

## Impact of ambient conditions on SMP elimination and rejection in MBRs

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### Abstract

The widespread application of the membrane assisted activated sludge process is restricted by membrane fouling which increases investment and operating costs. Soluble microbial products (SMP) are currently considered as the major cause of membrane fouling in membrane bioreactors (MBR). This study aims at elucidating and quantifying the effects of varying environmental conditions on SMP elimination and rejection based on findings in a pilot MBR and in well defined lab trials. Several factors are thought to influence the concentration of SMP and their fouling propensity in one way or other but findings are often inconsistent or even contradictory. Here, SMP loading rate was found to have the greatest effect on SMP elimination and thus on concentration in the MBR. The degree of elimination decreased at very low DO and low nitrate concentrations. On average, 75 % of influent SMP were eliminated in both pilot and lab trials, with the elimination of polysaccharides (PS) mostly above 80 %. Rejection of SMP components by the used membrane (PAN, 37nm) ranged mainly from 20 - 70 % for proteins and from 75 - 100 % for PS. Especially protein rejection decreased at higher temperatures and higher nitrification activity. The increased fouling rates at lower temperatures might therefore partly be explained by this increased rejection. Apparently mainly the nitrite oxidising community is responsible for the formation for smaller SMP molecules that can pass the membrane.

**Keywords:** Membrane bioreactor, SMP, dissolved oxygen, nitrate, temperature, fouling

### Nomenclature

#### Symbols

$c$	concentration	$\text{mg L}^{-1}$
$FR$	fouling rate	$(\text{m d})^{-1}$
$J$	flux	$\text{L} (\text{m}^2 \text{h})^{-1}$
$k_d$	decay constant	$\text{d}^{-1}$
$K_S$	half-saturation constant	$\text{mg L}^{-1}$
$R$	rejection	-
$\dot{i}$	reaction/elimination rate	$\text{mg} (\text{L h})^{-1}$
$t$	time	h
$TMP$	transmembrane pressure difference	Pa
$\dot{V}$	volumetric flow rate	$\text{L h}^{-1}$
$\mu$	dynamic viscosity	Pa s

#### Subscripts

<i>i</i>	component
<i>I</i>	inhibitor
<i>meas</i>	measured
<i>R</i>	reactor
<i>Rec</i>	recycle

#### Abbreviations

AE	aerobic
AN	anaerobic
AX	anoxic
BSA	bovine serum albumin
DO	dissolved oxygen
EPS	extracellular polymeric substances
PS	polysaccharides
SMP	soluble microbial products
VSS	volatile suspended solids

## Introduction

The membrane assisted activated sludge process in a membrane bioreactor (MBR) offers many advantages over the conventional activated sludge process. Its widespread application, however, is restricted by membrane fouling which reduces permeate yield and increases investment and operating costs. Extracellular polymeric substances (EPS) in either bound or soluble/colloidal form are currently considered as the major cause of membrane fouling in MBRs. It was shown that the soluble or colloidal, often called the “suspended” fraction is equal to soluble microbial products (SMP). Several operational factors like the type of wastewater, sludge loading rate, sludge age, MLSS concentration, and mechanical stress are thought to influence the concentration of EPS/SMP and their fouling propensity in one way or other (Chang and Lee, 1998, Chang et al., 2002, Rosenberger and Kraume, 2003, Trussell et al., 2004). Despite the large number of technical publications on membrane fouling and EPS/SMP, due to the complex nature of the biological system and the difference in experimental and analytical methods applied many questions remain unanswered to date. Findings frequently are inconsistent or even contradictory and often are only of a qualitative nature. It is mostly agreed that soluble/colloidal EPS, i.e. SMP, are of higher importance than the bound form (e.g. Rosenberger and Kraume, 2003). However, SMP uptake/formation kinetics and the influences of ambient conditions like oxygen and nitrate concentration and temperature, which are subject to large variations in wastewater treatment plants, especially in decentralised systems, have rarely been studied quantitatively.

While often EPS or SMP formation or release is addressed, Drews et al. (2006) observed that soluble compounds analysed as polysaccharides and proteins are actually eliminated to a large extent. By “elimination”, either biodegradation, adsorption or any other mechanism is understood which leads to a disappearance of SMP from the liquid phase. In a pilot MBR fed with domestic wastewater and operated with irregular sludge withdrawal, net elimination was around 80% as long as dissolved oxygen (DO) concentration was above 1 mg L<sup>-1</sup>. DO also seems to influence EPS/SMP properties. In a study on the distribution of EPS in aerobic granules, Wang et al. (2005) found that the granules’ aerobic outer shell contained poorly soluble, noneasily biodegradable and rather hydrophobic EPS whereas the anoxic inner core was filled with readily soluble and biodegradable EPS. Recently, a study on biofilm structure in aerobic/anoxic MBRs was conducted investigating porosity and EPS surface coverage using image analysis techniques (Yun et al., 2006). The authors reported that not only the amount of EPS but also its spatial distribution inside the biofilm may affect membrane filterability. They found the amount of polysaccharide extracted from the aerobic

biofilm was greater than that from the anoxic biofilm despite the smaller resistance of the aerobic film. The confocal images of anoxic biofilms showed that they were highly spread out and that the distribution of polysaccharides was more uniform than the aerobic biofilm where the porosity of the PS structure was high.

