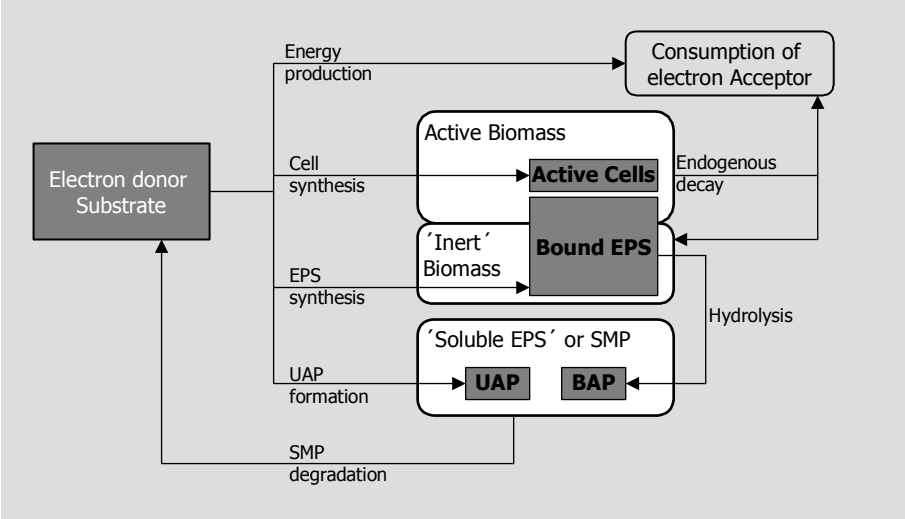

Figure 3-3 *Schematic representation of the model by Laspidou et al. (2002a)*

Their conclusion is that soluble EPS is actually SMP. They further make a distinction for utilization-associated products (UAP) and biomass-associated products (BAP).

3.4.3. EPS and fouling

Ishiguro *et al.*(1994) describe filtration experiments with supernatant after centrifugation of activated sludge from an MBR. The concentrations of Dissolved Organic Carbon (DOC), proteins and sugars were measured both in the concentrate and the permeate. The difference between these concentrations was plotted against stable flux values and an exponential relation was found: high stable fluxes occurred with low concentration differences for soluble proteins, sugars and DOC. Also Nagaoka *et al.*(1996) revealed a relation between EPS and membrane fouling, although they determined total EPS concentrations in the mixed liquor. Furthermore they analysed the amount of EPS attached to the membrane and modelled the attachment process to compute cleaning intervals (Nagaoka *et al.,* 1998).

Wisniewski *et al.*(2000) also pointed at the importance of the soluble fraction of the activated sludge; see also Wisniewski and Grasmick (1998) for the influence

of shear rates on concentrations of soluble material in the water phase, leading to more fouling. Ognier *et al.*(2002) filtered the total mixed liquor and the supernatant after centrifugation; they conclude that 'the irreversible part (of the fouling) is due to the soluble fraction of the suspension and is independent of the filtration time'. The adsorption phenomenon shows specific hydraulic resistance of the same order of magnitude as the clean membrane resistance.
Cho and Fane (2002) ascribe the slow rise of TMP during the first operational period (375 hour) of an anaerobic MBR to EPS deposition, confirmed by membrane autopsy. Chang and Lee (1998) ascribe the observed decrease in filterability during stirred cell filtration experiments to amount of EPS bound to flocs. This amount can be related to the physiological state of the biomass.
Cicek *et al.*(2002) found out that soluble organic compounds could be related to permeate flux. Organic matter smaller than 0.1 μm exhibited the strongest correlation to permeate flux.
One of the possibilities to reduce fouling is the addition of alum or zeolites to the biomass (Lee *et al.*(1999). The alum dose was determined by the amount of phosphate. The zeolite concentration was kept at 1 g/L. Both these additives resulted in a slower fouling of the membrane.

A recent development is the analysis of the water phase of the activated sludge with respect to proteins and polysaccharides as also applied in this thesis, Rosenberger *et al.*(2000, 2005); Rosenberger and Kraume (2002); Rosenberger (2003); Lesjean *et al.*(2004)). They describe a linear relationship between the fouling rate in pilot plant and the amount of polysaccharides in the water phase.

3.5. Fouling and biological operating parameters

Bouhabila *et al.* (1998) found that SRT of 30 days induced more fouling than SRT of 10 and 20 days. Lee *et al.*(2003) found that SRT of 60 days led to more membrane fouling than 40 days; SRT of 20 days resulted in the least fouling. Chang and Lee (1998) investigated the influence of physiological states of the biomass on membrane fouling. By varying the SRT from 3, 8 to 33 days the filterability of the biomass got better. Rosenberger *et al.*(2002) experienced no difference in filtration behaviour with SRT's ranging from 5 to 40 days. Choi *et*

al.(2002) measured higher fouling propensity under filamentous sludge bulking conditions compared to normal conditions.

Chang *et al.*(1999) describe experiments in which different types of activated sludge flocs are promoted by varying the F/M ratio and HRT. Subsequently the filterability of the activated sludge is measured in a stirred cell experiment. It was concluded that the composition and size distribution of organic compounds in activated sludge ultimately determine the effectiveness and feasibility of membrane filtration in wastewater treatment. Controlling the soluble organic matter content in a MBR is essential for improving filtration performance and reducing operational costs.

During the experiments described by Ng *et al.*(2000) the filterability of the biomass changed concurrent with changes in microbial population.

3.6. Fouling and hydrodynamic operating parameters

3.6.1. Shear rate and cross flow

The most frequently used methods to control fouling in MBR are the coarse bubble aeration of the membranes (in submerged configuration) or the cross flow, sometimes in combination with air bubbles (in side stream configuration).

The hydraulic configuration of the membrane filtration step is of major importance in the types of fouling that may occur and the extent to which this will be the case. Most important quantity is the scouring effect of the cross flow, or the aeration of the membranes. Increasing the air flow will increase attainable stable permeate flux up until a threshold value where no more performance increase is observed (Hong *et al.*, 2003).

Hong and co-workers (2002) describe the influence of introducing a relaxation period (intervals of zero production), resulting in slower flux decline, due to enhanced removal of foulants accumulated at the membrane surface. Chua *et al.*(2002) examined the effectiveness of relaxation with continuous aeration in order to obtain long term sustainability with high fluxes.

The effectiveness of periodic relaxation was observed by many others (e.g. Hütter *et al*, 2000) and is at the moment state of the art for e.g. Kubota and Zenon MBR systems (Meraviglia *et al*, 2003, Kraume and Bracklow, 2003).

The relaxation period can be made more effective by reversing the permeate flow with a backflush. Usually, the backflush flow is higher than the permeate flow e.g. 1.25 times higher (DeWilde *et al.*, 2005). Backflushing is usually impossible with plate and frame systems, because of the risk of removing the membrane from the support layer. The interval between backflushing usually is in the range of minutes. Some experiments were performed with high frequency backpulsing, e.g. Kuberkar *et al.*(1998). The membranes were backflushed with backpulse durations between 0.1 and 1 sec., which resulted in net flux increase up to 1000%. It seems difficult to realise a backpulse with such a frequency at large scale. However, these experiments point out that the cake layer formation takes place very fast.

A side–effect of increased shear rate is the release of 'dispersed primary particles' from activated sludge flocs (Mikkelsen and Keiding, 2002), thus changing the particle size distribution and filterability of the mixed liquor.

3.6.2. Membrane material and pore size

Filtering activated sludge with two membranes with the same pore size but with different hydrophobicity showed different fouling mechanisms to occur (Chang *et al.*, 2001). The hydrophobic membrane always had more cake layer formation. The same membrane showed higher solute rejection indicating that the cake layer acts as an additional membrane with smaller pore size.

Hydrophobic membranes tend to have lower critical fluxes than hydrophilic membranes, which suggests surface interaction to play a role, even prior to convective deposition of cake layers (Madaeni *et al.*(1999)).

Wu *et al.*(1999) measured a decreasing critical flux with increasing pore size, filtering silica colloidal suspensions, BSA solutions and baker's yeast suspensions.

Le-Clech *et al.*(2003) found out that pore size almost did not affect critical flux for MLSS concentrations ranging from 4-12 g/L. At low MLSS concentrations the membrane with the smallest pore size (0.1 μm) showed the lowest critical flux.

3.7. Concluding Remarks on Literature Review

The filtration of activated sludge is a complex process, with many interacting parameters. Therefore many attempts were made to identify dimensionless numbers, specific fouling species or sub-critical operating conditions.
Considering the general filtration equation,

$$J = \frac{TMP}{\eta_p R_t} \quad \textbf{(3-10)}$$

it is clear that, given a certain set of hydraulic conditions, three parameters determine filtration performance: TMP, η_p and R_t.

Connected with these three parameters, three groups of flux influencing parameters can be distinguished, determined by the filtration process: the feed flow (i.e. the activated sludge), the process conditions, and the membrane. Each element in these groups affects one or two of the parameters in eq. 3-1. Even this arrangement leaves out some interactions, but still it gives insight in the many factors that have to be taken into account in research on MBR fouling.
In table 3-2 these relationships are summarised, according to the three groups of influencing parameters and their effect on TMP, η_p and R_t.

This study will primarily focus on the first group of factors: Activated Sludge Properties and their influence on filtration performance. With the current state-of-the-art in cross flow filtration theory and membrane manufacturing, the other two groups can be kept constant to the level needed for this purpose.

This is achieved by doing experiments under well defined conditions, as described in *Chapter 4*.

This results in accurate filtration data that lay the foundation for a fair comparison of filtration performance of different installations, or the monitoring of filtration characteristics of an activated sludge that has been subjected to different conditions.

Table 3-2 *Factors Influencing Flux in MBR*

Activated Sludge property	**Affects**	**via**
PSD	R	Cake/gel layer formation
Floc Type	R	Cake layer formation
η_{sludge}(TSS,…)	TMP	Shear stress
Solutes (amount and quality)	R	Adsorption, Pore blocking, Cake layer
pH	R	Adsorption of some species, crystallisation, denaturation of proteins
Temperature	$\eta_{permeate}$(T)	
Process condition	**Affects**	**via**
u_{cr}	R	Shear stress and cake layer formation
Constant J/Constant TMP	TMP, R	Shape of R(V); implicitly also R - cake layer formation, adsorption and pore blocking
Membrane	**Affects**	**via**
Material	TMP, R	CWF; adsorption, pore blocking
Pore size, Porosity	TMP, R	CWF; cake layer formation, adsorption and pore blocking
Configuration	TMP	Determines CWF, cleaning strategy, process conditions.

However, the results of the mentioned research do not yet enable conclusive results from interpreting filtration data on its own. This requires additional analyses of the feed flow in terms of chemical composition and its physical properties.

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' We must not think of the things we could do with, but only of the things we can't do without.'
J.K. Jerome, Three men in a boat (to say nothing of the dog)

4
MATERIALS AND METHODS FOR CHARACTERISATION OF ACTIVATED SLUDGE MEMBRANE FILTRATION

This chapter describes the materials and methods applied for the measurement of filtration characteristics of activated sludge membrane filtration. It ends with a short description of the results that are obtained by measuring according to this protocol.

4.1. Installation Requirements

The installation which will be used for the assessment of the filterability of activated sludge from MBRs must answer several requirements. Basically, a filtration unit consists of the following elements:

- membrane
- pumps
- measuring and control facilities
- data acquisition facilities
- possibility to perform cleanings and maintenance of the membrane.

These elements will be discussed in the following paragraphs.

4.1.1. The Membrane

Membrane configuration

The filtration step in an MBR system can be designed in different ways. Many membrane configurations are available on the market, which can be divided in two classes: 1. Submerged membranes, in the form of hollow fibres or plate and

frame membranes, and 2. Side stream membranes, commonly tubular membranes, see also Chapter 2.
Submerged membranes are operated outside-in, which means that the permeate flows from the outside of the membrane structure to the inside of the fibres. The driving pressure is a vacuum at the inside of the membrane.
Side stream membranes are operated inside-out, which means that permeate is forced through the membrane wall by hydraulic pressure, caused by the circulating flow. There are also systems that apply suction at the permeate side, to control permeate flux and crossflow velocity independently.

Operation and maintenance of these two systems can be quite different. Generally, submerged systems are operated with lower fluxes, whereas the required circulation flow in the side stream configuration demands an extra energy input. On the other hand, submerged systems are equipped with coarse bubble aeration to ensure continuous cleaning of the membranes. This also negatively influences the energy efficiency of the process.
The most important difference between these systems lies in the degree of controllability of the flow pattern and permeation. In the case of a bundle of hollow fibres, bubble aeration will not always be sufficient to keep the whole permeation area clean. The parts that are fouled, i.e. (partially) blocked, become unavailable for permeation. The remaining part will face a higher permeation rate, since the overall average permeation rate is kept constant (in constant flux operation). Locally, the flux will be much higher than average. Second, the flow pattern around the membrane bundle is not constant; every now and then pieces of cake layer will be removed, increasing the available permeation area. This process contains too many unknown parameters to be used at small scale for filtration characterisation. Therefore, for filtration characterisation purposes preference is given to a side stream membrane configuration, which ensures a continuous and well defined flow pattern along the membrane.

Membrane material
The most commonly applied membranes in MBR are made of hydrophilic or hydrophilised organics. Some authors report an importance of pore size, others

deny major influence of this factor, see also chapter 3. In this study it is assumed that the most determining factor in the filterability of activated sludge is the quality of the water phase, and the membrane properties are of minor importance, given that it is a hydrophilic membrane.

Membrane module

The above considerations led to the conclusion that a tubular hydrophilic membranes may be used for filtration characterisation.

The membranes applied in this study were provided by the X-Flow company. The company sells several types of side stream membranes for activated sludge filtration. During the preliminary experiments, F5385 modules were used (see appendix III for technical data). This module constists of a 1 inch PVC tube with 12 membrane tubes, each with a diameter of 5.2 mm., see Figure 4-1. The membrane module is placed vertically with upward flow direction. The twelve membrane tubes are glued with resin at both ends of the PVC tube.

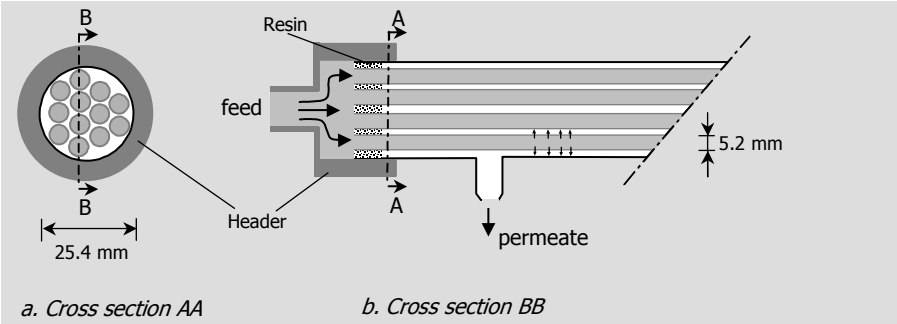

Figure 4-1 *Cross section and explanation of working principle of standard membrane module*

In the course of the experiments it turned out that with low crossflow velocities some tubes got clogged with sludge. The module was therefore changed to a hand-made single tube module, containing one membrane tube with a diameter of 8 mm.

Another drawback of the common module for this application is the header that is used to divide the feed stream over the twelve membrane channels. Firstly, this causes additional turbulence for the sludge, as well as additional headloss

and it also disturbs the flow profile in the first part of the tubes. The measurement of the TMP is complicated, since the pressure measurements are fixed in the circulation line, before the inflow-header and after the outflow-header.

These drawbacks are overcome in the single tube module. The diameter of the membrane tube is bigger, allowing lower crossflow velocities, with less chance of channel clogging. The header is reduced to a tube of the same diameter as the membrane tube, ensuring undisturbed entrance of the membrane tube, see Figure 4-2. The pressure measurement is fixed in the header which makes the TMP measurement as accurate as possible.

The membrane is glued with resin in a 15 mm PVC tube in order to decrease the amount of permeate volume in the module (compared to one membrane tube in a 25.4 mm PVC tube).

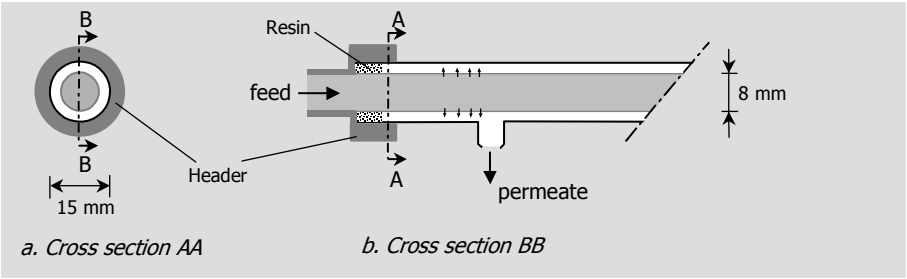

Figure 4-2 *Cross section and explanation of working principle of customised membrane module*

4.1.2. Pumps

The installation uses four pumps: two pumps for circulation, one for backflushing and one for permeate extraction.

The two circulation pumps are used:

1. For the circulation of clean water during clean water measurements and during a clean water forward flush. This pump must be able to produce the same crossflow velocity during the clean water measurements as is applied during activated sludge filtration. Furthermore, during the forward flush with clean water the crossflow velocity must be increased up to > 4 m/s. To this aim a centrifugal pump is applied.

2. For the circulation of activated sludge a positive displacement pump is used. In this way the floc structure of the sludge is not damaged. To this aim a peristaltic pump is used, see the picture in Figure 4-3.
   This pump operates at a rather low circulation speed to produce the desired crossflow velocity. This leads to considerable fluctuations both in crossflow velocity and pressure. An absorber was constructed which sufficiently damped these fluctuations. The range of crossflow velocities that can be applied is 0.6 to 2 m/s.

Figure 4-3 *Peristaltic pump for circulation of activated sludge*

3. The backflush pump must be able to produce a reverse TMP at least as big as the TMP during filtration. In these experiments the maximum TMP is 0.7 bar and the backflush pump was able to produce a TMP of –0.75 bar.

4. The permeate pump is also a peristaltic pump in order to allow accurate and continuous permeate extraction. The pump is connected to the permeate outlet of the membrane module. The permeate flux rate could be set between 0 and 190 L/m^2·h.

4.1.3. Measuring and control facilities

Since the installation is used for scientific research, special care was taken to ensure accurate measurement of the relevant parameters and their control. Some parameters must be available online in order to control the process:

- Crossflow velocity. This requires a flow meter in the main stream with a range 0.6 – 5 m/s. With a tube of 8 mm, this means a maximum flow of 905 L/h. A Krohne electromagnetic flow meter (IFC 010K) is used.

- TMP. Three pressure transmitters are used to measure TMP:
    - Immediately before the entrance of the membrane tube, P1
    - Immediately after the outlet of the membrane tube, P2
    - In the permeate stream, P3.

  The first two are used to measure the pressure drop through the membrane element. The third one measures the pressure in the middle of the membrane module, at the permeate side. Transmembrane pressure is calculated as the difference between the mean of the pressure in the feed flow and the pressure at the permeate side:

$$TMP = \frac{P1 + P2}{2} - P3 \qquad \textbf{(4-1)}$$

  The applied pressure transmitters are from the Labom company, type CB3010, see Appendix IV. The pressure transmitters in the feed flow cover a range from 0 to 2.5 bar; the pressure transmitter in the permeate from –1 to 0.6 bar.
- Permeate flux is measured by collecting the permeate on a mass balance connected to a PC. The PC calculates the permeate flux with a time interval of 10 sec.

For online process control filtration resistance is calculated each time step. To this aim also temperature is measured. During an experiment the activated sludge is aerated with a aeration stone which keeps the oxygen content above 2 mg/l. pH and oxygen content in the activated sludge batch are measured with standard electrodes from the company WTW.

The feed flow to the membrane module can be either clean water (demineralised water or MBR permeate) or activated sludge. The switch between the several modes of operation (e.g. activated sludge filtration, clean water measurement,

backflush) is performed by a PLC which can switch pumps on and off as well as open or close two- and threeway valves. The valves are operated with pressurised air.

4.1.4. Data acquisition

The software Testpoint was used to develop a programme with which the data can be presented online and logged on a hard disk. The programme consists of an input screen for the installation characteristics and a presentation screen showing the filtration curve. The programme calculates for each time step:

- crossflow velocity [m/s]
- transmembrane pressure [bar]
- permeate flux [$L/m^2 \cdot h$]
- filtration resistance [m^{-1}]

These data are recorded on the hard disk for later evaluation.

4.1.5. Cleaning and maintenance

Apart from hydraulic cleaning of the membrane by crossflow, the membranes can be cleaned with chemicals. Usually the membrane is soaked in a solution or chemicals are added to the backflush stream. In this case the backflush flow was too low to add chemicals to the backflush stream. Therefore a cleaning system was developed in which cleaning agents were inserted in and/or circulated through the membrane module (at the feed side) with a peristaltic pump.
The applied cleaning agent was NaOCl with an active chlorine content of 500 ppm.

4.2. Installation

Based on the above considerations, an installation was built with which crossflow filtration characteristics can be assessed, see Figure 4-4.
The core of the installation is formed by the tubular membrane element, with an inner diameter of 8 mm and a length of 950 mm. To avoid damage of the floc structure, the activated sludge is circulated with a peristaltic pump. The fluctuations in flow and pressure, caused by this pump are equalised in an absorber.

The circulation flow is regulated with the rotation speed of the peristaltic pump, and the permeation flow rate is regulated with a second peristaltic pump, which allows a flux range of 0 – 190 l/m² ·h.

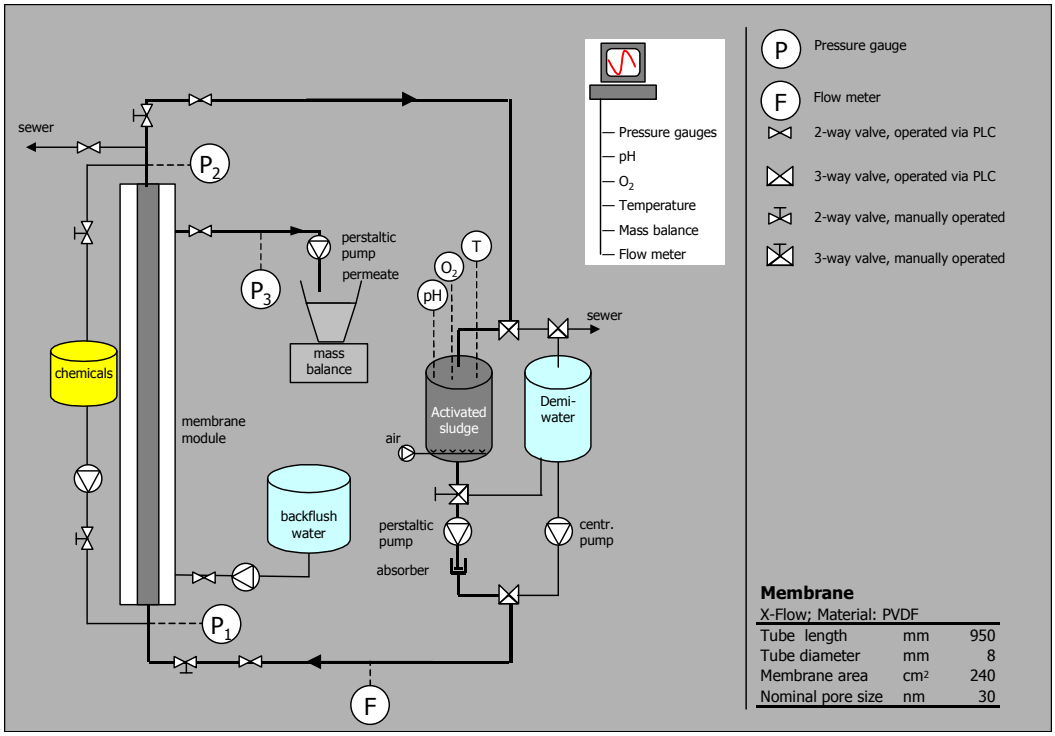

Figure 4-4 *Schematic drawing of filtration characterisation unit*

Clean water resistance can be measured by filtration of demineralised water, which is circulated with a centrifugal pump allowing crossflow velocities up to 4 m/s.

A backflush can be performed with a transmembrane pressure of 0.75 bar, and a third peristaltic pump is applied for circulating chemicals for a chemical cleaning.

The vessel for the clean water contains some 100 litres; the vessel for activated sludge contains some thirty litres. During a filtration experiment, which lasts

about 15 to 20 minutes, the content of the activated sludge vessel is circulated 2 times.

4.3. Hydraulic circumstances during filtration characterisation

The choice of hydraulic operating conditions during filtration characterisation is mainly determined by two considerations:

- The time needed to carry out a filtration characteration measurement should not be too long, to allow quick assessment of filtration behaviour. The aim is to develop a measurement which enables characterisation within 60 minutes.
- Occurring fouling mechanisms should be the same as in the original MBR. One or both parameters (flux and cross flow velocity) should be adjusted in such a way as to provoke the occurrence of fouling. This should not be exaggerated too much, to avoid the shift towards types of fouling which are not typical for MBR. The idea is to accelerate the fouling process to such extent that it can be measured accurately.

The accuracy of the characterisation method can be evaluated by meeting the following conditions:

- The obtained filtration characteristics can be correlated to the long term filtration behavior of the MBR system it is taken from. Supposed that the filtration characterisation only exaggerates processes that occur in the 'source'-MBR, the results should reflect phenomena that are encountered there.
- The measurements are reproducible with an accuracy of +/- 5%. The filtration characterisation should not change filtration characteristics, which can be assessed by repeatability of the measurement.

Tubular cross flow filtration enables control over the crossflow velocity, and also over the permeation rate. Transmembrane pressure can be determined straight forward. The next question is under which hydraulic circumstances filtration characterisation should take place. There are two parameters that can be set in constant flux crossflow filtration:

- crossflow velocity, u_{cr}, and
- permeation rate, J.

It is difficult to find representative values for crossflow velocities in activated sludge filtration, since the applied range is quite large: 1 to 6 m/s (Evenblij, 2001). Submerged systems are usually operated with a representative crossflow velocity smaller than 1 m/s (Stephenson *et al.*, 2000), determined by the rising speed of air bubbles. Also side stream applications for the treatment of municipal wastewater apply this magnitude of crossflow. In this thesis the crossflow velocity during filtration characterisation experiments was set to 1 m/s.
Another consideration leading to this value is the observed clogging of membrane tubes at velocities lower than 1 m/s.

The permeate flux in submerged MBR systems is usually kept low, in the range 10 – 35 $L/m^2 \cdot h$ (Stephenson *et al.*, 2000), in order to avoid fouling. Side stream systems are mostly operated with higer fluxes, 20 to 200 $L/m^2 \cdot h$ (Stephenson *et al.*, 2000) since fouling can be controled by increasing the crossflow velocity. For filtration characterisation fouling should take place in a relatively short time span, otherwise the accuracy will be too low. Since the crossflow velocity was chosen to be 1 m/s it turned out that in most cases permeate fluxes had to be higher than 50 $L/m^2 \cdot h$ in order to provoke fouling.
Based on experiences with different types of activated sludge, the permeate flux for a standard experiment was chosen as 80 $L/m^2 \cdot h$. Depending on the filterability additional experiments could be done with higher or lower fluxes.

