M. Verhülsdonk and K. Glas

Comparative Investigation on the Influence of Fouling and Scaling on the Membrane Filtration of Waste Water

Wastewater treatment should be viewed as one element of a holistic system targeted on the sustainable use of water. Recycling and the multiple utilization of not only water but also of the substances contained increasingly become an essential necessity. The application of a modular wastewater recycling was investigated for the treatment of anaerobic wastewater to process water. After the pretreatment, ultrafiltration and reverse osmosis are necessary to meet drinking water quality. The goal of this study is the stable operation of the membrane plants. In order to study the feasibility of the membranes, chemical and technical parameters as well as biofilm detection were observed during operation. During the operation period, biofilm development had no significant effect on the membrane performance. Scaling was the central issue problem. A calculation of the scaling potential showed that phosphate and carbonate salts tend to deposit on the membrane surface of the reverse osmosis. Based on the calculation of the scaling potential, a test was carried out with additional acid dosage to increase the solubility of salts. Within this test, a constant operation of the reverse osmosis without significant reduction in permeability was achieved. After 80 days of successful operation, the membranes were removed and analyzed by SEM/EDX to determine the scaling on the membrane surface.

Descriptors: membranes, biofouling, scaling, wastewater treatment, ultrafiltration, reverse osmosis

1 Introduction

An example of a water management concept developed for a brewery has proven that about 30 % of its fresh water demand can be covered by recycled water in non-sensitive that cannot come in contact with the product [1]. To explore the concept of waste water recycling in the beverage industry, a pilot plant on a semi-industrial scale (1.0–1.5 m³/h) was installed in a brewery to investigate the advanced treatment of the effluent from an anaerobic reactor by means of modular plant components (Fig. 1).

The end-of-pipe solution, where all production effluent water of the brewery is treated, has the advantage of a constant water quantity and quality. The disadvantage is the more complex multi-step treatment. However, it is possible to come up with a treatment concept for a particular brewery, independently of strategy and water supply design, and the process steps developed can be retrofitted into the production process without major changes.

The flotation removes the fluctuating content of settleable solids in the outlet of the anaerobic reactor. The pretreated wastewater is pumped via a hopper into the membrane bioreactor. The membrane bioreactor consists of a denitrification, a nitrification and the membrane basin. The next treatment step provides an ultrafiltration, removing turbidity, colloids and microorganisms. The following reverse osmosis removes dissolved salts and organic compounds thus drinking water quality can be reached.

The aim of the project is the reuse of treated water by means of reliable process technology with a minimum addition of chemicals. The core concept of this research project is its modular principle which allows for best possible adaption to the specific requirements of the beverage industry.

A part of this research project is the examination of the membrane plant performance (ultrafiltration and reverse osmosis) for the purpose of process optimization. In membrane processes, an essential part of operational costs can directly or indirectly be traced back to biofouling/scaling and associated counteractive measures. In order to investigate the influence of biofouling and

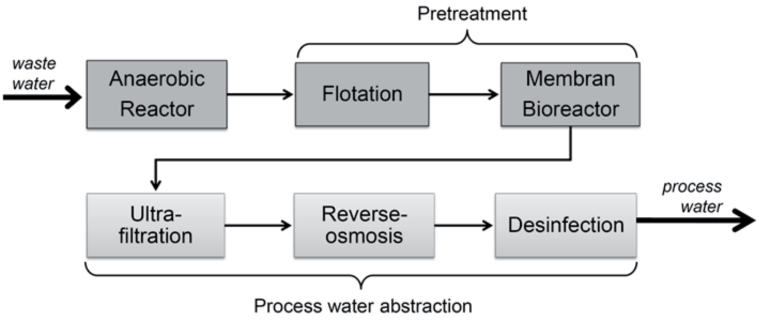

Fig. 1 Process scheme of sustainable water recycles

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scaling on the membrane performance, the following examinations were carried out:

- Detection of the technical parameters as flux, pressure drop, membrane permeability
- Integration of biofilm sensors in the inlet of the membrane elements
- Analysis of the membrane surface by scanning electron microscope (SEM) and Energy-dispersive X-ray spectroscopy (EDX)
- Water analysis of the feed, permeate and concentrate of the reverse osmosis plant

2 Background

2.1 Membrane separation

In the area of water treatment, pressure driven membrane processes are mainly used.

