



Fouling minimised reclamation of secondary effluents with reverse osmosis (ReSeRO)

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ABSTRACT

A group of researchers from Israel and Germany investigates fouling minimized reclamation of secondary effluent of municipal waste waters for unrestricted irrigation. Countries with increasing water scarcity including Israel are forced to use secondary or tertiary effluents for irrigation. The quality of the effluents can be increased to the levels of drinking water with multistage membrane process where low pressure membranes, mostly ultrafiltration (UF) are used as a pretreatment to reverse osmosis (RO). The minimisation of the organic and biofouling on UF and RO membranes is achieved by different pretreatments including biofiltration, coagulation and adsorption on powdered activated carbon. The pretreatment options are considered separately and in various combinations. The reduced fouling will also be achieved by modification of the membrane surface by the manufacturer. This study incorporates field and lab experiments, the latter are performed for a better understanding of biofouling-induced increased scaling in RO membranes. In order to enable the study of RO foulants on the membrane sheets, RO test-cells were developed for detailed analysis of fouled RO membranes by autopsy. The relevance of the lab-observed trends to the treatment of the secondary effluents of Sede Teiman wastewater treatment plant will be verified. Studies of flow conditions and the influence of flow conditions on transport and fouling layer formation are performed using computational fluid dynamics (CFD). Further modelling will be done on the biological elimination of organic substrates within biofilters.

Keywords: Biofiltration; Coagulation; Desalination; Fouling; Membrane processes; Reverse osmosis; Scaling; Ultrafiltration

1. Introduction

The research is a joint effort of Israeli and German partners, and includes field experiments at Sede Teiman wastewater treatment plant (near Beer Sheva), theoretical

modelling, and lab analysis. The experimental part is based on the knowledge accumulated in the Unit of Environmental Engineering at BGU, Water Supply Group at TU Dresden and the membrane manufacturer Inge AG. Studies of scaling and extracellular polymeric substances (EPS) formation is performed with various analytical tools including Langmuir isotherms, ATR-FTIR,

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quartz crystal microbalance with dissipation compensation (QCM-D), powder X-ray diffraction, HR-SEM, transmission electron microscopy (TEM), grazing incidence small angle neutron scattering (GISANS), small angle neutron scattering (SANS), and grazing incidence X-ray diffraction (GIXD) using synchrotron source in direct cooperation between the Departments of Biotechnology Engineering and the Zuckerberg Institute for Water Research at BGU and the Institut fuer Festkoerperforschung at the Forschungszentrum Juelich. However, this paper is more focused on the general field experimental setup and modelling.

Existing water resources in Israel are extensively used or some may even say overused. Facing pressing water scarcity, in April 2008 the Israel ministry of infrastructure developed a program aimed at using reliable water resources such as desalinated water for potable use and tertiary treated wastewater for irrigation. The Israeli water authority is looking into a possibility to perform a decentralized desalination of secondary effluents from municipal wastewater plants using Multiple Integrated Membrane System (MIMS). The MIMS consists of UF membrane pretreatment followed by low fouling or energy saving RO membranes. With low concentration of dissolved solids, the cost for desalination of secondary effluents is estimated as one third of the cost for seawater desalination (18.9 vs. 48 cents m^{-3} [1]). However, with high organic and biological loads, both UF and RO membranes are expected to experience high degrees of fouling that might disturb normal operation.

In order to minimise fouling problems a closer understanding of fouling mechanisms is needed. As fouling mechanisms are better understood, this knowledge can be used to minimise fouling, for example, by an adapted pretreatment step. Thus, a combination of applied research and fundamental investigations are performed in close cooperation within this project.