4.4. Measuring protocol

Before the start of a filtration characterisation measurement about 30 L of activated sludge is sampled from an MBR installation. This sample is inserted in the respective vessel and aeration is started.
The measuring protocol consists of seven steps:

1. **Measuring Clean Water Resistance.** In order to assess the starting condition of the membrane demineralised water is filtrated under 'standard conditions': J=80 l/m² ·h, u_{cr}=1 m/s.
2. **Filtrating Activated Sludge**. The activated sludge sample is filtrated under standard conditions (J=80 l/m² ·h, u_{cr}=1 m/s). By keeping the flux constant, the needed TMP will increase until a value of about 0.70 bar is reached, after which activated sludge filtration is stopped.
3. **Forward flush**. The membrane tube is flushed with demineralised water with a crossflow velocity of 4 m/s, whereas the permeation flux is lowered until 40 l/m² ·h. When dR/dt=0, the next step is taken.
4. **Measuring Clean Water Resistance.** In order to assess the condition of the membrane demineralised water is filtrated under 'standard conditions': J=80 l/m² ·h, u_{cr}=1 m/s.
5. **Backflush**. If the clean water resistance at this point is higher than the initial value, a backflush is carried out, with TMP= -0.75 bar.
6. **Measuring Clean Water Resistance**. In order to assess the condition of the membrane, demineralised water is filtrated under 'standard conditions': J=80 l/m² ·h, u_{cr}=1 m/s.
7. **Chemical Cleaning**. If the clean water resistance at this point is still higher than the initial value, measured under 1., a chemical cleaning is peformed with NaOCl (500 ppm active chlorine) until the membrane is clean.

During steps 1 to 6 the following data are stored in the connected computer:

- Pressure data
- Value of mass balance
- Crossflow rate
- Temperature, pH and dissolved oxygen concentration in feed vessel

With these data, TMP and permeation flux can be calculated, see eq. 4-1 for TMP; the following equation was used to calculate the permeate flux:

$$J = \frac{dM}{dt}\frac{3600}{A_m \cdot \rho} \qquad \textbf{(4-2)}$$

where	M = produced mass of permeate	[g]
	t = time	[s]
	A_m = membrane are	[m^2]
	ρ = permeate density	[kg/m^3]

The filtration resistance is calculated as follows:

$$R_t = \frac{TMP}{J}\frac{\eta_{ref}}{\eta_{act}} \quad \textbf{(4-3)}$$

The reference temperature in this study is 15 Celsius degrees, see also eq. 2-8.

For a good comparison of the obtained data and an unequivocal representation, the results are presented as filtration resistance (m^{-1}) against specific produced volume of permeate (L/m^2), i.e. the filtration curve: R(V).
The clean membrane resistance to filtration of clean water ($R_{m,0}$) can show small variations. In order to have all the filtration curves start at the same point, each resistance value was subtracted with $R_{m,0,}$ see further 4.6 *Elaboration of Results*.

4.5. Sludge Analyses

4.5.1. Activated Sludge

TSS, VSS

For any measurement on activated sludge, the total amount of Suspended Solids was determined, sometimes together with Volatile Suspended Solids. These analyses were done according to NEN.

Viscosity

In order to assess the influence of viscosity on filtration performance, a limited amount of viscosity measurements was performed. An Anton Paar Rheometer was used for this, allowing shear rate controlled experiments. The shear rate was varied between 5 and 1000 s^{-1}. Viscosity measurements were performed on the activated sludge broth, since this value determines to a great extent the characteristics of the boundary layer.

4.5.2. Water Phase

EPS Measurements

Since EPS consists of a wide range of substances, it is almost impossible to determine its concentration straightforward. Based on results from other researchers (Rosenberger, 2003) a choice was made to determine protein and polysaccharide concentrations. These analyses were performed according to the methods described by Dubois *et al.*(1956) and Lowry *et al.*(1951) , applying colorimetry. As stated before, the interest was mainly in the quality of the water phase; therefore the water phase and biomass were separated with a washed paper filter, Schleicher&Schuell 589², with pore size ranging from 7 to 12 µm. Very small quantities (about 5 ml) were filtered in dead mode.

4.6. Elaboration of Results

4.6.1. Constant Flux

An example of the obtained data with constant flux operation is presented in Figure 4-5 and Figure 4-6.

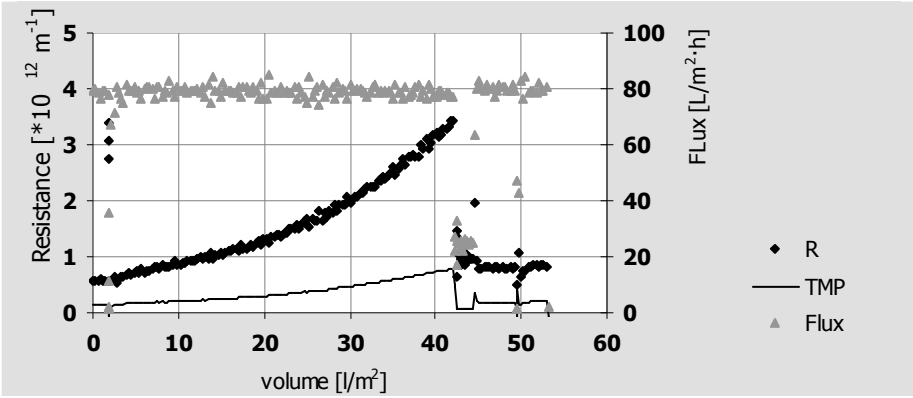

Figure 4-5 *Example of Results with Filtration Characterisation Measuring Protocol: TMP, permeate flux and filtration resistance*

In Figure 4-5, the grey triangles, plotted on the right hand axis, show the flux, the black dots show the (calculated) resistance and the drawn line shows the

(calculated) TMP, both plotted on the left hand axis. The different cleaning steps can be distinguished quite well.
After production of 42 L/m² a forward flush with clean water is applied, followed by the back flush and chemical cleaning. The forward flush is enough to remove all fouling: back flushing does not lead to a further decrease of the clean water resistance.

Filterability
The filterability of the activated sludge under consideration is expressed in the form of the filtration part of the graph in Figure 4-5: the filtration curve.
Generally the temperature of the activated sludge only slightly changed during an experiment. The energy input by the peristaltic pump was negligible or was maybe nullified by the cooling capacity of the aeration. For example, during the experiment presented in Figure 4-5 the temperature of the activated sludge batch increased from 17.3 to 17.6 Celsius degrees, see Figure 4-6.

Depending on the type of fouling that occurs, the volume-resistance graph will display different forms. A water with inert particles building a cake layer will cause a straight line, the slope of which is determined by the packing density of the cake layer.
Water with a wide range of particles, forming a compressible gel-layer will show an exponential increase in filtration resistance per volume produced permeate. A combination of different types of fouling is most likely to occur when filtrating a suspension of biomass. In this example the start of filtration follows the curve of a compressible cake layer, but after a certain produced volume the crossflow is able to control the growing cake layer and equilibrium would be reached if the experiment were extended long enough.

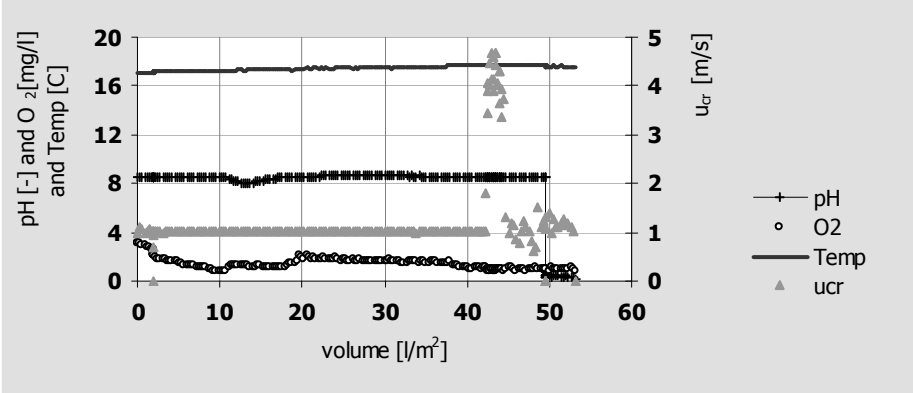

Figure 4-6 *Example of Results with Filtration Characterisation Measuring Protocol: Dissolved Oxygen, pH, Temperature and crossflow velocity during the experiment.*

These different (hypothetical) types of filtration curves, when filtrating under constant flux conditions are presented in Figure 4-7. Here the initial membrane resistance is not subtracted from R(V).

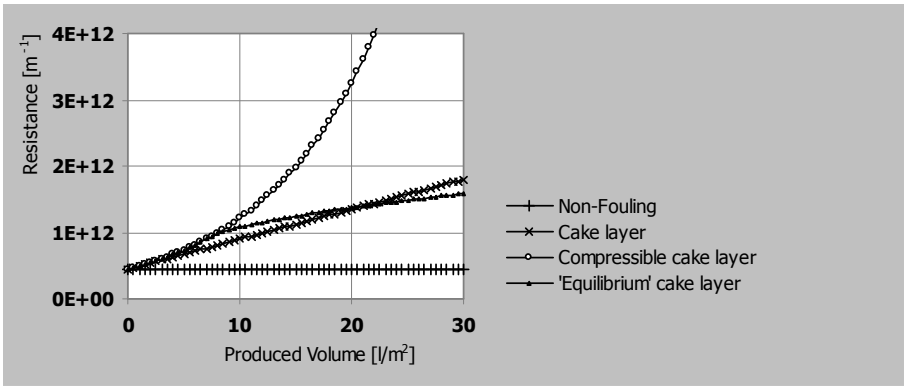

Figure 4-7 *Different Types of (Hypothetic) Filtration Curves Resulting from Constant Flux Experiments (Presented as Resistance Against Produced Volume)*

Reversibility

By cleaning the membranes with different means, information can be obtained on the reversibility of the fouling. Starting with the clean membrane resistance, $R_{m,0}$, the filtration resistance after sludge filtration is $R_{m,0}+\Delta R_{sl}$. The effect of

forward flushing is called ΔR_{ff}, the effect of back flushing ΔR_{bf}, and the effect of chemical cleaning ΔR_{cc}.
Generally, the following relation will be valid:

$$\Delta R_{sl} = \Delta R_{ff} + \Delta R_{bf} + \Delta R_{cc} \quad \textbf{(4-4)}$$

Operating under constant flux conditions allows small variations in $R_{m,0}$, since the most important fouling phenomena are exclusively dependent on the applied flux. This is contrary to constant TMP experiments where small variations in membrane resistance will cause high fluctuation in permeate flux at the start of an experiment.

4.6.2. Constant TMP

Operating with constant TMP will lead to a totally different R(V) graph. It will also start with the membrane resistance, but it will in almost all cases increase to a plateau, where there is equilibrium between fouling and scouring processes. The transport of foulants towards the membrane decreases with time. Since this is a process of which the speed is only decreasing, it requires longer time and more accurate equipment to visualise and evaluate differences.
The influence of the initial membrane resistance is much bigger, since the starting flux of the experiment is dependent on this value.

Characteristic of this curve would be the time, or amount of produced permeate, for a certain dR/dV to be reached. In this case a critical TMP could be determined, similar to the critical flux determination.

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' If you never try a new thing, how can you tell what it's like? It's men such as you that hamper the world's progress. Think of the man who first tried German sausage!'
J. K. Jerome, Three men in a boat (to say nothing of the dog)

5
EVALUATION OF CHARACTERISATION METHOD

5.1. Introduction

This chapter describes the results obtained by applying the proposed measuring protocol. In the start up phase of the project some experiments were performed operating with constant TMP. The different sampling methods are discussed in 5.2. Although the results were not totally satisfying, some features are presented in paragraph 5.3.
For more accurate data and higher representativity the mode of operation was shifted to constant flux as described in paragraph 5.4.

Since much of the work described in this Chapter was really a 'trial and error' story, almost all experiments must be evaluated individually. Practically all parameters were varied in the course of this process, teaching how to do these experiments as well as which method has to be avoided. Also the installation that was used for filtration characterisation evolved continuously: different types of valves were tested, different membrane modules, the data acquisition system was implemented, the activated sludge circulation (centrifugal) pump was exchanged for a peristaltic one, etc. An integral chronological description of these experiments would be useless; on the other hand, only presenting the 'smooth' results would leave out interesting findings and instructive failures. Therefore a compromise is presented here: a selection of the most relevant experiments and measurements.

5.2. Sampling

5.2.1. Measuring on site

The experiments described in this chapter were all performed on activated sludge taken from MBR pilots treating municipal wastewater. The pilot installations were located at the Beverwijk wwtp, which was at that time treating 317,000 P.E. of municipal wastewater (54 g BOD/P.E.·day). The pilots were being operated as part of a comparative study to assess the feasibility of the MBR technique for the extension of the Beverwijk wwtp. Four pilot plants (with capacities of 2-10 m^3/h) with four different membrane types were tested, as described extensively in the report by STOWA (2001), which was also published by IWA (Van der Roest *et al.*, 2002). Two of the pilot installations, one equipped with Zenon and the other with Kubota membranes were used most frequently as a source for activated sludge. This was mainly done in order to construct a database with good data from a few systems, and because Zenon and Kubota are worldwide the most commonly applied membrane systems for MBRs. Activated sludge from the pilot plant equipped with Mitsubishi membranes was used only few times.

The goal of this chapter is to describe how activated sludge filtration can be characterised in a repeatable and representative way, within in a short time span. A detailed description of the pilot plants is therefore not presented here. The activated sludge was sampled from the installations just after the sludge had passed the membrane tank. It had just 'seen' the membrane and was therefore supposed to be representative in terms of its composition and filterability determining properties.
The sludge samples were taken in batches of about 20-25 litres, and were aerated during the experiments. The experiments were performed immediately after sampling.

5.2.2. Measuring in the lab

Since the pilot installations were located in a non-heated Nissen-hut, the filtration characterisation installation had to be returned to the laboratory during the winter months in order to avoid (further) damage by freezing.
During this period, activated sludge was sampled at the pilot site and transported to the laboratory in closed vessels (travel time around 90 minutes) where it was aerated again before filtration characterisation took place. In this period also the activated sludge circulation pump was changed from a centrifugal to a peristaltic type of pump.

5.3. Constant TMP Experiments

5.3.1. Pressure and TMP drop

In a crossflow membrane filtration system the TMP is determined by the pressure drop along the membrane tube and the pressure at the permeate side. Since the absolute permeate flow is quite low in the permeate collector, permeate pressure is assumed to be constant. The pressure along the membrane tube is assumed to decrease linearly. This gives the following expression for TMP:

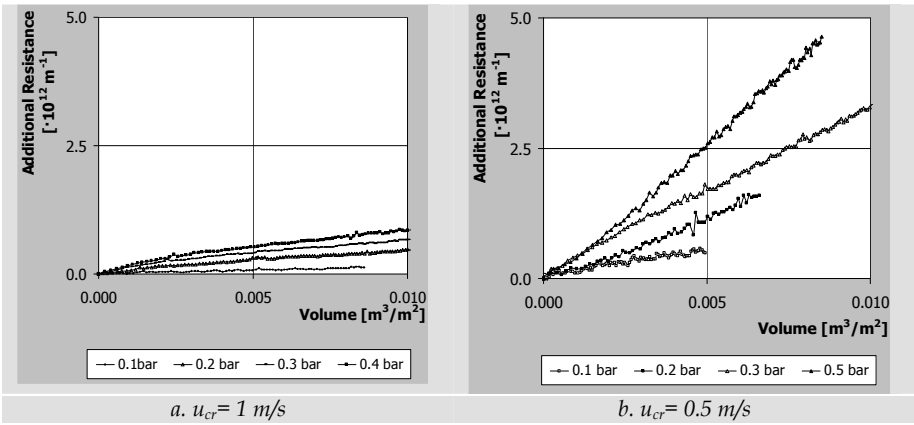
$$TMP = \frac{P_1 - P_2}{2} - P_3 \qquad \textbf{(5-1)}$$

where P_1 = pressure at membrane channel inlet
P_2 = pressure at membrane channel outlet
P_3 = pressure at permeate side

Thus, TMP is an average value, at the inlet TMP will be higher, and at the outlet TMP will be lower than the value calculated with equation 5-1, depending on the pressure drop along the membrane channel.
A higher cross flow velocity will lead to a higher pressure drop. In the set-up that was used for the constant TMP experiments it was quite difficult to regulate TMP, since each adjustment also affected cross flow velocity. During an experiment the TMP had to be adjusted which makes some filtration curves unsteady.

5.3.2. General results

The first experiments with constant TMP show a resistance development starting with a certain dR/dV, which is then either continuously decreasing, or (almost) constant. Two examples of experiments with different values for TMP are presented in Figure 5-1.



a. u_{cr}= 1 m/s *b. u_{cr}= 0.5 m/s*

Figure 5-1 Example of typical results from constant TMP experiments (u_{cr}= crossflow velocity)

The initial value of the fouling rate (dR/dV) strongly depends on the starting condition of the membrane. Starting with a fouled membrane (i.e. lower permeability) will cause the experiment to start with a lower flux, corresponding with a lower fouling rate. This is one of the drawbacks of working with constant TMP: the membrane has to be totally clean at the start of each experiment.

Another important aspect, illustrated by Figure 5-1, is the influence of the applied crossflow velocity. Bearing in mind that the clean membrane resistance ($R_{m,0}$) is around $0.5 \cdot 10^{12}$ m^{-1}, it is noteworthy that using a high crossflow velocity leads to ultimate additional resistance values of the same order as $R_{m,0}$. This is important for the question which operating conditions have to be chosen as standard for a filtration characterisation method.

5.3.3. Characterisation of Filterability

Figure 5-1 shows the general form of a filtration curve and also gives indications how to characterise it. The most interesting part is the starting phase, distinguished by the initial value of dR/dV: R'(0). This value indicates the filterability of the activated sludge. Two other values that can be used to compare filterability are:

- The additional resistance after production of a certain volume of permeate, ΔR_x.
- The slope of R(V) after production of a certain volume of permeate, R'(x).

It should be noted that the first part of the graph is important, because here the differences are most pronounced. Furthermore, during operation of an MBR in practice, a cleaning is always applied after a relatively short production interval (like back flush or relaxation), which favours focusing on the start of the measurement. Lastly, it was never observed that filtration curves, measured under identical operating conditions crossed each other. Sometimes two or more curves overlapped when reaching equilibrium, i.e. dR/dV became zero. It never happened that a curve with higher initial R'(0) would end with a lower R_∞ than another curve with lower initial R'(0). The initial development of R(V) was decisive for the whole experiment.

For the remaining part of this paragraph the additional resistance after 7.5 l/m² is used ($\Delta R_{7.5}$) , and also dR/dV at this point: R'(7.5). This (arbitrary) value was chosen because almost all filtration curves can be avaluated with this and the differences can be illustrated in this way.

5.3.4. Sensitivity for Variations in Operational Conditions

The filterability of activated sludge is sensitive for changes in operating conditions, like TMP and crossflow velocity. For example $\Delta R_{7.5}$ could be evaluated for different values of TMP, yielding graphs like the one presented in Figure 5-2, calculated from the curves presented in Figure 5-1. For those curves that were stopped before reaching 7.5 l/m² the trend line, calculated by Microsoft EXCEL was extrapolated to obtain a value.

The value of $\Delta R_{7.5}$ increases with increasing TMP in the range 0.1 to 0.5 bar. With increasing crossflow velocity, the influence of TMP increase becomes smaller and absolute values also decrease. As will be discussed further on, the value of $\Delta R_{7.5}$ as a function of applied TMP can be regarded as characteristic for a specific activated sludge.

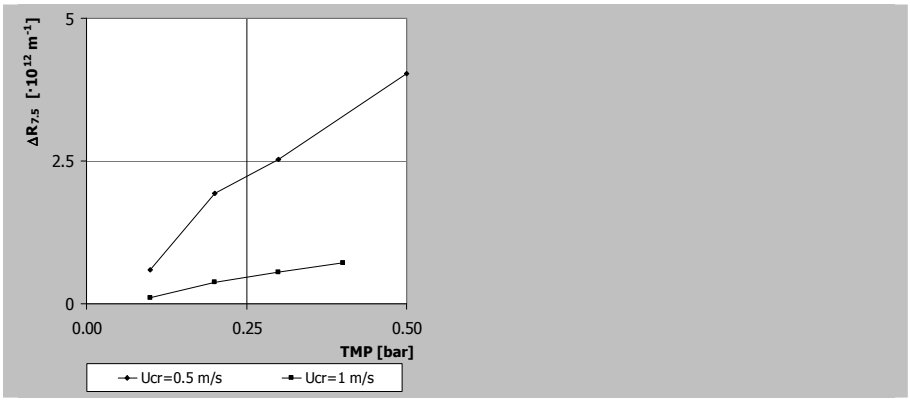


Figure 5-2 Summarising constant TMP experiments

5.3.5. Reproducibility

The reproducibility of the method was tested by filtrating a sample more than once. A sample was kept aerated for a few hours and was filtrated two or three times. In between the filtration experiments the membrane was cleaned with a 500 ppm sodium hypochloride solution.

For comparison, the additional resistance after producing 7.5 l/m² ($\Delta R_{7.5}$) was calculated for each of these tests. The average value of $\Delta R_{7.5}$ was calculated, as well as the relative deviation from the first measurement:

$$\Delta R_{7.5,n} / \Delta R_{7.5,1}$$

where n=2 or 3.

The results are summarised in Table 5-1.

Table 5-1 Results from reproducibility experiments

System	Measurement #	TMP [bar]	Average $\Delta R_{7.5}$ [10^{12} m^{-1}]	$\Delta R_{7.5,n}/\Delta R_{7.5,1}$ [-]
Kubota	2	0.30	1.04	1.01
Mitsubishi	2	0.30	1.24	1.09
Mitsubishi	3	0.30		1.08
Kubota	2	0.10	0.40	1.14
Zenon	2	0.30	0.71	0.94
Mitsubishi	2	0.30	1.16	1.04
Mitsubishi	2	0.30	1.11	1.08

The first column shows the origin of the activated sludge, the last two columns show the average $\Delta R_{7.5}$ and the ratio between the n^{th} measurement and the first.
Since the permeate flux rate increases with increasing TMP, also the transport of foulants will increase. This is reflected by the increase of $\Delta R_{7.5}$ with increasing TMP.
The last column shows that in all cases except one, the filterability decreases when filtering a sample more than once, $\Delta R_{7.5,n}/\Delta R_{7.5,1} > 1$. This probably means that the maintenance procedure and the measurement itself were detrimental for the filterability. If this is true, it might mean that the accuracy is higher than indicated here and cannot be determined in this way.
A last encouraging conclusion is the rather broad range of values for $\Delta R_{7.5}$, which indicates that differences can be measured. The filtration experiments with activated sludge from the Zenon installation yielded a value of $\Delta R_{7.5}$ of $0.71 \cdot 10^{12}$ m^{-1}. The same experiment performed with activated sludge from the Kubota installation resulted in a value of $1.04 \cdot 10^{12}$ m^{-1} and with sludge from the Mitsubishi installation even higher values of about $1.15 \cdot 10^{12}$ m^{-1} and $1.24 \cdot 10^{12}$ m^{-1}. These differences are bigger than the deviations within the results from the repeatability experiments. This is an indication that the filterability may be measured accurately with this method.

5.3.6. Influence of MLSS concentration

The sludge samples were taken from MBR-pilots treating municipal wastewater, resulting in fluctuations in MLSS concentration. Since MLSS is assumed to play a role in membrane fouling, this parameter was measured and compared to the filterability of the respective activated sludge. Figure 5-3 shows the results of

these measurements. For four values of TMP, $\Delta R_{7.5}$ is plotted against MLSS concentration. The range of MLSS concentrations is from 7.5 g/L up to 17 g/L, and $\Delta R_{7.5}$ values vary between 0 and 4 $\cdot 10^{12}$ m^{-1}

There is no general relation between MLSS concentration and $\Delta R_{7.5}$ in these experiments.

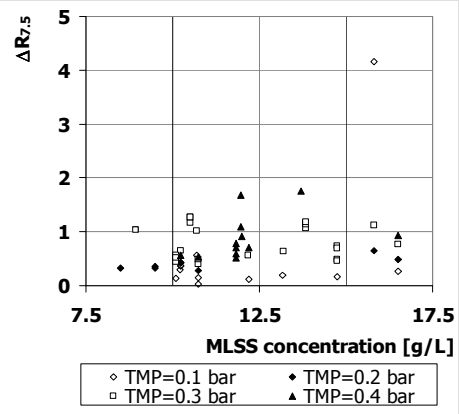


Figure 5-3 *$\Delta R_{7.5}$ against MLSS concentration for different values of TMP*

5.3.7. Sample maintenance

The sludge samples were transported from the MBR pilot location to the laboratory for examination. During transportation, which took 1.5 hours, no aeration was applied. After arrival at the laboratory aeration was started and filtration experiments were carried out. In some experiments the sludge was kept aerated over night, to be filtered again on the day after sampling. In this way measurements were done with sample ages of 0 and 1 day. For each experiment that was done under the same conditions $\Delta R_{7.5}$ was calculated. The values of day zero and day one are divided, and thus the normalised $\Delta R_{7.5,n}$ was obtained.

Results are summarised in Table 5-2. In 3 cases $\Delta R_{7.5}$ at day 1 is about 80% of the value at day zero. One sample however showed the opposite behaviour; here the normalised $\Delta R_{7.5}$ increased with maintenance time.

Table 5-2 Effect of Sludge maintenance on Filterability

System	Sample number #	Sample age [d]	TMP [bar]	$\Delta R_{7.5,n}/\Delta R_{7.5,1}$ [-]	$(dR/dV_{7.5,n})/(dR/dV_{7.5,1})$ [-]
Kubota	1	1	0.37	0.78	0.84
Kubota	1	1	0.80	0.80	0.94
Kubota	2	1	0.10	0.78	1.15
Zenon	3	1	0.35	0.93	1.11
Zenon	4	1	0.30	0.70	0.89
Zenon	5	1	0.10	1.30	1.38
Zenon	5	1	0.20	1.51	1.25
Zenon	5	1	0.30	1.61	1.37

From these results it is clear that merely aerating the activated sludge leads to changes in filtration characteristics.

5.3.8. Results with three values of TMP

During the period of measurements with constant TMP, a series of characterisation experiments was performed with TMP of 0.10 bar, 0.20 bar and 0.30 bar. These experiments were performed with activated sludge from three different installations, one equipped with Zenon membranes, one with Kubota membranes and one with Mitsubishi membranes. Since the aim of these measurements was merely to discover whether any differences could be measured, the results are presented anonymous, the installations are referred to as System A, B and C. Therefore the measurement numbers in the three graphs are not corresponding in the sense that measurement 1 with TMP=0.10 bar is the same sludge as used for measurement 1 with TMP=0.20 bar.

The results are presented in Figure 5-4 and show that variations can be observed. A clear distinction between the different installations cannot be seen, maybe the filterabilities overlap. With increasing TMP also $\Delta R_{7.5}$ increases.

