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# Impact of inflow conditions on activated sludge filterability and membrane bioreactor (MBR) operational performance

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#### Abstract

Operation and performance of MBRs for wastewater treatment is influenced by changes in activated sludge properties. Therefore, understanding of the factors influencing sludge filterability is of major importance for efficient operation of MBR installations. This paper assesses the impact of the influent characteristics on activated sludge filterability and treatment performance of full-scale MBRs. The full-scale MBRs were closely monitored both in summer and winter. The measurement campaign, consisting of activated sludge filterability tests and physicochemical analyses, is supplemented with operational performance data evaluation of the various full-scale plants. Based on these data, it is shown that an undesired and refractory composition of incoming wastewater, hydraulic and/or organic load shocks, as well as abrupt temperature changes of the influent, lead to operational problems and affect sludge filterability.

#### Keywords

Membrane bioreactor (MBR); full-scale operation; activated sludge filterability; inflow characteristic; loading rate;

#### List of abbreviations and symbols

α <sub>R</sub> ·C <sub>i</sub>	product of the specific cake layer resistance
α <sub>R</sub>	specific cake resistance at reference filtration resistance [m/kg],
Ci	solids concentration involved in the fouling process [kg/m3]
$\Delta R_{20}$	added resistance after filtration of 20 L/m <sup>2</sup> in the DFCm [m <sup>-1</sup> ]
CAS	conventional activated sludge
COD	chemical oxygen demand [mgO <sub>2</sub> /L]
DFCm	Delft Filtration Characterization method
DO	dissolved oxygen [mgO <sub>2</sub> /L]
DSVI	diluted sludge volume index [mL/gTSS]
EPS	extracellular polymeric substance [mg/L]
MBR	membrane bioreactor
MLSS	mixed liquor suspended solids [g/L]
MLVSS	mixed liquor volatile suspended solids [g/L]
MT	membrane tank
NaCl	sodium chloride
SMP	soluble microbial products [mg/L]
TDS	total dissolved salts [g/L]
TMP	transmembrane pressure [bar]
WWTP	waste water treatment plant
XRD	X-Ray powder Diffraction
XRF	semi-quantitative X-ray Fluorescence

# 1. INTRODUCTION

Filterability of activated sludge is the connecting parameter between membrane bioreactor 'biology' and membrane operation. The changes in activated sludge properties will undoubtedly result in changes in MBR operation and performance. Therefore, understanding of the factors influencing sludge filterability is of major importance for efficient operation of the MBR installations.

In previous work [1], seasonal changes in the composition of raw domestic wastewater and their impact on activated sludge filterability were discussed. However, the characteristics of raw wastewater not only vary between the seasons, but they are also subjected to continuous, e.g., hourly and/or daily, fluctuations. In consequence, feed water quality, and resulting activated sludge filterability [2], will change continuously [3]. In addition, organic loading plays an important role in membrane fouling and sludge filterability [4-7]. Evenblij et al. [8] also observed variations in the filterability as a result of sudden changes in the influent characteristics. Moreover, a continuous deterioration of the sludge filterability with increasing addition of a carbon source was reported. Furthermore, according to Geilvoet [9], continuous variation in the flow rate, temperature and/or composition of influent flow may lead to stress conditions, also impacting activated sludge quality. Consequently, non-stabilised operation and fluctuating process conditions are causing deterioration of activated sludge filterability.

# **1.1. Hydraulic loading rate effect**

Variations in feed water flow rate, and thus changes in hydraulic load, can cause nonstabilised operation of the MBR [10]. As distinct from industrial installations, municipal MBRs are exposed to storm flows that may drastically change the hydraulic loading and decrease pollutant concentrations. Short hydraulic shock periods are usually compensated by operation at higher fluxes, e.g. up to double the design flux [10]. However, those periods of increased flux should not exceed 1-2 hours and often need to be followed by a relaxation period. Therefore, long or prolonged periods with high influent flow rate might be problematic for MBR operation due to the potential increment of membrane fouling. Yet, the influence of hydraulic loading was hardly discussed in literature [11].

In order to assess the impact of hydraulic shocks on activated sludge filterability, development of the filterability during periods of elevated flow rates was investigated. The MBR Varsseveld was designed for a treatment capacity of 250-300 m<sup>3</sup>/h during dry weather flow and to treat a rain weather flow up to 755 m<sup>3</sup>/h. MBR Varsseveld is connected to a combined sewer system and has a stand-alone configuration. Therefore, incoming storm water is treated exclusively by the MBR. This provided good conditions for the hydraulic load effect investigation.

# **1.2. Temperature effect**

As has been previously stated [1, 12], temperature has a significant influence on activated sludge filterability and wastewater composition, causing seasonal sludge filterability fluctuations. Temperature, and rapid temperature changes in particular, likely demand changes in MBR operation. However, thus far, it has attracted little research attention [13]. Therefore, the impact of temperature changes on activated sludge filterability and operation of full-scale MBRs was evaluated.

## **1.3. Influent composition effect**

It was hypothesized that road salt, generally applied during the European winter months, could have a detrimental effect on activated sludge filterability and thereby aggravating or masking temperature effects. In literature, contradicting results are presented with respect to the impact of salt concentrations on flocculation. While some researchers observed a

negative impact [14, 15], others found no effect [16] and some reported positive influence of salt concentration, most likely due to compression of the electric double layer surrounding particles, enhancing flocculation [17]. Furthermore, as pointed out by Yang et al. [18], Na<sup>+</sup> may act as a fouling promoter but also as a cleaning agent. Filtration deterioration could be ascribed to ion exchange with the floc sustaining matrix, i.e. monovalent cations replacing bivalent cations, causing the microbial flocs to disrupt [8]. It also has been reported in literature, that high concentrations of salts can affect membrane performance [19, 20], increase fouling propensity [20, 21], disturb biotreatment processes [15, 22] and consequently reduce water quality [19, 20, 23, 24]. However, the impact of the typical winter phenomenon 'road salt' on activated sludge characteristics and on the filterability in particular, has been largely overlooked in the past. Therefore, the potential impact of road salt on activated sludge filterability was further investigated.