Microfiltration (MF) and ultrafiltration (UF) membranes belong to the group of pore membranes. Here, convective mass transport is dominating. Substances with a greater diameter than pore size will be rejected by the membrane while dissolved substances will permeate.

Nanofiltration (NF) and reverse osmosis membranes (RO) are solution diffusion membranes with a diffusive mass transport through the membrane. Dissolved substances, for which the membrane is non-permeable, will concentrate on the membrane surface. NF and RO are characterized by a different rejection of ions. Monovalent ions such as chloride can pass nanofiltration membranes easily while multivalent ions (phosphate, carbonate) will be rejected. In contrast, reverse osmosis membranes have a salt rejection of more than 90 %.

2.2 Fouling and scaling mechanisms in membrane processes

Fouling and scaling have a direct effect on the performance of membrane plants. Caused by fouling, the membrane blocks and the transmembrane pressure drop increases. As a result, the frequency of cleaning intervals rises and the membrane performance is reduced. Beside the loss of profits, used chemicals cause additional costs. Furthermore, premature aging reduces the membrane's life cycle.

Several forms of fouling, which can influence each other are known [2]:

- Scaling as the deposition of precipitated salts in supersaturated solution;
- colloidal Fouling as the forming of a filter cake by suspended particles;
- organic Fouling as the deposition of organic material;
- biofouling as the accumulation of biomass over a tolerable level by biofilm growth.

Scaling is a typical problem in RO membrane plants, because of the supersaturation of salts during the filtration process. Colloidal fouling can be prevented by using an UF as pretreatment system in front of the RO. But biofouling remains a major concern in UF and RO membrane plant operation. An important factor is the flow velocity inside the membrane. Picioreanu and Radu [2,3] proved that fouling starts in areas with low flow velocity, as behind the spacer geometry.

Scaling

In contrast to biofouling, scaling is the development of a surface layer caused by precipitation of dissolved salts after exceeding the limit of solubility. An acidic cleaning for the removal of the crystalline surface layer is often insufficient because of an enduring decreased permeate flow after cleaning. This is caused by an incomplete removal of the surface layer by acids. Furthermore, it is difficult to flush the crystalline sludge out of the membrane module. For this reason, membrane blocking should be avoided by the removal or stabilization of scaling triggering substances. Scaling can be prevented by scaling inhibitors, acids or electrochemical processes. Scaling inhibitors, also known as antiscalants, decelerate the precipitation of dissolved salt by avoiding the crystallization. The effect of acid dosing is a higher limit of solubility of dissolved salts. If these measures should be insufficient, an additional softening pretreatment can be considered [4, 5]. The following section presents the calculation of the scaling potential of the main salts expected to precipitate.

Calcium phosphate scaling

The scaling potential of calcium phosphate is determined by comparing the ion-product *IP* with the solubility product *SP*. The solubility is the balance between solid calcium phosphate and the saturated solution of calcium phosphate. The calculation of the ion product is shown in equation 1. In a saturated solution the *IP* is equal to *SP*. If IP is larger than *SP*, the salt precipitates.

$$IP = [Ca^{2+}]^3\,[PO_4^{3-}]^2 \qquad (1)$$

Dependent on the pH, different phosphorous phases in water are known (Fig. 2). For strongly alkaline pH, PO_4^{3-} is dominant. At a slightly acidic pH, dihydrogen phosphate H_2PO_4 is present. The solubility is dependent on the temperature and ionic strength of the water. The solubility products in water at 25 °C are outlined in table 1.

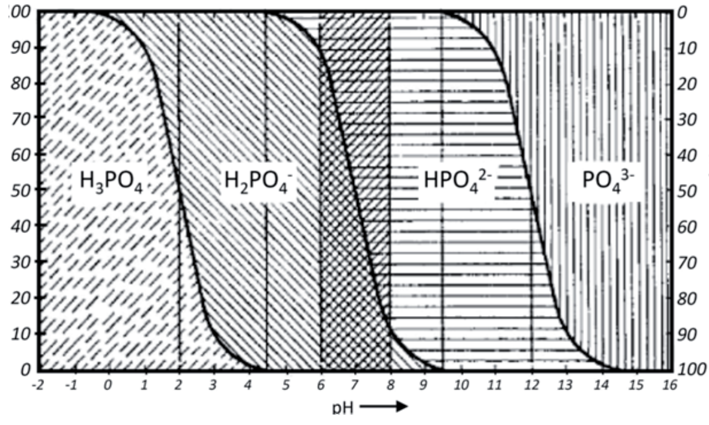

Fig. 2 Phosphate phases depending on the pH [7]

