Performance of a MBR for the treatment of blackwater

H. Knerr, A. Rechenburg, T. Kistemann and T. G. Schmitt

ABSTRACT

The paper describes the experience gained in operating a membrane bioreactor (MBR) for the treatment of blackwater. Beside a complete characterization of blackwater, operational conditions and removal efficiency concerning parameters such as COD, BOD_5, nitrogen and phosphorus as well as microbiological parameters were determined. Furthermore the membrane performance was investigated. The results show that in blackwater treatment nitrogen removal is limited in the biological process, because of the blackwater matrix (BOD_5:TKN = 1.1:1.0). Blackwater contains a high fraction of soluble, inert COD, which is not degradable by biological operation, only. Phosphorus elimination was negligible, probably induced by precipitation of cellular phosphorus. Although the released permeate was free of the fecal indicators *E. coli* and streptococcus and met guideline values, a direct reuse as service water is not recommended due to the yellowish coloration.

Key words | blackwater, membrane bioreactor, reuse

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INTRODUCTION

Domestic drinking water demand in industrialized countries ranges between 100 and 150 L per capita and day. The resulting wastewater can be divided into 60 to 70% of greywater and 30 to 40% of blackwater. Greywater is characterized as the lowest polluted domestic wastewater stream and a lot of alternative sanitation models pursue concepts with reuse of treated greywater (Friedler *et al.* 2005; Nolde 2005 etc.). Most of the blackwater in households can be allocated to toilet flushing. As a consequence blackwater consists of a mixture of feces, urine, toilet paper and transportation water. It contains high amounts of organic matter, nitrogen, phosphorus and potassium. Therefore most studies on blackwater reuse concentrate on anaerobic systems in order to apply treated blackwater in agriculture as a fertilizer or to generate biogas (Kujawa-Roeleveld *et al.* 2005, Luostarinen *et al.* 2005 etc.). On the other hand, the aerobic treatment of blackwater, e.g. for reuse in toilet flushing, is an alternative approach (Otterpohl *et al.* 1999). In this context membrane bioreactors (MBR) have a key function, because high chemical and microbiological effluent qualities can be achieved.

Biological treatment of source-separated blackwater using MBR technology was applied by Lindner *et al.* (2004) already. But the focus of these investigations was the development of a process scheme for the purification of blackwater for its reuse as a toilet flushing water (Lindner 2008), not the investigation of biological treatment processes. Nevertheless, within the biological treatment process the matrix of blackwater have to be examined in greater detail (Knerr *et al.* 2007). Therefore, a MBR in semi-technical scale was operated with separated blackwater to analyze fundamental aspects of aerobic blackwater treatment processes. The results of these investigations, with a focus on the overall removal performance and the membrane performance are presented in this paper.

MATERIALS AND METHODS

Experimental setup and operation conditions

Raw blackwater used in the study originated from conventional flushing toilets (9 L/flush). The toilets were located in 8 apartments with 15 inhabitants in downtown Kaiserslautern (Germany). The blackwater was conveyed gravitationally to a pumping station. From there it was pumped to the pilot plant, which was located outside the building in a container.

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To protect the membranes from clogging the pilot plant was equipped with a rotation screen (0.1 mm). By this, toilet paper and feces were removed from the blackwater influent. Furthermore the plant was fitted with a storage tank (V = 500 L) serving as a flow buffering tank. The semi-technical scale MBR (V = 1,200 L) operated in the study used continuous flow reactor technology, realized as pre-denitrification (V = 500 L). The system included a submerged ultrafiltration capillary module with a nominal pore size of 0.05 m and a total membrane area of 10 m. The membrane material was polyether sulphone (PES). Nitrification zone (V = 500 L) and membrane tank (V = 200 L) were aerated by compressed air. While the tank with the submerged membrane was aerated by large bubble aeration (crossflow aeration), the nitrification reactor was arranged with fine bubble aeration. The schematic layout of blackwater pilot plant is given in Figure 1.