Furthermore, the differences and the influence of municipal wastewaters on sludge filterability in full-scale MBRs were investigated in order to find a plausible relation between filterability of the activated sludge and the inflow characteristics with respect to organic loading.

In our present work, we aimed to assess the impact of the influent characteristics on activated sludge filterability and operational performance of full-scale MBRs. The effect of influent flow rate, temperature and composition on the activated sludge filterability is discussed in this paper.

## 2. MATERIALS AND METHODS

## 2.1. Experiments description

To assess the impact of the influent flow rate, temperature and influent composition on activated sludge filterability and operation, full-scale MBRs treating municipal wastewater, namely MBR Varsseveld, MBR Ootmarsum and MBR Heenvliet, were investigated. A detailed description of the investigated plants is presented in Table 1. The experiments were carried out during summer and winter periods. During those measurement periods, activated sludge filterability tests and physicochemical analyses were carried out. The filterability of the activated sludge was monitored in different compartments of the MBR, e.g. membrane tank, aerobic, anaerobic and anoxic.

Location	Unit	Varsseveld	Heenvliet	Ootmarsum
WWTP configuration	-	MBR (stand-alone)	CAS+MBR (parallel and serial)	CAS+MBR (parallel)
Membrane configuration	-	Submerged	Submerged	Sidestream
Membrane type	-	Hollow fibre (HF)	Flat sheet (FS)	Tubular (MT)
Membrane supplier	-	Zenon-GE	Toray	Norit
Total membrane area	m²	20,160	4,115	2,436
Membrane pore size	μm	0.035	0.08	0.038
Biological capacity	p.e.	23,150	3,333	7,000
Hydraulic capacity (DWF)	m <sup>3</sup> .h <sup>-1</sup>	250-300	38-50	75
Hydraulic capacity (RWF)	m <sup>3</sup> .h <sup>-1</sup>	755	100	150
Average Flux (DWF)	l.m <sup>-2</sup> .h <sup>-1</sup>	15-25	12-24	26-40
SRT	days	24-26	31-40	40-42
HRT	hours	4-14	20 (serial) 28 (parallel)	-
MLSS	g.L <sup>-1</sup>	6-10	8-13	10-12
F/M	gBOD/gMLVSS*d	0.03-0.04	0.027-0.045	0.05

Legend: DWF - dry weather flow; RWF - rain weather flow; P.E. – person equivalent;

The mixed liquor suspended solids (MLSS) and mixed liquor volatile suspended solids (MLVSS) measurements were conducted in accordance with the procedures described in

APHA Standard Methods [25]. The diluted sludge volume index (DSVI) test was carried out in accordance with the protocol given by Koopman and Cadee [26] and Jenkins et al. [27]. In order to assess the impact of influent characteristics with regard to organic loading on sludge filterability, MBRs listed in Table 1 were studied. To this end, sludge filterability, MLSS, MLVSS, temperature, COD concentration, COD load and specific COD loading were analysed.

## 2.2. The Delft Filtration Characterization method (DFCm)

The Delft Filtration Characterization method (DFC<sub>m</sub>) was used to study the activated sludge filterability. The DFCm is a small-scale filtration characterization installation operated on the basis of a standardised measuring protocol allowing characterisation of different samples of activated sludge under the same conditions. The cross flow velocity is 1 m/s and the applied flux is 80 L/m<sup>2</sup>.h. The installation, accompanying protocol and example of research results were described elsewhere [3, 28-30]. For easy comparison between different tests, the value  $\Delta R_{20}$  is used (Table 2) based on the classification proposed by Geilvoet [9]. This value is defined as the increase in resistance after a specific permeate production of 20 L/m<sup>2</sup>.

ΔR <sub>20</sub> [10 <sup>12</sup> m <sup>-1</sup> ]	Classification
0 - 0.1	Good
0.1 - 1.0	Moderate
> 1.0	Poor

**Table 2**: Classification of activated sludge filterability based on  $\Delta R_{20}$ 

Besides the  $\Delta R_{20}$  value, more detailed information about the membrane cake layer can be extracted from the DFCm outcome after detailed data analysis is performed. According to Geilvoet [9], when the cake layer filtration theory is fitted to the DFCm output the cake layer can be further characterized by the product of the product of the specific reference cake layer resistance and the concentration of solids accumulating in the cake layer, i.e.,  $\alpha_{R} \cdot c_i$ . The  $\alpha_{R} \cdot c_i$  was calculated from:

$$(\alpha_R \cdot c_i) = a^{1/b} = a^{1-s} \tag{1}$$

Where  $\alpha_R$  is specific cake resistance at reference filtration resistance [m/kg],  $c_i$  is solids concentration involved in the fouling process [kg/m<sup>3</sup>], s is compressibility coefficient [-] and a and b are coefficients available in the DFCm output.

### 2.3. Data collection and processing

Parallel to the filterability tests, plant operations and performances were monitored, analysed and linked with influent characteristics and sludge filterability. For this purpose, both the plant operational data and membrane performance data, such as influent flow, permeate flow, flux, TMP, permeability, temperature, pH, MLSS concentration and DO concentration, were collected for the respective periods. Hereafter, the impact of influent characteristics on sludge filterability and operation of full-scale MBRs was evaluated.