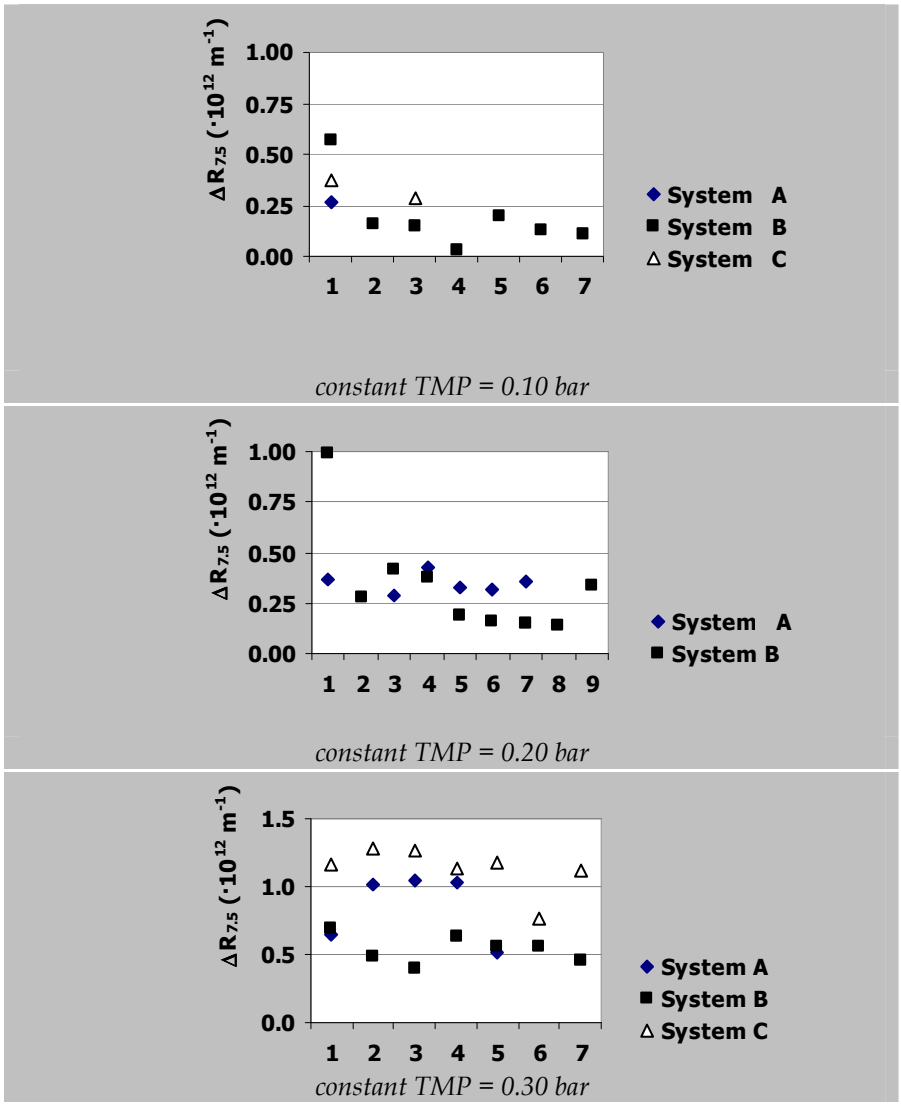


Figure 5-4 *Results from constant TMP experiments performed with three values of TMP and activated sludge from three systems. Numbers indicate respective experiment number with a new batch of sludge.*

5.4. Constant Flux Experiments

With a suction pump at the permeate side of the membrane, experiments can be carried out under constant flux conditions. The induced TMP is monitored and calculated to filtration resistance. The results are also presented as an additional resistance (ΔR_x) against the produced volume per square metre of membrane surface (L/m²).

5.4.1. Standard Conditions and General Results

For the constant flux experiments standard conditions had to be chosen again, to enable comparison of different samples. Based on the experience with sludges originating from different installations and the needed representativity the following values were chosen for flux and cross flow velocity: J=80 l/m²·h, u_{cr}=1 m/s. These values represent a reasonable compromise between ensuring the occurrence of fouling mechanisms that are characteristic for MBR and accelerating these processes to minimise measuring time.

As discussed in Chapter 3, the particles that may be involved in the fouling process will be bigger when the flux is increased. In a real large scale MBR installation the actual local permeate flux will be higher than the averaged set point, since an unknown amount of membrane area may be clogged, thus increasing the flux for the rest of the membrane area. Compare for example to the ´local critical flux´ concept described by Ognier *et al*. (2004).

The length of a characterisation experiment is determined by the limitations of the installation; in this case the accuracy of the permeate pressure gauge. As a result of fouling processes, the permeate pressure had to be decreased in order to extract the constant permeate flux. An experiment therefore lasted until at least 40 l/m² of permeate was produced, or until a TMP of 0.7 bar was reached.

Depending on the activated sludge fouling characteristics typical results with constant flux will look like those represented in Figure 5-5. After a first jump to reach the TMP for the desired flux, a seemingly linear phase is followed by an increasing fouling rate. The whole curve can be fitted to a third order polynome, starting at zero m^{-1}. When the influence of the first jump is left out, sufficient accuracy is kept when fitted to a second order polynome.

As shown in the mentioned figure, considerable differences can be found between different types of sludge and different points in time.

When the curves from Figure 5-5 (constant flux) and Figure 5-1 (constant TMP) are compared, the following differences can be observed. Firstly, the scale differences in the horizontal axes. Permeate production during constant flux experiments was usually much higher than during constant TMP experiments. This is of course a consequence of the mode of operation; during constant flux experiments the volume increases linearly with time, whereas with constant TMP the permeate production rate decreases.
Secondly, it is noteworthy to see that constant flux experiments result in a curve that starts with a gradual resistance increase, (almost constant dR/dV), and ends like this or with an ever increasing fouling rate. Most cases show a visibly concave development.

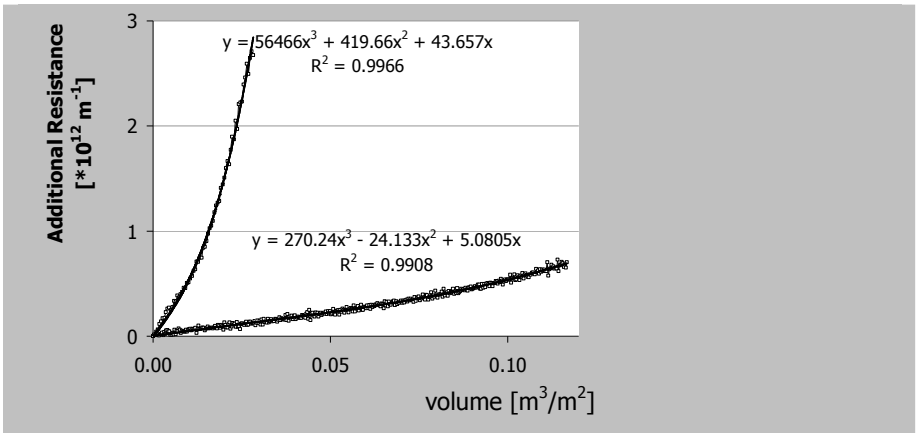


Figure 5-5 *Two examples of results obtained with constant flux experiments*

5.4.2. Characterisation of Filtration Curve

An activated sludge filtration curve, determined under constant flux conditions, has the following characteristics:
A first stepwise resistance increase until the desired flux is achieved (usually in about 15 seconds, in which the rotation speed of the permeate suction pump was

smoothly increased). In some cases this phase would coincide with an initial amount of fouling, causing the first jump of filtration resistance to end somewhat higher than $R_{m,0}$. This phase is followed by an almost linear increase of filtration resistance with produced volume, followed by an increasing fouling rate, indicated by a resistance increase with the square of the produced volume.
Since the best fit is a second order polynome starting at 0, at least two parameters must be used to mathematically characterise a filtration curve. Usually, the first jump in filtration resistance did not occur.

Since the produced permeate volumes with constant flux experiments are much higher than with constant TMP, it seems better to use as reference point a higher volume than 7.5 l/m^2 that was used in those experiments. Under standard conditions, ($J = 80\ l/m^2 \cdot h$ and $u_{cr} = 1\ m/s$) sufficiently accurate and discriminating results can be obtained when curves are compared after 20 l/m^2, i.e. after 15 minutes of filtration. This value (ΔR_{20}) will be used in the further description of the results. Bearing in mind that the resistance of a clean membrane is about $0,4 – 0,5 \cdot 10^{12}\ m^{-1}$, it is easy to estimate the significance of any value.

For the cases depicted in Figure 5-5, assuming a reference value for permeate production of 20 l/m^2, this yields for the steep curve:
$\Delta R(0.020) = 1.49^{*}10^{12}\ m^{-1,}$ and $dR/dV(0.020) = 128 \cdot 10^{12}\ m^{-2}$, and for the bottom one: $\Delta R(0.020) = 0.09^{*}10^{12}\ m^{-1,}$ and $dR/dV(0.020) = 4 \cdot 10^{12}\ m^{-2}$.

From almost all constant flux filtration curves that were measured in this project not two curves crossed each other. The initial fouling rate determined the course of the filtration curve, and it's 'ranking' in terms of filterability. This quite remarkable feature means that in order to compare filterability of different activated sludges, the filtration curves can be characterised by one value, either the mentioned ΔR_{20} or the derivative to produced volume, dR/dV_{20}.

5.4.3. Variations during one day

In order to assess the variations in filterability, a sample was taken from the MBR pilot at three or four times during a day, with an interval of about 1.5 hour. As is shown in Figure 5-6, ΔR_{20} fluctuated considerably during the experimental periods; so it seems to be a rather dynamic parameter. On the other hand differences between measuring days and different MBR pilots are obviously shown.

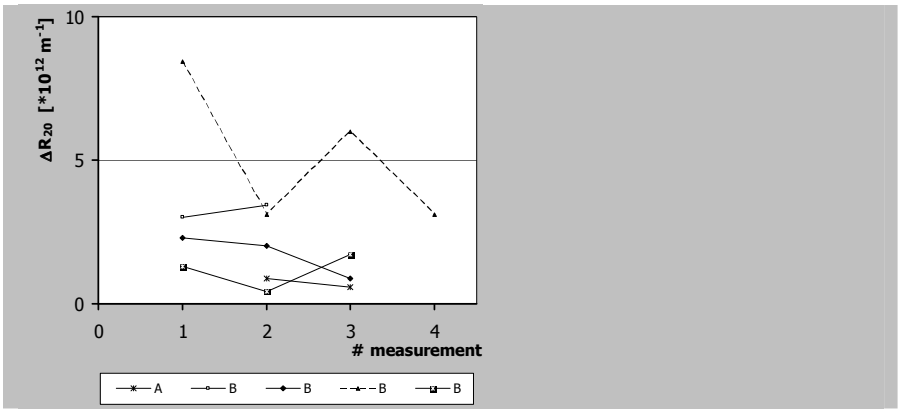


Figure 5-6 *Development of ΔR_{20} during different days; J=80 l/m²h; u_{cr}=1 m/s; A, B signify different MBR pilots*

5.4.4. Influence of circulation pump type

In Chapter 3 the influence of pump type on filterability was concisely discussed. It is known that using a centrifugal pump may lead to floc damage and successively deterioration of filtration behaviour. This was investigated in the following experiments that were carried out two times. These experiments are also important to determine the value of the experiments that were carried out using the peristaltic circulation pump.

An experiment consisted of four characterisation experiments, as follows. First, the activated sludge was filtered under standard constant flux conditions (J=80 $L/m^2 \cdot h$, u_{cr}=1 m/s) applying the peristaltic pump for sludge circulation. Subsequently, the sludge was circulated over the feed vessel with the centrifugal pump for 15 minutes (the time span for an average constant TMP experiment).

Table 5-3 *Experimental procedure and results of experiments with centrifugal and peristaltic circulation pump*

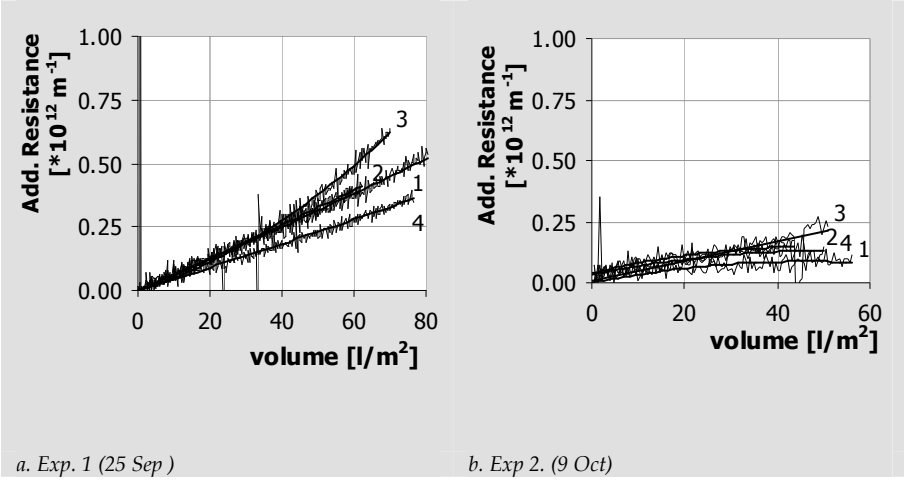
Day	Curve no.	Circulation pump	J	u_{cr}	Exp. 1 (25 Sep) ΔR_{20}	Exp. 1 (25 Sep) ΔTMP_{20}	Exp. 2 (9 Oct) ΔR_{20}	Exp. 2 (9 Oct) ΔTMP_{20}
	#	-	$L/m^2 \cdot h$	m/s	$\cdot 10^{12}\ m^{-1}$	bar	$\cdot 10^{12}\ m^{-1}$	bar
1	1	peristaltic	80	1.0	0.10	0.026	0.06	0.013
15 min of circulation with peristaltic pump								
1	2	peristaltic	80	1.0	0.15	0.036	0.12	0.042
1	3	centrifugal	80	1.0	0.12	0.030	0.08	0.023
2	4	peristaltic	80	1.0	0.10	0.027	0.09	0.020

Next to that, the activated sludge broth was again filtered with the peristaltic pump as circulation pump. As a third filtration experiment, the sludge was filtered, while using the centrifugal pump as circulation pump.
Then the activated sludge was aerated over night and the following day a standard experiment was performed with the peristaltic pump as circulation pump, as summarised in Table 5-3.
The influence of circulating with a peristaltic pump can be evaluated as an increase in ΔR_{20}. In the first experiment the value of ΔR_{20} increased by a factor 1.5 and in the second experiment by a factor 2.

If the filtration curves are observed closely, more information is obtained, see Figure 5-7. This can be seen most clearly in the first experiment, Figure 5-7a. The first two measurements result in filtration curves of the same type, they almost overlap each other. The third one shows a faster resistance increase, which accelerates with produced volume. One night of aeration increased the filterability considerably, ΔR_{20} is lower than during the first filtration.

As can be seen from the filtration curves it was rather difficult to measure the third curve in these experiments (while using the centrifugal pump for circulation). This was partly caused by the difficulties in measuring permeate flux accurately. For this reason also the values of TMP are presented in Table 5-3. TMP measurements are more accurate and show fewer fluctuations and as can be seen, the influence of the circulation with the centrifugal pump is detrimental for the filterability. After one night of 'rest', however, the filterability increases. Apparently, substances being involved in membrane fouling are over night

being adsorbed to the microbial flocs or may have been degraded by the biomass.



a. Exp. 1 (25 Sep) *b. Exp 2. (9 Oct)*

Figure 5-7 *Filtration curves under standard condition measured with different types of circulation pump; for description of the four filtration curves from each experiment, see text.*

It must be remarked that during these experiments two of the few cases were observed of filtration curves crossing each other. The second and third filtration curves were of a really different type than the first and the fourth.

5.4.5. Comparing activated sludge from two installations

Some exploratory investigations were performed to obtain an indication of the differences in filterability between two installations. To this aim activated sludge was sampled from the pilot plant equipped with Zenon membranes and the installations with Kubota membranes. Figure 5-8 presents the values of ΔR_{20} for measurements performed under standard conditions during a period of four months. The different systems are signified as A and B.

The variation in ΔR_{20} is quite high, especially for system B. It must be remarked that some values were not actually measured but are extrapolated values, because the experiment had to be stopped before reaching 20 L/m² of permeate production. Each data point in Figure 5-8 represents a filtration curve, made

with a fresh batch of activated sludge. Thus, the interval between two measurements is at least 2 hours.
There seems to be a gradual decrease in filterability of system B (ΔR_{20} increases) in the second half of April, whereas the second half of May shows a gradual increase in filterability (ΔR_{20} decreases).

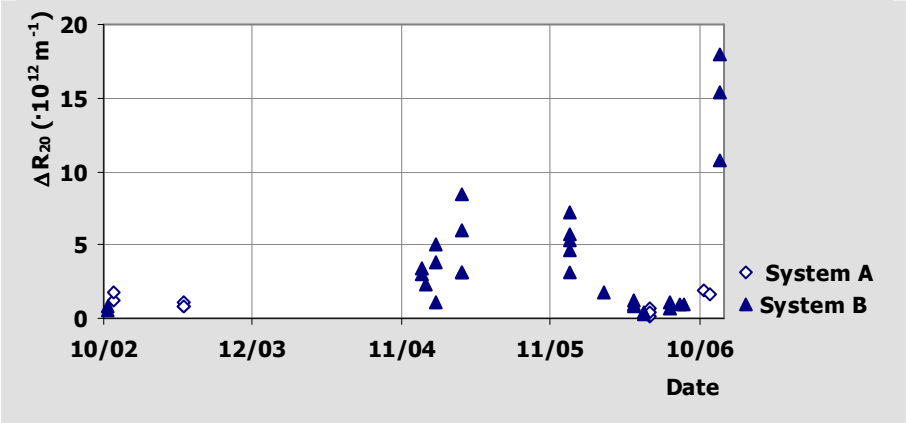
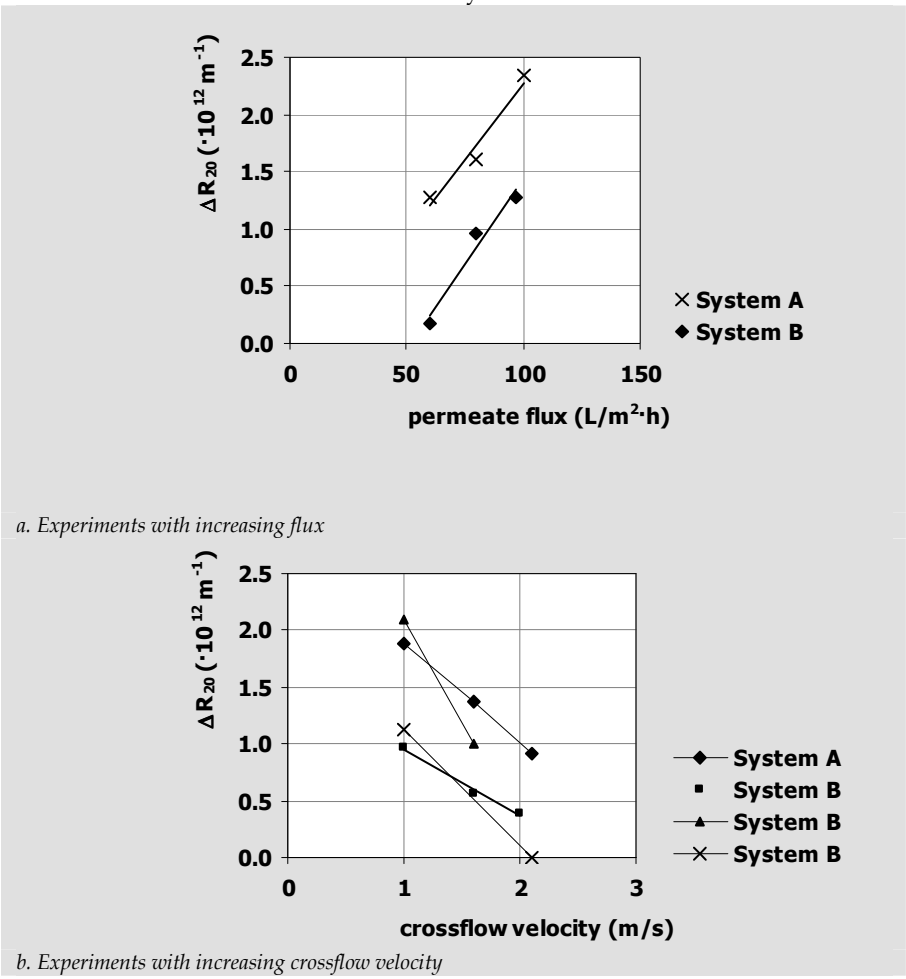


Figure 5-8 *Values of ΔR_{20} measured under standard conditions during a period of four months with activated sludge sampled from two MBR pilot plants.*

The activated sludge from system A seems to have a better filterability. Although the amount of data in this graph is smaller for this system, compared to system B, the rest of the measurements confirm this trend (not presented here).

Both sludges were also filtered under varying conditions in order to assess the sensitivity of the fouling behaviour for these changes. In one set of experiments the crossflow velocity was increased, while the flux was constant. The results are presented in Figure 5-9 as ΔR_{20} under different conditions. Increasing the flux leads to an almost linear increase in ΔR_{20}. The rate at which this happens is almost equal for the two types of sludge that were tested.
Increasing crossflow velocity shows similar behaviour. There seems to be a critical crossflow value for each activated sludge sample, for which ΔR_{20} will be zero. The same could possibly be said for flux; if flux is decreased far enough, ΔR_{20} will become zero. This approach to filtration characterisation is not further

followed in this thesis, since it requires at least three filtration runs to determine a critical flux or a critical crossflow velocity.



a. Experiments with increasing flux

b. Experiments with increasing crossflow velocity

Figure 5-9 Sensitivity of filterability for changes in operational conditions

5.5. Fouling Reversibility

All experiments ended with a standard cleaning procedure, consisting of:

- a forward flush with permeate or demineralised water,
- a backflush with permeate and

- a chemical cleaning; a 500 ppm NaOCl solution, in which the membrane was soaked for 30 minutes.

In between these cleaning steps the membrane resistance to clean water filtration was measured, indicating the efficiency of the respective procedure. Thus, five values of filtration resistance are of importance:

- $R_{m,0}$ Membrane resistance at the start of the experiment
- R_{as} Filtration resistance at the end of activated sludge filtration
- R_{ff} Filtration resistance after forward flushing
- R_{bf} Filtration resistance after backflushing
- R_{cc} Filtration resistance after standard chemical cleaning

With these five values, five filtration resistance intervals can be calculated, summarised in Table 5-4.

Table 5-4 *Five resistance intervals calculated from filtration characterisation curve*

Resistance interval	Formula	Comment
ΔR_{as}	$R_{as} - R_{m,0}$	Total resistance increase caused by activated sludge filtration
ΔR_{ff}	$R_{as} - R_{ff}$	Resistance *de*crease, caused by forward flushing with u_{cr}=4 m/s
ΔR_{bf}	$R_{ff} - R_{bf}$	Resistance *de*crease, caused by back flushing with TMP=0.75 bar
ΔR_{cc}	$R_{bf} - R_{cc}$	Resistance *de*crease caused by standard chemical cleaning, 30 min. with 500 ppm NaOCl
ΔR_{ir}	$R_{cc} - R_{m,0}$	Remaining resistance increase after standard cleaning procedure, or, the irreversible fouling resistance

Each of these resistance intervals, except ΔR_{as}, gives information about the reversibility of the fouling.

When interpreting the values calculated according to Table 5-4, the following consideration has to be taken into account. The duration of each experiment was dependent on the filterability of the activated sludge under consideration. This means that the cleaning procedure was performed at different stages in the fouling process. Thus, from a scientific point of view, values cannot be compared since the circumstances under which they are measured are not the same. On the other hand, the circumstances are implicitly taken into account by the fact that filtration time depends on filterability. A well filterable activated sludge induces a small value of ΔR_{as}, with equally low values for the other resistance intervals.

All the same, conclusions should be drawn with care and for further application a thorough standardisation should take place.
A first step towards standardisation can be taken by dividing the resistance intervals by the amount of permeate that was produced during the experiment, yielding values with unity $m^{-1}/(L/m^2) = m/L$, or m^{-2}.

Although this type of information is not further used in this thesis, some of the results are presented here to illustrate the possibilities (and difficulties) of this method. Figure 5-10 presents the relative contribution of the values for ΔR_{ff}, ΔR_{bf}, and ΔR_{ir} to the total amount of fouling that was built up during the experiment. Usually, ΔR_{bf} forms the major part, followed by ΔR_{bf} and ΔR_{ir} contributes the least to the fouling process.

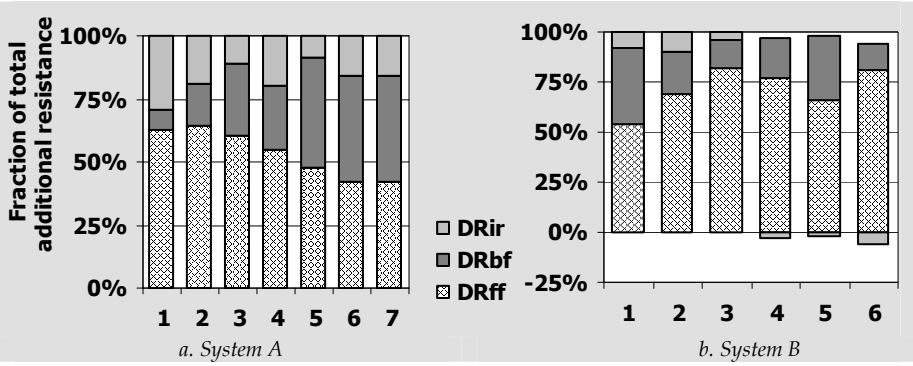


Figure 5-10 *Results from fouling reversibility calculations for measurements under standard conditions (DRir in this figure equals $\Delta R_{cc} + \Delta R_{ir}$)*

Figure 5-10 also shows that sometimes the clean water resistance after the chemical cleaning was lower than at the start of the experiment (measurements 4, 5 and 6). This leads to a negative contribution of ΔR_{ir} to the overall filtration resistance.

5.6. Conclusions

Filtration characterisation with the method proposed in Chapter 4 leads to interesting results. Constant flux experiments lead to useful data and the

filtration curve can generally be characterised by the value of additional membrane resistance (i.e. actual filtration resistance minus the membrane resistance).
Standard conditions for a constant flux experiment were identified as:

- permeate flux of 80 L/m^2·h
- crossflow velocity of 1 m/s

With the described method differences in filterability between installations could be quantified as well as variations in time. The next chapter will describe experiments in which these results are verified and the first steps are taken to identify the factors that determine the filterability of any given activated sludge broth.

References Chapter 5

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The process of living seems to consist in coming to realize truths so ancient and simple that, if stated, they sound like barren platitudes. They cannot sound otherwise to those who have not had the relevant experience: that is why there is no real teaching of such truths possible and every generation starts from scratch…
C.S. Lewis, Letters

6
MANIPULATING THE FILTERABILITY OF ACTIVATED SLUDGE

6.1. Introduction

Knowing that the activated sludge filterability in an MBR installation may change, for example during a season or on a shorter time scale of days or even hours, the constant operation of an MBR system requires knowledge of the parameters that determine filterability of activated sludge. The filtration characterisation method described in the former chapters can be utilised well to investigate the sensitivity of activated sludge filterability to different conditions. The conditions can be varying at the level of hydrodynamics or at the level of microbiological parameters. Some experiments with varying hydrodynamic conditions were described in the former chapter. This chapter describes the experiments in which the filterability of activated sludge was manipulated by means of substrate addition, in paragraph 6.3.

Secondly, the aim of this chapter is to identify whether the changes in filterability as measured with the filtration characterisation method can be correlated to water quality parameters. The idea behind this is that each type of fouling is caused by *some* constituent(s) in the feed flow. In order to optimise filtration performance it is necessary to determine these constituents, find out the concentrations in which they occur and the circumstances that cause their presence. From the literature review (see chapter 3.4. *Identifying Foulants and Fouling Situations in MBR*) extracellular polymeric substances (EPS) in the water phase of activated sludge turn out to be among the group of substances which most likely are involved in membrane fouling in MBR. Their microbial origin,

physical and chemical properties seem to confirm this hypothesis. Firstly, a measurement method to determine EPS in activated sludge was identified. With this method the occurrence of EPS in MBR was investigated, as described in paragraph 6.2.

6.2. Occurrence of EPS in an MBR treating municipal wastewater

6.2.1. EPS measurement method

To determine protein concentrations, the method proposed by Lowry *et al.*(1951), modified by Frølund *et al.*(1996) and improved by Brahner (2000) was used. This method is based on a two-step colour reaction in which the peptide of the protein reacts with a copper solution. Subsequently the copper-peptide bonding is reduced by the Folin-Ciocalteaus reagens. The colouring is measured spectrophotometrically as extinction at 750 nm.