The MBR was fed over 10 months up to 50.6 L/h constantly, with an average flow rate of 26.4 L/h accordingly 501 L/d, regulated depending on the fluid level in the MBR. The volume of the storage tank was adjusted to achieve a hydraulic retention time (HRT) in the range of 8-10 h. The volume of the biological reactor was operated with an average HRT of about 2 d. Permeate was extracted by applying negative pressure on the membrane using an eccentric screw pump. Operational cycle lasted 10 min with 9 min of permeation and 1 min relaxation.

The MBR was seeded with 200 L sludge adapted to real blackwater from another pilot plant. During the first three months of investigations MBR operation circumstances were varied until stable conditions and low effluent concentrations were reached. Hence, the start-up phase is neglected in the following examination.

Sampling and analyses

To asses the physical-chemical performance of the pilot plant, samples were taken twice a week. From the influent 24 h composite samples were taken. Effluent was monitored by applying random sampling. All samples where analyzed for 15 parameters. Ammonium-nitrogen (NH_4-N), nitrite-nitrogen (NO_2-N), nitrate-nitrogen (NO_3-N) and chemical oxygen demand (COD, total and dissolved) were analyzed using cuvette tests (Hach Lange). Analyses of total nitrogen (TN), total organic carbon (TOC), dissolved organic carbon (DOC), total phosphorus (TP), orthophosphate (PO_4-P), biological oxygen demand (BOD_5), alkalinity ($S_{KS4.3}$), organic bounded nitrogen (N_{org}), total suspended solids (TSS) and volatile suspended solids (VSS) were carried out according to German standard methods (DIN). For TN-, TOC- and DOC-measurements an autoanalyzer (Dimatec) was used. For measurements of pH, temperature (T) and electrical conductivity (EC) several probes (WTW) were applied. All samples were taken using plastic bottles (PE), cooled at 4°C, immediately transported to the laboratory and analyzed within 8 hours.

The microbiological analysis took place on a bi-weekly scheme. Random samples were taken from the storage tank and permeate, where flammable sampling fitting was installed. All samples were immediately stored in a cooling box and transported to the laboratory within 4 h. Processing of the samples was done latest within 16 h after sampling.

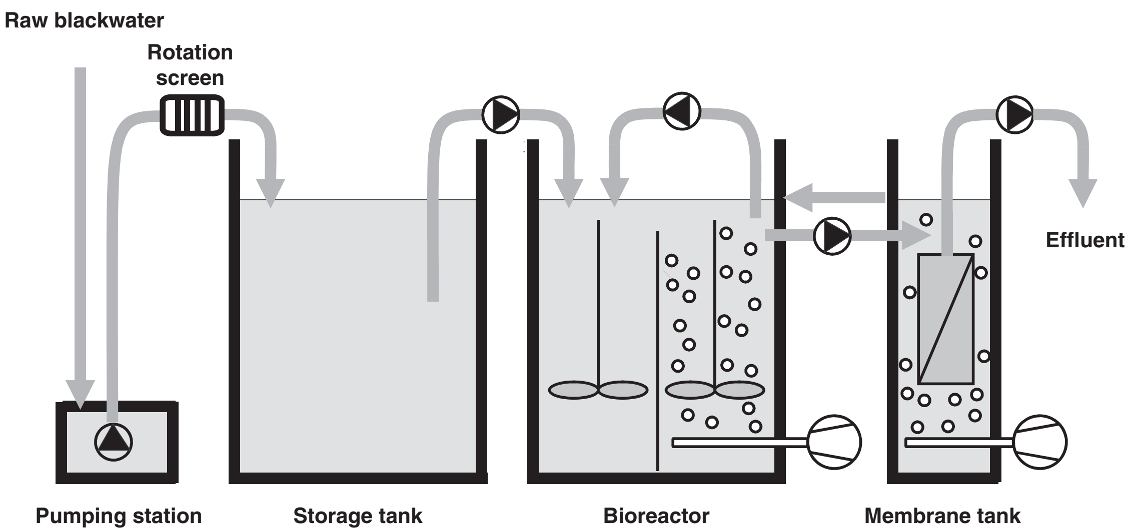

Figure 1 | Schematic layout of blackwater pilot plant.