Coagulation, biofiltration, adsorption on powdered activated carbon and UF membrane are the investigated pretreatment options that help to reduce the RO fouling. The most popular view on UF fouling is that it is the undesirable process which reduces permeability, increases operational costs and requires chemical cleaning. The cleaning might eventually result in partial loss of integrity in UF membranes [2–4]. Here, however an increased UF fouling might be necessary to prevent RO fouling. Sacrificing more stable UF membrane that can be cleaned chemically, we might be able to actually improve the daily operation of the entire MIMS system. Further pretreatment, for example, higher coagulant and PAC dosages or membranes with lower molecular weight cut-off (MWCO), will result in higher UF fouling and therefore operational costs but might reduce RO fouling significantly. Frequent UF cleaning including

chemicals, will be efficient in restoring UF flux but might negatively affect RO performance too. Varying UF characteristics will affect the degree of UF fouling, and might affect RO fouling as well. Biofiltration will reduce carbon sources to reduce biofouling, and might at the same time reduce some other organic matter that is responsible for organic RO fouling as well. The current study will answer questions on how RO performance will be influenced by:

- The degree of UF pretreatment, that is, varying coagulant and PAC, biofiltration prior UF
- Biofiltration as stand alone pretreatment
- UF characteristics, that is, varying hydrophilicity, pore size, capillary geometry (diameter and length)

2. Analysis

Analysis of the filtrates, RO permeate, the membrane characteristics including fouling layer and fouled membrane surface. Generally, the quality of biofiltration filtrate, UF and RO effluents will be compared by total organic carbon (TOC), UV absorption at 254 nm (UV254), attenuated total reflection–Fourier transformed infrared spectroscopy (ATR-FTIR), gas and liquid chromatography–mass spectrometry (GC/LC-MS) and liquid chromatography–organic carbon detection–organic nitrogen detection (LC-OCD-OND). UF membrane characterization will be performed with pure water flux measurements, transmembrane streaming potential, pore size and pore size distribution determination, and tensile strength at break. In order to enable the study of RO foulants on the membrane sheets, RO test-cells will be developed which can host flat sheet RO membrane with surfaces big enough to be directly operated downstream of the pilot scale UF-units and to supply sufficient surface for detailed analysis of fouled RO membranes in the lab. Fouled RO membrane analysis will be performed with contact angle measurements, ATR-FTIR, atomic force microscopy (AFM), and scanning electron microscopy (SEM). Studies of flow conditions and layer formation will be performed using CFD. Further modelling will be done on the biological elimination of organic substrates within the biofilters.

Analysis of the biofilms. A problem encountered in operating RO and nanofiltration (NF) on secondary/tertiary and other effluents is fouling of the membrane by sparingly soluble inorganic salts, colloids, and organic macromolecular compounds whether already present in the water or produced by bacteria attached to the membrane surface. As fouling frequently involves more than a single component, the question on how its various components affect each other on the membrane surface becomes highly relevant. It is known that organic molecules, including those of biological origin, can induce

precipitation of sparingly soluble materials, such as hydroxylapatite and silica and control their crystal habit. Accordingly, fouling or scaling will also be investigated on the molecular level, that is, the effect of various components in the biofilm and organic fouling layer on mineralization of calcified and silicate minerals on the membranes surface. At first, defined model systems of solutions representative domestic effluent water will be prepared. The mineralization studies will be monitored in aqueous solutions as well as using thin films compounds representatives of biofouling, as for example, Langmuir films of peptides, proteins, fatty acids and polysaccharides at an air-solution interface or bound to solid surfaces such as gold or silica. The mineralization that will be induced by these surfaces will be monitored closely with respect to precipitation rates, crystalline phases and their morphology at the interfaces. As the research develops, the model surface components will be replaced by a real biofilm extracted EPS. Various analytical tools will be employed including Langmuir isotherms, ATR-FTIR, QCM-D, powder X-ray diffraction, SEM, TEM, GISANS, SANS, and GIXD using synchrotron source. As SANS is a “transmission” technique, the experiments can be performed in aqueous solutions of gold or silica surfaces, coated with the above mentioned proteins etc.