Table 1 **Solubility product *SP* of the different calcium phosphates [6]**

Phosphate phase		Solubility product *SP*
Tricalcium phosphate	$Ca_3(PO_4)_2$	8.46×10^{-32} mol/l
Dicalcium phosphate	$CaHPO_4$	1.83×10^{-7} mol/l
Monocalcium phosphate	$Ca(H_2PO_4)_2$	soluble

Calcium carbonate scaling

In order to avoid calcium carbonate scaling, the pH of the concentrate stream in an RO system should be lower than the pH of saturation (pH_s) where the water of the concentrate stream is in equilibrium with $CaCO_3$ [8]. Well known calculation methods to predict the scaling potential of calcium carbonate are the Langelier Saturation Index (LSI) for brackish waters with a TDS<10.000 mg/l [9] and the Stiff & Davis Saturation Index (S&DSI) for sea waters [10].

The LSI is determined as the difference between the water pH and the saturation pH_s (Eq. 2). A positive value is an indicator for scaling, a negative value indicates that calcium carbonate is dissolved in water.

$$LSI = pH - pH_s \qquad (2)$$

Calcium sulfate, barium sulfate, strontium sulfate scaling

The scaling potential of calcium, barium and strontium sulfate is calculated by comparison of the *IP* with the solubility product K_L (e.g. $CaSO_4$ in Eq. 3). The solubility products (Eq. 6–8) in the water are a function of the ion strength *U* pending on the molar ion concentration M_{li} [mol/1000 g], the concentration of the Total dissolved solids (TDS) and the molar concentration *M* of the ion *i* [mol/l] (Eq. 4–5). The TDS concentration is the sum of the concentration of all ions in water [11].

$$IP_{(CaSO_4)} = \mathrm{M_{Ca}} \cdot \mathrm{M_{SO_4}} \qquad (3)$$

$$U = 0.5 \sum \left(M_{li}\, Z_i^2\right) \qquad (4)$$

$$M_{li} = \frac{10^6}{10^6 - TDS} \cdot M_i \qquad (5)$$

$$K_{L(CaSO_4)} = 0.0016 \cdot U^{\,0.6742} \qquad (6)$$

$$K_{L(SrSO_4)} = 0.00001 \cdot U^{\,0.6916} \qquad (7)$$

$$K_{L(BaSO_4)} = 7.0 \cdot 10^{-9} \cdot U^{\,0.835} \qquad (8)$$

3 Material and Methods

Technical structure of the pilot plant

Figure 3 shows the process scheme of the UF/RO. The used hollow fiber membrane in the UF (Hyflux K-600 ETN 0812) has a molecular weight cut off (MWCO) of 120 kDa. The plant is operating in „dead-end“ mode. For the removal of particles on the membrane surface, a combined air-water backwash is carried out every 30 minutes. Any necessary chemical cleaning is done via the mobile CIP tank. The produced filtrate is stored in a second hopper. The RO plant is supplied from the hopper. A level-regulated booster-pump transports the water to the high-pressure pumps. The installation consists of two parallel modules. NF and RO membranes can be tested with the plant. Prior to the modules, antiscalants can be dosed. To compare different stabilisation methods, the chemicals can also be dosed separately in front of each single module. A partial flow of the concentrate is recirculated to guarantee the essential flow over the membrane surface. The membrane performance is regulated (permeate flow, recovery) via three manual valves per module at the feed and concentrate side.