All samples were analyzed for the parameter *E. coli*, (ISO 9308-1), fecal streptococci (ISO 7899-2), *Pseudomonas aeruginosa* (ISO 16266) and somatic coliphages (ISO 10705 2).

RESULTS AND DISCUSSION

Blackwater characterization

The analysis of the major parameters of raw and mechanically treated blackwater, including pH, EC and microbiological parameters in comparison with raw blackwater from vacuum toilets (1 L/flush) and high strength domestic wastewater is summarized in Table 1.

With regard to the organic content, the mechanically treated blackwater showed similar characteristics as domestic wastewater, because toilet paper and feces were removed from the influent and dilution takes place by toilet flushing. Average COD concentration was 720 ± 143 mg/L. The average ratio of BOD_5:COD was 0.43, which is identical as the ratio of 0.45 for domestic wastewater. Hence, the blackwater fed to the MBR showed a similar biological treatability.

Concerning nutrients obvious differences to domestic wastewater appeared in the mechanically treated blackwater. The concentrations of nitrogen and phosphorus were very high. Average total Kjeldahl nitrogen (TKN) concentration was 278 ± 34 mg/L (83% resulted from NH_4-N). TP concentration was about 29.2 ± 3.3 mg/L in average, of which 87% was soluble PO_4-P. These results indicated almost 3 to 4 times higher nutrient concentrations than domestic wastewater. So the relation between carbon and the nutrients was shifted towards nitrogen and phosphorus resulting in a BOD_5:TKN:TP ratio of about 100:92:9.8. In contrast the BOD_5:TKN:TP ratio reported by Metcalf & Eddy (2003) for high strength domestic wastewater is 100:9.0:1.5.

A comparison of the measured raw blackwater characteristics with raw blackwater originating from vacuum toilets using 1 L water per flush (Kujawa-Roeleveld *et al.* 2005) gives

Table 1 | Measured blackwater characteristics (mean values incl. standard deviation) compared with raw blackwater from vacuum toilets (Kujawa-Roeleveld *et al.* 2005) and high strength domestic wastewater (Metcalf & Eddy 2003); n.d. = no data

Parameter	Unit	This study		Kujawa-Roeleveld *et al.* (2005)	Metcalf & Eddy (2003)
		raw	mechanically treated		
pH	[-]	9.0 ± 0.1	8.44 ± 0.3	8.81 ± 0.2	7.5
EC	[mS/cm]	2.3 ± 0.2	2.6 ± 0.2	n.d.	n.d.
BOD_5	[mg/L]	524 ± 118	323 ± 92	n.d.	400
COD	[mg/L]	2,887 ± 793	720 ± 143	9,503 ± 6,460	1,000
TOC	[mg/L]	1,178 ± 272	306 ± 62	n.d.	n.d.
TKN	[mg/L]	273 ± 39	278 ± 34	1,025 ± 130	85
NH_4-N	[mg/L]	202 ± 32	231 ± 22	708 ± 101	n.d.
NO_3-N	[mg/L]	2.2 ± 0.7	1.8 ± 0.3	n.d.	n.d.
TP	[mg/L]	34.4 ± 6.0	29.2 ± 3.6	114 ± 63	15
PO_4-P	[mg/L]	23.3 ± 2.4	25.4 ± 2.6	29.0 ± 17	n.d.
TSS	[mg/L]	1,697 ± 395	67 ± 64	n.d.	350
VSS	[%]	96.1 ± 1.8	72.9 ± 15.3	n.d.	275
E. coli	[cfu/mL]	n.d.	1.70E + 06	n.d.	1.0E + 06 to 1.0E + 08[a]
Fecal streptococci	[cfu/mL]	n.d.	1.63E + 06	n.d.	1.0E + 03 to 1.0E + 04[b]
Ps. aeruginosa	[cfu/mL]	n.d.	9.54E + 04	n.d.	1.0E + 01 to 1.0E + 02[b]
Coliphages	[pfu/mL]	n.d.	1.60E + 03	n.d.	n.d.

