Filterability Assessment of Membrane Bioreactors at European Scale

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Proefschrift

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Summary

Due to the implementation of a membrane stage, MBR technology can ensure superior effluent quality and footprint reduction compared to the conventional activated sludge process. However, even if the development of the immersed membrane technology succeeded to reduce significantly MBR operational costs, fouling mitigation costs still hampered further market development.

The main contributors of fouling still need to be investigated and more accurately quantified in order to develop efficient counter measures and fouling prevention protocols. The measurements of filterability can significantly contribute to these matters. Filterability measurements can be used to ascertain whether a permeability decrease should be attributed to poor activated sludge filterability or inadequate operations of the filtration process. A better understanding of the mechanisms involved in the filtration process can then be achieved. However, the lack of standard methods for the activated sludge filterability remains a limitation of such characterisation. A large quantity of methods has been developed by different research groups making the reliable comparisons of their results difficult. Therefore, general conclusions cannot be formulated.

Due to its participation to two broad European projects, namely EUROMBRA and MBR-Train, Delft University of Technology decided to organise a large filterability measurement campaign at a European scale.

Pilot and full-scale MBR plants of a large number of partners (Company, research institute and university) were investigated using the Delft Filtration Characterisation method, i.e. a standardize filterability test called the DFCm. Reliable data concerning filterability of pilot and full-scale MBR plants have been collected under the same hydraulic circumstances. Results coming from each research group could then be relevantly and accurately compared. Furthermore, a set of analyses was performed in combination with each filterability measurement. Design, operational and membrane performance data were also collected for each MBR plant.

The DFCm proved to be in practice *a user-friendly, quick and accurate* tool for activated sludge characterisation. Results obtained during the measurement campaign were *consistent* with the plant operations, *reliable* and *reproducible* along the experimental periods. Compared to other filterability characterisation methods, the DFCm advantages are its extremely well-defined and *well-controlled protocol* and its short term duration allowing dynamical monitoring of the activated sludge filterability of the MBR plants.

Due to the uniqueness of each MBR plant, significant differences in filterability had been found between plants. Large fluctuations in filterability were observed during this research and were partly explained by different factors. A classification of importance of the effect of several parameters on filterability was formulated. Firstly, the impacts of uncontrolled conditions and wastewater temperature can be noticed:

- The *feedwater* quality is likely to be the *dominant factor* in terms of activated sludge filterability. Difficult wastewater, and stress on the activated sludge and foam occurrence have a strong influence on the activated sludge filterability.
- The *seasonal fluctuations* and the *temperature* of the wastewater also have a *significant influence* on activated sludge filterability. A correlation was demonstrated statistically between filterability and temperature in full-scale plant applications. The main cause for the filterability deterioration during the winter time is likely to be a *submicron particle release* occurring due to poor flocculation conditions under *low temperature conditions*. In the same way, the main reason for the improvement of the activated sludge filterability during the summer time is likely to be a reduction of the sub-micron particle concentration in the free water due to the entrapment of these particles in the floc network. This strong floc network is likely to be due to a better flocculation state of the activated sludge under warm temperature conditions.

Operating conditions and MBR design parameters can also be considered factors affecting activated sludge filterability:

- Under low recirculation ratio conditions, activated sludge upconcentration was observed within the membrane tank. This activated sludge upconcentration resulted in a significant filterability improvement. The *activated sludge upconcentration* is likely to be a *process* responsible for filterability improvement under these specific operating conditions.
- Low activated sludge loading systems showed a better filterability than high loading systems. Therefore, *activated sludge loading* influences the activated sludge filterability. However, it should be considered a fouling control parameter of *second order of influence* due to its relative effect on filterability compared to seasonal fluctuations and toxicity.

Some others parameters did not show significant correlation and impacts on activated sludge filterability:

- SMP did not show any significant correlation with activated sludge filterability. It is
 mostly due to the colorimetric methods chosen to quantify the SMP concentration.
 The conventional colorimetric methods should not be considered anymore
 appropriate for MBR membrane fouling investigations. New methods more orientated
 towards characterising specific properties like size should be implemented to
 comprehend the degree of involvement of the SMP in the fouling process.
- Activated sludge apparent viscosity did not show any significant correlation with activated sludge filterability. Activated sludge filterability and MBR plant permeability were not affected by activated sludge apparent viscosity variations. It cannot be considered a relevant parameter to optimising membrane fouling control and membrane performances of current, full-scale municipal MBR applications. However, it still should be considered a predominant factor in regard to clogging issues.

• The membrane configurations of the immersed MBR plants did not affect the activated sludge filterability. Activated sludge filterability is likely to be related with the biological process more than with the membrane filtration process. The differences in shear and hydraulic regimes promoted by different immersed configurations are not sufficient to significantly affect the activated sludge filterability.

Finally, interesting results were ascertained concerning the impact of the scaling-up of MBR plants on activated sludge filterability. Significant differences in terms of filterability could be observed between pilot and full-scale MBR plants. The differences in capacities (buffer), operating conditions, stress and the difficulty to maintain steady conditions lead to different behaviours in terms of activated sludge filterability in pilot-scale MBRs.

A general framework based on the results of the measurement campaign was formulated. The general framework showed that the activated sludge of an MBR plant does not significantly differ from a conventional activated sludge. Furthermore, proper flocculation is likely to be essential for proper MBR operations. As a consequence, conservative design ensuring proper flocculation should be implemented in MBR applications. Regarding fouling control, current solutions are based on flux enhancer additions and high turbulence promotions at the membrane wall. In their current states, both techniques are likely not to be effective at a reasonable cost. Therefore, new protocols should be tested based on the observation that filterability could be enhanced by a local activated sludge upconcentration in the membrane tank.

Recommendations were also formulated regarding the use of lab and pilot-scale tests and the design of full-scale MBR plants. Membrane costs remained one of the major bottlenecks of the MBR market development growth. In order to become competitive, membrane costs should drop or membrane flux production rate should increase significantly. Therefore, further efforts should be put into membrane material research in order to develop mass production membrane at low cost or/and high permeable membrane.

MBR pilot-scale research should be reconsidered. Significant differences in terms of filterability could be observed between pilot and full-scale MBR plants in our study. It is mostly due to differences in surrounding conditions and to the lack of redundancy which prevent continuous and stable operations at the pilot-scale. Therefore, feasibility tests at the pilot-scale before the building of a full-scale plant installation are likely not to be compulsory anymore. However, pilot-scale studies can still be useful in terms of operating condition optimisation. Rather than building a complete pilot-scale plant with its own specific biology, it is likely that a separate parallel membrane tank coupled to an existing full-scale MBR plant can bring more conclusive information in order to develop cost effective MBR operating modes.

Finally, an optimal use of the membrane surface area implemented can be achieved if the design of the MBR is based on the dry weather flow or for plants connected to a separate sewage network. The full potential of MBR applications can then be used for upgrading and retrofitting of existing wastewater treatment plants.

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1 Introduction

Sanitation is the single most important medical advance since 1840 (British Medical Journal survey, 2006)

1.1 Sanitation: State of the World

In November 2002, the United Nations Committee on Economic, Social and Cultural Rights affirmed that "access to adequate amounts of clean water for personal and domestic uses is a fundamental human right of all people" (United Nation website). One of the eight Millennium Development Goals (MDGs) is to halve the proportion of the population without sustainable access to safe drinking water and basic sanitation by 2015.

The latest coverage statistics show that the targets for the MDG concerning clean water should be reached. However, it might not be the case for sanitation (Figure 1). Two and half billion people -more than a third of the world's population- are still without access to proper sanitation, including 1.2 billion who have no facilities at all. In the developing world the lack of effective sanitation facilities is considered the primary cause of diseases linked to water. About 90 per cent of sewage and 70 per cent of industrial wastes in developing countries are discharged without treatment, deteriorating available water source quality. Therefore, new facilities are strongly needed. Current technologies but also new innovative and cost effective technological solutions need to be developed and implemented in order to tackle sanitation challenges and meet the MDG targets.



Figure 1: current state of the sanitation development in the world (UN website)

1.2 Membrane bioreactors: "catching two birds with one stone"

As presented in the previous section, new sanitation facilities need to be implemented. Even if current technologies are robust and allowed the production of a good quality effluent, there is still a need for innovation in the water sector, especially in term of water reuse. Membrane bioreactor process is considered one of the most promising technologies of this last decade. Associating a conventional activated sludge process and a membrane separation stage, membrane bioreactor technology allows a high sanitation quality as well as water reuse perspectives. The produced permeate by a membrane bioreactor can indeed be reused for agricultural or, after a last disinfection step, for drinking purposes. Therefore this innovative technology can be seen as a powerful tool to contribute to the MDG clean water and MDG sanitation target achievements. However, high total costs do not make membrane bioreactor technology easily applicable at this moment, especially in the developing world.

Regarding the potential of membrane bioreactor processes, the European Union has decided to finance three research programs dedicated to membrane bioreactors, namely EUROMBRA (Membrane bioreactor technology for advanced municipal wastewater treatment strategies: 4.2 million euro), AMEDEUS (Accelerate Membrane Development for Urban Sewage Purification: 5.9 million euro) and MBR-TRAIN (Marie-Curie Host Research Fellowship: 2.05 million euro) (MBR network website). The three projects were financed to promote the European MBR industry and to participate in the development and optimisation of the membrane bioreactor technology. The first goal of this project was to develop new alternatives to compete with the American and Japanese leaders of the MBR market. The next aim was to develop a common and competitive treatment alternative for future applications and thus contribute to some extents to the MDG achievements. This thesis is one of the contributions.

1.3 Problem statement, objectives and structure of this thesis

1.3.1. Problem statement and research approach

Due to the direct contact between the activated sludge and the membrane stage, fouling of the membrane cannot be completely avoided and results in high operational costs. A better understanding of the fouling phenomena is a crucial first step towards MBR optimisation and efficient membrane operations. Once the fouling mechanisms are better understood, preventing measures can be taken in order to set the best operating conditions in regard to fouling prevention and cost minimisation. Fouling was therefore intensively investigated by many research groups in the past decade. Their research focus essentially on the three main factors considered responsible for fouling:

- The membrane properties
- The membrane operation
- The activated sludge characteristics

However, due to the fact that each MBR set up is a unique combination of these three factors, general conclusions were rarely formulated. Depending on the scale of the experiments, the membrane type or the type of influent used in their research, authors reported contradictory results and no consensus could be found. Furthermore, short term fouling simulations under laboratory conditions were hardly representative or related with

the actual fouling mechanisms occurring in full-scale plants which consisted of short and long term components.

Another major drawback of MBR research is the *non-existence* of standard methods to quantify accurately the fouling rate. Each research groups developed their own peculiar methods. As a consequence, the comparison of results coming from different research groups became hardly feasible and hampered the development of practical general knowledge on MBR fouling.

Because all these previous aspects cannot be tackled at once, the research work performed during this thesis was based on several assumptions:

- Well-filterable activated sludge is fundamental to achieve cost effective and high membrane performances
- Research needs to be performed as closely as possible to real full-scale MBR operations
- A large set of data needs to be collected in order to obtain general conclusions
- An accurate activated sludge characteristic parameter needs to be implemented in each set of experiments in order to compare different activated sludge samples

A filtration test unit and an associated method (the Delft Filtration Characterisation method - DFCm) were developed by previous researchers to qualify activated sludge filterability (Evenblij, 2005, Geilvoet, 2010). Activated sludge samples were filtered in the DFCm under identical hydraulic circumstances and with similar initial membrane conditions. The fouling potential of different activated sludge samples could then be quantified and compared accurately. Due to its participation in two broad European projects, namely EUROMBRA and MBR-Train, Delft University of Technology decided to organise a large campaign of filterability measurements at a European scale.

Pilot and full-scale MBR plants of a large number of partners (Company, research institute and university) were investigated using the DFCm. Reliable data concerning filterability of pilot and full-scale MBR plants have been collected under the same hydraulic circumstances. A set of analyses was performed in combination with each filterability measurement. Design, operational and membrane performance data were also collected for each MBR plant.

Results coming from each plant can then be compared relevantly and accurately. Local differences in filterability could be analysed and compared in order to formulate more general conclusions.

1.3.2. Objectives

The main objective of this research work is to get a better understanding of design and operational parameters influencing activated sludge filterability, in order to get a step closer to optimum membrane performance conditions.

The research objectives described in this thesis can be divided in two parts. In the first place the filterability results collected during this research can be analysed *locally*. Local filterability variations observed can be analysed depending on their feedwater

characteristics and the operating conditions set in the MBR plants. The second step of this research is to *inter-link* the results collected at different MBR locations and subsequently to *generalise* the conclusions.

1.3.3. Thesis outline

A general background in wastewater treatment will be given in the literature review presented in *Chapter 2*. Fundamentals and conventional processes will be discussed firstly; membrane bioreactor technology basics will then be presented, with a special focus on fouling and filterability.

Chapter 3 will recapitulate the materials and methods used during this research work. The Delft Filtration Characterisation methods will be described from its first development till its recent assessment performed by previous researchers. The European DFCm tour and the analytical tools used will then be presented.

Chapter 4 will present the full set of data collected during this research work. Filterability variations within and between different experimental periods will be commented. The reliability of the data collected with the DFCm will be assessed in *Chapter 5*. The correlations with the plant configurations, the operating and surrounding conditions will be discussed in *Chapter 6*.

Chapter 7 will globally evaluate the local conclusions formulated in *Chapter* 6 and conclude this thesis, developing practical and useful recommendations for MBR endusers.

2 Literature Review

The basics concerning the activated sludge process will be briefly introduced in this chapter. Membrane technology and its application to the membrane bioreactor process will then be discussed in detail, followed by an updated state of the knowledge on membrane fouling in MBR. Finally, the different fouling and membrane performance indicators will be presented, with a special focus on filterability.

2.1 Fundamentals

The wastewater produced by a community is defined by Metcalf&Eddy (2003) as "a combination of the liquid or water carried wastes removed from residences, institutions, and commercial and industrial establishments, together with such groundwater, surface water, and stormwater as may be present". Due to the presence in wastewater of organic substituents, nutrients, toxic compounds and numerous pathogenic microorganisms, its collection and treatment is essential to protect public health and the environment. The current trend in the urban water cycle in Europe is to combine a centralised sewer network and a centralised wastewater treatment plant (wwtp) composed of a series of mechanical, chemical and biological processes (EUROSTAT, 2003).

2.1.1. Activated sludge process

The activated sludge process was developed in the early 1910s (Ardern and Lockett, 1914) and is now commonly used for biological treatment of municipal and industrial wastewaters. The activated sludge process is based on the observation that the aeration of wastewater leads to the growth of biological matters which reduce the organic content of the sewage. The basic activated sludge process illustrated in Figure 2 is composed of three steps:

- A reactor containing the microorganisms or active biomass kept in suspension and aerated,
- A liquid-solids separation step usually based on the settling properties of the biomass
- A recycle system for returning solids from the separation unit back to the reactor.



Figure 2: Schematic view of the activated sludge process

The biological process is essential in the wastewater treatment train for removing soluble, colloidal and particulate organic substances. Furthermore, nutrient elimination through biological nitrification-denitrification and biological phosphorus removal can be achieved in its more advanced configuration.

2.1.2. Conventional design of WWTP

A wwtp is composed of successive processes, each removing a specific fraction of the targeted substances (Figure 3). Primary clarification, usually composed of mechanical processes sometimes combined with chemical addition, is most efficient in removing rough materials and large particles. The secondary treatment, mostly composed of the activated sludge process, is targeting organic matters. Biological nitrification-denitrification and biological phosphorus removal can be achieved using staged reactors in series, alternating aerobic, anaerobic / anoxic conditions and proper internal recirculation.



Figure 3: Schematic representation of a conventional activated sludge plant

2.1.3. Achievements and limitations

A high treatment quality in terms of organic compounds and nutrient removal can be achieved by means of a conventional wwtp (Table 1). Furthermore, activated sludge processes can be designed from household purposes up to megalopolis-scale (several million people equivalents), matching a satisfactory effluent quality for main water body discharges.

However, some limitations remain mostly resulting from the final settling stage. In order to achieve high effluent quality, good settling properties of the activated sludge are compulsory. Unfortunately, due to dynamics inherent to the incoming wastewater (pH, loading, seasonal variations), operational problems like bulking sludge, rising sludge or Nocardia growth can occur. These operational problems can result in inadequate separation of activated sludge in the final settling process leading to discharges of activated sludge particles into the main water stream. Furthermore, effluent of a conventional wwtp cannot be considered free of pathogen even if the removal efficiency of pathogenic microorganisms reaches 99,9%.

Due to the growing water stress, especially in terms of quality deterioration, the European Union introduced new regulations in 2000: The Water Framework Directive (WFD)

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2000/60/EC. The aims of the WFD are to achieve "a good ecological and biological state for all surface waters and groundwater in Europe by 2015". New regulations were introduced, like the Dutch directive NW4, defining new surface water quality standards (Table 1). Consequently, tertiary treatments are likely to become compulsory in order to reach the new total nitrogen and total phosphorus requirements, possibly 2.2 mg.L⁻¹ and 0.15 mg.L⁻¹, respectively.

Therefore, innovative technologies need to be developed and implemented in wastewater treatment to overcome the limitations of the activated sludge process and meet the environmental requirements. The membrane process is one of them and membrane bioreactors one of the applications.

Table 1. effluent	discharge limite	s for municinal	l wwtns with (canacity su	nerior at 100 0	
Table L. elliuelli	uischarge minic	s ior municipa	i wwips with	capacity su	penor at 100,00	JUFE

Parameters	Typical efflluent quality of wwtp in the Netherlands	Current discharges limits (2008)	Expected dischages limits by 2015 (NW4)
BOD (mg.L ⁻¹)	5 - 20	20	20
COD (mg.L ⁻¹)	20 - 60	125	125
SS (mg.L ⁻¹)	5 - 20	30	5
N _{tot} (mg.L ⁻¹)	2 - 12	10	2.2
P_{tot} (mg.L ⁻¹)	0.1 - 0.5	1.0	0.2

2.2 Membrane filtration technology applied to MBR

2.2.1. Fundamentals and process description

Membrane basics

Membrane technology was implemented in wastewater treatment due to its high *separation* properties. Acting as a *physical barrier* between two phases, a driving force is needed in order to get a transmembrane flux. The transmembrane flux can be caused by a temperature gradient, a concentration gradient or an hydraulic pressure gradient. The driving force in water treatment membrane processes usually is a hydraulic pressure gradient called *Transmembrane Pressure* (TMP) (Mulder, 2000). It is equal to the difference between the pressure on the feed side and the pressure on the permeate side. Assuming laminar flow conditions through the membrane, the permeate flux rate can be calculated from the Darcy's law (Lojkine et al, 1992):

$$J = \frac{TMP}{\eta_p \cdot R_{tot}}$$
(2-2-1)

Where:

J

= permeate flux, $[m.s^{-1}]$

 η_p = apparent viscosity of the permeate assumed identical to pure water, [Pa.s]

 R_{tot} = total resistance to filtration composed of the membrane resistance and the additional resistance resulting from fouling mechanism, $[m^{-1}]$

Membrane classification

A *total retention* of specific contaminants can be achieved depending on the membrane classes. Membranes are classified according to the membrane pore size, the applied pressure and the Molecular Weight Cutoff (MWCO) (see Figure 4). Microfiltration (MF) and Ultrafiltration (UF) membranes work essentially by size exclusion whereas nanofiltration (NF) and reverse osmosis (RO) achieve removal by diffusion and charge exclusion as well as size exclusion (Metcalf&Eddy, 2003).

Low-pressure membranes, i.e. MF or UF are commonly used in MBR applications. Membrane bioreactors build using MF and UF can retain bacteria, pathogenic microorganisms and large viruses. The effluent produced can thus be considered free of particles and macrocolloidal materials. However, soluble organic compounds, metal ions, various pharmaceutical contaminants and endocrine disrupters are not removed and still remain in the treated water.



Figure 4: Retention of specific contaminants by mean of membrane filtration (adapted from Metcalf&Eddys, 2003)

Membrane materials

The different materials used in the membrane manufacturing can be categorized as organic and inorganic. Inorganic materials are usually ceramics and most likely used in industrial niches due to their prohibitively high costs. Organic materials consisting of modified polymers are mostly used in MBR applications. Most of the membranes used in MBR are anisotropic, presenting variations in the pore size distributions and asymmetric (Judd, 2006).

MBR membranes also need to be mechanically robust and chlorine resistant with regard to the high chlorine concentration used during cleaning. In order to limit membrane fouling, membranes also need to have hydrophilic properties. Hydrophobic membranes get turned into hydrophilic membranes due to surface modifications (Judd, 2006).

Based on these previous aspects, the most suitable polymers used as membrane materials in MBR applications are presented in Table 2.

Material	Advantages	Disadvantages
Polyvinylidene difluoride (PVDF)	Excellent strengh and flexibility Reasonnable permeability	Limited ability to modify properties
Polyethylsulphone (PES)	Good thermal and chemical (Chlorine) stability Easy to fabricate	Low pressure limits Poor chemical resistance to aromatics
Polyethylene (PE)	Inexpensive good flexibility	Sensitive to oxidation
Polypropylene (PP)	High temperature resistance	Moderate chemical resistance Sensitive to chlorine

Table 2: Advantages and disadvantages of several membrane materials (adapted from Pearce, 2008)

Clean water permeability is not as important in MBR processes as in conventional filtration applications, since the accumulation of foulant particles at the membrane surface will strongly affect the membrane filtration properties (Le-Clech et al., 2006). Whereas membrane performances are a critical factor in membrane filtration, module configurations, process design, operating conditions and biology performances are more crucial in MBR applications.

Membrane configurations

Geometry and membrane configurations are key process factors to determine the process performances. Three configurations are currently leading the MBR market (presented in Figure 5):

- The hollow fibre
- The flat sheet
- The tubular

The advantages of each configuration in terms of packing density, turbulence promotion, energy requirements, costs and cleaning strategy are presented in Table 3.

Table 3: Specificity of each MBR membrane configuration (Pearce, 2008)

Configuration	Packing density (m ² .m ⁻³)	Turbulence promotion	Cleaning strategy	Energy requirements	Costs
Hollow fibre (HF)	200-450 (High)	Poor	Backflush Weekly chemical cleaning	Low /moderate	Low
Flat Sheet (FS)	100-150 (Low)	Fair	Relaxation Monthly chemical cleaning	Very low	High
Tubular (MT)	150-300 (moderate)	Good	Backflush Weekly chemical cleaning	moderate/ high	High



Figure 5: picture of (a) a tubular membrane (b) hollow fibre membrane (c) flat sheet membrane

2.2.2. Membrane bioreactors for wastewater treatment

The membrane bioreactor technology is composed of a biological process and a membrane separation step. The major difference with a conventional activated sludge process is the absence of the secondary clarifier. As a consequence, the activated sludge settling properties are not a limiting factor anymore. Higher MLSS content can thus be achieved in MBRs. In combination with the replacement of the secondary clarifier, this results in more compact systems.

MBR configurations

As already reported in *Section 2.2.1*, the module configuration is strongly influencing the overall performances of MBR processes. The module configuration has to promote an even flow distribution, a high packing density, a high turbulence at the membrane surface, a low energetic cost, an easy cleaning strategy and an easy maintenance (Judd, 2006). Considering those aspects, two general trends emerged in practise:

- The sidestream MBR (sMBR)
- The immersed MBR (iMBR)



Figure 6: Schematic view of a sidestream and an immersed MBR (Judd, 2006)

Both configurations are presented in Figure 6 and were intensively discussed by Pearce (2008). Sidestream MBRs are usually designed with a tubular membrane configuration. sMBRs were originally operated under crossflow conditions. In this concept, the activated sludge is pumped into the membrane modules placed on the side of the biological tank resulting in high performances and high fluxes, but at a significant energy cost and a larger footprint. Therefore, sMBR technology was preferred for difficult wastewaters and small-scale high strength water applications.

The sMBR concept has been extended in recent years by the development of the air-lift technology (Pearce, 2008). Air-lift MBRs use air to recirculate the activated sludge into the membrane modules and thereby significantly reduces the energy demand. As a consequence, lower fluxes can be achieved. Due to this low energy alternative, sMBR configuration might become competitive with immersed MBRs for large scale applications due to its advantages in term of cleaning strategy, maintenance and turbulence promotion.

Immersed MBRs are usually designed with hollow fibre or flat sheet membrane configurations. Originally the membrane modules were directly immersed in the biological tank. Part of the air used to maintain aerobic conditions in the tank was then also used to promote proper turbulences and circulation around the membrane modules.

As a result, iMBRs tend to be more cost effective for large scale applications than sMBRs (Pearce, 2008). Consequently, due to the lower turbulence promoted around the membranes, iMBRs tend also to be favoured for low strength wastewater treatment, like municipal wastewater.

In general terms, iMBRs designed with hollow fibre membranes have been found to provide the most cost effective solution for large scale and low strength wastewater treatment plants. This is mostly due to the low cost of hollow fibre membranes, the relatively low aeration demand and the high packing density that can be achieved. However, intensive pretreatments and regular maintenance cleanings are compulsory to maintain stable performances and avoid operational difficulties like fibre clogging due to hairs or poor turbulence promotion.

iMBRs designed with flat sheet membranes have been found to be able to treat the same type of wastewater. However, due to the higher air demand and the relatively high flat sheet membrane cost, this configuration tends to be selected only for small to medium scale treatment plants. Operational advantages like monthly chemical cleaning strategy and easier maintenance are also contributing to its implementation in smaller scale plants (Pearce, 2008).

It is important to notice that differences between hollow fibre, flat sheet, and even sidestream configurations (with the air-lift concept) tend to slightly disappear.

iMBRs were originally designed with the membrane modules integrated in the biological tank. Most of the membrane suppliers are currently offering MBR solutions with membrane modules immersed in a separated tank aside, increasing slightly the plant footprint, but significantly improving the turbulence promotion and facilitating the maintenance and cleaning actions. Hollow fibre packing density tends to decrease as well in order to promote a better activated sludge distribution around the membranes and therefore limit the energy demand. Finally, flat sheet membranes were originally designed to be almost free of chemical cleaning. However, regular chemical cleanings are currently also needed in flat sheet configurations in order to get stable performances at a minimum energy use.

MBR advantages and limitations compared to conventional activated sludge process

As presented in the previous section, the MBR process has many advantages:

- Excellent and stable effluent quality, including disinfection;
- High volumetric loading resulting in *compact* designs and/or low excess sludge productions;
- High potential for *water reuse*.

However, due to the membrane separation stage some drawbacks arise:

- High investment and operation costs compared to conventional activated sludge process due to the membrane costs, the relatively low production flux and the need of qualified operators;
- *Membrane fouling* resulting in:

- More extensive pre-treatments required,
- High energy input required to maintain turbulent conditions near the membrane (aeration),
- Regular chemical cleanings.

For all these reasons, membrane fouling in MBR needs to be comprehended in order to allow further development of the MBR technology.

MBR technology development

The first commercial MBR applications can be found in the 1960s, where side-stream MBRs were applied to treat high strength industrial wastewater (Stephenson et al., 2000). The MBR market mostly grew in the USA and Japan in niche-applications due to its high cost in terms of membrane installation and energy consumption. From 1989 on, a new growth period started for the MBR technology with the concept developed by Yamamoto et al. (1989). Yamamoto et al. presented the idea that membranes could directly be immersed in the activated sludge and operated at modest flux reducing drastically the operating costs. Thanks to this new concept and the reduction of the membrane costs which occurred during the 1990s, the MBR market grew rapidly in the period from 2000 to 2005 (\in 166 million, Hanft, 2006).

Lesjean & Huisjes (2008) published a survey about the trends and perspectives of the European Market. They showed the MBR development has started in the early 1990s concerning the industrial sector whereas it only took off in 1999 in the municipal one. 154 MBRs could be counted in total in Europe in 2002 which included 85 % of industrial applications. From this date on, a sharp increase of the number of installations in both sectors is noticeable and can be explained by the commercial success of the immersed MBR technologies offering lower capital and operating costs.

It is expected that the global progression rate is likely to remain at least constant in the coming years. Up till now, the MBR market is largely dominated by two actors, namely GE-Zenon and Kubota providing hollow fibre and flat sheet membranes, respectively.

De Wilde et al. (2009) reported a survey presenting that further MBR market growth could be expected if several standardisation measures would be taken. They underlined the need for standards on the *interchangeability* of MBR filtration modules, standard *pre-treatment* and *standard characterization methods* (membrane fouling monitoring, membrane aging and membrane performance assessment). Their survey reported that these standardisations should help decision makers to invest in MBR technology and therefore contribute to the (municipal) MBR market growth.

Brepols et al. (2008) also reported that a large potential market for MBR technology could be the upgrading and the retrofitting of the current municipal treatment plants.

2.3 Membrane Fouling

Each requirement and chosen design presented in *Section 2.2* is mostly dictated by one goal:

 Prevent the accumulation of foulant materials at the membrane surface in order to operate MBRs on a long term at the highest flux as possible and at minimum energy use, in other words: *prevent fouling*.

Fouling is defined by the Union of Pure and Applied Chemistry (UPAC) as "the loss of performance of a membrane due to the deposition of suspended or dissolved substances on its external surfaces, at its pore openings or with in its pores" (Koros et al., 1996). It can then be considered a plural phenomenon where each mechanism is inducing some losses of membrane performances.

2.3.1. Fouling mechanisms

Fouling is generally separated in four principal fouling mechanisms. However, it is commonly admitted that the contribution of concentration polarisation can be considered negligible in UF applications (Vyas et al., 2001, Geilvoet, 2010). Therefore, the concentration polarisation will be left out of consideration in this section. The three remaining mechanisms illustrated in Figure 7 are:

- *Pore blocking* (R_{pb}): the number of pore channels available for permeation during filtration is reduced due to particles obstructing membrane pores.
- *Absorption* (R_a): the cross section available for permeation is reduced due to particles, colloids and molecules entering the membrane pores and absorbing at the membrane wall.
- *Cake (or Gel) layer formation* (R_{cl}): the membrane surface is covered by a more or less dense and porous layer resulting from the accumulation of particles and other materials that could not enter the membrane.



Figure 7: Schematic representation of fouling mechanisms

According to the resistance-in-series model, the total membrane resistance is composed of the addition of the membrane resistance itself (R_m) with the resistances due to the fouling mechanisms and can be calculated as followed:

$$R_{tot} = R_m + R_{pb} + R_a + R_{cl} \tag{2-3-1}$$

2.3.2. Fouling steps

The three mechanisms presented in the previous section are interacting and becoming more or less dominant during the filtration process, depending on the membrane operational conditions or the filtration time period. However, different fouling steps can be identified over a longer filtration period. The theoretical fouling steps are illustrated in Figure 8. They are commonly defined and classified depending on the cleaning strategy needed to remove them:

- *Reversible fouling*: it can be removed by physical cleaning. The cake layer mechanism is considered to be the dominant mechanism taking part in this step (Meng et al, 2009).
- *Irreversible fouling*: it can be removed by chemical cleaning and not by physical cleaning. Pore blocking and absorption mechanisms are considered to be predominant in this step.
- Irrecoverable (long-term irreversible) fouling: it can not be removed by cleaning means. Since irrecoverable fouling is inevitable it can also be considered a part of membrane characteristics (membrane aging).



Figure 8: Fouling rates during long-term MBR operations under constant flux condition (adapted from Kraume, 2007, Geilvoet, 2010)

2.3.3. Factors affecting fouling

Judd (2006) presented in a schematic way inter-connections between different fouling mechanisms and numerous factors involved in Figure 9. As a simplified approach, parameters can be grouped in three variables (Le-Clech et al, 2006):

- The membrane characteristics,
- The MBR operating conditions,
- The feed water /biomass characteristics.

The three mentioned groups and their impacts on fouling will be discussed separately in the following section.



Figure 9: Inter-relationships between MBR parameters and fouling (Judd, 2006)

Membrane characteristics

Numerous publications tried to define the optimal membrane configuration and materials to limit fouling and clogging. Meng et al. (2009) published a review in order to organise and highlight the more salient research results of the past years. As already presented in *Section 2.2.1*, pore size, porosity, roughness, surface charge and hydrophilicity are decisive characteristics due to their large impacts on fouling build up and MBR performances. Pore size distribution is considered a crucial parameter. A narrow pore size is likely to be more effective for fouling prevention. Different materials used in the membrane fabrication do not seem to be equal with respect to reversible and irreversible fouling. For instance, Yamato et al. (2006) showed that PVDF membrane were superior to PE membrane in terms of irreversible fouling prevention in MBR treating municipal wastewater. Zhang et al. (2008) also investigated the affinity between foulants and different membrane materials. PVDF membranes showed less affinity with extra-cellular polymeric substances (EPS) than PES membranes in this study.

MBR operating conditions

Constant flux operation

Current full-scale MBR applications are usually operated under constant flux conditions (Judd, 2006). The imposed flux, leading with time to a rise in TMP, results in three fouling stages illustrated in Figure 10 (Zhang et al., 2006):

- Stage 1 where an initial short-term and rapid rise in TMP occurs
- Stage 2 where a long term rise in TMP occurs either linear or weakly exponential
- Stage 3 where a sudden TMP jump occurs resulting in maintenance actions.



Figure 10: illustration of the TMP jump concept (Zhang et al., 2006)

The *stage 1* is mostly due to colloidal absorption and pore blocking mechanisms and is considered to lead to irreversible fouling (Ognier et al., 2002). However, on a longer term, this fouling seems to be negligible compared to the total membrane resistance (Choi et al., 2005). The *stage 2* is characterised by a slow fouling propensity due to the colloidal matter deposition, especially polysaccharides (Zhang et al., 2006). The length of the *stage 2* is mostly conditioned by the extraction flux and the system hydrodynamics. The *stage 2* ends when the increase in fouling propensity inherent to the mode of operations at constant flux results in a TMP jump (*stage 3*). The rapid loss of performances occurring during the *stage 3* is most likely due to an inhomogeneous distribution of the fouling layer (Zhang et al., 2006). In order to maintain constant flux conditions, local flux increases strongly in the less fouled zone. The fouling is then characterised by a self-acceleration mechanism resulting at the end in a TMP jump.

It is therefore crucial to carefully choose the imposed flux. Le-Clech et al. (2006) advise to operate MBRs at a modest flux in order to maintain sustainable operating conditions and therefore postponed as long as possible the occurrence of the *stage 3*.

Sludge retention time

Sludge retention time (SRT) is considered one of the major parameters affecting MBR process operations. Due to the complete retention of solids in an MBR, it is possible to achieve a long SRT with MBR technology. However operating an MBR at long SRT leads inevitably to an increase of the MLSS content (Le-Clech et al., 2006). Operating MBRs at long SRT was intensively investigated in order to assess limits and potential of the technology (Laera et al., 2007, Cote et al., 1997). In regard of fouling, it is not recommended to operate an MBR at long SRT (more than 40 days) due to the large increase of the activated sludge apparent viscosity (Laera et al., 2007, Rosenberger et al., 2002). Furthermore, operating at long SRT requires an increase of the aeration intensity to maintain the high MLSS content in suspension and provides proper activated sludge oxygenation. Nowadays, full-scale municipal MBRs are operated sustainably with SRT

varying between 20 and 30 days allowing nutrient removal and moderate operating costs (Brepols et al., 2003, De Wilde et al., 2003, Lyko et al., 2007).

Aeration

Aeration is currently used in immersed MBR configurations to provide oxygen to the biomass, to maintain the activated sludge in suspension and to mitigate fouling by turbulence promotion and membrane surface scouring, i.e avoid membrane clogging and prevent as much as possible the occurrence of fouling. Whereas crossflow filtration in tubular membranes is well known and efficiently modelled, the bubble effect in hollow fibre (and to some smaller extent flat sheet) modules is still under investigation. Pollet (2009) performed an intensive work to get a better understanding of the air and liquid flow distributions in hollow fibre configurations. The uneven distribution of the intensity of the turbulent shear remains a major issue to optimize energy input in MBRs. Moderate packing density is considered a major factor promoting low fouling conditions. The increase of the aeration improves filtration conditions till an optimum value where further increase do not result in an improvement of the membrane performances. Therefore, an optimum value for the aeration between the membrane performances and the energy costs needs to be found for each MBR configuration. Furthermore, it is also quite common to use intermittent aeration in MBR applications in order to reduce operational costs.