Despite the vast activity aiming at better understanding membrane fouling, not much is known today on the ways biofouling and organic fouling on membranes affect the precipitation of sparingly soluble minerals in the treated water. This is particularly true in the case of wastewater treatment where calcium phosphate minerals tend to precipitate on the membrane surface. The authors are not aware of studies aimed at elucidating, on the molecular level, the mutual interactions between biofouling components and minerals on membranes for water treatment. It is hoped that the research will open a window towards better understanding of biofouling induced mineralization that will enable the development of more efficient ways to tackle the fouling problem in water treatment processes.

3. Scientific and technological background

Major constituents of concern in secondary effluents include pathogens, residual organics, ammonia, phosphorus, particles and certain ions (calcium, magnesium, iron, silica, boron). A conventional tertiary treatment by means of filtration and disinfection will be effective in removal of pathogens, particles, ammonia and phosphorus. To be implemented for industrial needs, the quality should be improved by addition of ion exchangers to remove salts, or by activated carbon to remove organics. Removal of both salts and organics can be achieved by

RO membranes [5]. During the last decade, RO membrane treatment has been approved as the best available technology for the production of high quality recycled water for indirect potable reuse (e.g., the Water Replenishment Project in Orange County, California; Wulpen Aquifer Recharge Project in Belgium; NeWater Project in Singapore).

One of the more persistent, well-known and extensively documented problems associated with RO membranes is the problem of fouling. Fouling can be coped by pretreating the water prior to RO. The problem of fouling has to be resolved before the membrane because no effective membrane cleaning has been found yet, and therefore highly fouled RO membranes will be simply replaced with a higher frequency. In the past, most RO plants used conventional pretreatment, which is defined as chemical and physical pretreatment without the use of membrane technologies. Conventional pretreatment generally uses coagulation/flocculation, settling, sand filtration and a following cartridge filtration as physical pre-treatment. It is usually accompanied by a disinfection stage by means of ozonation, chlorination, and/or UV irradiation, depending on the chosen membrane material, to reduce biofouling impact. Membrane separation processes, such as UF, are emerging technologies that can be used for RO pretreatment instead of the filtration and disinfection stages described above. Recent studies performed by Mekorot, Israel national water company, with Norit/X-Flow membranes showed the potential of the inside-out pressure driven hollow fibres with a stable flux of $90 \text{ l m}^{-2} \text{ h}^{-1}$ and high recovery rates of more than 90%. Mekorot applied UF pretreatment with FeCl_3 . Two main bottlenecks in application of UF pretreatment are the low retention of organic contents and of nutrients and irreversible fouling if applied as stand alone treatment process. The problem of fouling demands frequent cleaning operations that result in gradual aging and eventual loss of integrity of the UF membranes [3,4,6]. To address the fouling problem, a pretreatment stage (this time to UF) is therefore necessary.

A number of studies have shown that coagulation/flocculation followed by adsorption on PAC is one of the most effective pretreatments for UF membrane filtration. Using turbidity as a measure of organic content removal, Abdessemed and Nezzal [7] found that introduction of CaCl_2 to the effluent followed by adsorption on PAC reduced chemical oxygen demand (COD) in secondary effluents of domestic wastewater by 80%. This pretreatment step was followed by UF through Carbosep tubular inorganic membranes with MWCO values of 10 and 15 kDa. In an earlier study by the same group [8], 86% removal of COD was obtained by application of $40 \text{ mg l}^{-1} \text{ FeCl}_3$ and $20 \text{ mg l}^{-1} \text{ PAC}$. In that study, cellulose acetate membranes with a MWCO of 50 kDa

