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Cleaning of reverse osmosis membranes

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ABSTRACT

This presentation tries to answer this question by comparing the cleaning efficiency of inorganic acid and alkali with different Osmotech membrane cleaners. Further scopes of work were to investigate the effect of different cleaner combinations and the sequence of their application, e.g. acidic followed by alkaline cleaning or vice versa. The tests have been carried out on a reverse osmosis pilot plant using flat sheet membrane samples cut from an 8´´ membrane element which has been in operation for several years. The element has been autopsied prior to the tests to investigate the type of deposit.

Keywords: Membrane cleaning; Reverse osmosis (RO); SWRO; Scaling; Fouling; Biofouling; Cleaning chemicals; Osmotech membrane cleaners; Cleaning products; Membrane autopsy; Pretreatment

1. Introduction

1.1. Challenges in SWRO operations

The sea water reverse osmosis (SWRO) has become the most popular and widespread technique for sea water desalination in the last decades. However, the operation of a SWRO plant even today after many years of constant technical development is still not free of problems.

One challenge which every SWRO application faces is the formation of deposits on the membrane surface during operation, often generalized as "fouling." This fouling layer inevitably leads to a performance loss of the membrane plant over a certain time span due to the blockage of the membrane surface and consequently to higher operational costs caused by increasing energy consumption.

In real field where surface waters like river water or seawater are purified via passing through RO membranes, only a special kind of fouling or scaling with a constant composition takes place. Precipitation and deposition of the different substances are normally coincident. Furthermore, the quality of intake water changes in different seasons of the year with changing amount of rainfall, temperature, or environmental and local factors.

2. Deposits on reverse osmosis membranes

2.1. Scaling, fouling, and biofouling

The main task of an RO membrane is to remove dissolved solids, (e.g. calcium, sodium, chloride, and sulfate) from the water as it acts as a barrier against them. The retention of the membrane regarding these dissolved salts usually ranges between 98.0 and 99.9%, depending on the membrane specifications and

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the type of the rejected ions. Bivalent ions like Ca^{2+} or SO_4^{2-} are generally better rejected than monovalent ions such as Na^+ or Cl^-. Undissolved particles which slipped through the pretreatment or precipitated thereafter in consequence of pH variations or exceeding of solubility limits are rejected completely by RO membranes. All these rejected substances accumulate on the surface of the membrane and affect, i.e. reduce the filtration performance. The general name of this unwanted obstacle is fouling. The term "fouling" generally includes several different kinds of layers building up on the membrane and the feed spacer surface and can basically be classified into three different kinds of deposits: scaling, fouling, and biofouling.

2.2. Scaling

Scaling is typically caused by the excess of the solubility limit of sparingly soluble salts contained in the feed–concentrate stream. If the dissolved salts are concentrated during the process beyond their solubility limit they tend to precipitate. This phenomenon will start at the point of the highest concentration—on the membrane surface in the last element of the plant. The tendency for precipitation can be influenced by physicochemical parameters like the pH (e.g. for calcium carbonate) or the temperature of the water. Typical scalants are for example: calcium carbonate, alumino silicate, barium sulfate, and magnesium hydroxide (Brucite). An effective countermeasure to control the scaling tendency is the continuous use of antiscalants.

2.3. Particulate, colloidal, and organic fouling

This includes the deposition of all kinds of nondissolved substances flushed in by the feed stream into the membrane elements surface such as iron flocs, silt, humic substances, etc. The typical ways of deposit building are the sedimentation of particles in areas of low flow, the plugging of the feed channels by an overload of particles, or the adhesion on the membrane surface due to interactions between the electrical charges of particles/ions and the membrane surface. These kinds of fouling start usually from the feed side, at the first elements of the plant.

2.4. Biofouling

Biofouling means the formation of biofilm on the membrane surface by the growth of bacteria. Biofilms are a very complex matrix composed of micro-organisms [1] and extracellular polymeric substances [2], as well as other organic molecules, such as humic substances [2,3]. Biofilms provide an appropriate environment for the growth and the survival of micro-organisms, which become—protected by the surrounding biofilm—highly resistant to sanitizing measures like biocide treatment. Depending on the origin of the raw water and the operating conditions of a membrane plant, biofouling is more or less likely to occur. In case of low operating temperatures and groundwater as raw water, problems caused by biofouling might be more unlikely whereas surface water, potentially carrying a high load of micro-organisms and nutrients, in combination with high ambient temperatures strongly favor biofilm formation.

2.5. Concentration polarization

The formation of deposits is further supported by the concentration polarization of the feed stream directly on the membrane surface. Once a solution is passed through a selective membrane that separates components from the solution, the concentration of the separated compounds starts to increase on the surface of the membrane. This phenomenon occurs not only on reverse osmosis membranes, but also in other membrane filtration applications. For the RO process, the membrane is the separating interface, the solution is saline or brackish water, and the separated substances are dissolved (or undissolved) solids of the feed water.