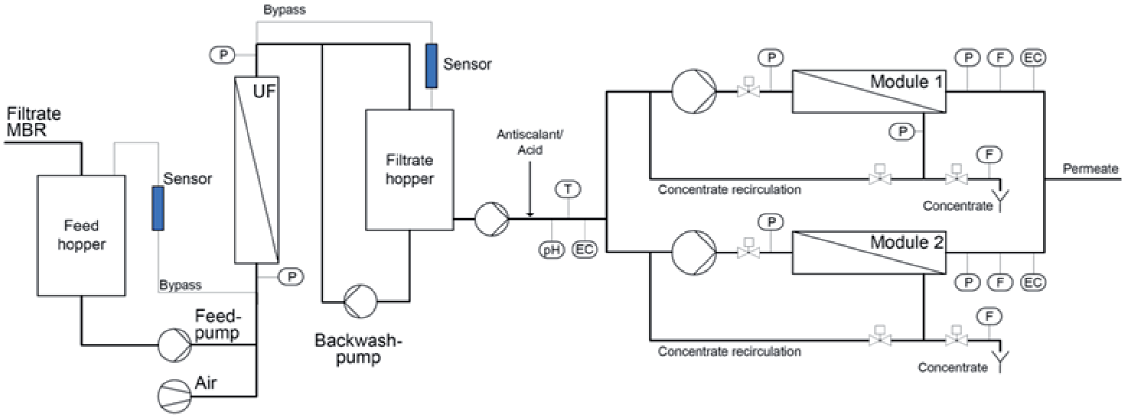

Fig. 3 **UF and RO plant process scheme**

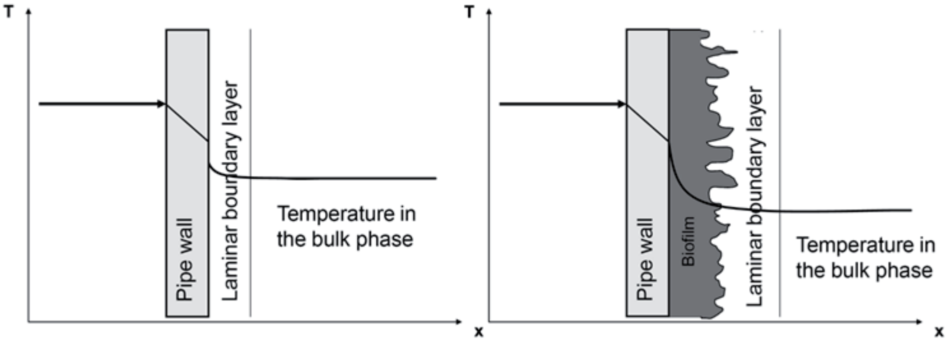

Fig. 4 **Influence of biofilms on the heat transfer [15]**

Biofilm detection

The development of biofilms in the pipes in front of the membrane plants is detected by two sensors (Sensor Deposens 2.0, Lagotec GmbH) installed in a bypass in the intake and outlet of the ultrafiltration plant. Thus, differences between the untreated water and the filtrate are detectable. A part of the intake is piped along the sensor back to the storage tank. The flow through the

Table 2 Specification of the used membranes in the RO plant [16]

Specification	Unit	Module 1	Module 2
Model		NE404070	RE4040CE
Membrane type		Nanofiltration	Reverse osmosis
Material		PA	PA
Dimensions (Ø x length)	inch	4 x 40	4 x 40
Effective membrane area	m²	7.9	7.9
NaCl rejection	%	40–70	99,5
Maximum transmembrane flux	l/(m²h)	24	24

Table 3 Measuring equipment

Mode	Measure	Equipment
Inline	Electrical conductivity	AQUIS 500 CR Typ 202565 (Measuring converter) Jumo 01478772 010 1035 0001 (Measuring sensor)
	pH	AQUIS 500 pH Typ 202560 (Measuring converter) pH 01461891 010 1027 0056 211500 (Measuring sensor)
	Flow rate	Buerkert Flow SE35/8035 Hall Sensor with Fitting S030/S035
	Pressure	Endress + Hauser Ceraphant T PTP31
Offline	Electrical conductivity	HANNA Combo pH + EC Typ HI98129
	pH	Hach HQ 11d
	Temperature	Hach HQ 11d

bypass can be controlled manually by a valve and an integrated flowmeter (Fig. 3).

In heat exchangers, with increasing thickness, the biofilm accumulation reduces the heat transfer between a flowing medium and the inner pipe wall [12,13] (Fig. 4). This effect is also used in the biofilm sensors. The sensor surface is form-closed integrated in the pipe wall and measures the thermal diffusion barrier caused by organic and inorganic residuals. The received signal is proportional to the biofilm thickness [14]. The generated data is saved continuously by an internal micro-controller. Via a cleaning access, the biofilm sensors were cleaned mechanically with a brush, after a constant biofilm thickness had been detected. After each cleaning, a zero point calibration was necessary.