Activated sludge characteristics

Activated sludge properties and their impacts on membrane fouling have been intensively investigated (Judd, 2006). As a simplification, this section will mostly focus on the particle size distribution aspect and the major constituents considered being involved in the fouling process, namely extracellular polymeric substances and soluble microbial products.

Particle Size Distribution

The constituents of the activated sludge are usually classified in three fractions (Metcalf&Eddy, 2003):

- The suspended solids (> 1μm)
- The colloids (0.1 to 1µm)
- The soluble constituents ($< 0.1 \, \mu m$)

The physical characteristic *size* of the activated sludge solids plays a major role in the fouling process (Judd, 2006). Mean floc sizes mentioned in literature, main components of the suspended solids fraction, vary from 240 μ m for Cabassud et al. (2004) to 3.5 μ m for Cicek et al. (1999) whereas Bae et al. (2005) report mean floc size of 25 μ m. However, it is currently admitted that the floc size of the activated sludge does not have a direct correlation with fouling (Le-Clech et al., 2006). It is mostly due to the fact that the turbulence promoted close to the membrane surface by aeration is preventing large particle deposition onto the membrane.

Colloids are nowadays considered the main contributor to membrane fouling due to their size range close to the membrane pore size (Rosenberger et al., 2005). Geilvoet (2010)

demonstrated during his research work that the particles determining the cake layer volume, i.e. the filtration resistance, are predominantly in the size range between 0.1 and $0.5\mu m$.

The soluble fraction contribution to fouling through their absorption on the membrane pores remains unclear (Bae et al, 2005, Wisniewski et al., 1998). The contrary results collected and reported by Judd (2006) concerning the contribution of the soluble fraction to membrane fouling are likely due to differences in definitions of each fraction used by different research groups.

Extracellular polymeric substances

The Extra cellular Polymeric Substances (EPS) provide the physical framework which keeps microbial aggregates together. They form a layer that protects the micro-organisms against influences from the outside. The characteristics of EPS are depending on various factors, such as gas sparging, substrate composition, loading rate and solids retention time (Laspidou and Rittmann, 2002). It results in a large diversity of EPS network possibilities.

Two forms of EPS are currently distinguished in MBR fouling research:

- *The bound EPS* which derives from the active cell
- *The soluble EPS* which are solubilised in the free water.

Figure 11 shows a schematic representation of the different mechanisms. Soluble EPS are released from the bacterial cell during cell lysis and diffuse through the cell wall into the free water. As a major difference with soluble microbial products, part of the soluble EPS can also be coming from the influent.





Many research groups tended to comprehend the relation between EPS and fouling. However, the conclusions are not consistent.

Firstly, it is likely due to the lack of a standard measuring protocol for the determination and the extraction of the bound EPS concentration. Several extraction methods are mentioned in literature like cation exchange resins (Jang et al., 2005, Frølund et al., 1996), heating methods (Morgan et al. (1990)) and centrifugation with formaldehyde (Zhang et al., 1999). This large variety in extraction methods leads to a wide range of bound EPS concentration, difficult to compare or to link in a common model. Secondly, the inconsistent results concerning soluble EPS are likely due to the fact that the colorimetric methods used to characterise soluble EPS does not allow a clear differentiation between the colloidal and the dissolved fractions of the soluble EPS. Therefore the contribution of each EPS fraction to fouling cannot be properly established.

Researchers succeeded still to correlate EPS to fouling (Cho and Fane, 2002, Rosenberger and Kraume, 2002b). Drews et al. (2005) and Lesjean et al. (2005) reported that an increase of the polysaccharide contents showed a good correlation with a fouling rate increase. Furthermore Cho et al. (2005) demonstrate that bound EPS have some influences on the specific cake resistance.

However, controversial results were reported concerning bound EPS depending on the SRT and their impact on fouling tendency (Meng et al., 2009). Lee et al. (2003) and Ng et al. (2006) present results were the optimum SRT related with the lower fouling propensity is around 20 days whereas Zhang et al. (2006) and Han et al.(2005) show results where the optimum SRT is fluctuating between 30 and 50 days. Their diverging results underline the fact that the role of EPS in the fouling process is still not fully understood, notably due to the lack of analytical standard methods.

Soluble microbial products

Soluble microbial products (SMP) were firstly introduced by Namkung and Rittmann in the mid 80s. They are commonly considered "soluble cellular components that are released during cell lysis, diffuse through the cell membrane, are introduced with the influent, are lost during synthesis or are excreted for some purpose" (Laspidou et al. 2002). SMP are usually low formula weight compounds and biodegradable. Due to the fact that SMP and soluble EPS are composed of the same components (except that a part of the soluble EPS fraction is coming from the influent), SMP are currently measured analytically with the same colorimetric methods used to quantify soluble EPS. They are now widely acknowledged as a major contributor to MBR fouling.

The influence of SMP on MBR fouling was demonstrated by several research groups. Zhang et al. (2006) found that SMP were more important than the MLSS content in order to explain the fouling process. Jeong et al. (2007), Sperandio et al. (2005), Ye et al. (2005) and Trussell et al. (2006) presented results where the fouling rate was influenced by the SMP content.

However, like in the EPS case, the role of SMP in the fouling process is still not clear and controversial results can be found in literature. Drews et al. (2007) tried to sum up the SMP research results of several research groups. However they did not succeed to underline clear trends, notably due to the difference in SMP extraction methods, membrane characteristics and scales of the research performed.

Furthermore, Geilvoet (2010) reported that the volume of the particles in the free water with a diameter ranging from 0.4 to $1\mu m$ showed a more significant correlation with filterability, i.e. fouling potential, than the SMP concentration.

2.4 Physical characterisation of activated sludge: focus on filterability

As already presented in *Section 2.2*, activated sludge properties are strongly influencing the membrane fouling process. Tools were developed by different research groups to characterise physically and chemically the activated sludge. Activated sludge is usually physically characterised using four parameters, namely the settleability, the compressibility of the activated sludge, the apparent viscosity and the filterability (Metcalf & Eddy, (2003)).

2.4.1. Activated sludge settleability

Activated sludge settling properties are crucial for conventional wwtp operations. Good settling properties need to be enhanced in order to ensure a low suspended solids concentration in the effluent and sufficient biomass return into the aerobic tank. The activated sludge settleability is usually quantified by the sludge volume index (SVI in mL.g⁻¹). The SVI represents the ratio between the volume of an activated sludge sample after a well-defined settling time and its MLSS concentration. It gives valuable information about the activated sludge flocculation state and floc structure. SVI values close to 120 mL.g⁻¹ are indicators of good settling properties in while SVI values above 150 mL.g⁻¹ indicate poor settling properties.

2.4.2. Activated sludge compressibility – thickening

The thickening process is currently used to reduce the volume of activated sludge needing post-treatment. The thickening process reduces the operating costs of the activated sludge treatment line and offers several advantages (Degremont Water Treatment Handbook, 2007):

- The increase of the degradation rate of organic matters in digesters
- The improvement of the reliability along the entire water treatment line
- The reduction of the volume of conditioning works

The thickening process usually results in an increase of the concentration of the activated sludge collected in the clarifiers which should remain in the limits of activated sludge pumpability. The basic thickening technique is the thickening by settling or gravity thickening.

During thickening by settling, the activated sludge suspension is fed into a tank with a long retention time so that the activated sludge gets compacted. Compacted activated sludge can then be withdrawn from the bottom and supernatant from the top of the tank. This type of thickener is designed based on the sedimentation curve developed in the Kynch Theory (Kynch, 1952) illustrated in Figure 12.

Kynch theory is based on the hypothesis that the falling velocity of a particle depends solely on the local particle concentration. Different zones can be identified during the settling process presented in the Figure 12 by letters A to E. Each zone presents its own settling properties in term of velocities and flocculation state and is mostly depending on the concentration of particles.



Figure 12: Interfaces in sedimentation (a), illustration of the Kynch Theory (b) (Tiller, 1981, Degremont Water Treatment Handbook, 2007)

From the Kynch curve the concentration in each zone can be determined as a function of time. Each zone or section can then be used to design and to size the settling units like solids contact settling tanks or activated sludge thickening units (Degremont Water Treatment Hanbook, 2007).

2.4.3. Activated sludge apparent viscosity

The relatively high mixed liquor suspended solids content (MLSS) in an MBR results in higher viscosity values compared to conventional systems. Activated sludge is considered a non-Newtonian fluid behaving as a pseudo-plastic fluid (Rosenberger et al., 2002, Le-Clech et al., 2006). It is composed of a floc network which tends to get disrupted under high shear rate conditions, resulting in a decrease of apparent viscosity.

Laera et al. (2007) investigated activated sludge rheology that depended on sludge age (40 to 80 days). A model based on the Ostwald model was proposed to determine apparent viscosity depending on TSS content:

$$\eta_a = \exp(0.882 \cdot MLSS^{0.494}) \cdot (\gamma_0)^{-0.05 \cdot MLSS^{0.631}}$$
(2-4-2)

With:

 $\begin{array}{ll} \eta_{a} & = \mbox{the apparent viscosity of the activated sludge in [Pa.s]} \\ MLSS & = \mbox{the mixed liquor suspended solid concentration of the activated} \\ sludge in [g.L^{-1}]. \\ \gamma_{0} & = \mbox{the shear rate at the wall in [s^{-1}]} \end{array}$

Rosenberger et al. (2002) investigated apparent viscosity for a large number of smallscale MBR plants. The pseudo-plastic behaviour of MBR activated sludge was confirmed and apparent viscosity variations were correlated with TSS. The model proposed is:

$$\eta_{a} = \exp(1.9 \cdot MLSS^{0.43}) \cdot (\gamma_{0})^{-0.22 \cdot MLSS^{0.37}}$$
(2-4-3)

Other parameters which showed a lower impact on the apparent viscosity during Rosenberger study were the EPS concentration and mechanical stress prior to the measurements.

However, in both studies, the wide range in MLSS content (5 to 40g.L⁻¹) and the relatively high sludge age (SRT) investigated cannot be considered representative of municipal full-scale MBR operating conditions that are mostly operated around MLSS of 8-12g.L⁻¹ and SRTs of 20 days (Brepols et al., 2003, De Wilde et al., 2007).

In other studies, Wu et al. (2007) and Wang et al. (2006) investigated the effects of activated sludge characteristics on membrane fouling in a pilot-scale submerged MBR. During this long term experiment, EPS and soluble COD were considered the main contributors to membrane fouling whereas apparent viscosity and MLSS showed a significant statistical influence on membrane fouling. Meng et al. (2007) operated three identical lab-scale membrane bioreactors in order to determine the impact of hydraulic retention time on the MBR performances. They observed growth of filamentous bacteria at low hydraulic retention time, resulting in an increase in the apparent viscosity which affected the performances of the membrane process. Kornboonraksa et al. (2009) investigated factors affecting MBR performances when treating high strength wastewater. MLSS and floc size were found to be the dominant factors affecting membrane filtration performances, while activated sludge apparent viscosity and EPS were secondary factors affecting membrane fouling.

However, even if several studies (Wu et al., 2007), Wang et al., 2006) found same impact of apparent viscosity on MBR filtration performances on a lab-scale, to our knowledge there is no data currently available concerning the apparent viscosity impact on full-scale municipal MBRs.

2.4.4. Activated sludge filterability

Activated sludge filterability usually refers to the dewaterability of the activated sludge. However, due to the fact that this research work focused on the membrane bioreactor technology, this section will focus on activated sludge filterability related with membrane processes, i.e. membrane filterability.

Filterability (or fouling potential) can be defined as the specific contribution of the activated sludge to the overall filtration resistance encountered (Rosenberger et al., 2005). Filterability is usually measured under constant hydraulic conditions and is therefore considered mostly dependant on the membrane and the activated sludge characteristics. This is a major difference with the membrane permeability which is a result of the membrane properties, the module hydrodynamics and all filtration resistances (Judd, 2006, Rosenberger et al., 2005).

Filterability was therefore intensively used in MBR research. Rosenberger et al. (2002) reported that the filterability of activated sludge was mostly influenced by the

composition of the free water phase whereas no significant impact of the MLSS content and of the bound EPS concentration could be found. Mikkelsen (2001) reported in its experiments that a deflocculation resulting in an increase of the concentration of the dispersed mass caused deterioration in filterability. It was also reported that filterability can be considered a dynamic parameter continuously subjected to changes due to various factors (influent flow rate, influent composition, temperature, biological treatment, etc.) (Judd, 2006, Evenblij, 2006).

Geilvoet (2010) also concluded that a good filterability was a required condition in order to ensure good operating conditions in MBRs. He also showed that the activated sludge filterability was strongly related with submicron particles (colloids) present in the freewater phase (see *Chapter 3*).

As no standardised protocol exists, several protocols, based on fouling rate or TMP measurements, were developed or adapted from dewaterability tests by different research groups in order to quantify activated sludge filterability. The most commons are:

• The flux step method based on the critical flux concept (Le-Clech et al. (2003)). As presented in Figure 13, the fouling rate evolution is quantified by monitoring the increase in TMP during several filtration steps at different fluxes. However, the lack of a standard set-up and protocol is the main drawback of the flux-step method, since it allows no unequivocal comparison between different data. Furthermore, results obtained are also dependent on the membrane initial state. It is important to notice that Van der Marel (2010) also developed an improved flux step method which can be used to investigate both the reversible and irreversible fouling potential of an activated sludge.



Figure 13: Schematic representation of the critical flux determination by the flux-step method (Le-Clech et al., 2003, Geilvoet, 2010)

• The Capillary Suction Time (CST) originally used as a dewaterability test, it is also used in MBR research. CST is the time interval required by a sludge sample to saturate a certain fixed area of a filter paper under the influence of the capillary suction (Standard methods, 1998). However, a major drawback of the CST is its

dependency on the MLSS concentration. Furthermore, in a recent study, Lyko et al. (2007) did not succeed to monitor filterability changes using CST.

• The Time to Filter (Standard Methods, 1998) is a method where an activated sludge sample is filtered in dead end mode over a paper filter under well-defined constant pressure conditions (Figure 14). Unfortunately, due to its MLSS dependency and the constant pressure mode used during this test; the Time to Filter is likely not to bring any representative knowledge on filterability of MBR activated sludge.



Figure 14: Schematic representation Time-to-Filter method (Geilvoet, 2010)

• The Delft Filtration Characterisation method (DFCm, Evenblij et al. (2005)) and the MBR-VITO fouling measurement method (MBR-VFM, Huyskens et al., (2008)) are considered the most known methods quantifying filterability of an activated sludge sample under well-defined hydraulic conditions. These different methods will be evaluated in *Chapter 5*.

As a last remark, filterability is usually measured on short term basis. Therefore, a filterability measurement does not give any direct indication in term of long term or irreversible fouling behaviour. However, combined with permeability data, it does help to figure out the contribution of each fraction, namely the membrane aging (irrecoverable fouling) and the actual (reversible) fouling.

2.4.5. Membrane performance indicators and filterability

Membrane performances are usually expressed in tern of fouling rate, i.e. TMP increase over time, and permeability in current MBR applications. The permeability can be expressed as presented in equation (2-4-4):

$$L = \frac{J}{TMP} = \frac{1}{\eta_p \cdot R_{tot}}$$
(2-4-4)

With:

- L = permeability in $[m.s^{-1}.Pa^{-1}]$ or $[L.m^{-2}.h^{-1}.Bar^{-1}]$ J = flux in $[m.s^{-1}]$
- η_p = apparent viscosity of the permeate in [Pa.s]
R_{tot} = total resistance to filtration in $[m^{-1}]$

An overall overview of the plant performances can be obtained with these measurements. However, Geilvoet (2010) already emphasized the weakness of the permeability parameter:

- Its dependence to flux and pressure when cake layer filtration is involved
- Its lack of differentiation between membrane resistance and fouling resistance after some time of operation
- Its calculation based on the total membrane surface and not the effective membrane surface.

As a consequence, the measurements of the fouling rates and permeability decline can be considered a consequence of the deterioration of the filtration process but can hardly explain the causes of it.

The measurements of filterability can however bring additional knowledge. Whereas permeability measurements gave an overall overview of the performances of the MBR plants, filterability measurements can help to distinguish between the performance loss due to the activated sludge quality itself and the performance loss due to poor membrane operations. Therefore, having an indication of the activated sludge filterability, MBR filtration cycle, aeration rate or flux can be optimised or set dynamically as a function of the fouling propensity of the activated sludge.

2.5 Summary

Due to the implementation of the membrane stage, MBR technology can provide superior effluent quality and footprint reduction compared to the conventional activated sludge process. However, even if the development of immersed membrane technology succeeded to reduce significantly MBR operational costs, the capital and fouling mitigation costs still hampered further market development.

The main contributors of fouling still need to be more precisely investigated and more accurately quantified in order to develop efficient counter measures and fouling prevention protocols.

As a last remark, the measurements of filterability can indicate whether a permeability decrease should be attributed to poor activated sludge filterability or inadequate operations of the filtration process. They can therefore significantly contribute to the further optimisation of the MBR performances.

3 Materials and methods

The DFCm, the analyses performed and the European tour organised during this research work will be described in this chapter.

3.1 The Delft Filtration Characterisation method (DFCm)

3.1.1. Background

The first research on MBR technology performed at Delft University of Technology started in 2001 with the PhD thesis of Evenblij (2006). The aim of his research was to get a better understanding of the relations between activated sludge properties and membrane fouling. Evenblij work focused on the development of a method which allowed accurate characterisations of MBR activated sludge filterability. This research resulted in the creation of the Delft Filtration Characterisation method (DFCm), composed of a filtration unit and a measuring protocol.

Thanks to the DFCm, different activated sludge samples could be filtered under identical standardised hydraulic circumstances (Figure 15). The resulting additional resistances can then be compared reliably and differences in filterability behaviour can be attributed exclusively to the activated sludge properties.

Geilvoet (2010) took the follow-up of this work in 2005. He firstly assessed the possibilities and limitations of the DFCm. These results will be summed up in *Section 3.1.4*. Geilvoet also monitored the filterability of two full-scale MBRs in Varsseveld and Heenvliet over a period of several months and succeeded to comprehend and explain the development of the permeability of the full-scale plants using the filterability results. As a final work, the author performed stress experiments that emphasised the correlation between filterability deterioration and activated sludge deflocculation.





3.1.2. Filtration characterisation unit

The DFCm is presented schematically in Figure 16. The unit used during this thesis was the second version of the filtration characterisation unit. Its design was identical to the unit developed by Evenblij except that it was made mobile. Pumps, valves, membranes

and tube diameters remained identical. The structure of the frame was made lighter and easier to build and carry. As a major improvement, the new version of the unit can be located directly on the MBR site in order to perform the filterability tests as close as possible to the sampling point (in time). It remained a short term experiment used to assess activated sludge filterability at a given time. As already emphasised by Geilvoet (2010), experiments need to be performed on a short term in order to exclude the role of biological processes on the activated sludge. As a last remark, the filtration characterisation unit should not be considered a lab-scale membrane bioreactor aiming at simulating the process but merely a measuring device.



Figure 16: Schematic representation of the DFCm Filtration Characterisation unit (Geilvoet, 2010)

A single sidestream tubular PVDF ultrafiltration membrane tube (X-flow F5385, diameter = 8 mm, length = 1 m, membrane area = 0.025 m^2 and nominal pore size = 0.03μ m) forms the basic filtration system of the unit. A peristaltic pump (Watson-Marlow 620) is used for the activated sludge recirculation with a cross-flow velocity (CFV) of 1 m.s⁻¹ (single-phase flow). The permeate is extracted at a constant flux of 80 L.m⁻².h⁻¹ and the permeate production rate is monitored thanks to a mass balance (Mettler Toledo). During the filtration, several parameters (TMP, flux, temperature, pH) are monitored and stored in a computer file using the software application Testpoint (National Instrument).

The main output of an experiment is the progression of the resistance during filtration. This resistance is calculated using Darcy's law (detailed in *Section 1*):

$$R_{total} = \frac{TMP}{\eta_p \cdot J} \ [\text{m}^{-1}]$$
(3-1-1)

In this equation the viscosity of the permeate is taking into account. Based on the work of Manem and Sanderson (1996), the permeate viscosity can be assumed equal to pure water and is only depending on temperature. A correction for temperature is then performed

during our experiments using the empirical relationships between temperature and pure water viscosity presented by Janssen and Warmoeskerken (1997):

$$\eta_p = 0.001 \cdot e^{(0.580 - 2.520 \cdot \theta + 0.909 \cdot \theta^2 - 0.264 \cdot \theta^3)}$$
(3-1-2)

with

$$\eta_{p}$$
 = apparent permeate viscosity [Pa·s]
 $\theta = 3.6610 \cdot \frac{T}{273.1 + T}$ (empirical factor) [-]
T = temperature [°C]

The filtration resistance is plotted as a function of the permeate production per unit of membrane surface. The initial resistance (i.e. membrane resistance) can be considered identical for all experiments and is left out of consideration when analysing the results.

At the end of each filtration experiment, the membrane of the filtration unit is cleaned by a mechanical and/or chemical cleaning procedure. These two steps will be described in detail in *Section 3.1.3*.

3.1.3. DFCm measuring protocol

The DFCm measuring protocol is the key point to get comparable results between various sets of filtration experiments. It needs to be performed always according to the same procedure. Therefore, it was formulated and explained in detail by Evenblij (2005) and Geilvoet (2010). An overview of these key points will be presented in this section.

- Determination of the membrane resistance
 - \circ Recirculation of clean water at a crossflow velocity of 1 m.s⁻¹
 - Permeate extraction at a flux of 80 L.m⁻².h⁻¹
 - Monitoring of the filtration resistance
 - \circ If the membrane resistance is higher than $0.5*10^{12}$ m⁻¹, mechanical or chemical cleaning needs to be implemented
- Filtration of the activated sludge sample
 - \circ Recirculation of the activated sludge at a crossflow velocity of 1 m.s⁻¹
 - Permeate extraction at a flux of 80 $L.m^{-2}.h^{-1}$
 - $\circ\,$ Permeate extraction until a permeate production of 25 L.m⁻² or a TMP value of 0.6 Bar
 - Reconsideration of the flux value in case of extreme poor or good filterability
- Cleaning of the membrane
 - Forward flush with clean water at a crossflow velocity higher than 5 m.s⁻¹
 - At the end of the set of experiments or if the starting resistance is higher than $0.5*10^{12}$ m⁻¹, a chemical cleaning is performed with NaOCl (1500 ppm) for at least 15 min.

3.1.4. DFCm Assessment

This section will be a summary of a part of the work performed by Geilvoet (2010) during his PhD thesis.

Geilvoet (2010) first emphasised that the DFCm goal is the characterisation of the *activated sludge filterability* and not the measurement of *fouling simulation*. It is important to notice that filterability is an activated sludge *characteristic* whereas fouling is a *process*. As a consequence, filterability can be determined and measured accurately on a *short term* basis. However, as a major drawback, the potential relations between filterability and irreversible or irrecoverable fouling cannot be directly assessed. It is therefore not a decisive parameter with respect to the filtration process, but can still be considered a good starting point for a proper filtration process. As an additional remark, Geilvoet (2010) specified that the DFCm can clarify the role of the activated sludge filterability in the fouling process whereas permeability measurements can not.

The second part of the DFCm assessment performed by Geilvoet (2010) was focusing on the representativeness of the filtration unit for the MBR filtration process in practice. Geilvoet agreed on the fact that the operational circumstances in the DFCm membrane tube are not completely identical to the ones during immersed MBR filtration. However, it does not appear to be a strong objection regarding the fact that the aim of the DFCm is not a simulation of the membrane operations but the characterisation of the activated sludge properties. Furthermore, experimental results confirmed the reliability of the method in full-scale MBR plants.

The rheological study presented in his work also demonstrated that for the operating conditions in the DFCm and for the range of MLSS concentrations encountered in the MBR process, the flow regime in the membrane tube of the DFCm is laminar. This implies that the rheological circumstances in the membrane tube do not affect the experimental results.

Geilvoet (2010) finally concluded his assessment by demonstrating that the dominant fouling mechanism in the DFCm experiments was the cake layer formation. As also presented in Figure 17, theoretical calculations indicated that the cake layer resistance was mostly due to the *concentration of substances* with a diameter inferior to 5μ m accumulating on the membrane surface, while larger particles were being retained in the bulk flow due to the backtransport phenomena.



Figure 17: Backtransport velocity (V_{BT}) as a function of the particle size in the DFCm (Geilvoet, 2010)

Based on this demonstration, the DFCm can be considered a powerful and practiceoriented tool. The DFCm can be used in-situ at a full-scale MBR plant. Coupled with the permeability measurements of the full-scale plant and the operational conditions set in the MBR, accurate optimisation actions can be taken. The filterability measurements can determine if low permeability values are due to poor filterability or inappropriate operating conditions. In case of poor filterability, process operations can then be adjusted to minimise fouling consequences.

3.1.5. DFCm output

General output

The main step of the measuring protocol is the activated sludge filtration step. The main output of an experiment is the progression of the resistance during filtration. This resistance is calculated using Darcy's law (see previous section). The filtration resistance is plotted as a function of the permeate production per unit of membrane surface. As a result of the fouling of the membrane during filtration, filtration resistance will increase. The slope of the curve gives an indication of the activated sludge filterability; e.g. a steep curve corresponds to poor filterability (Figure 18).



Figure 18: Example of DFCm output

For easy comparison of different curves, the value ΔR_{20} can be used. This value is defined as the increase in resistance after a specific permeate production of 20 L.m⁻². The ΔR_{20} can be considered a finger print of the filterability sample. Due to the fact that the curves obtained during the experiments followed the same trend, the ΔR_{20} values extracted from each curve can be considered representative of the filterability of the activated sludge sample investigated. Therefore, the filterability comparison between different activated sludge samples will be expressed in terms of ΔR_{20} values in the rest of this thesis.

Based on the DFCm results gathered during numerous filtration campaigns, a practical classification has been made by Geilvoet (2010) and is presented in Table 4.

Table 4: ΔR_{20} and corresponding filterability qualification (for the standard measuring protocol, CFV = 1.0 m.s⁻¹, J = 80 L.m⁻².h⁻¹)

ΔR ₂₀ (*10 ¹² m ⁻¹)	Filterability
0 - 0.1	Good
0.1 - 1.0	Moderate
>1	Poor

Fitting cake layer filtration theory (Geilvoet, 2010)

Based on the assumption that the dominant fouling mechanism in a short-term DFCm experiment is cake layer formation, Geilvoet (2010) showed that the DFCm outputs can be fitted to a power function with two coefficients p and q:

$$\Delta R = p(V)^q \tag{3-1-3}$$

With

$$p = (\alpha_R \cdot c_i)^{\frac{1}{1-s}}$$
(3-1-4)

$$q = \frac{1}{1-s} \tag{3-1-5}$$

Where:

 α_r = specific cake resistance at reference resistance, [m·kg⁻¹]

 c_i = solid concentration involved in the fouling process, [kg·m⁻³]

s = compressibility coefficient, [-]

Considering the power relationship between the resistance increase and the specific permeate production, the DFCm outputs can then be plotted in logarithmic scale on both axes as presented in Figure 19. This representation allows a better understanding of the contribution of the compressibility coefficient *s* and the product $\alpha_r \cdot c_i$ to the total filtration resistance (Geilvoet, 2010).



Figure 19: Example of DFCm output in double logarithmic scales

Both parameters α_r and *s* are related to the properties of the activated sludge. Under constant flux filtration, the compressibility coefficient *s* expresses the compression potential of the cake layer and its values are varying between 0 and 1, where 0 corresponds to no compression and 1 to a complete one.

This further distinction of the DFCm outputs provides significant information in order to better understand the DFCm data set. Thanks to this separation, the contribution of the compressibility and the contribution of the concentration of foulants involved in the cake layer formation can be differentiated within the total resistance increase monitored during the DFCm experiments.

3.2 The European DFCm tour

3.2.1. Background

As already underlined in *Chapter 2*, the lack of standardised methods for the activated sludge characterisation, i.e. EPS, SMP or filterability, is one of the hindrances of the MBR process optimisation. Regarding filterability measurements, a large quantity of methods has been developed by different research groups making the reliable comparisons of their results difficult. Therefore, general conclusions were rarely formulated.

With respect to its participation to two broad European projects, namely EUROMBRA and MBR-Train, Delft University of Technology decided to organise a large filterability measurement campaign at a European scale.

Pilot and full-scale MBR plants of a large number of partners (company, research institute and university) were investigated using the DFCm. Reliable data concerning filterability of pilot and full-scale MBR plants have been collected under the same hydraulic circumstances. Results coming from each research group can then be relevantly and accurately compared. Furthermore, a set of analyses was performed in combination with each filterability measurement. Design, operational and membrane performance data were also collected for each MBR plant. Finally, each MBR plant was investigated at least twice, once during summer period and once during winter period, in order to get information on the modifications of the activated sludge morphological properties depending on the periods of the year (seasonal variations), and their impacts on the activated sludge filterability and the MBR performances.

3.2.2. MBR plant overview



Figure 20: Locations of the MBR plants investigated during the European DFCm tour

The location of each MBR investigated during the European DFCm tour is presented in Figure 20. Fifteen MBR plants were investigated in total, from Norway to Italy, passing by England, Germany, Belgium, the Netherlands and Switzerland.

General specifications of each MBR plant are presented in Table 5 and Table 6. Seven full-scale and eight pilot-scale MBRs were investigated during this study. MBRs were operated with a sludge age varying from 13 to 200 days, with nine of them operated at 25 days. Nine of the investigated MBRs were designed with a hollow fibre (HF) membrane configuration, four with a flat sheet (FS) membrane configuration and two with a multi-tube (MT) membrane configuration. Nine of the MBR plants in terms of design and performance characteristics will be discussed in *Chapter 4*.

Table 5: General data of the pilot plants

	AMEDEUS (D)	EAWAG B70 (CH)	EAWAG B80 (CH)	EAWAG B90 (CH)	ENREM MTB (D)	Trento (I)	Tromdheim (N)	Cranfield (UK)
Country	Germany	Switzerland	Switzerland	Switzerland	Germany	Italy	Norway	England
Scale	Pilot	Pilot	Pilot	Pilot	Pilot	Pilot	Pilot	Pilot
Туре	FS	FS	HF	HF	FS	HF	HF	MT / FS / HF
Brend	A3	Kubota	Zenon	Puron	Kubota	Zenon	Zenon	
Pore size (µm)	0.2	0.1	0.04	0.08	0.1	0.04	0.04	0,03 / 0,08 / 0,04
SRT (d)	13	13	13	13	200	25	27	20
SADm (m ³ .m ⁻² .h ⁻¹)	0.86	1.25	0.43	0.5	n.c.	0.4-0.5	0.65	variable
TSS (g.L ⁻¹)	5.3-9.0	6.1-13.6	8,98-12,5	8,20-11	5,1-6,4	7.5-10.2	0.3	5.15-12.3
Filterability samples	41	34	52	30	10	55	36	69

Table 6: General data of the full-scale plants

	ENREM MTK (D)	Heenvliet (NL)	Monheim (D)	Nordkanal (D)	Ootmarsum (NL)	Varsseveld (NL)	Schilde (B)
Country	Germany	The Netherlands	Germany	Germany	The Netherlands	The Netherlands	Belgium
Scale	Full Scale	Full Scale	Full Scale	Full Scale	Full Scale	Full Scale	Full Scale
Туре	FS	HF	HF	HF	MT	HF	HF
Brend	A3	Toray	Zenon	Zenon	NORIT	Zenon	Zenon
Pore size (µm)	0.2	0.08	0.04	0.04	0.03	0.04	0.04
SRT (d)	25	24	30	24	42	33	21
SADm (m ³ .m ⁻² .h ⁻¹)	0.33	0.44	0.1-0.3	0.4	n.c.	0.27	0.375
TSS (g.L ⁻¹)	14.5-16.5	11,2-16	6.8-10.1	11.6-14.8	9-9,5	9,5-11	6.9-13
Filterability samples	42	>100	52	68	72	74	98

3.3 Physical-chemical analyses

Each filterability experiment performed with the DFCm was accompanied by various physical-chemical analyses, namely mixed liquor suspended solids (MLSS), temperature, pH, dissolved oxygen concentration and soluble microbial product (SMP) characterised as proteins and polysaccharides in the free-water phase.

3.3.1. Activated sludge analyses

The analysis of MLSS (g.L⁻¹) was performed according to standard methods NEN6621. The temperature, pH and dissolved oxygen concentration were monitored during filtration experiments with standard electrodes (type WTW).

3.3.2. Soluble microbial products in the free-water phase

The free-water phase was defined as the filtrate of a paper filter with a nominal pore size between 7 and 12 μ m (Schleicher&Schuell 589²). SMP were quantified as the sum of the protein and the polysaccharide concentration present in the samples.

Proteins in the free-water phase were measured according to Te Poele's (2006) improved method. This is a variant of Rosenberger's method (2000), also a modified form of the photometric method of Lowry et al. (1951). Absorption of the formed colour is measured at 750 nm in a 4 cm glass cuvette. The amount of proteins is expressed in mg.L⁻¹.

Polysaccharides in the free-water phase were also using the method of Te Poele (2006). The colorimetric method is based on Dubois et al. (1956). The absorption of the formed colour is measured at 487 nm in a 4 cm glass cuvette. The amount of polysaccharides is expressed in $mg.L^{-1}$.

3.4 Statistical analyses

Statistical analyses were carried out using SPSS 16.0 (*SPSS Corporation*). The Pearson product momentum correlation coefficient was used to estimate linear correlations. Pearson's correlation coefficient is usually signified by r_p , and can take on the values in the range -1.0 to 1.0, where -1.0 is a perfect negative (inverse) correlation, 0.0 means an absence of a correlation, and 1.0 is a perfect positive correlation. The Pearson coefficient between -0.4 and 0.4 stands for weak correlation between two parameters and the interrelation can be ignored in this situation. In this study, correlations were considered statistically significant at a 95% confidence interval (p< 0.05).

3.5 General remarks

The DFCm tour was performed from February 2007 to November 2008. Each experimental period performed in the DFCm tour was lasting a week. This duration allows numerous measurements. The set of performed experiments was always started with a DFCm experiment performed at a flux of 80 L.m⁻².h⁻¹. In case of good filterability or poor filterability, experiments at 40 L.m⁻².h⁻¹ or 120 L.m⁻².h⁻¹ were also performed in order to improve the quality of the collected data set. However, the need for such measurements was exceptional.

The filterability was considered "steady" along a week of experiments when the filterability quality (defined as poor, moderate and good) remained identical between the different experiments performed. In case of filterability variations implying a change in terms of quality, i.e. from poor to moderate or moderate to good for instance, the filterability variations were defined as "significant".

Thanks to the long stay, the consistency of the measurements could also be checked by repeating several times the measurements on the same activated sludge sample in case of suspicious values. Unexpected events were also investigated and related operational data were collected directly in order to figure out their causes. However, filterability remained consistent along the week in most cases. Therefore, a "representative curve" was chosen empirically in order to compare the different experimental periods (see *Chapter 4*).

The general state of the MBRs investigated during this research was defined as "steady state" when the treatment efficiency of the MBR plants remained above the discharge limits set for the plants and if the permeability remained stable along the week of experiment. The state of the MBRs was qualified as "unsteady state" when the treatment efficiency requirements were not met and if strong drops in permeability were observed during the week of experiments.

4 Results

The filterability data and a large part of the data collected during the European DFCm tour will be presented in this chapter. The filterability data will be discussed and compared with the permeability of the MBR plants and the operating conditions in each plant separately.

4.1 Filtration characterisation at MBR Schilde



Schilde is located in the sub-urbs of Antwerpen. The MBR Schilde was commissioned in 2004. It is the first full-scale municipal MBR built in the Benelux. The MBR was built in order to retrofit the existing WWTP of Schilde and therefore increase the biological capacity of the wastewater treatment plant and achieve better nutrient removal. The MBR is operated by the Belgium Company Aquafin NV. Five sets of experiments were performed with the DFCm at Schilde MBR plant. Three of them were week-term experiments; they were performed in February 2007, April 2008, and August 2008. The two other experimental periods daily experiments performed were in November 2007 and April 2009. The MBR plant and the DFCm results are presented in this section.