## 2.4. Road salt effect

For a salinity influence study, an activated sludge sample of 30 L was taken directly from the membrane tank of the nearby MBR Heenvliet. After immediate transportation, samples were placed in a continuously aerated DFCm container. The DFCm was used to measure the activated sludge filterability. Prior to the filterability measurement, a sludge sample was taken to perform further sludge analyses, i.e., MLSS, MLVSS and DSVI.

The road salt sample used during experiments was exactly the same salt used to de-ice roads and was provided by courtesy of the Rijkswaterstaat, an agency responsible for maintenance of the Dutch roads. The exact road salt composition was determined by X-Ray

powder Diffraction (XRD) and semi-quantitative X-ray Fluorescence (XRF) analysis. As expected, sodium chloride was the main constituent of the used road salt. (see supplementary data).

After performing a reference filterability test without addition of road salt, 10 g of salt was diluted in hot tap water and added to the activated sludge sample. Typically, raw sewage has a salinity of about 0.01-0.11% [31-33], sewage following road gritting has a salt content of about 0.2% [34] and the waters with a salt concentration above 1% are considered to have high salt concentration [19]. In this research, road salt was added to create the following salt concentrations in activated sludge samples: 0.03%, 0.10%, 0.16%, 0.48% and 1.27%. Corresponding salt quantities were diluted in hot tap water. The cumulative volume of tap water used to dissolve the road salt was less than 0.7% of the total sludge volume. After initial manual mixing to distribute salt in the sludge sample, continuous aeration keeps the sample well-mixed during the course of the experiment. The conductivity was measured with the multi-meter (WTW 340i) equipped with a conductivity measuring cell (WTW TetraCon 352).

## 3. RESULTS & DISCUSSION

### 3.1. Hydraulic loading rate effect

The first period of detailed analysis has taken place during the 'winter 2009' campaign experiments in MBR Varsseveld. Due to heavy storm and intensive rainfall the influent flow had increased more than 5 times from a daily average of 130 m<sup>3</sup>/h to nearly 700 m<sup>3</sup>/h within an hour and reached maximum capacity within 2.5 hours. During the whole measurement period, the average  $\Delta R_{20}$  values of 3.7·10<sup>12</sup> m<sup>-1</sup>, 3.4·10<sup>12</sup> m<sup>-1</sup>, 3.1·10<sup>12</sup> m<sup>-1</sup> and 3.5·10<sup>12</sup> m<sup>-1</sup> were measured for samples originating from the membrane tank #1, #2, #3, and #4 respectively. The average daily filterability results of the sludge samples originating from all of the membrane tanks are plotted on Figure 1a. In addition, a single measurement of the sludge sample from the membrane tank #1 (MT1), the only one corresponding to the highest peak flow is plotted. Before the hydraulic shock prevailed, the activated sludge originating from the membrane tank was considered to have poor filterability, characterised by a  $\Delta R_{20}$  of 3.5  $\cdot$  10<sup>12</sup> m<sup>-1</sup>. Although the standard deviation for the measurements of samples with  $\Delta R_{20}$ above  $1 \cdot 10^{12}$  m<sup>-1</sup> is up to  $\pm 0.3 \cdot 10^{12}$  m<sup>-1</sup>, there is a variation of  $\Delta R_{20}$  that can be related to changes in the hydraulic loading rate. At first, filterability deteriorates during the peak flow to improve after the first peak has passed (Figure 1a). Although only one measurement was made during the highest peak flow conditions, whereas the other measurements were all made afterwards, however, considering the dynamic nature of the mixed liquor [35] this filterability change is possible. The filterability improvement observed after the first peak might be attributed to the dilution effect caused by heavy rainfall. The subsequent filterability changes are rather just variations around average sludge filterability due to the uncertainty associated to the equipment measure. The measured changes in filterability might be also caused by the hydraulic load change but also by melting snow and concomitant temperature drop of the influent. The temperature and snow melt aspect is discussed further in the 'Temperature effect' section. Nevertheless, a similar pattern of filterability changes during the period of increased hydraulic loading was observed for samples originating from the aerobic, anoxic and other membrane tanks (Figure 1b). We may conclude that distinct changes in hydraulic loading have a potential to impact activated sludge filterability. Our findings are in accordance with Trussell et al. [36] and Trussell [37] who found that deflocculation of sludge during rain weather flows caused an increase in colloidal content in the mixed liquor, resulting in severe membrane fouling. The change in the hydraulic loading rate resulted in operational problems that will be discussed as well in the below section.

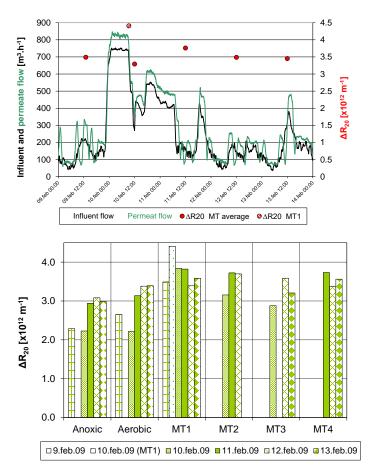


Figure 1: (a) Influent and permeate flows as well as average daily ΔR<sub>20</sub> values for membrane tank samples versus operational time and (b) corresponding activated sludge filterability development in the various compartments of the MBR Varsseveld during different days of the 'winter 2009' campaign. On Figure 1a, a single measurement of the sludge sample from the membrane tank #1 (MT1), the only one corresponding to the actual peak flow is plotted.