were used. In other studies [9–12], it was found that introduction of $120 \text{ mg l}^{-1} \text{ FeCl}_3$ followed by adsorption with $1 \text{ g l}^{-1} \text{ PAC}$ removed 91% of dissolved organic carbon (DOC) from a biologically treated wastewater effluent. In those studies, polysulfone membranes with a 17.5 kDa MWCO were used. A slightly lower organic matter removal of 88% for treatment of synthetic wastewater was obtained when the FeCl_3 dose was reduced to 50 mg l^{-1} and PAC concentration was reduced from 1 to 0.5 g l^{-1} [13]. In general, apart from the reports of the group of Abdessemed [7,8], there is agreement as to the dose ranges of FeCl_3 ($50\text{--}150 \text{ mg l}^{-1}$) and PAC ($0.5\text{--}1 \text{ g l}^{-1}$) that should be applied to obtain a successful reduction of organic content. However, there does not appear to be general consensus about the optimal membrane characteristics; membranes of virtually every polymer material, cellulose acetate, polyethylene, polysulfone, polyethersulphone and polyvinylidene difluoride, with MWCOs ranging from 4 to 500 kDa have been used [7–17]. In addition, majority of the studies were performed in lab conditions thus not taking into account effects of prolonged operation.

More than that, the studies did not consider biofiltration that might be an additional pretreatment step to reduce organic substrate and therefore prevent following membrane processes from biofouling [18,19]. As shown by Uhl, 2007, biofiltration can be used to achieve lower concentrations of bacteria, TOC and BDOC and resulted in a profoundly decreased biofilm formation on glass slides compared to rapid media filtration. Used as a pretreatment for NF, it was shown that the transmembrane pressure increase was in average 5-fold lower for the nanofilter with biofiltration as pretreatment (NF-Bio) compared to the nanofilter with rapid media filtration as pre-treatment (NF-RMF). The results of this study indicate that biofiltration, as a simple pretreatment option, can significantly mitigate fouling problems in low flux NF installations for NOM removal from surface water. Above those drawbacks, UF pretreatment was studied as a function of UF fouling, not related to RO performance.

In general, the main UF foulants can be subdivided into proteins and polysaccharides, humic substances, low molecular weight acids and low molecular weight neutrals. Although it is assumed that the main foulants are the first two groups of biopolymers and humic substances [20], each group contains a wide variety of compounds that can be hydrophilic or hydrophobic, charged or neutral, with supercoiled and hare-like shapes, and with different molecular weights. Increased removal of organic compound in front or directly at the UF stage might have a profound effect on fouling at RO membrane level, and therefore more expensive UF pretreatment might significantly influence functioning of RO

membranes. It is therefore worth to perform more intensive pretreatment to prolong RO membrane lifetime.

4. Objectives and preliminary results

For the minimisation of fouling, different aspects have to be taken into account, which also relate to different fouling mechanisms. Organic fouling is mainly caused by the deposition of high molecular weight compounds. Colloidal fouling is caused by particles in the sub-micrometer size range. Biofouling is caused by microorganisms mineralising organic substrates, that is, biodegradable organic substances. Thus, in order to minimise fouling, strategies to remove high molecular weight compounds, to remove submicrometer particles and to remove biodegradable organic substances need to be followed. Consequently, treatment options to be considered are biofiltration for the removal of high molecular weight and biodegradable organics, coagulation/flocculation for removal of particles and organics, PAC for removal of organics and UF for removal of particles and high molecular weight organics. Of course, combinations of these treatment processes might be ideal, as each process has its strengths and weaknesses with regard to the removal of the different target constituents.

In the experimental investigations to be carried out at Sede Teiman wastewater treatment plant (near Beer Sheva), the three different pretreatment options will be investigated as sole pretreatment as well as in combination in order to minimise fouling. Consequently, in the pilot plant operated on site of the wastewater treatment plant, three treatment trains will be applied, all of them with an RO-membrane which can be analyzed closely for foulants, downstream.