4.1.1. Plant information

Background and MBR design

The wwtp Schilde serves a conglomeration of 28,000 inhabitants and is operated by Aquafin (Flemish wastewater Treatment Company). It is composed of two treatment lanes (Fenu et al., 2009):

The conventional activated sludge (CAS) lane was built in 1989 with a nominal capacity of 18,000 population equivalents (PE). The facility was built to comply with carbonaceous and particulate discharge regulations. The primary treatment consists of screens, a sand trap and rectangular primary clarifiers. Secondary treatment is achieved by a conventional single-stage activated sludge system (2x594 m³). Phosphorus is removed by simultaneous chemical precipitation. Activated sludge-effluent separation is achieved by three round settling tanks (total surface area of 993 m²).

The membrane bioreactor (MBR) lane was built in 2003 and commissioned in 2004 with the aim of meeting more stringent water quality regulations including nutrient removal implementation. The biological capacity was increased to 28,000 PE. The MBR lane is composed of a 1mm drum-sieve to protect the downstream system, a pre-denitrification tank (504 m³), an aeration basin (504 m³) and a filtration unit (200 m³). The filtration unit is composed of 4 Zenon MBR filtration trains having a total membrane surface area of 10,560 m² and being able to treat an average flow of 250 m³.h⁻¹ and maximum peak flow of 355 m³.h⁻¹. The filtration cycle lasts 330 s including 25 s backwash and 5 s relaxation. Maintenance cleanings with sodium hypochlorite are performed on weekly bases.

Detailed design and operational parameters are presented in Table 7. The scheme of the wwtp is presented in Figure 21.

As Aquafin NV was also involved in several Research projects (AMEDEUS and MBR-Train), the MBR was also used as an experimental tool for several research programs. Garces et al. (2007) reported cost optimisation tests performed at Schilde MBR plant. After pilot-scale experiments and implementation of the new optimised aeration cycle at full-scale, they could decrease the membrane aeration energy requirement with 35%. Furthermore, De Wilde et al. (2007) developed a predictive permeability model and concluded that membrane life expectancy at MBR Schilde will be a minimum of 11 years.



Figure 21: General scheme wwtp Schilde (Garces et al., 2007)

Schilde		Schilde	
Location	Belgium	Biological capacity (P.E.)	10000
Scale	full	Average flow (m ³ h ⁻¹)	230-355
Membrane type	hollow fibre	F/M ratio (kg _{BOD} kg _{MLSS} ⁻¹ d ⁻¹)	0.01-0.03
Membrane configuration	submerged	SRT (d)	21
Membrane supplier	Zenon ZW500c	HRT _{total} (h)	3.5-5.5
Membrane surface (m ²)	10560	V _{anoxic} (m ³)	504
Membrane pore size (µm)	0,04	V _{aerobic} (m ³)	504
Pretreatment (mm)	drum sieve 1mm	Recirculation aerobic to anoxic (m ³ h ⁻¹)	2160
Aeration (m ³ h ⁻¹)	3960	Recirculation ratio	8,6
Design net flux (Lm ⁻² h ⁻¹)	24-26	Chemical addition for denitrification (m ³ h ⁻¹)	Butyric acid
Energy consumption (kWh m ⁻³)	0.55-0.62	V _{MT} (m ³)	200 (4*50)
SADm (m ³ m ⁻² h ⁻¹)	0,375	Recirculation MT to aerobic (m ³ h ⁻¹)	1450
SADp (m ³ m ⁻³)	10-17.2	Recirculation ratio	5,8



Operating conditions and influent characteristics

Detailed characteristics and membrane performances of the MBR plant for the experimental periods are presented in Table 8. The MBR was under steady state conditions in February 2007 and August 2008 whereas an uncommon growth of *Nocardia amarae* (filamentous bacteria) was reported in November 2007 (no data available) and April 2009.

It is noticeable that a relatively large amount of COD was measured in the permeate of the MBR in April 2008 (76 mg.L⁻¹). At this period of the year, a malfunctioning of the carbon source addition for nutrient removal was reported. A too large amount of butyric acid was dosed in the anoxic tank during this experimental period resulting in strong and uncontrolled foaming events. As a result, the complete activated sludge in the MBR was intoxicated and needed to be removed and changed a month after our measuring campaign. This point will be discussed in *Chapter 6*.

	Schilde					
	Period	feb-07	apr-08	aug-08	apr-09	
Influent	COD (mg L ⁻¹)	175	140-200	170	140-195	
milluent	NH_4^+ (mg L ⁻¹)	17	15-19	8.0-13.1		
	COD (mg L ⁻¹)	27	76	23	15-16	
Permeate	$NH_4^+ (mg L^{-1})$	-	-	-	-	
	NO_{3}^{-1} (mg L ⁻¹)	1.9-2.7	2.2	2.2		
	P (mg L ⁻¹)	-	-	-	-	
TSS (g L ⁻¹)		6.9-7.9	11.1-11.5	10.5-13	8.4-12.9	
T(°C)		10.5-10.8	11.9-12.5	16.5-17.2	13.75-14.1	
Permeability (L m ⁻² h ⁻¹ Bar ⁻¹)		123	100-105	178	110	
М	BR state	steady	unsteady / ?	steady	foam	

Table 8: Detailed characteristics from the experimental periods at wwtp Schilde

4.1.2. Research approach

Objectives

The first experimental period performed at Schilde was the first test with the mobile DFCm unit outside of the TU Delft lab facilities. Therefore, the first aim of this experimental period was to investigate the feasibility and the reliability of the in-situ measurements with the new mobile unit.

The second aim of the filtration characterisations performed at MBR Schilde was to quantify the activated sludge filterability of a full-scale MBR plant and to investigate the impact of seasonal fluctuations.

Approach

The DFCm was installed directly at the MBR Schilde site during one week in February 2007, April 2008 and August 2008. The experiments performed in November 2007 and April 2009 were daily trials where an activated sludge sample was taken and brought back to the TU Delft lab facilities. Only the long-term monitoring (week of experiments) will be reported in detail in this section.

Each activated sludge filterability measurement was accompanied by the analyses summed up in Table 9.

Table 9: Activated sludge quality analyses

Analysis	Reference to method
Activated sludge	
Delft Filtration Characterisdation method (DFCm)	Chapter 3.1
Mixed Liquor Suspended Solid (MLSS)	Chapter 3.2
Free water	
Soluble Microbial Product (SMP) proteins	Chapter 3.2
Soluble Microbial Product (SMP) polysaccharides	Chapter 3.2

4.1.3. Filtration characterisation

First experimental period - February 2007

The activated sludge of MBR Schilde was only sampled in the first membrane tank line of the plant during the first experimental period. Filterability results are presented in Figure 22. The ΔR_{20} values are close to $1*10^{12}$ m⁻¹ during the whole week. The filterability can therefore be considered poor but steady along the week of experiments. The permeability data of the MBR plant are presented in Figure 23. They are in accordance with the filterability results. The permeability remained steady along the week with values around 120 L.m⁻².h⁻¹.Bar⁻¹.



Figure 22: Filterability data of the first experimental period



Figure 23: Permeability data in the full-scale plant of the first experimental period

At the end of this experimental period, the DFCm tests were considered a success. Reliable filterability data were intensively monitored directly on a full-scale MBR site. Furthermore, the unit was easy to carry and build directly on the MBR location.

Second experimental period - April 2008

The filterability data collected during the second experimental period are presented in Figure 24. Activated sludge was sampled in each membrane tank, i.e. MT1 to MT4, and in the recirculation loop in order to investigated filterability quality variations between tanks. The filterability can be considered poor in each membrane tank and during the whole experimental period. The ΔR_{20} values were always above $3*10^{12}$ m⁻¹. Heterogeneous results can be observed. Filterability data collected in each tank on the first day of experiment (15th of April) are similar. The same filterability is measured in each membrane tank and in the recirculation. However, significant variations can be observed between the different membrane tanks and the recirculation during the other days of the experimental period. However, no clear pattern can be identified. It is important to notice that ideally every membrane tanks should present the same filterability. It is likely that differences in filterability observed between the tanks.

The filterability data are in accordance with the operational state of the MBR. An uncontrolled foaming event was reported by the plant operators during this experimental period. Furthermore due to a toxic event (see *Chapter 6*), the activated sludge needed to be replaced a month after this experimental period.



Figure 24: Filterability data of the second experimental period

The permeability data are presented in Figure 25. Permeability values are close to 100 L.m⁻².h⁻¹.Bar⁻¹. In comparison with the activated sludge filterability quality monitored, it can be said that the MBR is performing relatively well. However, as already reported previously, the MBR was not still able to handle the foaming event due to the poisoning

of the activated sludge and operations had to be shut down and the activated sludge replaced a month after this experimental period.



Figure 25: Permeability data of the second experimental period

Third experimental period - August 2008

The third experimental period took place in order to investigate the seasonal fluctuations (summer period) in terms of filterability at MBR Schilde. The filterability data are presented in Figure 26. The filterability can be considered moderate in each tank and all along the week of experiments with ΔR_{20} comprised between 0.2 and 0.64*10¹² m⁻¹. The filterability can be considered steady along the week even if slight filterability variations are noticeable between each membrane tank.

The permeability data for each line of the MBR are presented in Figure 27. The permeability remained steady along the week and consistent with the filterability data in each line with values between 150 and 200 $L.m^{-2}.h^{-1}.Bar^{-1}$.



Figure 26: Filterability data of the third experimental period



Figure 27: Permeability data in the full-scale plant of the third experimental period

Experimental period comparison

A representative curve of each set of experiments is plotted along with the permeate production in Figure 28.



Figure 28: DFCm outputs concerning wwtp Schilde experimental periods.

Significant differences in activated sludge filterability can be observed between experimental periods. ΔR_{20} varied from $0.31*10^{12}$ m⁻¹ in August 2008 to $4*10^{12}$ m⁻¹ in April 2009. Filterability changed from poor to moderate between February 2007 and August 2008 whereas the filterability can be considered very poor in November 2007, April 2008 and April 2009.

The significant differences in terms of filterability observed between February 2007 and August 2008 can likely be considered seasonal variations due to the fact that in both cases the MBR was under steady-state conditions. The only relevant difference in operating parameters was the temperature of the wastewater changing from 10.5°C for February 2007 to 17.2°C in August 2008.

The filterability results from November 2007, April 2008 and April 2009 can mostly be explained by filamentous bacteria growth reported by the plant operators (after analyses) and activated sludge poisoning due to operational issues. This point will be discussed in *Chapter 6*.

4.2 Filtration characterisation at MBR Monheim



Monheim is located in the South of Germany. The building of an MBR was commissioned in order to comply with the low discharge limits in this area. Two sets of experiments were performed with the DFCm at Monheim MBR plant. They were performed in March 2007 and August 2008. As a preliminary experiment, an activated sludge sample coming from Monheim MBR had been shipped to Delft University of Technology for filterability characterisation in September 2006. The MBR plant and the DFCm results are presented in this section.

4.2.1. Plant information

Background and MBR design

The Monheim MBR is designed to treat the wastewater of 9,700 PE. The MBR technology was chosen for this plant due to peculiar soil and risks of infiltration of the water discharged by the wwtp. The MBR lane presented in Figure 29 is composed of a 1 mm sieve followed by a pre-denitrification tank (680 m³), an aerobic tank (680 m³) and a filtration unit (300 m³). The filtration unit is composed of four lines of 7 Zenon ZeeWeed 500c modules. The total membrane surface area is 12,320 m². The full-scale MBR is treating an average flow of 96 m³.h⁻¹ with a yearly average flux close to 8 L.m⁻².h⁻¹ (maximum net flux is 23.4 L.m⁻².h⁻¹). The permeate extraction cycle lasts 550 s, 500 s permeate extraction and 50 s backflush. The aeration is intermittent with cycle of 13 s. Phosphorus removal is enhanced thanks to poly-aluminium chloride addition.

Detailed operational parameters are presented in Table 10. The scheme of the pilot plant is presented in Figure 29.







Monheim			Monheim	
Location	Germany		Biological capacity (P.E.)	9700
Scale	full		Average flow (m ³ h ⁻¹)	96-480
Membrane type	hollow fibre		F/M ratio (kg _{BOD} kg _{MLSS} ⁻¹ d ⁻¹)	0.05-0.06
Membrane configuration	submerged		SRT (d)	30
Membrane supplier	Zenon ZW500c		HRT _{total} (h)	6.5-17
Membrane surface (m ²)	12320		V _{anoxic} (m ³)	680
Membrane pore size (µm)	0,04		V _{aerobic} (m ³)	680
Pretreatment (mm)	1mm sieves		Recirculation aerobic to anoxic (m ³ h ⁻¹)	1360
Aeration (m ³ h ⁻¹)	1250		Recirculation ratio	2.8-14.2
Design net flux (Lm ⁻² h ⁻¹)	8,1-23,1		Chemical addition	AICI ₃
Energy consumption (kWh m ⁻³)	1		V _{MT} (m ³)	300
SADm (m ³ m ⁻² h ⁻¹)	0.1-0.3		Recirculation MT to aerobic (m ³ h ⁻¹)	5120
SADp (m ³ m ⁻³)	12.5-36		Recirculation ratio	10.7-53.3

Operating conditions and influent characteristics

Detailed characteristics and membrane performances of the MBR plant for the experimental periods are presented in Table 11. The MBR was under steady state conditions during both experimental periods in March 2007 and September 2008. Treatment efficiency remained identical during both experimental periods. Fluctuations in MBR membrane performances are then likely due to seasonal variations.

	Monheim					
	Period	mrt-07	sep-08			
	COD (mg L ⁻¹)	440	455			
Influent	NH4 ⁺ (mg L ⁻¹)	18.4	29.9			
muent	PO4-P (mg L ⁻¹)	5	7.4			
	Flow (m ³ d ⁻¹)	4000	1300			
	COD (mg L ⁻¹)	20	15			
Permeate	NH_4^+ (mg L ⁻¹)	0.02	0.07			
i enneate	$NO_{3}^{-1}(mg L^{-1})$	6	4.1			
	P (mg L ⁻¹)	0.5	0.12			
	TSS (g L ⁻¹)	9-10,1	6,8-7,1			
	T(°C)	8,4-9,2	17,5-18,3			
Perme	ability (L m ⁻² h ⁻¹ Bar ⁻¹)	100-120	125-145			
	MBR state	steady	steady			

Table 11: Detailed characteristics from the experimental periods

4.2.2. Research approach

Objectives

The aim of the filtration characterisation performed at MBR Monheim was to quantify the activated sludge filterability fluctuations of a full-scale MBR plant and to investigate seasonal fluctuations especially.

Approach

The DFCm was installed directly at MBR Monheim site during one week for each experimental session. Each activated sludge filterability measurement was accompanied by the analyses summed up in Table 12.

Table 12: Activated sludge quality analyses

Analysis	Reference to method
Activated sludge	
Delft Filtration Characterisdation method (DFCm)	Chapter 3.1
Mixed Liquor Suspended Solid (MLSS)	Chapter 3.2
Free water	
Soluble Microbial Product (SMP) proteins	Chapter 3.2
Soluble Microbial Product (SMP) polysaccharides	Chapter 3.2

4.2.3. Filtration characterisation

First experimental period - March 2007

The filterability results of the samples taken in the membrane tank and the permeability data of the pilot plant for this experimental period are presented in Figure 30 and Figure 31, respectively. The filterability remained steady and can be considered moderate along the week with ΔR_{20} values close $0.60*10^{12}$ m⁻¹. The permeability of the plant remained also steady with values oscillating around 90 L.m⁻².h⁻¹.Bar⁻¹. The value of permeability (garrow in Figure 31) can be considered low in regards to the filterability quality.



Figure 30: Filterability data evolution along the first experimental period



Figure 31: MBR permeability data evolution along the first experimental period (plant operator data)

Second experimental period - September 2008

The filterability results of the samples taken in the membrane tank and the permeability data of the pilot plant for this experimental period are presented in Figure 32 and Figure 33, respectively. The filterability remained steady and can be considered good along the week with ΔR_{20} values close $0.1*10^{12}$ m⁻¹. The permeability of the plant remained also

steady and consistent with the filterability data with values oscillating around 135 L.m⁻²h⁻¹.Bar⁻¹(both experimental period data are indicated by two green arrows in Figure 33).



Figure 32: Filterability data evolution along the second experimental period



Figure 33: MBR permeability data evolution along the second experimental period (plant operator data)

Tank comparison

The filterability was monitored in each tank during the second experimental period. The filterability measurements are presented in Figure 34. No significant difference in filterability can be noticed depending on the sampled tanks. The filterability was homogenous along the whole process with ΔR_{20} values close $0.1*10^{12}$ m⁻¹. It is likely due to the high recirculation rates fixed between the membrane tank and the aerobic tank (10Q). The recirculation results in short contact time in the membrane tanks (10 min) and therefore a good homogenisation of the activated sludge in the process can be obtained.



Figure 34: Evolution of the filterability along the MBR process

Experimental period comparison



Figure 35: DFCm outputs concerning Monheim MBR plant experimental periods.

A representative curve of each set of experiment is plotted along with the permeate production in Figure 35. Significant differences in activated sludge filterability can be observed between experimental periods. ΔR_{20} values fluctuate between 0.07-0.1*10¹² m⁻¹ in September 2006 and August 2008 and 0.65*10¹² m⁻¹ in March 2007. Filterability can be considered very good in September 2006 and August 2008 and March 2007. The differences in filterability observed are likely due to seasonal variations.



4.3 Filtration characterisation at MBR EAWAG

The MBR plant of the EAWAG aquatic research institute is located in Dübendorf (ZW), close to Zurich. The MBR was built within the framework of the European project EUROMBRA in order to investigate the impact of the activated sludge loading on the membrane performances of MBRs equipped with different membrane configurations.

Three sets of experiments were performed with the DFCm at EAWAG MBR pilot plant. They were performed in May 2007 and during the second and fourth week of November 2007. The MBR plant and the DFCm results are presented in this section.

4.3.1. Plant information

Background and MBR design

The MBR plant was designed in order to investigate the membrane performances of different MBR configurations under short contact time conditions and organic peak load events. The pilot plant was therefore designed to compare different membrane configurations. Boehler et al. (2009) presented the results concerning long term permeability experiments. They concluded that high sludge loading, seasonal variations and foam events clearly impact permeability whereas moderate gross flux variations did not show significant effects. However, contradictory results were observed depending on the membrane configurations and unknown operational factors seem to have a relevant impact on membrane permeability. Zwickenpflug et al. (2009) also reported results from experiments about peak load effects on membrane permeability. They concluded that organic peak loads had a minor effect on permeability compared to other unidentified factors (activated sludge characteristics). The authors also observed that the fouling created during these events was mostly reversible.

The MBR pilot plant was designed to treat the equivalent of 100 PE. The MBR lane is composed of a 3 mm screen followed by an anoxic tank B50 (2 m³), an aerobic tank B60 (2 m³) and three membrane tanks in parallel. Each membrane tank is equipped with a specific membrane configuration. B70 membrane tank is equipped with a Kubota flat sheet module FS50 (40 m²), B80 membrane tank is equipped with a Zenon hollow fibre ZW500A module (46 m²) and B90 membrane tank is equipped with a Puron hollow fibre A-30-HS module (30 m²). The average flow treated by the pilot plant is 1.4-2 m³.h⁻¹.

filtration cycle lasts 600 s; 480 s permeate extraction and 120 s relaxation. Maintenance cleanings with sodium hypochlorite and citric acid are performed every two weeks. Detailed design and operational parameters are presented in Table 13. The scheme of the wwtp is presented in Figure 36.



Figure 36: General scheme of EAWAG MBR plant (Joss et al., 2008)

Zurich	B70	B80	B90
Location	Switzerland	Switzerland	Switzerland
Scale	Pilot	Pilot	Pilot
Membrane type	Flat sheet	Hollow fibre	Hollow fibre
Membrane configuration	submerged	submerged	submerged
Membrane supplier	Kubota	Zenon ZW500A	Puron
Membrane surface (m ²)	40	46	30
Membrane pore size (µm)	0,4	0,04	0,1
Pretreatment (mm)	3mm sieve	3mm sieve	3mm sieve
Aeration (m ³ h ⁻¹)	50	46	15
Design net flux (Lm ⁻² h ⁻¹)	12	10	16
Energy consumption (kWh m ⁻³)	5	5	5
SADm (m ³ m ⁻² h ⁻¹)	1,25	0,43	0,5
SADp (m ³ m ⁻³)	73	22	32

Table 13: General design and operational parameters at EAWAG MBR plant

Zurich	B70	B80	B90
Biological capacity (P.E.)	100 (total)	100 (total)	100 (total)
Average flow (m ³ h ⁻¹)	0.47	0.47	0.47
F/M ratio (kg _{BOD} kg _{MLSS} ⁻¹ d ⁻¹)	0.1-0.375	0.1-0.375	0.1-0.375
SRT (d)	13	13	13
HRT _{total} (h)	1-3.5	1-3.5	1-4.75
V _{anoxic} (m ³)	2	2	2
V _{aerobic} (m ³)	2	2	2
Recirculation aerobic to anoxic (m ³ h ⁻¹)	-	-	-
Recirculation ratio	-	-	-
Acetate addition for denitrification (m ³ h ⁻¹)	-	-	-
V _{MT} (m ³)	1.41	1.61	0.67(+1.48)
Recirculation MT to aerobic (m ³ h ⁻¹)	0.94	0.94	0.94
Recirculation ratio	2	2	2

Operating conditions and influent characteristics

Average characteristics and membrane performances of the MBR plant for the experimental periods are presented in Table 14 and Table 15. The MBR pilot plant was under high sludge loading conditions in May 2007 ($0.25 \text{ kg}_{COD}.\text{kg}_{TS}^{-1}.\text{d}^{-1}$). During both experimental periods performed in November 2007, the MBR pilot plant was running under low sludge loading conditions ($0.1 \text{ kg}_{COD}.\text{kg}_{TS}^{-1}.\text{d}^{-1}$). The volume of the B90 tank was also increased from 0.67 to 2.15m^3 between the second experimental period and the third experimental period. A foaming event was reported during the third experimental period resulting in a loss of TS in the whole MBR plant. No significant differences were observed in terms of treatment efficiency depending on the experimental period. However, large variations in MLSS content can be observed between tanks and experimental periods. It is likely due to difference in hydraulics between the membrane tanks. This point will be discussed in *Chapter 6*.

Zurich					
Period		Global	stdv		
Influent	COD (mg L ⁻¹)	412	91		
muent	$NH_4^+ (mg L^{-1})$	28.6	5.7		
Permeate	COD (mg L ⁻¹)	25-30	-		
	$NH_4^+ (mg L^{-1})$	0.7	0.7		
	NO_{3}^{-} (mg L ⁻¹)	2.4	1.4		
	P (mg L ⁻¹)	0.5	0.8		
TSS (g L ⁻¹)		6,1-13,6	-		
Т	(O°)	15,4-20,2	-		

Table 14: Average characteristics from the experimental periods

Zurich		B70			B80			B90	
Date	mei-07	nov-07	nov-07	mei-07	nov-07	nov-07	mei-07	nov-07	nov-07
Days of operation	150-153	341-344	352-356	150-153	341-344	352-356	150-153	341-344	352-356
MBR state	High load	low load	low load	High load	low load	low load	High load	low load	low load
V _{MT} (m ³)		1.41			1.61		0.67	0.67	2.15
HRT per configuration (min)	107	177	185	115	185	182	76	140	275
COD load (kg _{COD} .d ⁻¹)	16	10	9	16	10	9	16	10	9
Sludge loading (g _{COD} .Kg _{TSS} ⁻¹)	250	143	151	250	143	151	250	143	151
average TS (g.kg ⁻¹)	7.2	8.5	6.5	9.1	11.7	6.37	8.7	10.1	8.2
Temperature (°C)	19.4	15.6	16.1	19.4	15.6	16.1	19.4	15.6	16.1
Permeability (L m ⁻² h ⁻¹ Bar ⁻¹)	125	75-150	110	200	185-205	220	185	95	110

Table 15: Permeability data detailed characteristics for each membrane module

4.3.2. Research approach

Objectives

The aim of the filtration characterisation performed at EAWAG MBR plant was to quantify the activated sludge filterability fluctuations depending on the different membrane configurations. Then, the second aim of these experimental periods was to quantify the impact of sludge loading on activated sludge filterability.

Approach

The DFCm was installed directly on site during all the experimental periods. Each activated sludge filterability measurement was accompanied by the analyses summed up in Table 16.

Table 16: Activated sludge quality analyses

Analysis	Reference to method			
Activated sludge				
Delft Filtration Characterisdation method (DFCm)	Chapter 3.1			
Mixed Liquor Suspended Solid (MLSS)	Chapter 3.2			
Free water				
Soluble Microbial Product (SMP) proteins	Chapter 3.2			
Soluble Microbial Product (SMP) polysaccharides	Chapter 3.2			

4.3.3. Filtration characterisation

First experimental period - May 2007

During this experimental period the MBR pilot plant was under high sludge loading conditions. The filterability data for each tank are presented in Figure 37. Significant fluctuations depending on the membrane tank and the time of week are noticeable. The filterability quality can be considered poor in the aerobic tank, the tank B70 and B90 with ΔR_{20} values in average around $2*10^{12}$ m⁻¹, $1.1*10^{12}$ m⁻¹ and $1.0*10^{12}$ m⁻¹, respectively. The filterability in the tank B80 can be considered moderate with ΔR_{20} values varying between 0.1 and $0.84*10^{12}$ m⁻¹. In addition, large variations can be observed between measurements from the same tank, especially the tank B80.

Permeability data for each membrane configurations are presented in Figure 38. Permeability data are most of the time consistent with the filterability measurements. The higher permeability values were monitored for the B80 tank with values close to 200 L.m⁻².h⁻¹.Bar⁻¹, which also presents the best filterability. The tank B70 and B90 presented permeability values of 125 and 185 L.m⁻².h⁻¹.Bar⁻¹, respectively. However, whereas steady permeability was monitored along the week for the tank B80, noticeable variations were measured in terms of filterability.

As a last remark, the filterability quality measured in each membrane tank improved compared to the filterability measured in the aerobic tank (B60).



Figure 37: Filterability and data evolution along the first experimental period



Figure 38: Permeability data evolution along the first experimental period

Second experimental period - 2nd week of November 2007

During this experimental period the MBR pilot plant was under low sludge loading conditions. A chemical cleaning in each membrane tank was performed in the morning of the 15th of November 2007. The filterability data for each tank are presented in Figure 39.

Significant fluctuations depending on the membrane tank and the time of week are noticeable. The filterability quality can be considered poor in the aerobic tank and the tank B70 with ΔR_{20} values in average around $2.24*10^{12}m^{-1}$, $1.23*10^{12}m^{-1}$, respectively. The filterability in the tank B80 can be considered moderate with ΔR_{20} values varying between 0.1 and $0.48*10^{12}m^{-1}$. The filterability quality in the tank B90 varied from poor to good with ΔR_{20} values varying between 0.98 and $0.08*10^{12}m^{-1}$.

Permeability data for each membrane configurations are presented in Figure 40. The higher permeability values were monitored for the B80 tank, which also presents the best filterability. A membrane performance improvement due to the chemical cleaning was mostly significant in the tank B70 equipped with a Kubota membrane configuration with permeability values increasing from 75 to 150 L.m⁻².h⁻¹Bar⁻¹. A slight improvement in membrane performance was also noticeable in the tank B80 after the chemical cleaning (Zenon configuration) with permeability values varying from 185 to 205 L.m⁻².h⁻¹Bar⁻¹ whereas the permeability measured in the tank B90 (Puron) remained unaffected by the chemical cleaning with permeability values close to 95 L.m⁻².h⁻¹Bar⁻¹.

As a last remark, the filterability quality measured in each membrane tank improved compared to the filterability measured in the aerobic tank (B60).


Figure 39: Filterability data evolution along the second experimental period



Figure 40: Permeability data evolution along the second experimental period

Third experimental period - last week of November 2007

During this experimental period the MBR pilot plant was under low sludge loading conditions. However, a foaming event resulting in TS loss was reported for this experimental period.

The filterability data for each tank are presented in Figure 41. The filterability data measured during this experimental period are homogenous. No significant variations were noticeable between different membrane tanks along the week. The filterability quality can be considered poor in every tank with ΔR_{20} values close to $1*10^{12}$ m⁻¹.

Permeability data for each membrane configurations are presented in Figure 42. Permeability data are steady along the week. The tank B80 presents the best membrane performances with permeability data steady around 220 L.m⁻².h⁻¹Bar⁻¹ whereas the permeability data values for the B70 and B90 are stable around 110 L.m⁻².h⁻¹Bar⁻¹.

As a last remark, the filterability quality measured in each membrane tank can be considered identical to the filterability measured in the aerobic tank (B60).



Figure 41: Filterability data evolution along the third experimental period



Figure 42: Permeability data evolution along the third experimental period

Experimental period comparison

Average filterability values for each tank of the MBR pilot and for each significant event or experimental period are presented in Figure 43. The data reported concerning the experimental period of May 2007, November 2007 before and after chemical cleaning are mostly identical. Variations between each membrane tank and the aerobic tank are similar. This effect of the sludge loading will be discussed in *Chapter 6*.

As illustrated in Figure 44, the filterability data of the 4th week of November 2007 (3^{rd} experimental period) show significant changes in terms of filterability compared to the previous experiments. A homogenisation of the activated sludge filterability in the whole MBR can be observed. Whereas significant differences in terms of filterability were measured between the tanks during the previous experimental period, a homogenous filterability quality was monitored during the 4th week of November 2007. It is likely due to a foaming event which occurred during this period. This point will also be discussed in *Chapter 6*.



Figure 43: Average filterability measurements for each tank and each experimental period



Figure 44: Impact of the foaming event on the filterability in each tank (a) steady state operation (b) foaming event

Filterability variations in each tank depending on the experimental period

A representative curve for each membrane tank of each set of experiments is plotted along with the permeate production in Figure 45, Figure 46 and Figure 47 for the tank B70, B80 and B90, respectively.



Figure 45: Representative DFCm outputs for each experimental period concerning the B70 tank (Kubota)

Representative DFCm outputs of each experimental period concerning the tank B70 are presented in Figure 45. The filterability in this tank remains constant. Seasonal fluctuations, changes in sludge loading or incidental foaming events did not seem to have impacts on the filterability. The filterability remains identical during the different experimental periods.



Figure 46: Representative DFCm outputs for each experimental period concerning the B80 tank (Zenon)

Representative DFCm outputs of each experimental period concerning the tank B80 are presented in Figure 46. The filterability in this tank remains constant during the first two experimental periods. The changes in sludge loading do not seem to affect the activated sludge filterability in the tank. The foaming event is in contrary affecting negatively the activated sludge filterability.

Representative DFCm outputs of each experimental period concerning the tank B90 are presented in Figure 47. The filterability in this tank remains almost constant during the experimental periods. The seasonal variations (temperature variations) do not seem to have an impact on the filterability. The change in sludge loading seems to slightly affecting the activated sludge filterability. The increase of the contact time (tank volume) occurring during the third experimental period is positively affecting the filterability in this tank despite the foaming event reported.



Figure 47: Representative DFCm outputs for each experimental period concerning the B90 tank (Puron)

4.4 Filtration characterisation at MBR ENREM



The MBR ENREM is located in Margaretenhöhe in the sub-urb of Berlin where no connection to the sewage network is available. The MBR was built in order to test the potential of the MBR technology as semi-central treatment plant wastewater in an environmentally sensitive area. The MBR needs to ensure complete disinfection and advanced biological phosphorus removal down to 0.1mgP. L^{-1} in order to comply with European guidelines on bathing water.

Two sets of experiments were performed with the DFCm at ENREM MBR plant. They were performed in June 2007 and January 2008. The MBR plant and the DFCm results are presented in this section.

4.4.1. Plant information

Background and MBR design

The ENREM project (Enhanced Nutrients Removal in Membrane bioreactor) was aiming at demonstrating the feasibility of MBR applications for advanced treatments of wastewater in a sensitive and remote area, especially, high nutrient removal. During the project, Gnirss et al. (2003) identified and tested various phosphorous removal process combinations in order to establish efficient P-removal strategies for small sewage treatment units.

As a result, the demonstration MBR pilot plant was designed to treat the equivalent to 250 PE and enhanced biological phosphorus removal and nitrogen removal in a post-denitrification step.

The MBR lane is composed of a 1 mm sieve and solids cutting feed pump to protect downstream process , then follow an anaerobic tank (0.70 m³), two aerobic tanks (3.7 m³ in total), two anoxic tanks (3.86 m³ in total) and a filtration unit (2 m³). The filtration unit is composed of three A3 MBR filtration modules having a total surface area of 75 m² and being able to treat an average flow of 0.33 m³ h⁻¹ and maximum peak flow of 0.54 m³ h⁻¹. The filtration cycle lasts 1,146 s; 1,000 s permeate extraction and 146 s relaxation. Maintenance cleanings with sodium hypochlorite are performed every month.

The plant is designed to treat the complete incoming flow without any possibility of bypass. Therefore, storage tanks of 800 to 1,200 L were installed in each house and a buffer tank of 10 m^3 was built before the MBR plant in order to help to stabilise the

hydraulic flow and homogenize the composition of the influent entering the MBR. Thanks to this storage capacity, the MBR can be operated under constant and steady operating conditions. As reported by Gnirss et al. (2008), the combination of a buffer capacity and MBR can achieve high quality effluent under smooth filtration conditions. They demonstrated that a buffer tank flattened the hydraulic loading profile and provided and homogenisation rate of 25%.

Detailed design and operational parameters are presented in Table 17. The scheme of the wwtp is presented in Figure 48.



Overall design

Figure 48: General scheme of ENREM MBR (Gnirss et al., 2008))
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Table 17: General design and operational parameters at MBR ENREM

			ENREM	
I	ENREM		Biological capacity (P.E.)	250
	Location	Germany	Average flow (m ³ h ⁻¹)	0.33-0.54
	Scale	Pilot	F/M ratio (kg _{BOD} kg _{MLSS} ⁻¹ d ⁻¹)	0.11-0.12
	Membrane type	Flat sheet	SRT (d)	25
	Membrane configuration	submerged	HRT _{total} (h)	15-24
	Membrane supplier	A3	V _{anaerobic} (m ³)	0,7
	Membrane surface (m ²)	75	V _{anoxic} (m ³)	3,86
	Membrane pore size (µm)	0,2	V _{aerobic} (m ³)	3,71
	Pretreatment (mm)	1mm sieve	Recirculation anoxic to anaerobic (m ³ h ⁻¹)	3
	Aeration (m ³ h ⁻¹)	25	Recirculation ratio	1,5
	Design net flux (Lm ⁻² h ⁻¹)	16-19	Chemical addition (m ³ h ⁻¹)	Acetate
	Energy consumption (kWh m ⁻³)	6.0-9.0	V _{MT} (m ³)	2 (3*0.69)
	SADm (m ³ m ⁻² h ⁻¹)	0,33	Recirculation MT to aerobic (m ³ h ⁻¹)	1.66-2.7
	SADp (m ³ m ⁻³)	20,5	Recirculation ratio	4

Operating conditions and influent characteristics

Detailed information about the MBR plant concerning each experimental period are presented in Table 18. The MBR was under steady state conditions in June 2007 whereas a foaming event was observed and lead to unsteady membrane operations during the experimental period of January 2008. The plant loading remained in the same order of magnitude for both experimental periods. The major difference seems to be in the nitrification step. Higher nitrate concentration were measured in the permeate in January 2008. A nitrate concentration of 10.3 mg.L^{-1} was measured in June 2007 whereas the nitrate concentration was above 25 mg.L⁻¹ in January 2008. The low temperature conditions (around 12 °C) are likely to be responsible for the limitation of optimal nitrification step achievement.