Another intensive rainfall period, allowing studying the effect of hydraulic load, was analysed in the course of the 'winter 2010' experimental campaign in MBR Varsseveld. During this rainfall period, influent flow had increased more than 3 times from 150 m<sup>3</sup>/h and reached instantaneously, almost the maximum hydraulic capacity, i.e., 700 m<sup>3</sup>/h, within an hour. The average flow rate during the entire period of hydraulic peak was about 500 m<sup>3</sup>/h. The first filterability measurements were performed during the treatment of a storm flow. Therefore, the quality of activated sludge before the period of excessive hydraulic load could not be assessed. Nevertheless, filterability development showed a clear tendency. During the whole research period, the average  $\Delta R_{20}$  values of  $0.74 \cdot 10^{12} \text{ m}^{-1}$ ,  $0.75 \cdot 10^{12} \text{ m}^{-1}$ ,  $0.76 \cdot 10^{12} \text{ m}^{-1}$  and  $0.76 \cdot 10^{12} \text{ m}^{-1}$  were measured in the one to four membrane tanks, respectively. The average daily filterability results of the sludge samples originating from all of the membrane tanks are plotted on Figure 2a. The worst, yet classified as moderate, sludge filterability was measured during the period of increased flow rate. After the hydraulic peak had passed, an improvement in filterability was observed. At first, the filterability improved slowly, whereas in the next 3 days it improved more rapidly, reaching a  $\Delta R_{20}$  of 0.58  $\cdot 10^{12}$  m<sup>-1</sup> (Figure 2a). A similar filterability development pattern was observed in all compartments of the MBR (Figure 2b). It is of course possible that the filterability simply improved despite the storm flow and is not related to the peak flow event. Nevertheless, also in this case, the impact of hydraulic loading on activated sludge filterability was visible. However in this case, the positive influence of a decreasing hydraulic loading rate on sludge filterability was clearly observed.

The operation of the MBR was not affected seriously during this rainfall event. The fluxes increased from average 24 L/m<sup>2</sup>·h up to 44 L/m<sup>2</sup>·h to compensate higher influent flow rates. Permeability had improved from 120-140 L/m<sup>2</sup>·h·bar to 160-210 L/m<sup>2</sup>·h·bar between the periods of elevated and regular hydraulic loads, respectively. Further permeability improvement to values of 180-230 L/m<sup>2</sup>·h·bar was achieved after maintenance cleaning was performed.

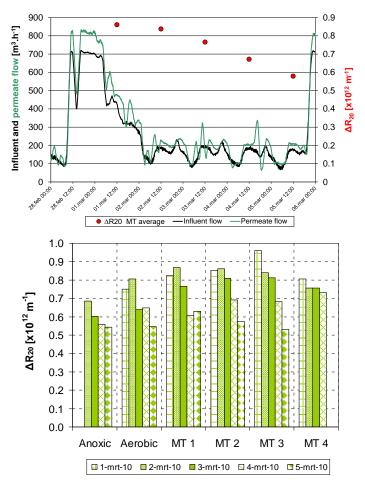
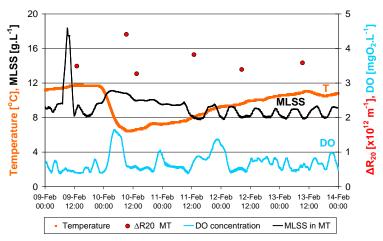


Figure 2: (a) Influent and permeate flows as well as average daily ΔR<sub>20</sub> values for membrane tank samples versus operational time and (b) corresponding activated sludge filterability development in the various compartments of the MBR Varsseveld during different days of the 'winter 2010' campaign

#### 3.2. Temperature effect

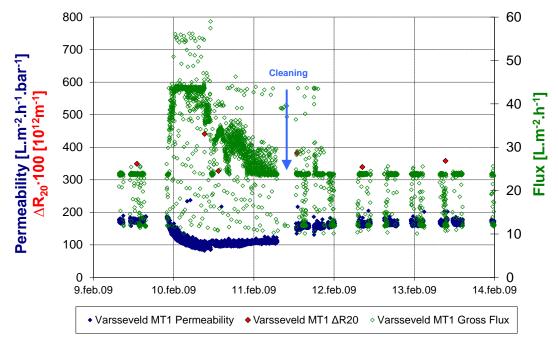
The negative impact of quickly decreasing temperature was observed during the 'winter 2009' campaign at the MBR Varsseveld. During the night between 9<sup>th</sup> and 10<sup>th</sup> of February, a rapid temperature drop of 7°C within 7 hours had occurred, seriously affecting the activated sludge filterability (Figure 3). The immediate filterability improvement, probably caused by the dilution effect associated with the heavy rainfall, was followed by filterability deterioration. Afterwards, recovery of the sludge filterability can be further observed.



**Figure 3**: Temperature, MLSS, dissolved oxygen profiles and  $\Delta R_{20}$  values measured at MBR Varsseveld during 'winter 2009' campaign

This abnormal situation was caused by the heavy storm event that happened after a prolonged dry and cold period with snow cover. Consequently, melting snow was transported to the plant together with fresh and cold rainwater. As a result, the temperature of the incoming wastewater had dropped from about 12°C to 6°C and the hydraulic load rapidly increased from an average 160 m<sup>3</sup>/h to the maximum capacity of 750 m<sup>3</sup>/h and lasted about 12 hours (Figure 3). Furthermore, it is possible that a large amount of salt was present in the influent. The road salt, i.e., sodium chloride, used to de-ice roads at that period was dissolved in melt water and was also transported to the plant. The impact of road salt on activated sludge filterability was investigated and is discussed in the following 'Salinity' section. Thus, when the cold and possibly salty inflow reached the treatment plant bioreactor, the microorganism population, their activity, and morphology of the sludge flocs were likely to be affected. In effect, activated sludge filterability deteriorated in each section and the permeability achieved at the plant was approximately 50% lower than under stabilised operation conditions (Figure 4). The actual permeability decreased from about 200-230 L/m<sup>2</sup>·h·bar under undisturbed operation circumstances to values below 100 L/m<sup>2</sup>·h·bar during the storm event. At that time, the applied flux had increased from typical 20±6 L/m<sup>2</sup>·h to about 35±8 L/m<sup>2</sup>·h. Moreover, at high flows, the MLSS concentration in the membrane tanks in MBR Varsseveld is higher than at dry weather flow (Figure 3), due to the lower ratio of permeate and recirculation flows. According to critical MLSS concentration theory proposed by Lousada-Ferreira [38], the observed extreme values also might have contributed to the membrane permeability drop at the heavy storm event.