The polysaccharides are measured according to the method described by Dubois *et al.*(1956) and improved by Brahner (2000). The method is based on the acid hydrolisation of polysaccharides at high temperature. The monosaccharides subsequently show a colour reaction with phenol which is measured as extinction at 490 nm. For a further discussion on measuring methods, see Rosenberger (2000) and Te Poele (2005).

6.2.2. Size distribution and fouling processes

The particle size distribution of the boundary layer in membrane filtration of activated sludge depends on many factors, as pointed out in Chapter 3. Tardieu (1997) calculated the hydrodynamic circumstances in the filtration installations applied in his thesis. By applying the expressions for Brownian diffusion, lateral migration and shear induced diffusion, the length averaged permeate flux (along a membrane with length L) as function of particle size can be calculated. The formulas are as follows (Belfort *et al*, 1994):

Brownian diffusion:
$$0.114 \cdot \left(\frac{\gamma_0 \cdot \mathrm{k}^2 \cdot \mathrm{T}^2}{\eta_0^2 \cdot d^2 \cdot L} \right)^{1/3} \cdot \ln\left(\frac{c_w}{c_b} \right) \qquad \textbf{(6-1)}$$

Lateral migration: $$\frac{0.036 \cdot \rho_0 \cdot d_p^3 \cdot \gamma_0^2}{\eta_0} \quad \textbf{(6-2)}$$

Shear induced diffusion: $$0.078 \cdot \left(\frac{d_p^4 \cdot \gamma_0}{L} \right)^{1/3} \cdot \ln\left(\frac{c_w}{c_b} \right) \quad \textbf{(6-3)}$$

where

γ_0= wall shear rate		s^{-1}
k = Stefan Boltzmann constant		J/K
T = absolute temperature		K
D = particle diameter		m
L = length of membrane module		m

c_w = suspension concentration at the membrane wall
c_b = suspension concentration in the bulk

According to Tardieu (1997) the length averaged flux can also be regarded as a back transport velocity. As long as the back transport velocity is larger than the permeate flux 'velocity', a particle will not deposit on the membrane. In this way the magnitudes of the different back transport mechanisms can be compared to the convective flow, caused by the permeate flux, as is presented in Figure 6-1.

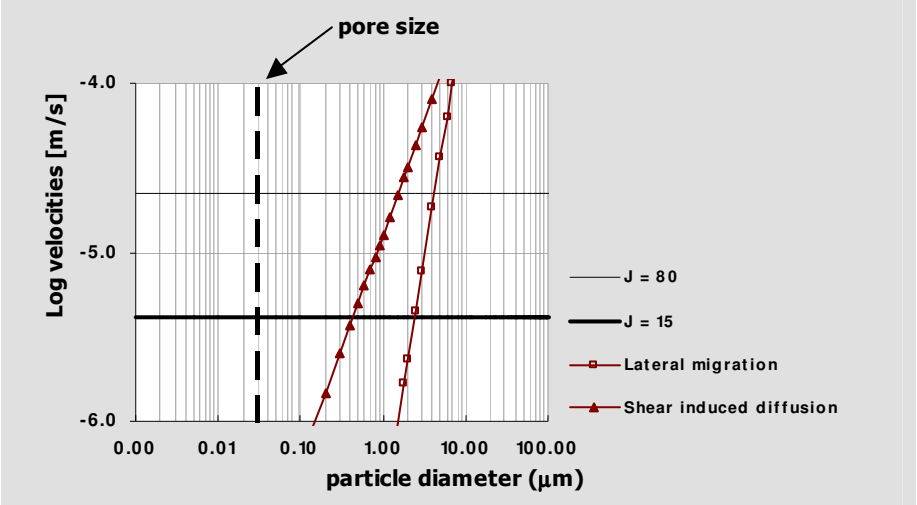


Figure 6-1 *Magnitude of three backtransport mechanisms, calculated according to Belfort (1994) and two values of permeate flux; crossflow velocity=1 m/s; wall shear rate=4000 s^{-1}; c_w/c_b=20; L=0.95 m; T=293 K.*

According to Metcalf&Eddy (2003) the average floc size of activated sludge is 50 – 200 µm. Figure 6-1 illustrates that the back transport velocity for particles of this size is much higher than the permeation velocity. Although the assumptions for the calculations of the back transport model are in more than one respect not valid for activated sludge (spherical particles, non-interacting) and more assumptions have to be made in order to make the calculations (wall shear rate, sludge viscosity) Figure 6-1 provides a good indication of the relative magnitudes of the velocities during filtration.

Together with the results from the literature Review, pointing at the importance of the liquid phase of activated sludge for its filterability, it was decided not to analyse the activated sludge flocs, but only substances in the sub-micron range.

6.2.3. Separation of floc phase and water phase

For the separation of the water phase from the floc phase, several methods are available, among which the following were tested for this study, described in Evenblij and van der Graaf (2005):

- centrifugation
- filtration

- gravitary sedimentation

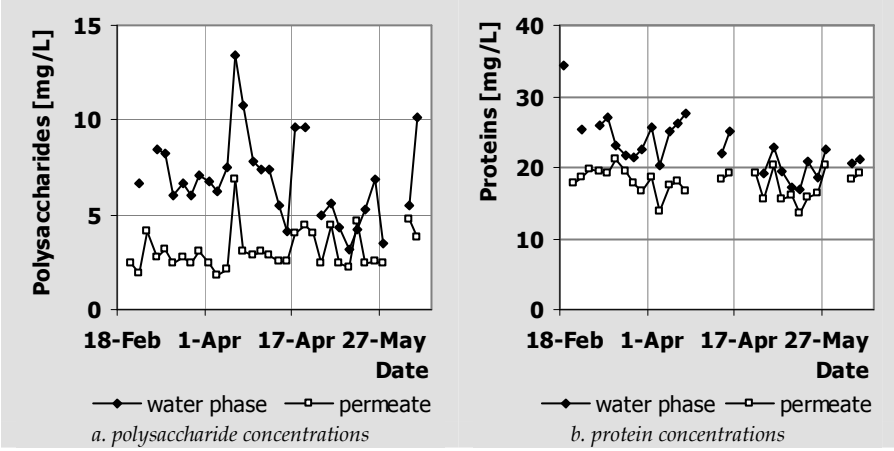
Comparison of these methods led to the conclusion that for a proper separation of particles smaller than about 2 µm, filtration over a washed paper filter leads to useful results. In this study Schleicher&Schull 589/2 paper filters were used. According to the specifications the pore size of this filter is non-definite, in the range of 7-12 µm. Since this type of separation is carried out as dead-end filtration special care has to be taken to minimise capturing particles in the cake layer, by filtering only small quantities of sludge.

6.2.4. EPS in water phase of an MBR

Over a period of three months the water phase of the activated sludge taken from an MBR treating municipal wastewater was analysed for EPS. Each time a sample was taken from the recirculation flow from the membrane tank to the aeration tank. The activated sludge sample was filtered over a paper filter and the obtained filtrate was analysed. Each time also the permeate of the MBR was sampled and analysed. Both proteins and polysaccharides were measured as described in 6.2.1.

The results of the measurements are presented in Figure 6-2, both for polysaccharides and proteins. The measured protein concentration in the water phase varied from 16 to 35 mg/L and in the permeate from 13 to 21 mg/L.

The measured polysaccharide concentration in the water phase varied from 3 to 14 mg/L and in the permeate from 2 to 7 mg/L.



a. polysaccharide concentrations *b. protein concentrations*

Figure 6-2 *EPS concentrations in water phase and permeate of an MBR installation treating municipal wastewater*

The average ratio proteins/polysaccharides in the water phase was 3.0 and in the permeate 6.3.

The retention of the UF membrane in the MBR (30 nm pore size) is for the proteins some 15% and for the polysaccharides about 40%.

Table 6-1 *Average values for EPS concentrations in Zenon pilot MBR at Beverwijk (2002)*

		Water phase	Permeate	colloidal fraction*
Polysaccharides (average)	mg/L	7.3	3.1	4.2
Proteins (average)	mg/L	22.9	17.8	5.1
Ratio proteins/polysaccharides	-	3.1	5.7	1.2

*calculated as: (water phase) – (permeate)

The absolute retention is in the same order for proteins and polysaccharides, as shown in Figure 6-3. For each measurement the difference in concentration between the water phase and the permeate ($c_{water\ phase} - c_{permeate}$) is calculated. These values ($\Delta_{proteins}$ and $\Delta_{polysaccharides}$) represent the concentrations that are retained in the system by the membrane. This fraction can not be signified according to size distribution, since it is unknown which part of the substances smaller than the pore size do actually pass the membrane. In this sense, the term 'retention' is

strictly spoken also incorrect. For convenience purposes and failing a better term, these substances are furthermore indicated by the term 'colloidal fraction' of the EPS in the water phase.

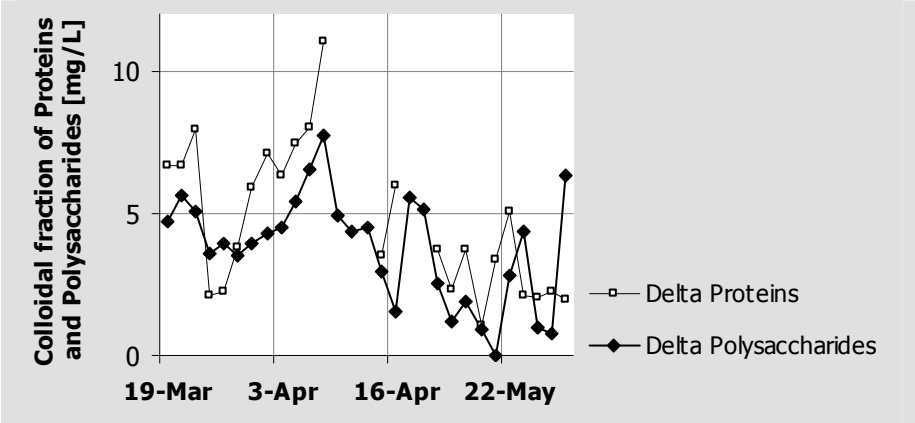
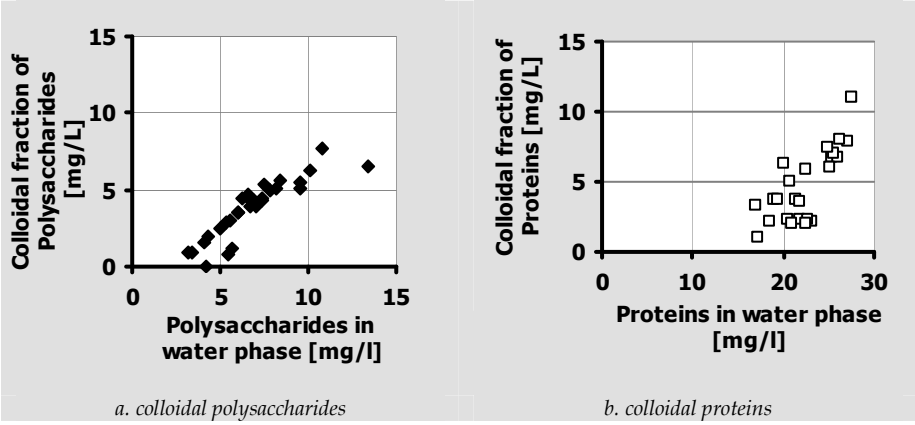


Figure 6-3 *Colloidal fraction of proteins and polysaccharides in the water phase versus time*



a. colloidal polysaccharides *b. colloidal proteins*

Figure 6-4 *Colloidal fraction of EPS versus concentration in the water phase*

Apart form a few exceptions, both $\Delta_{proteins}$ and $\Delta_{polysaccharides}$ follow the same trend. Assuming that all substances that pass the paper filter are small enough to enter the boundary layer, the values of $\Delta_{proteins}$ and $\Delta_{polysaccharides}$ represent the concentrations of EPS available for membrane fouling.

The colloidal fraction of EPS increases linearly with the EPS concentration in the water phase. When the concentration in the water phase is low enough, all present EPS is in colloidal form, as illustrated by Figure 6-4.

6.3. Influence of substrate addition

Activated sludge treatment is based on the ability of biomass to convert and mineralise pollutants (substrate) present in the wastewater. The biomass adapts itself to the wastewater that it is treating. Depending on how the treatment installation is operated, i.e. high or low loaded, the biomass will be in a certain physiological state. The physiological state of the biomass may influence its filterability to a high extent.

Chang and co-workers describe experiments in which activated sludge in different physiological states was tested with respect to its filterability [Chang and Lee, 1998]. The filtration experiments were performed in stirred cell tests, with a quite high TMP and up to high concentrations. The different physiological states of the biomass were determined by different combinations of SRT and F/M ratio.

Furthermore, the activated sludge was fractionated in three fractions: bulk, cells and EPS. Their conclusion was that higher sludge ages resulted in less fouling, and the major qualitative parameter, responsible for fouling was EPS. It must be remarked here that for the determination of the influence of EPS on filterability, the total amount of EPS, present in the microbial flocs, was extracted and separately analysed.

6.3.1. Reference measurements: no substrate

In order to have a reference measurement the first experiments with substrate addition were all paralleled by an experiment in which the activated sludge was only aerated. One or two times a day a filtration characterisation measurement was performed. Results from one 'reference batch' are presented in Figure 6-5.

The left hand graph shows the filtration curves with a 'standard' y-axis. To demonstrate the small differences, the right hand graph shows a magnification of the y-axis. The filterability did not change during a period of four days.

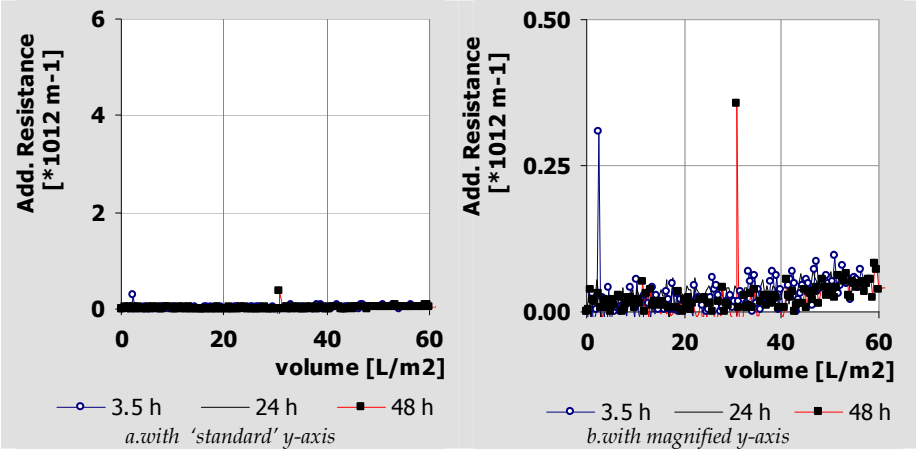


Figure 6-5 *Filtration curves of batches of activated sludge which were aerated for two days*

Because the changes in filterability of the reference batch were so small, they will not always be presented.

6.3.2. Substrate

To investigate the extent to which the filterability of activated sludge can be influenced by substrate addition several experiments were carried out. To this aim activated sludge was sampled at an MBR pilot installation and transported to the laboratory of Sanitary Engineering in Delft. Subsequently, the activated sludge was aerated and after varying intervals of time substrate was added to the biomass.

The substrate used in this study was a standard substrate, used for biomass maintenance during practical courses. It consisted of several components, listed in Table 6-2.

Table 6-2 *Constituents of Standard Substrate solution, to be added per 250 g of suspended solids*

- 100 g $CH_3COONa \cdot 3H_2O$
- 50 ml of solution containing 200 g/L NH_4Cl and 25 g/L $CaCl_2 \cdot 2H_2O$
- 50 ml of solution containing 1 g/L $FeCl_3 \cdot 6H_2O$
- 50 ml of solution containing 22.5 g/L $MnSO_4 \cdot H_2O$ and 40 g/L $MgSO_4 \cdot 7H_2O$

- 50 ml of solution containing 20 g/L KH_2PO_4 and 25 g/L $Na_2HPO_4 \cdot 2H_2O$

By applying a common rule for biomass maintenance, the resulting biomass loading rate for different substances is summarised in Table 6-3. These values are based on the experience with the former experiments and assure a certain amount of nutrients per gram MLSS. This amount of substrate is further called: the Standard Amount. Because the MLSS concentration in MBR is about three times higher than in conventional activated sludge, also the amounts of substrate for a sample of the same volume are about three times higher.

Table 6-3 F/M ratios after adding one time Standard Amount of substrate

Substrate	Food/Mass ratio [mg substrate /gr MLSS]
COD	157
Carbon	70.6
Nitrogen	10.5
Phosphorus	1.8

6.3.3. Dilution Experiments

During pilot experiments with MBR treating municipal wastewater discharging from a mixed sewer system, problems may occur after a long dry weather period followed by a heavy rainstorm event. 'Problems' in this sense mean a decrease in filterability, leading to a reduction of membrane permeability.

The mechanisms that cause this type of system malfunctioning were supposed to be related to changes in the water phase of the activated sludge. Probably due to changes in conductivity (salt content) and osmotic pressure, or otherwise, the quality of the biomass could deteriorate, reflected as a decrease in filterability. This assumption was partially tested under lab conditions.

Activated sludge was sampled at the Zenon MBR pilot installation at Beverwijk wwtp and brought to the laboratory. Here the activated sludge was divided over two batches and both were simply diluted with the same volume of tap water. One of the vessels was provided with the Standard Amount of substrate, based on the new MLSS concentration.

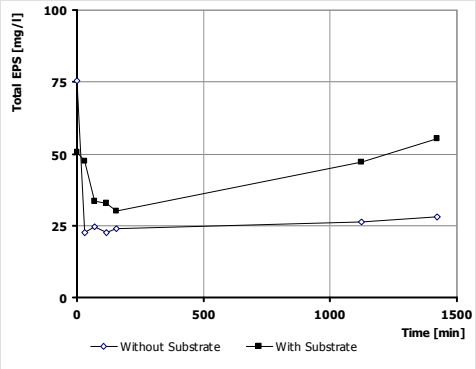


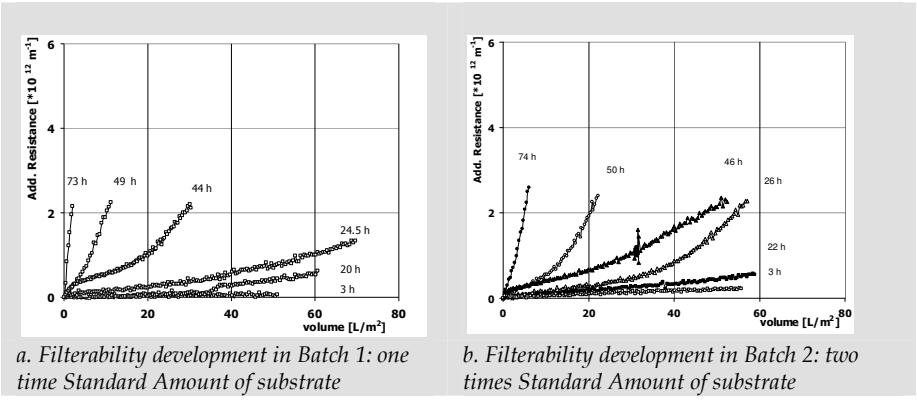
Figure 6-6 *Development of EPS concentration in water phase after dilution, with and without substrate addition*

The batch with substrate addition showed an increase of about 1 gram in MLSS concentration in one day, whereas the pH also increased from 7.7 to 8.7. EPS concentrations in the batch without substrate remained almost constant, as well as the pH (7.9 to 8.1). In the batch with substrate, polysaccharides first decreased from 9 down to 6 mg/l and subsequently increased during 24 hours up to 15 mg/l. The same development was observed for proteins. The sum of proteins and polysaccharides is presented as an indication of total EPS in Figure 6-6.
Interestingly the EPS concentration almost did not change in the batch without substrate addition. Compared to the original concentration, only a very small increase was observed. It was expected that the water phase would be in a kind of equilibrium with the biomass, resulting in a release of EPS to the water phase after dilution.
When dilution is accompanied by substrate addition, the EPS concentrations start to increase during a day. This phenomenon is further investigated in the next paragraphs.

6.3.4. Substrate Experiments: one and two times Standard Amount

To test the reaction of two types of activated sludge in terms of filterability, samples were taken from two MBR pilots, treating the same influent. Figure 6-7 shows the filtration curves of experiments carried out with activated sludge

from the Zenon pilot. *Figure 6-7a* shows the reaction after adding one time Standard Amount of substrate. The time between sampling and substrate addition was 2 hours. In a second batch the same experiment was carried out, but with two times Standard Amount of substrate.



a. Filterability development in Batch 1: one time Standard Amount of substrate

b. Filterability development in Batch 2: two times Standard Amount of substrate

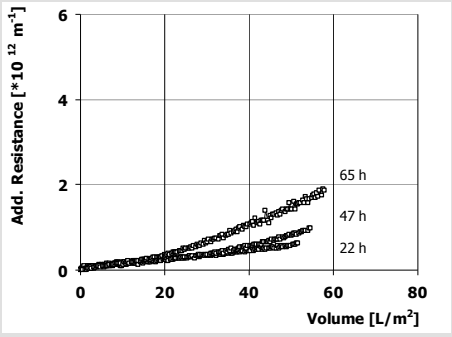
Figure 6-7 *Filterability development during two experiments with and two times Standard Amount of substrate added; sludge sampled from Zenon MBR pilot. Time indication means: hours since sampling from the MBR installation*

The two experiments resulted in a comparable decrease in filterability. However, the second experiment showed a slower filterability decrease but with the same trends. The ultimate filterability after 50 hours in the second case is comparable to the filterability after 50 hours in the first case.

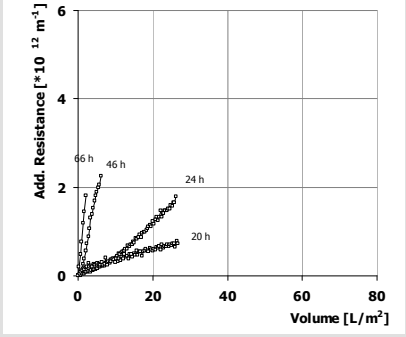
Remarkable is the filtration curve after 44 hours, *Figure 6-7a*. The filtration curve shows an initial jump until a produced volume of some 5 L/m^2, followed by a decreasing fouling rate for about 5 L/m^2, which is succeeded by a continuously increasing fouling rate.

The initial jump may be ascribed to adsorption phenomena, which are usually not encountered in filtration characterisation of MBR activated sludge. This hints at the release to the water phase of adsorptive substances caused by e.g. disruption of microbial flocs, or active excretion of these substances by the biomass. This effect can also be observed in *Figure 6-7b* at some 50 hours after substrate addition.

Figure 6-8 shows the results of the same experiments carried out with activated sludge taken from a MBR pilot equipped with Toray membranes. Both samples were taken at the same day but, different from the former experiment, the substrate was added 30 hours after sampling.



a. Filterability development after addition of one time Standard Amount of substrate



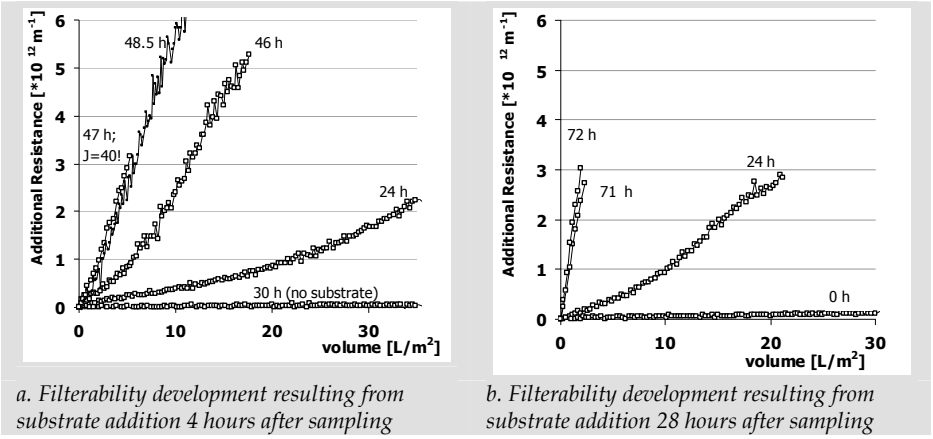
b. Filterability development after addition of two times Standard Amount of substrate

Figure 6-8 *Filterability development during two experiments with one and two times Standard Amount of substrate added; sludge sampled from Toray MBR pilot*

The effect in the first case (Figure 6-8a) is much smaller than in Figure 6-7a. The effect of the second experiment is much bigger than that of Figure 6-7b.

6.3.5. Filterability development after adding three times the Standard Amount of substrate

The same type of experiments was performed with increased substrate concentrations. The amount of added substrate was three times the Standard Amount. In this case the time point of substrate addition was varied: 4 hours after sampling (*Figure 6-9a*) and 28 hours after sampling (*Figure 6-9b*). In this case activated sludge was sampled at two different days from the same installation.
Results are similar to those of the first experiment.
As an interesting feature of these experiments the last three filtration curves of the experiment represented in *Figure 6-9a* must be considered in more detail. After 46 hours the fouling rate was as shown in the graph. In order to obtain a longer filtration period, a second filtration curve was measured, however, with a lower flux. Instead of the standard 80 L/m²h, this measurement was performed with a flux of 40 L/m²h (depicted as '47 h; J=40!').



a. Filterability development resulting from substrate addition 4 hours after sampling

b. Filterability development resulting from substrate addition 28 hours after sampling

Figure 6-9 Filterability development during two experiments with three times Standard Amount of substrate added (activated sludge taken from a Zenon-pilot)

As can be seen, the filterability showed the same or even worse behaviour. This means that the fouling rate in this case is totally dependent on produced volume, and not on operating conditions, since the filtration resistances are presented against produced volume.

The third filtration experiment of this day (48.5 hours after sampling) showed filtration behaviour comparable to the former experiment. Compared to the one after 46 hours, this indicates an ongoing deterioration of the filterability of the activated sludge.

6.4. Influence of Salts

6.4.1. Only salt addition

Because the standard solution contained a high amount of salts, the filtration deterioration might be ascribed to osmotic stress imposed on the biomass, or to ion exchange with the floc-sustaining matrix, causing the microbial flocs to disrupt. To exclude this influence, several experiments were performed 1. with only salt addition, (described in this paragraph) and 2. with very low salt addition and also acetate addition (described in 6.4.2).

Figure 6-10 shows the results of two experiments in which only salts were added to the biomass. The amount was calculated as two times the Standard Amount.

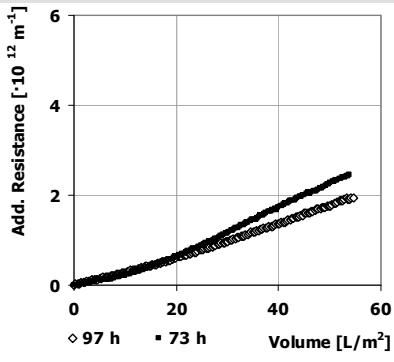
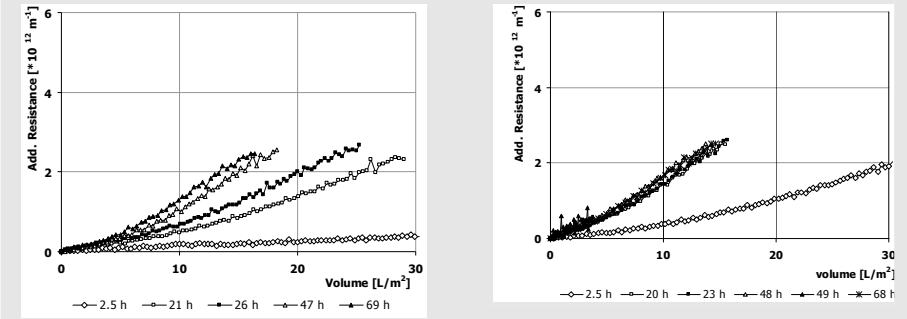


Figure 6-10 Filterability 3 days after adding two times Standard Amount of salts, without carbon source

Only one filtration curve per experiment is shown, measured 72 hours after salt addition. The influence of the salts is considerable: after 3 days the filterability of the activated sludge has become quite bad in both cases, compared to the filtration curves after two days merely aerating the activated sludge (as presented in Figure 6-5).