As a last remark, the permeability values reported in this section can be considered relatively high. It is due to the fact that the operators in the plant normalised the permeability for temperature and pressure. They separated the TMP data monitored into the head loss caused by the membrane and the head loss caused by the pipes. The head loss due to the membrane is taken into account in their permeability calculations.

ENREM					
P	eriod	jun-07	jan-08		
Influent	COD (mg L ⁻¹)	1100	870-910		
IIIIucin	$NH_4^+ (mg L^{-1})$	120	110		
	COD (mg L ⁻¹)	45	40-45		
Permeate	$NH_4^+ (mg L^{-1})$	0	-		
Terricate	NO_{3}^{-1} (mg L ⁻¹)	10.3	25-30		
	P (mg L ⁻¹)	0.02	1,3-2		
TSS	6 (g L ⁻¹)	14,5-16,4	14,9-16,5		
T(°C)		20,8-24	11,9-13,1		
Permeability (L m ⁻² h ⁻¹ Bar ⁻¹)		843-1237	550-900		
MBI	R state	steady	foam		

Table 18: Detailed characteristics from each experimental periods

4.4.2. Research approach

Objectives

The aim of the filtration characterisation performed at MBR ENREM was to quantify the activated sludge filterability fluctuations of the demonstration MBR pilot plant and to figure out the impact of post-denitrification on activated sludge filterability. The second experimental period was also used to compare several filtration characterisation methods based on a study on four pilot-scale MBRs. The results were published by De la Torre et al. (2009) and will be discussed in *Chapter 5*.

Approach

The DFCm was installed directly on site during one week in June 2007. The DFCm was installed at University facilities close to the treatment plant in January 2008. Each activated sludge filterability measurement was accompanied by the analyses summed up in Table 19.

Table 19: Activated sludge quality analyses

Analysis	Reference to method
Activated sludge	
Delft Filtration Characterisdation method (DFCm)	Chapter 3.1
Mixed Liquor Suspended Solid (MLSS)	Chapter 3.2
Free water	
Soluble Microbial Product (SMP) proteins	Chapter 3.2
Soluble Microbial Product (SMP) polysaccharides	Chapter 3.2

4.4.3. Filtration characterisation

First experimental period - June 2007

The filterability results of the samples taken in the membrane tank and the permeability data of the pilot plant for this experimental period are presented in Figure 49 and Figure 50, respectively. The filterability remained steady and can be qualified as good along the week with ΔR_{20} values below $0.1*10^{12}$ m⁻¹. The normalised permeability of the plant remained also rather steady with values oscillating between 850 and 1200 L.m⁻²h⁻¹.Bar⁻¹. These good filterability and membrane performance results were likely due to the high temperature monitored during the week of experiments, namely 22 to 24 °C for the activated sludge temperature. The data are also in accordance with the general state of the MBR during this month of operation. The MBR plant was operating under steady state conditions and no unexpected events occurred during this experimental period.



Figure 49: Filterability data evolution along the first experimental period



Figure 50: MBR permeability data evolution along the first experimental period

Second experimental period - January 2008

The second experimental period was firstly planned in order to investigate the differences in filterability between warm and cold conditions. As already presented previously, the second experimental period was also used to compare several filtration characterisation methods based on a study on four pilot-scale MBRs. The results were published by De la Torre et al. (2009).



Figure 51: Filterability evolution along the second experimental period



Figure 52: MBR permeability evolution along the second experimental period

The filterability results of the samples taken in the membrane tank and the permeability data of the pilot plant for this experimental period are presented in Figure 51 and Figure 52, respectively. Variations in terms of filterability were noticeable with ΔR_{20} values

ranging from 1.16 to 3.27*10¹² m⁻¹. The filterability for this experimental period can be qualified as poor. The results are in accordance with the operational state of the MBR plant during this time. The MBR was under unsteady state conditions and subject to strong foaming events resulting in MLSS loss and unstable permeability data. The permeability of the plant oscillated between 550 and 900 L.m⁻²h⁻¹.Bar⁻¹. Strong variations in permeability values can be observed in Figure 52. However, the absolute permeability values remained high with respect to the operational conditions of the MBR.

Tank comparison

During the first experimental period, activated sludge samples were taken in different tanks of the MBR plant, namely the anoxic, the aerobic and the membrane tanks. The filterability data concerning each tank are presented in Table 20 and Figure 53.

Table 20: ∆R₂₀ values in each MBR tank

ENREM			
Tank	$\Delta R_{20} (*10^{12} \text{ m}^{-1})$		
Anoxic	0.028		
Aerobic	0.056		
Membrane	0.048		



Figure 53: Filterability evolution along the MBR process

Regarding the ΔR_{20} values, the filterability can be considered constant in the process. It is likely due to the two high recirculation rates fixed between the membrane tank and the aerobic tank (4Q) and the anoxic and the anaerobic tank (1.5Q). The two recirculations result in short contact times in each tank (around 40 min for the anaerobic, aerobic and anoxic tank and 15 min for the membrane tank) and therefore a good homogenisation of the activated sludge in the process can be obtained.

Experimental period comparison

A representative curve of each experimental period is plotted along with the permeate production in Figure 54.



Figure 54: Representative DFCm outputs concerning MBR ENREM experimental periods.

Significant differences in activated sludge filterability can be observed between the two experimental periods. ΔR_{20} varied from $0.05*10^{12}$ m⁻¹ in June 2007 to $1.5*10^{12}$ m⁻¹ in January 2008. The scattering of the data points for the experiment performed in June 2007 is due to the very good filterability measured. Due to the very small variations observed, each data point remained in the error detection limit of the sensors and therefore results in some noise in the measurements.

Filterability was good in June 2007 whereas the filterability can be considered poor in January 2008. The filterability differences are likely due to a combination of phenomena. Firstly, the state of the MBR was different during both experimental periods. The MBR was under steady state conditions in June 2007 whereas it was under a foaming event impacting the denitrification process in January 2008. Furthermore the temperature and seasonal fluctuations should be taken into account. The temperature of the activated sludge was around 12°C in June 2007 whereas it was around 22°C in January 2008.

4.5 Filtration characterisation at MBR AMEDEUS



The MBR pilot-scale plant investigated during this research period is located in Berlin close to the TU Berlin University. This pilot-scale plant was built within the framework of the AMEDEUS project.

Two sets of experiments were performed with the DFCm at AMEDEUS MBR pilot plant. They were performed in June 2007 and January 2008. The MBR plant and the DFCm results are presented in this section.

4.5.1. Plant information

Background and MBR design

Within the framework of AMEDEUS, the TU Berlin leads a comprehensive screening of 30 different flux enhancers in order to identify their potential for MBR filtration performance optimisation (Iversen et al., 2009a). Lab tests experiments were firstly performed to determine the effect of the additives on activated sludge respiration, nitrification, denitrification and activated sludge characteristics (Iversen et al., 2009b). Batch tests demonstrated that most of the tested substances did not inhibit oxygen uptake, nitrification and denitrification except polyaluminium chloride and powdered activated carbon due to a shift of pH.

Koseoglu et al. (2008) also performed filtration test cell experiments showing that synthetic polymers strongly enhanced the filtration compared to metal salts and natural polymers. After these lab trials, three additives were finally tested at pilot-scale. One specific type of synthetic polymer seems to enhance filtration on a long term. However, controversial results were found underlining the difficulties of scaling up research and of the additive concentration choices (Iversen et al., 2009b).

The MBR pilot plant used in this research was designed to treat the equivalent to 150 PE, fed with municipal domestic wastewater pumped directly from the Berlin sewage network. The pilot plant was built with two identical lines in order to investigate the impact of flux enhancers and different coagulants on membrane performances, MBR plant operations and removal efficiency.

Each MBR lane is composed of a sand trap followed by an anoxic tank (0.80 m³) and an aerobic tank (0.80 m³) with filtration modules directly immersed in it. Each filtration unit is composed of an A3 MBR filtration module having a total surface area of 22 m² and being able to treat an average flow of 0.20-0.22 m³ h⁻¹. The filtration cycle lasts 720 s; 600 s permeate extraction and 120 s relaxation. Maintenance cleanings are performed every 3 months.

Detailed design and operational parameters are presented in Table 21. The scheme of the wwtp is presented in Figure 55.



Figure 55: General scheme of AMEDEUS MBR (Iversen et al., 2009)

Table 21: General design and operational parameters at MBR AMEDEUS

AMEDEUS		AMEDEUS	
Location	Germany	Biological capacity (P.E.)	100-150
Scale	Pilot	Average flow (m ³ h ⁻¹)	0,20-0,22
Membrane type	Flat sheet	F/M ratio (kg _{BOD} kg _{MLSS} ⁻¹ d ⁻¹)	0.08-0.3
Membrane configuration	submerged	SRT (d)	13.3
Membrane supplier	A3	HRT _{total} (h)	7.3-8
Membrane surface (m ²)	22	V _{anoxic} (m ³)	0.8
Membrane pore size (µm)	0,2	V _{aerobic} (m ³)	0.8
Pretreatment (mm)	sand trap	Recirculation aerobic to anoxic (m ³ h ⁻¹)	1
Aeration (m ³ h ⁻¹)	19	Recirculation ratio	4-4.55
Design net flux (Lm ⁻² h ⁻¹)	16	Chemical addition (m ³ h ⁻¹)	various
Energy consumption (kWh m ⁻³)	6.0-8.0	V _{MT} (m ³)	-
SADm (m ³ m ⁻² h ⁻¹)	0,86	Recirculation MT to aerobic (m ³ h ⁻¹)	ŀ
SADp (m ³ m ⁻³)	52	Recirculation ratio	-

Operating conditions and influent characteristics

Detailed characteristics of the MBR plant for each experimental period are presented in Table 22. The MBR was under steady state conditions in June 2007 and flux enhancers were dosed in line 2. The MBR was under steady state conditions during the experimental period of January 2008. No unexpected events occurred during both experimental periods. The MBR presented identical treatment efficiency and close membrane performances for both experimental periods. No significant difference can be underlined.

AMEDEUS					
P	eriod	jun-07	jan-08		
Influent	COD (mg L ⁻¹)	1260	740-780		
mildent	$NH_4^+ (mg L^{-1})$	60	50		
	COD (mg L ⁻¹)	30-35	25-27		
Permeate	NH_4^+ (mg L ⁻¹)	0	0		
Termeate	NO_{3}^{-1} (mg L ⁻¹)	2,3-6	7,8-8		
	P (mg L ⁻¹)	5-7,5	3,6-9		
TSS	δ (g L ⁻¹)	5,3-5,8	8,2-9		
T(^o C)		24,6-26,8	17-18,7		
Permeability	(L m ⁻² h ⁻¹ Bar ⁻¹)	380-620	500-600		
MBR state		flux enhancers	steady		

Table 22: Detailed characteristics of each experimental period

4.5.2. Research approach

Objectives

The aim of the filtration characterisation performed during the first experimental period at AMEDEUS MBR was to quantify the activated sludge filterability fluctuations due to the flux enhancer additions. The aim of the second experimental session was to compare the filterability of both lines without flux enhancer addition and to investigate the filterability seasonal variations. The results of the second experimental period were also used in a joined study with the TU Berlin and the Kompetenzzentrum Wasser Berlin (KWB). The aim of the study was to assess several filtration characterisation methods. The results of this study were published by De la Torre et al. (2009) and will be discussed in *Chapter 5*.

Approach

The DFCm was installed for a week at the TU Berlin facilities close to the treatment plant in June 2007 and January 2008. Each activated sludge filterability measurement was accompanied by the analyses summed up in Table 23.

Table 23: Activated sludge quality analyses

Analysis	Reference to method
Activated sludge	
Delft Filtration Characterisdation method (DFCm)	Chapter 3.1
Mixed Liquor Suspended Solid (MLSS)	Chapter 3.2
Free water	
Soluble Microbial Product (SMP) proteins	Chapter 3.2
Soluble Microbial Product (SMP) polysaccharides	Chapter 3.2

4.5.3. Filtration characterisation

First experimental period - June 2007

During this experimental period additives were dosed in Line 2 (presented as AX2 and AE2 for the data from the anoxic and aerobic tanks, respectively). The Line 1 was operated without any chemical substance addition in order to be used as reference.

Results concerning the filterability experiments in each tank performed along the week are presented in Figure 56. The filterability can be qualified as moderate for the whole period and for each tank with ΔR_{20} value comprise between 0.15 and 0.37*10¹² m⁻¹. The filterability monitored in the anoxic tank of the line 1 (AX1) increased slightly during the experimental period.

The filterability variations monitored along the week in both lines were in accordance with the permeability measurements presented in Figure 57. The permeability values decreased when the filterability got worse.

Based on the data collected on the 19^{th} of June 2007, the filterability was better in the MBR line 1 compared to MBR line 2, with ΔR_{20} values around 0.2 and $0.37*10^{12} \text{ m}^{-1}$, respectively. Therefore, it is likely that the dosage of this specific flux enhancer in the MBR line 2 at this specific concentration has a detrimental effect on the membrane filtration performances of the MBR plant.

As a next point of comparison, the ΔR_{20} values measured in the aerobic tank of each line (AE1 and AE2) are lower than the one in the anoxic tank. The filterability in both line improved in the aerobic tank.



Figure 56: Evolution of the filterability along the first experimental period



Figure 57: Evolution of the MBR permeability along the first experimental period

Second experimental period - January 2008

During this second experiment session the filterability was only monitored in the aerobic tank of both lines. Filterability and permeability data monitored along the week are presented in Figure 58 and Figure 59, respectively. The filterability can be considered poor and remained steady along the week of measurements where both lines showed close behaviour with ΔR_{20} values around $1*10^{12}$ m⁻¹. It is accordance with the pilot plant permeability data monitored during the experimental period. The permeability of the MBR plant remained steady in both lines during the experiments with a value close to 550 L.m⁻².h⁻¹.Bar⁻¹. The pilot plant could therefore be considered in a steady state.



Figure 58: Evolution of the filterability along the second experimental period



Figure 59: Evolution of the MBR permeability along the second experimental period

Experimental period comparison

A representative curve of each set of experiment is plotted along with the permeate production in Figure 60.



Figure 60: DFCm outputs concerning both experimental periods.

Significant differences in activated sludge filterability can be observed between experiments. ΔR_{20} varied from $0.18*10^{12} \text{ m}^{-1}$ in June 2007 to $0.98*10^{12} \text{ m}^{-1}$ in January 2008. Filterability was moderate in June 2007 whereas the filterability can be considered poor in January 2008.

Furthermore a difference in filterability is noticeable between tanks in June 2007. The activated sludge from the line 1 presented a ΔR_{20} value close to $0.18*10^{12} \text{ m}^{-1}$ whereas the ΔR_{20} of the line 2 is close to $0.31*10^{12} \text{ m}^{-1}$. Besides the fact that no flux enhancers addition were taking place in January 2008, the major differences between the two set of experiments are the MLSS content and the activated sludge temperature. The temperature was around 25°C in June 2007 and closed to 17°C in January 2008. We can observe that it is still a warm sludge for winter time. It is mostly due to the location of the pumps. The pumps are heating up the activated sludge tank due to the fact that the complete MBR set up is located in a single container. The MLSS content varied from 5.5.g.L⁻¹ to 8.5 g.L⁻¹ between June 2007 and January 2008.



4.6 Filtration characterisation at MBR Nordkanal

The MBR Nordkanal in located in Kaarst in North Rhine-Westphalia (Germany). It started operation in the beginning of 2004 and is operated by the Erftverband. It is the largest fullscale MBR in Europe. Two sets of experiments were performed with the DFCm at Nordkanal MBR plant. They were performed in July 2007 and November 2008. The MBR plant and the DFCm results are presented in this section.

4.6.1. Plant information

Background and MBR design

The Nordkanal full-scale MBR is operated by Erftverband and is designed to treat the wastewater of 80,000 PE. The composition of the influent can be considered standard for German municipal wastewater (Lyko et al., 2007). The MBR lane presented in Figure 61 is composed of a 6 mm screen, an aerated sand-grit chamber and then 1 mm sieve followed by a pre-denitrification tank (3,525 m³), an aerobic tank (5,789 m³). The full-scale MBR is operated with simultaneous sludge stabilisation and pre-dinitrification. The Zenon ZeeWeed 500c filtration units are directly immersed in the nitrification tank. The nitrification tank is separated in four lines containing in total 84,840 m² of membrane surface area. The full-scale MBR is treating an average daily flow of 1,024 m³ h⁻¹. The permeate extraction cycle lasts 450 s, 400 s permeate extraction and 50 s backflush. The aeration is intermittent with cycle of 13 s. Phosphorus removal is enhanced chemically by ferric chloride addition. Detailed design and operational parameters are presented in Table 24.

The full-scale MBR was also used for research purposes. Lyko et al. (2008) presented results related with a long term experiment. They reported pronounced seasonal variations due to the colloidal fraction of the sludge and suggested that cleaning frequency adjustments should be implemented during low temperature periods. Different cleaning strategies were also tested at Nordkanal MBR in order to delay the intensive membrane ex-situ chemical cleaning (Brepols et al., 2008).





Nordkanal		Nordkanal	
Location	Germany	Biological capacity (P.E.)	80000
Scale	Full	Average flow (m ³ h ⁻¹)	750-1180
Membrane type	hollow fibre	F/M ratio (kg _{BOD} kg _{MLSS} ⁻¹ d ⁻¹)	0.04
Membrane configuration	submerged	SRT (d)	25-28,6
Membrane supplier	Zenon ZW500c	HRT _{total} (h)	8-12.4
Membrane surface (m ²)	84480	V _{anoxic} (m ³)	3525
Membrane pore size (µm)	0,04	V _{aerobic} (m ³)	5789
Pretreatment (mm)	1mm sieves	Recirculation aerobic to anoxic (m ³ h ⁻¹)	3320
Aeration (m ³ h ⁻¹)	34000	Recirculation ratio	4.0
Design net flux (Lm ⁻² h ⁻¹)	12,0-23	Chemical addition	FeCl₃
Energy consumption (kWh m ⁻³)	0,9	V _{MT} (m ³)	-
SADm (m ³ m ⁻² h ⁻¹)	0,4	Recirculation MT to aerobic (m ³ h ⁻¹)	-
SADp (m ³ m ⁻³)	17	Recirculation ratio	-

Table 24: General design and operational parameters at MBR Nordkanal

Operating conditions and influent characteristics

Detailed characteristics and membrane performances of the MBR plant for the experimental periods are presented in Table 25. The MBR was under steady state during both experimental periods in July 2007 and November 2008. No significant membrane performance fluctuations were monitored in both experimental periods. The treatment efficiency remained identical between both experimental periods. The slight decrease in permeability observed during winter period is likely due to temperature and seasonal variations.

Nordkanal				
	Period	jul-07	nov-08	
Influent	COD (mg L ⁻¹)	1017-1210	997-1118	
mucht	$NH_4^+ (mg L^{-1})$	38.9	38.9	
	PO4-P (mg/L)	8.7	8.7	
	COD (mg L ⁻¹)	22.1	17.4	
Permeate	NH4 ⁺ (mg L ⁻¹)	0	0	
1 cimeate	NO ₃ ⁻ (mg L ⁻¹)	3.5	2.57	
	P (mg L ⁻¹)	0.26	0.3	
TS	S (g L ⁻¹)	14,3-14,8	11,6-12	
T(°C)		17,8-19,5	9,5-12,7	
Permeabilit	Permeability (L m ⁻² h ⁻¹ Bar ⁻¹)		150-175	
MBR state		steady	steady	

Table 25: Detailed characteristics from each experimental period

4.6.2. Research approach

Objectives

The aim of the filtration characterisation performed at MBR Nordkanal was to quantify the activated sludge filterability fluctuations of a full-scale MBR plant and to investigate seasonal fluctuations especially.

Approach

The DFCm was installed directly at MBR Nordkanal site during one week for each experimental session. Each activated sludge filterability measurement was accompanied by the analyses summed up in Table 26.

Table 26: Activated sludge quality analyses

Analysis	Reference to method
Activated sludge	
Delft Filtration Characterisdation method (DFCm)	Chapter 3.1
Mixed Liquor Suspended Solid (MLSS)	Chapter 3.2
Free water	
Soluble Microbial Product (SMP) proteins	Chapter 3.2
Soluble Microbial Product (SMP) polysaccharides	Chapter 3.2

4.6.3. Filtration characterisation

First experimental period - July 2007

The filterability results of the samples taken in the membrane tank and the permeability data of the pilot plant for this experimental period are presented in Figure 62 and Figure 63, respectively. The filterability increased slightly but can still be qualified as good along the week with ΔR_{20} values around $0.1*10^{12}$ m⁻¹. The permeability of the plant remained also steady with values oscillating around 200 L.m⁻².h⁻¹.Bar⁻¹. The good filterability and membrane performance results were likely due to the high temperature monitored during this week of experiments, namely 17.8 to 21°C for the activated sludge temperature. These data are also in accordance with the general state of the MBR during

this month of operation. The MBR plant was operating under steady state conditions and no unexpected events occurred during this experimental period.



Figure 62: Filterability data evolution along the first experimental period



Figure 63: MBR permeability data evolution along the first experimental period

Second experimental period - November 2008

The filterability results of the samples taken in the membrane tank and the permeability data of the pilot plant for this experimental period are presented in Figure 64 and Figure 65, respectively. The filterability remained relatively steady and can be considered moderate along the week with ΔR_{20} values between 0.28 and $0.85*10^{12}$ m⁻¹. The permeability of the plant remained steady with values close to 170 L.m⁻².h⁻¹.Bar⁻¹. The data are in accordance with the general state of the MBR during this month of operation and the period of the year. The MBR plant was operating under steady state conditions and no unexpected events occurred during this experimental period.



Figure 64 : Filterability data evolution along the second experimental period



Figure 65: MBR permeability data evolution along the second experimental period

Experimental period comparison

A representative curve of each set of experiment is plotted along with the permeate production in Figure 66.



Figure 66: DFCm outputs concerning Nordkanal MBR plant experimental periods.

Significant differences in activated sludge filterability can be observed between experiments. ΔR_{20} values changed from $0.08*10^{12}$ m⁻¹ in July 2007 to $0.47*10^{12}$ m⁻¹ in November 2008. Filterability can be considered good in July 2007 and moderate in November 2008. In regard to the MBR state during both experimental periods, the fluctuations in filterability are likely due to seasonal variations and temperature differences.

4.7 Filtration characterisation at MBR Trondheim



The MBR Trondheim is located in the lab facilities of the department of hydraulic and environmental engineering of the Norwegian University of Science and Technology (NTNU). The small pilot-scale plant was developed in order to assess the potential of the combination of the bed biofilm reactor with a membrane step. A set of experiments was performed with the DFCm at Trondheim MBR plant. It was performed in October 2007. The MBR plant and the DFCm results are presented in this section.

4.7.1. Plant information

Background and MBR design

The moving bed biofilm reactor (MBBR) is a process developed at NTNU Norway. It consists of small plastic biofilm carriers used to create a large surface area for the biofilm to grow on. The carriers are suspended by aeration (Leiknes et al., 2007). As a result the concentration of activated sludge in suspension in the free water phase (MLSS) can be considered rather low. This process can achieve high soluble organic matter biodegradation. This technology was then coupled with a submerged membrane reactor. Ivanovic et al. (2006) investigated the effect of the loading rate on the submicron particle production and characteristics. Results showed that high loading rate conditions were detrimental for membrane performances and were resulting in higher submicron particle production and undesirable floc structures. Some further researches were practised in order to optimise the aeration in the MBBR. Ivanovic et al. (2008) proposed an approach to define optimal operating conditions between minimisation of membrane fouling and colloidal particle formation due to shear intensity.

The MBR is composed of a primary clarifier, 3 aerobic tanks (0.18 m³ in total) and a filtration unit (0.027 m³). The filtration unit is composed of Zenon Zeeweed 10 module with a total surface area of 1 m² and being able to treat an average flow of 0.031 m³ h⁻¹. The filtration cycle lasts 285 s including 15 s backwash. Maintenance cleanings with sodium hypochlorite are performed every month.

Detailed design and operational parameters are presented in Table 27. The scheme of the pilot plant is presented in Figure 67. The complete set up was composed of two lines.

However, only the line with three reactors in series was investigated during this experimental period.



Figure 67: General scheme of Trondheim MBR pilot plant

Table 27: General design and operational parameters at MBR Trondheim

Trondheim		Trandhaim	
Location	Norway		
Scale	nilot	Biological capacity (P.E.)	5,1-8
Mambrana tuna	hallow fibro	Average flow (m ³ h ⁻¹)	0.031
iviendrane type		F/M ratio (kg _{BOD} kg _{MLSS} ⁻¹ d ⁻¹)	0.01-0.03
Membrane configuration	submerged	SRT (d)	-
Membrane supplier	Zenon ZW10	HRT _{total} (h)	2.0-6.0
Membrane surface (m ²)	1,86	V_{rest} (m ³)	-
Membrane pore size (μm)	0,04	V_{correlia} (m ³)	0.26
Pretreatment (mm)	primary sedimentation	Recirculation aerobic to anoxic (m ³ h ⁻¹)	-
Aeration (m ³ h ⁻¹)	1,21	Recirculation ratio	-
Design net flux (Lm ⁻² h ⁻¹)	35	Acetate addition for denitrification (m ³ h ⁻¹)	-
Energy consumption (kWh m ⁻³)	-	V _{MT} (m ³)	0.04
SADm (m ³ m ⁻² h ⁻¹)	0,65	Recirculation MT to aerobic (m ³ h ⁻¹)	-
SADp (m ³ m ⁻³)	18,7	Recirculation ratio	-

Operating conditions and influent characteristics

Detailed characteristics and membrane performances of the MBR plant for the experimental period are presented in Table 28. The MBR was under steady state conditions during the experimental period.

Trondheim		
Period		okt-07
Influent	COD (mg L ⁻¹)	290-495
	$NH_4^+ (mg L^{-1})$	16,7-25
Permeate	COD (mg L ⁻¹)	25-40
	$NH_4^+ (mg L^{-1})$	-
	NO_{3}^{-} (mg L ⁻¹)	13,4-18,2
	P (mg L ⁻¹)	-
TSS (g L ⁻¹)		-
T(°C)		12,1-15,3
Permeability (L m ⁻² h ⁻¹ Bar ⁻¹)		200-320
MBR state		steady

Table 28: Detailed characteristics from the experimental periods of Trondheim MBR

4.7.2. Research approach

Objectives

The aim of the filtration characterisation performed at MBR Trondheim was to investigate the activated sludge filterability of the biofilm process. Furthermore, investigations about the impact of the hydraulic retention time on activated sludge filterability were practised during this experimental period.

Approach

The DFCm was installed directly at MBR Trondheim site. Each activated sludge filterability measurement was accompanied by the analyses summed up in Table 29.

Table 29: Activated sludge quality analyses

Analysis	Reference to method
Activated sludge	
Delft Filtration Characterisdation method (DFCm)	Chapter 3.1
Mixed Liquor Suspended Solid (MLSS)	Chapter 3.2
Free water	
Soluble Microbial Product (SMP) proteins	Chapter 3.2
Soluble Microbial Product (SMP) polysaccharides	Chapter 3.2

4.7.3. Filtration characterisation

Experimental period - October 2007

The filterability results of the samples taken in the membrane tank and the permeability data of the pilot plant for this experimental period are presented in Figure 68 and Figure 69, respectively. The filterability quality can be considered moderate along the week with ΔR_{20} values ranging from 0.38 to $0.91*10^{12}$ m⁻¹. The filterability followed the permeability pattern which decreased from 320 to 200 L.m⁻².h⁻¹.Bar⁻¹. The MBR plant was operating under steady state conditions and no unexpected events occurred during the experimental period.



Figure 68: Filterability data along the experimental period



Figure 69: MBR permeability data along the experimental period

Tank comparison – hydraulic retention time comparison

The Trondheim MBR pilot plant is composed of three aerobic tanks in series. Therefore, it was possible to monitor the mixed liquor filterability along the purification process.

The retention of time in each tank was equal to 2 h and the filterability could then be presented as a function of the HRT of the MBR.



Figure 70: DFCm output for different hydraulic retention times

The fluctuations in activated sludge filterability depending on different hydraulic retention times in the MBR are plotted in Figure 70. ΔR_{20} varied from $0.7*10^{12} \text{ m}^{-1}$ to $1.87*10^{12} \text{ m}^{-1}$ depending on the retention time in the system. The filterability quality varied from poor to moderate along the MBR process. The longest the retention time in the system, the best the filterability. This point will be discussed in *Chapter 6*.



4.8 Filtration characterisation at MBR Trento

The MBR Trento pilot plant was operated continuously between September 2005 and September 2006 and is located at the full-scale plant of Lavis (30,000 PE) located 12 km north of Trento.

Two sets of experiments were performed with the DFCm at Trento MBR plant. They were performed in November 2007 and July 2008. The MBR plant and the DFCm results are presented in this section.

4.8.1. Plant information

Background and MBR design

The MBR plant of Trento was used to conduct some long term experiments especially within the framework of the European project EUROMBRA. Guglielmi et al. (2007) investigated permeability recovery using the flux step method. They concluded that aeration can significantly affected permeability recovery and help preventing fouling during peak hydraulic loads. They also concluded that the feedwater quality was the most determinant factor affecting fouling rate under sub-critical flux conditions. A model based on experiments performed at MBR Trento was proposed by Saroj et al. (2008). Their model enables them to predict accurately severe fouling occurrence (TMP jump) during subcritical flux operations.

The large MBR pilot plant (150-200 PE) is operated at the full-scale plant of Lavis (30,000 PE) located 12 km north from Trento. The influent is mainly coming from municipal sewage (80%) but a significant fraction (20%) of the total COD loading is coming from industrial discharges (landfill leachate and winery wastewater). Ferric chloride (FeCl₃) is dosed upstream of the full-scale wastewater treatment plant in order to promote chemical phosphorus precipitation. As a consequence, the MBR pilot plant influent contains FeCl₃.

The MBR lane presented in Figure 71 and Figure 72 (depending on the experimental period) is composed of a 2 mm fine screen, a grit chamber followed by a predenitrification tank (4.7 m³), an aerobic tank (8.7 m³) and a filtration unit (1.5 m³). The filtration unit in November 2007 (Figure 71) was composed of a Zenon ZeeWeed 500d and an Eidos (polypropylene, 1 mm pore size) filtration system for a total surface area of 100 m². The Eidos filtration system was removed in January 2008 and was replaced by a Zenon ZeeWeed 500d (Figure 72). The pilot plant is able to treat an average flow from 1.5 to 2.2 m³ h⁻¹. The permeate extraction regime is an alternate relaxation (1 min) followed by a suction phase (9 min). Intermittent aeration and mechanical cleaning is achieved by means of air bubble scouring with a specific air flow rate of 0.4-0.5 Nm³ m⁻².h⁻¹. Detailed design and operational parameters are presented in Table 30.



Figure 71: General scheme Trento MBR in November 2007 (Guglielmi et al., 2007)



Figure 72: General scheme Trento MBR in July 2008
Trento			
Location	Italy		
Scale	Pilot		
Membrane type	hollow fibre		
Membrane configuration	submerged		
Membrane supplier	Zenon ZW500d / Eidos	Trento	
Membrane surface (m ²)	100	Biological capacity (P.E.)	150-200
Membrane pore size (µm)	0.04 / 0.1	Average flow (m ³ h ⁻¹)	1.5-2.2
Pretreatment (mm)	2 mm fine screen	SRT (d)	23.8-25.6
Aeration (m ³ h ⁻¹)	60	HRT _{total} (h)	7-9.5
Design net flux (Lm ⁻² h ⁻¹)	10.5-15	V _{anoxic} (m ³)	4.7
Energy consumption (kWh m ⁻³)	0.2-0.3	V _{aerobic} (m ³)	8.7
SADm (m ³ m ⁻² h ⁻¹)	0.4-0.5	V _{MT} (m ³)	1.5
SADp (m ³ m ⁻³)	17-20	Recirculation ratio	5.9-11.1

Table 30: General design and operational parameters at MBR Trento

Operating conditions and influent characteristics

Detailed characteristics and membrane performances of the MBR plant for the experimental periods are presented in Table 31. The MBR was under steady state conditions in November 2007. In contrary, low membrane performances were monitored in July 2008. A higher contribution of industrial wastes (landfill leachate) was measured in the influent composition in July 2008. The influent specificity is likely to be responsible for the low MBR membrane performances observed during this experimental period. As a consequence, lower treatment efficiency can be observed in terms of COD and nitrogen removal.

P	Period	nov-07	jul-08
Influent	COD (mg L ⁻¹)	270-377	535
	$NH4^+$ (mg L^{-1})	14.2-19.8	35-48.4
	13-31	47	
Permeate	$NH4^+$ (mg L^{-1})	0.4-3	4.6
T cimeate	NO3 ⁻ (mg L ⁻¹)	6.3-13.1	24.2
	0.8-2.2	0.1	
TSS	S (g L ⁻¹)	9.6-10.2	7.50
٦	12.0-13.4	23.5-26.5	
Recirculation from	10	8	
F/M ratio (kg	0.16-0.277	0.12-0.21	
Q _{zenc}	0.8-1.5	0.8-1.5	
Q _{eido}	0-1.4	0	
Permeability	r (L m ⁻² h ⁻¹ Bar ⁻¹)	70-80	42
MB	steady	leachate	

Table 31: Detailed characteristics from the experimental periods

4.8.2. Research approach

Objectives

The aim of the filtration characterisation performed at MBR Trento was to quantify the activated sludge filterability fluctuations of a pilot-scale MBR plant depending on

seasonal and influent variations. Therefore, the results presenting in this section are only the one concerning the membrane tank equipped with the Zenon membrane configuration.

Approach

The DFCm was installed directly at MBR Trento site during one week for each experimental period. Each activated sludge filterability measurement was accompanied by the analyses summed up in Table 32.

Table 32: Activated sludge quality analyses

Analysis	Reference to method
Activated sludge	
Delft Filtration Characterisdation method (DFCm)	Chapter 3.1
Mixed Liquor Suspended Solid (MLSS)	Chapter 3.2
Free water	
Soluble Microbial Product (SMP) proteins	Chapter 3.2
Soluble Microbial Product (SMP) polysaccharides	Chapter 3.2

4.8.3. Filtration characterisation

First experimental period - November 2007

The filterability results of the samples taken in the membrane tank and the permeability data of the pilot plant for this experimental period are presented in Figure 73 and Figure 74, respectively. The filterability remained steady and can be considered poor along the week with ΔR_{20} values close to $1*10^{12}$ m⁻¹. The permeability of the plant remained also steady with values oscillating between 70 and 85 L.m⁻².h⁻¹.Bar⁻¹. Permeability data and filterability data are consistent, i.e. poor filterability quality and poor membrane performances, even if the permeability values can be considered low. It is likely due to the combination of cold temperature ($12^{\circ}C$ - winter period) and the mixed influent entering the treatment plant.



Figure 73: Filterability data along the first experimental period



Figure 74: MBR permeability data along the first experimental period

Second experimental period - July 2008

The filterability results of the samples taken in the membrane tank and the permeability data of the pilot plant for this experimental period are presented in Figure 75 and Figure 76, respectively. The filterability remained steady and can be considered poor along the

week with ΔR_{20} values varying between 1.91 and $3.1*10^{12} \text{ m}^{-1}$. The permeability of the plant remained also steady with a constant value of 42 L.m⁻²h⁻¹.Bar⁻¹. Permeability data and filterability data are consistent, i.e. poor filterability and poor membrane performances, even if the permeability values can be considered low, especially in regard to the temperature of the activated sludge and the period of the year. The major explanation for this low membrane performances advanced by the plant operators was a change in the influent composition. This point will be discussed in *Chapter 6*.



Figure 75: Filterability data along the first experimental period



Figure 76: MBR permeability data along the first experimental period

Experimental period comparison

A representative curve of each set of experiment is plotted along with the permeate production in Figure 77.



Figure 77: DFCm outputs concerning Trento MBR pilot plant experimental periods.

Significant differences in activated sludge filterability can be observed between experiments. Extrapolated ΔR_{20} values were equal to $1.38 \times 10^{12} \text{m}^{-1}$ in November 2007 and $2.49 \times 10^{12} \text{m}^{-1}$ in July 2008. Filterability can be considered very poor in both cases. Whereas the expected filterability behaviour would be a better filterability in summer compared to winter time, the contrary results were found in MBR Trento. This point will be discussed in *Chapter 6*.