The surrogate parameter SMP concentration alone is not sufficient to predict fouling. Not only its prevalence but also its properties depend on environmental factors. E.g., the PS and protein concentrations as commonly analysed photometrically according to Dubois et al. (1956) and Lowry et al. (1951) cannot be differentiated into microbial products or components already present in the feed. By fractionation, Geilvoet et al. (2006) recently showed that proteins and PS present in the influent or arising in denitrification, nitrification and membrane tanks can have rather different sizes. Thus a decrease in the sum parameters PS and protein concentration accompanied by a change in the composition and properties of these substances can go in parallel with increasing fouling rates. This indicates the importance of more detailed chemical as well as data analyses which also consider ambient conditions. In other words, to evaluate the role of SMP on fouling, the influence of various parameters on the two key aspects - occurrence and concentration of SMP on the one hand and their properties like rejection, molecular weight and fouling potential on the other (Fig. 1) - must be taken into account.

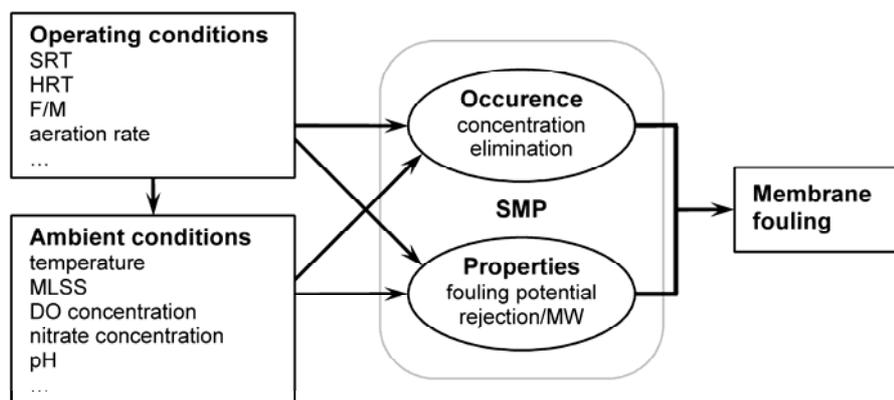


Fig. 1: Potential interactions between operating parameters, ambient conditions, SMP concentration and rejection.

This study aims at elucidating and quantifying the effects of environmental conditions on SMP elimination and rejection by the membrane in pilot scale field experiments and in well defined lab trials.

## Materials and Methods

### *Plants*

A 140 L MBR was operated for one year – initially with irregular, i.e., batchwise and later with regular sludge wastage (Vocks et al., 2007) – at a pumping station (separate sewer) located in a remote area on the outskirts of Berlin. Therefore, influent consisted basically of domestic wastewater devoid of industrial and storm water. This resulted in a highly concentrated influent with, e.g., COD concentrations of  $1,200 \text{ mg L}^{-1}$  on average. Enhanced biological phosphorus removal and post-denitrification without additional carbon dosing were installed (see Fig. 2). In the filtration tank, a  $1.4 \text{ m}^2$  PAN plate and frame module (GKSS, Germany, nominal pore size: 37 nm) was immersed. 14 minutes of permeate withdrawal were followed by a one minute filtration break, i.e., flux was set to  $10 \text{ L m}^{-2} \text{ h}^{-1}$  to achieve  $13 \text{ L h}^{-1}$ . Samples were taken from the buffer tank, from

the membrane chamber and from the permeate twice a week. In order to minimize the influence of daily and weekly fluctuations, samples were always taken periodically on the same days and hours.

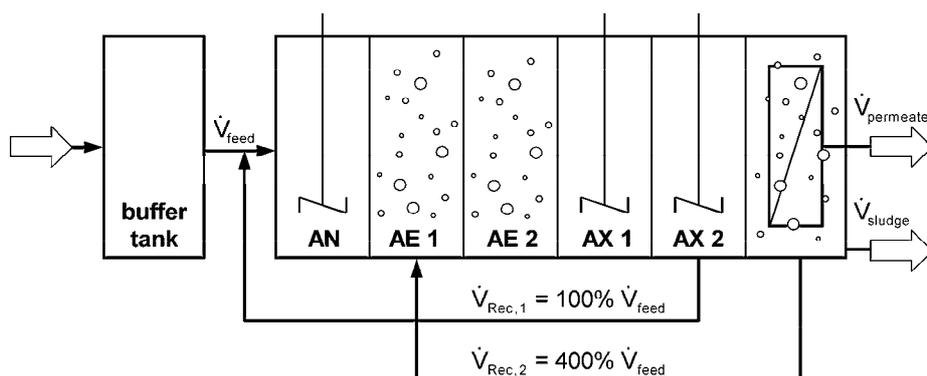


Fig. 2: Pilot MBR (140 L) basic flow sheet (AN: anaerobic, AE: aerobic, AX: anoxic tanks)