6.4.2. Low Salt addition

During the experiments described in 6.3 the activated sludge responded enormously to a substrate addition, which may be ascribed to the fact that it is an 'unknown' substrate. It can be imagined that the reaction would not be that extreme if a known substrate would be given in high quantities. This is confirmed by the following experiment. Activated sludge was sampled from two MBRs, that were operated differently. One of the installations was equipped with acetol addition for phosphorus removal, whereas the other was not. Both sludges were fed with a low concentration of salts and one time the Standard Amount of acetate. Filtration curves that were made with both of these sludges are presented in Figure 6-11.



a. Installation A *b. Installation B*

Figure 6-11 *Effect of low salt addition to activated sludge from two different pilot plants*

Activated sludge from the installation with acetol addition (Installation B) showed a relatively bad filterability already immediately after sampling. It decreased in the course of one day but afterwards remained constant for a few days. Although the absolute filterability of this sludge was quite low, it did not deteriorate further after day 1.

In the other case (Installation A) filterability decreased continuously during three days in response to the substrate gift. The ultimate filterabilty of both samples is more or less the same. The fouling kinetics of the sludge from Installation A is higher than that of Installation B.

6.5. Addition of Influent

The normal substrate for the MBRs that were used to sample activated sludge is the influent that is treated. To create a higher COD load to the sampled activated sludge batch with normal influent, the influent would have to be either concentrated or huge amounts of influent would have to be added. The latter is not well possible in the batch experiments, since by adding substrate the MLSS concentration is decreased, which is undesirable.

To investigate the influence of substrate containing the 'known' influent, some experiments were performed.

6.5.1. Influent plus 150 mg COD/g MLSS

Firstly, several experiments were carried out in which influent was added in different quantities. Two batches of activated sludge were provided with influent and substrate. The time points of sampling and substrate addition are summarised in Table 6-4.

Figure 6-12 represents the changes in filtration properties of the activated sludge for both batches.

Table 6-4 *Different steps in experiments with influent and C-source addition*

Time	Action
0:00	Batch 1: Addition of 1.8 L of Influent (MLSS goes down some 7%) batch 2: Addition of 5.4 L of influent (MLSS goes down some 20%)
3:22	Filtration characterisation
20:26	Filtration characterisation
45:25	Batch 1 and 2: Addition of 1.8 L influent and 150 mg COD/gr MLSS
49:41	Filtration characterisation
74:59	Filtration characterisation
93:59	Filtration characterisation

Only influent addition does not lead to any changes in filterability, on the contrary, filterability improves during the first 45 hours in both cases. If influent addition is accompanied by an additional C-source, filterability starts to decrease as will be described in the next paragraph.

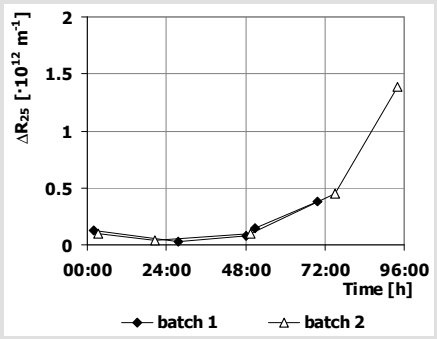


Figure 6-12 *Filterability development after addition of influent and substrate*

6.5.2. Influent plus 50 mg COD/gr MLSS

An experiment in which activated sludge was provided with 1 L of influent and 50 mg COD/gr MLSS resulted in no further changes in filterability. After one day the filterability had increased, indicated by a ΔR_{25} of $0.03 \cdot 10^{12}$ m^{-1}, which was constant for two days (not presented here).

6.6. EPS concentrations during substrate experiments

Almost all the experiments described in this chapter were accompanied by measurement of EPS concentration in the water phase of the activated sludge sample. Generally spoken, the activated sludge showed increasing EPS concentrations in the water phase after adding acetate.

6.6.1. EPS concentration without addition of substrate

The reference batches were also sampled for EPS measurements. Both proteins and polysaccharides were measured. The concentrations did not fluctuate very much as illustrated by Figure 6-13 and Figure 6-14.

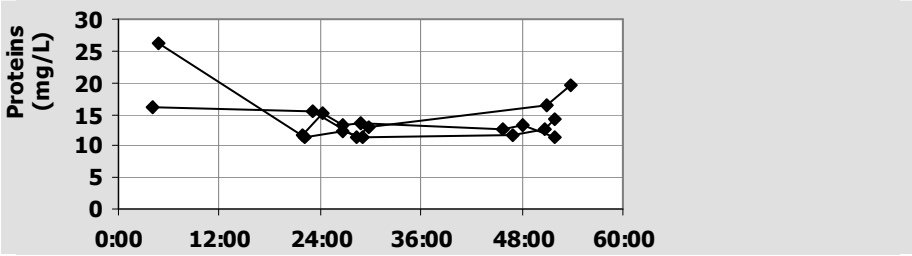


Figure 6-13 Protein concentrations in reference batches during three experiments

The polysaccharide concentrations fluctuated more than the protein concentrations. This is ascribed to sample conservation and a sampling method that was not optimal during these experiments.

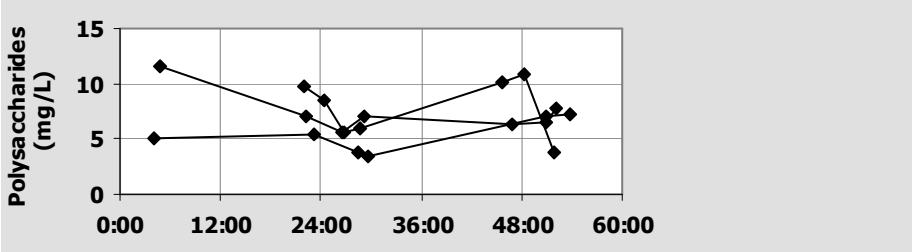


Figure 6-14 *Polysaccharide concentrations in reference batches during three experiments*

6.6.2. EPS concentrations after substrate addition

The EPS measurement that were carried out during the experiments described in 6.3.4 *Substrate Experiments: one and two times Standard Amount* (Figure 6-7 and Figure 6-8) are presented in Figure 6-15. The protein measurements are not available because of problems with the analyses

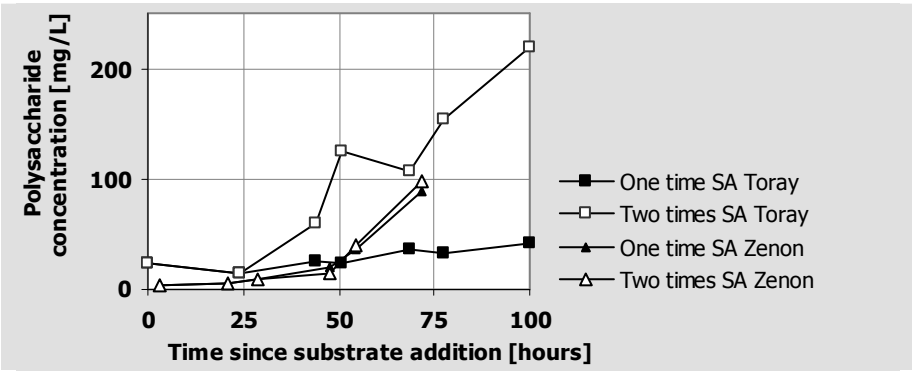


Figure 6-15 *Development of EPS concentrations in the water phase of activated sludge during the experiments from ch. 6.3.4 (SA Standard Amount of substrate; Toray, Zenon MBR pilot plants)*

After the addition of substrate the EPS concentrations in the water phase of the activated sludge increased very much, compared to the basic level measured in 6.2. It is interesting to see that the Toray sludge reacted different from the Zenon sludge to the substrate gift. A substrate gift of one time the Standard Amount resulted in a relatively small and gradual increase of polysaccharides. When the substrate gift was doubled, the polysaccharide concentration increased much faster and to higher ultimate values.

The Zenon sludge did not show much difference between one and two times the Standard Amount of substrate. The same differences were also measured with the filtration curves (see 6.3.4).

6.7. Conclusions

The filterability of activated sludge can be manipulated and the occurring changes can be measured with the developed filtration characterisation method. Furthermore, a first step is made to identify substances involved in the process of membrane fouling.

The addition of a standard substrate in high concentrations has a detrimental effect of filterability. Within two days a standard filtration experiment cannot be performed anymore because the membrane is completely fouled within 2 minutes.

The decrease in filterability is paralleled by an increase of the amount of EPS in the water phase. The EPS concentration increases to values up to 20 times more than at the start of the experiment.

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Time after time I attempted to get away from the torrent of isolated, particular facts: but anything tending to opinion, or discussion, to fancy, to ideas, even to putting some of his infernal facts together and making something out of them – anything like that was received in blank silence.
C.S. Lewis, Letters

7
APPLICATION AT THREE MBR PILOT PLANTS

The filtration characterisation method was tested at three MBR pilot sites in the Netherlands. Each time the installation was located at the respective treatment plant and during 6 to 8 days activated sludge was sampled and investigated.
The relation with the operational performance of the pilot installations and the filtration characterisation will be discussed in Chapter 9.
The measurements described in this chapter are also discussed in Geilvoet (2004) and Evenblij *et al.*(2005).

7.1. Three MBR pilots

7.1.1. Maasbommel

The first pilot site was located at Maasbommel wwtp. The measurements were performed in February, during a period of heavy snowfall and low temperatures. Due to problems with the equipment the pilot installation was not functioning properly, resulting in low system permeability.
Furthermore, there were some troubles with the sampling of activated sludge. Instead of sampling from the return flow from the membrane tank, activated sludge was sampled from an (anoxic) internal recirculation flow, which proved to have a high fouling potential. The results of this period will be discussed in paragraph 7.3.
A second series of measurements was performed in July; activated sludge was sampled from the membrane tank. The results of this period will be discussed in paragraph 7.4.

The pilot installation at Maasbommel wwtp is schematically represented in Figure 7-1. The hollow fibre membranes in this installation were manufactured by Zenon Environmental Inc. and have a nominal pore size of 40 nm.
The MBR pilot was operated by waterboard Rivierenland, and was designed to test the possibilities of MBR to comply with the new standards for nutrient removal; see for an extensive description STOWA (2004).

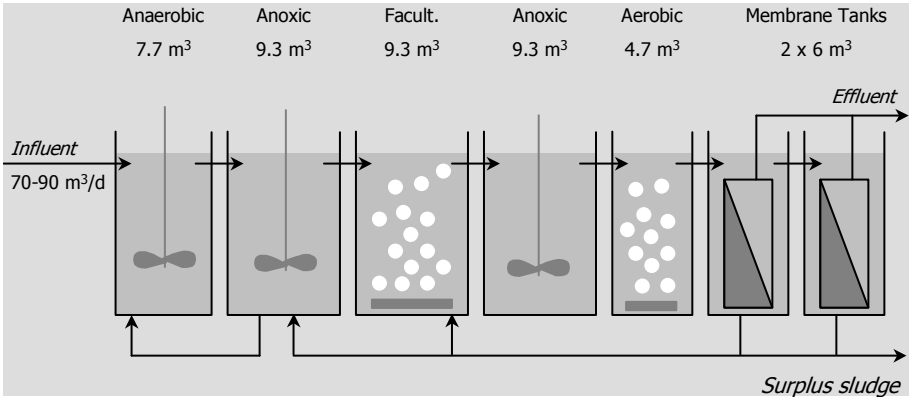

Figure 7-1 *MBR pilot at Maasbommel wwtp*

7.1.2. Hilversum

The second MBR pilot was located at Hilversum wwtp.

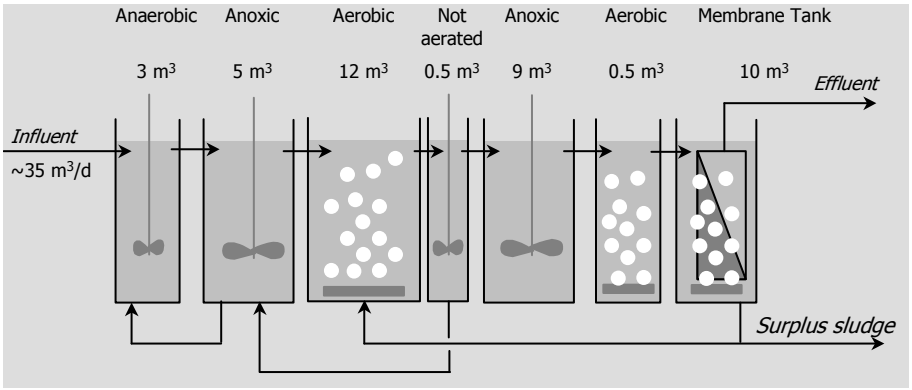

Figure 7-2 *MBR pilot at Hilversum wwtp*

The pilot had been in operation already 15 months and was equipped with Kubota plate and frame microfiltration membranes with a pore size of 500 nm.
The installation was equipped with a sodium acetate addition for nutrient removal. The total throughput was about 1.5 m^3/h; the schematic drawing of the installations is presented in Figure 7-2.

7.1.3. Beverwijk

Beverwijk wwtp was the location of several MBR pilots from 2000 until summer 2004. At the time of the measurements described in this chapter, only one MBR pilot was in operation. The installation was equipped with Huber ultrafiltration membranes with a nominal pore size of 40 nm. The installation had its own pre-treatment. The schematic overview of the installation is presented in Figure 7-3.

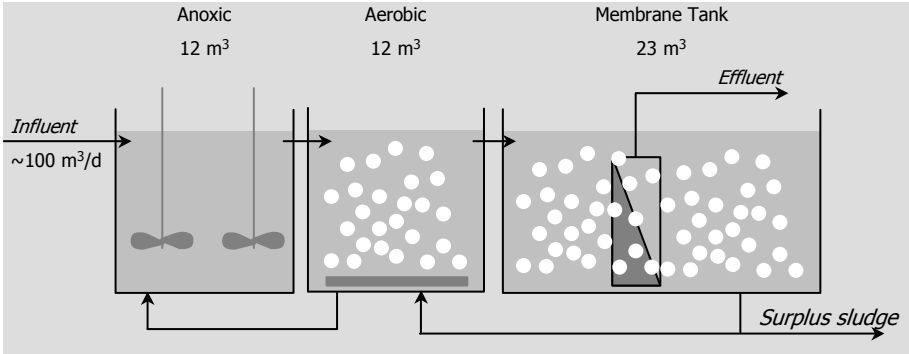

Figure 7-3 MBR pilot at Beverwijk wwtp

During the measurement campaign, the influent pump was in failure for a period of three days. This led to two periods of measurements that will be described and discussed separately. The second period, after restarting the influent pump is discussed in paragraph 7.6. The first period, which lasted for three days, is presented and discussed in the coming three paragraphs.

7.2. Filterability at three pilot sites

7.2.1. Characteristic filtration curves under standard conditions

Table 7-1 presents the average values and the range of ΔR_{20} that were measured with activated sludge from the three MBR pilot installations.

Table 7-1 Average values of ΔR_{20} for the three MBR pilot plants

	Permeate flux [L/m² ·h]	Average ΔR_{20} [·10¹² m⁻¹]	Range ΔR_{20} [·10¹² m⁻¹]
Hilversum	120	0.03	0.00 - 0.05
Maasbommel MT	80	0.13	0.05 - 0.27
Beverwijk	80	0.57	0.33 – 1.14

While measuring filtration characteristics at the different pilot sites it became clear that each installation had its 'own' filterability. Under standard conditions the filtration curves of an installation were found to be within a certain range. Figure 7-4 shows the filterability curves and ranges that were measured at the three pilot sites.

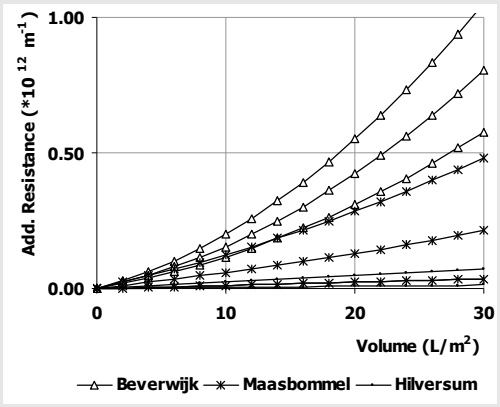

Figure 7-4 Differences in filterability of activated sludge from three MBR installations; Maasbommel and Beverwijk:J=80 L/m² ·h; Hilversum: J=120 L/m² ·h

Remarkably there was almost no overlap in the filterabilities, measured under standard conditions. The variations in filterability that were observed at each

pilot site did not change the type of filtration curve. Each curve followed the same trend and did not even cross one of the other curves. This means that the fouling mechanisms were of the same type for each activated sludge and did not change.

7.2.2. Sensitivity for Flux Variations

The filtration characterisation was carried out with varying values for permeate flux. This was partly standardised, in the sense that each day several experiments were performed under different conditions. In this way the sensitivity of membrane fouling with respect to these changes could be estimated. Thus for each value of permeate flux ΔR_{20} could be determined. The main conclusion of these experiments is that increasing the applied flux enlarged the differences between the three installations, see Figure 7-5. Thus, what was measured as a small difference at a flux of 80 L/m² ·h, could be a large difference when measured with a flux of 120 L/m² ·h. The relative 'ranking' of the filterability did not change as a result of changing the flux.

For each type of activated sludge there was a permeate flux at which there was no fouling, even after filtering during 30 minutes. This indicates the possibility of operating under so-called sub-critical operating conditions for the respective activated sludge.

It must be remarked that after each run in which fouling was observed, some fouling remained on the membrane that could not be removed by the standard chemical cleaning, which consisted of 30 minutes soaking in a 500 ppm sodium hypochloride solution. This probably means that although there may always be foulants in the activated sludge, these foulants will or will not reach the membrane, depending on the applied conditions. This again points to the idea of a critical flux.

This is in contrast with other situations, as described in *Chapter 6*, where in some cases fouling was found to be dependent on produced volume.

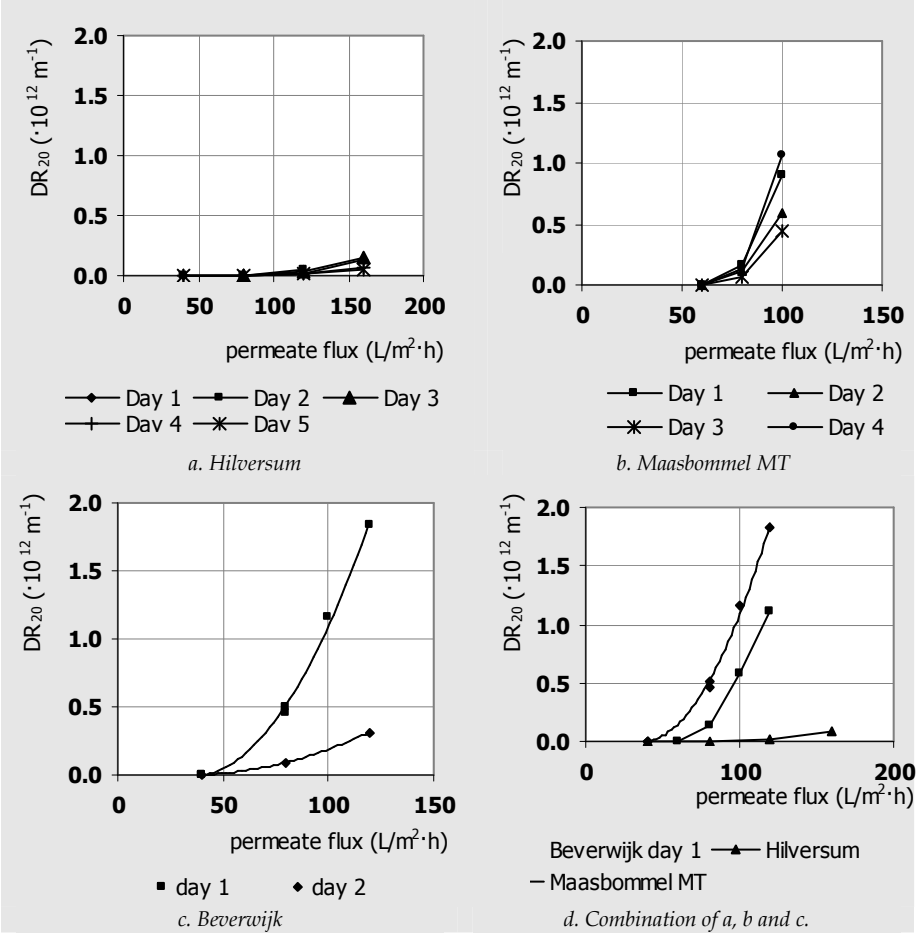

Figure 7-5 Sensitivity of ΔR_{20} for flux variations

If the fouling behaviour in MBR is generally totally dependent on applied flux, two remarks can be made. The fact that fouling is observed in almost all full scale submerged systems means that the membranes are operated below the optimum. This can be explained by supposing that the membranes will get blocked partially, leading to a higher net flux at the clean spots. Since a resistance increase is observed, it means that at these spots filtration takes places at super critical conditions.

7.2.3. Daily variations

Figure 7-4 and Table 7-1 show that considerable variations in filterability can occur during the measurements at one pilot site. When plotted on the same scale, the differences seem to become smaller with increasing filterability (i.e. lower ΔR_{20}). The relative variations however are in the same order of magnitude for each separate installation. For all measurements that were done at a flux of 80 L/m^2·h at Beverwijk and Maasbommel, and for the Hilversum case at 120 L/m^2·h, ΔR_{20} values are represented for each separate curve, see Figure 7-6. Remark the difference in scale of the vertical axes.

The filtration characterisation installation quantifies the differences between installations, therefore the changes in ΔR_{20} during one day are supposed not to be caused by the measuring method. This means that filterability really varies as shown in Figure 7-6.

The variations occurred in both directions: sometimes filterability became better, sometimes it became worse. There was no clear relation between the time of the day and the filterability, with an exception for the Hilversum case.

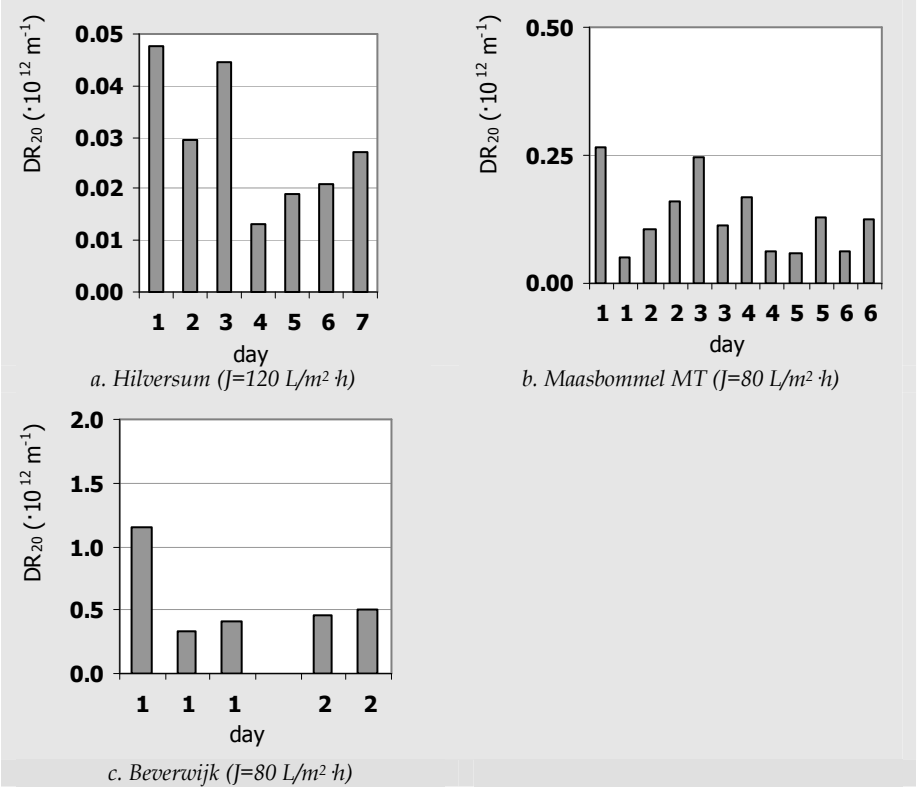

Figure 7-6 *Development of ΔR_{25} during the measurement campaigns; each bar represents one measurement. Remark the differences in the vertcial axes!*

Alternating Filterability at Hilversum

With the Hilversum activated sludge two 'types' of curves were obtained, one type showing a much faster resistance increase than the other. These two types of curves occurred over different days. At one day, both curves were measured within a time interval of 1.5 hours.

To show more clearly the difference in filterability, ΔR_{40} of these curves is presented in Figure 7-7a. From the seven curves that were measured under these circumstances during 6 days, four have a ΔR_{40} of about $0.1 \cdot 10^{12}$ m^{-1}, whereas the other three are about $0.35 \cdot 10^{12}$ m^{-1}. A closer look at the data shows that this phenomenon could be correlated to the time of sampling, see Figure 7-7b. The

lower values were all measured when the sample was taken at one o'clock in the afternoon. The other curves were measured either earlier or later.

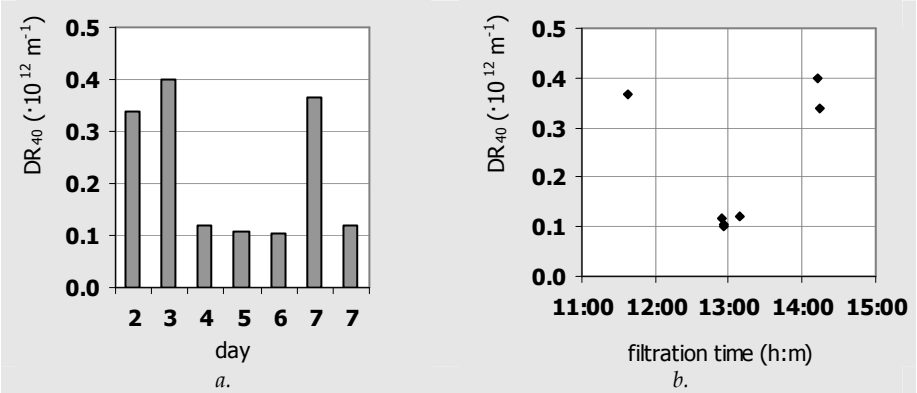

Figure 7-7 *Two types of fouling during filtration of Hilversum activated sludge at J=160 L/m²h*

The curves that were made with a flux of 120 L/m² ·h also showed the same trend, also with a minimum ΔR_{20} of 0 m^{-1} at about 13:00h, see Figure 7-8.

For the other installations the differences between filtration curves were not as pronounced as in the Hilversum case, which made correlation to the time point of sampling difficult. This may be explained by supposing that only in the case of Hilversum filterability is related to the operational cycle. The other two installations have other influencing factors far more important. This is supported by the fact that the absolute variations in the filterability of Hilversum activated sludge are orders of magnitude lower than the other two.