[a]total coilforms, [b]typical untreated wastewater

information about the dilution of the investigated blackwater by flushing water. A comparison of the raw and the mechanically treated blackwater of the analysis executed, illustrate the efficiency of the solid separation unit used.

Organic removal efficiency

The characteristics of COD and BOD_5 profiles in effluent and permeate during operation are presented in Figure 2(a). Predominantly, the sludge loading rate in MBR ranged between 0.010 to 0.030 g BOD_5/(g TSSd), but sometimes decreased to values $<$ 0.010 g BOD_5/(g TSSd). Hence, BOD_5 concentrations in the permeate were constant below the detection limit of 3.0 mg/L. Therefore BOD_5 removal efficiency was $>$ 99% and it can be assumed, that biological treatment of organic matter was nearly completed. In contrast, COD concentrations in the permeate were quite high. An average concentration of 136 ± 56 mg/L soluble COD was found. This is equivalent to an elimination rate of about 82%. Taking the long HRT (2 d) of the system and the low effluent BOD_5 concentrations into account, this portion was soluble inert organic material, which was not degradable by biological means.

The organic material contained in permeate will depend not only on the initial concentrations of organics, but also on refractory organic products generated through microbial metabolism. Caused by the sludge retention time (SRT) of the MBR, which was constantly 50 d or higher (see below), it is assumed that a main fraction of the soluble organics in permeate was not original substrate. E.g. Germirli *et al.* (1991) showed that the concentrations of inert soluble organics in influent can be doubled due to products of cell lysis and degradation. To avoid misinterpretations of the biological treatability of blackwater further examinations would be necessary to identify the inert soluble organics, which pass through the biological treatment in an unchanged form.

Nitrogen removal efficiency

Figure 2(b) illustrates the long-term characteristics of TKN and NO3-N in influent and permeate of the MBR. A complete nitrogen removal was not achieved. On the one hand there was not enough readily biodegradable carbon in the influent of the MBR available for the reduction of nitrate, as a result of the low BOD_5:TKN ratio. Accordingly the denitrification process was limited. On the other hand the oxidation of the high ammonium influent concentrations in combination with the carbon dioxide (CO_2) production due to the respiration of the organic content leads to a significant pH decrease, on the average about pH 6 and lower. Thus, nitrification was also incomplete. TKN-elimination rate was only 80%, which can be explained by inhibitation of the ammonia oxidizers Nitrosomonas/Nitrobacter by nitrous acid (HNO_2) (Udert *et al.* 2003). As a result the TN removal efficiency in the first month of observation was only 42% with average permeate concentrations of 64.6 ± 11.1 mg/L concerning TKN (89% resulted from NH_4-N), 104 ± 17.2 mg/L concerning NO_3-N and 0.07 ± 0.01 mg/L concerning NO_2-N. In this context the low NO_2-N concentrations in permeate have to be highlighted. It is assumed that denitritation (nitrogen removal via the nitrite pathway) took place in the anoxic zone, caused by the backflow from the nitrification zone, resulting in the low NO_2-N concentrations observed in permeate.