4.9 Filtration characterisation at MBR Cranfield

The MBR Cranfield is located in the Pilot hall of the Centre for Water of Cranfield University (UK). The pilot plant was built within the framework of the European project EUROMBRA in order to investigate and compare the impact of different operating conditions on membrane performances of different membrane configurations.

Two sets of experiments were performed at MBR Cranfield with the DFCm. They were performed in February 2008 and March 2008. The MBR plant and the DFCm results are presented in this section.

4.9.1. Plant information

Background and MBR design

The pilot plant research focuses on a direct comparison of three different full-scale sized membrane configurations operated in parallel as air-lift sidestream membrane module. The MBR is composed of a 2.2 m^3 aeration tank. Then, three air-lift sidestream modules are operated, namely a multi-tubular membrane (MT), a single flat sheet module (FS) and a hollow fibre module (HF). Two special vessels were designed to facilitate the operation of FS and HF membrane in an air-lift sidestream mode. Simulative separation distances prevalent in submerged modus were chosen for their design. For hydrodynamic comparison the filtration path length of each membrane module was fixed to 1.45 m. As a remark, it is important to note that the same air-lift velocity fixed may result in different hydraulic flow distributions in each membrane tank due to the differences in design of each membrane tank.

The aeration tank itself is also equipped with an internal submerged HF module (A_m =17.5 m²) which is functioning as a HRT control (volume control) module and enables the operation of the side-stream modules decoupled from the hydraulic overall performance sof the pilot plant. Variable aeration and cleaning procedures were implemented depending on the experiments performed.

Detailed design and operational parameters are presented in Table 33. The scheme of the wwtp is presented in Figure 78.



Figure 78: General scheme of the MBR plant

	Table 33: Genera	al design and	operational	parameters	at MBR	Cranfield
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Cranfield	FS	HF	MT
Location	England	England	England
Scale	Pilot	Pilot	Pilot
Membrane type	Flat sheet	Hollow fibre	Multi-tube
Membrane configuration	submerged	submerged	submerged
Membrane supplier	Toray	Puron	-
Membrane surface (m ²)	1,4	2,75	3,1
Membrane pore size (µm)	0,08	0,04	0,03
Pretreatment (mm)			
Aeration (m ³ h ⁻¹)	1.1-2.1	0.3-3.44	2.1-6
Design net flux (Lm ⁻² h ⁻¹)	9.0-30	9.0-40	9.0-30
Energy consumption (kWh m ⁻³)	-	-	-
SADm (m ³ m ⁻² h ⁻¹)	0.5-1.5	0.12-1.25	0.5-2
SADp (m ³ m ⁻³)	25-166.7	4.1-41.7	75-200

Cranfield	FS	HF	MT
Biological capacity (P.E.)			
Average flow (m ³ h ⁻¹)			
F/M ratio (kg _{BOD} kg _{MLSS} ⁻¹ d ⁻¹)			
SRT (d)	20	20	20
HRT _{total} (h)	12	12	12
V _{anoxic} (m ³)	-	-	-
V _{aerobic} (m ³)	2.2	2.2	2.2
Recirculation aerobic to anoxic (m ³ h ⁻¹)	-	-	-
Recirculation ratio	-	-	-
Acetate addition for denitrification (m ³ h ⁻¹)	-	-	-
V _{MT} (m ³)	0.0204	0.0125	0.008
Recirculation MT to aerobic (m ³ h ⁻¹)	variable	variable	variable
Recirculation ratio	variable	variable	variable

Operating conditions and influent characteristics

Detailed information about the MBR plant and the influent composition for each experimental period are unfortunately not available. The MBR was in a start-up phase in February 2008 resulting in a relatively low MLSS content (around 6 g.L⁻¹) and non steady state operations. The MBR plant was stabilized in March 2008 during the second experimental period.

4.9.2. Research approach

Objectives

The aim of the filtration characterisation performed at MBR Cranfield was to quantify the activated sludge filterability variations in each membrane configuration depending on flux and the air-lift velocity applied.

Approach

The DFCm was installed directly on site during both experimental periods. Each activated sludge filterability measurement was accompanied by the analyses summed up in Table 34. Particle size distribution analyses were also performed. Detailed information about the analyses was published by Moreau et al. (2009b).

Table 34: Activated sludge quality analyses

Analysis	Reference to method
Activated sludge	
Delft Filtration Characterisdation method (DFCm)	Chapter 3.1
Mixed Liquor Suspended Solid (MLSS)	Chapter 3.2
Free water	
Soluble Microbial Product (SMP) proteins	Chapter 3.2
Soluble Microbial Product (SMP) polysaccharides	Chapter 3.2

4.9.3. Filtration characterisation

First experimental period - February 2008 - Flux variations

The aim of this experimental period was to investigate the impact of the extraction flux on filterability in each membrane tank under identical air-lift velocity conditions (recirculation velocities). During this experimental period, the three air-lifted membrane chambers were operated with a fixed air lift of 35 L.min⁻¹ of air. The permeate flux of extraction in the pilot plant was fixed at 9 L.m⁻².h⁻¹ the first and the last days of this trial. The permeate flux was fixed at 18 L.m⁻².h⁻¹ on the second day. Due to poor filtration behaviour, the permeate flux of extraction of the DFCm was fixed at 30 L.m⁻².h⁻¹ instead of the usual 80 L.m⁻².h⁻¹.



Figure 79: Filterability daily variations in the aerobic tank

Figure 79 is presenting the variations in activated sludge filterability along the day. The fluctuations seem to follow the sewage loading flow pattern. A high loading peak can be observed in the early morning corresponding with a peak in term of filterability with a ΔR_{20} equal to 4.05×10^{12} m⁻¹. A ΔR_{20} decrease can then be observed during the day with a ΔR_{20} of 3.2×10^{12} m⁻¹ till a second peak in the evening with a ΔR_{20} equal to 3.6×10^{12} m⁻¹. During the whole experimental period the activated sludge quality can be considered poor.



Figure 80: Filterability data evolution along the first experimental period

The filterability results of the samples taken in the membrane tank and in the aerobic tank depending on the applied flux are presented in Figure 80. Different behavior depending on the membrane chamber can be observed:

- *Multi tube configuration*: there were almost no impacts on the activated sludge filterability due to the passage of the activated sludge in the membrane channel or due to the flux increase. The ΔR_{20} values measured in the aerobic tank and in the MT membrane tank remained identical, namely 4.05, 3.8 and 2.3*10¹² m⁻¹ during day 1, 2 and 3, respectively.
- *Flat sheet configuration*: No significant variations in activated sludge filterability were noticeable under low flux conditions. The ΔR_{20} values measured in the aerobic tank and in the FS membrane tank remained identical, namely 4.05 and $2.3*10^{12}$ m⁻¹ during day 1 and 3, respectively. However under high flux circumstances, the activated sludge filterability between the aeration tank and the membrane chamber improved. The ΔR_{20} values decreased from 3.8 to $3.2*10^{12}$ m⁻¹ between the aerobic tank and the FS membrane tank.
- *Hollow fibre configuration*: A decrease in filterability quality in the HF membrane tank was observed under low flux conditions with ΔR_{20} values increasing from 4.05 to 5.4*10¹² m⁻¹ in the day 1. However, contrary variations were monitored under high flux conditions. The filterability improved in the HF membrane tank during day 2 with ΔR_{20} values decreasing from 3.8 to 2.1*10¹² m⁻¹. From the three membrane configurations, the hollow fibre one seems to induce the more significant changes in activated sludge filterability.

Effect on permeability

The permeability data monitored during the first experimental period are presented in Figure 81. The permeability is also affected by the flux changes. A lower permeability was monitored when the flux increase, which is therefore in contradiction with the filterability data. However, this result is logical. The filterability variations observed remained relatively small and the permeability is calculated directly from the flux and the TMP values. The relation between flux increase and TMP increase is not linear due to the fouling layer at the membrane surface. Therefore, the permeability data reflected the flux increase but can thus not be compared with the filterability data. Permeability and filterability data should be compared under fixed flux operating condition in the MBR plant.



Figure 81: Permeability data evolution along the first experimental period

Second experimental period - March 2008 - Air-lift velocity variations

The aim of the second experimental period was to investigate the impact of Specific Aeration Demand (SAD_m) on the activated sludge filterability on each membrane module. The SAD_m variations, which directly depend on the air-lift velocity set, should affect the membrane scouring and the membrane performances of the module but also the recirculation velocity of the activated sludge within the modules. Under different SAD_m conditions, i.e. different air-lift velocities, the hydraulics and the retention time in each module should therefore be affected and the effect on filterability could be quantified. During this experimental period, the three membrane configurations were operated with a *fixed* permeate flux of 30 L.m⁻².h⁻¹ in order to accent the observed phenomenon during the flux experiments. Several SAD_m were tested for each membrane module varying between 0.25 and 2 m³.m⁻².h⁻¹. Due to a significant improvement of the activated sludge

filterability in the aerobic tank the permeate flux of extraction of the DFCm was fixed at 60 L.m^{-2} .h⁻¹.

The filterability results of the samples taken in each membrane tank depending on the airlift velocity are presented in Figure 82 Figure 83 and Figure 84 for the MT, FS and HF membrane module, respectively.



Figure 82: Filterability evolution in the MT membrane tank depending on the air-lift velocity



Figure 83: Filterability evolution in the FS membrane tank depending on the air-lift velocity



Figure 84: Filterability evolution in the HF membrane tank depending on the air-lift velocity

- *Multi tube configuration*: the filterability did not show significant variation in the range of tested air-lift velocities for this configuration. All the DFCm outputs showed the same trends with ΔR_{20} values close to $2.3*10^{12}$ m⁻¹ for SAD_m variations between 0.5 and 2 m³.m⁻².h⁻¹.
- *Flat sheet configuration*: significant improvements in filterability were measured under low air-lift conditions. The reduction of the SAD_m values from 1.5 to 0.5 $\text{m}^3.\text{m}^{-2}.\text{h}^{-1}$ is likely to favour good filterability behaviour. The ΔR_{20} values decreased from 2.4 to $0.7*10^{12} \text{ m}^{-1}$.
- *Hollow fibre configuration*: significant improvements in filterability were measured under low air-lift conditions. The reduction of the SAD_m values from 1.5 to 0.25 m³.m⁻².h⁻¹ is likely to favour good filterability behaviour. The ΔR_{20} values decreased from 2.5 to 0.13*10¹² m⁻¹.

Effect on permeability

The permeability data of each membrane module are plotted as a function of the SAD_m fixed in each of them in Figure 85 and as a function of the filterability in Figure 86.



Figure 85: Permeability evolution depending on the air-lift velocity

- Multi tube configuration: the permeability show significant variations in the range of tested SAD_m with a Pearson coefficient equal to 0.77 (p=0.00). The permeability values increased from 230 to 425 L.m⁻².h⁻¹.Bar⁻¹ for SAD_m variations between 0.5 and 2 m³.m⁻².h⁻¹. Whereas no filterability improvement can be noticed (Figure 86), the increase of the SAD_m, i.e. increase of the membrane scouring, was beneficial for the membrane performances of the MT configuration. Better membrane performances were thus obtained under high scouring conditions.
- Flat sheet configuration: improvements in terms of permeability can be observed under low SAD_m conditions even if the data points are quite scattered (no statistical correlation). The scattering of the data point might be due to the variations in feedwater quality (improvement along the experiment period, data not shown), which might have overcome the effect due to the SAD_m variations. The reduction of the SAD_m values from 1.5 to 0.5 m³.m⁻².h⁻¹ is likely to favour good membrane performances. High permeability values were only monitored under low SAD_m conditions. As presented in Figure 86, it is likely due to the improvement in filterability. The improvement in filterability is likely to overcome the lack of membrane scouring due to the decrease of the SAD_m in the flat sheet configuration.
- Hollow fibre configuration: significant improvements in terms of permeability were measured under low SAD_m conditions with a Pearson coefficient equal to 0.67 (p=0.00). The reduction of the SAD_m values from 1.5 to 0.25 m³.m⁻².h⁻¹ is likely to be responsible for good membrane performances in this configuration.

As for the flat sheet configuration, it is likely due to the improvement in filterability (see Figure 86).



Figure 86: Permeability as a function of the filterability in each membrane module All these results will be discussed in *Chapter 6*.

4.10 Concluding remarks

15 full-scale and pilot MBR plants were investigated during this research. 9 of them were presented in detail in this chapter.

The full-scale plants of Heenvliet, Varsseveld and Ootmarsum were not detailed in this chapter due to the fact that they were intensively investigated by Geilvoet (2010) and Krzeminski (2010). Krzeminski (2010) investigated these three MBRs in terms of filterability and design criteria in order to correlate both factors with the energy requirements of the MBR plants. However, the data collected concerning this three full-scale plants will be used in the next chapters for the general comparisons.

The pilot-scale MBR ENREM B was left out of consideration due to the lack of data collected (not any sensors implemented). Furthermore, as presented in *Chapter 5*, the filterability data collected with the DFCm on the MBR ENREM B are discussable due to the fact that the MBR plant was in a start up phase.

Large fluctuations in filterability and membrane performances have been observed. Local differences could be underlined and explained by seasonal fluctuations, organic sludge loading differences, the specificity of the feedwater or the changes in operating conditions. The data presented in this chapter will be discussed in *Chapter 5* and *Chapter 6*. *Chapter 5* will focus on the practical assessment of the DFCm. In *Chapter 6*, general conclusions will be formulated based on the local results presented in this chapter.

5 General observations

A practical assessment of the DFCm based on the campaign of measurements performed during this study will be presented in this chapter. As a second part, general observations will be formulated based on the results presented in Chapter 4.

5.1 The DFCm in practice

5.1.1. DFCm practical assessment

Based on the data presented in *Chapter 4*, several observations can be formulated concerning the DFCm:

- The DFCm was transported to and built successfully at 15 MBR locations. The only needs supplied by the local plants were a power supply and a sewage discharge. The DFCm was usually operational within 2 hours after arrival and never encountered a severe break down. The DFCm can therefore be considered a *user-friendly* and efficient tool.
- Van Meer (2007) performed an in-deep evaluation of the DFCm. The author showed that measurements in a series of the same activated sludge sample with the DFCm resulted in variations of ΔR_{20} values from 0.05 to $0.1*10^{12}$ m⁻¹ after 3 hours. These variations are likely due to the changes occurring in the activated sludge sample during this period of time, like variations in temperature. As presented in Table 35, two measurements performed in a row showed very consistent values with a variation lower than $0.05*10^{12}$ m⁻¹. The measurements performed with the DFCm can therefore be considered *reproducible*.

Table 35: Variations in ∆R ₂	values during in-series	measurements (same sample)
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Time (min)	$\Delta R_{20} \ (*10^{12} \ m^{-1})$	T(°C)
0	0,29	12,78
30	0,29	13,23
60	0,26	13,56

• More than 800 filterability measurements were performed during this research work. 330 measurements were performed at pilot-scale plant locations and more than 500 measurements at full-scale plant locations. The average ΔR_{20} values, associated standard deviations and relative deviations are presented in Table 36 and Table 37 for full-scale and pilot-scale plants, respectively. As presented in Table 36, steady state situations at the full-scale MBRs always correspond to ΔR_{20} values lower than $1*10^{12}$ m⁻¹. Furthermore, the standard variations of the ΔR_{20} values were at most $0.16*10^{12}$ m⁻¹ when the MBRs were under steady state conditions. These results showed that filterability measurements and filterability variations were consistent with the state of the MBRs during the week. In the case of unsteady situations, ΔR_{20} values above $1*10^{12}$ m⁻¹ were measured accompanied

with large standard deviation values varying between 0.4 and $1.5*10^{12}$ m⁻¹. These results underlined the fact that poor and unsteady filterability was monitored under unsteady state conditions. This is consistent with the state of the operation of the MBRs. The measurements performed with the DFCm can therefore be considered *reliable*.

From these previous remarks, it can be stated that steady filterability behaviour can be observed when the standard variations of the ΔR_{20} set of data remain below $0.2*10^{12} \text{ m}^{-1}$. Unsteady filterability behaviour can be observed when the ΔR_{20} values are above $1*10^{12} \text{ m}^{-1}$ accompanied with standard variations of the ΔR_{20} set of data above $0.4*10^{12} \text{ m}^{-1}$.

Table 36: Average ΔR_{20} values for the full-scale MBRs (Heenvliet, Ootmarsum data adapted from Krzeminski et al. (2010))

			$\Delta R_{20} (*10^{1})$	¹² m ⁻¹)	
Plants		Average	Std deviation	Relative deviation dR/R	State of the MBR
	feb-07	0.97	0.11	0.12	Steady
Schilde	apr-08	3.01	1.47	0.49	Unsteady
	aug-08	0.31	0.07	0.23	Steady
Monheim	mrt-07	0.56	0.04	0.07	Steady
Wonnenn	sep-08	0.08	0.02	0.18	Steady
ENDEM	jun-07	0.11	0.16	1.38	Steady
	jan-08	2.03	0.81	0.40	Unsteady
Nordkanal	jul-07	0.12	0.07	0.59	Steady
Norukanar	nov-08	0.43	0.07	0.16	Steady
Heenvliet	feb-07	0.31	0.12	0.39	Steady
ricenviet	jun-08	0.05	0.05	1.00	Steady
Ootmarsum	jun-08	0.18	0.04	0.23	Steady
Cotmarsum	feb-09	2.72	0.41	0.15	Unsteady
Varsseveld	jun-08	0.17	0.04	0.22	Steady
vaissevelu	mrt-09	3.41	0.55	0.16	Unsteady

			∆R ₂₀ (*10	¹² m ⁻¹)	
Plants		Average	Std deviation	Relative deviation dR/R	State of the MBR
	mei-07	0.91	0.32	0.35	High loading
EAWAG B70	nov-07	1.10	0.61	0.56	Low loading
	nov-07	0.91	0.10	0.11	Foaming
	mei-07	0.38	0.13	0.35	High loading
EAWAG B80	nov-07	0.29	0.13	0.45	Low loading
	nov-07	1.02	0.13	0.13	Foaming
	mei-07	0.99	0.16	0.16	High loading
EAWAG B90	nov-07	0.54	0.35	0.64	Low loading
	nov-07	0.80	0.20	0.25	Foaming
	jun-07	0.23	0.05	0.23	Steady
AMEDEOS (AET)	jan-08	0.96	0.11	0.12	Steady
Tromdheim	okt-07	0.61	0.17	0.29	Steady
Trento	nov-07	1.07	0.18	0.17	Steady
Tiento	jul-08	2.14	0.68	0.32	Unsteady
Crapfield (MT)*	feb-08	3.37	0.77	0.23	-
	mrt-08	2.33	0.17	0.07	-
Crapfield (ES)*	feb-08	3.11	0.78	0.25	-
Grannelu (FS)	mrt-08	2.42	0.25	0.10	-
Cranfield (HE)*	feb-08	3.18	1.44	0.45	-
	mrt-08	1.62	1.13	0.70	-

Table 37: Average ΔR_{20} values for the pilot-scale MBRs (*note that the measurements at MBR Cranfield were performed at a different flux, see Section 4.9)

- The results concerning the pilot-scale MBRs presented in Table 37 are in accordance with the results concerning the full-scale plants. For instance, standard deviations below $0.2*10^{12}$ m⁻¹ were calculated when the MBRs were under steady state situations. This result underlined the consistency of the filterability measurements with the state of the MBRs. However, due to various side effects, the tendency is not as clear as in full-scale plants. Poor filterability was also monitored under steady state conditions. This point will be discussed in *Chapter 6*.
- The filterability measurements were also consistent with the membrane performances of the MBR plants (Table 39 and Table 40). Poor filterability measurements matched with the poor membrane performances of the MBR plants whereas good filterability measurements matched with the steady MBR operations. The DFCm measurements can be considered *consistent with the membrane performances* of the MBR plant investigated. This point will be discussed in *Chapter 6*.
- The empirical scale established by Geilvoet (2010) and presented in Table 38 was based on batch test experiments and the monitoring of the full-scale MBR of Heenvliet. The scale's accuracy was confirmed in 15 pilot and full-scale MBR plant applications. ΔR_{20} values below $0.1*10^{12}$ m⁻¹ always corresponded to good operating conditions (i.e. steady permeability with reasonable values for the period of the year, good treatment efficiency and minimum maintenances) in the

MBR plants whereas ΔR_{20} values above $1*10^{12}$ m⁻¹ corresponded mostly with unsteady operations (i.e. foaming events, poor treatment efficiencies, need for more than regular cleanings) and/or poor MBR membrane performances. Furthermore, few of the measurements were completely off scale. The scale developed by Geilvoet (2010) can therefore be considered *accurate*.

Table 38: ΔR_{20} and corresponding filterability qualification (for the standard measuring protocol, CFV = 1.0 m.s⁻¹, J = 80 L.m⁻².h⁻¹)

$\Delta R_{20} (*10^{12} \text{ m}^{-1})$	Filterability
0 - 0.1	Good
0.1 - 1.0	Moderate
>1	Poor

5.1.2. Filtration characterisation method comparison

Geilvoet (2010) already published a comparison between the DFCm and several filtration characterisation methods. Geilvoet emphasised the major drawbacks of each method:

- The flux step method developed by Le-Clech et al. (2003) can be considered a useful tool to assess the fouling potential of the activated sludge of existing MBR plants. However, the lack of a standard set up and protocol, i.e. standard step height and step duration, prevents accurate and reliable comparisons between data obtained by different research groups. Furthermore, the flux step method is a relatively long experiment; it lasts at least 2h, and need specific schedule arrangements like regulation of the incoming flow. It can therefore not be performed during regular MBR operations.
- The Time-to-Filter method (TTF, Standard Methods, 1998) and the Sludge Filtration Index method (SFI, Raudies, 2007) are simple techniques to assess the fouling potential of activated sludge samples. However, the results are strongly dependent on the MLSS concentration of the activated sludge samples. Therefore, these two methods are likely not to be as reliable as the other available options.
- The MBR-VITO Fouling Measurement method (VFMm, Huyskens et al., 2008) presented by the Flemish Institute for Technological research (VITO) was developed to characterise the reversible and irreversible fouling potential of MBR activated sludge. Compared to the DFCm, the major differences lie in the mode of operations. They operated their filtration unit under constant pressure operation. Furthermore, the recirculation cross-flow in their unit is driven by coarse bubble aeration. As a consequence, identical hydraulic conditions are likely to be more difficult to reproduce in different experiments. Furthermore the constant pressure mode of operation can be susceptible to promote different fouling mechanisms as the one observed in current MBR applications.

In order to further develop the comparison between existing filtration characterisation methods, a joint study was organised by the Berlin Centre of Competence for Water (KWB), the TU Berlin and the TU Delft. In this study (De la Torre et al., 2009), a new method called Berlin Filtration Method (BFM) was introduced and compared with the DFCm and ex-situ filtration test cell measurements.



Figure 87: Scheme of the in-situ BFM test cell (De la Torre et al., 2009)

The BFM is presented schematically in Figure 87. The BFM test cell unit is composed of a UF flat sheet membrane (Microdyn-Nadir) with a surface area of $0.025m^2$ and an integrated aeration device. The test cell can be located directly in the activated sludge tank where the filterability needs to be measured. The test cell uses the modified critical flux step method to assess in-situ the filterability of the activated sludge in the tank. The modified critical flux step method is based on the method developed by Le-Clech et al. (2003) presented previously. However, as a major difference, relaxation periods are implemented between each flux step in order to get extra information on the irreversible fouling and the pore blocking influences on the final measurements.



Figure 88: Comparison between the BFM and DFCm outputs (De la Torre et al., 2009)

As presented in Figure 88, the BFM and the DFCm outputs showed similar trends for three MBRs, namely MBR 1, MBR 2 and MBR 4. The poor filterability monitored in the DFCm (high ΔR_{20} values) corresponded to low critical flux values. This is in accordance with the theory. Poor filterability resulted in a quick fouling occurrence even at very low flux and therefore in low critical flux values.

However, both methods showed a different trend for the MBR 3. The DFCm monitored a moderate filterability with ΔR_{20} values around $0.8*10^{12}$ m⁻¹ whereas a low critical flux

around 7 $L.m^{-2}.h^{-1}$ was measured by the BFM. This low critical flux value should correspond to poor filterability. Several hypotheses can be formulated to explain this difference:

- The MBR was in the start-up phase with an MLSS concentration close to 3.5 g.L^{-1} . As presented in *Section 6.8*, the flow regime in the DFCm is laminar as long as the MLSS content of the activated sludge is above 5 g.L⁻¹. Below this value, the flow regime gradually becomes turbulent. Therefore, with an MLSS content of 3.5 g.L^{-1} , it is likely that the flow regime was close to turbulent and therefore that the hydraulic regime in the DFCm was different from the conditions in standard experiments resulting in a lowering of the ΔR_{20} values. As a consequence, it is likely that the filterability measured in the MBR 3 was not comparable with the rest of the set of data.
- Due to the fact that hydraulic circumstances are not easy to control and reproduce in the BFM, an underestimation of the critical flux may have been measured in the MBR 3.

Based on MBR 1, MBR 2 and MBR 4, De la Torre et al. (2009) concluded that both methods were appropriate for accurate measurements of MBR activated sludge filterability. Authors also concluded that on site measurements were more reliable than the ex-situ test cell measurements. De la Torre et al. (2009) also underlined the importance of the step height and duration choices in order to get reliable data.

The BFM can be seen as an efficient tool to characterise the activated sludge filterability. However, three major drawbacks can be underlined compared to the DFCm:

- The BFM is based on the flux step method and therefore is confronted with the same disadvantages as presented previously, i.e. relatively long experiments and need for proper step height and step duration definition.
- As in any air-lift driven devices, the hydraulic conditions around the membrane cannot be easily controlled and reproduced.
- The sensitivity of the method. Whereas the DFCm can quantify small changes in filterability, it is likely that it will not be possible with the BFM due to its small range of variations (inherent to the flux step method).

Compared to other filterability characterisation methods, the DFCm major advantages appears to finally be its extremely *well-defined* and *well-controlled* hydraulic flow regime in the membrane tube and its short term duration allowing *in-series* measurements and *dynamical monitoring* of the activated sludge filterability in MBR plants. However, as illustrated previously, the DFCm measurements are only relevant if the MLSS content of the activated sludge is above 5 g.L⁻¹ due to the different flow regime created under low MLSS content.

5.1.3. Sensitivity of the permeability parameters

As presented in *Chapter 2*, the permeability parameter is currently used in pilot and fullscale applications to monitor the membrane performances of the MBR plants. As presented in Table 39 and Table 40, poor filterability measurements did match with poor monitored permeability data most of the time during the measurement campaign.

 Table 39: Filterability and permeability data of the full-scale MBR plants (Heenvliet, Ootmarsum, Varsseveld, data adapted from Krzeminski et al. (2010))

		$\Delta R_{20} (*10^{12} \text{m}^{-1})$	Permeability	
Plants		Average	(L.m ⁻² .h ⁻¹ .Bar ⁻¹)	
	feb-07	0,97	123	
Schilde	apr-08	3,01	45	
	aug-08	0,31	178	
Monheim	mrt-07	0,56	110	
Monheim	sep-08	0,08	135	
	jun-07	0,11	1050	
ENREW	jan-08	2,03	800	
Nordkanal	jul-07	0,12	215	
Norukanai	nov-08	Average 0,97 3,01 0,31 0,56 0,08 0,11 2,03 0,12 3,01 0,31 0,012 0,43 0,05 0,018 2,72 0,17 3,41	165	
Heenvliet	feb-07	0,31	160	
	jun-08	0,05	215	
Ootmarsum	jun-08	0,18	520	
	feb-09	2,72	220	
Varsseveld	jun-08	0,17	350	
	mrt-09	3,41	100	

		∆R ₂₀ (*10 ¹² m ⁻¹)	Permeability
Plants		Average	(L.m ⁻² .h ⁻¹ .Bar ⁻¹)
	mei-07	0.91	125
EAWAG B70	nov-07	1.10	90
	nov-07	0.91	110
	mei-07	0.38	200
EAWAG B80	nov-07	0.29	185
	nov-07	1.02	220
	mei-07	0.99	185
EAWAG B90	nov-07	0.54	95
	nov-07	0.80	110
AMEDEUS (AE1)	jun-07	0.23	450
	jan-08	0.96	530
Tromdheim	okt-07	0.61	250
Trento	nov-07	1.07	75
Trento	jul-08	2.14	42
Cranfield (MT)*	feb-08	3.37	-
	mrt-08	2.33	-
Cranfield (FS)*	feb-08	3.11	-
	mrt-08	2.42	-
Cranfield (HE)*	feb-08	3.18	-
	mrt-08	1.62	-

Table 40: Filterability and permeability data of the pilot-scale MBR plants

However, as illustrated by an example in Figure 89, permeability is likely not to be sensitive enough to predict the dynamic changes occurring in MBR applications. Figure 89a shows significant variations in terms of filterability which occurred in MBR ENREM during the second experimental period. These variations in filterability were in accordance with the state of the MBR where an extra cleaning was necessary due to a foaming event.

As presented in Figure 89b, the permeability monitored during this event did not follow the filterability changes or the variations in the MBR state. Even if some strong drops in permeability can be observed, the general permeability trend does not give any information of the poor state of the MBR. Therefore, during this specific case, the permeability parameter was likely to not be sensitive enough to represent the state of the MBR.

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Figure 89: Example of the sensitivity of the filterability parameters (a) and permeability parameter (b)

The lack of sensitivity of the permeability parameter is due to several factors:

- The membrane age or initial membrane permeability
- The state of the cleaning, i.e. time since the last chemical cleaning
- Its strong dependency on the applied flux when the membrane is fouled, i.e. under filtration operations.

In the case of a fouled, i.e. used, membrane, the resulting permeability is the sum of the state of the membrane itself, the applied flux and the resulting pressure and the activated sludge filterability. Therefore the permeability is not a constant value but a function of the three main factors involved in fouling, i.e. membrane characteristics, membrane operation and activated sludge properties, *without possible distinction of the contribution of each of them.*

From the set of data gathered during this study, the permeability could monitor different *states* for each MBR whereas the filterability measurements were able to *follow the evolution* of the activated sludge filterability. Comparing permeability and filterability, the permeability can evaluate the state of the global membrane performances of an MBR but without any distinction between the activated sludge quality and the operating condition contributions. On the other hand, filterability gives only information of the activated sludge quality and none about the membrane performances of the plant itself. However, due to the fact that filterability is a more dynamic parameter than permeability, filterability can be used efficiently to optimize the MBR plant dynamically.

5.2 MBRs in practice

Municipal wastewater treatment can be represented in theory by roughly constant characteristics in terms of COD loads, MLSS and nutrient contents. However, large variations in filterability depending on each MBR can be observed in practice. Not any MBRs show the same results and tendencies in term of filterability. Due to many reasons, each MBR shows a particular filterability pattern but also great differences in primary data in addition to the scale difference.

Firstly, as illustrated in Figure 90 through Figure 97, MBRs can be designed in many different ways. Depending on the elimination targets in term of nutrients, anaerobic (AN) and anoxic (AX) tanks can be implemented or not and placed differently, namely prior to the aerobic tank (AE), combined with it or as a post treatment. Furthermore, the membrane stage (MT) can be directly integrated in the aerobic tank or placed in a separated tank depending on the chosen design.



Figure 90: Simplified schematic view of MBR Schilde and MBR Monheim



Figure 91: Simplified schematic view of MBR EAWAG (3 membrane tanks in parallel with the incoming flow divided equally between them) and MBR Trento

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Figure 93: Simplified schematic view of MBR Nordkanal



Figure 94: Simplified schematic view of MBR Trondheim



Figure 95: Simplified schematic view of MBR Heenvliet







Figure 97: Simplified schematic view of MBR Cranfield (3 parallel membrane tanks)

As presented in Table 41 and Table 42 for the full-scale and pilot scale MBRs, respectively, significant differences in terms of plant design can also be observed concerning the recirculation ratio fixed between the membrane tank and the aerobic/anoxic tank in each MBR.

Table 41: Recirculation ratio, activated sludge temperature and F/M ratio for the full-scale MBR plants investigated during this study (data from Varsseveld, Heenvliet and Ootmarsum are adapted from Krzeminski et al. (2010))

	Monheim	Nordkanal	Schilde	Varsseveld	Heenvliet	Ootmarsum	ENREM
Recirculation ratio from the MT	>10	4	6	-	1.5	-	4
Temperature max (°C)	18.3	19.5	17.2	20.3	22.1	-	24.0
Temperature min (°C)	8.4	9.5	10.5	11.5	11.9	-	11.9
F/M ratio winter (g _{COD} .kg _{TSS} ⁻¹ .d ⁻¹)	118.0	160.5	126.9	77.2	92.0	92.1	68.2
F/M ratio summer (g _{COD} .kg _{TSS} ⁻¹ .d ⁻¹)	54.8	195.5	90.8	96.8	27.9	83.2	68.2

 Table 42: Recirculation ratio, activated sludge temperature and F/M ratio for the pilot-scale

 MBR plants investigated during this study

	AMEDEUS	B70	B80	B90	Trento	Trondheim	Cranfield
Recirculation ratio from the MT	4.0	2.0	2.0	2.0	6-11.5	-	various
Temperature max (°C)	26.8	19.6	19.6	19.6	26.5	15.3	15.1
Temperature min (°C)	17.0	15.4	15.4	15.4	12.0	12.1	9.7
F/M ratio winter (g _{COD} .kg _{TSS} ⁻¹ .d ⁻¹)	311.3	143.0	143.0	143.0	97.6	-	-
F/M ratio summer (g _{COD} .kg _{TSS} ⁻¹ .d ⁻¹)	401.8	250.0	250.0	250.0	154.0	-	-

As an example, the MBR Schilde and MBR Monheim were designed with the same criteria in terms of tank sequences and recirculation locations (see Figure 90). However, both plants are operated in a different way. The recirculation from the membrane tank to the aerobic tank is around 6 times the incoming flow at the MBR Schilde whereas this recirculation is above 10 times at the MBR Monheim.

As presented in Table 41, the recirculation ratio can vary from 1.5 to more than 10 times the incoming flow depending on the MBR full-scale plants. This difference in design does have a strong impact on the hydraulics of the plants and especially, as presented below, on the MLSS content in the membrane tank. A mass balance on a membrane tank can be calculated as follow for steady state situations:

$$g_{AE}(Q_I + Q_R) = g_{MT}Q_R + g_PQ_P$$
(5-2-1)

With : g_{AE} = the MLSS content in the aerobic tank in g.L⁻¹ g_{MT} = the MLSS content in the membrane tank in g.L⁻¹ g_P = the MLSS content in the permeate in g.L⁻¹ Q_I = incoming flow in m³.h⁻¹ Q_R = recirculation flow from the membrane tank in m³.h⁻¹

$$Q_P$$
 = permeate flow in m³.h⁻¹

Assuming the membrane tank as a completely mixed reactor, the MLSS content in the permeate equal to 0 g.L⁻¹ and the incoming flow equal to the permeate flow ($Q_{I=} Q_P$), the equation (5-2-1) can be rewritten:

$$g_{MT} = g_{AE} (1 + \frac{Q_I}{Q_R})$$
(5-2-2)

As a consequence, it can be concluded that the MLSS content in the membrane tank of an MBR solely depends on the initial MLSS content in the aerobic tank and the ratio between the incoming flow and the recirculation flow from the membrane tank. A low recirculation ratio will then result in a significant increase of the MLSS content in the membrane tank whereas high recirculation ratio will result in MLSS content in the membrane tank close to the one in the aerobic tank. Therefore recirculation ratio, i.e. design choices, can strongly influence the hydraulics in the MBR and consequently their operating conditions due to differences in activated sludge viscosity and biomass splitting between tanks. As illustrated in Figure 98, a poor splitting of the activated sludge in several membrane tanks can result in significant variations in MLSS content depending on the recirculation ratio. This point will be discussed in *Chapter 6*.