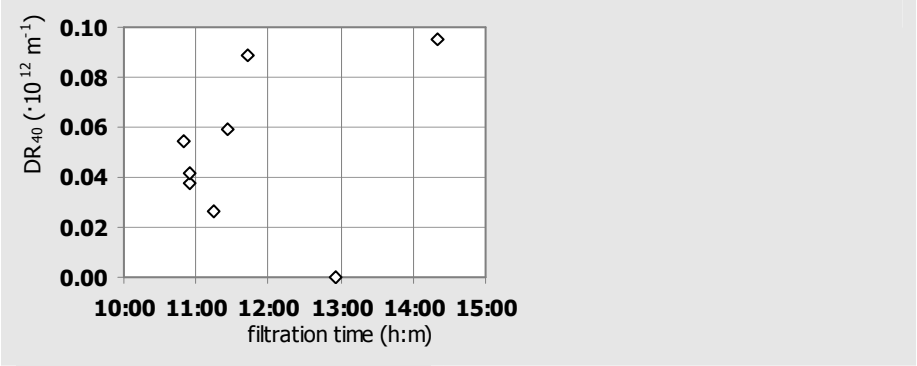

Figure 7-8 *Variation of ΔR_{40} as a function of time of day; Hilversum activated sludge at J=120 L/m² ·h*

Another explanation may be that daily variations in filterability do actually occur in the MBR pilots. In the other installations again these variations are insignificant because of other more important factors.
However this may be, every factor influencing filterability will eventually be 'translated' to the presence or absence of certain substances that play a role in the membrane fouling process. This is the subject of Chapter 8.

7.3. Filtration Characterisation in Exceptional Situations

7.3.1. Activated Sludge from Anoxic Recirculation (Maasbommel)

During the first measurement period at Maasbommel wwtp, activated sludge was erroneously taken from an internal recirculation flow, rather at the beginning stage of the process. Furthermore the measurements were carried out in a period of cold weather, heavy snowfall and troubles in operating the installation.

Filterability Results

The values of ΔR_{20} were 5 to 100 times higher than those obtained at the other installations, see Figure 7-9.
Most important to note is the order of magnitude that ΔR_{20} reaches in this case, almost 20 times higher than the Maasbommel activated sludge from the membrane tank in July. The differences may have several causes. Apart from

weather and temperature influences, the difference may be ascribed to the fact that the water phase of the activated sludge still contains untreated influent components. Although the ratio influent/activated sludge in this tank is quite low (1/13) the influence may be measurable quite well, which statement is supported by influent filtration experiments described by Ravazzini and van der Graaf (2005).

Another influence may be that the activated sludge is in anoxic conditions. This can have detrimental effect on a short term, since part of the activated sludge floc may break down, increasing the amount of free cells and other components in the water phase.

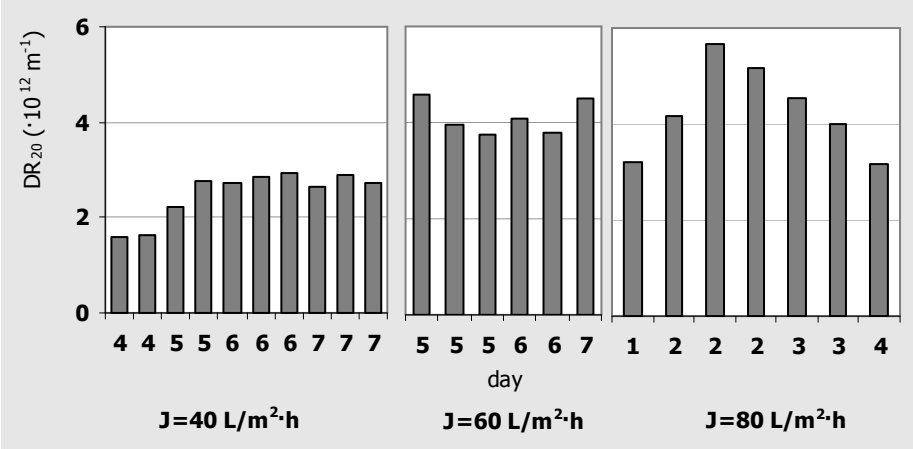

Figure 7-9 *Values of ΔR_{20} for Maasbommel CT measured with different fluxes*

Sensitivity for variation in permeate flux

With respect to the sensitivity to flux variations, it can be concluded that increasing flux above 60 L/m^2·h does not lead to an increase of ΔR_{20}, which means that the type of fouling that occurs becomes independent of the applied flux, see Figure 7-10. This figure presents the values of ΔR_{10} of all filtration curves that were measured. In this case ΔR_{10} is taken as reference value because the filtration curves with J=40 L/m^2·h and 60 L/m^2·h had to be interrupted at about 12 L/m^2. For the curves measured with J=40, 60 and 80 L/m^2·h, the average values of ΔR_{10} were calculated and plotted as 'average'.

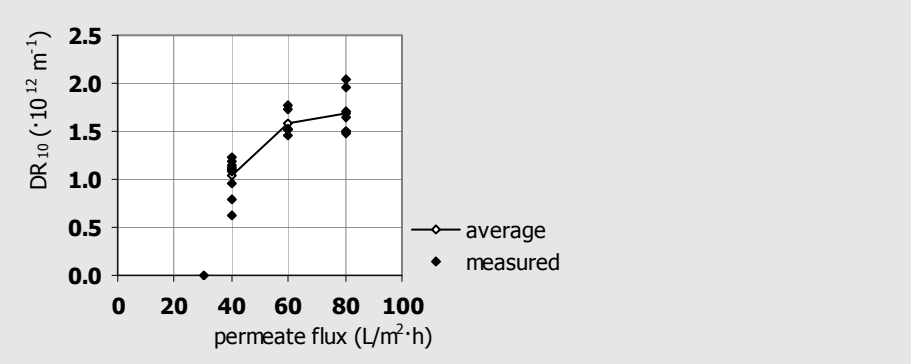

Figure 7-10 *Sensitivity of ΔR_{10} for variations in permeate flux*

The trend is somewhat different from the results obtained with the results presented in 7.2; the increase of ΔR_{10} becomes smaller with increasing flux.

7.3.2. Activated Sludge after Interruption Influent Flow

During the measurements at Beverwijk wwtp there were problems with the influent pump. This happened at the third day of the measurement period and resulted in operation of the installation without influent feed for three days.

Filterability Results

During the first of these three days the filterability increased dramatically. ΔR_{20} decreased from $0.51 \cdot 10^{12}$ m^{-1} at the end of day 2, down to $0.13 \cdot 10^{12}$ m^{-1} at day 3. After three days without influent addition, filtration under standard conditions was even sub critical (ΔR_{20}=0 m^{-1}). During these three days the MLSS concentration of the activated sludge decreased from 12.6 g/L to 10.9 g/L on day 4, and the temperature in the bioreactor increased considerably as a consequence of aeration, from 17 °C to values of about 27 °C.

In the days after reparation of the influent pump, at day 4 of the measurements, the filterability started to decrease during the remaining 4 days. The ultimate level of ΔR_{20} however was still lower than at the start of the measurement period.

Results of the measured filtration curves, summarised as values of ΔR_{20} are presented in Figure 7-11.

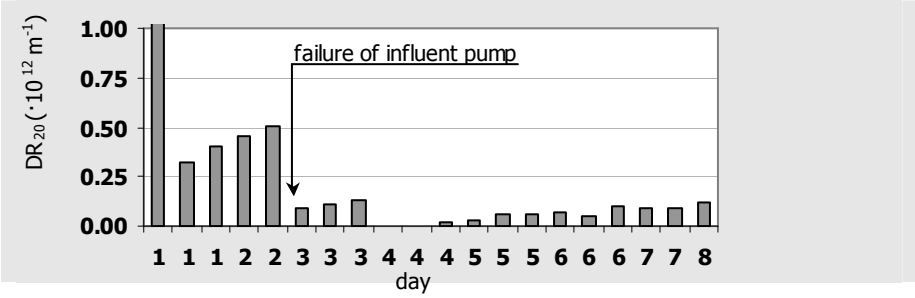

Figure 7-11 Development of ΔR_{20} after failure of influent pump (J=80 L/m² ·h)

7.4. Conclusions

Filtration characterisation can quantify differences in filterability between different installations. The filtration characterisation method was applied at three pilot sites and the measured differences are considerable.

The method can also quantify the influence of changes in operating conditions, like the case at Beverwijk wwtp, where the influent flow was interrupted for three days.

For each installation sub-critical conditions could be reached in the filtration characterisation unit.

The value of ΔR_{20} increases with increasing permeate flux; the relative ranking in terms of ΔR_{20} does not change with increasing permeate flux.

References Chapter 7

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If you see through everything, then everything is transparent. But a wholly transparent world is an invisible world. To `see through' all things is the same as not to see.
C.S. Lewis, The Abolition of Man

8
CORRELATING FILTRATION BEHAVIOUR TO EPS AND INSTALLATION CHARACTERISTICS

8.1. Introduction

The differences in activated sludge filterability between the three MBR pilot installations as described in chapter 7 are quite large. The next step in this research is to try to connect these differences to physical or chemical properties of the respective mixed liquor that was filtered.

One of the conclusions from the literature review (chapter 3) is that extracellular polymeric substances may be related to fouling processes in membrane bioreactors. More specifically, the polysaccharides and proteins in the water phase of activated sludge were shown to play a role in membrane fouling.

This chapter describes the experiments in which the role of colloidal and soluble EPS in membrane fouling during filtration characterisation is investigated. To this aim, the activated sludge of the pilot plants and permeate of filtration characterisation experiments described in Chapter 7 were analysed for EPS. The question is: can EPS in the water phase of activated sludge be correlated to fouling behaviour during ultrafiltration of the same activated sludge?

Firstly, the results of the EPS measurements during the measurement periods in which the installations were operated properly are presented, in paragraph 8.2.1. For the Beverwijk occasion where the influent pump went in failure during the weekend, only the first two days are presented. The remaining days are discussed in 8.2.2

The measurements carried out with activated sludge from the Maasbommel CT are mentioned briefly in paragraph 8.4 and the EPS values will be presented in 8.2.3.

Also other factors, such as membrane pore size and characteristics of the pilot plant are concisely discussed as possible causes of changes in filterability.

8.2. EPS measurements

Based on theoretical considerations as to which constituents may be involved in membrane fouling in MBR (see also Chapter 3), only the water phase of the activated sludge samples was analysed for EPS. The water phase is separated from the biomass by filtrating small amounts of the mixed liquor (some 25 ml) over a paper filter that is initially rinsed with demineralised water (to flush out lose fibres and other material that could interfere with the analysis).
Also the permeate that is produced during filtration characterisation is analysed for EPS. At the end of the filtration run permeate was sampled directly from the permeate outlet.

8.2.1. EPS measurements during normal operation

EPS in the water phase of activated sludge

Figure 8-1 shows the average amount of EPS (proteins and polysaccharides) that was measured in the activated sludge water phase of the three MBR pilot installations, as well as the breakthrough of these substances, as measured in the permeate; also the calculated colloidal fraction is presented. Table 8-1 presents as a reference the average values of ΔR_{20} for the three installations.

Table 8-1 Average values of ΔR_{20} for the three MBR pilot plants, discussed in Chapter 7

	Average ΔR_{20} [$\cdot 10^{12}$ m^{-1}]
Hilversum	0.02
Maasbommel MT	0.13
Beverwijk	0.57

Hilversum activated sludge shows the highest amount of EPS in the water phase, followed by Beverwijk and Maasbommel. The ranking of filterability for the three types of activated sludge is totally different from the amounts of EPS in the water phase, in the permeate and the calculated colloidal fraction.

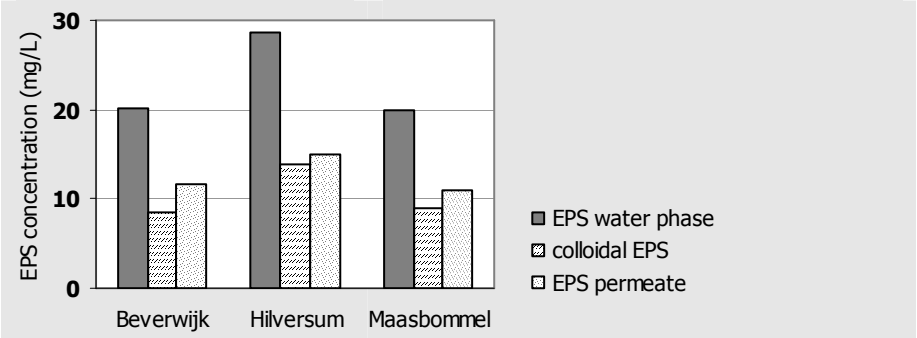

Figure 8-1 *Average EPS concentrations in permeate (J=80 L/m² ·h),water phase, and colloidal fraction*

Figure 8-2 shows the two groups of substances that were measured (proteins and polysaccharides) in the water phase of the activated sludge samples. For each filtration curve the analysed EPS values are presented for the three installations.

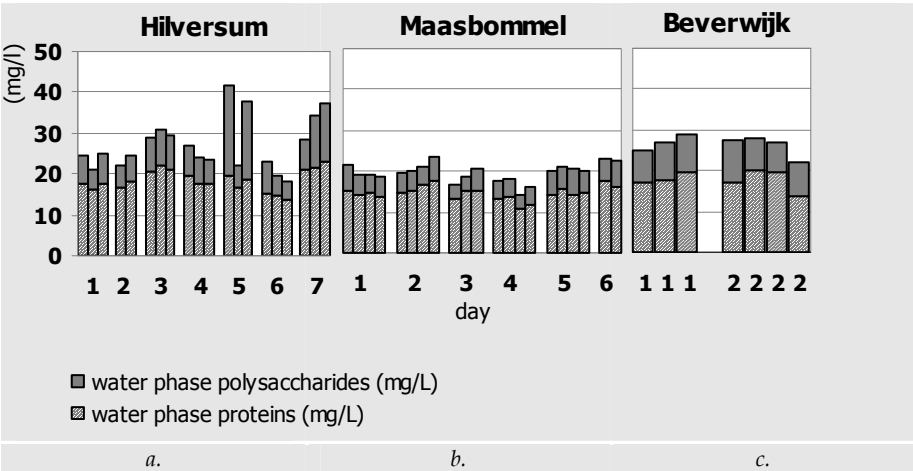

Figure 8-2 *Protein and Polysaccharide concentration in the water phase of three MBR pilot installations*

Proteins form the greater part of the EPS in the water phase, around 75% of the total EPS. The remaining 25% consist of polysaccharides, see also Table 8-2.

Table 8-2 *Ratios of proteins and polysaccharides in the different activated sludge water phases and permeates*

	Water phase		Permeate*	
	proteins [%]	polysaccharides [%]	proteins [%]	polysaccharides [%]
Hilversum	72	28	83	17
Maasbommel	74	26	86	14
Beverwijk	75	25	82	18

* Average values at J=80 L/m² ·h

EPS in the permeate of filtration characterisation experiments

The permeate of the filtration characterisation had a slightly different composition in terms of proteins and polysaccharides, compared to the water phase. The relative amount of proteins increased to about 83% and the remaining 17% consisted of polysaccharides, see Table 8-2.

The absolute values of the proteins and polysaccharides in the permeates, measured during the experiments with a permeate flux of 80 L/m² ·h are presented in Figure 8-3.

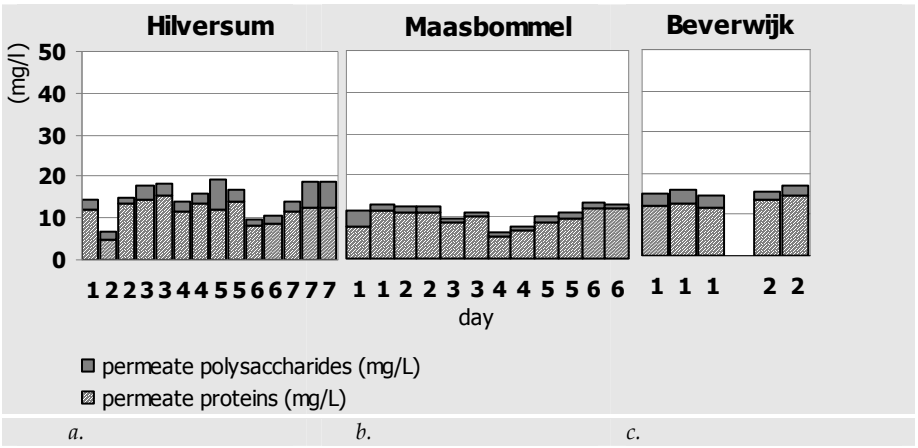

Figure 8-3 *Protein and Polysaccharide concentration in the permeate of fitlration characterisation experiments with activated sludge from three MBR pilot installations(J=80 L/m² ·h)*

Colloidal EPS

The extent to which the EPS pass the membrane during ultrafiltration provides probably an indication of the particle size distribution. Those substances that have a characteristic size smaller than the pore size of the membrane (or the cake layer) will be found in the permeate. The larger particles (the colloidal fraction) will be available for membrane fouling. Figure 8-4 presents the colloidal proteins and polysaccharide concentrations in the water phase of activated sludge during the filtration characterisation experiments. In the case of Beverwijk, sometimes the EPS concentrations in the permeate equalled the concentration in the water phase, resulting in a colloidal fraction of 0 mg/L.

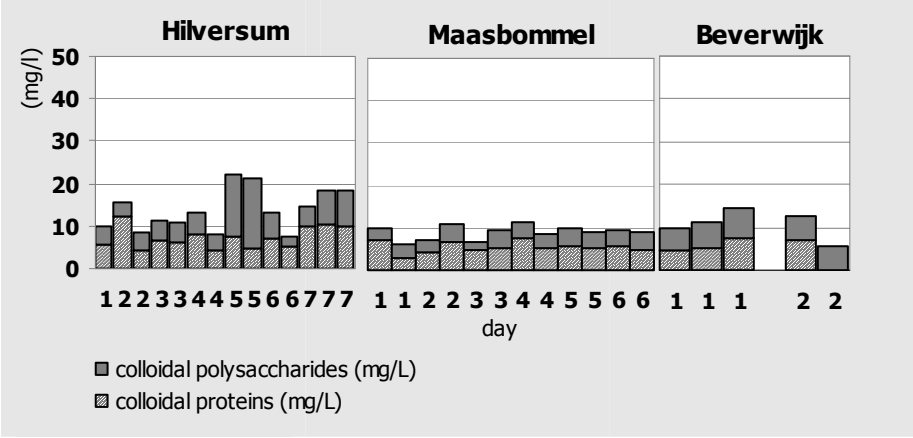

Figure 8-4 *Colloidal Protein and Polysaccharide concentrations during filtration characterisation of activated sludge from three MBR pilot installations (J=80 L/m² ·h)*

The absolute concentrations of potential foulants (colloidal EPS) are quite low. If these values are for example compared to those measured with the experiments described in Chapter 6, these values are almost insignificant. Furthermore, the differences between the installations are much smaller than the differences in filtration behaviour. Lastly, the small differences that can be identified yield to another ranking than was measured with the filtration experiments.

8.2.2. EPS concentrations during influent pump failure

During a weekend the influent pump of the Beverwijk pilot plant was in failure. The installation was not provided with new influent and no permeate was extracted.

As a consequence, the filterability drastically increased, reflected by values of ΔR_{20} of almost 0 m^{-1} on Monday. On Monday the pump was reset and influent was fed again to the bioreactor. The four subsequent days showed a gradual decrease in filterability. The corresponding EPS measurements are presented in Figure 8-5.

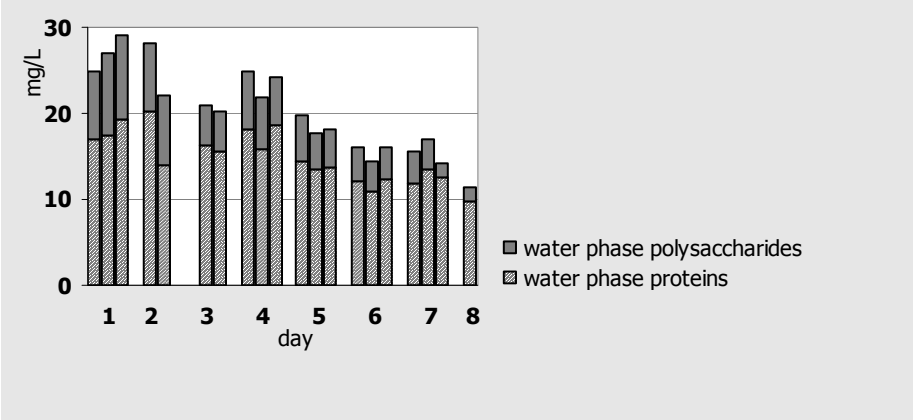

Figure 8-5 *EPS concentration in water phase of activated sludge; at the end of day 2 the influent pump went in failure*

The EPS concentrations do not follow the trend that was observed with filtration characterisation. On the contrary, during day 4 to 8, where ΔR_{20} gradually increased, the total amount of EPS decreased; both the polysaccharide and protein concentrations decreased.

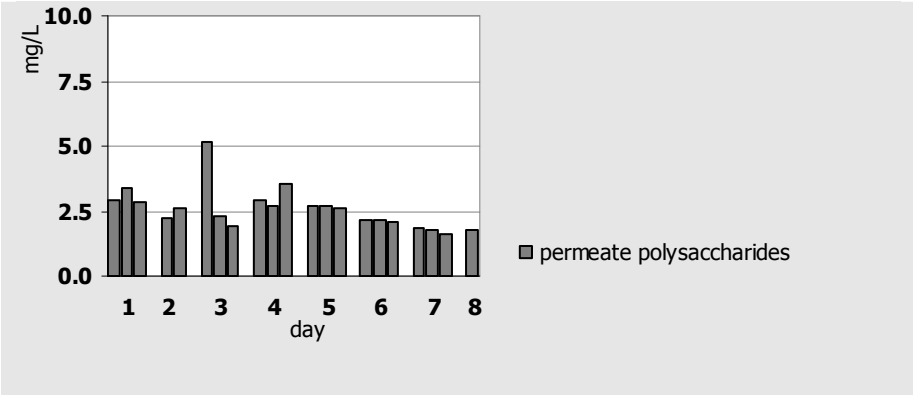

Figure 8-6 *EPS concentration in permeate of activated sludge; at the end of day 2 the influent pump went in failure*

The protein measurements of the permeate were not carried out correctly, so only the polysaccharide concentrations in the permeate are presented here, as well as the resulting retention, see Figure 8-6 and Figure 8-7.

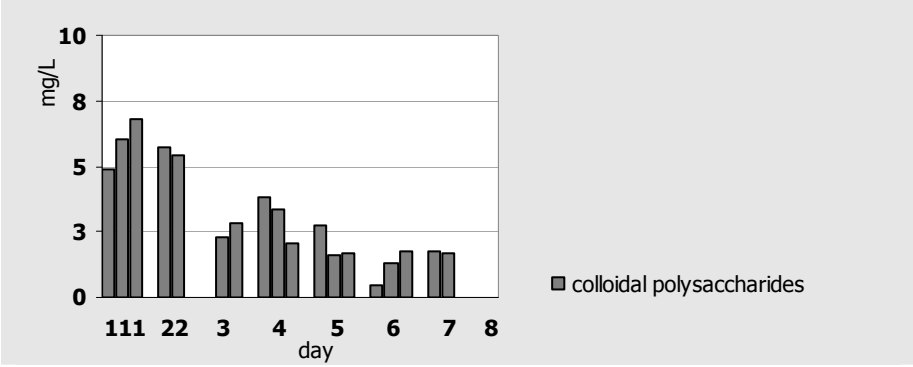

Figure 8-7 *Colloidal EPS concentration in water phase of activated sludge; at the end of day 2 the influent pump went in failure*

8.2.3. EPS concentrations in activated sludge from internal circulation

The first measurements at the Maasbommel pilot site were carried out with activated sludge from the facultative anoxic zone, indicated as Maasbommel CT. The EPS concentrations were quite high, as well as the colloidal fraction. This may be caused by the application of high shear pumps, leading to floc damage. The average values of EPS in the water phase, the colloidal fraction and EPS in the permeate are presented in Figure 8-8.

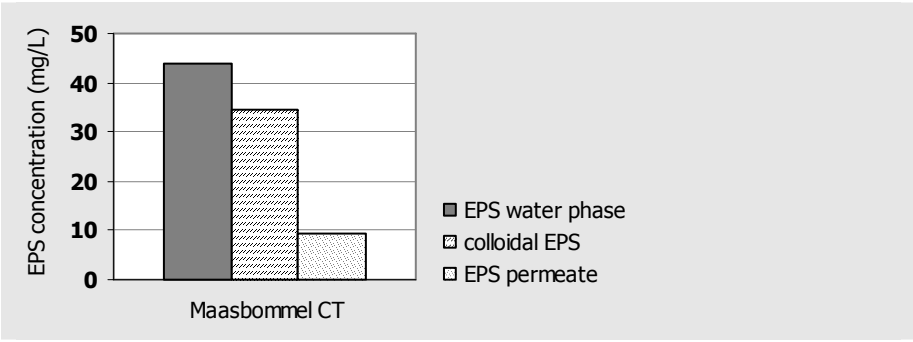

Figure 8-8 *EPS concentrations in the water phase, permeate and the calculated retention*

The fact that the concentration of EPS in the permeate was quite low, indicated the formation of a cake layer which may have acted as a secondary membrane. This approach is further discussed in paragraph 8.4 *Break through and fouling*.

8.3. Colloidal EPS vs. ΔR_{20}

The colloidal fraction, as calculated from the measured EPS concentrations, can be plotted against the filterability, expressed as ΔR_{20}, see Figure 8-9.

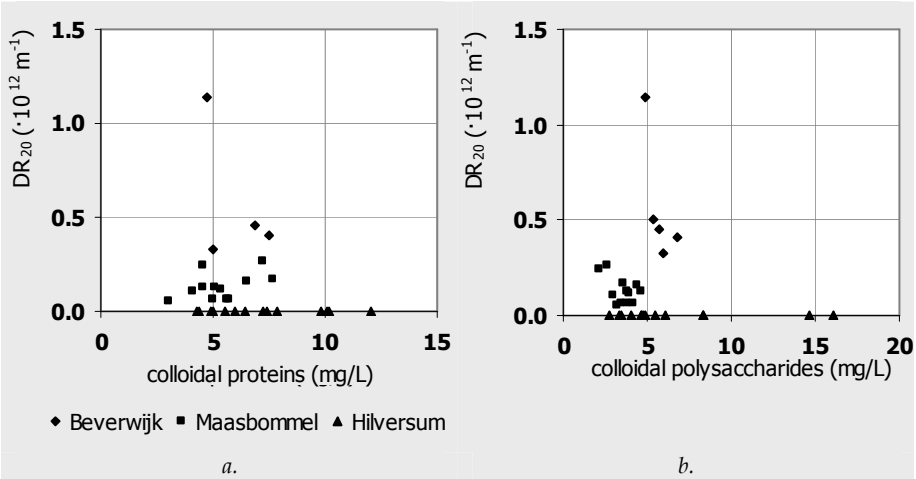

Figure 8-9 *ΔR_{20} against concentrations of colloidal EPS, calculated as the difference between concentrations in the water phase and the permeate; J=80 L/m² ·h*

Only when one 'outlier' is excluded, there is a weak relation between colloidal polysaccharides and filterability, most clearly in the case of Beverwijk and Maasbommel. Also in this case, Hilversum forms an unexpected exception. Since all filtration curves at 80 L/m² ·h yielded a ΔR_{20} of 0 m^{-1}, it was expected that the colloidal EPS fraction would be low. One of the two highest colloidal polysaccharide concentrations however is obtained with activated sludge of the Hilversum pilot plant.

8.4. Break through and fouling

Apart from correlating EPS concentrations to filtration behaviour, the EPS concentrations may reveal information about fouling phenomena. In this respect it is interesting to consider the permeate concentrations of EPS as 'breakthrough' of these substances.
Figure 8-10 shows the results of calculating the ratios of $c_{permeate}$ and c_{water} for different values of permeate flux. The calculations are made both for proteins and polysaccharides.