Chemicals

To prevent scaling, hydrochloric acid (33 %) and the antiscalant MT3000 by Grünbeck Wasseraufbereitung GmbH were dosed in the feed flow of the RO plant.

Membrane specifications and pilot operation

Membranes from the CSM Woongjin Chemical Co. Ltd were tested in the first experiment. Due to good results with the NF membrane, the RO membrane in module 2 was installed 10 days later. The specification of the membranes is displayed in table 2. At the beginning of the experiment, the pH in the feed was adjusted to 6.5 with hypochloric acid. During the operation, the pH was increased to 6.9. After 80 days of operation, the experiment was stopped and the membranes were removed for SEM/EDX analysis.

Table 4 Ion composition of the UF filtrate

Ion	Concentration [mg/l]
Ba^{2+}	0.042
Ca^{2+}	46–58
K^{+}	13–39
Mg^{2+}	20–24
Na^{+}	423–503
Si^{4+}	9.2
Sr^{2+}	0.58
Cl^{-}	193–315
F^{-}	0.22
HCO_3^{-}	1220
P^{3-}	12.6
SO_4^{2-}	11–30

Process parameters

To supervise the process, pressure, flow rate, temperature, pH value and electrical conductivity are measured and recorded online. In addition to the online measurement, scheduled measures of the electrical conductivity, temperature and pH of the feed flow, permeate and concentrate of each module were carried out. The used equipment is listened in Table 3.

Water analysis

To determine the salt rejection of the membranes, samples of the feed flow, the permeate and the concentrate of the RO modules were analyzed, based on the German and International Standard Methods (DIN EN ISO).

SEM/EDX

After the experiment a sample of the feed and the concentrate side of each membrane were removed and air dried. For the scanning electron microscope (SEM) and Energy-dispersive X-ray spectroscopy (EDX) analysis, the samples were coated with a 5 nm gold layer with a BAL-TEC SCD 005 Sputter Coater. The SEM/EDX was performed with a Joel JSM-5900 LV Scanning Electron Microscope at 15 keV.

4 Results and discussion

4.1 Water analysis and calculation of scaling potential

For calculation of the scaling potential and dimensioning of the RO, the ion composition of the UF filtrate was analyzed (Table 4).

Calcium phosphate scaling

The UF filtrate has a pH value of 7.8 to 8.0. Concerning to figure 2, at this pH, 90 % of the phosphorous is HPO_4^{2-} and 10 % is $H_2PO_4^{-}$. Supposing the worst case, a 100 % salt rejection and calculating with the highest ion concentrations, the *IP* is in all cases higher than

Table 5 **Calcium carbonate scaling; pH_s at different recoveries**

Recovery	pH_{sat}
0 %	7.1
20 %	7.0
40 %	6.8
60 %	6.5

Table 6 **Sulfate scaling; *IP* and *SP* at 60 % recovery**

Scaling compound	Ion product IP [mol²/L²]	Solubility product SP [mol²/L²]
$CaSO_4$	2.82×10^{-06}	3.27×10^{-04}
$SrSO_4$	1.29×10^{-08}	1.96×10^{-06}
$BaSO_4$	5.97×10^{-10}	9.78×10^{-10}