To enhance the nitrification process, a dosage of sodium hydroxide (NaOH) was integrated into the biological treatment process. By this, constant pH values between 7.5–8.5

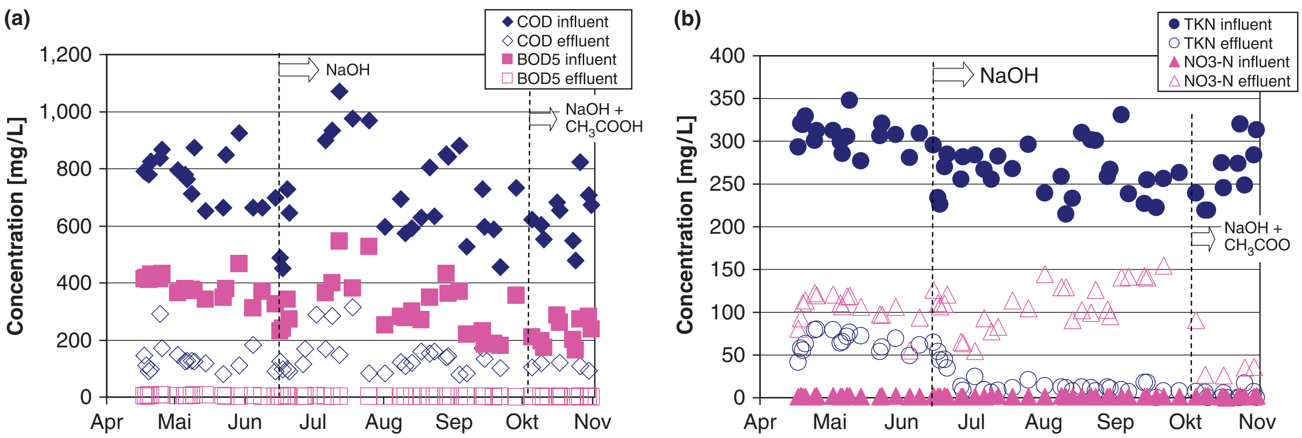

Figure 2 | COD and BOD_5 profiles (a) and TKN and NO_3-N profiles (b) during operation.

were achieved, which resulted in a TKN elimination rate at stable values of about 93% and average TKN permeate concentration of 18.7 ± 15.7 mg/L. Furthermore TN removal efficiency increased to an average rate of about 52%. A complete nitrogen removal through improved denitrification was achieved dosing acetic acid (CH_3COOH), which was implemented in the last four weeks of operation. Accordingly, TN elimination rate during this phase reached 88% with an average permeate concentration of 49.0 ± 3.0 mg/L.

Phosphorus removal efficiency

Because the pilot plant operated without chemical precipitation, phosphorus elimination mechanisms were mainly bioassimilation. TP permeate concentration were 29.2 ± 2.7 mg/L in average, corresponding to an elimination rate of 0% (results not shown). Therefore total TP removal efficiency was negligible. It is assumed, that precipitation of cellular phosphorus, during endogenous respiration is responsible for this observation. But further examinations will have to support this presumption.

Removal of conductivity

Besides enormous loads of carbon, nitrogen and phosphorus, blackwater contains quite a number of other nutrient salts. Most of these soluble salts are sodium compounds such as sodium chloride and sodium nitrate (Carden et al. 2001), which originate mainly from urine (Udert et al. 2003). Accordingly, the concentration of soluble salts has to be considered within blackwater treatment.

The EC of the feed water was 2.6 ± 0.2 mS/cm on average. During the phase with enhanced nitrification through dosage of NaOH, the EC in permeate was 1.7 ± 0.3 mS/cm, which corresponded to a removal rate of 33%. In the last for weeks of investigations, where also CH_3OOH was dosed, the removal of EC increased up to 58% (1.1 ± 0.04 mS/cm). This can be explained by the improvement of the denitrification process. These results indicate that a complete nutrient removal is highly influencing the saline conditions in MBR and therefore the long term performance of biological treatment process of blackwater.