The three different treatment trains comprise:

1. biofiltration – RO
2. coagulation – biofiltration – UF – RO
3. coagulation – UF – RO

Furthermore, the option is given to combine the biofiltration from train one and train three to give

4. coagulation – biofiltration – UF – RO

Or to combine biofiltration from train one with train three while switching off coagulation to give

5. biofiltration – UF – RO

These five combinations will enable the researchers to compare differently modified UF-membranes for their pretreatment effect on RO-fouling. If two different membranes are to be compared, this can be done in two—besides the membrane types—similar trains like the combination of

- trains 2 and 4
- trains 2 (w/o biofiltration) and 5

From the experimental results regarding

- fouling behaviour of UF- and RO-membranes
- removal of organic substances of different groups (like polysaccharides/biopolymers, humics, low molecular weight substances) in different treatment stages
- removal of biodegradable organic substances in different treatment stages
- removal of particles of different sizes in different treatment stages
- proof of biomineralisation in the presence of metals like calcium
- precipitation of salts (scaling)

Conclusions can be drawn for the optimisation of pretreatment to RO, thus potentially lowering the cost for the entire application. There is a pronounced need for such investigations which will deliver scientifically proven understanding on the mechanisms in the pretreatment of RO feed, as this knowledge is then to be applied for targeted optimisation of the entire process.

In water treatment processes usually spiral wound cartridges of one meter length are used and assembled as 6 cartridges in a row into one pressure tube. It is known that over the length of each pressure vessel and in the cartridge a steady decrease of the feed volume stream and increase of dissolved substances and salinity can be found. This change also leads to a variation of the filtration conditions (flow velocity, concentration polarisation, osmotic pressure) [21]. This will significantly influence the fouling and scaling behaviour along the membrane.

In order to focus on the variation of the filtration conditions the set up of the RO unit in this project consist of long channel test cells. Six of them are set in a row to simulate a pressure vessel consisting of six cartridges. The length of the channel is 1 m with a membrane area of 0.91×0.04 m. The channel height is 0.8 mm and includes a feed spacer, simulating the flow conditions in spiral wound modules available on the market. Five permeate outlets are constructed on each bottom part of the cell by dividing the permeate bed into 5 sections (each lowered, separated and equipped with a permeate spacer) to systematically analyse the rejection and flow conditions along the membrane channel. Fig. 1 exposes the outlets along the cell.

It is also possible to take samples of the feed before each RO-channel. The membrane cell is equipped with X20 RO-membranes (Polysep, NL). The membrane, facing the top of the module with the active membrane area, is gripped between top and bottom of the module. The test cells consist of acrylic glass with a stainless steel inlet and a steel grid on top and bottom of the channel. The grid was added to prevent a bending of the channel by high pressure forces and assure the lasting of the acrylic glass beyond the screws due to high surface pressure.

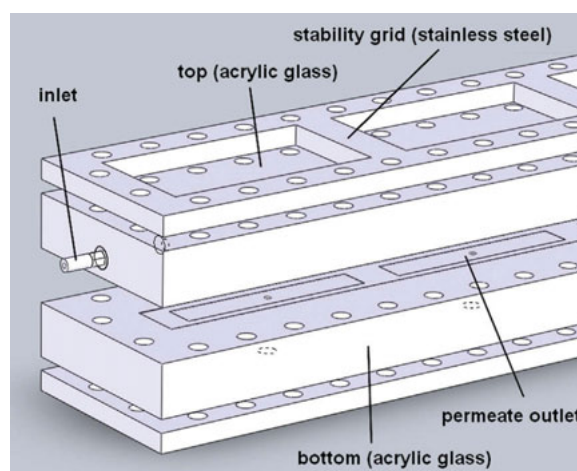


Fig. 1. Computer model of the long channel test cell with visible outlets.

Acrylic glass was chosen to enable visualizing of the flow conditions along the channel. For minimizing the impacts of the inlet stream a broad inlet was constructed. The manufactured test cell is shown on Fig. 2.