To uncouple the effects of ambient conditions (temperature, nitrate and dissolved oxygen concentration) on SMP formation/elimination and thus to study them under more defined conditions, 9 lab-scale trials were carried out with sludge from the above mentioned pilot MBR using a 1 L stirred bioreactor with the same ultrafiltration membrane (PAN, 37 nm) placed around the inside of the wall (Fig. 3). Table 1 shows the good comparability of operating conditions for both plants, i.e., the 9 lab trials represent the whole range of conditions throughout the pilot's operation period of one year. The lab-scale reactor was continuously fed with the same domestic wastewater at HRT = 3.6 h to represent the residence time in the pilot's aerobic tanks. This HRT was chosen because aerobic elimination of SMP was thought to be completed in these tanks. Dissolved oxygen (DO) concentration was controlled via a needle valve. Temperature was controlled by a cooling coil connected to a thermostat. To study the effect of nitrate, sodium nitrate was dosed to the feed. In order to inhibit nitrifier activity, in some runs allylthiourea was added.

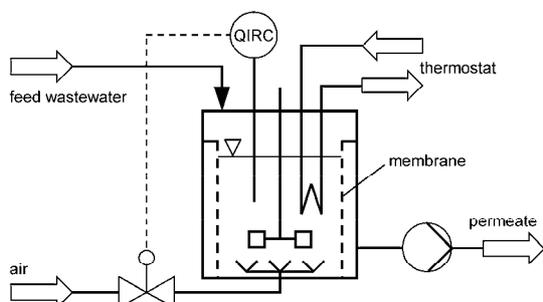


Fig. 3: Lab MBR (1 L) flow sheet

Table 1: Operating parameters for the pilot and lab-scale MBR (\*large variations due to irregular sludge wastage, \*\* averaged for the period of irregular sludge wastage)

		Pilot MBR (one year)	Lab-scale MBR (9 trials)
TS concentration	[g L <sup>-1</sup> ]	3.1 – 13.9*	2.5 - 13
sludge age	[d]	30 – 35**	30
HRT	[h]	10.8 (3.6 in AE)	3.6
temperature	[°C]	11 – 32	10 – 31
influent protein concentration	[mg L <sup>-1</sup> ]	87 – 228	37 - 245
influent PS concentration	[mg L <sup>-1</sup> ]	10 – 70	8 - 40

Again, samples were taken in the feed, reactor and permeate. Step increases and decreases of DO concentration ( $0.1$  to  $5 \text{ mg L}^{-1}$ ) and temperature were performed and each new value was kept constant for approx. 24 h. Each lab trial lasted for approx. one week, during which time the feed was cooled to  $7^\circ\text{C}$  to minimise biodegradation in the feed tank. Trials were repeated with fresh sludge to avoid its acclimatisation to the specific conditions in the 1 L MBR and to ensure reproducibility of field conditions.

## Analyses

Suspended solids were separated from the liquid phase containing soluble and colloidal substances by paper filtration (black ribbon, Schleicher & Schuell). Polysaccharide (PS) concentration in the sludge filtrate, influent filtrate and in the plant permeate was measured according to the photometric method proposed by Dubois et al. (1956) which yields results in glucose equivalents. Protein concentrations were measured according to Lowry et al. (1951). Total SMP concentration was assumed to be the sum of PS and protein concentrations which typically constitute the main fractions.

Calibration for PS was carried out with glucose in the range of  $2 - 80 \text{ mg L}^{-1}$ . Nitrate and nitrite in the sample were found to impair the photometric measurement and to lead to elevated PS values. Therefore, nitrate and nitrite were measured (ion chromatography) in each sample and the measured value was corrected according to eq. (1):

$$c_{PS} = c_{PS,meas} - 0.099 \cdot c_{NO_3-N} - 1.9 \cdot c_{NO_2-N} \quad (1)$$