However it should be pointed out that, permeability is considered a weak parameter to monitor MBR performances and to determine the cause of poor filtration process. In our case, according to Figure 4, the  $\Delta R_{20}$  value does not seem to correlate with membrane permeability. This is in accordance to the statements of other researchers [2, 9, 28, 29, 39].



**Figure 4**: Varsseveld MBR operation monitoring – flux, permeability and filterability – during winter 2009. The graphs illustrate process permeability plotted on y-axis (in blue), sludge filterability expressed as, and for better visualization multiplied by a factor 100,  $\Delta R_{20}$  parameter on y-axis (in red), gross flux on second y-axis (in green) and time on x-axis.

Following the operator's logbook, the weekend before the temperature drop, an increase in MLSS concentration was observed at the plant. The MLSS concentration in the aerobic tank increased from the regular 8.0 g/L on 5th of February up to 13.0 g/L on 8th of February (results not illustrated graphically). Subsequently, concentrations of up to 19 g/L were measured in the membrane tanks (Figure 3). This concentration increase is attributable to changing incoming influent characteristic. In the neighbourhood of the WWTP, the ditches alongside roadways were cleaned and accumulated materials from the ditches were removed to provide proper patency and drainage capacity. Subsequently, this mainly organic material was discharged into the sewerage, feeding the plant with more concentrated wastewater, increasing MLSS in the MBR and probably caused an organic overloading condition for the activated sludge microbial community. The high activated sludge loadings could have negatively affected membrane performance [6]. Moreover, an improvement in sludge quality/filterability under low organic loadings was reported by others [2, 5]. The positive impact of reduced loading on filterability was more visible in summer than in winter [2]. On the other hand, Zwickenpflug et al. [40] did not observe significant effects of organic loading changes on filtration performance compared to seasonal fluctuations. Apparently, seasonal temperature changes or unexpected events have stronger influence on filterability than the sludge loading. Therefore, as previously stated by Moreau [2], activated sludge loading likely affects sludge filterability but should not be considered a predominant parameter with respect to filterability.

In addition, at that time the oxygen concentration in the aerobic tank was low reaching values of about 0.4 mg/L (Figure 3). Prolonged low DO concentrations are reported to be detrimental for activated sludge filterability due to deflocculation processes leading to excessive growth of filamentous bacteria and release of fine particles [41, 42]. The low DO concentration was most likely a consequence of increased oxygen uptake by the aerobic bacteria consuming available and excessive organic material. During the rest of the investigated period, oxygen

concentration was in the range of 0.5-1.0 mg/L, while in the comparable period in the previous year the concentration was in the range of 1.0-1.5 mg/L.

Therefore, the combination of unpredictable and uncontrollable circumstances, e.g. heavy rain fall, quick snows melt and organic load increase, likely affected activated sludge filterability and caused serious operational problem for the MBR. In contrast, during the 'winter 2010' period, aforementioned circumstances did not occur at the same time and MBR operation was not disturbed.

#### 3.3. Influent composition effect

### 3.3.1. Salinity

From both XRD (Figure A.1) and XRF (Table A.1) analyses it could be concluded that the road salt sample is rather pure with 98 weight% sodium chloride (NaCl). Apart from potassium, calcium and magnesium, together 1.8 weight% of total mixture, no other compounds in significant quantities were detected.

The initial conditions of activated sludge sample prior to sodium chloride injection are presented in Table 3.

Temperature	рΗ	MLSS	MLVSS	DSVI	$\Delta R_{20}$
°C	-	g/L	g/L	mL/g	m⁻¹
18.9	7.7	9.1	6.7	132	0.79
17.1	8.5	9.4	7.2	117	1.02
17.8	7.7	8.3	6.3	115	1.37
18.6	7.8	10.1	7.6	114	1.39
	°C 18.9 17.1 17.8	°C - 18.9 7.7 17.1 8.5 17.8 7.7	°C         -         g/L           18.9         7.7         9.1           17.1         8.5         9.4           17.8         7.7         8.3	°C         -         g/L         g/L           18.9         7.7         9.1         6.7           17.1         8.5         9.4         7.2           17.8         7.7         8.3         6.3	°C         -         g/L         g/L         mL/g           18.9         7.7         9.1         6.7         132           17.1         8.5         9.4         7.2         117           17.8         7.7         8.3         6.3         115

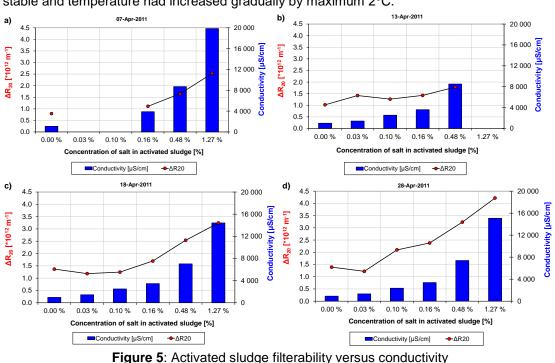
Table 3: Characteristics of activated sludge samples prior to the salinity shock test

The conductivity and total dissolved salts (TDS) results together with corresponding salt concentrations are presented in Table 4.