Using the developed test-cell, conclusions can be drawn for the optimisation of pretreatment to RO, thus potentially lowering the cost for the entire application. There is a pronounced need for such investigations delivering scientifically proven understanding on the mechanisms of pretreatment of RO feed. This knowledge will then be applied for targeted optimisation of the entire process. In order to start our investigations at the waste water treatment side at Sede Teiman, Israel, on the pretreatment part as described above in train 3 an UF test unit, equipped with a multibore dizzer P module of the company inge AG, Germany, was installed and brought into operation. Following preliminary results are exemplarily shown.

The optimal coagulant concentration was determined with jar tests. Ferric chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) in concentration range of $0\text{--}40 \text{ mg l}^{-1}$ was pored into

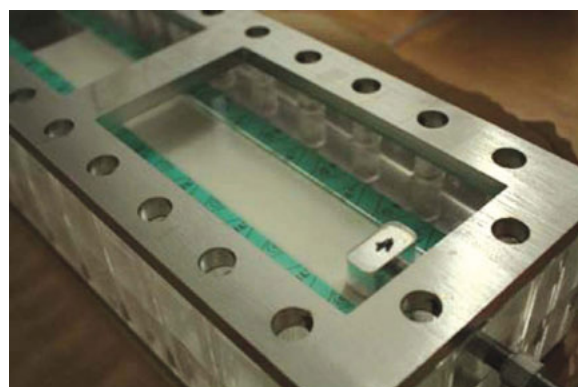


Fig. 2. The manufactured test cell.

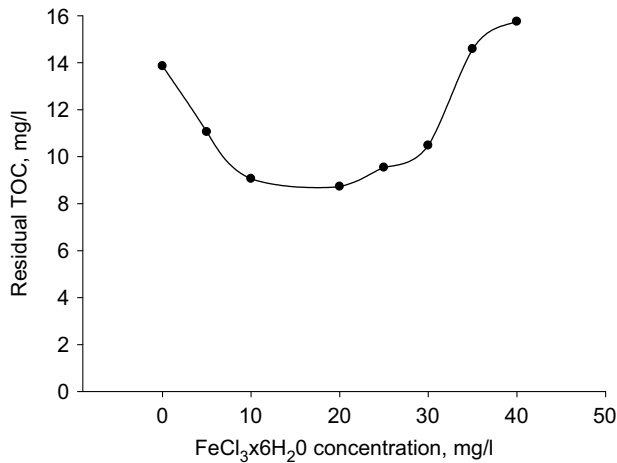


Fig. 3. Retention of organics from secondary effluents of Sede Teiman wastewater treatment plant by coagulation and sedimentation, as a function of the coagulant concentration.

6 well stirred 800 ml glass jars in a conventional multiple stirrer (PHIPPS & BIRD STIRRER, 7790-402). The slurry was (i) mixed rapidly at 100 rpm for 1 min; (ii) mixed slowly at 30 rpm for 20 min; and (iii) allowed to settle quiescently for 30 min. Samples collected by slow decantation from the upper part of the test jars were analysed for conductivity (Cyberscan con 10, Eutech Instruments, Singapore), pH (PHM210, Radiometer Analytical SAS, France), total suspended solids (WTB Binder, Class 2ED 115/E2 #00-17337, Binder Inc., USA)

and DOC (Apollo 9000 TOC analyzer, Tekmar Company, Cincinnati, OH). The results for residual TOC are present on Fig. 3.

The lowest 8 mg l^{-1} DOC level was measured at 10 and 20 mg l^{-1} $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ concentrations. The obtained concentration is much lower than reported before [22], and can be considered low enough for the daily operations of the treatment plant.

A small pilot UF plant is set into operation at Sede Teiman wastewater treatment plant to investigate the UF pretreatment performance as discussed above. The UF experiments on the secondary effluents were performed with standard dizzer P module with 0.9 mm Multibore membranes (inge AG, Greifenberg, Germany). The results of transmembrane pressure (TMP), flux and normalised permeability are presented on Fig. 4.