Proteins were calibrated with BSA in the range of  $5 - 200 \text{ mg L}^{-1}$ . Since the method according to Lowry et al. (1951) is not protein specific but also responds to humic substances, the modified method by Frolund et al. (1996) is often used. In this study, the modified method could not be employed because precipitation occurred during analyses which led to higher absorption. Hence, the presented protein data include humic substances which in comparison to protein concentrations in this study usually are only present in small concentrations (approx.  $10 \text{ mgC L}^{-1}$ ). Also, they generally pass the membrane unhindered and therefore do not contribute too much to rejection. All protein concentrations were corrected for allylthiourea. From concentration data, the respective elimination rate  $\dot{r}_i$  was calculated using the following mass balance:

$$\dot{r}_i = \frac{\Delta c_{i,R}}{\Delta t} - \frac{\dot{V}_{feed}}{V_R} \cdot \bar{c}_{i,feed} + \frac{\dot{V}_{permeate}}{V_R} \cdot \bar{c}_{i,permeate} + \frac{\dot{V}_{sludge}}{V_R} \cdot \bar{c}_{i,R} \quad (2)$$

This rate was divided by MLVSS to yield a specific elimination rate which – like specific growth rates or F/M ratios – can be used for comparison of trials with different biomass concentrations.

## Results and Discussion

### SMP elimination

Throughout the study, PS concentrations ranged from  $3 - 146 \text{ mg L}^{-1}$  in the sludge filtrate and from  $0 - 8 \text{ mg L}^{-1}$  in the permeate. Protein concentrations were  $0 - 210 \text{ mg L}^{-1}$  and  $0 - 42 \text{ mg L}^{-1}$ , respectively. Fig. 4 shows a summary of results from all lab experiments. Specific elimination of total SMP, PS and protein as calculated using eq. (2) is plotted over the respective sludge loading rate. Due to different batches of feed used throughout the study, biomass growth during the

experiment, and slight changes in the feed despite cooling, specific SMP load varied between 1.2 and 7.7 mgSMP (gVSS h)<sup>-1</sup>. Results exhibit some scatter (all data far from bulk were identified as measurement errors) but as can be seen, linear relations exist between loading and elimination rates. Therefore, to evaluate the influence of other conditions, the degree of elimination (specific elimination rate over specific loading rate) was used instead of the specific elimination rate. Average elimination degrees were 75.1 % (total SMP), 87.4 % (PS) and 72.5 % (proteins). Fig. 4c) also includes pilot data which fits well into the lab-scale results. Total SMP and PS elimination in the pilot was around 75 % and 80 % on average, i.e., lab trial results can be considered as representative.

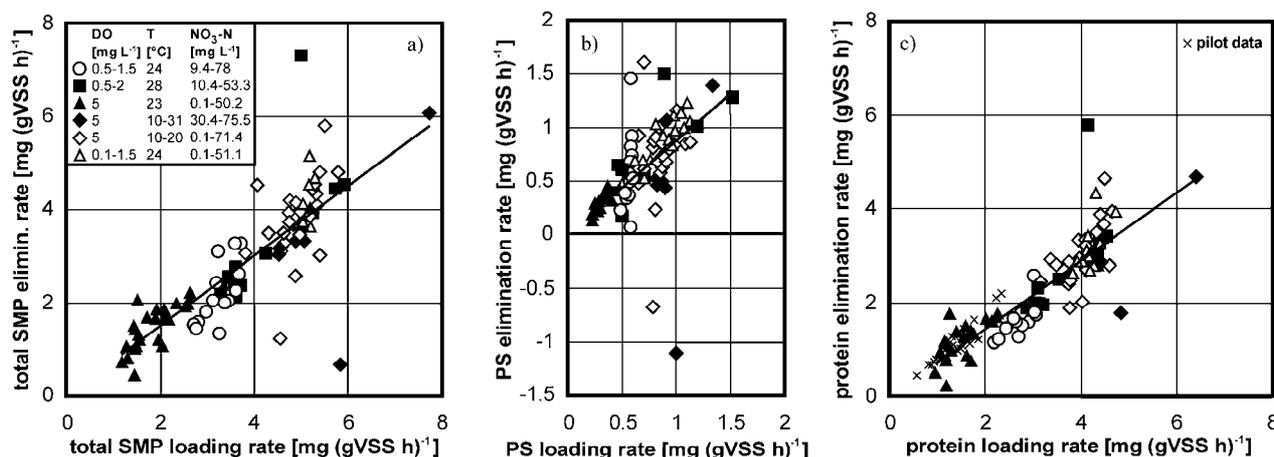


Fig. 4: Specific elimination rate over respective sludge loading rate: a) total SMP, b) PS, c) proteins. — average over all lab trials.

Besides DO as an oxygen source for mineralisation of SMP, nitrate seems to influence SMP elimination. Especially during a period of irregular sludge wastage, a decrease of SMP concentration with increasing nitrate content in the membrane tank was found (Drews et al., 2006). This indicates higher elimination rates in the presence of nitrate or during nitrification and is in agreement with Rosenberger et al. (2006) who found that PS are mainly degraded in the first anoxic tanks of a pre-denitrification MBR. Fig. 5 shows elimination degrees as a function of different oxygen sources, DO and nitrate, as observed in lab trials.