Salt addition	Salt concentration		Electric conductivity		Total dissolved salts		
g	mg <sub>NaCL</sub> /L	%	μS/cm		g/L		
			Average	Standard deviation	Average	Standard deviation	
0	0	0.00	1,001	72	0.6	0.05	
10	300	0.03	1,402	63	0.9	0.04	
30	1,000	0.10	2,473	96	1.6	0.06	
50	1,700	0.16	3,588	222	2.3	0.1	
150	5,000	0.48	7,917	827	5.1	0.5	
400	13,300	1.27	16,455	2,965	10.5	1.9	

 Table 4: Conductivity and total dissolved salts results of the salt injection experiments

During all experiments, filterability deteriorated when the salt was added (Figure 5). The deterioration of activated sludge filterability was directly linked to the increase in salinity, expressed as conductivity, in the sludge sample. The measured conductivity increased from approximately 1,000  $\mu$ S/cm for the samples without the road salt to about 16,500  $\mu$ S/cm for the samples with the highest salt concentrations. The TDS concentrations were in the range



of 0.6±0.05 g/L and 10.5±1.9 g/L for the samples without added salt and the sample with the highest salt concentrations, respectively. During the course of experiments the pH remained stable and temperature had increased gradually by maximum 2°C.

The results of these experiments clearly demonstrate that addition of the sodium chloride to activated sludge results in high  $\Delta R_{20}$  values, and as such indicating worsening of activated sludge filterability (Figure 6). It could be of interest, to establish on-line conductivity measurement of the influent as an indicator for changes in influent and/or indicator of the process state and allow countermeasures when needed.

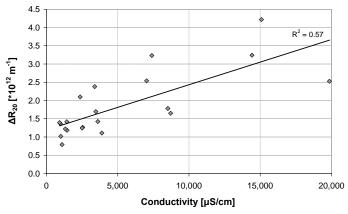


Figure 6: Impact of salt concentration on activated sludge filterability

The results are in accordance with the previous findings and suggest that NaCl, the main road de-icing agent, can have detrimental effects on activated sludge filterability [19]. The activated sludge quality is most probably affected by disturbed floc formation [19, 34, 43], decreased settleability [19, 44, 45], leaching of multivalent cations from the flocs (Ca<sup>2+</sup>, Mg<sup>2+</sup>, Fe<sup>3+</sup>) disturbing the bridging function of these ions in the flocs leading to dispersed sludge, release of EPS and SMP from the floc under high salinity conditions [19], and reduced bacterial activity [46]. Furthermore salinity leads to changes in surface charge, hydrophobicity and bioflocculation [19], all being influencing parameters on filterability [28]. Therefore, although

salt ions are smaller than the membrane pore sizes and thus pass through an MBR membrane, salinity will influence MBR performance indirectly by affecting the biotreatment processes and changes in activated sludge filterability.

## 3.3.2. Toxicity

Peculiar composition of the incoming wastewater can also affect activated sludge filterability and disturb MBR process operation. For example, during the 'winter 2009' experiments in Ootmarsum, membrane performance was affected by an abnormal chemical composition of the incoming wastewater. The substance present in the inflow was polymeric, greasy, oil like similar to candle wax. The polymer was supplied via the sewer network and could be discharged to the sewer system by a local industry or local community inhabitants with the toilet-water. As a consequence of the presence of this polymer, the membrane permeability dropped and there was a need to reduce the flux in order to recover expected levels of performance. It can be assumed that the biological degradation of the polymer was limited and that it was retained on the membrane, likely causing additional fouling problems. The permeability dropped from the 400 L/m<sup>2</sup>·h·bar to approximately 250 L/m<sup>2</sup>·h·bar under the applied flux of 44 L/m<sup>2</sup>·h. The problem was also observed in the CAS system as the sand filter operation was also affected. In order to retain typical MBR operation, cleanings and drainage procedures had to be performed more frequently. This process disturbance started in January 2009 and lasted till June 2009. Thus, it took approximately 6 months and a number of intensive chemical cleanings to recover to normal MBR operation and performance. These operational problems were accompanied by serious filterability deterioration, resulting in poor sludge filterability along the whole membrane bioreactor: 1.9·10<sup>12</sup> m<sup>-1</sup> in anaerobic tank, 2.1.10<sup>12</sup> m<sup>-1</sup> in anoxic and aerobic tanks and 2.2.10<sup>12</sup> m<sup>-1</sup> in the membrane compartment.

Consequently, exceptional poor filterability results were obtained in the winter 2009 campaign in MBR Ootmarsum. During the next experimental campaign in August 2009, thus approximately 2 months after normal operation was restored, the filterability was considered as good and process operation was not hampered anymore. The filterability differences between the two periods were ascribed to the differences in the coefficient  $\alpha_R \cdot c_i$  was  $100 \pm 8 \cdot 10^{-3} \text{ m}^{-2}$  during the polymer incident period,  $47 \pm 6 \cdot 10^{-3} \text{ m}^{-2}$  during the following winter period and about  $20 \pm 9 \cdot 10^{-3} \text{ m}^{-2}$  in the summer periods.