For the Hilversum case the critical flux is higher than 80 L/m^2·h, and measurements are available for 40, 80, 120 and 160 L/m^2·h. When filtering below critical conditions $c_{permeate}/c_{water}$ decreased somewhat while increasing the permeate flux. Afterwards, $c_{permeate}/c_{water}$ increased, indicating the permeation of more proteins smaller than the pore size. For the permeate fluxes that were investigated, the amount of proteins that passed the membrane increased slightly, from 65 to 73%. The polysaccharides show a somewhat different picture, the fraction passing the membrane is constant when flux is increased.

Although there are less data points for the Maasbommel MT case, a comparable trend can be observed.
For the Beverwijk case, the differences are bigger. For the proteins, $c_{permeate}/c_{water}$ increased considerably, from 32% at J=40 L/m^2·h to 78% at J=120 L/m^2·h. A somewhat smaller increase is observed with the polysaccharides: from 27% to 58%. Thus the breakthrough of polysaccharides is highest for the Beverwijk sludge.

The first period of measurements at the Maasbommel pilot plant are also presented here (as Maasbommel CT). Both for proteins and polysaccharides the $c_{permeate}/c_{water}$ is smallest for the highest applied flux. Furthermore there is a strong relation with the flux in the case of proteins: from 58% at 40 L/m^2·h to 26% at 80 L/m^2·h. This may point to the increasing involvement of larger particles, forming a dense layer that is non-permeable to EPS.

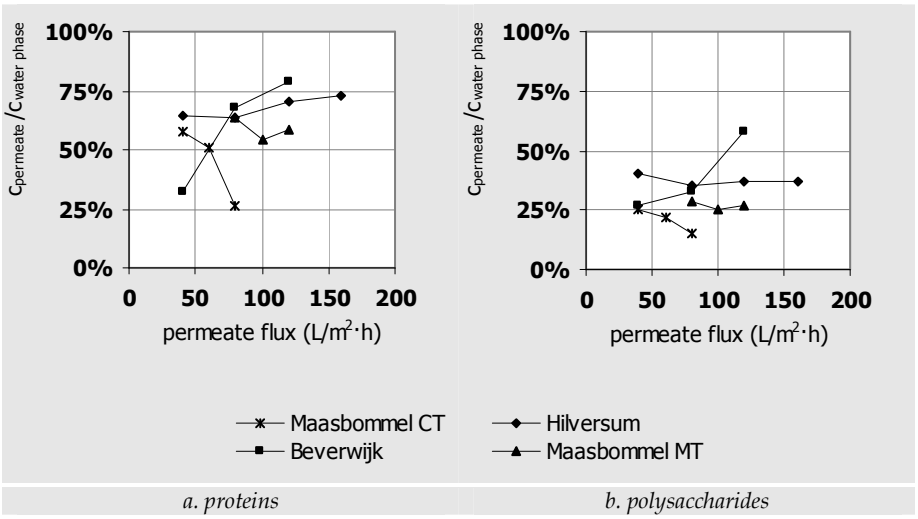

Figure 8-10 *Ratios of $c_{permeate}/c_{water}$ at different permeate fluxes and for different substances*

The conclusion may be that the value of $c_{permeate}/c_{water}$ as a function of permeate flux indicates the type of fouling mechanisms that occur, where a decrease indicates a higher fouling propensity. The best filterable activated sludge (Hilversum) had more or less constant $c_{permeate}/c_{water}$ values, even when fluxes up to 160 L/m² ·h were applied.

8.5. Assessment of other fouling influencing factors

Apart from EPS concentrations in the water phase of the activated sludge there are numerous points in which the MBR pilot installations differ from each other. Almost all of them may somehow influence filtration behaviour, although quantification of them is difficult. Some of these points are considered in the next paragraphs.

8.5.1. Pilot Membrane pore size

Each of the three installations where activated sludge was sampled had its own membrane type with which it was operated. Hilversum was operated with plate and frame microfiltration membranes. Beverwijk and Maasbommel were

operated with ultrafiltration membranes, with plate and frame membranes in the first and hollow fibre membranes in the latter, see Table 8-3.

Table 8-3 *Membrane characteristics of pilot plants*

pilot plant	membrane system	pore size (nm)
Hilversum	fixed plate and frame, outside-in, Kubota	500
Maasbommel	hollow fibre, outside-in, Zenon	40
Beverwijk	rotating plate and frame, outside-in, Huber	40

Since the membrane in the filtration characterisation unit was a tubular ultrafiltration membrane, it may be that this difference determines the filtration curves that were obtained. Firstly the influence of the pilot membrane on the activated sludge filterability has to be assessed.

If this influence is interpreted in terms of particle size distribution of the mixed liquor, i.e. the cut off of the membrane, it is important to realise that each activated sludge floc enters the membrane tank only once in 10 to 20 hours. On the other hand there are numerous other factors that will most likely influence the quality of the water phase much more than this. For example the mixing with influent, its subsequent aeration, taking part in recirculation flows tens of times, etc. All this will certainly overrule the influence of one membrane passage.

The effect of the membrane would be to keep particles in the system that would otherwise be washed out in a secondary clarifier. Theoretically, the activated sludge from the Hilversum plant would contain the smallest amount of particles, since they would pass the relatively large membrane pores. As was shown in Figure 8-1 the water phase of Hilversum activated sludge contained a quite high fraction smaller than 30 nm, the pore size of the filtration characterisation membrane.

8.5.2. Pumping and Recirculation

The amount of pump passages mentioned in the former paragraph will probably influence the sludge quality. Furthermore, the hydraulic regime in the installation is supposed to be of influence. If throughout the installation the

shear rate is kept as low as possible, flocculation takes place to the highest possible degree, which seems favourable for good filtration properties. The number of pump passages amount to several hundreds during the SRT for each installation and do not differ that much for the installations.

8.5.3. Hydraulic Retention Time

The hydraulic retention time can be regarded as a characteristic of the water phase, and it could therefore possibly be related to filterability.

The hydraulic retention time can be regarded as the lifetime of the water phase in the installation. During this lifetime sedimentation, flocculation, biological uptake, degradation or stripping will take many substances out of it. Other processes will add substances to the water phase, like microbiological excretion products and floc breakage by shear forces. All these processes and the time that is provided by the operating conditions to perform them, will lead to a certain quality of the water phase that can be measured as filterability. This hypothesis is tested by plotting the values of ΔR_{20} against HRT of the different installations, see Figure 8-11.

There seems to be a trend: higher HRTs lead to a better filterability. Although there are only three points, these can be extended with two other values that were obtained accidentally.
The first one was obtained at the pilot site Beverwijk. Here the influent pump broke down just before the weekend, and during three days the sludge was without influent feed and was only recirculated. Filterability enormously improved already after one day, with ΔR_{20}values as low as $0.01 \cdot 10^{12}$ m^{-1}. This can be considered as a case where HRT went up to values of 70 hours, providing another point in the graph of Figure 8-11, see also paragraph 8.2.2.

The second point was obtained at the Maasbommel pilot plant. Due to a mistake in the first measuring period, activated sludge was sampled from an internal recirculation stream, originating from the third step in the treatment train. Thus, the water phase had had a retention time of approx. 45 minutes, and the

measured values of ΔR_{20} were averagely $4.3 \cdot 10^{12}$ m^{-1}. This provides a point to the left and beyond the scale of the vertical axis in Figure 8-11.

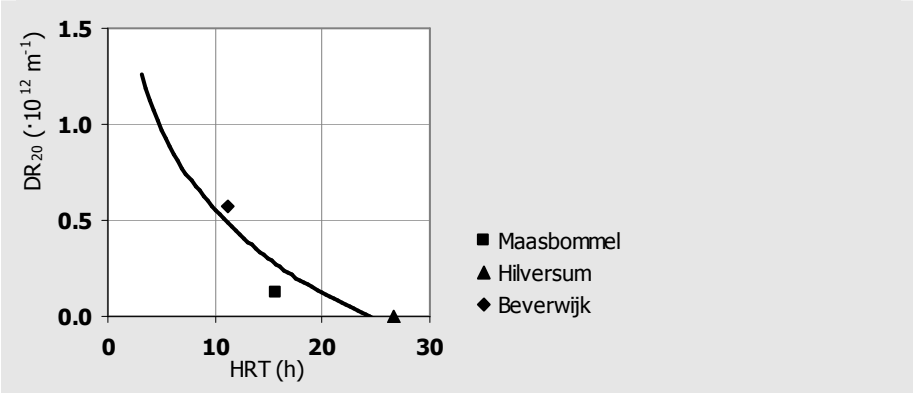

Figure 8-11 *Relation between HRT and ΔR_{20}*

8.6. Conclusions

The results described in Chapter 7 can only difficultly be correlated to the analysed EPS concentrations in the water phase and permeate.
Neither for the summed EPS, nor for its components a relationship was observed between fouling and EPS.

Membrane pore size of the pilot plant cannot be correlated to the fouling behaviour in the filtration characterisation unit.

There seems to be a relation between HRT and ΔR_{20}, although only three points are available.

Do not draw this conclusion from your experiment, that there remains nothing for you to know; but rather that there remains an infinity for you to know.[*]
Blaise Pascal, Pensées

9
DISCUSSION

9.1. Introduction

This chapter discusses and summarises the results from chapters 4 to 8 and the questions that arise from their evaluation. The main outcomes of the research will be discussed according to the following questions:

- is the filtration characterisation method strong enough to quantify filterability of activated sludge (§9.2);
- can the filterability of activated sludge be influenced by varying substrate gifts (§9.3);
- are the circumstances during filtration characterisation representative for filtration behaviour in an MBR installation (§9.4);
- can the filtration characterisation method discriminate between different MBR installations (§9.5)
- can the filtration behaviour as measured during filtration characterisation be correlated to the presence of soluble EPS in the water phase of activated sludge or to other parameters (§9.6).

9.2. Quantifying filterability

The filtration behaviour of activated sludge can be quantified by the filtration characterisation method described in chapter 4. Constant flux experiments are relatively easy to perform and yield useful results within a limited period of time. The obtained filtration curve can be summarised by one value of additional resistance, which is the filtration resistance minus the clean membrane resistance. The value of additional resistance after producing 20 L/m² of permeate (ΔR_{20}) is considered a useful value for this purpose.

* The whole paragraph runs like this : Do you believe it to be impossible that God is infinite, without parts? Yes. I wish therefore to show you an infinite and indivisible thing. It is a point moving everywhere with an infinite velocity; for it is one in all places and is all totality in every place.
Let this effect of nature, which previously seemed to you impossible, make you know that there may be others of which you are still ignorant. Do not draw this conclusion from your experiment, that there remains nothing for you to know; but rather that there remains an infinity for you to know.

Application of a single tubular membrane ensures control over the hydraulic circumstances during filtration.
Constant TMP experiments are difficult to carry out and cannot be regarded as being representative for filtration in an MBR.

Although the reproducibility of the filtration characterisation method could not be verified by simply filtrating the same sample more than once, the accurateness of the measurements is proved by the results from chapter 7. For the Hilversum case the filterability at 80 $L/m^2 \cdot h$ (14 times measured) invariably was 0 m^{-1}. Furthermore, the range of ΔR_{20} as measured with 120 $L/m^2 \cdot h$ went from 0 to $0.05 \cdot 10^{12}$ m^{-1}; for the MBR installation with the worst filterability (Beverwijk) the range was 0.33 to $1.14 \cdot 10^{12}$ m^{-1} during normal operation. This means that the accuracy of the method is determined by the accuracy of the measuring devices for TMP and flux measurements.

9.3. Manipulating filterability

The filterability of activated sludge can be manipulated by adding an easily degradable substrate in high dosages. After dosing a one-time gift of substrate, ΔR_{20} increases over a period of three days. Depending on the exact amount of substrate and the installation where the sludge is sampled, the sludge will react faster and more or less severe to the substrate gift.
In these experiments the increase of ΔR_{20} coincided with an increase of EPS in the water phase. The ultimate values of EPS in the water phase could be hundreds of mg/L, whereas in a normal operating MBR the EPS concentrations amounted to around 40 mg/L.
Firstly it was supposed that these findings confirmed the results found by Rosenberger and Kraume (2002) and published as such in Rosenberger *et al.*(2005). The results of the further following research seem to falsify this conclusion, see §8.2 and §9.6.
This occasion also shows the risks of misinterpreting and generalising the results of the filtration characterisation method. It can be defended that in the case of the substrate experiments EPS were involved in the process of membrane

fouling, since the EPS concentrations were very high. On the other hand this does not necessarily mean that these EPS are always the cause of and involved in fouling processes.

9.4. Representativity

The most obvious difference between MBR installations in practice and the filtration characterisation method is the applied permeate flux. Full scale MBR systems apply permeate fluxes of about 10-40 L/m^2·h (Stephenson *et al*, 2000), whereas permeate flux during filtration characterisation is much higher and standardised at 80 L/m^2·h. This difference can be defended by realising that

1. the difference between actual permeate flux (in full scale) and filtration characterisation flux is smaller than the difference between design flux and filtration characterisation flux; this is further discussed in 9.4.1 and 9.4.2 and,
2. an increase in flux usually does not lead to changes in fouling mechanisms, fouling is only more intensive; this is further discussed in 9.4.3.

9.4.1. Local critical flux

During filtration characterisation high fluxes are applied (in a standard experiment: 80 L/m^2·h), which is high compared to nominal permeate fluxes that are applied in full-scale operation. Membrane manufacturers advise fluxes in the range 10-40 L/m^2·h, with practical values up to 30 L/m^2·h (Stephenson *et al*, 2000; Kraume and Bracklow, 2003).

Permeability

Full scale MBR operation is usually designed to operate under sub-critical conditions, i.e. no increase in TMP is experienced. If TMP increases too much during a filtration run, measures are taken to avoid further performance decline.
A commonly used parameter to evaluate system performance is membrane permeability, expressed as the ratio of permeate flux and operational TMP, L/m^2·h·bar, see eq. 8-1.

$$P = \frac{J}{\Delta P} = \frac{dV}{dt \cdot A_m \cdot \Delta P} \qquad \textbf{(9-1)}$$

where

P	= Permeability	[L/m² ·h ·bar]
J	= Permeate Flux	[L/m² ·h]
ΔP	= Trans Membrane Pressure	[bar]
A_m	=Membrane area	[m²]
V	=Produced permeateVolume	[L]

For example, German guidelines for MBR in municipal wastewater treatment prescribe an intensive cleaning when permeability becomes lower than 100 L/m² ·h ·bar, (MUNLV 2003, cited in Kraume and Bracklow, 2003). Permeability is a useful parameter for operators and for scientists as well, as long as the working conditions under which it is calculated are mentioned. Furthermore, with fouled membranes the permeability depends on the TMP because the fouling layer is compressible.

It is important to realise that permeability is an average value, calculated for a membrane compartment, or membrane module. This can lead to underestimation of the filtration performance, since the membrane area (A_m) available for permeation may be less than the installed amount.

Many MBR systems are operated below a so called secondary critical flux, which means that the membrane permeability is lower than the clean water permeability but is not decreasing in time. For example, a membrane with an initial clean water permeability of 1000 L/m² ·h ·bar, may be producing a constant flux of 25 L/m² ·h, not at the expected TMP of 0.025 bar, but with a TMP of 0.05 bar. This means that

1. a certain amount of fouling was accumulated at the membrane, causing an overall permeability decline, or,
2. part of the membrane is clogged, and the remaining clean part of the membrane is producing the permeate. In this example, 50% of the membrane surface is clogged, because production of the mean amount of 25 L/m² ·h is achieved at a TMP two times higher than was expected from the clean water permeability.

It can be imagined that in this way local permeate flux will exceed the critical value at some locations of the total membrane area, inducing transient fouling behaviour. This concept of local fluxes higher than the net flux is described by Ognier (Ognier *et al*, 2004). It is expected that in every day practice the two situations described above can occur at the same time and that the clogged part of the membrane can all the same contribute to permeate production to a small extent (Heijman *et al,* 2004).

Ineffective Cross flow aeration

The locally exceeding of the critical flux is a consequence of the way in which MBR systems are operated, particularly submerged membrane systems. In submerged systems membrane cleaning is performed by a (dis)continuous air flow along the membrane plates or hollow fibre bundles, sometimes combined with the moving of the membrane itself. It can be expected that cleaning of these membranes will be discontinuous in time. Part of the membrane will be clean and other parts will get fouled. A third mechanism of membrane fouling can be defined:

3. Part of the membrane is clogged and the rest is clean. The location of these parts may change with time, depending on the effectiveness of the cleaning with air.

Those parts of the membrane that are involved in permeation will thus face a higher flux than designed and have to cope with a higher load of foulants. The fact that the nominal critical flux in many installations is so low, sustains this model. For example, activated sludge from the Hilversum MBR pilot plant was filtered under sub critical conditions in the filtration characterisation unit with a flux of 80 L/m^2·h. The pilot installation itself operates with fluxes as low as 10-15 L/m^2·h. During storm events the flux is increased to 20-30 L/m^2·h, leading (locally) to super critical operation, indicated by an increase in TMP. The mere fact that with relatively low permeate fluxes a permeability drop is experienced indicates an ineffective cross flow.

9.4.2. Cross flow velocity

The cross flow velocity of the bulk flow will cause turbulence near the membrane and will disturb the boundary layer. It promotes back transport mechanisms, which results in reduction of filtration resistance. Furthermore, the shear stress that is imposed can drag away cake layer parts. As was mentioned before, the effectiveness of the cross flow can be very low in submerged systems. It seems likely that because air and water follow paths with a minimum of energy loss, those places where cross flow is 'needed' most, i.e. the already fouled parts of the membrane, will have a lower amount of cross flow, speeding up the process of fouling and clogging.

In the tubular membrane of the filtration characterisation unit the cross flow can be supposed to cause a constant shear stress along the membrane wall. Although membrane flux is a function of tube length (Song, 1998, Kromkamp *et al*, 2002), the absolute variation is small. Furthermore, the permeate extraction is taking place under well-defined conditions. The activated sludge filtration in the filtration characterisation unit is therefore regarded as representative of what is happening at those spots where filtration rates are highest in full scale MBRs.

9.4.3. Fouling mechanisms

The fouling mechanisms that occur during cross flow ultrafiltration of activated sludge are dependent on the applied operating conditions, i.e. the ratio between fouling forces (permeate flux) and 'de-fouling' forces (back transport by cross flow) which can be expressed by the ratio of crossflow velocity and permeate flux: u_{cr}/J. Those parts of the membrane in a full scale installation partaking in permeation will experience a certain value of u_{cr}/J. The fouling mechanisms that occur at those spots are comparable to those that occur in the filtration characterisation. The only difference is the extent to which the fouling is allowed to take place.

It is however possible that other fouling mechanisms occur in submerged membrane systems. Every time a certain spot at the membrane is cleaned by the discontinuously effective cross flow, it is again involved in the filtration process. Because the cross flow may not be effective at that time, activated sludge will be

transported to the membrane unhindered and either form an impermeable layer, or a secondary membrane which acts as a barrier for potential foulants. It seems undesirable to work under these conditions since activated sludge flocs tend to adhere to the membrane surface. Once this takes place removal is very difficult, as exemplified in the case of both plate and frame membranes and hollow fibres. In the case of plate and frame modules it was experienced that an ineffective cross flow led to a 50% coverage of the membrane surface with activated sludge deposits.

From these considerations it is concluded that an MBR installation can be regarded as functioning properly if the equipment for turbulence provides a continuously and equally distributed amount of energy at the membrane surface, to assure that back transport mechanisms will be activated. For those parts of the membrane where this is true, the filtration characterisation will accurately mimic what is going on in the full-scale installation.

9.4.4. Constant Flux or Constant TMP

For a proper evaluation of results obtained with Constant TMP experiments, the starting-point should be the same for each measurement. The occurring fouling mechanisms seem to be dependent on the ratio between permeate flux and shear force. This ratio is changing during Constant TMP experiments, since flux is decreasing with time.
During Constant Flux experiments this ratio is constant, since only TMP is increased. A drawback of this method is that fouling layer compression can be expected to take place from a certain value of TMP. When operating a full scale MBR this will be avoided at all times and can therefore be regarded as not representative for MBR operation.

9.5. Relation with Operation of MBR installation

By applying filtration characterisation as described in *Chapter 7 Application at three MBR pilot plants* a relation is presumed between the phenomena in the pilot and those measured in the filtration characterisation unit. This paragraph will go deeper into this relation.

The follow up of the research described in this thesis has already paid a lot of attention to correlate results from filtration characterisation experiments to the performance of the respective full-scale installation (MBR at Varsseveld wwtp, the Netherlands). There was a very good agreement between the filtration characterisation experiments and the development of long-term permeability of the system. Results will be published by Delft University of Technology in the course of 2006.

9.5.1. Fouling intensification

When filtering activated sludge from different pilot plants in the filtration characterisation unit, it became clear that each plant had its own characteristic filtration curve. Under standard conditions there was a certain ranking in terms of ΔR_{20}. By increasing the flux this ranking did not change, only absolute differences became bigger, see §7.3, Figure 7-6.

This probably means that working at higher fluxes only intensifies processes occurring at lower fluxes. In that sense filtration characterisation could be seen as a way to 'speed up time', in order to see what might happen if no further action is taken. In practice filtration is always interrupted by a periodic backwashing, a relaxation time and maintenance cleanings

Filtration characterisation could then possibly help to identify to which extent the membrane filtration in the pilot installation is functioning properly. In the case of Hilversum for example, the conclusion could be that the effective membrane area is much lower than the installed membrane area. Optimising aeration efficiency or developing other ways of membrane cleaning could be carried in order to make use of the good filterability of this sludge.

For other installations, depending on the experiences with peak flows, new values for flux set points could be defined according to the results of filtration characterisation.

9.5.2. ΔR_{20} as a Predictive Value

It can be hypothesised that long term fouling phenomena in MBR installations are dependent on the total produced volume of permeate (Wintgens *et al.*, 2003). The water phase of the activated sludge contains low concentrations of foulants

that will find their way to the membrane and accumulate there. In the course of weeks or months, these substances will cause a long-term permeability decline. This is partly verified by observations described in the STOWA report 2004-28 (STOWA, 2004) where a logarithmic relation between total permeated volume and membrane permeability is described. Howell and co-workers describe experiments that indicate a strong relationship between residual fouling and cross flow velocity (Howell *et al*, 2004). Each decrease in cross flow velocity (gas flow rate) results in an increased residual fouling, see Figure 9-1. Shifting the operational parameters towards values where fouling is more severe will speed up the long-term process.

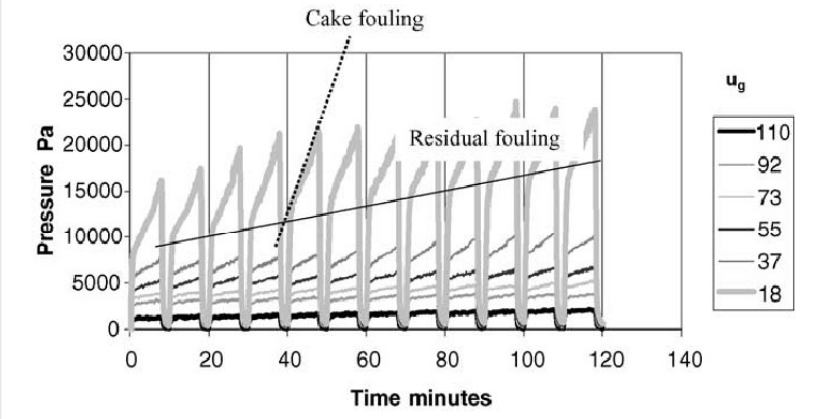

Figure 9-1 *Multiple Cycles of intermittent permeation at an MLVSS of 17.42 g/L, J=24 L/m² h and different gas flow velocities u_g (18,37,55,73,92,110 mm/s)The slopes of the 30-40 min cake fouling line and the residual fouling lines are shown for the lowest gas flow rate (from: Howell et al., 2004). Membrane: Kubota; activated sludge treating a synthetical wastewater*

In the case of working at an increased permeate flux, substances with a fouling potential are transported to the membrane in much higher quantities compared to normal operation. Such an experiment gives information about the presence of these substances, and provides indications to define a maintenance regime.
In this sense filtration characterisation could act as a warning system, since it simulates the uninterrupted filtration of a large volume of permeate. If at a

certain moment ΔR_{20} increases suddenly, this indicates an increased presence of these substances in the mixed liquor.

9.5.3. Long term Permeability development

With few exceptions, all MBR installations will face a certain decrease in system permeability as a result of ongoing fouling processes. Also membrane ageing may play a role but this process is considered to be beyond the scope of this thesis. The long-term permeability decrease is probably related to the filtration characterisation as ΔR_{20}. If the filtration characterisation is carried out in a representative period, it should be possible to correlate it to the actual MBR performance.

From pilot experiences at Beverwijk wwtp some examples were obtained from the long-term permeability decline (van der Roest *et al*, 2002). The Kubota system for example showed an average permeability decline of 415 L/m² ·h ·bar over a period of 324 days, about 1.3 L/m² ·h ·bar ·day. For the Zenon installation at the same location a decrease of 235 L/m² ·h ·bar was observed over a period of 246 days, a rate of 0.96 L/m² ·h ·bar ·day. In the case of Beverwijk there were large differences in the rate of permeability decrease. There were also extended periods in which permeability did not change, or even increased. For the Mitsubishi system a more or less representative period showed a decrease of 275 L/m² ·h ·bar over a period of 126 days: 2.2 L/m² ·h ·bar ·day.

MBR pilot at Maasbommel wwtp

The MBR pilot installation at Maasbommel was tested two times, first in February 2004 and secondly in July 2004. In February the permeability decline was about 0.4 L/m² ·h ·bar ·day, at a permeability of around 135 L/m² ·h ·bar. In July the rate of permeability decline was about 0.6 L/m² ·h ·bar ·day (STOWA, 2004-28; Figure 17 p. 27). Although the rate was higher in July, absolute permeability was higher, about 160 L/m² ·h ·bar. For the evaluation in this chapter only the values from the second period can be used.

MBR pilot at Beverwijk

The MBR pilot at Beverwijk wwtp started with a permeability of more than 400 L/m² ·h ·bar (Bentem, 2004). In the first period of about 80 days the rate of permeability decline was about 1.3 L/m² ·h ·bar ·day. Then, after a period of technical problems, permeability stabilised at about 225 L/m² ·h ·bar, with peaks up to 275 L/m² ·h ·bar.

MBR pilot at Hilversum

During the period in which measurements were carried out at wwtp Hilversum, the permeability increased with around 3 L/m² ·h ·bar ·day. This period lasted for about 20 days.

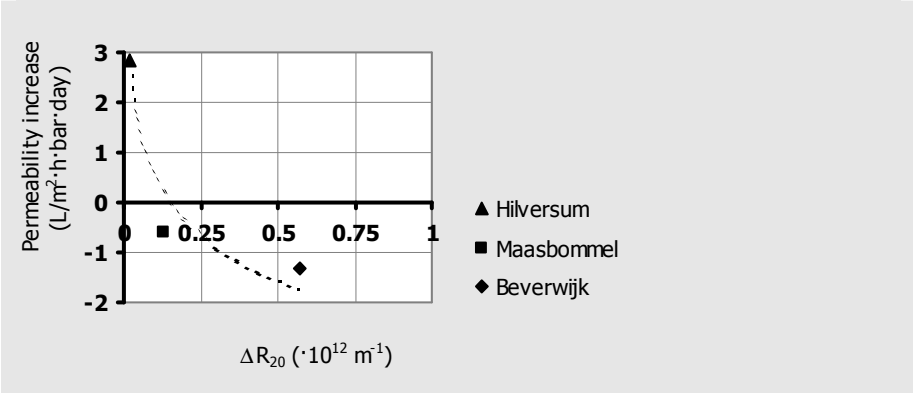

Figure 9-2 Differences in filterability of activated sludge from three MBR installations;

The average values of ΔR_{20} at the three pilot installations are plotted against the system permeability increase in the period in which the measurements were carried out, see Figure 9-2.