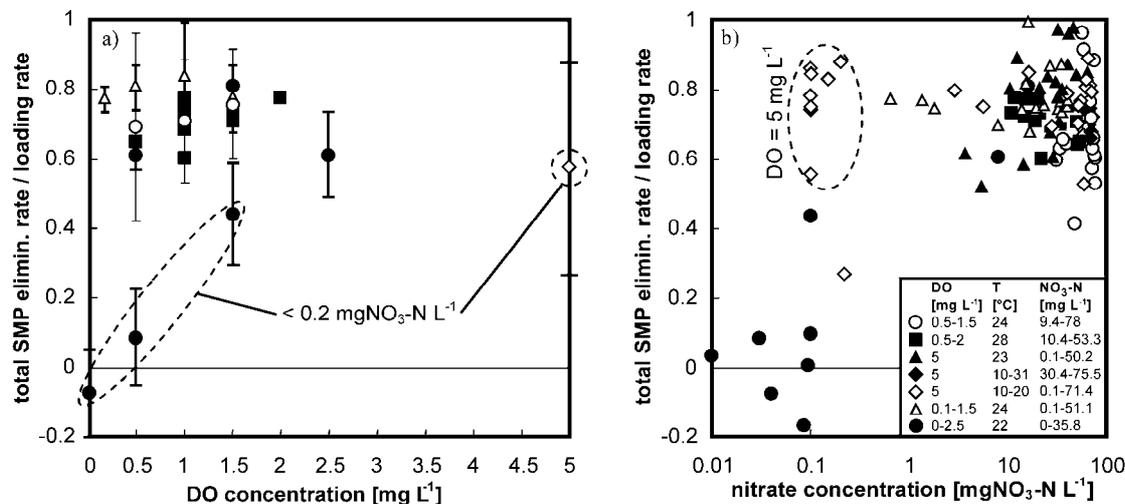


Fig. 5: Degree of total SMP elimination over concentration of oxygen sources (lab trials, same symbols as in Fig. 4, • data from Drews et al. (2006)): a) DO, b) nitrate.

As can be seen in Fig. 5a), like in the pilot, influent SMP was eliminated by approx. 75 % on average, but only as long as nitrate concentration was above approx.  $0.2 \text{ mgNO}_3\text{-N L}^{-1}$ . This observation is confirmed by Fig. 5b), where all eliminations with nitrate concentrations above approx.  $0.5 \text{ mgNO}_3\text{-N L}^{-1}$  range between 40 and 100 %. Only the combination of low nitrate and low DO concentrations leads to small eliminations, in one case even to a net formation of SMP. Due to the sludge and influent characteristics, only a few data points could be generated so far for very low nitrate concentrations. These are too few to be able to make a final judgment and should only be seen as an indication of a possible influence.

Due to a number of still combined and interacting effects results show some scatter, but Fig. 5 suggests that SMP uptake kinetics can be described by the simultaneous uptake of two non-essential substrates (DO and nitrate) and parallel decay (i.e., SMP formation) with the dependence on DO concentration represented by a Monod-type term and that of nitrate represented by non-competitive inhibition. This is in agreement with Lu et al. (2001) who from numerical studies proposed a similar mechanism:

$$\frac{\dot{r}_{SMP}}{c_{VSS}} = \frac{\dot{r}_{SMP,max}}{c_{VSS}} \cdot \left( \frac{c_{DO}}{c_{DO} + K_{S,DO}} + \frac{c_{NO_3}}{c_{NO_3} + K_{S,NO_3}} \cdot \frac{K_{I,DO}}{c_{DO} + K_{I,DO}} \right) - k_d \quad (3)$$

SMP measurements in paper filtered samples from all tanks showed that protein elimination was already accomplished in the first aerobic tank while PS gradually diminished in AE2 and AX1 and then rose again slightly in AX2 where both DO and nitrate are missing. By a fractionation it was shown that SMP components indeed changed by degradation/formation: Only 40-50% of the influent PS which had passed the paper filter passed a membrane filter (cellulose acetate,  $0.2 \mu\text{m}$ ) while 80-95% of the membrane tank sludge supernatant did.