The presented data is for the runs with a constant flux of $60 \text{ l (m}^2 \text{ h)}^{-1}$. The filtration runs were 30 min long, and during that time the TMP increased from 0.4 to 0.5 bar. The coagulation was performed with 5 mg l^{-1} Fe^{3+} (24 mg l^{-1} $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$). The hydraulic backwash was sufficient to maintain the permeability on the level of $150 \text{ l (m}^2 \text{ h bar)}^{-1}$. To increase the permeability to the initial levels the membrane rack was washed once every 12 h with a combination of H_2SO_4 and NaOH . The chemical cleaning successfully restored the flux to the initial levels of $250 \text{ l (m}^2 \text{ h bar)}^{-1}$, but dropped afterwards to the level of $150 \text{ l (m}^2 \text{ h bar)}^{-1}$ again. However, the combination of hydraulic and chemical cleaning options was sufficient to run the UF membrane system with a good stable flux.

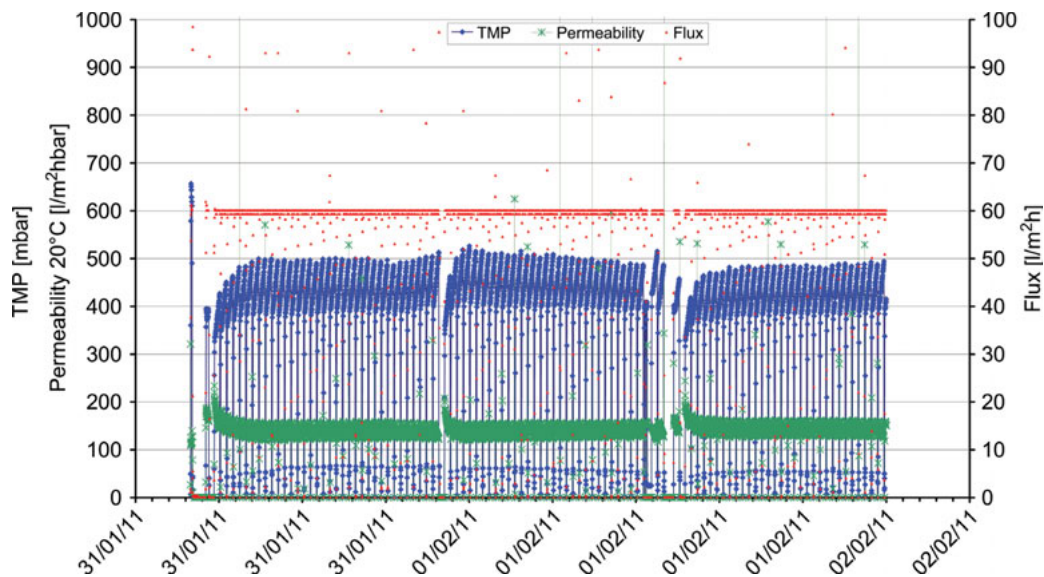


Fig. 4. Flux, permeability and TMP in UF treatment of the secondary effluents of Sede Teiman wastewater treatment plant.