9.5.4. Preliminary Conclusions

The filtration characterisation method turns out to quantify the long-term filterability development of the MBR system where the sludge is sampled. This conclusion is sustained by measurements referred to earlier where filtration characterisation was carried out four or five time in the course of a year. Within this period ΔR_{20} varied dramatically, and afterwards it turned out that these

variations very well corresponded to the ups and downs in the operation of the MBR plant.
The effect of aeration for membrane cleaning in MBR is limited. In practice almost all MBR systems apply low permeate fluxes, at least when compared to the 80 L/m² ·h used in this thesis. Unfortunately these low fluxes are necessary, because an increase in permeate flow leads to a decrease in system permeability. In other words, the required TMP to sustain the flux increases with time: fouling takes place. In many cases membrane permeation is intermittently stopped in order to have the bubble aeration and sometimes membrane movement remove the formed cake layer.

The Hilversum case seems to point out that a) the bubble aeration was insufficient and/or b) the membrane was partially clogged. During the filtration characterisation experiments the critical flux was higher than 80 L/m² ·h. In the installation however, increasing the usual permeate flux of 12 L/m² ·h to values up to 18 L/m² ·h already leads to a permeability decrease. The applied membrane aeration is not able to avoid the increase of filtration resistance. This effect is most likely exaggerated by the effect of exceeding the ´local critical flux´. Due to partial clogging of the membrane surface the remaining membrane area has to produce a higher flux.

9.6. Substances influencing filtration behaviour

The applied colorimetric analysis of EPS in the water phase of activated sludge and the permeate of filtration characterisation does not explain the differences in filtration behaviour.
The concentration in the water phase cannot be correlated to the filtration behaviour. The same holds for the calculated retained EPS concentrations.
Firstly, the absolute concentration values are of the same order for the three tested sludges, and the differences result in a ranking which is different from the results obtained with filtration characterisation.
This may have several causes (see Figure 9-3):

1. Proteins and polysaccharides are not involved in the membrane fouling process. Of course, the subsequent question must be which other substances

are available? An interesting option could be the group of humic substances, although also for this group of substances contradictory results can be found in literature.

2. Proteins and polysaccharides may be the only substances involved in the fouling process.
3. Proteins and polysaccharides are among other substances involved in the membrane fouling process.

For the cases 2. and 3. the following two options are available:

a) Some of the analysed proteins and polysaccharides present in the water phase are involved in the fouling process. It can be imagined that a specific group of proteins or polysaccharides is detrimental for crossflow filtration of activated sludge. A more or less constant concentration of EPS may consist of changing types of substances with varying concentrations.

b) The analysed proteins and polysaccharides in the water phase are only part of the total amount of proteins and polysaccharides involved in membrane fouling. Maybe the cut-off of the filter used for separating the water phase from the activated sludge was too low.

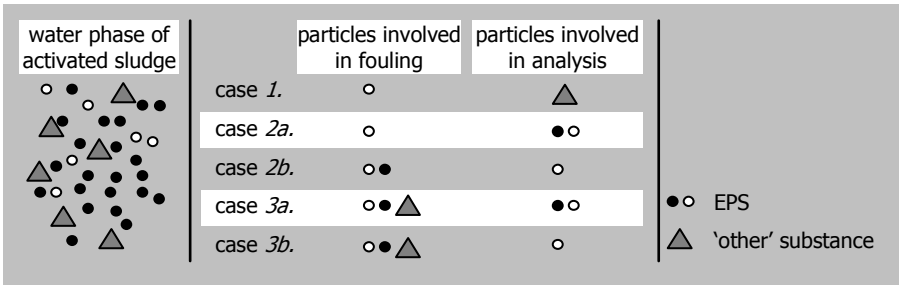

Figure 9-3 *Schematic overview of water phase of activated sludge and five possibilities for analysing foulants*

Unfortunately, the measurement method applied in this thesis cannot discern between these four options. Other techniques, such as chromatography, particle size analysis may throw light on this issue.

The size of a particle is not the only characteristic determining its involvement in fouling. The reasoning only works out one way: a particle that is too large will not be involved in membrane fouling. A particle small enough to enter the boundary layer will only under certain conditions take part in the fouling process. Other properties of the particles/substances are also decisive for its inclusion in the fouling process. Among others, hydrophilicity for example may play a role in this respect.

9.7. Recommendations for further research

The filtration characterisation method can be applied in several ways. It can be used to investigate the causes of membrane fouling in MBR processes. It can also be used to

9.7.1. Phenomenological approach

The application of the filtration characterisation method should be extended to other installations. In this way a matrix of ΔR_{20} values and installation characteristics will be obtained. This phenomenological approach will give information on the influence on ΔR_{20} of choices in design and operation of MBR installations. To this aim, the installations must of course be described uniformly. Parameters to be included in the description are:

- biological characteristics; division of the different compartments, circulation flows, biological loading rate, MLSS concentration, influent composition, etc.
- operational characteristics: operation of the membrane separation step and biology.
- 'hardware' characteristics: membrane characteristics, type of pumping devices, type of pre-treatment, size of compartments, etc.

9.7.2. Pin-pointing foulants

The exact determination of the foulants in activated sludge filtration can be addressed by performing additional analysis of the water phase of the activated sludge. Several approaches can be followed here:

- by using very small batches of activated sludge and filtering them for a long time, changes in the water phase may be measured. Also the possibilities for analysis of the formed cake layer should be investigated in order to come as close as possible to the actual foulants. This can be done by very specific cleaning methods (e.g. enzymatic cleaning, as demonstrated by Te Poele (2005)) or by resuspending the cake layer by rinsing it with clean water or removing it with a back flush and analysing the produced water.
- 'spiking' of the biomass with 'suspected' substances and subsequently filtrating it. In this way the interaction between the substance, the biomass and the fouling behaviour is as close to reality as possible.
- other analysis techniques for the water phase, such as particle size analysis, size exclusion chromatography, broadband UV analysis, etc.

9.7.3. Modelling filtration behaviour

The occurring mechanisms at the membrane in terms of particle transport, cake layer formation, viscosity-effects, etc. can at the moment only be described in general terms. The activated sludge broth is very complex and it seems worthwhile to endeavour constructing a model in which physical-chemical processes are described as well as the biological processes determining filtration behaviour.

9.7.4. Optimising MBR systems

The filtration characterisation installation can also be applied for system optimisation. By extending the feed vessel towards a simplified model of the bioreactor (e.g. with intermittent aeration, influent addition, etc.) the sensitivity of the system may be assessed at a small scale. Also possible optimisation measures may be tested before application at full scale.

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Summary

Over the past decades, the application of Membrane BioReactors for the treatment of municipal wastewater has drawn a lot of attention. Most important reason for this interest is the expected increase in effluent quality, achieved by the membrane. Furthermore, a reduction of footprint can be achieved by the replacement of the secondary clarifier by membranes and the higher MLSS concentration compared to conventional activated sludge systems.

One of the main concerns when operating an MBR is the performance of the membranes. Due to the nature and composition of the mixed liquor, membrane fouling may occur. The extent to which fouling occurs varies with time, depending on inflow conditions, membrane configuration, membrane operation, etc. This thesis describes the development of a tool to quantify the filterability of activated sludge.

A filtration apparatus was constructed enabling the cross flow filtration of activated sludge in a single tubular membrane. The conditions under which filtration takes place can be controlled in order to accurately measure the characteristics of the filtration curve. By doing short-term filtration tests of maximum 1 hour, a 'finger print' of the activated sludge can be made.
Subsequently, a measuring protocol was set up, involving clean water measurements and three steps of membrane cleaning after a filtration experiment.

A useful parameter to characterise filterability is the resistance additional to the clean membrane resistance, after filtering a fixed volume (20 L per square meter of membrane) under standard conditions (constant flux of 80 $L/m^2 \cdot h$ and a cross flow velocity of 1 m/s), expressed as $\Delta R_{20,}$ [m^{-1}].

The filtration characterisation method was applied in two ways during the research:

1. Comparison of pilot installations in terms of filterability.

Three pilot installations were visited and during a period of about 14 days the filtration characterisation unit assessed the filterability of the biomass in the respective MBR pilot. The results show that each pilot installation promotes its own activated sludge with a wide range of filterabilities. One of the tested sludges incurred no fouling at all, when filtered under standard conditions. This indicates filtration in the so-called sub-critical mode.
Furthermore, changes in operating conditions (interruption of influent flow) could be measured as a change in filterability.

The values of ΔR_{20} could be correlated to the longer-term development of system permeability of the pilot installations where the activated sludge was sampled. In the case where ΔR_{20} was 0 m^{-1}, the system permeability increased.

2. Assessment of factors influencing sludge filterability.

In order to assess factors influencing the filterability of activated sludge, experiments were carried out in which the filterability was manipulated. Addition of an easily degradable substrate to the activated sludge resulted in an increase in ΔR_{20}.

Ultimately, experiments were performed in order to pinpoint specific (groups of) substances involved in the membrane fouling process.
To this aim, the concentration of extracellular polymeric substance was determined in the water phase of the activated sludge. Protein concentrations and polysaccharides were analysed. It turned out that the measured concentrations could not be correlated to the fouling phenomena observed during the filtration tests.

Samenvatting

De toepassing van membraanbioreactoren voor de zuivering van stedelijk afvalwater heeft in de afgelopen 10 sterk in de belangstelling gestaan. De belangrijkste reden hiervoor is dat verwacht werd dat met deze techniek een grote sprong gemaakt kon worden in te behalen effluentkwaliteit. Daarnaast kan MBR kleiner gebouwd worden dan met het conventionele actiefslibsysteem, enerzijds door een hoger slibstofgehalte in de actiefslibtank en het feit dat de grote nabezinktanks vervangen kunnen worden door de kleinere membraanscheidingsstap.

Eén van de belangrijkste aandachtspunten bij de bedrijfsvoering van een MBR is de membraanscheidingsstap. Vanwege de samenstelling en eigenschappen van het actiefslibmengsel kan membraanvervuiling ontstaan. De mate waarin dit gebeurt varieert met tijd, afhankelijk van aanvoercondities (hoeveelheid en samenstelling), membraanconfiguratie, bedrijfsvoering van de membranen, etc. Dit proefschrift beschrijft hoe een meetmethode en een meetinstallatie zijn ontwikkeld om de filtreerbaarheid van actiefslib te kwantificeren.

Een filtratie-opstelling werd ontworpen en gebouwd waarmee actiefslib in de crossflow modus gefiltreerd kan worden middels een enkelvoudig tubulair membraan. De omstandigheden waaronder filtratie plaatsvindt kunnen worden gecontroleerd en nauwkeurig gemeten zodat een representatieve filtratiecurve verkregen wordt. Met kortdurende experimenten van maximaal 1 uur kan zodoende een 'vingerafdruk' van het actiefslib gemaakt worden.
Vervolgens is een meetprotocol opgesteld, samengesteld uit schoonwaterfluxmetingen en drie reinigingsstappen rondom het eigenlijke filtratie-experiment.

Een waardevolle parameter waarmee de filtreerbaarheid gekarakteriseerd kan worden, blijkt de additionele weerstand (t.o.v. de schoon-membraanweerstand) te zijn, na filtratie van een vast volume (20 l permeaat per vierkante meter membraanoppervlak) onder vaste condities, zijnde een constante permeaatflux

van 80 l/m^2·h en een crossflow snelheid van 1 m/s. Deze additionele weerstand wordt genoteerd als ΔR_{20} en heeft de eenheid van hydraulische weerstand, [m^{-1}].

De filtratie-karakteriseringsmethode is in dit onderzoek op twee manieren toegepast:

1. Voor de vergelijking van een drietal pilot installaties.

Een drietal pilot-installaties is bezocht en gedurende een periode van 14 dagen werd de filtreerbaarheid van het actiefslib uit deze installaties gemeten met de filtratie-karakteriseringsmethode. De resultaten laten zien dat in elke pilot-installatie een 'eigen' actiefslib ontstaat met een specifieke filtreerbaarheid. De verschillen in filtreerbaarheid tussen de verschillende installaties zijn aanzienlijk. Het slib uit één van de installaties vertoonde zelfs helemaal geen vervuiling gedurende het standaardexperiment.
Verder kon aangetoond worden dat veranderingen in de bedrijfsvoering van de pilot gemeten kunnen worden als veranderingen in filtreerbaarheid.

De gemeten waarden van ΔR_{20} bleken te corresponderen met de lange termijn ontwikkeling van de permeabiliteit van de betreffende installaties. In het geval van het slib dat geen vervuiling veroorzaakte, bleek de permeabiliteit van de pilot-installatie stijgend geweest te zijn in de betreffende periode.

2. Onderzoek naar factoren die de filtreerbaarheid van actiefslib beïnvloeden.

Om inzicht te verkrijgen in de factoren die de filtreerbaarheid van actiefslib bepalen zijn experimenten uitgevoerd waarin de filtreerbaarheid werd gemanipuleerd. Door toevoeging van een gemakkelijk afbreekbaar substraat bleek de filtreerbaarheid af te nemen, ΔR_{20} nam toe.
Uiteindelijk zijn experimenten uitgevoerd met als doel het vaststellen van bepaalde substanties die betrokken zijn bij het membraanvervuilingsproces.
Hiertoe zijn de concentraties extracellulaire polymere substanties bepaald in de waterfase van het actiefslib. Proteïne- en polysaccharideconcentraties werden geanalyseerd. Het bleek dat de gemeten concentraties niet te correleren zijn aan de vervuilingsverschijnselen zoals die in de filtratie-experimenten zijn waargenomen.

Nomenclature

Symbols

η	dynamic viscosity [Pa·s]
J	permeate flux [L/m^2·h]
u_{cr}	crossflow velocity [m/s]
V	volume [m^3]
P	permeability [L/m^2·h·bar]
Q	flowrate [m^3/h] or [m^3/s]
R	rejection factor [-]
R	filtration resistance [m^{-1}]
τ_m	shear stress along membrane [Pa]

Abbreviations

BOD	Biochemical Oxygen Demand, see 2.2.2
CP	concentration polarisation
COD	Chemical Oxygen Demand, see 2.2.2
CWF	Clean water flux
DWF	Dry Weather Flow
EPS	Extracellular Polymeric Substances, see 3.4.2
F/M	ratio of Food to Microorganisms, see 2.2.2
J	Permeate flux
MBR	Membrane BioReactor, see 2.5
MF	Microfiltration, see 2.3
MLSS	Mixed Liquor Suspended Solids, see 2.2.2
MWCO	Molecular weight cut-off
NF	Nanofiltration, see 2.3
OR	Surface overflow rate
p.e.	population equivalent
PSD	Particle size distribution
PVDF	polyvinylidenfluoride (membrane material)
RWF	Rain Weather Flow

SLR	Solids loading rate
SMP	Soluble Microbial Products
SRT	Sludge Retention Time, see 2.2.2
SVI	Sludge Volume Index, see 2.2.2
TMP	Trans Membrane Pressure, see 2.3.1
TSS	Total Suspended Solids, see 2.2.2
UF	Ultrafiltration, see 2.3
WWTP	Wastewater treatment plant

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2005

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Curriculum Vitae

Herman Evenblij was born on the 21st of October 1972 in Rotterdam, the Netherlands. After finishing secondary school at the Guido de Brès (VWO) he started the MSc.-study Civil Engineering at Delft University of Technology.
He finished his study at the section of Sanitary Engineering (department Watermanagement) in 1999 with a master thesis, entitled 'Preliminary study into membrane filtration of raw wastewater at wwtp Bennekom'.
From October 1999 until May 2000 he worked as a teacher Chemistry and Physics at the secondary school 'Farel College' in Ridderkerk, the Netherlands.
In the Summer of 2000 he started as a researcher at Delft Univsersity of Technology. He worked in the research group of prof. ir. J.H.J.M. van der Graaf at the department of Sanitary Engineering, within the project ´Filtration Characterisation in MBR´, which was funded by DHV Water bv. The results of this research are described in this thesis.

Since February 2005 he is employed as specialist MBR and water treatment at the engineering and consultancy firm Witteveen+Bos in Deventer, the Netherlands.

Appendices

Appendix I Municipal MBRs in Europe

By Renze van Houten; updated until August 15, 2004. From: www.waterforum.net

Year	Location	Capacity p.e.	Membrane manufacturer	Flow m^3/h
1991	Kingston (UK)	<1,000	Kubota	5
1998	Porlock (UK)	4,000	Kubota	80
1998	Erlangen (Ger)		Kubota	8
1999	Rödingen (Ger)	3,000	Zenon	100
1999	Büchel (Ger)		Kubota	40
2000	Markranstädt (Ger)	12,000	Zenon	150
2000	Swanage (UK)	23,000	Kubota	530
2001	Campbeltown (UK)	24,000	Zenon	110
2001	Knautnaundorf (Ger)	900	Huber/VRM	
2001	Altenberge (Ger)	1,000	Huber/VRM	
2001	Moneyreagh (IRE)		Kubota	24
2002	South Wraxall Sewage (UK)		Kubota	23
2002	Schwägalp (CH)	780	Huber/VRM	
2002	St. Peter ob Judenburg. (A)	1,500	Mitsubishi	
2002	Lowestoft (UK)	46,000	Zenon	300
2002	Wessex Water (UK)		Kubota	290
2002	Brescia (I)	46,000	Zenon	1580
2002	Llanranog (UK)		Kubota	12
2002	Welsh Water (UK)		Kubota	15
2002	Skipsea (UK)		Kubota	55
2002	Gairloch (UK)		Kubota	60
2002	Cromarty (UK)		Kubota	43
2002	Greyabbey (IRE)		Kubota	49
2003	Kircubbin		Kubota	72
2003	Schilde (B)	20,000	Zenon	350
2003	Gardenstown (UK)		Kubota	30
2003	Longbridge (UK)		Kubota	65
2003	Lynmouth (UK)		Kubota	68
2003	Merkendorf (Ger)		Kubota	8
2003	St. Anna Alfaeda (I)		Kubota	12
2003	Velo Veronese (I)		Kubota	13
2003	Erbezzo (I)		Kubota	12
2003	Revore Veronese (I)		Kubota	19
2004	Varsseveld (NL)	23,000	Zenon	755
2004	Mohnheim (Ger)	9,700	Zenon	75
2004	Seelscheidt (Ger)	11,000	Kubota	355

2004	Waldmössingen (Ger)	2,600	Zenon	90
2004	Kaarst-Nordkanal (Ger)	80,000	Zenon	1850
2004	Buxton (UK)		Zenon	450
2004	La Bisbal (I)		Zenon	135
2004	Cardigan (UK)		Kubota	360
2004	Riells I Viabrea (E)		Kubota	215
2004	Fairbourne (UK)		Kubota	27
2004	Severn Trent (UK)		Kubota	94
2004	San Martino di Castrozza (I)		Kubota	42
2004	Aghalee & Aghagallon (IRE)		Kubota	60
2004	Guilvinex (F)		Kubota	110
2004	Heenvliet (NL)	2,500	Seghers/Keppel	100
2004	Ootmarsum (NL)	14,000	?	650
200?	Hilversum, (NL)	91,000	?	1400

Appendix II Industrial MBRs in the Netherlands

By Renze van Houten; updated until January 30, 2004. From: www.waterforum.net

Year	Location	Manufacturer	Flow m^3/h
1996	Landfill, Middenmeer	Grontmij/Stork	5
1997	Landfill, Rotterdam	Grontmij/Stork	5
1997	Tankcleaning, Hengelo	Septo Biotechniek	3
1997	Landfill, Amersfoort	Zenon	5
1998	Agricultural, Dronten	Triqua	7.5
1999	Broek op Langedijk, Agricultural	Triqua	0.15
1999	Destruction, Bergum	Zenon	40
1999	Marine wastewater, Den Helder	Triqua	5
1999	Tankcleaning, Alblasserdam	RWB/Mitsubishi	1.2
2000	Tankcleaning, Rijssen	RWB/Mitsubishi	2.5
2000	Paper Industry, Apeldoorn	Triqua	10
2000	Pharmaceutical, Oss	Triqua	16
2001	Solid Waste Treatment, Weert	Triqua	1,5
2001	Tankcleaning, Veendam	Nijhuis	12
2002	Food industry	Kubota	2
2002	Landfill	Kubota	10
2003	Solid Waste Treatment, Moerdijk	Self operated/X-Flow	50
2003	Destruction, Son	Zenon	80
2004	Tankcleaning, Botlek	RWB/Mitsubishi	7.5
2004	Chemical, Tilburg	Seghers Keppel	35

Appendix III Membrane specifications F5385

COMPACT MEMBRANE PRODUCTS

X-Flow COMPACT membranes are only available as complete membrane modules. A product code consists of a combination of the membrane type (Table 1) and the module type (Table 2).

Table 1	COMPACT MEMBRANES (5.2, 8.0 mm)	
Duty	UF	
Membrane type	F4385	F5385
Membrane material	PVDF	PVDF
Internal Diameter [mm]	5.2	8.0
Clean water flux in module at 25°C [l/m^2.h at 100 kPa]	> 1000	> 750
Pore size [nm]	30	30
Max.pres. [kPa]	500	500
Max.temp.(*) [°C]	70	70
pH feed	2-10	2-10
PVDF = polyvinylidene fluoride		

(*) *Maximum temperature of operation is determined by the module used. See module technical datasheet for more information.*

NOTES:

1. For further information, see membrane and module technical datasheets.
2. Final maximum operating limits are determined by the lowest values of the membrane and module pressure and temperature specifications.
3. It is not advisable to operate a membrane module at any combination of the maximum limits of pH, concentration, pressure, or temperature, during cleaning or production; doing so will severely influence the membrane lifetime.
4. The product code is composed as follows (for example):
   - 3 meter, 8 inch GFR housing, radial permeate outlet
   - PVDF ultrafiltration membrane, 5.2 mm

   gives:
   - 38GRH/F4385

150704

COMP-ALL-0429
(replaces: COMP-ALL-0308)

Table 2	COMPACT PRODUCTS (5.2, 8.0 mm)				
Module type	Length [m]	Diameter [inch]	Membrane ID [mm]	Membrane area [m^2]	Permeate outlet
33GE (*)	3	3	5.2	5.1	radial
33GE (*)	3	3	8.0	4.0	radial
38PRV	3	8	5.2	29	radial
38GRH (*)	3	8	5.2	33	radial
38GRH (*)	3	8	8.0	27	radial
PRV : PVC			GE/GRH : glass fibre reinforced epoxy		

(*) *33GE and 38GRH, also available as refill*

The maximum allowable temperature for a PVC module housing is 40°C, and 70°C for a glass fibre reinforced module.

For more information please write or call to:

X-Flow B.V.
P.O.Box 739
7500 AS Enschede
The Netherlands

Phone: + 31 (0)53 4287350
Fax: + 31 (0)53 4287351
E-mail: info@xflow.nl
Web site: www.x-flow.com

COMP-ALL-0429

Appendix IV Data sheet pressure transmitters

PRESSURE TRANSMITTER
- INDUSTRY DESIGN -

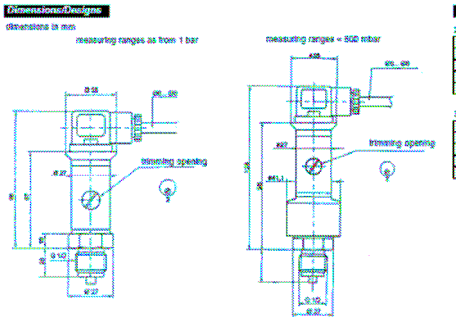
TYPE SERIES
CB3010

Design Features

- measuring ranges 0...250 mbar up to 0...600 bar
- measuring system ceramic cell
- signal output: 4...20 mA, 2-wire circuitry
  0...20 mA, 3-wire circuitry
  0...10 V, 3-wire circuitry
- easily zero-point adjustment
- stainless steel case, degree of protection IP 65
- encapsulated electronics
- process temperature up to 100 °C
- stainless steel process connection

Dimensions/Designs

dimensions in mm



Connection diagram

Application

The pressure transmitter is fitted with a ceramic measuring cell as sensor. The measuring signal is converted by the electronics to an impressed current signal of 4...20 mA (optional 0...20 mA or 0...10 V). Due to their robust design, these transmitters are suited for general industrial applications. The process temperature may reach 100 °C.

Design and Function

The electronics is encapsulated to protect it against moisture and vibrations. Zero-point can be adjusted with an internal trimming potentiometer. The potentiometer may be accessed from outside by loosening a casing screw. The output is available in 2- or 3-wire circuitry. Pressure is compensated through an opening in the top of the case and the connection plug.

Mounting and installation instructions

The installation position has no effect on the measuring procedure. However, it is recommended that the transmitter be protected against all forms of stressing from its service environment (vibrations, temperature, corrosion).
The adjustment for the zero-point is situated behind the casing screw. The potentiometer can be accessed through the encapsulation by undoing the screw.

Order Details - please give additional specifications for models not listed -

Pressure transmitter - industry design -		CB3010	
measuring range	per table		
process connection*	G 1/2 (standard)		K1010
	G 1/4		K1012
	1/2 NPT		K1030
	1/4 NPT		K1032
additional features (to be indicated in case of need, only)			
output signal	0...20 mA, 3-wire		H3
	0...10 V, 3-wire		H4
order code (example):		CB3010	A1055	K1010	

standard measuring ranges		
measuring range		order code
0...250	mbar	A1010
0...400	mbar	A1011
0...600	mbar	A1012
0...1	bar	A1083
0...1.6	bar	A1084
0...2.5	bar	A1085
0...4	bar	A1086
0...6	bar	A1087
0...10	bar	A1088
0...10	bar	A1089
0...20	bar	A1090
0...40	bar	A1091
0...60	bar	A1043
0...100	bar	A1093
0...160	bar	A1094
0...250	bar	A1095
0...400	bar	A1096
0...600	bar	A1098
-1...0	bar	A1066
-1...0.6	bar	A1067
-1...1.5	bar	A1068
-1...3	bar	A1069
-1...5	bar	A1070
-1...9	bar	A1071
-1...15	bar	A1072

* further process connections upon request

Technical Data

Case design
material no.: 1.4404
internal trimming potentiometer for zero-point and span, encapsulated electronics, vent port in the case

Electrical connection
right angle plug per DIN 43650
further connections upon request

Degree of protection
IP 65

Process connection
material no.: 1.4404, G 1/2

Wetted parts
ceramic, stainless steel and NBR-sealing washer

Measuring system
ceramic cell

Temperature ranges
- process temperature -25...+100 °C
  with max. 50 °C ambient temperature
- storage temperature -40...+85 °C
- allowed ambient temperature -25...85 °C
- compensated temperature range -10...55 °C

Temperature effect
on zero point: < 0.25 % f.s./10 K
on span: < 0.15 % f.s./10 K

Auxiliary power supply

- nominal voltage	24 V DC
- function range	11...40 V DC
- max. allowed rated span	40 V DC
with signal output 0...10 V:	
- function range	14...40 V DC

Signal output
- 4...20 mA, 2-wire circuitry
- 0...20 mA, 3-wire circuitry
- 0...10 V, 3-wire circuitry

Current limitation in the output signal
at 110 % of the pressure range

Measuring ranges
see order details

Adjusting range
zero point ± 10 %

Charateristic curve deviation
(linearity, hysteresis, repeatability)
< 0.5 % f.s. (fixed point adjustment)

Reaction time
< 3 ms

Load
- R_L max.= $\frac{\text{Vsers}-11}{0.02}$
with signal output 0...10 V:
- > 2.5 kOhm

Weights
standard housing: approx. 300 g

EMC-test
noise trimming as per EN 50082 part 1 and 2

Vibration resistance according to IEC 68-2-6

- amplitude	1.5 mm at 10-55 Hz
- acceleration	20 g at 55-2.000 Hz

Mechanical shock loading according to IEC 68-2-32
- 25 impact shocks from 1 m height

Information on other models upon request or see order details