Microbiological removal efficiency

For the microbiological analysis parameters representing fecal indicators and potential pathogens were chosen. Both, *E. coli* and fecal streptococci entered the MBR at concentrations higher than 10^6 cfu/100 mL. This was expected as the blackwater contains most of the fecal material. The MBR effluent was free of any fecal indicator. Within the analysed volume of 100 mL no fecal bacteria remained detectable. Therefore, a removal efficiency of 100% can be stated (Table 2).

Same as for the fecal bacterial indicator, somatic coliphages could not be detected in 50 mL after the MBR. As the median in the influent was 1,600 pfu/100 ml at least a reduction of three log10 for phages can be achieved with the MBR. A breakthrough of viruses was not detected during the operation of the plant, but due to the short operation period any statements about long term performance efficacy cannot be given. It is known from the literature that the membranes of the MBR might develop microcracks where viruses could pass (Hu et al. 2003).

Pseudomonas aeruginosa was taken as an example for bacteria which are able to form biofilms, being highly

Table 2 | Removal efficacy of the MBR with respect to fecal indicator bacteria and *Ps. aeruginosa*

Parameter		Influent	Permeate
E. coli (n = 12)	cfu/100 mL	1.70E + 06	0
	Removal [%]	–	100
Fecal streptococci (n = 12)	cfu/100 mL	1.63E + 06	0
	Removal [%]	–	100
Ps. aeruginosa (n = 12)	cfu/100 mL	9.54E + 04	2.32E + 03
	Removal [%]	–	97.57
Coliphages (n = 12)	pfu/100 mL	1.61E + 03	0
	Removal [%]	–	100

resistant to disinfection methods and very modest. During the operation of the treatment plant it was always possible to detect *Ps. aeruginosa* after in the MBR effluent. The concentrations ranged between 32 and 64,000 cfu/100 mL. As *Ps. aeruginosa* were detected from the beginning on, it can be assumed, that a contamination of the treatment system occurred at a very early stage. The calculated reduction that could be achieved in the MBR was 97.57%. Nevertheless, it is not possible to exclude that the *Ps. aeruginosa* in the permeate resulted from persisting biofilms in the pipe system of the effluent and therefore the reduction efficacy might have been higher for this bacteria, too.

Biomass evolution and aerobic sludge stabilization

Also TSS- and VSS-concentrations were monitored during operation (Figure 3(a)). The MBR was started with an initial TSS-concentration of 2.5 g/L (data not shown). The concentrations increased gradually and after 5 month operation TSS reached a value of about 15 g/L. Hence, the net growth rate of the biomass was very low with 0.019 d^{-1} on average. The low growth rate was caused by the low organic sludge load. Consequently, the microorganisms were permanently starving in the range of endogenous respiration. Because no excess sludge was taken out of the system, except for sampling, the SRT during this period was >50 d. As a result foaming was monitored and negligible sludge loss was observed. In the following TSS concentrations in MBR was regulated to constant levels between 10–12.5 g/L by regular excess sludge extraction. Consequently, the SRT decreased on values of about 50 d. The resulting organic sludge load ranged between 0.010–0.025 g BOD_5/(g TSSd) and foaming was reduced.

Despite of the low sludge loading rates and the high SRT, the organic fraction in activated sludge were continuously quite high. Measured VSS was constant >80% of TSS therefore characterising an unstabilized sludge. A slight increase of VSS was observed during the period without excess sludge removal and a slight increase during the period with excess sludge removal. Endogenous respiration of the activated sludge rates were determined in the range of 30.7 to 46.1 g O_2/(kg TSSd). These values characterized a completely stabilized activated sludge. Accordingly, the VSS in biological reactor resulted rather from the accumulation of particulate, organic material (e.g. cellulose) than from a lack of aerobic sludge stabilization.