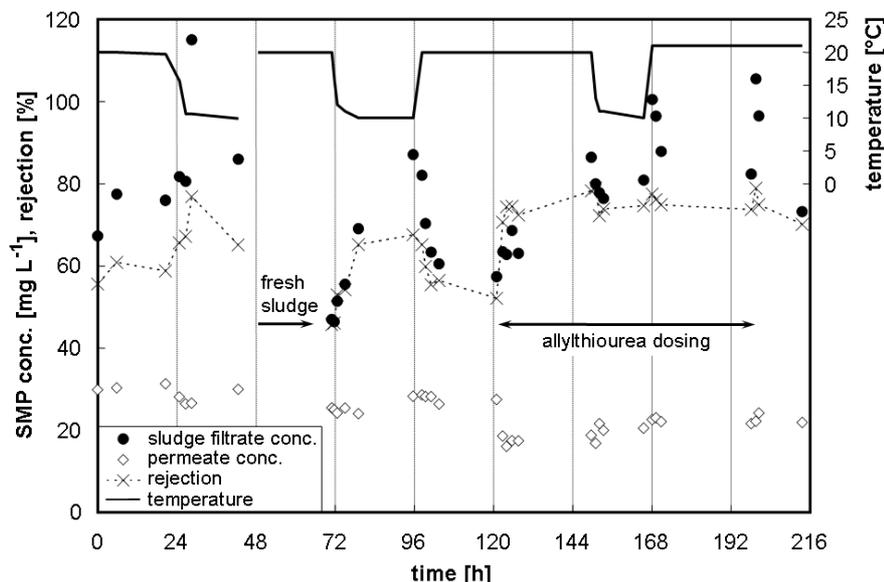


Fig. 6: Effect of temperature changes on total SMP concentrations and rejection (two lab scale experiments)

Like all reaction kinetics, temperature is expected to influence the elimination rate. However, the effect of sudden temperature changes on SMP concentration was sometimes found to be higher than the influence of steady state temperature (see overshooting of sludge filtrate concentration at 28 and 168 h in Fig. 6). Temperature drops caused an increase in sludge SMP concentration which was reversible when temperature was increased. At 121 h, allylthiourea was added. Immediately, the

SMP concentration in the permeate dropped by approx. 40 % while that in the sludge filtrate increased. As a result, SMP rejection which initially followed the sludge filtrate profile instantly increased and then remained almost constant irrespective of temperature. Thus, it can be assumed that nitrification might be connected to the production of small SMP molecules that can pass the membrane, i.e. the stress due to inhibition (presence of allylthiourea) might lead to a decreased production of smaller SMP. Influences on rejection will be discussed in more detail in the following.

### SMP rejection

The concentrations of SMP fractions in the MBR are not only governed by influent and elimination rates but also by their respective rejection. In lab trials, rejection ranged mainly between 20 and 65 % for proteins and between 75 and 95 % for PS, values similar to those observed in the pilot. Since elevated temperatures lead to stronger movement of the molecules – whereby solubility is increased and adhesive forces are decreased – a higher mass flux of SMP through the membrane was expected at higher temperatures. Fig. 6 and Fig. 7 confirm this especially for proteins in the lab trial. Pilot data scatter more but show a similar trend. While a slight tendency can be observed for PS in the lab trial, none can be seen in the pilot. Another explanation for a rejection decrease could be the formation/release of smaller SMP fractions at higher temperatures. During all lab experiments shown, temperature was first decreased and later increased (see below), i.e., aging effects of the membrane or the development of a selective layer on the membrane can be ruled out as causes for the observed trends. In addition, according to Ye et al. (2006) the achievement of constant rejection only takes approx. 1 h after start-up and therefore is thought to have been accomplished in each new condition period.

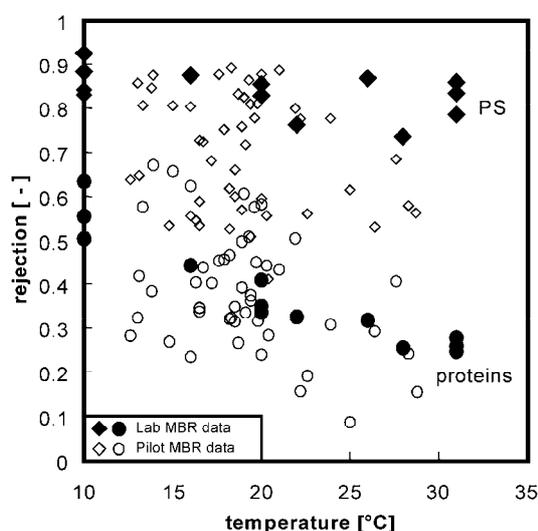


Fig. 7: SMP rejection by the membrane over temperature (lab trial conditions:  $c_{DO} = 5 \text{ mg L}^{-1}$ ,  $c_{NO_3-N} > 30 \text{ mg L}^{-1}$ ).

DO concentration showed no unambiguous influence on either rejection. However, the concentration of nitrogen compounds did (see Fig. 8). As can already be seen in Fig. 6, the addition of allylthiourea caused a steep increase in rejection. Especially protein rejection was found to be higher with inhibited ammonia oxidation through either addition of allylthiourea or temperature decrease, i.e., when the concentration of ammonia rose (Fig. 8a)) or when ammonia oxidation rate decreased (Fig. 8b)). Since rejection was almost unaffected by temperature in the presence of allylthiourea (see Fig. 6), it is concluded that temperature only has an indirect effect on rejection as a nitrification inhibiting factor. In three lab trials, however, no dependence of protein rejection on

ammonia oxidation rates was observed. In these runs, significant amounts of nitrite were detected, i.e., apparently nitrite oxidation was inhibited. Fig. 8c) shows protein rejection over nitrite concentration for one of them (for the other two runs, trends were similar but not as pronounced). The inhibition of nitrite oxidisers such as *Nitrobacter* therefore seems to be the most relevant one for the lack of SMP molecules that are small enough to pass the membrane.