Figure 98: Example of the impact of different activated sludge splitting on the MLSS content in the membrane tanks

In addition to the design criteria, several others differences between MBR plants presented in Table 41 and Table 42 can be observed:

- The temperature of the incoming wastewater can strongly vary depending on the period of the year. These seasonal variations are obviously location dependent.
- The organic sludge loading varies also significantly from plant to plant
- The type of wastewater can be significantly different like in MBR Trento where leachate needs to be treated. Furthermore, the industrial discharges in the municipal wastewater stream are likely to be different between plants.

From all these previous points, it can be stated that numerous differences can be observed between MBRs. Therefore, each MBR can be considered unique resulting from the incoming wastewater, the seasonal variations and the design choices.

These significant differences between plants may result in differences in activated sludge filterability. The relationship between these aspects and the filterability variations will be discussed in *Chapter 6*.

5.3 Summary

The DFCm proved in practice to be *a user-friendly, quick and accurate* tool for activated sludge characterisation. Results obtained during the measurement campaign were *consistent* with the plant operations, *reliable* and *reproducible* along the experimental periods. Compared to other filterability characterisation methods, the DFCm advantages are its extremely well-defined and *well-controlled protocol* and its short term duration allowing dynamical monitoring of the activated sludge filterability in MBR plants. Furthermore, compared to permeability measurements which can indicate the well or poor operation of an MBR plant, filterability measurements can be used to explain the causes of this poor operation and therefore efficiently contribute to the optimisation of the MBR performances.

Each MBR plant is unique. Significant differences in filterability had been found from plant to plant. Due to the possibility of accurate and reliable comparisons, further investigations could be performed. The relationship between these differences and the filterability variations will be discussed in detailed in *Chapter 6*.

6 Discussions

The results presented in Chapter 4 will be discussed in relation to the operating and process conditions of each MBR plant in this chapter. From specific MBR cases, more general conclusions will be presented concerning the effects of various parameters on activated sludge filterability.

6.1 Scale dependency

A general overview of the MBR activated sludge filterability data obtained during the measurement campaigns is presented in Figure 99. The filterability for each MBR is represented as an average value for the summer period and an average value for the winter period. Furthermore, MBRs are classified depending on their scales, pilot or full-scale plants. Large fluctuations in filterability were observed during the measurement campaign. The ΔR_{20} varies from 0.05 $*10^{12}$ m⁻¹ till 3.5 $*10^{12}$ m⁻¹. The significant differences observed depending on the scale of the plants will be discussed in this section.



Figure 99: Overview of activated sludge filterability of all MBR sites (membrane tank samples)(data for MBR Heenvliet, Varsseveld and Ootmarsum adapted from Krzeminski et al. (2010))

Filterability is plotted as a function of the plant scale in Figure 99 and a comparison of the filterability quality depending on the scale of the plant is presented in Figure 100. Significant differences in behaviour can be noticed between pilot and full-scale plants. Pilot-plant filterability data are more homogeneous than full-scale plant filterability data, fluctuating between moderate and poor quality for both periods of the year. Furthermore, extremes like good or very poor quality were rarely measured compared to full-scale MBR applications. Whereas the average improvement in filterability in full-scale plants

during the summer period is around 85%, the average filterability improvement in pilotscale plants is only around 27% (illustrated in Figure 100). In three cases (EAWAG pilots and Trento pilot plant), even filterability deteriorations were measured during the summer period.



Figure 100: Filterability quality depending on the period of the year in full-scale and pilot plants

In addition, the filterability data are plotted as a function of the biological capacity of the MBR plants in Figure 101. It can be clearly seen that the ΔR_{20} measurements of the full-scale plants, i.e. biological capacity above 200 p.e., are more scattered than the measurements performed at the pilot-scale, i.e. biological capacity below 200 p.e..

Table 43: Average and temperature fluctuation as a function of the scale of the plant

Temperature (°C)	Average	stdv
Pilot-scale	17.8	3.6
Full-scale	15.2	4.7


Figure 101: Filterability as a function of the biological capacity of the MBR plants

The filterability differences due to scale could be explained by several factors:

- Pilot-scale plants are less sensitive to seasonal fluctuations. Most of the pilot-scale plants investigated were indoor pilot MBRs, with smaller temperature variations between the summer and the winter periods. As presented in Table 43, the average temperature for the pilot-scale plants investigated is higher than for the full-scale plants and the standard deviation is smaller indicating smaller temperature variations depending on the period of the year in pilot-scale plants.
- Design and operational conditions are significantly different between full-scale and pilot-scale plants. Whereas full-scale plant designs are currently conservative, i.e. low loading process, and full of redundancy to ensure continuous operation, pilot-plants are designed to investigate a specific aspect of wastewater treatment, like the sensitivity of the process to high loading conditions, a focus on specific membrane materials and membrane configurations and so forth. Furthermore, due to an economic factor, few redundancies are provided in pilot-scale studies. Therefore, breakages can happen easier and steady state conditions are more difficult to maintain in pilot-scale plants.
- In keeping with the previous point, less care is taken during the building of most pilots compared to full-scale plants. During the building of a full-scale plant, great care is taken in terms of flow distributions (plug flow for instance), good activated sludge repartition between different zones (anoxic, aerobic) whereas pilot-scale plants are usually designed as a function of what is available on site.

The differences in capacities (buffer), operating conditions, stress and the difficulty to maintain steady conditions lead to different behaviours in terms of activated sludge filterability in pilot-scale MBRs.

From this point of view, scaling up of research results seems to remain a major issue in MBR applications. Kraume et al. (2009) already underlined that lab-scale experiments can be meaningful for full-scale MBR applications only under well-specified conditions, namely fresh activated sludge and comparable operating conditions. Iversen et al. (2009) also stressed the importance of long term experiments at full-scale after their inconsistent results obtained at lab and pilot-scale. These results are also in accordance with the work of van der Gast et al. (2006) where the authors reported that bacterial diversity is determined by the volume of the membrane bioreactors. Taking into account those reports and the results of our study, the use of pilot-scale study should be reconsidered:

- MBR technology is nowadays well-established. Several full-scale MBR plants are operated successfully at competitive costs (Brepols et al., 2007). Therefore MBR can be considered a robust technology. MBR feasibility tests at pilot-scale are likely not to bring more information than what can be found in literature.
- Due to the fact that a significant different activated sludge is grown during pilotscale study compared to the one obtained at full-scale, pilot-scale studies do not seem relevant to determine the fouling potential of a specific wastewater.
- Pilot-scale studies can still bring valuable information if used for membrane configuration comparisons, aeration and cleaning strategy tests or operating cost optimisations.



6.2 Seasonal fluctuations

Figure 102: Overview of activated sludge filterability of all MBR sites

Variations in filterability between the experiments performed with full-scale installations during either the summer or the winter period are obvious. They are schematically represented in Figure 103. Good and moderate filterability was monitored in full-scale plants during the summer period whereas all of the full-scale plants showed poor to moderate filterability during the winter period. As presented in Figure 102, the filterability fluctuations between summer and winter were location dependant. Concerning full-scale plant data especially, the larger improvement in filterability was observed in full-scale MBR Ootmarsum and Varsseveld and was up to 95%. Full-scale MBR Schilde showed the smallest variation with 68% improvement.



Figure 103: Filterability quality of MBR full-scale plants as a function of the period of the year

The deterioration of the activated sludge filterability in winter in full-scale applications is a known fact supported by MBR installation operational experiences (Wang et al., 2006, Lyko et al., 2007, De Wilde et al., 2007). The authors also assessed that seasonal variations have a major influence on membrane permeability (Zwickenpflug et al., 2009). They usually correlated the permeability loss observed with low temperature conditions (Wedi, 2006, De Wilde et al., 2007) presuming the importance of the activated sludge viscosity changes.

From all the parameters currently presented as responsible for changes due to seasonal variations, i.e. apparent viscosity changes, bacterial population evolutions, influent fluctuations: temperature is likely to be the most relevant one. Mulder (2000) already reported the impact of temperature on membrane filtration performances due to permeate viscosity changes. However, Jiang et al. (2005) demonstrated that the proposed temperature correction was not sufficient to explain their results obtained at different temperatures. Judd (2006) summarised the contributing phenomena implied by temperature variations:

- The temperature also impacts the activated sludge viscosity and not only the permeate viscosity reducing then the shear stress generated by coarse bubble aeration
- Intensified deflocculation, release of EPS and floc size reduction has been reported at low temperature
- Particle back transport velocity decreases with the lowering of the temperature
- Lower COD biodegradation at low temperature results in higher particulate and soluble COD (Jiang et al, 2005)

The ΔR_{20} measured during our experimental campaign is presented as a function of the temperature in Figure 104. Results are in accordance with the literature and a reasonable

trend can be observed. A correlation was statistically demonstrated between filterability and temperature in the full-scale plant applications with a Pearson coefficient equal to -0.82 (p=0.00).

As recounted previously, some research groups tried to explain the impact of temperature through the apparent viscosity of the activated sludge (Wang et al., 2006, De Wilde et al., 2007). This aspect was investigated in another research study (Moreau et al., 2009) presented in *Section 6.8*. The temperature impact on apparent viscosity was found to be not significant and did not explain filterability variations. The changes in activated sludge apparent viscosity at low temperature were not sufficient to be considered responsible for the activated sludge filterability deterioration.

Other phenomena need to be taken into account to explain the filterability deterioration under low temperature conditions. Lower COD biodegradation in the sewage network takes place under low temperature conditions. Furthermore, even some polymeric substances are not eliminated at low temperature. As a consequence, a different influent composed of more hardly biodegradable compounds is likely to enter the MBR during winter periods. A significantly more complex influent needs therefore to be treated during winter time. The change in influent is likely to form a different biomass presenting different morphological characteristics compared to the biomass formed during summer (Metcalf&Eddy, 2003). These morphological changes are likely to be responsible for the poor filterability.

Furthermore, Lyko et al. (2007) and Drews et al. (2007) reported soluble EPS release during the winter period whereas Wilen et al. (2000) observed that a decreased temperature reinforced the deflocculation phenomenon. It is likely that a *sub-micron particle release* occurred under *low temperature conditions*, resulting in activated sludge filterability deterioration. This deflocculation process was observed in batch tests and reported by Geilvoet (2010) where a significant correlation between sub-micron particle release under low temperature conditions and filterability was demonstrated.



Figure 104: Filterability plotted as a function of the temperature

A huge impact of the temperature on filterability was figured out during this study. When the temperature dropped from 20 to 10° C, a change in ΔR_{20} from 0.1 to $1*10^{12}$ m⁻¹ was measured (1000%). No statistical direct correlation between activated sludge viscosity and temperature was found in the range of MLSS investigated during this study (see *Section 6.8*). It is likely that the deterioration of the filterability under low temperature conditions is due to the combination of several effects:

- The changes in the influent composition and biomass characteristics due to the lower degradation of COD and specific substances in the sewage network,
- The deflocculation of the activated sludge or the different flocculation properties of the biology under low temperature conditions resulting in sub-micron particle and SMP like particle release.

It is likely that the physical-chemical properties, i.e. like surface activities, of the activated sludge change as a function of the temperature. The flocculation properties of the activated sludge can then be considered a related process temperature and are therefore likely to be affected by these changes. As a consequence, the changes in flocculation properties depending on the temperature are likely to affect the activated sludge filterability.

6.3 MBR permeability

The effect of the seasonal fluctuations on membrane performances of the MBR plants investigated during the measurements campaign are presented in Figure 105a and Figure 106a for the full-scale plants and the pilot-scale plants, respectively. The permeability data of the MBR plants are also plotted along with temperature in Figure 105b and Figure 106b for full-scale and pilot-scale plants, respectively. Significant variations between summer and winter periods can be observed.

The permeability variations appeared to be less pronounced than the ones expressed in term of filterability. As previously reported in *Section 4.4*, the high values of permeability for MBR ENREM are due to the way the operators of the plants are calculating it. A correction for temperature and for pressure is included in their permeability values.

The full-scale MBR plants showed permeability trends in accordance with the filterability measurements. The permeability is better in summer than in winter. This, however, is not always the case for the pilot-scale MBR plants. In some case, even a better permeability was measured in winter than in summer.

Furthermore, large fluctuations in permeability (from 400 to 150 L.m⁻².h⁻¹.Bar⁻¹) can be observed for the same temperature depending on the plants. There is no clear pattern between permeability and temperature even if significant variations in permeability can be observed between different periods of the year.



Figure 105: Permeability as a function of the period of the year (a) permeability plotted along with temperature (b) for full-scale plants



Figure 106: Permeability as a function of the period of the year (a) permeability plotted along with the temperature (b) for pilot-scale plants

The insignificant correlation observed between the permeability of the MBR plants and the temperature underlined the weakness of the permeability parameter. As already presented in *Chapter 2*, filterability measurements have several advantages compared to the monitoring of the permeability only. In this specific case, the filterability measurements help to determine the importance of the activated sludge temperature on the process performances whereas the permeability does not. Due to the non-distinction between the membrane state itself and the activated sludge quality, permeability measurements are insufficient to comprehend fully what is occurring during the MBR filtration process.



Figure 107: Permeability data as a function of the filterability for the full-scale plants

Permeability data of the full-scale plant and the pilot-scale plants are plotted along with the filterability data in Figure 107 and Figure 108, respectively. A statistical correlation with a Pearson coefficient equal to -0.882 (p=0.00) was demonstrated between the filterability measurements and the permeability data of the full-scale MBRs equipped with hollow fibre membranes. The correlation between the filterability measurements and the permeability of the MBRs equipped with flat sheet membranes is not demonstrated statistically. A weaker Pearson coefficient between the filterability and the permeability data of the plants equipped with hollow fibre membranes was also noticed in the pilotscale plants. Furthermore it can be noticed that small variations in permeability correspond to large changes in filterability underlining the sensitivity of the filterability parameter.



Figure 108: Permeability data as a function of the filterability for the pilot-scale plants

The configuration's impact on the significance of the correlation is likely due to two factors:

- The imposed *cleaning strategy* varied depending on the membrane type. MBRs equipped with hollow fibre membranes are operated on a weekly based chemical cleaning schedule whereas MBRs equipped with flat sheet membranes are operated on a monthly or yearly based chemical cleaning schedule (Judd, 2006). Due to the weekly cleanings implied by hollow fibre configurations, filterability measurements could be linked with permeability data of the MBRs equipped with this configuration, as reversible fouling remains the major contributor to fouling on a weekly basis. However, in the case of low cleaning frequency, i.e. flat sheet configuration, reversible fouling does not remain as the main contribution to the total membrane fouling. Under these cleaning conditions, filterability measurements are not relevant anymore to describe the full membrane performance loss (or the duration between the last cleaning and the measurement campaign has to be taken into account).
- The difference in clean membrane permeability. The clean membrane permeability of hollow fibre membranes is usually relatively close to 200 L.m⁻².h⁻¹.Bar⁻¹ and does not strongly vary depending on the membrane suppliers. It is not the case for flat sheet membranes. Large variations in clean membrane permeability can be noticed depending on the membrane suppliers making permeability data comparison difficult.

Due to these two factors, filterability is likely to accurately predict membrane performances of MBRs mostly equipped with hollow fibre configurations.

6.4 Feedwater quality and toxicity

Le-Clech et al. (2006) stated in their review that fouling in MBRs was mostly due to the interactions between the MBR membranes and the biological suspension. The influence of the wastewater itself should not be considered significant (Choi et al. (2005)). The authors explained that higher fouling rates were observed for specific cases, i.e. sal sewage as a feed or synthetic feed which could lead to different EPS contents. Le-Clech et al. (2006) finally concluded that the fouling propensity of the wastewater was indirectly taken into consideration during the characterisation of the biomass. Guglielmi et al. (2007) reported different findings, where the feedwater quality should not be considered insignificant, especially in the case of synthetic influents.

Due to the fact that all the MBR plants investigated in our study were fed with real municipal wastewater, few correlations between filterability and feedwater quality were therefore expected. However, results obtained in several MBR locations seem to show different trends. The results concerning incoming feedwater and unexpected toxic events and their effects on activated sludge filterability will be discussed in this section.

6.4.1. Feedwater - MBR Trento

As presented in Table 44, the filterability monitored during the winter period (November 2007) in MBR Trento presented ΔR_{20} values in accordance with the temperature, namely values close to $1*10^{12}$ m⁻¹ for a temperature around 13°C. Furthermore, small standard deviations were calculated corresponding to steady operations. Based on the results of temperature influence on filterability presented in *Section 6.2*, a large improvement in filterability was expected for the summer period (July 2008) where the temperature was close to 27° C. As presented in Table 44 and Figure 109, ΔR_{20} values close to $0.1*10^{12}$ m⁻¹ were therefore expected. However, the filterability results were different in practice.

An unexpected filterability was monitored in the MBR Trento pilot-scale plant during the summer period (July 2008). As presented in Figure 109 and Table 44, the activated sludge filterability can be considered poor with an average ΔR_{20} value of 2.14*10¹² m⁻¹ whereas the temperature of the activated sludge is close to 27°C. The filterability measured during the summer period can therefore be considered to be worse than during the winter period.

	Trento		
Period	$\Delta R_{20}(*10^{12} \text{m}^{-1})$	stdv	T(°C)
nov-07	1.07	0.18	12-13.4
jul-08	2.14	0.68	23.5-26.5
Expected summer value	around 0.1	based on Se	ction 6.2

Table 44:	Filterability	measurements	for both	experimental	period
				•	•



Figure 109: Representative additional resistance increase of the Trento MBR pilot plant during the summer and winter periods and an illustration of the summer filterability data expected

After the collection of a maximum of information, the only significant explanation for the deterioration of the activated sludge filterability was that a higher concentration of discharges of industrial wastes (landfill leachate) was measured in the influent in July 2008. Part of it had to contribute to the filterability deterioration.

As shown in Table 45, the COD and Ammonia load were higher in July 2008 compared to November 2007. The increase was reported from the end of the first week of July (data not shown). A high concentration of ammonia is specific for landfill leachate wastewater. Furthermore the lower COD removal compared to November and the incomplete nitrification achieved underlined the loss of treatment efficiency during this period.

Table 45: Summar	y of the MBR	plant efficiency	for the different	t experimental	period
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Trento								
	Period	nov-07	jul-08					
Influent	COD (mg L ⁻¹)	270-377 🥖	535					
	NH4 ⁺ (mg L ⁻¹)	14.2-19.8	35-48-4					
	COD (mg L ⁻¹)	13-31	47					
Permeate	NH4 ⁺ (mg L ⁻¹)	0.4-3	4.6					
1 cifficate	NO3 ⁻ (mg L ⁻¹)	6.3-13.1	24.2					
	P (mg L ⁻¹)	0.8-2.2	0.1					
	TSS (g L ⁻¹)	9.6-10.2	7.50					
	T(°C)	12.0-13.4	23.5-26.5					

The feedwater quality did strongly affect the treatment efficiency, the filterability and the membrane performances of the MBR (see *Section 4.8*). The feedwater quality can be in

this case be, therefore considered to be the major factor influencing activated sludge filterability before seasonal fluctuations and temperature.

6.4.2. Activated sludge poisoning - MBR Schilde

An unexpected low filterability quality was monitored in the full-scale MBR Schilde in April 2008. The filterability output is presented in Figure 110. The ΔR_{20} values went up to $5*10^{12}$ m⁻¹ indicating a poor filterability. Such high ΔR_{20} values were rarely measured in municipal MBR applications. None of the operating parameters set in the plants could explain such a poor filterability. Low temperature was measured (12°C) during the experimental period. However, previous experiments performed under the same temperature and operating conditions in February 2007 did not reveal such poor filterability behaviour. The data is presented in Table 46. A high COD concentration (76 mg.L⁻¹) was measured in the permeate during this experimental period whereas the COD load coming from the influent remained in the usual range compared to other experimental periods. Several explanations were formulated like growth of filamentous bacteria preventing good treatment efficiency or presence of a toxic in the influent trapped in the MBR system due to the complete retention provided by the membrane steps. However none of those explanations were conclusive. As a consequence, the activated sludge needed to be completely replaced a month after our experimental period implying a new start up of the MBR plant.

		Sch	ilde		
	Period	feb-07	apr-08	aug-08	apr-09
Influent	COD (mg L ⁻¹)	175	140-200	170	140-195
miluent	$NH_4^+ (mg L^{-1})$	17	15-19	8.0-13.1	
	COD (mg L ⁻¹)	27	76	23	15-16
Permeste	$NH_4^+ (mg L^{-1})$	-		-	-
T criticate	NO_{3}^{-} (mg L ⁻¹)	1.9-2.7	2.2	2.2	
	P (mg L ⁻¹)	-	-	-	-
TSS (g L ⁻¹)		6.9-7.9	11.1-11.5	10.5-13	8.4-12.9
T(°C)		10.5-10.8	11.9-12.5	16.5-17.2	13.75-14.1

Table 46: Summary of the MBR plant efficiency for the different experimental period



Figure 110: Representative additional resistance increase of the full-scale MBR Schilde depending on the period of the year

A problem with the dosage pump for the carbon source addition was finally ascertained to be the cause of the problem. A carbon source, namely butyric acid, is added in the anoxic tank of the MBR in order to enhance denitrification. The dosage was set proportional to the nitrogen concentration in the permeate. However, it seems that a minimal amount of very concentrated carbon source was dosed even though the nitrogen concentration was below the discharge limits. It resulted in a poisoning or an overloading of the activated sludge due to the highly concentrated dosed solution.

The treatment efficiency of the plant was not ensured anymore in this specific case due to the over-loading of the activated sludge. An excess of the carbon source resulted in a toxic event (inhibition) which strongly deteriorated the activated sludge filterability and finally the MBR membrane performances and the plant operations. In this case, the state of the activated sludge and the specific load that the plant had to handle were the major factors influencing the activated sludge filterability.

6.4.3. Foaming event - MBR EAWAG

As already presented in *Section 4.3*, a foaming event was reported in MBR EAWAG during the third experimental period (November 2007). The causes of the foaming event are unknown. As a consequence, activated sludge got spilled on the floor resulting in total solid loss in the whole MBR (see Table 47).



Figure 111: Filterability evolution in MBR EAWAG during different experimental period

The filterability data for the different periods in the MBR EAWAG plant are presented in Figure 111. As already reported in *Section 4.3*, changes in activated sludge loading did not seem to significantly affect the activated sludge filterability. The filterability quality in each tank remained identical, i.e. poor in the tank B70, moderate in tank B80 and close to poor in tank B90.

However, as presented in Figure 111, a significant change in filterability can be observed during the foaming event, especially in tanks B60 and B80. Whereas the activated sludge filterability remained in the same order of magnitude in tank B70 and B90 with a ΔR_{20} value close to $1*10^{12}$ m⁻¹, the filterability improved in the aerobic tank (B60) with ΔR_{20} values decreasing from 2 to $1*10^{12}$ m⁻¹ and the filterability got worse in the tank B80 with ΔR_{20} values increasing from 0.4 to $1*10^{12}$ m⁻¹. The foaming event led to a change in the TS content in each tank which therefore resulted in changes of the biological properties of the activated sludge. These changes in biological properties strongly influence the activated sludge filterability. Thanks to a very dynamic behaviour between each tank, a uniform filterability in the whole MBR was monitored during the foaming event.

Zurich		B70			B80			B90	
Date	mei-07	nov-07	nov-07	mei-07	nov-07	nov-07	mei-07	nov-07	nov-07
Days of operation	150-153	341-344	352-356	150-153	341-344	352-356	150-153	341-344	352-356
MBR state	High load	low load	low load	High load	low load	low load	High load	low load	low load
V _{MT} (m ³)		1.41			1.61		0.67	0.67	2.15
HRT per configuration (min)	107	177	185	115	185	182	76	140	275
COD load (kg _{COD} .d ⁻¹)	16	10	9	16	10	9	16	10	9
Sludge loading (g _{COD} .Kg _{TSS} ⁻¹)	250 💋	143	45 1	250	143	-154 🔍	250	143	+51- 🔪
average TS (g.kg ⁻¹)	7.2 🔪	8.5	6.5	9.1 🕓	11.7	6.37 🦯 🦊	8.7	10.1	8.2
Temperature (°C)	19.4	15.6	16.1	19.4	15.6	16.1	19.4	15.6	16.1
Permeability (L m ⁻² h ⁻¹ Bar ⁻¹)	125	75-150	110	200	185-205	220	185	95	110

Table 47: Summary of the MBR plant efficiency for the different experimental period

As presented in Table 47, the permeability of the MBR plant itself does not seem to be affected by the foaming event. The permeability values monitored in each membrane tank remained close to the value monitored during the other experimental periods.

A foaming event occurred in MBR EAWAG. This foaming event resulted in TS loss and leaded to significant changes in the biological properties of the activated sludge. These changes in the activated sludge properties were underlined by significant variations with the monitored filterability during the other experimental periods. Whereas very dynamic fluctuations of the filterability were measured in each tank during the other experimental periods, the foaming event resulted in a homogenisation of the activated sludge filterability in all the MBR tanks.

6.5 Impact of the organic loading on filterability

Full scale MBR plants are currently designed and operated at a very low food to mass ratio, i.e. very low activated sludge loading, in order to achieve high nutrient elimination. The study presented in this section is the combination of the experimental works performed at two pilot-scale MBRs, namely MBR Trondheim and MBR EAWAG. In both pilot plants, the activated sludge filterability could be measured at the different stages of the purification process. Furthermore, the activated sludge loading was changed between two experimental periods at MBR EAWAG.

The details of each MBR plant are presented in *Section 4.3* and *Section 4.7*. The impact of activated sludge loading on its filterability will be discussed in this section.

6.5.1. MBR Trondheim

Ivanovic et al. (2006) reported results concerning the influence of activated sludge loading rates on the characteristics of the retentate in the BF-MBR. These results were obtained with the BF-MBR investigated in Trondheim. Due to the specificity of their biofilm MBR (see *Chapter 4*), they expressed the "activated sludge loading" in terms of organic loading per surface area of carrier ($gO_2.m^{-2}d^{-1}$). Based on two operating conditions, namely a low loading rate (12.1 $gO_2.m^{-2}d^{-1}$) with 4h retention time and a high loading rate (47.9 $gO_2.m^{-2}d^{-1}$) with a 1h retention time, they concluded that low organic loading conditions produced a retentate with more favourable characteristics in term of dewatering and activated sludge filterability. They observed a lower fouling rate and required less frequent cleaning under low loading conditions.

The Trondheim MBR pilot plant is composed of three aerobic tanks in a series. Therefore, it was possible to monitor the mixed liquor filterability during the purification process (after each tank) in order to confirm the results presented by Ivanovic et al. (2006). The retention of time in each tank was equal to 2h and due to COD measurements at the entrance of each tank it was possible to calculate the specific organic loading in each tank. Results are presented in Table 48 and Figure 112.

	Trondheim										
	Tank	HRT (h)	COD (mgO ₂ . L^{-1})	Organic loading $(gO_2.m^{-2}.d^{-1})$	$\Delta R_{20} (*10^{12} \text{ m}^{-1})$						
ſ		0	500	-	1,87						
I	1	2	248	28,6	1,05						
I	2	4	62	14,2	0,98						
	3	6	25	3,5	0,7						

Table 48: Organic loading along the MBR Trondheim process



Figure 112: Impact of the organic loading $(gO_2.m^{-2}.d^{-1})$ on the activated sludge filterability in Trondheim pilot plant (October 2007)

The results presented in Figure 112 show a clear impact of the organic loading $(gO_2.m^{-2}d^{-1})$ on the filterability. When the organic loading rate decrease from 28.6 to 1.4 $gO_2.m^{-2}d^{-1}$ the ΔR_{20} decreased from 1.87 to 0.70 *10¹² m⁻¹. A large improvement occurred in the first aerobic tank (from a 0 to 2h retention time). A second improvement step occurred between a 4 and 6h retention time. It is likely that the filterability improved during the treatment due to the degradation of the organic components of the wastewater. Due to the fact that there is not any recirculation in the pilot plant, the MBR Trondheim plant can be considered a plug flow reactor. The improvement in filterability can then be considered a result of the purification of the wastewater stream.

Our study performed at Trondheim MBR confirmed and strengthened the previous results they obtained (Ivanovic et al., 2006). A long hydraulic retention time, which induced lower organic loading, enhanced activated sludge filterability. The improvement in filterability can directly be correlated with the decrease in organic loading per surface area in the BF-MBR plant.

6.5.2. MBR EAWAG

As presented in *Section 4.3*, MBR EAWAG was built to investigate the impact of activated sludge loading on membrane filtration performances in MBRs. Activated sludge loading could be changed in the plant by varying the contact time between the wastewater and the activated sludge, the volume of the biology (amount of activated sludge) or the influent COD load. The COD load in the plant was changed from 16 to 10 kg_{COD}.d⁻¹ between the first experimental period (May 2007) and the second one (November 2007).

As a consequence, the activated sludge loading decreased from 250 to 143 g_{COD} .kg_{TSS}⁻¹.d⁻¹(Table 49).

Zurich	B70		B	80	B90		
Date	mei-07	nov-07	mei-07	nov-07	mei-07	nov-07	
Days of operation	150-153	341-344	150-153	341-344	150-153	341-344	
MBR state	High load	low load	High load	low load	High load	low load	
V _{MT} (m ³)	1.41		1.61		0.67		
HRT per configuration (min)	107	177	115	185	76	140	
COD load (kg _{COD} .d ⁻¹)	16	10	16	10	16	10	
Sludge loading (g _{COD} .Kg _{TSS} ⁻¹ .d ⁻¹)	250	143	250	143	250	143	
average TS (g.kg ⁻¹)	7.2	8.5	9.1	11.7	8.7	10.1	
Temperature (°C)	19.4	15.6	19.4	15.6	19.4	15.6	
Permeability (L m ⁻² h ⁻¹ Bar ⁻¹)	125	75-150	200	185-205	185	95	

Table 49: MBR EAWAG loading data for both experimental periods

Activated sludge filterability in each tank is plotted as a function of the activated sludge loading in Figure 113. No significant changes in terms of filterability can be observed between both experimental periods. The activated sludge filterability can be considered poor for both periods in the aerobic tank B60, the membrane tank B70 and the membrane tank B90 with ΔR_{20} values close to $2*10^{12}$ m⁻¹, $1*10^{12}$ m⁻¹ and $0.95*10^{12}$ m⁻¹, respectively. The activated sludge filterability in the membrane tank B80 remained moderate for both periods with ΔR_{20} values oscillating around $0.45*10^{12}$ m⁻¹.

However, the temperature of the activated sludge changed significantly between both experimental periods. The temperature was around 19 °C in May 2007 and around 15 °C in November 2007. As presented in Figure 113, expected ΔR_{20} values can be calculated based on the trend line obtained in Figure 104 (*Section 6.2*). Expected ΔR_{20} values were 4.5, 2.4. 1.1 and 2.2*10¹² m⁻¹ for the tanks B60, B70, B80 and B90, respectively. Based on the results presented in *Section 6.2*, the decrease of 4 °C should have resulted in a significant deterioration of the activated sludge filterability.

It is likely that the change in organic loading from 250 to 143 $g_{COD}.kg_{TSS}^{-1}.d^{-1}$ during the second experimental period compensated for the filterability deterioration due to the temperature decrease. The organic loading can therefore be considered a factor which has a significant influence on activated sludge filterability in MBR EAWAG. The reduction of the organic loading is likely to result in a filterability improvement.



Figure 113: Activated sludge filterability in each tank as a function of the activated sludge loading and expected winter values based on temperature differences

6.5.3. General overview

Meng et al. (2009) reported in their review that high activated sludge loading could be detrimental in terms of membrane filtration performances (Cho et al., 2005). It is mostly due to the fact that both parameters are strongly affecting the bound EPS production since they govern biomass growth and decay (Men et al., 2007). Le-Clech et al. (2006) reported the same trend. However, they specified that the correlation between activated sludge loading and increase in fouling propensity was only demonstrated for unsteady state conditions, when biomass stabilisation had not yet been reached.

During our study, the DFCm measurements partly confirmed the results already presented by MBR plant operators. Ivanovic et al. (2006) reported an improvement of the activated sludge quality when the organic loading was reduced.

However, concerning the MBR EAWAG, Boelher et al. (2009) and Zwickenpflug et al. (2009) reported that activated sludge loading changes did not have a significant impact on the MBR membrane filtration performances compared to seasonal fluctuations and uncontrolled foam events. In contrary, the DFCm study underlined the fact that the reduction of the organic loading from 250 to 143 $g_{COD}.kg_{TSS}^{-1}.d^{-1}$ compensated for the filterability deterioration due to the decrease of the temperature. It is likely that Boelher et al. (2009) and Zwickenpflug et al. (2009) could not accurately differentiate the effect of temperature and sludge loading during their study.

A strong statement cannot be made based on this study. Activated sludge loading is likely to influence the activated sludge filterability. However, it should not be considered a predominant parameter affecting filterability due to the different results reported.

The activated sludge loading of each investigated MBR was calculated depending on the experimental periods in order to check its impact on filterability from a general point of view. Filterability data are plotted along with activated sludge loading in Figure 114.

A weak correlation can be demonstrated statistically between the filterability measurements and the activated sludge loading calculated for the experimental summer periods with a Pearson coefficient equal to 0.54 (p=0.05). However, a complete different behaviour can be observed during the experimental winter periods. Activated sludge loading is likely to positively affect the activated sludge filterability but its action is mostly visible under good (summer-like) and steady operations. From the moment unexpected events or low temperature conditions occurred, the effect of activated sludge loading became hardly visible due to the predominant effects of the other factors on filterability. Therefore, activated sludge loading is likely to influence filterability but cannot be considered a major parameter to control fouling propensity in MBRs.



Figure 114: Filterability as the function of the activated sludge loading in the different MBR plants

6.6 Indirect effect on activated sludge filterability – activated sludge upconcentration

As already presented in *Chapter 5* and recalled in the Equation (6-6-1), the MLSS content in the membrane tank of an MBR is solely a function of the MLSS content in the aerobic tank and the recirculation ratio from the membrane tank. Under a low recirculation ratio, a significant upconcentration of the activated sludge can occur in the membrane tank. For instance, if an MBR is designed with a recirculation ratio of 2 and operated with an MLSS content in the aerobic tank close to 8 g.L⁻¹, the MLSS content in the membrane tank of an MBR should be close to 12 g.L⁻¹ under the assumption that the membrane tank is completely mixed and the MBR under steady state conditions.

$$g_{MT} = g_{AE} (1 + \frac{Q_I}{Q_R})$$
(6-6-1)

With : g_{AE} = the MLSS content in the aerobic tank in g.L⁻¹

 g_{MT} = the MLSS content in the membrane tank in g.L⁻¹

 Q_I = incoming flow in m³.h⁻¹

 Q_R = recirculation flow from the membrane tank in m³.h⁻¹

As reported in *Chapter 2*, filtration and gravity thickening are governed by an identical and general force balance (Wakeman, 1981). During the thickening process, the packing arrangement of the particles and the applied pressure, a more or less porous activated sludge layer will be formed leading to different dewaterability properties depending on the activated sludge concentration. The same phenomena can be assumed for the filtration process during the upconcentration of the activated sludge. The effect of activated sludge upconcentration on filterability was already investigated by Lousada Ferreira et al. (2009) during batch test experiments. The study developed in this section will discuss significant changes in filterability observed during the occurrence of the upconcentration process in the membrane tanks of two pilot-scale plants.

6.6.1. MBR Cranfield

As already presented in *Section 4.9*, strong variations in activated sludge filterability were observed depending on the membrane configurations and the air-lift velocity set (or specific aeration demands-SAD_m) in the MBR. As presented in Figure 115 and Figure 116, the filterability and the permeability in the membrane chambers, especially the one equipped with hollow fibre modules, improved when the SAD_m was reduced. Therefore a better filterability and better membrane filtration performances were observed under low SAD_m conditions.

Result overview



Figure 115: Example of filterability improvement observed in MBR Cranfield (hollow fibre membrane configuration)



Figure 116: Influence of the aeration on the module permeability.

An overview of the different parameters monitored during the experimental periods is presented in Table 50. Significant fluctuations in the MLSS content in each membrane module can be observed. The MLSS concentration varied from 5.5 to 12.3 g.L⁻¹ depending on the membrane modules and the SAD_m set.