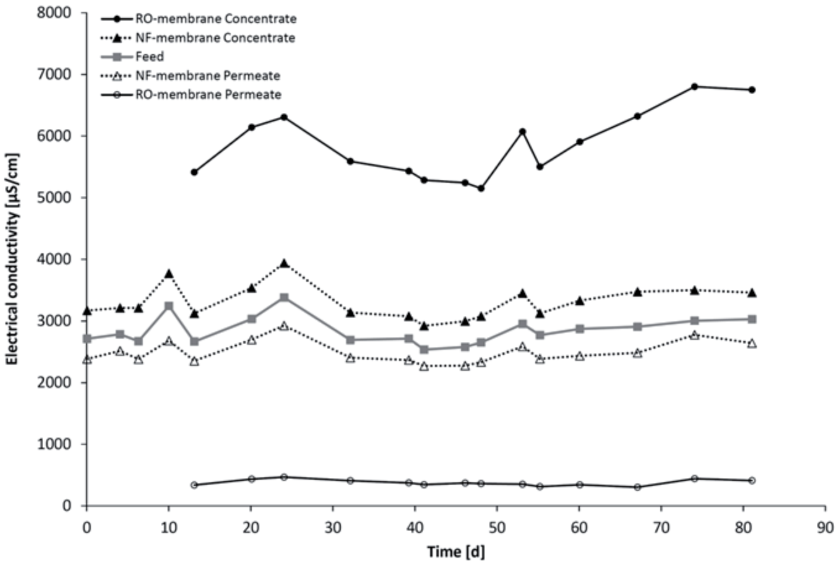

Fig. 5 **Electrical conductivity of the feed, permeate and concentrate of the NF/RO**

the *SP*, so that scaling is expected. At lower pH, the ratio moves in the direction of the soluble $H_2PO_4^-$. Thus, one way to eliminate the scaling is the pH reduction in the feed of the membranes.

Calcium carbonate scaling
The calculation of the pH_s is presented in table 5. Because the *pH* of the feed is higher than pH_s, the LSI is positive in all cases and scaling is expected. Similar to calcium phosphate scaling, a reduction of the pH with hypochloric acid would increase the solubility.

Calcium sulfate, barium sulfate, strontium sulfate scaling
At 60 % recovery and 100 % salt rejection, the *SP* is higher than the *IP* in all cases. Thus, sulfate scaling seems to be no problem (Table 6).

4.2 Operation of the RO plant

With both membranes, the NF and the RO membrane, a stable operation was reached. The recovery was 55–60 % in both modules. The permeability of the NF membrane was 7–10 l/(m² h bar). The permeability of the RO membrane was about 2.5–4.1 l/(m² h bar). The conductivity measurements, shown in Figure 5, display a clear difference in the membrane salt rejection. The electric conductivity of the NF permeate was 2200–2900 µS/cm, the RO permeate had a conductivity of 300–460 µS/cm. These numbers were confirmed by a water analysis (Table 7).

4.3 SEM/EDX Analyse

The SEM images (Fig. 6) show the concentrate side of the RO membrane as well as the membrane after a cleaning with a UF-filtrate/HCl solution at pH 2.0. Scaling on the membrane surface is clearly visible. After the cleaning the scaling was removed

Table 7 **Water analysis of the feed, permeate and concentrate of the NF/RO**

			NF Membrane		RO-Membrane	
Parameter	**Unit**	**Feed**	**Permeate**	**Concentrate**	**Permeate**	**Concentrate**
pH	[–]	6.6	6.4	6.7	5.5	6.8
Temperature	[°C]	21.6	23.1	24.0	23.9	24.3
Conductivity	[µS/cm]	2712	2366	3075	373	5430
KS4.3	[mmol/l]	13.3	10.2	–	1.2	–
Ba2+	[mg/l]	0.024	0.015	0.038	<0.001	0.048
Ca2+	[mg/l]	54.6	35.5	82.0	0.36	124
K+	[mg/l]	27.0	25.1	33.1	3.621	74.6
Mg2+	[mg/l]	21.8	10.17	39.3	0.094	51.5
Na+	[mg/l]	547	485	647	68.9	1470
Si4+	[mg/l]	9.3	8.9	9.7	0.677	25.3
Sr2+	[mg/l]	0.80	0.50	1.3	0.003	1.7
Cl–	[mg/l]	361	369	392	37.6	681
P3–	[mg/l]	17.4	1.7	40.9	0.092	37.7
SO42–	[mg/l]	14.1	<0.1	32.1	<0.1	27.03