Similar observations were also made by Geilvoet [9] based on comparable experiences encountered during measurements in Varsseveld. At that time, discharges from the local cheese factory containing a synthetic polymer was affecting activated sludge filterability and operation of the MBR. The polymer was not biologically degraded and was retained on the membrane causing serious membrane fouling. The filterability improved significantly from  $5.4\pm2.5\cdot10^{12}$  m<sup>-1</sup> to  $0.3\pm0.2\cdot10^{12}$  m<sup>-1</sup> after the cheese factory was uncoupled from the sewer. Geilvoet [9] observed that differences in the  $\Delta R_{20}$  values were attributed to the differences in the coefficient  $\alpha_{R}$ ·c<sub>i</sub>. During the period of disturbed operation, the coefficient  $\alpha_{R}$ ·c<sub>i</sub> was 190±76·10<sup>-3</sup> m<sup>-2</sup>, whereas during undisturbed operation it was 19±3·10<sup>-3</sup> m<sup>-2</sup>, which is in accordance with our observations. Also in our case, the coefficient  $\alpha_{R}$  c was higher during the period of disturbed operation compared to the period without problems with influent composition, i.e., 100±8·10<sup>-3</sup> m<sup>-2</sup> and 20±9·10<sup>-3</sup> m<sup>-2</sup>, respectively. Furthermore, also in our case, process improvement was observed after the polymer inflow was stopped. The improvement in permeability from about 300-350 L/m<sup>2</sup>·h·bar and fluxes from 13 L/m<sup>2</sup>·h to permeability of approximately 450 L/m<sup>2</sup>·h·bar and the fluxes of 20 L/m<sup>2</sup>·h under stable operation conditions were reported.

Based on the described two cases, we conclude that disturbances in incoming wastewater composition are crucial with regard to MBR operation and activated sludge filterability. If a

toxic or refractory compound is discharged into the sewer network, and the chemical composition of the influent is drastically differs, process disturbances are very likely. Furthermore, duration of the disturbances is directly linked to the time that the compound is supplied and to the hold-up at the plant. Intensive chemical cleanings are necessary to allow day-to-day operation but effectiveness of cleaning in place cannot be guaranteed. When reactor perturbation sustains, detailed analyses of the cause is indispensable. Depending on exposure time, the recovery period could last from few weeks up to few months.

#### 3.3.3. Organic loading rate effect

The COD concentrations in the influent of municipal MBRs were in the range of 100-900 mg/L. The COD load and specific biomass loading varied significantly between the locations:,values between 50-3,300 kg/day and 0.02-0.48 kgCOD/kgMLSS.day were measured Figure 7 illustrates filterability values versus influent COD concentrations for municipal MBRs. The relationship between the contaminant concentration and the sludge filterability for the MBRs treating municipal wastewater is not visible surveying all measurements. Possibly, this can be attributed to the fact that at municipal plants, COD measurements are not performed on a daily basis and average values for the corresponding experimental periods had to be used. However, for the municipal wastewater MBR Heenvliet, where COD measurements were performed on the exact same days as filterability tests, the relation was more apparent [1].

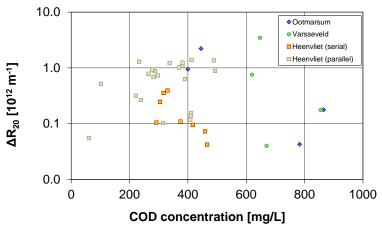
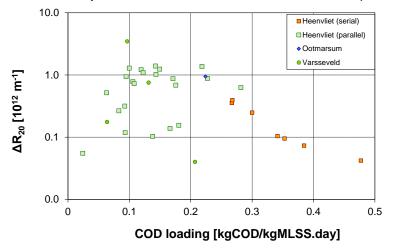


Figure 7: Filterability versus influent COD concentration in municipal MBRs



**Figure 8**: Filterability versus specific COD loading in municipal MBRs. Term 'serial' refers to serial MBR configuration and indicates operation of the MBR in series to the CAS system. Figure 8 illustrates filterability values versus specific COD loading for municipal MBRs. The

results and tendencies of the supposed correlation between the specific organic loading in kg<sub>COD</sub>/kg<sub>MLSS</sub>.day and  $\Delta R_{20}$  were similar to the ones based on the influent concentrations. Again, due to the previously discussed reasons, the relationship between the contaminant concentration and the sludge filterability for the MBRs treating municipal wastewater is not visible when looking at all measurements (Figure 8). Nevertheless, when influent COD measurements are taken parallel to the filterability tests, a clear relationship is observed between the contaminant specific loading and filterability for installations fed with municipal wastewater (Figure 8 and Figure 9).

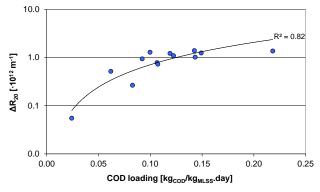


Figure 9: Filterability of activated sludge versus influent specific COD loading in Heenvliet municipal MBR

Although the organic loading of the incoming waste streams appears to not have major influence on sludge filterability, two cases can be distinguished. In one (Figure 9), when an MBR is operated in parallel to the CAS system, the increase in organic loading has negative influence on sludge filterability most likely as a consequence of coinciding seasonal temperature variations. In that case, MLSS concentration varied between 8.3-12.9 g/L and on average was 10.5±1.5 g/L. In the second case (Figure 8), when an MBR is operated in series to the CAS system, the increase in organic loading has a positive influence on sludge filterability most likely due to improved flocculation properties [47]. One of the uncertainties related to serial operation analysis is that since the MBR is placed after the CAS system, CAS sludge has probably utilized higher loading prior reaching MBR. Therefore, keeping in mind the relatively limited number of data points, a general conclusion cannot be drawn without further studies. Nevertheless, under steady operational conditions, good sludge filterabilities are likely to be found even at high specific organic loadings. Furthermore, some factors such as dilution during winter (i.e., low loading) and temperature decrease may occur together. Hence, it is expected that MBRs operated with municipal wastewaters are subjected to synergistic effects of such factors on activated sludge filterability.

Summarizing, there is a relationship between sludge filterability and influent specific COD loading for municipal MBRs.