Reuse potential of biological treated blackwater for toilet flushing

It was intended to reuse the treated blackwater for toilet flushing (complete recycle). With respect to this application, different water quality requirements exist worldwide (FBR 2005, EPA 2004 etc.). Due to the observed *Ps. aeruginosa* concentrations and the intensive yellowish coloration, the obtained permeate quality did not meet the limits stated by the German FBR (2005), while other prescriptive limits (bacteriological and chemical) are fulfilled as well as the prescriptive limits given in EPA (2004). Consequently, the treatment process has to be expanded by advanced treatment methods in order to meet non restricted water reuse standards.

Gayh (2007) investigated the application of ozone (O_3) and hydrogen peroxide (H_2O_2) and provided recommendations on necessary steps for advanced blackwater treatment especially on the aspects of decoloration of treated blackwater. Lindner (2008) used UV-C radiation for his pilot plant examinations but also investigated activated carbon adsorption, ozonation and UV-A radiation combined with titanium dioxide (TiO_2) and H_2O_2 in laboratory experiments.

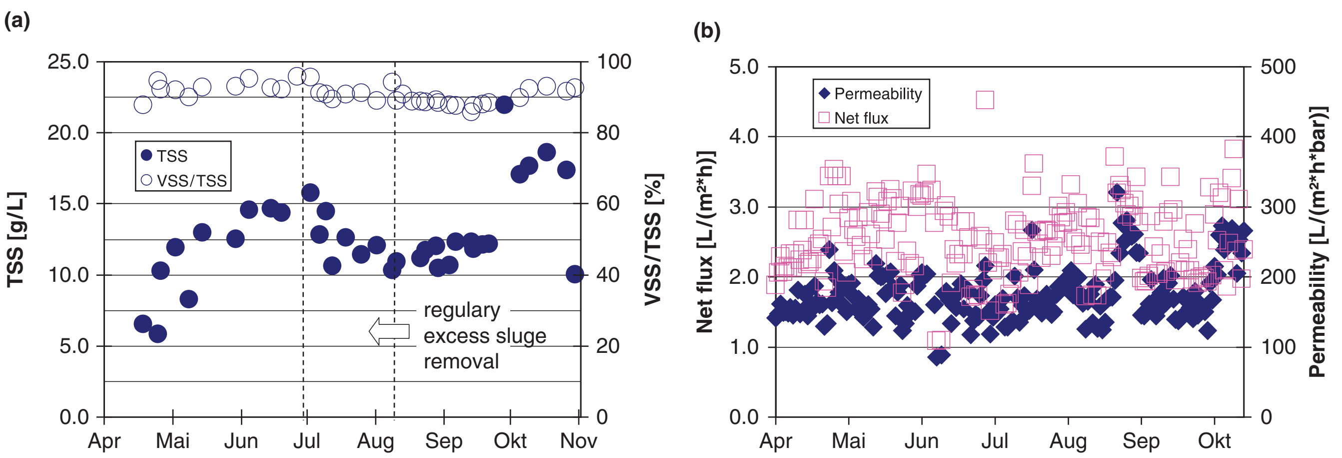

Figure 3 | Development of TSS and VSS (a) and permeability and flux (b) over operation time.

Knerr *et al.* (2009) applied a combination of O_3 and UV-C radiation. Investigations of laboratory and field experiments on different advanced treatment methods (activated carbon, O_3, H_2O_2 and chlorine) for waste- and blackwater are published by Böhler *et al.* (2007) and Abegglen *et al.* (2009).

The results of the latter investigations showed a good decoloration by using powdered and granulated activated carbon. But, due to the high nutrient and organic content in MBR effluent, biofilms in the permeate mains were observed (Abegglen *et al.* 2009). However, the dosage of activated carbon seems to be a suitable option for decoloration and sorption of dissolved carbon, especially in single house applications. Efficient decoloration and disinfection along with advanced chemical oxidation of organic components can be achieved by using O_3, H_2O_2 and UV-C radiation or a combination of these treatment steps. An additional effect is the degradation of trace organics. Also the elimination of pharmaceutical residues can be expected by application of O_3, H_2O_2 and UV-C radiation as advanced technologies for blackwater treatment. But, because of the technical limits of these technologies, application will be limited on larger applications, mainly.