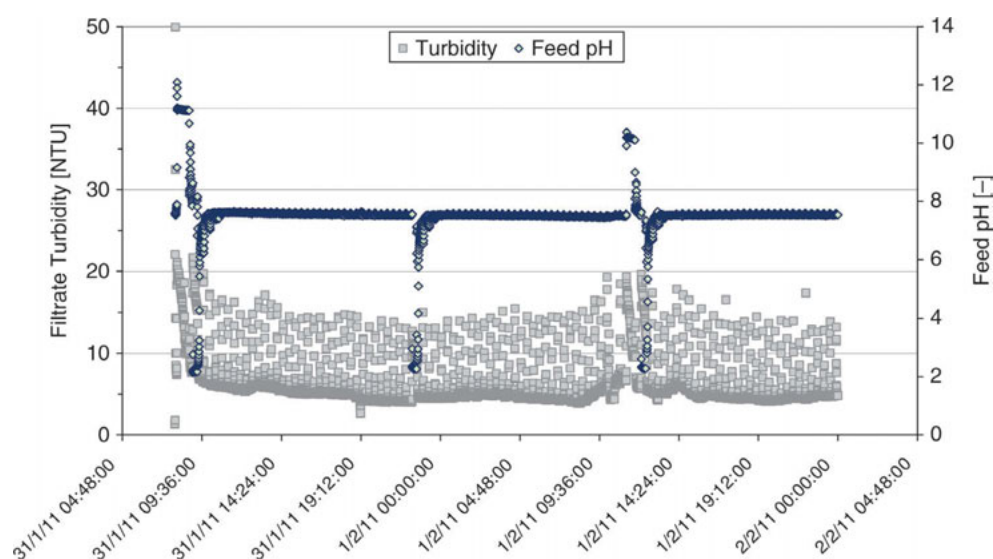


Fig. 5. The turbidity of the permeate after UF filtration of the secondary effluents of Sede Teiman wastewater treatment plant with dizzer P membrane modules (inge AG, Greifenberg, Germany).

The quality of the UF permeate was evaluated with the turbidity measurements. The results are depicted on Fig. 5.

An initial turbidity level of 50 NTU was detected. The measured turbidity levels in UF permeate were surprisingly high and showed the insufficient levels of turbidity removal. However the major source of turbidity in the secondary effluents is the colour from both, the secondary effluents and ferric ions in water, and therefore the turbidity in that case may not be the representative parameter for assessment of the permeate quality.

5. Modelling

The degree of membrane fouling will be assessed by calculations of the flux drop that can be determined as a percentage reduction of clean water flux before, within and after filtration. Calculations of fouling will be accompanied by calculations of the degree of removal expressed as TOC removal ratio. A CFD model will be applied for the description of the complete flow field in the RO test cells and the UF capillaries. The existing model [23] can be used for UF capillaries of arbitrary cylindrical geometry, alignment, that is, vertical or horizontal, and operation condition, that is, cross-flow or dead-end, using the software COMSOL Multiphysics (COMSOL AB, Sweden). For the description of the flow inside the test cells a new model has to be established first based on the work of Lerch [23]. This is done by numerical calculations of the Navier–Stokes and continuity equations first for stationary and second for transient flows, including the effects of dynamic fouling layer formation. Based on

the modelled flow field, particle or floc velocities and trajectories can be derived by balancing the forces and torques acting on the flocs in this flow field. The forces and torques considered arose from sedimentation, shear induced and Brownian diffusion, double layer repulsion and van der Waals attraction, drag by filtration, virtual mass and lateral migration. Floc agglomerates here are considered being ideally spherical, rigid and of constant shape, consisting of primary particles embedded in iron or aluminium hydroxide, revealing an inner porosity, low density and low zeta potential. To account for the inner porosity of the flocs an equivalent hydrodynamic floc radius is used. The effect of the porous aggregates on floc suspension viscosity is considered by a correction factor. However, the model is able to describe both, floc transport and solid particle transport. The derived particle/floc velocities can be used to model stationary floc volume concentration distributions by numerical calculations of the convection and diffusion equation. In a first step, the model can be used to describe initial and stationary floc transport and deposition behaviour.

Additional biological reaction kinetic modelling will be performed at the TU Dresden to describe and predict the biological elimination of organic substrates within the biofilters, using LC-OCD analysis. This will help to answer the question about the origin of membrane biofilms, which is crucial for the development of successful biofouling prevention strategies. If the EPS forming the biofilm on the membranes is mainly synthesized by deposited bacteria, then a possible strategy is the successful removal of substrates which are converted to EPS by bacteria. These may be easily biodegradable low

molecular weight substances or cosubstrates like nitrogen sources (the nitrogen source can impact the biomass yield from organic substrates drastically). In these cases a successful strategy could be the implementation of a bioreactor in front of the RO-system which is sometimes already done with success.

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