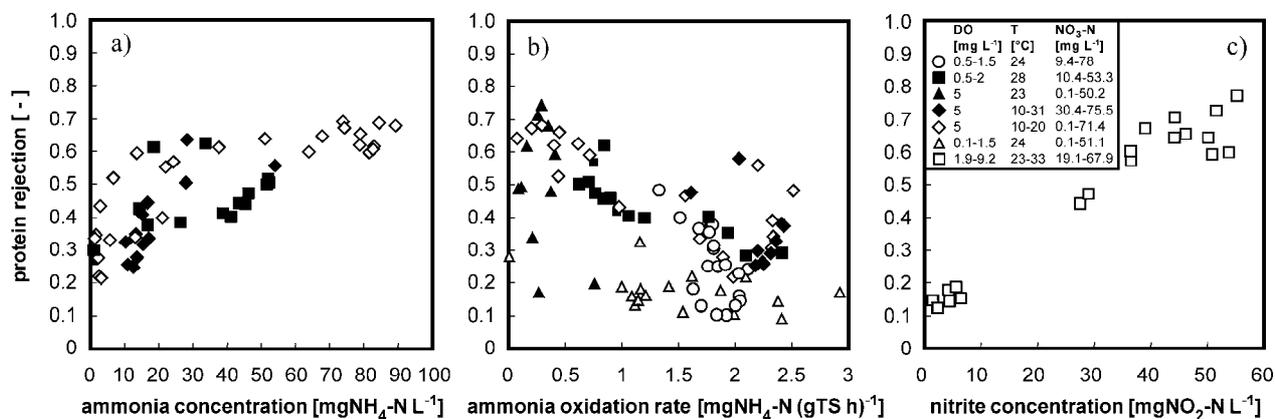


Fig. 8: Protein rejection by the membrane over a) ammonia concentration, b) ammonia oxidation rate, c) nitrite concentration (lab trials, same symbols as in Fig. 4).

Dropping temperatures or addition of allylthiourea increases the pH-value. SMP concentrations and rejection are therefore plotted over pH in Fig. 9. A clear trend can be observed for permeate concentration and rejection, i.e., as pH is increased through either decreasing temperature or addition of allylthiourea/inhibition of nitrification, less SMP pass the membrane. Again, over more than 10 days of operation conditions were changed “back and forth” which means that Fig. 9 does not show a time effect. Interestingly, the observed trend was completely different in the three runs where nitrite was detected: In the presence of significant amounts of nitrite (> 1 mg L<sup>-1</sup>), rejection decreased with increasing pH.

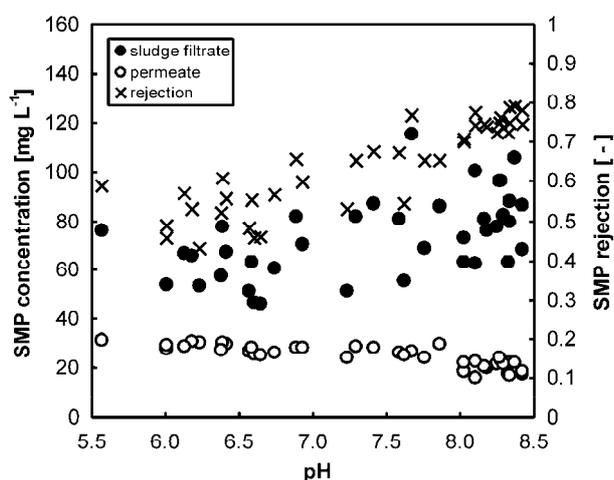


Fig. 9: SMP concentrations and rejection over pH (data from two lab trials with low nitrite concentrations)

#### Relation to fouling rate

The relation between rejection and fouling is yet unclear. When SMP on average are big enough to be rejected by a high percentage, they are likely to form a cake which can give rise to external

fouling. On the other hand, if rejection is low, i.e., SMP are small enough to pass the pores, it is quite likely that a certain amount also gets stuck inside the pores or adsorbs to the inner walls of the pores and thereby causes internal fouling. In agreement with the hypothesis that an inhibition of nitrification increases protein rejection (see above), elevated PS rejection and very high PS concentrations of up to  $150 \text{ mg L}^{-1}$  were found in the pilot plant during a period of low nitrification activity. However, at the same time fouling was rather low (Drews et al., 2007). This could indicate that under these conditions SMP were mainly too large to cause internal fouling but rather formed a loose cake.