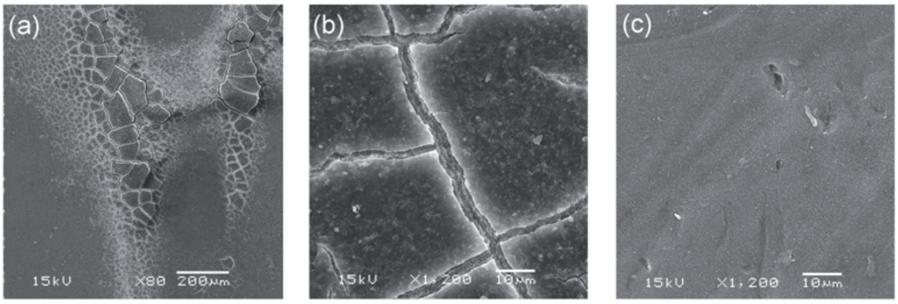

Fig. 6 SEM images
(a) Concentrate side of the RO membrane (80x)
(b) Concentrate side of the RO membrane in detail (1200x)
(c) Concentrate side of the RO membrane after cleaning with HCl (1200x)

almost completely. The EDX studies (Fig. 7–9) are consistent with the SEM images. In all investigations carbon, oxygen and sulfur were detected. These peaks can be effected by deposits on the membrane surface as well as the membrane material. An assignation to a particular group is not possible with the EDX. The deposits of calcium, phosphorus and magnesium suggest scaling, caused by phosphate and carbonate salts. On the feed side no major differences between the NF and RO membrane were detected. On the concentrate side, on the RO membrane, higher phosphorous peaks were measured, caused by the higher rejection of the RO membrane. On the cleaned membrane surface, only the elements of the membrane material were proven.

The results of the SEM/EDX analysis are congruent with the calculation of the scaling potential. Phosphate and carbonate salts precipitate on the membrane surface, sulfate scaling is not a problem. Further, the removal of the precipitated salts from membrane surface at a low pH can be used for a cleaning procedure within the RO plant.

4.4 Biofilm detection

With the biofilm sensors, the biofilm development in the feed and the filtrate of the UF was detected. Figure 10 shows the detection of biofilm thickness with the biofilm sensor in the inlet of the UF. In this figure, a typical biofilm development, as described by Flemming [17], is visible:

- Initial settlement
  Settling of microorganisms and macromolecules at the surface
- Exponential growth
  Adsorption of new microorganisms (passive aggregation) and growth of already existing cells (active aggregation)

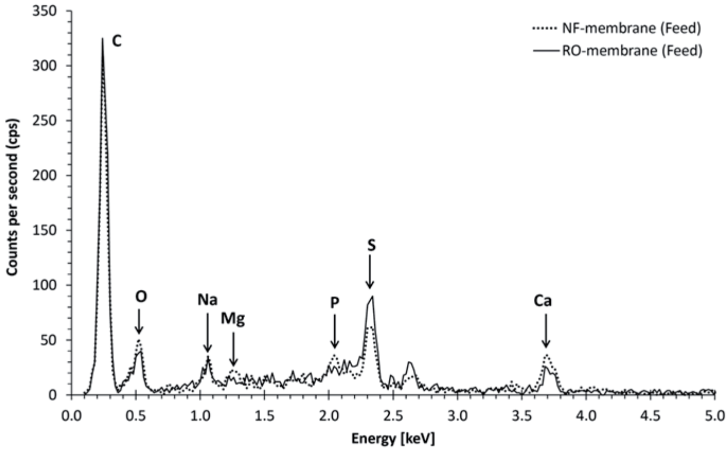

Fig. 7 EDX Analysis of the scaling on the membrane surface at the feed side

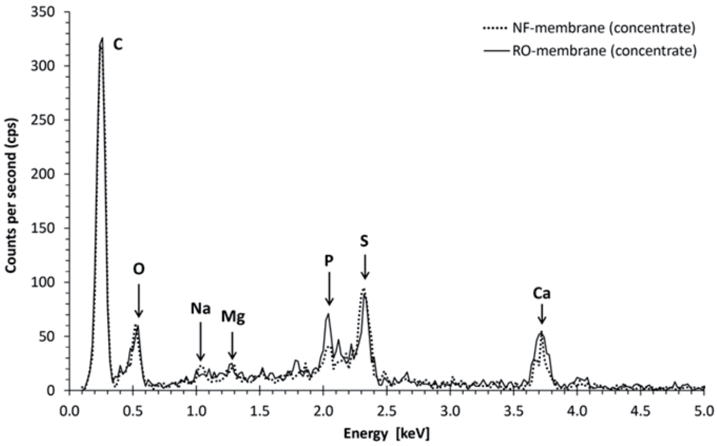

Fig. 8 EDX Analysis of the scaling on the membrane surface at the concentrate side