Cranfield	FS	HF	MT
Membrane type	Flat sheet	Hollow fibre	Multi-tube
Membrane surface (m ²)	1.4	2.75	3.1
Membrane pore size (µm)	0.08	0.04	0.03
Aeration (m ³ h ⁻¹)	1.1-2.1	0.3-3.44	2.1-6
Design net flux (Lm ⁻² h ⁻¹)	9.0-30	9.0-40	9.0-30
SADm (m ³ m ⁻² h ⁻¹)	0.5-1.5	0.12-1.25	0.5-2
Temperature (°C)	10.7-14.9	10.5-14.9	10.2-14.9
MLSS (g.L ⁻¹)	6.1-12.3	6.4-11.8	5.45-10.1
pH	6.8-7.4	7-7.6	6.9-7.5
DO (mg.L ⁻¹)	0.2-2.2	0.2-1.6	0.2-2.4

Table 50: Overview of the monitored parameters during the experimental period

The MLSS concentration plotted as a function of the SAD_m in each membrane module are presented in Figure 117. A general tendency can be observed. Higher MLSS concentrations were measured under low SAD_m conditions. It is likely due to the lower recirculation ratio from the membrane tanks induced under low SAD_m conditions.

The membrane filtration performances of each module as a function of the MLSS content are presented in Figure 118. As in the filterability case, a good correlation can be observed between MLSS concentration and membrane module permeability for the hollow fibre and the flat sheet configurations. For these two configurations, the membrane permeability improved from 400 to 750 L.m⁻².h⁻¹.Bar⁻¹ when the MLSS concentration increased from 5.5 to 12.3g.L⁻¹. Only the data gathered with the multi tube configuration do not seem to follow the pattern.



Figure 117: MLSS concentration in each module as a function of the aeration



Figure 118: Influence of the MLSS concentration on the permeability of the plant



Figure 119: Filterability as a function of the MLSS concentration

As presented in Figure 119, a good correlation between filterability and MLSS content can be demonstrated statistically with a Pearson coefficient equal to -0.97 (p=0.01) for the hollow fibre membrane configuration. Filterability improvements in the flat sheet and in the hollow fibre configurations can be clearly linked with MLSS content. The increase in MLSS (activated sludge upconcentration) can be correlated with the decrease of the SAD_m and thus also the improvement in filterability.

Effect of the SAD_m

The air-lift velocity in MBR Cranfield pilot plant can be adjusted to investigate the impact of the aeration, i.e. $SAD_m (m^3.m^{-2}.h^{-1})$ on the membrane performances. During the experimental period, different SAD_m were fixed and correlated with the filtration performances of the MBR plant.

It is important to notice that in the very peculiar set up of the MBR Cranfield the air-lift velocity, i.e. the SAD_m, is controlling two parameters:

- The recirculation ratio from the membrane tank. Under low SAD_m, the recirculation ratio can be considered low as well and result in a significant increase of MLSS content in the membrane tank as presented previously.
- The vertical gas and liquid flow inducing scouring at the membrane. Under low SAD_m, it is likely that low vertical gas and liquid flow will be created resulting in low shear at the membrane wall.

Based on this first observation, it is likely that the activated sludge upconcentration observed in Figure 117 is due to the low recirculation ratio imposed in the membrane tank by the low SAD_m .

Based on the second observation, the effect of the SAD_m on the vertical liquid flow can be investigated. As a result of the SAD_m set, very low vertical liquid flow velocities were measured, especially in the hollow fibre and flat sheet membrane configurations. The measured liquid flow velocities varied between $1.3*10^{-5}$ and $4*10^{-4}$ m.s⁻¹ depending on the air-lift velocity. Under current submerged MBR operating conditions, the liquid flow velocities close to the membrane are usually comprised between 0.1 and 0.4 m.s⁻¹ (Prieske et al., 2008, Berube et al., 2006). Consequently, a low shear, i.e. weak scouring, can be expected at the membrane wall.

Secondly, the permeate flow velocity, i.e. 30 L.m⁻².h⁻¹ or 8*10⁻⁶ m.s⁻¹, was equal to 60% of the lower measured liquid flow velocity and cannot be considered negligible as it is usually the case in current immersed MBR applications. A local upconcentration effect can therefore be expected. However, as presented in Figure 118, the local upconcentration and the low shear generated at the membrane wall do not seem to be detrimental in terms of membrane performances.

Conclusions

As already reported in literature (Le-Clech et al., 2003), high MLSS concentration might favour the formation of a high porous media or a loosely bound cake layer. However, these hypotheses were not substantiated by the particle size distribution analyses. No trend between floc size and filterability were underlined during our study. Furthermore, during all these experimental periods, no correlation between SMP and filterability were emphasized.

More than the MLSS content itself, the *activated sludge upconcentration* occurring under specific aeration conditions is likely responsible for the activated sludge filterability improvement.

Two mechanisms can be distinguished:

- Due to practical arrangements (volume of the tank, activated sludge residential time), hydraulic conditions in each membrane unit are *different* and an *activated sludge upconcentration* occurred under low SAD_m conditions in the hollow fibre and flat sheet membrane configurations due to the low recirculation ratio.
- This *activated sludge upconcentration* leads to margins in MLSS content inducing differences in filtration behaviour.

Due to the *activated sludge upconcentration in the membrane tank* under low recirculation ratio conditions, sub-micron particles (foulants) are likely to be *trapped within the dense floc network* resulting in a clarification effect. This process is likely to be beneficial in terms of filterability.

In addition, it can also be assumed that the low local shear generated in this specific set up did not break the floc at the membrane wall and was not detrimental in terms of filterability and membrane performances.

6.6.2. MBR EAWAG

Significant variations in the TS content (Total Solids, i.e. MLSS, colloids and solubles, expressed in g.kg⁻¹) in each membrane tank were observed in MBR EAWAG during the second experimental period.

Result overview

Detailed information about the plant and the state of the MBR during this experimental period is recalled in Table 51 and Table 52. As presented in Figure 120, significant variations in terms of filterability were monitored during the second experimental period in November 2007.

Zurich	B70	B80	B90
Membrane type	Flat sheet	Hollow fibre	Hollow fibre
Membrane configuration	submerged	submerged	submerged
Membrane supplier	Kubota	Zenon ZW500A	Puron
Membrane surface (m ²)	40	46	30
Membrane pore size (µm)	0.4	0.04	0.1
Aeration (m ³ h ⁻¹)	50	46	15
Design net flux (Lm ⁻² h ⁻¹)	12	10	16
SADm (m ³ m ⁻² h ⁻¹)	1.25	0.43	0.5
SADp (m ³ m ⁻³)	73	22	32
SRT (d)	13	13	13
HRT _{total} (h)	1-3.5	1-3.5	1-4.75
V _{anoxic} (m ³)	2	2	2
V _{aerobic} (m ³)	2	2	2
V _{MT} (m ³)	1.41	1.61	0.67(+1.48)
Recirculation ratio	2	2	2

Table 51: Design parameters of MBR EAWAG

Table 52: Operating parameters at MBR EAWAG during the second experimental period (November 2007)

Zurich	B70	B80	B90
MBR state		low load	
HRT per configuration (min)	177	185	140
COD load (kg _{COD} .d ⁻¹)		10	
Sludge loading $(g_{COD}.Kg_{TSS}^{-1}.d^{-1})$		143	
average TS (g.kg ⁻¹)	8.5	11.7	10.1
Temperature (°C)		15.6	
Permeability (L m ⁻² h ⁻¹ Bar ⁻¹)	75-150	185-205	95

Firstly, a significant improvement in filterability can be noticed between the aerobic tank B60 and the filterability measured in all membrane tanks. The ΔR_{20} values decreased from 2 to $0.1*10^{12}$ m⁻¹ (in the most extreme cases). Therefore, it seems the filterability is improving from poor to good just by passing from the aerobic tank to the membrane tank in specific cases.

Then, large filterability fluctuations within each membrane tank can also be observed whereas the filterability of the B60 (aerobic tank) remained constant. The ΔR_{20} values

varied from 0.45 to $1.89*10^{12}$ m⁻¹, 0.1 to $0.48*10^{12}$ m⁻¹ and 0.1 to $0.98*10^{12}$ m⁻¹ in the tank B70 (Kubota), B80 (Zenon) and B90 (Puron), respectively.



Figure 120: Filterability variations in each tank in MBR EAWAG during the second experimental period (November 2007)

Activated sludge upconcentration and recirculation ratio

As presented in Figure 121, the total solids (TS) concentration measured in the tank B60 (7.9g.kg^{-1}) is significantly lower than the TS concentration measured in the membrane tanks (from 8 to 14.5 g.kg⁻¹). Based on Equation (6-6-1) and on the recirculation ratio reported in Table 51, i.e. equal to 2 in each membrane tank, the TS content in every membrane tanks should be close to 12 g.kg⁻¹. However, strong variations in TS content between tanks can be observed.

Firstly, the recirculation ratio may be different from the designed values. If the recirculation ratios reported are effective, it is likely that the membrane tank B70, B80 and B90 are not uniformly mixed and each present a different hydraulic flow distribution. These differences in mixing and flow distribution result in significant variations in TS content between the membrane tanks. These variations in TS content also correspond to different degrees of activated sludge upconcentration in the membrane tank.



Figure 121: Results obtained in the EAWAG pilot plant also composed of several membrane tanks equipped with different membrane configurations

A significant correlation can be demonstrated statistically between the TS concentration and the filterability with a Pearson value equal to -0.90 (p=0.03). As for the results concerning MBR Cranfield, activated sludge upconcentration is likely to be responsible for the filterability improvement.

As presented in the previous section, the TS concentration in the membrane tank, and therefore the activated sludge upconcentration process, depends on the recirculation ratio set from the membrane tank. However, in the case of MBR EAWAG, the TS measurements do not match with the theory. Whereas similar TS contents should be found in every membrane tank, significant variations were measured. The significant variations in TS content however result in significant differences in filterability behaviour. It can still be assumed that a significant activated sludge upconcentration occurred in the membrane tank (based on the TS measurements) and that activated sludge upconcentration resulted in an improvement of the activated sludge filterability due to some clarification effect (see *previous section*).

6.6.3. General overview - MLSS and filterability

In regard to the data presented in the previous section, MLSS content could be considered a parameter showing a direct correlation with activated sludge filterability measured with the DFCm. The filterability data are plotted along with the average MLSS value for each experimental period in Figure 122.



Figure 122: Filterability as a function of the average MLSS content

No significant statistical correlation was underlined between MLSS content and filterability. Good filterability and poor filterability were measured for the same MLSS content (11 g.L⁻¹ for instance). This is also in accordance with the work of Geilvoet (2010). During his research performed with the DFCm, Geilvoet (2010) did not determine any direct correlation between MLSS and filterability. The influence of MLSS content on activated sludge filterability is likely to be at the most of a secondary order.

Therefore, the filterability improvement presented in the previous section cannot be due to the MLSS content. The *activated sludge upconcentration* has to be considered in the *process* which leads to a filterability improvement. It is not the MLSS content as a *state* which influences the filterability of the activated sludge, but the upconcentration *process* of the activated sludge occurring under specific conditions.

6.7 SMP and filterability

As already mentioned in *Chapter 2*, soluble microbial products (SMP) are considered by many research groups to be a predominant foulant in the MBR filtration process. SMP are usually investigated in terms of protein and polysaccharide concentrations, measured with the well-established colorimetric methods of Dubois et al. (1956) and Lowry et al. (1951). However, contradictory results were often reported (Drews et al., 2007) and no clear consensus was made between SMP and MBR membrane performances up to date. The SMP concentrations in the free water phase determined during our study are presented in this section. SMP concentrations, expressed as protein and polysaccharide concentrations are plotted along with the filterability data in Figure 123 and Figure 124.



Figure 123: Filterability plotted along with the protein concentration in the free water phase

During this experimental study, large fluctuations in filterability quality and SMP concentrations were observed. The ΔR_{20} values varied between 0.05 and 5*10¹² m⁻¹. The protein and polysaccharide concentrations varied between 1 and 35 mg.L⁻¹ and 1 and 20 mg.L⁻¹, respectively. However, for an identical protein or polysaccharide concentration, a wide range of filterability qualities can be observed. For instance in a polysaccharide concentration of 15 mg.L⁻¹, ΔR_{20} values ranges from 0.05 to 2*10¹² m⁻¹. Therefore, no significant direct correlations could be found between filterability and SMP during this study (Figure 123 and Figure 124).



Figure 124: Filterability plotted along with the polysaccharide concentration in the free water phase

These results confirmed the suggestion of Kimura et al. (2009). The authors reported that monitoring SMP concentrations by conventional colorimetric methods is insufficient to elucidate membrane fouling in MBR due to the heterogeneity of different MBR mixed liquors. Authors suggested that methods like excitation-emission matrix (EEM) spectroscopy which delivered more detailed information about SMP characteristics should be considered. De la Torre et al. (2009) also tried to figure out a new indicator in order to better comprehend SMP involvement in MBR fouling. The authors reported good correlations between TMP increase and a specific type of polysaccharide. Finally, Geilvoet (2010) reported that the sub-micron particles which greatly caused the fouling process represented at the most a concentration of 1 mg.L⁻¹. It is therefore likely that the colorimetric methods used in this study were not specific enough.

With respect to all these aspects, SMP are possibly involved in the MBR fouling process due to their physical properties (Drews et al., 2007). However, new methods more orientated towards characterising specific properties like size or different components should be implemented to comprehend the degree of involvement of the SMP in the fouling process. The conventional colorimetric methods cannot be considered anymore appropriate for MBR membrane fouling investigations.

6.8 The significance of apparent viscosity in full scale MBR

In the scope of this research, a study was performed concerning the significance of apparent viscosity in full-scale MBR applications. Ten large-scale MBR plants were investigated over the past two years, and data needed to calculate the apparent viscosity of activated sludge for each plant were collected. The first aim of this study was to quantify variations of activated sludge apparent viscosity in full-scale municipal MBR applications. The second part of this study investigated whether this range of variations in apparent viscosity of activated sludge impacts MBR activated sludge filterability and can be correlated with full-scale MBR permeability in order to determine its relevance as a fouling control parameter.

6.8.1. Apparent viscosity calculation

As many parameters were needed for the calculations of the apparent viscosity, the dimension of all of them is summarized in Table 53.

Table	53:	Nomenclature	of	the	parameters	needed	for	the	activated	sludge	viscosity
calcul	atior	ı									

Nomenclature	
d_{mem}	Membrane diameter (m)
f_{mem}	Fanning friction factor of the membrane (-)
g	Gravity acceleration (= 9.81 m s^{-2})
h	Column height (m)
J	Permeate flux $(m \cdot s^{-1})$
k	Flow consistency index (Pa·s ⁿ)
L _{mem}	Membrane length including pressure sensor headers (m)
n	Flow behaviour index (-)
U_{g}	Superficial gas velocity $(m \cdot s^{-1})$
ΔP_{mea}	Pressure drop measured by the sensor (Pa)
ΔP_{mem}	Pressure drop along the membrane (Pa)
Re	Reynolds number (-)
Re _{MR}	Metzner and Reed Reynolds number (-)
TSS	Total suspended solids $(g \cdot L^{-1})$
U _{mem}	Velocity at the membrane (m)
Ϋ́w	Shear rate at the wall (s^{-1})
η_{a}	Apparent viscosity (Pa·s)
ρ	Density (kg·m ⁻³)
τ	Shear stress at the wall (Pa)

For each experiment conducted with the DFCm, the pressure loss along the membrane tube was monitored with two pressure sensors. The DFCm was operated under a single-phase flow and well-defined hydrodynamic conditions. Therefore, pressure drop values and shear stress present in the membrane can be theoretically derived.

The pressure drop along the membrane can be determined from the readings of the pressure sensors (ΔP_{mea}):

$$\Delta P_{mem} = \Delta P_{mea} - \rho g h \tag{6-8-1}$$

where ρ is the density, g is the gravity acceleration and h is the column height. The column height in this particular case, due to the membrane's vertical orientation, is the same as the length of the membrane (L_{mem}). The sludge density was determined using the following relationship (Metcalf&Eddy, (2003)):

$$\rho = 1000 + 0.2 MLSS \tag{6-8-2}$$

Subsequently, the shear stress (τ_w) can be calculated based on Bernoulli's law and mass conservation:

$$\tau_{w} = \frac{1}{4} \frac{d_{mem}}{L_{mem}} \Delta P_{mem}$$
(6-8-3)

where d_{mem} is the membrane diameter (= 0.008 m). Alternatively, shear stress can be defined as (Coulson et al., (1999)):

$$\tau_{w} = \frac{1}{2} f_{mem} \rho u_{mem}^{2}$$
(6-8-4)

where u_{mem} is the liquid cross-flow velocity in the membrane (1 m·s⁻¹). Hence, the Fanning friction factor (f_{mem}) can be determined by combining Equations (6-8-3) and (6-8-4):

$$f_{mem} = \frac{1}{2} \frac{d_{mem}}{L_{mem}} \frac{\Delta P_{mem}}{\rho u_{mem}^2}$$
(6-8-5)

From the Fanning friction factor, the Reynolds number can be calculated depending on the flow regime:

Laminar (
$$\text{Re}_{MR} \le \text{Re}_{MR,c}$$
): $f = 16 \,\text{Re}_{MR}^{-1}$ (6-8-6)

Turbulent (
$$\operatorname{Re}_{MR} \ge \operatorname{Re}_{MR,c}$$
): $f = 0.0792 \ n^{0.6/5} \operatorname{Re}_{MR}^{-0.25}$ (6-8-7)

where the Reynolds number is defined by Metzner and Reed for a power-law (non-Newtonian) fluid as:

$$\operatorname{Re}_{MR} = \frac{\rho u_{mem} d_{mem}}{\eta_a}$$
(6-8-8)

where η_a is the apparent viscosity of the fluid and the critical Reynolds number (Re_{*MR,c*}) is defined by:

$$\operatorname{Re}_{MR,c} = \frac{6464 n \left(2+n\right)^{\frac{2+n}{1+n}}}{\left(3 n+1\right)^2}$$
(6-8-9)

The apparent viscosity cannot be calculated from Equation (6-8-6) without knowing the Reynolds number. The latter can be determined from Equations (6-8-4) or (6-8-5), requiring the determination of the flow regime. A model proposed by Rosenberger et al. (2002) was used to estimate Re_{MR} and Re_{MR,c}. In this model, the fluid consistency index *k* and the flow behaviour index *n* for MBR activated sludge used in the pseudo-plastic fluid model ($\tau_w = k \dot{\gamma}^n$) can be calculated as:

$$k = 0.001 \exp\left(2 MLSS^{0.41}\right) \tag{6-8-10}$$

$$n = 1 - 0.23 MLSS^{0.37} \tag{6-8-11}$$

and the apparent viscosity for a flow in a pipe can be calculated as:

$$\eta_a = k \left(\frac{3n+1}{4n}\right)^n \left(\frac{8u_{mem}}{d_{mem}}\right)^{n-1}$$
(6-8-12)

Varying theoretically the total suspended solids from 0 (water) to 20 g.L⁻¹, the Reynolds number (Equation (6-8-7)) and the critical Reynolds number can be calculated. Results are presented in Figure 125.


Figure 125: Evolution of the Reynolds number as a function of the MLSS and the crossflow velocity

Based on Figure 125, a laminar regime can be achieved at a CFV of 1 m.s^{-1} with an MLSS content above 6 g.L⁻¹. Full-scale MBRs are usually operated with an MLSS content between 8 and 12 g.L⁻¹. Therefore, a laminar regime in the DFCm can be assumed for our research study.

Equation (6-8-6) can be used to calculate the Reynolds number and Equation (6-8-8) can then be used to calculate the apparent viscosity of the activated sludge from the experimental data.

As a remark, it can be observed that the experiments practised with the mixed liquor collected at MBR Trondheim were performed under a different flow regime (very low MLSS). Therefore, these results can not be relevantly compared with the results from other MBR plants.

6.8.2. Shear rate in the DFCm: Comparison with a submerged MBR

By definition, the shear rate γ in a tube is equal to:

$$\dot{\gamma} = \frac{\tau_w}{\eta_a} \tag{6-8-13}$$

A constant shear rate in the DFCm can be calculated based on a laminar regime and is equal to 1000 s^{-1} . In a bubble column bioreactor, the shear rate is defined by the following expression (Pereza et al., 2006):

$$\dot{\gamma} = \left(\frac{g\,\rho U_g}{\eta_a}\right)^2 = \left(\frac{g\,\rho U_g}{k}\right)^{\frac{1}{n+1}} \tag{6-8-14}$$

where U_g is the superficial gas velocity. The gas velocity for a submerged system usually ranges between 0.25 and 0.1 m.s⁻¹ (Verrecht et al., 2008). Figure 126 shows the relation between MLSS and shear rate at different superficial gas velocities. Depending on the gas velocity, different shear rate values are obtained, varying between 100 and 450 s⁻¹. It is important to point out that Equation (6-8-14) is usually used to calculate shear rate values in bubble column reactors, i.e. without membrane modules included. Therefore, shear rate values might be under predicted, but still give a fair estimation of the expected shear rate order of magnitude. This estimation is supported by Chan et al. (2007) who observed an average shear rate value of 500 s⁻¹ in submerged hollow fibre membrane modules. They measured maximal shear rate values or peak values up to 3000 s⁻¹. Iversen et al. (2008) reported shear rate values between 500 and 1500 s⁻¹ in a submerged flat sheet module. Apparent viscosity values calculated also seem to be in the range of activated sludge apparent viscosity values measured by Itonaga et al. (2004) and Khongnakorn et al. (2008).

Therefore, keeping in mind the non-Newtonian behaviour of activated sludge and the shear rate applied in both systems (i.e. the DFCm and a submerged MBR), apparent viscosity values calculated from the DFCm experiments can be considered representative for submerged MBR processes.



Figure 126: Evolution of the shear rate in a bubble column depending on the MLSS and the gas velocity

6.8.3. Apparent viscosity versus MLSS, temperature and SMP

Effect of MLSS

Calculated apparent viscosities are plotted along with the MLSS in Figure 127. As already shown by Rosenberger et al. (2002) and Seyssiecq et al. (2007), apparent viscosity values are strongly correlated with MLSS content. The apparent viscosity values varied between 0.0058 and 0.0146 Pa.s when the MLSS increased from 5.2 to 17.4 g.L⁻¹. It is between six and fourteen times the apparent viscosity of water at 20° C.

Table 54: Pearson correlation (r_p) coefficient of operational parameters and apparent viscosity

(** correlation significant at the 0.05 level, 2-tailed)

	Pearson coefficient (r _p)	p value
MLSS	0.5813**	0,00
Temperature	0,0527	0,53
ΔR_{20}	(-)0.1766	0,06
Permeability	0.24075	0,06
Proteins	0.2040	0,09
Polyssacharides	(-)0.1021	0,39

Statistical analysis results are presented in Table 54. The Pearson coefficient for the correlation between apparent viscosity and MLSS is equal to 0.581 (p=0.00), demonstrating a significant statistical correlation between apparent viscosity and MLSS. The latter was also observed by Wu et al. (2007) and Kornboonraksa et al. (2009). Moreover, a strong increase in the MLSS content of a municipal MBR could lead to an increase in apparent viscosity up to double its initial value. As already reported in the literature (Cicek et al., (2001);Lubbecke et al., (1995);Marrot et al., (2005)), high MLSS content and high apparent viscosities lead to mass and oxygen transfer limitations, and thus impact hydraulic regimes and energy requirements (Cicek et al., (2001), Manem et al., (1996)). These high apparent viscosity conditions, therefore, do not seem to be suitable for achieving low energy requirement in MBRs.



Figure 127: Apparent viscosity as a function of the MLSS

Effect of the temperature

Due to the impact of temperature on water apparent viscosity, an effect might be expected on activated sludge apparent viscosity as well (Metcalf&Eddy, (2003)).

The apparent viscosity values calculated in this study are plotted along with temperature in Figure 128a. Even normalized for MLSS (apparent viscosities expressed in Pa.s/(g.L⁻¹) in Figure 128b), there is no clear correlation between apparent viscosity values and temperature in the range of investigated MBR plants (from 9.7 to 27.4°C). The Pearson coefficient is equal to 0.053 (p=0.53). Therefore, no significant statistical correlation with apparent viscosity and temperature was determined in this study. This is in contradiction with the results presented by Wu et al. (2007) and Wang et al. (2006). A statistically significant correlation was found between temperature and apparent viscosity during their long-term pilot-scale study. However, during each DFCm short-term experiment, the MBR plants investigated were in steady-state conditions whereas the correlation between temperature and apparent viscosity was observed in their study when a change in sludge retention time, i.e. MLSS, was occurring. Therefore, combined effects are very likely responsible for the different results obtained.

From a statistical point of view and based on the DFCm data set, temperature does not have a direct significant impact on apparent viscosity within the range 9.7 to 27.4°C. Given the relatively small impact of temperature on the apparent viscosity of water in this temperature range, this could have been expected. Apparent viscosity of activated sludge results from a combination of various parameters. Therefore, the impact of the MLSS seems predominant, making the temperature impact irrelevant. However, the temperature might have an indirect impact on apparent viscosity through the sludge growth and composition and, hence, the MLSS.



Figure 128: Apparent viscosity versus (a) temperature (b) temperature normalized for MLSS for all full-scale MBRs

Effect of the SMP

The statistical correlation between apparent viscosity and SMP characterised in this study as proteins and polysaccharides are presented in Table 54. Pearson coefficients equal to 0.204 (p=0.09) and -0.102 (p=0.39) for proteins and polysaccharides, respectively, were calculated. Correlation between apparent viscosity and SMP is not statistically significant in this study.

Different findings about the impacts of SMP and bound extra cellular polymeric substances (bound EPS) were presented in the literature. Bound EPS showed usually a better correlation with apparent viscosity than SMP. Rosenberger et al. (2002) showed a fairly good correlation between total EPS (bound EPS + SMP). Kornboonraksa et al. (2009) showed also an indirect correlation between bound EPS and apparent viscosity through MLSS. However, because both studies focus at least to some extent, on MBRs treating high-strength wastewater, a relatively high amount of bound EPS was measured (100 to 200 mg/gVSS). Meng et al. (2007) also observed an increase in activated sludge apparent viscosity when the bound EPS amount increased from 45 to 160 mg/gVSS in lab-scale MBRs due to the growth of filamentous bacteria.

Unfortunately, a bound EPS analysis was not performed during this study. However, the bound EPS content in steady-state, full-scale municipal MBRs is expected to be lower

than in the cited studies, i.e. between 10 and 30 mg/gVSS (Stowa report). Furthermore, even if bound EPS can be related to apparent viscosity for some specific industrial MBRs with specific influent or small-scale membrane bioreactors, no relevant relation between SMP and apparent viscosity was found in steady-state, full-scale municipal MBR plants. Therefore, in accordance with the literature and based on the DFCm data set, no statistical correlation was found between apparent viscosity and SMP.

6.8.4. Apparent viscosity versus activated sludge filterability and large-scale MBR permeability

Apparent viscosity is plotted along with activated sludge filterability data in Figure 129a. Large fluctuations in activated sludge filterability were observed during this measurement campaign with variations in the ΔR_{20} from $0.05*10^{12}$ m⁻¹ up to $4*10^{12}$ m⁻¹. However, no clear correlation was found between apparent viscosity and filterability during this study.

The apparent viscosity variations from 0.0058 to 0.0146 Pa.s do not seem to significantly impact the activated sludge filterability. The Pearson coefficient evaluating the correlation between the apparent viscosity and the filterability is presented in Table 54. The Pearson coefficient is equal to -0.177 (p=0.06) and indicated a weak correlation between filterability and apparent viscosity. Due to the low Pearson coefficient value, the correlation was considered statistically non-significant. Hence, apparent viscosity in the bulk should not be considered a major contributor for activated sludge filterability deterioration within the range of full-scale municipal MBR plant applications.

Apparent viscosity values are plotted along with pilot and full-scale membrane permeability in Figure 129b. Depending on the location and the season, permeability of the MBR plants varied between 50 and 600 $\text{L.m}^{-2}.\text{h}^{-1}.\text{Bar}^{-1}$. The Pearson coefficient revealing the correlation between apparent viscosity and permeability is presented in Table 54. It is equal to 0.241 (p=0.06) and indicated a weak correlation between permeability and apparent viscosity. Therefore, apparent viscosity does not correlate significantly with the permeability data in the permeability range of the investigated MBR plants in this study.

These findings are in accordance with the current MBR literature. Kornboonraksa et al. (2009) considered apparent viscosity as a sub-factor affecting membrane filtration process through its good correlation with MLSS. Li et al. (2008) confirmed this finding; they found a moderate statistical correlation between apparent viscosity and membrane fouling resistance, mostly due to the impact of MLSS content on the increase in membrane resistance in their study. Furthermore, Rosenberger et al. (2002b) also concluded that, despite strong variations in apparent viscosity, there was no influence on filterability.

Wu et al. (2007) correlated apparent viscosity and filterability. However, as discussed previously, the pilot-scale MBR operated in these researchers' study reached relatively high MLSS values compared to full-scale municipal MBRs and was not always operated under steady-state conditions.

Yamamoto et al. (1994) and Rosenberger et al. (2002) reported that critical concentrations of MLSS of 30-40 g.L⁻¹ were detrimental to membrane performances and had strong impact on membrane operations, especially due to membrane module clogging. Itonaga et al. (2007) advised not to work far beyond 15g.L⁻¹ in order to avoid high apparent viscosity increase and the formation of a sticky cake layer on the membrane surface.

However, in the range of current municipal full-scale application (6-15 g.L⁻¹), no clear links were underlined between apparent viscosity and membrane permeability loss in the literature. Khongnakorn et al. (2007) reported that even if an MBR should not be operated with MLSS content above $15g.L^{-1}$ due to the strong risk of membrane clogging, a sudden increase in TMP during their experiments could not be attributed to apparent viscosity or MLSS content increase from 4 to 16 g.L⁻¹. Hong et al. (2002) also reported a very small influence of MLSS and apparent viscosity on permeate flux decline for the range 3.6-8.4 g.L⁻¹.

Therefore, based on the statistical analysis performed and on the cited literature, apparent viscosity fluctuations observed in this study are not statistically significant enough to impact large-scale plant permeability behaviour in the ranges of MLSS, SRT and temperature investigated in the studied pilots and full-scale municipal MBRs.



Figure 129: Apparent viscosity versus (a) activated sludge filterability and (b) membrane permeability for all full-scale MBRs

6.8.5. Concluding remarks

Activated sludge apparent viscosity values for each experiment could be calculated experimentally. Within the representative range for full-scale applications (in terms of SRT, MLSS and temperature), the apparent viscosity values were found to be in the range of 0.0058 to 0.0146 Pa.s. The main factor influencing activated sludge apparent viscosities was MLSS content confirming the findings in the literature.

Temperature did not directly affect activated sludge apparent viscosity within the temperature range at pilot and full-scale municipal MBR plants (9.7 to 27.4 °C). In terms of reversible fouling potential and membrane performance, activated sludge filterability and MBR plant permeability were not affected by activated sludge apparent viscosity

variations. Therefore, even if apparent viscosity plays a major role in terms of oxygen transfer efficiency, it is not relevant when optimising membrane fouling control and membrane performance of current, full-scale municipal MBR applications. However, it still should be considered a predominant factor in regard to clogging issues.

6.9 Membrane configuration and filterability

As already presented in *Chapter 2*, membrane characteristics trough membrane materials, chosen shape, and hydrophobicity have a determinant effect in terms of membrane fouling potential. Furthermore, as reported by Meng et al. (2009) in their review, aeration of the membrane modules, through shear force, turbulence and their impact on the floc size distribution, play a key role in membrane fouling prevention. Therefore, the different membrane configurations applied to *immersed MBRs*, namely hollow fibre, flat sheet and multi-tube, due to their own specificity in terms of membrane material, turbulence promotion and aeration devices might have an impact on the activated sludge filterability. This assumption will be tested in regards of the results of the DFCm in this section.



Figure 130: Effect of the membrane configuration on activated sludge filterability

The filterability expressed as ΔR_{20} is plotted as a function of the membrane configurations of the MBR plants investigated in Figure 130. Average filterability values and standard variations are reported in Table 55. The filterability is also plotted as a function of the membrane configurations in Figure 131. Average values for the hollow fibre and the flat sheet membrane configurations are almost identical with ΔR_{20} values equal to 0.78 and 0.79*10¹² m⁻¹, respectively. The average ΔR_{20} value for the multi tube configurations. It is likely due to the fact that many fewer experiments were performed with activated sludge coming from an MBR equipped with a multi-tube configuration.

Table 55: Detailed information concerning the filterability for each configuration

$\Delta R_{20} (*10^{12} \text{ m}^{-1})$					
Configuration	Average	Stdv	Max	Min	
Flat sheet	0.79	0.56	1.70	0.02	
Hollow fibre	0.78	1.17	4.31	0.04	
Multi tube	1.29	1.28	3.24	0.11	

Large standard deviations can also be observed for all the configurations. Good and poor filterability data were measured for each MBR configuration. Therefore, it can not be stated that a specific configuration favoured good or poor activated sludge filterability from our set of experiments.



Figure 131: Filterability as a function of the MBR membrane configuration

Based on this observation, activated sludge is likely to be related to the biological process more than to the membrane filtration process in immersed MBR applications. Activated sludge filterability can be therefore considered independent of the membrane filtration step and can then be optimised or influenced independently from it.

6.10 Specific cake resistance, accumulation of sub-micron particle and compressibility

6.10.1. DFCm outputs

As presented in *Chapter 3*, the filterability data obtained can be separated into two side effects according to the work of Geilvoet (2010).

One, $\alpha_R c_i$ is the product of the concentration of substances accumulating in the cake layer and the specific cake resistance caused by these substances. It gives an indication of the mass deposition involved in the cake layer build up. The compressibility coefficient *s* gives information about the compressibility of the cake layer under the operating conditions set in the DFCm. Thanks to this separation, extra information can be gathered concerning the activated sludge filterability and the cake layer build up especially. Values for $\alpha_R c_i$ and *s* are presented in Table 56.

It is important to notice that a correlation coefficient equal to 0.63 was calculated between $\alpha_R c_i$ and *s*. $\alpha_R c_i$ and *s* can therefore be considered correlated. This should be taken into account when interpreting the data.

Location	Period	$\alpha_{\rm R} c_{\rm i} (10^{12} {\rm m}^{-2})$	S
Schilde	feb-07	0.048	0.18
	nov-07	0.144	0.17
	apr-08	0.110	0.17
	aug-08	0.021	0.08
	apr-09	0.138	0.16
	jun-07	0.003	0.00
	jan-08	0.075	0.00
AMEDEUS	jun-07	0.009	0.00
	jan-08	0.048	0.06
	sep-06	0.005	0.00
Monheim	mrt-07	0.033	0.24
	aug-08	0.004	0.00
Nordkanal	jul-07	0.003	0.00
Norukariai	nov-08	0.015	0.00
Trento	nov-07	0.073	0.10
Tiento	jul-08	0.102	0.13
	0	0.072	0.04
Trondheim	2	0.069	0.08
rionaneim	4	0.061	0.04
	6	0.038	0.02
	mei-07	0.05	0.06
B70	nov-07	0.05	0.06
	nov-07	0.05	0.06
B80	mei-07	0.02	0.00
	nov-07	0.02	0.00
	nov-07	0.04	0.05
B90	mei-07	0.05	0.00
	nov-07	0.04	0.00
	nov-07	0.04	0.04

Table 56: $\alpha_R c_i$ and s values for the different experimental periods and MBR plants.

 $\alpha_R c_i$ is plotted along with the filterability data in Figure 132. A significant correlation is demonstrated statistically between the filterability and the mass involved in the cake layer build up with a Pearson coefficient equal to 0.97 (p=0.00). It is in accordance with the results of Geilvoet (2010) which reported that the total cake layer resistance was predominantly determined by the concentration of substances accumulating in the cake layer.

The DFCm test is a short term measurement. As already reported in *Chapter 3*, the main fouling mechanism involved in the fouling process during DFCm tests is the cake layer formation. Therefore, DFCm tests are too short to be able to underline significant changes due to a long term fouling mechanism like absorption.