In this study, fouling rate

$$FR = \frac{dTMP}{dt} \cdot \frac{1}{\mu(T) \cdot J} \quad (4)$$

in the pilot was mostly below  $10^{11} \text{ (m d)}^{-1}$  (Drews et al., 2007). It was found that possibly due to the high sludge ages employed, fouling rate was not solely affected by SMP but also by other sludge properties (compare Lee et al., 2002, Grelier et al., 2006, Drews et al., 2006). It should also be noted that the applied flux of  $10 \text{ L (m}^2 \text{ h)}^{-1}$  was rather low.

At a similar flux, fouling rates in the lab MBR were higher due to the membrane arrangement and thereby the smaller air scour efficiency. Therefore, they cannot be compared to pilot values but give qualitative indications of fouling progression. Fig. 10 shows the change of fouling rate in a lab-scale run where temperature was first decreased from  $20 \text{ }^\circ\text{C}$  to  $11 \text{ }^\circ\text{C}$  and later increased again to  $21 \text{ }^\circ\text{C}$  (hours 122 to 214 in Fig. 6). Despite the fact that the permeate viscosity was corrected for temperature (compare eq. (4)), fouling rate increased during cooling and later decreased again when temperature was increased. Since these fouling rates were determined during a period when rejection was not influenced by temperature (during allylthiourea dosing) it is concluded that indeed rejection alone is not an indication of fouling. Possibly adhesive forces are reduced and the rejected cake is not as sticky at higher temperatures.

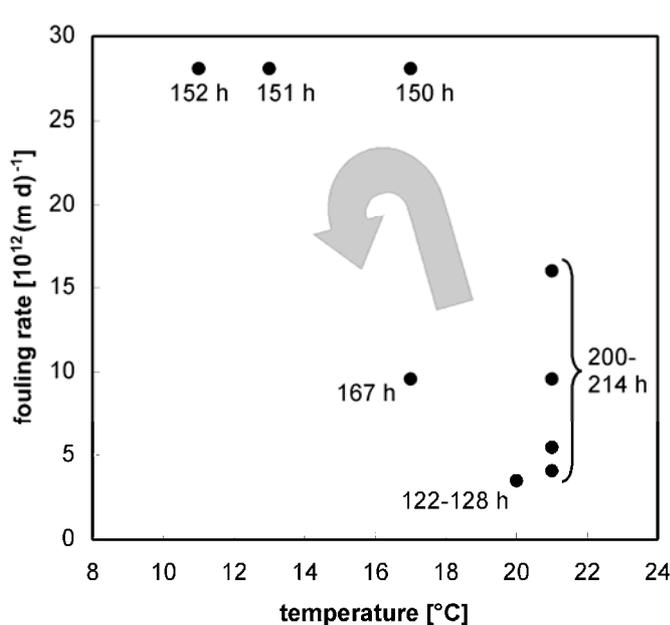


Fig. 10: Fouling rate over temperature in a lab trial ( $c_{DO} = 5 \text{ mg L}^{-1}$ ,  $c_{NO_3-N} = 0.1 - 71.4 \text{ mg L}^{-1}$ )

## Conclusions

Multiple and complex interactions occur between ambient conditions and SMP elimination and rejection. Therefore, no final conclusions can be drawn but results indicate the following relationships:

- Through field experiments and well-defined lab trials it was shown that SMP loading rate has the strongest influence on SMP elimination rate in MBRs. For a comparison of different studies, the degree of elimination should therefore be used in the future. Here, average polysaccharides and proteins eliminations were 87% and 73% of the influent, respectively. It should be noted that sample preparation was done by paper filtration, so values could be somewhat different when applying centrifugation.
- Besides SMP load, DO and nitrate concentrations appear to have an impact on SMP elimination and thereby on SMP concentration with SMP elimination being lower at low availability of oxygen sources. To achieve low SMP concentrations, a sufficient supply of oxygen is therefore required in the membrane tank.
- Especially protein rejection was found to be influenced by ammonia oxidation activity and thus by temperature. Rejection was higher at low temperatures or otherwise inhibited nitrification. Here, nitrite oxidisers seem to be responsible for the formation of smaller SMP compounds that can pass the membrane. The build up of nitrite can thus be used as an indication of increased SMP rejection or lower permeate concentration. Two opposite effects of pH on protein rejection were observed. Normally, rejection increased with pH while in the presence of nitrite it decreased at higher pH.
- Sudden temperature changes yield spontaneous SMP release and increase in fouling rates. Therefore, to avoid fouling in MBRs, special attention needs to be paid to SMP concentrations in small or decentralised plants where operating and environmental conditions are subject to higher fluctuations than in large plants. If possible, steep temperature gradients should be avoided.

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