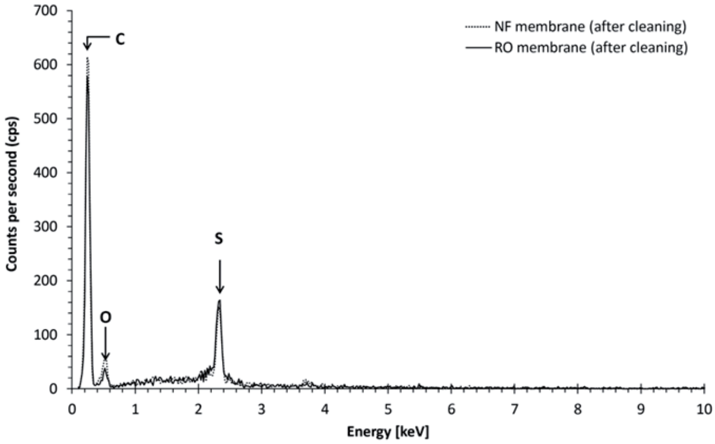

Fig. 9 EDX Analysis of the membrane surface after cleaning

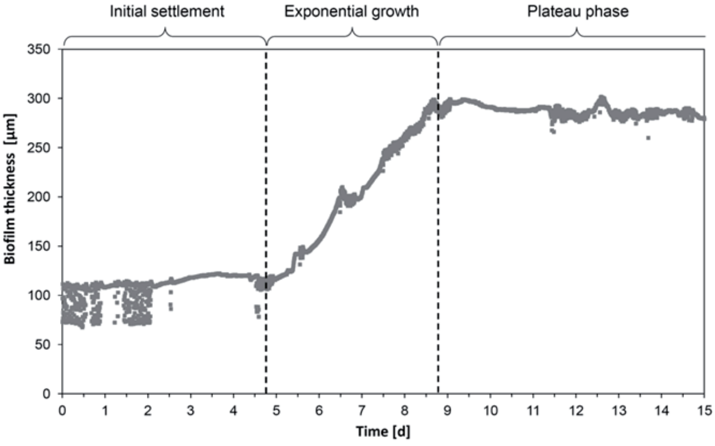

Fig. 10 Detection of the biofilm development in the intake of the UF

- Plateau phase
  Balance between the regeneration by adsorption and accretion and the detachment by erosion and sloughing

During the experimental period, biofilms with a thickness up to 350 microns were detected. The development of biofilms from the initial settlement to the plateau phase was between 5 and 10 days.

Regardless of the development of biofilms on the sensor surface within a few days, during the first six months of operation of the UF no significant increase in transmembrane pressure of the UF was detected. The biofilm development within the sensor was not comparable to the performance of the UF and RO plant. A reason for the poor comparability is assumed in the different operation modes. While a continuous flow passes the sensor, the UF is cleaned by the backwash cycles every 30 minutes. Thus, the biofilm development on the surface of the UF was avoided.

5 Conclusion

During the operation period, biofilm development had no significant effect on the membrane performance. Scaling on the RO surface was the central issue. In preliminary tests of the RO plant, a blockage of the membranes within 2–3 days was detected. It was assumed that the blockage is caused by scaling because a biofilm cannot block a membrane this quickly. This assumption was confirmed by the analysis of the ion composition of the water and the calculation of the scaling potential. Calcium carbonate and calcium phosphate were detected as the salts with the highest scaling potential.

Based on the calculation of the scaling potential, a test was carried out with additional acid dosage to increase the solubility of salts. Within this test, a constant operation of the NF and RO membrane without significant reduction in permeability was achieved. Fouling on the membrane surface was no problem during the operation because the pressure drop from the feed to the concentrate side of the membrane was constant. With biofouling, this pressure drop would have been increased because of additional friction losses as well as the transmembrane pressure drop. After 80 days of operation, the experiment was stopped and SEM/EDX analysis of the feed and concentrate side of the membranes were performed. On the membrane surface, a salt layer was distinguished. The salt layer could be removed with acidulous UF filtrate at pH 2.0.

The first test showed, that the recycling of wastewater with the presented concept is possible and a constant operation could be reached. The next step is the investigation of the fouling/scaling on the membranes in detail by CLSM and SEM/EDX analysis and the optimization of the operating conditions to make wastewater recycling more economically.

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