Due to the fact that mostly cake layer filtration is involved in the DFCm measurements, it is logical that an increase in the mass of the particles involved in the cake layer shows a significant correlation with the filterability.



Figure 132: Filterability as a function of the mass involved in the cake layer build up

The compressibility coefficient *s* is plotted along with the filterability data in Figure 133. No significant correlation was demonstrated statistically. However, few conclusions based on graphic analyses can be drawn. Most of the compressibility coefficient *s* were equal to 0 when the ΔR_{20} values measured were below $0.5*10^{12}$ m⁻¹. Therefore, well filterable activated sludge is likely to form an incompressible cake layer at the membrane (*s*=0). Most of the ΔR_{20} values above $1*10^{12}$ m⁻¹ were composed of a compressibility component (*s*>0). Therefore, poor filterable activated sludge is likely to form a compressible cake layer.



Figure 133: Filterability as a function of the compressibility of the cake

6.10.2. Impact of the MLSS concentration

 $\alpha_R c_i$ coefficient is plotted as a function of the MLSS concentration in Figure 134. As expected, no statistical correlation can be found. As presented in *Chapter 3*, the particles involved in the cake layer formation are likely to be smaller than 1µm due to the dragging forces resulting from the crossflow velocity applied in the DFCm. MLSS are mostly composed of flocs with a size ranging between 30 and 100 µm (*Chapter 3*). Therefore, it is in accordance with the literature that no correlation can be found between the activated sludge MLSS concentration and the amount of material involved in the cake layer build-up.

However, some interesting remarks can be drawn from this comparison. Based on Figure 134, it seems that the larger cake layer build up is obtained by an MLSS concentration around 10 $g.L^{-1}$. Above this concentration, the cake layer is likely to be composed of a smaller particle amount (in mass). Therefore, it is likely that a large amount of particles (potential foulants and cake layer components) remained trapped within the activated sludge floc network when the MLSS concentration increased.



Figure 134: $\alpha_R c_i$ as a function of the MLSS content

The compressibility coefficient *s* is plotted as a function of the MLSS concentration in Figure 135. No statistical correlation between compressibility and MLSS concentration was demonstrated during our study. However, similar to the $\alpha_R c_i$ coefficient, a maximum compressibility coefficient was found in an MLSS concentration close to 10 g.L⁻¹. It is then likely that different cake structures are formed at the membrane depending on the MLSS concentration of the activated sludge. Above a certain MLSS concentration, the cake layer formed at the membrane is likely to be less compressible. As a consequence, better filtration performances can be expected under relatively high MLSS content.



Figure 135: s coefficient as a function of the MLSS content

As already reported in *Chapter 2* and *Section 6.6*, MLSS concentration is not considered to have a direct impact on activated sludge filterability and membrane filtration performances, mostly due to the fact that smaller particles have predominant fouling potential compared to the flocs themselves. However, due to the separation of the filterability term into two different components, the amount of material involved in the cake layer build-up $\alpha_{\rm Rc_i}$ and a compressibility coefficient *s*, an indirect effect of the MLSS concentration can be underlined.

Depending on the MLSS concentration, different cake layer in terms of mass of material and structure are formed. From the results presented in Figure 134 and Figure 135, it is likely that the cake layer formed by an activated sludge with an MLSS concentration above a certain value (10 g.L⁻¹ in our study) will be composed of fewer particles (in mass) and with a less compressible structure than the cake layer formed by an activated sludge with a lower MLSS content. It is likely due to the fact that sub-micron particles with a diameter below 1 μ m have more chance to be trapped within the floc network under high MLSS concentration conditions (clarification effect).

6.11 Summary

The results of the DFCm measurement campaign were discussed in this chapter. Large fluctuations in filterability were observed during this research and were partly explained by different factors. A classification of importance of the effect of several parameters on the filterability of the MBR investigated can be formulated. Firstly, uncontrolled conditions and wastewater temperature can be identified:

- The *feedwater* quality is likely to be the *dominant factor* in terms of activated sludge filterability. Difficult wastewater, and stress on the activated sludge and foam occurrence have a strong influence on the activated sludge filterability.
- The *seasonal fluctuations* and the *temperature* do also have a *significant influence* on activated sludge filterability. A correlation was demonstrated statistically between filterability and temperature in the full-scale plant applications. The main cause for the filterability deterioration during winter time is likely to be a *sub-micron particle release* occurring due to poor flocculation conditions at *low temperatures*. In the same way, the main reason for the improvement of the activated sludge filterability during the summer time is likely to be a reduction of the sub-micron particle concentration in the free water due to the entrapment of these particles in the floc network. This strong floc network is due to a better flocculation state of the activated sludge at high temperatures.

Operating conditions and MBR design parameters can also be considered factors affecting activated sludge filterability:

- Under low recirculation ratio conditions, activated sludge upconcentration was
 observed within the membrane tank. This activated sludge upconcentration
 resulted in a significant filterability improvement. The activated sludge
 upconcentration is likely to be a process responsible for filterability improvement,
 i.e. entrapment of the sub-micron particles resulting in a clarification effect, under
 these specific operating conditions.
- Low activated sludge loading systems also showed better filterability quality than high loading system. Therefore, *activated sludge loading* influences the activated sludge filterability. However, it should be considered a fouling control parameter of *second order of influence* due to its relatively low effect on filterability compared to seasonal fluctuations or unexpected toxicity.

Some others parameters did not show significant correlation and impacts on activated sludge filterability:

• *SMP* did *not* show any *significant correlation* with activated sludge filterability. It is mostly due to the colorimetric methods chosen to quantify the SMP concentration. The conventional colorimetric methods should not be considered anymore appropriate for MBR membrane fouling investigations. New methods

more orientated towards characterising specific properties like size should be implemented to comprehend the degree of involvement of the SMP in the fouling process.

- Activated sludge apparent viscosity did not show any significant correlation with activated sludge filterability. Activated sludge filterability and MBR plant permeability were not affected by activated sludge apparent viscosity variations. Even if apparent viscosity plays a major role in terms of oxygen transfer efficiency, it cannot be considered a relevant parameter optimising membrane fouling control and membrane performances of current, full-scale municipal MBR applications. However, it still should be considered a predominant factor in regard to clogging issues.
- The membrane configurations of the immersed MBR plants did not affect the activated sludge filterability. Activated sludge is likely to be related to the biological process more than with the membrane filtration process. The differences in shear and hydraulic regimes promoted by different immersed configurations are not sufficient to significantly affect the activated sludge filterability.

Finally, interesting results were ascertained concerning the impact of the scaling-up of MBR plants on activated sludge filterability. Significant differences in terms of filterability could be observed between pilot and full-scale MBR plants. The differences in capacities (buffer), operating conditions, stress and the difficulty to maintain steady conditions lead to different behaviours in terms of activated sludge filterability in pilot-scale MBRs.

Based on these results and on the data collected during the research study, hypothesis will be formulated in *Chapter 7* in order to define the process set up leading to the best activated sludge filterability.

7 General framework and recommendations

From the measurement campaign presented in Chapter 4, ten discussion points were formulated and presented in Chapter 6. Wastewater temperature and properties, scale of the plants, feedwater and toxicity influences, activated sludge organic loading, activated sludge viscosity, membrane configurations, SMP concentrations, turbulence promotions and their indirect effects were discussed in relation to their impacts on activated sludge filterability. Based on the results of Chapter 6, a general framework can be formulated in order to define the process set up leading to the best activated sludge filterability.

7.1 From experiments to a general framework

7.1.1. MBR - activated sludge

Major differences between MBR activated sludge and conventional activated sludge can be resumed shortly:

- MBR activated sludge has a higher MLSS content
- This higher MLSS content results in a smaller average floc size (Le-Clech et al., 2006)
- The change in apparent viscosity due to the higher MLSS content may have a direct impact on the oxygen transfer efficiency

The research performed during this study and presented in *Chapter 6* also demonstrated some similar behaviour between MBR activated sludge and conventional activated sludge:

- Organic loading does play a role in MBR activated sludge filterability quality
- Differences in activated sludge filterability quality can be observed depending on the scale of the plants.

It was also demonstrated in this study that wastewater properties like temperature strongly affect activated sludge filterability. Furthermore, the research results showed that feedwater quality and toxicity have a predominant impact on the activated sludge filterability in MBR applications.

As a consequence, like in conventional activated sludge plants, more difficult operating conditions can be expected during the winter time, if complex wastewater needs to be treated and if toxic events occur.

However, in contrast with conventional applications, the quality of the permeate should not be affected under these described conditions whereas a slight degradation of the effluent quality can be expected, i.e. an increase of TS concentration in the effluent, in conventional systems. The deterioration of the filterability in MBR applications under these previous conditions will only result in permeability losses and therefore in additional operating and cleaning costs. In a worst case scenario, the deterioration of the filterability can lead to the complete shut down of the MBR for a limited period of time in order to perform an intensive cleaning protocol.

Like in conventional activated sludge applications, wastewater properties, feedwater quality and toxicity have a strong impact on the activated sludge filterability. However, as a major difference, the deterioration of the filterability in MBR applications should not affect the permeate quality as would be the case in conventional applications but might eventually lead to extreme case scenario where the MBR plants need to be shut down.

Furthermore, the results presented in *Section 6.9* showed that the membrane configuration in itself does not have a significant impact on the activated sludge filterability in immersed MBR applications. As a consequence, it is likely that the variations in activated sludge filterability in immersed MBRs are mostly due to changes in the biological part of the MBRs. The shear created in immersed configurations and the current operating protocols and filtration cycles implemented are likely not to have any influences on the activated sludge filterability.

The membrane configurations do not have a significant impact on the activated sludge filterability in immersed MBR applications. Consequently, it is likely that the large variations in filterability observed during this study are due to changes in the state of biology of the MBRs, i.e. flocculation state and particle size distribution.

As already reported by Geilvoet (2010), even if a non settleable activated sludge, namely due to an higher MLSS content, can be handled in an MBR applications, it is still crucial to obtain a well flocculated activated sludge. Therefore, membrane bioreactor technology remained simply an *elaborated activated sludge process*. Hence, all the rules which apply to conventional activated sludge plants in terms of treatment efficiency and favourable flocculation conditions remain *identical*.

As a consequence, operating conditions in MBRs can to be set based on conventional activated sludge knowledge in order to obtain a well-flocculated activated sludge. The conventional activated sludge process controls, which obtain high levels of treatment performances, can be recalled quickly (Metcalf&Eddy, 2003):

- Plug flow design with good tank separation
- Use of a selector
- Dissolved oxygen levels need to be maintained around 1.5 to 2 mg.L⁻¹ in the aeration tanks
- The amount of return sludge needs to be regulated
- The waste activated sludge amount needs to be well known and well controlled
- Routine microscopy can be implemented in order to detect activated sludge changes

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• In addition to the previous points, variations in loading and wastewater composition should be avoided, pH and temperature should be well controlled, a good mixing, sufficient nutrients and soluble BOD need to be provided in order to prevent bulking sludge occurrence

Based on the measurement campaign, other specific criteria may be formulated to achieve good activated sludge filterability. As presented in Figure 136, the MBRs showing the best activated sludge filterability are MBR Heenvliet, MBR Monheim and MBR Nordkanal. The simplified schemes of each of these MBRs are recalled in Figure 137 to Figure 139 and further criteria are presented in Table 41.



Figure 136: General overview of the activated sludge filterability of the MBRs investigated

With respect to the data presented, there are no clear patterns between the three MBRs. Significant differences can be observed between the three plants in terms of *membrane configurations*, i.e. flat sheet for MBR Heenvliet and hollow fibre for MBR Nordkanal and MBR Monheim, *tank adjustments*, i.e. separated for MBR Heenvliet and MBR Monheim and integrated for MBR Nordkanal and *recirculation ratio implemented*, namely 1.5, above 10 and 4 for MBR Heenvliet, MBR Monheim and MBR Nordkanal, respectively.

The according criteria of the three plants are:

- The three MBR plants are designed to achieve biological nutrient removal with well separated anoxic and aerobic tanks
- The three MBR plants present a relatively conservative design in terms of sludge loading

- The three MBR plants present some relevant buffer capacity
- The three MBR plants were not subjected to any toxic or foaming event during the measurement campaign.

It seems difficult to formulate more specific criteria than these general ones. The uniqueness of each MBR and the complexity of the process involved make it difficult to formulate clear statements.



Figure 137: Simplified schematic view of MBR Heenvliet



Figure 138: Simplified schematic view of MBR Monheim



Figure 139: Simplified schematic view of MBR Nordkanal

Table 57: Recirculation ratio, activated sludge temperature and F/M ratio for the full-scaleMBR plants investigated during this study (data from Varsseveld, Heenvliet andOotmarsum are adapted from Krzeminski et al. (2010))

	Monheim	Nordkanal	Schilde	Varsseveld	Heenvliet	t Ootmarsum	ENREM
Recirculation ratio from the MT	>10	4	6	-	1.5	-	4
Temperature max (°C)	18.3	19.5	17.2	20.3	22.1	-	24.0
Temperature min (°C)	8.4	9.5	10.5	11.5	11.9	-	11.9
F/M ratio winter (g _{COD} .kg _{TSS} ⁻¹ .d ⁻¹)	118.0	160.5	126.9	77.2	92.0	92.1	68.2
F/M ratio summer $(g_{COD}.kg_{TSS}^{-1}.d^{-1})$	54.8	195.5	90.8	96.8	27.9	83.2	68.2

Due to the fact that the membrane configurations of immersed MBR applications do not significantly affect the activated sludge filterability, the activated sludge of an immersed MBR can be simply considered an elaborated activated sludge process. As a consequence, besides the settling properties affected by higher MLSS contents, design and operating rules in terms of treatment efficiency and flocculation state should remain identical in immersed MBR applications. Organic loading and the scale of the plant should be considered determinant factors in terms of filterability. Wastewater properties like temperature, feedwater characteristics and toxicity are likely to affect MBR activated sludge in the same way as conventional activated sludge.

However, the consequences will be different. Poor activated sludge quality in conventional applications will likely result in the deterioration of the effluent quality. Poor activated sludge filterability will not necessarily result in the deterioration of the permeate quality but could lead to strong operational problems resulting in an increase of the operational and cleaning costs and, in a worst case scenario, the shutting down of the MBR plant for a limited period of time in order to perform an intensive cleaning.

7.1.2. MBR – membrane stage

As already presented in *Chapter 2*, the presence of a membrane stage in the MBR technology and its direct contact with the mixed liquor inevitably induce a fouling occurrence. However, several types of fouling mechanisms can be differentiated. The cake layer mechanism which is predominant on a short term basis can be separated from the pore blocking and absorption mechanisms occurring on a long term basis.

Short term fouling

One of the main conclusions of Geilvoet (2010) was that good activated sludge filterability was a primary prerequisite for an efficient MBR filtration process, especially due to the predominant role of activated sludge quality on reversible fouling occurrence.

As already presented by Geilvoet (2010), Lesjean et al. (2009) and discussed in *Section* 6.6, activated sludge filterability is strongly dependent on the amount of sub-micron particles present in the free-water phase with a diameter ranging from the membrane pore size (0.03 to 0.1μ m) up to 1.0μ m. Significant filterability improvements could be observed due to the retention of the sub-micron particles within the floc network (Lesjean et al, 2009). It is important to notice that the sub-micron particles due to their size can be

considered only a small fraction of the SMP, soluble EPS and loose EPS. Concerning short term fouling, it can be assumed that the membrane is mostly working as a rejecting barrier. Therefore, foulants in this research are characterised as a function of their physical properties, i.e. size, rather than as a function of their chemical specificities.

The sub-micron particles in the free-water phase do play a predominant role in terms of cake layer build up (Geilvoet, 2010). Therefore, the concentration of these particles needs to be controlled and sub-micron particles with a diameter below 1µm should be kept out of the free-water as much as possible.

Several techniques have been tried in order to retain the sub-micron particles within the floc network and therefore prevent them from being in the free-water phase:

- Chemical coagulants may be used to enhance activated sludge flocculation (metal salts, or natural and synthetic polymers). However the latest results showed some inconsistency (Iversen et al., 2009). Results can be considered arbitrary depending on the coagulant and the activated sludge. Furthermore, cautious statements should be formulated concerning their efficiency on the long term. It is likely due to the fact that the current coagulant or flux enhancers available are *not size specific*. They mostly target, in the best case, specific charge compounds. The coagulation process occurs randomly. As a consequence, bigger flocs are produced but there is no certitude that sub-micron particles will be trapped.
- Theoretically, strong turbulent promotion can prevent the sub-micron particles from reaching the membrane due to the back-transport phenomena induced. However, in regard to the diameter of these particles, an efficient turbulence promotion is likely not to be feasible at a reasonable cost. Based on the theory of backtransport velocity, the shear rate needed at the membrane wall to prevent particles of a specific size to deposit can be calculated. Results of these calculations are presented in Figure 140. For a production flux of 20 L.m⁻².h⁻¹, the shear rate which need to be provided by air scouring have to be superior to 5000 s⁻¹ in order to prevent the deposition of the particles with a diameter larger than 0.5µm. As presented in *Section 6.8*, current submerged MBR applications are operated under air-scouring conditions providing in average a shear rate around 500 s⁻¹ with peak values close to 1500 s⁻¹. Therefore, preventing sub-micron particle deposition by means of air-scouring is likely not to be achievable at a reasonable production flux.



Figure 140: Back-transport flux as a function of the shear implied by the turbulence and the diameter of the particles

Because flux enhancers are not operational at full-scale yet and turbulence promotions by means of air-scouring is likely not to be efficient in preventing the sub-micron particle deposition, new concepts or new filtration cycles need to be considered.

Based on the results of the *Section 6.6*, another technique can be developed to trap the sub-micron particles within the floc network. Local artificial flocculation can be achieved by activated sludge upconcentration in the membrane modules.

Under low recirculation ratio conditions from the membrane tank, an activated sludge upconcentration can be achieved in the membrane modules. It was presented in *Section* 6.6 that activated sludge upconcentration resulted in good MBR membrane performances and filterability improvement. As a consequence, radically new operating protocols and MBR design could be implemented and tested.

Tests need to be performed in order to define the adequate recirculation ratio allowing proper activated sludge upconcentration and *clogging prevention*. Indeed, under these operating conditions, the performance issue is switched from a fouling problem to a clogging prevention issue. Membrane suppliers have in the recent years made great efforts to improve the hydraulic flow distribution in their membrane modules. For instance, Kubota improved their air diffuser whereas Koch-Puron enclosed channels inbetween their fibres to enhance the activated sludge flow distribution. Therefore, activated sludge upconcentration in the membrane module may likely be achieved with the current MBR membrane technologies available without clogging occurrence.

Furthermore, as it was observed in *Chapter 6, low local shear* generated at the membrane wall is *not detrimental* for membrane performances and activated sludge filterability. New protocols could then be implemented like filtration cycle with limited aeration followed by a strong mechanical cleaning, i.e. back flush and/or a relaxation period with aeration for instance. The mechanical cleaning should then be set to enhance the vertical liquid flow velocity and a homogenous activated sludge flow distribution rather than being set to limit fouling occurrence.

The sub-micron particles in the free-water phase do play a predominant role in short term fouling. The concentration of sub-micron particles needs to be controlled and be kept out of the free-water. Effective coagulants are currently not available and turbulence promotion at the membrane wall is likely not to be able to prevent sub-micron particle deposition at a reasonable cost. Therefore new concepts need to be considered. As presented in Chapter 6, the activated sludge upconcentration observed in the membrane tank resulted in an improvement of the filterability and an improvement of the membrane performances. It is likely due to the entrapment of sub-micron particles in the floc network by the "artificial flocculation" created during the upconcentration process. New filtration cycles and operating modes with low recirculation and low aeration demand should be considered on these bases in order to reduce short term fouling.

Long term fouling

As already presented in *Chapter 2*, long term fouling can be considered a result of pore blocking, absorption and biofilm growth mechanisms onto the membrane. Due to the nature and composition of the activated sludge, particles and specific species will always deposit *with time* at the membrane. Whatever the operating conditions set in the MBR plants, deposition of particles and microorganism growth on the membrane will occur on the *long term*. Therefore, these mechanisms can hardly be manipulated in current MBR applications, except maybe in membrane coating or quorum sensing control (Yeon et al., 2008).

Lon term fouling should then be considered simply a cleaning issue. As hardly any efficient preventing strategy can be implemented, long term fouling should be considered one of the variables to take into account during chemical membrane cleaning. Long term fouling should be considered a consequence of the lack of effectiveness of the current chemical/physical cleaning protocols.

Therefore, more efficient chemical cleaning protocols have to be developed in order to counter balance long term fouling.

7.2 From the general framework to the next practise

Based on the framework formulated previously, recommendations for the next practices can be announced. Recommendations for further research perspectives and new design criteria for full-scale MBR applications will be presented in this section.

7.2.1. Lab-scale research

Membrane costs remained one of the major bottlenecks of the MBR market development growth. In order to become competitive, membrane costs should drop or membrane flux production rate should increase significantly. Therefore, further efforts should be put into membrane material research in order to develop mass production membrane at low cost or/and high permeable membrane.

Membrane coating is also a part of the MBR research which should receive special focus. Improvement of the current membrane by coating addition could limit particle deposition and enhance correct sustainable production flux.

As a last remark and as already presented in the previous section, lab research should also focus on developing new chemical cleaning protocols to prevent long term fouling. Enzymatic cleaning might be one of the technologies which can be investigated (Te Poele et al., 2006).

7.2.2. Pilot-scale research

Based on the results of *Chapter 6*, MBR pilot-scale research should be reconsidered. Significant differences in terms of filterability could be observed between pilot and full-scale MBR plants in our study. It is mostly due to differences in surrounding conditions and to the lack of redundancy which prevent continuous and stable operations at the pilot-scale. Furthermore, the MBR process is currently well-established and is considered a robust technology. Therefore, feasibility tests at the pilot-scale before the building of a full-scale plant installation are likely not to be compulsory anymore. Due to the difference in filterability between both scales, pilot-scale studies are likely not to bring any additional knowledge from feasibility tests compared to what can be found in the literature or in the plant history.

A pilot-scale study can still be useful in terms of operating condition optimisation. However, rather than building a complete pilot-scale plant with its own specific biology, it is likely that a separate parallel membrane tank coupled to an existing full-scale MBR plant can bring more conclusive information in order to develop cost effective MBR operating modes.

As an example, the operating mode proposed in the previous section, i.e. filtration mode without aeration followed by a strong mechanical cleaning, could be tested in a parallel pilot-scale membrane tank and, after figuring out the proper operating sequences, could later on be implemented in the full-scale plant aside. The fact that the pilot-scale trials could be performed with the activated sludge from the full-scale MBR plant will increase the chance of success of the scaling up.

7.2.3. Full-scale plant applications

As already presented in *Chapter 2*, water discharge regulations are becoming more and more stringent. Coupling this to the fact that the MBR process is becoming a well accepted robust technology, the MBR market should grow steadily in the coming years. However, several factors have still strongly hampered this development:

- As reported previously membrane investment costs remain high
- Operating costs and especially energy requirements are still too high
- Permeate production flow remains relatively low
- Design of a new MBR plant has to be based on the maximum flow

Due to all these factors, MBR market potential development in the municipal sector has to come from retrofitting and upgrading existing wastewater treatment plants. Several essential design criteria can be formulated to help further this development:

- An optimal use of the membrane surface area implemented can be achieved if the design of the MBR is based on the dry weather flow or for plants connected to a separate sewage network.
- As presented in the previous section, a pilot-scale study could be coupled or replaced by a good history of the plants. Thorough and accurate knowledge of the surrounding conditions is compulsory and should be well documented in order to produce a proper design. A good knowledge of the extreme conditions which can be encountered is determinant to predict the plant performances dynamically.
- Thorough and accurate knowledge of the sewage network can also be provided from previous experiences. Good knowledge of the influent composition is essential. Data concerning what is coming into the plant is crucial. Possible toxic and peak load events can then be tracked down and their strong and uncontrollable effects on activated sludge filterability can be limited. It can first be done by collecting data concerning the influent composition and the sewage network, like the type of industries connected and the composition of their discharges.
- As presented in the previous section, a careful biological design resulting in good flocculation properties would also be essential to ensure steady operations.
- SVI analyses and total nitrogen analyses present in the effluent can also be considered useful indicators of operational problems or toxicity. Nitrifying organisms are very sensitive to a wide range of organic and inorganic compounds and can therefore be easily inhibited (Metcalf&Eddy, 2003). For instance, the unexplained loss of the nitrification in a plant history can therefore be seen as an indicator of potential toxicity and be used to predict potential unstable operations.

7.3 Final remarks

A measurement campaign in several European pilot and full-scale MBRs was organised during this research work in order to ascertain the relevancy of different parameters on MBR fouling and MBR membrane performances. Due to the implementation of an identical measuring protocol during the whole measurement campaign, i.e. the DFCm, local observations could result in general conclusions. This intensive filterability study hopefully helped to get a step closer to optimum MBR design and operations, especially concerning the biological part.

However, even if general conclusions could be formulated, the MBR issues are far from being solved. The lack of data (truly depending on the plant facilities) and the difficulties to collect accurate and relevant information or comparable data between plants remain a major issue. On line sensors, for MLSS or influent characterisation for instance, could solve a large part of this problem.

Several research targets can be formulated out of this thesis work. The improvement of filterability observed in the membrane tank during the upconcentration process should be further investigated. A better understanding of the involved mechanisms could allow significant energy savings.

Furthermore, as it was observed during this research work, steady state conditions are crucial in order to obtain efficient membrane performances. Critical control parameters of the biological process need to be clearly identified and a better characterisation of the incoming flow of the MBR plants should be implemented in order to understand further the interactions between the biology and the membrane stage.

The DFCm finally proves to be an efficient tool to optimise MBR plant operations, especially under unsteady state conditions. However, this analysis can only be fully relevant if combined with an adequate set of analyses.

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Samenvatting

Dankzij het toepassen van een membraanfiltratiestap kan met MBR technologie een superieure effluentkwaliteit en ruimtebesparing bereikt worden ten opzichte van het conventionele actiefslib proces. Hoewel door de ontwikkeling van ondergedompelde membraansystemen de operationele kosten van het proces aanzienlijk teruggebracht konden worden, blijven de kosten die gerelateerd zijn aan het voorkomen van membraanvervuiling (fouling) een belemmering voor de marktontwikkeling van MBR technologie.

De belangrijkste oorzaken van fouling moeten nog steeds onderzocht en gekwantificeerd worden om efficiënte tegenmaatregelen en protocollen ter voorkoming van fouling te kunnen ontwikkelen. Het meten van de filtreerbaarheid kan hierbij een significante bijdrage leveren. Het meten van de filtreerbaarheid kan gebruikt worden om na te gaan of een daling van de permeabiliteit te wijten is aan een slechte filtreerbaarheid van het actiefslib of aan slechte bedrijfsvoering van het filtratieproces. Hierdoor kan een beter begrip verkregen worden van de mechanismen die een rol spelen in het filtratieproces. Echter, het gebrek aan gestandaardiseerde methoden voor het karakteriseren van filtreerbaarheid blijft een beperking. Een grote hoeveelheid methoden is ontwikkeld door verschillende onderzoeksgroepen, wat betrouwbare onderlinge vergelijking bemoeilijkt. Als gevolg hiervan is het niet mogelijk om algemeen geldende conclusies te formuleren.

In verband met twee omvangrijke Europese onderzoeksprojecten, EUROMBRA en MBR-Train, heeft de Technische Universiteit Delft besloten tot het organiseren van een grootschalige meetcampagne op Europese schaal. Pilot-schaal en praktijkschaal MBR installaties van diverse partners (bedrijven, onderzoeksinstituten en universiteiten) zijn onderzocht met behulp van een gestandaardiseerde filtratietest, de Delft Filtration Characterisation method (DFCm). Gegevens betreffende de filtreerbaarheid van het actiefslib in de pilot- en praktijkinstallaties zijn verzameld onder identieke hydraulische omstandigheden. Op deze wijze kunnen resultaten van verschillende onderzoeksgroepen op een relevante en accurate wijze met elkaar vergeleken worden. Daarnaast is een set actiefslib analyses uitgevoerd in combinatie met de filtreerbaarheidsmetingen. Ook zijn gegevens betreffende het ontwerp, de bedrijfsvoering en het presteren van de membranen verzameld voor alle MBR installaties.

De DFCm heeft zich in de praktijk bewezen als *gebruiksvriendelijke, snelle* en *nauwkeurige* methode voor het karakteriseren van de filtreerbaarheid van actiefslib. De resultaten die verzameld zijn gedurende de meetcampagne zijn consistent met de bedrijfsvoering van de betreffende installaties en *betrouwbaar* en *reproduceerbaar*. In vergelijking met andere karakteriseringsmethoden zijn de voordelen van de DFCm het scherp gedefinieerde en goed *beheersbare* protocol en de korte duur van de experimenten, waardoor de dynamiek van de filtreerbaarheid in MBR installaties ondervangen kan worden.

Als gevolg van de uniciteit van elke MBR installatie zijn significante onderlinge verschillen wat betreft de filtreerbaarheid aangetoond. In het onderzoek zijn grote fluctuaties in de filtreerbaarheid geobserveerd en gedeeltelijk verklaard door verschillende factoren. Deze factoren kunnen gerangschikt worden op basis van de mate waarin ze de filtreerbaarheid beïnvloeden.

Ten eerste kan de invloed van de oncontroleerbare samenstelling en temperatuur van het afvalwater genoemd worden:

- De influentkwaliteit is waarschijnlijk de dominante factor die de filtreerbaarheid van het actiefslib beïnvloedt. Moeilijk afvalwater, stress omstandigheden voor het actiefslib en schuimvorming hebben een sterke invloed op de filtreerbaarheid.
- Temperatuurverschillen van het afvalwater door seizoenen hebben ook een significante invloed op de filtreerbaarheid. Een statistisch verband is aangetoond tussen filtreerbaarheid en de temperatuur in MBR installaties op praktijkschaal. De belangrijkste oorzaak van de verslechtering van de filtreerbaarheid tijdens de winter is het vrijkomen van colloïdale deeltjes als gevolg van de ongunstige omstandigheden voor flocculatie bij lage temperaturen. Op dezelfde manier is de belangrijkste reden voor de verbetering van de filtreerbaarheid tijdens de zomerperiode de afname van het aantal colloïdale deeltjes, doordat ze in hogere mate in de slibvlok worden opgenomen.

De bedrijfsvoering van het proces en MBR ontwerpparameters kunnen ook beschouwd worden als factoren die de filtreerbaarheid beïnvloeden:

- Bij een laag recirculatiedebiet treedt indikking van het actiefslib in de membraantank op. Deze indikking resulteerde in een aanzienlijke verbetering van de filtreerbaarheid.
- Laagbelaste systemen vertonen een betere filtreerbaarheid dan hoogbelaste systemen. Hierbij moet wel rekening gehouden worden met het feit dat de belasting van het systeem een kleiner effect heeft op de filtreerbaarheid dan de temperatuur of toxiciteit van het afvalwater.

Enkele andere parameters vertoonden geen significante correlatie met de actiefslib filtreerbaarheid:

SMP vertoont geen verband met de filtreerbaarheid. Dit is waarschijnlijk te wijten aan de methode (spectrofotometer) die gekozen is om de SMP concentratie te bepalen. Deze conventionele methode moet niet meer als geschikt beschouwd worden met betrekking tot MBR fouling onderzoek. Nieuwe methoden die georiënteerd zijn op het karakteriseren van specifieke eigenschappen zoals de deeltjesgrootte moeten geïmplementeerd worden om na te kunnen gaan in welke mate SMP het fouling proces beïnvloedt. De viscositeit van het actiefslib vertoont geen significante relatie met de filtreerbaarheid. Filtreerbaarheid en permeabiliteit van de beschouwde MBR installaties werden niet beïnvloed door fluctuaties in de filtreerbaarheid. De viscositeit kan niet beschouwd worden als een te optimaliseren parameter met betrekking tot de bedrijfsvoering van de huidige MBR installaties op praktijkschaal. Aan de andere kant vormt de viscositeit wel een belangrijke factor met betrekking tot clogging (slibophoping in de membraanmodule).

De membraanconfiguratie van ondergedompelde MBR systemen vertoont geen relatie met de filtreerbaarheid. De filtreerbaarheid wordt eerder beïnvloed door het biologische proces dan door het filtratieproces. De verschillen in schuifspanningen en het hydraulische regime als gevolg van verschillende modules zijn niet voldoende om een significant effect te hebben.

Ten slotte zijn interessante resultaten vastgesteld met betrekking tot het opschalen van MBR technologie. Aanzienlijke verschillen zijn geconstateerd tussen pilot-schaal en praktijkschaal MBR installaties. De verschillen in (buffer)capaciteiten, bedrijfsvoering, stressomstandigheden en stabiliteit van het proces leiden tot verschillen in filtreerbaarheid.

Een algemeen kader gebaseerd op de resultaten van de meetcampagne is opgesteld. Dit kader toont aan dat het actiefslib uit een MBR installatie niet significant verschilt van actiefslib uit een conventioneel actiefslib systeem.

Een goed functionerend flocculatieproces is essentieel voor efficiënte bedrijfsvoering van MBR installaties. Hier moet rekening mee gehouden worden bij het ontwerp van MBR systemen. Met betrekking tot het beheersen van fouling zijn de huidige systemen gebaseerd op toevoeging van chemicaliën ter bevordering van de flocculatie en het bevorderen van de turbulentie bij het membraan. Beide technieken lijken niet haalbaar tegen aanvaardbare kosten. Daarom moeten nieuwe protocollen getest worden die gebaseerd zijn op de waarneming dat de filtreerbaarheid verbeterd kan worden door het actiefslib in de membraantank in te dikken.

In dit proefschrift worden aanbevelingen geformuleerd met betrekking tot het gebruik van lab- en pilot-schaal experimenten en het ontwerp van MBR installaties op praktijkschaal. Membraankosten blijven een belangrijk knelpunt wat betreft de marktontwikkeling van het MBR proces. Om competitief te worden moeten de membraankosten omlaag of de toepasbare flux aanzienlijk verhoogd worden. Daarom is meer onderzoek nodig naar membraanmateriaal om goedkoop en op grote schaal membranen te kunnen produceren.

MBR onderzoek op pilot-schaal moet heroverwogen worden. Aanzienlijke verschillen in filtreerbaarheid tussen pilot- en praktijkschaal MBR installaties zijn aangetoond. Stabiele bedrijfsvoering op pilot-schaal wordt vooral bemoeilijkt door verschillen in omgevingsfactoren en een gebrek aan redundantie. Om deze reden zijn haalbaarheidsstudies op pilot-schaal voor de bouw van een full-scale installatie niet noodzakelijk meer. Wel kunnen pilot-studies nuttig zijn met betrekking tot het optimaliseren van de bedrijfsvoering van het proces. In plaats van het bouwen van een volledige pilot-schaal installatie met zijn eigen specifieke biologie, is het waarschijnlijker dat een aparte parallel geschakelde membraantank die gekoppeld is aan de bestaande fullscale installatie betere informatie oplevert over hoe het MBR-proces kosteneffectief bedreven kan worden.

Ten slotte kan gesteld worden dat het geïnstalleerde membraanoppervlak optimaal gebruikt kan worden als het ontwerp van de MBR is gebaseerd op de droogweerafvoer of als de MBR is aangesloten op een gescheiden rioolstelsel. Op deze manier kan het potentieel van het MBR proces optimaal benut worden voor het retrofitten van bestaande afvalwaterzuiveringsinstallaties.

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Curriculum Vitae



Adrien Moreau holds a Master of Science degree in Process and Chemical Engineering (2005) from the National Institute of Applied Science (INSA Toulouse, France). He was then a Ph.D. student at Delft University of Technology (the Netherlands) in the framework of a European Union Marie Curie research training fellowship. He was part of the research project MBR-TRAIN which deals with process optimisation and fouling control in membrane bioreactors for water treatment. His research focused on MBR activated sludge characterisation full-scale performance and MBR optimisations. He is now working as a research engineer for the research center for water of Veolia in Paris (France).

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