Furthermore, in complete blackwater cycles the accumulation of non-degraded substances and their effects on the recycling-system have to be considered, to evaluate the reuse potential of biological treated blackwater. Firstly, the non-biodegradable organic components found in blackwater, which are expected to be mainly humins, will accumulate in the system. This may lead to a further increase of coloration due to the decomposition of these substances (Böhler *et al.* (2007)), followed maybe by double-flushing of the toilets and therefore increasing treatment costs (Abegglen *et al.* 2009). Secondly, the incomplete nutrient elimination will result in an accumulation of nitrate-nitrogen, phosphate and salts in the system until equilibrium with the influent is reached. While inhibition of bacteria by nitrate is usually described as negligible (Udert *et al.* 2003), the increasing salinity may impair the activity of microorganisms and cause precipitations in the system. Although Antholz *et al.* (2009) showed that the biological treatment process can be conducted at such conditions the integration of a complete biological elimination of nutrients has to be examined based on the boundary conditions (blackwater composition, dilution with other stream etc.).

Membrane performance

During stable operation times the permeate flow ranged between 10.9–45.2 L/h regulated depending on the fluid level in MBR and the inflow circumstances. Average permeate flow was about 25 L/h. Therefore, in the average the net flux was only 2.5 L/(mh) with a range of 1.1–4.5 L/(m h). Transmembrane pressure (TMP) was on average - 9.0 mbar, which led to an average permeability of 268 L/(m hbar) standardized at a temperature of 10°C and constant satisfactory filtration results (Figure 3b).

The found flux values are clearly below reported values in the literature for critical flux (e.g. McAdam *et al.* 2005; LeClech *et al.* 2003). The advantage of operating the membrane far below the critical flux is that the filtration process was stable over a period of 10 month. Influence of permeability caused by fouling through the low flux was not observed. Backwashing of the membrane or chemical cleaning outside the reactor was not necessary.

CONCLUSIONS

The results of the study presented show that MBRs are applicable for blackwater treatment and satisfactory results in terms of water quality, process stability and membrane performance can be achieved. However, because of the blackwater matrix the biological process requires operational optimization. Caused by high nitrogen influent concentration the loss of alkalinity in the MBR must be compensated to achieve high and steady nitrification rates. Due to the low BOD_5:TKN ratio, denitrification can not be achieved in the biological process. As a result total nitrogen content and soluble salts in the permeate remained quite high. The low load rate resulted assumedly in precipitation of cellular phosphorus through bacteria. Furthermore, the investigated blackwater contains high concentrations of inert COD. As a consequence, in complete blackwater cycle (reuse for toilet flushing) the non-degradable components will accumulate in the system, which may result in high salinity restricting the biological treatment processes and efficiency. To achieve advanced desalination in biological blackwater treatment, a dosage of external organic carbon was integrated to enhance denitrification. As a further advantage, advanced phosphorus removal through heterotrophic bacteria can be achieved by this and consequently alkalinity compensation can be minimized. Permeate was free of suspended solids, clear, meeting hygienic guideline values for the fecal indicators *E. coli* and fecal streptococci, but still had an intensive coloration similar to urine. Therefore the treatment process requires to be upgraded by further processing units, e.g. by ozonation, to minimize aesthetic problems with reclaimed backwater.

Due to the low flux, backwashing of the membrane and chemical cleaning outside the reactor was not necessary throughout the study and no operational problems appeared. However, the process is a relatively new application for MBR technology. Thus, additional long-term investigations and detailed monitoring are necessary. Furthermore micro pollutants (e.g. endocrine distributors) have to be observed. Nevertheless, reuse of blackwater can enhance local water availability, especially in semi arid or arid regions and the reuse of blackwater can reduce the overall urban water consumption significantly.

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