## 4. CONCLUSIONS

Regarding the impact of the inflow characteristics on activated sludge filterability the following conclusions can be drawn.

- Low temperatures and sudden changes in the influent concentration and composition are the most common causes leading to poor sludge filterability.
- The combination of undesirable events, e.g., hydraulic and/or organic load shocks, harmful composition of the incoming wastewater, is extremely difficult to overcome without filterability deterioration and operational problems.
- Activated sludge loading should not be considered a predominant parameter with respect to filterability in municipal MBRs. The changes in the organic loading are not

solely responsible for the changes in measured filterability, which are most likely a consequence of temperature variations or unexpected events.

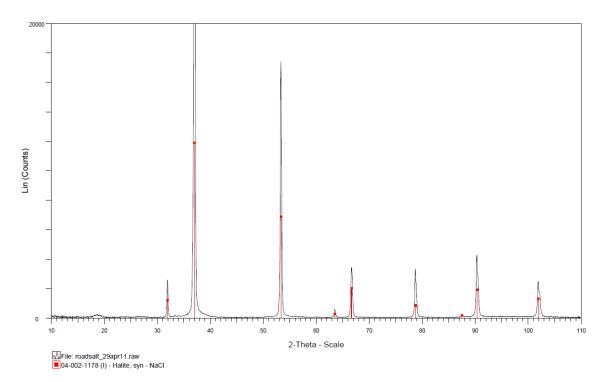
• Temperature can be defined as a major influencing parameter with respect to activated sludge filterability in municipal MBRs.

# Supplementary info: Determination of road salt sample composition by X-ray analysis

The X-Ray powder Diffraction (XRD) and semi-quantitative X-ray Fluorescence (XRF) analysis were performed to precisely determine composition of the road salt. The analysis was carried out by Ruud Hendrikx from the Department of Materials Science and Engineering of the Delft University of Technology.

The XRD patterns were recorded in Bragg-Brentano geometry in a Bruker D8 Advance diffractometer equipped with a Vantec position sensitive detector and graphite monochromator. Data collection was carried out at room temperature using monochromatic Co K $\alpha$  radiation ( $\lambda = 0.179026$  nm) in the 2 $\theta$  region between 10° and 110°, step size 0.0426 degrees 2 $\theta$ . Step time 2 s. The samples were placed on a Si {510} substrate and rotated during measurement. Data evaluation was done with the Bruker program EVA. In the figures the measured XRD patterns are shown in black. The coloured red lines show the peak positions and intensities of the identified phases, such as found using the ICDD pdf4 database [48]. All patterns are background-subtracted, meaning the contribution of air scatter and possible fluorescence radiation is subtracted. The XRF analysis was conducted with Philips PW2400 X-ray wavelength dispersive Fluorescence Spectrometer and data evaluation was done with UniQuant 5.0 software.

The measured XRD pattern is presented in Figure A.1; red lines show the peak position and intensity of halite, NaCI, according to the PDF4 ICDD database [48]. Apart from potassium, calcium and magnesium, together 1.8 weight% of total mixture, no other compounds in significant quantities were detected. The XRF results are in weight% of total mixture and are presented in Table A.1.



**Figure A.1**: Results of X-ray powder Diffraction (XRD) analysis of the road salt sample. The intensity of reflected X-rays, expressed in counts, is shown on the x-axis, and increasing values of 2-theta, i.e., diffraction angle in degrees, are shown on y-axis. The XRD provides qualitative analysis to identify present minerals, whereas, XRF quantitatively determines detailed composition of the salt sample as presented in Table A.1.

Atomic	Element	w/w%	Standard error	Atomic	Element	w/w%	Standar
number				number			error
9	F	<		47	Ag	<	
11	Na	41.6	0.55	48	Cd	<	
12	Mg	0.524	0.058	49	In	<	
13	Al	<		50	Sn	<	
14	Si	0.0151	0.003	51	Sb	<	
15	Px	<		52	Те	<	
15	Р			53	I	<	
16	Sx	0.667	0.074	55	Cs	<2e	
16	S			56	Ba	<2e	0.0053
17	Cl	56.42	0.55	57	La	<	0.0072
18	Ar	0.0483	0.0051	58	Ce	<	
19	К	0.154	0.017	59	Pr	<	
20	Ca	0.47	0.052	60	Nd	<	
21	Sc	<		62	Sm	<2e	
22	Ti	<		63	Eu	0.0098	0.0083
23	V	<		64	Gd	0.0103	0.0043
24	Cr	<		65	Tb	<	0.0047
25	Mn	0.0061	0.0014	66	Dy	<2e	
26	Fe	0.0269	0.003	67	Но	<	0.0075
27	Со	<		68	Er	<	
28	Ni	<		69	Tm	<	
29	Cu	<		70	Yb	<	
30	Zn	<		71	Lu	<	
31	Ga	<		72	Hf	<	
32	Ge	<		73	Та	<	
33	As	<		74	W	<	
34	Se	<		75	Re	<	
35	Br	0.0206	0.001	76	Os	<	
37	Rb	<		77	lr	<	
38	Sr	0.0073	0.0008	78	Pt	<	
39	Y	<		79	Au	<	
40	Zr	<		80	Hg	<	
41	Nb	<		81	TÎ	<	
42	Мо	0.0053	0.0019	82	Pb	<	
44	Ru	<	-	83	Bi	<	
45	Rh	<		90	Th	<	
46	Pd	<		92	U	<	

Table A.1: Results of X-ray fluorescence analysis of the road salt sample

Legend: < - concentration is lower than 50 mg/kg; <2e – weight% lower than two standard errors.

ICDD International Centre for Difraction Data (ICDD), Powder Diffraction File PDF-4, 2005.

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