Fig. 1 shows the principle of concentration polarization in a cross-flow membrane application. It is shown that the concentration increases over a certain distance (r_B) called "boundary layer." The thickness of

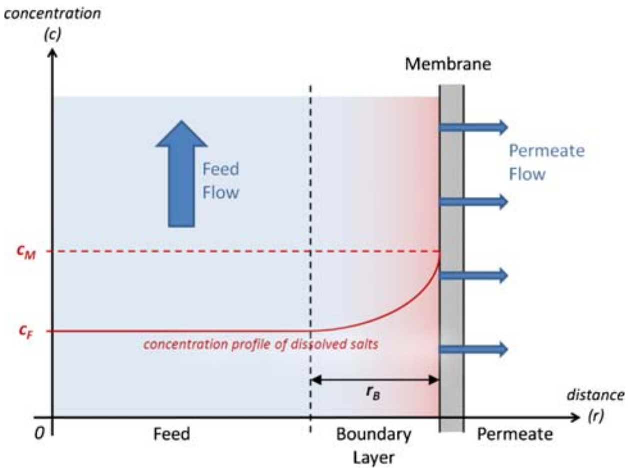

Fig. 1. Concentration profile of dissolved salts and suspended solids from the feed solution (c_F) to the membrane surface (c_M).

this boundary layer is highly dependent on the turbulence in the feed stream. The more turbulent the feed flow, the thinner is the boundary layer. The phenomenon is in fact a preliminary stage for fouling and scaling, so it should be kept as low as possible. Effective countermeasures are a turbulent feed flow created by high flow velocity and appropriate spacer design. A higher permeate flux increases the concentration polarization—so flux or applied pressure should not exceed a certain level—while the flow velocity of the feed stream should not fall below a certain minimum. The phenomenon illustrated in Fig. 1 also reduces the observed rejection and permeates flux because the existing concentration as well as osmotic pressure on the active surface area is higher than that of feed solution (which can be monitored by process measuring and control technology).

Although the deposits on RO membranes are quite similar in some cases, (e.g. from SWRO operating under comparable conditions), every fouling layer in field applications can be considered as unique, because it is always a result of combined fouling of different types of deposit. Furthermore, the formation of each fouling layer is influenced by many local factors, e.g. plant parameters, fluctuations in operating factors, variations in pretreatment, seasonal changes of the feed water quality, etc.

3. Cleaning of reverse osmosis membranes

3.1. Cleaning frequency

Although in theory the causes for these deposits can be prevented by a perfect pretreatment of the membrane feed water, in practice these problems can only be retarded to a certain degree. This fact requires facing appropriate actions to counter the negative effects of fouling and to ensure a maximum system lifetime. Therefore, membrane cleaning operations at regular intervals are inevitable.

As mentioned before, the formation of deposits on membrane surfaces has multiple reasons. The related consequences on the other hand are much easier to observe as they all show the same effect: a significant performance loss of the RO plant by reduced permeate flow and quality. By close monitoring of the development of some key performance indicators (KPI) like permeate flux, differential pressure (feed to concentrate), and salt passage, the right time to start a cleaning can be chosen. Before evaluation of the KPI, it is of highest importance to normalize the measured operating data, in order to eliminate the fluctuations caused by changes in feed water quality, (e.g. salinity), temperature, or pressure during operation.

Membrane manufacturers usually recommend conducting a cleaning when:

- The normalized permeate flow drops 10%.
- The normalized salt passage increases 5–10%.
- The normalized pressure drop (feed pressure minus concentrate pressure) increases 10–15% [4].

However, in field operation these recommendations are often not kept with, but cleaning is done not before there is a much higher impact on the key performance indicators, for different reasons. At this point, it should be considered that the deposits usually do not distribute evenly over the surface of the membrane. In other words, a drop in permeate flow of 10% caused by scaling does not mean that every element has lost 10% of its performance. Scaling starts at the point of highest concentration (in the last elements of the plant) and may not even affect the first elements at all. This means that the last elements probably already are subject to severe scaling, while the normalized overall permeate flow dropped "only" by 10%.

If a cleaning is delayed too long, it can be difficult to remove the foulants completely from the surface. This can make things worse in the long-run because remaining deposits can enhance new fouling, which in the end will increase the necessary membrane cleaning frequency.

3.2. Choice of cleaner and cleaning sequence

Beside the standard cleaning chemicals like inorganic acids and alkalis, today there are various brands and products of specialty membrane cleaners available, which are formulated and produced by different companies. These products are generally classified as acidic and alkaline cleaners and can furthermore contain various active ingredients beside the basic acids and alkalis.

The best basis to select the right cleaning program is the detailed knowledge about the composition of the foulants present on the membrane. However, this can only be achieved by performing a membrane autopsy which is a time consuming operation and comes along with the additional costs for one or more destroyed membrane elements. To get as much useful information as possible, the following points should be checked:

- Feed water analysis.
- Development of normalized key performance indicators.

- Results of previous cleanings (analysis of cleaning solutions available?).
- Analysis of silt density index (SDI)—filters or cartridge filters.
- Any other related results that give indications about possible foulants, e.g. deposits of the inner surfaces of feed pipes or pressure vessels, prior membrane autopsy reports, etc.

In general, alkaline cleanings are recommended to remove organic fouling and biofouling, while acidic cleanings are more effective against inorganic deposits. As in most cases, the fouling layer exists of a combination of several foulants, consisting of inorganic, organic, and biological components, a two-step cleaning is generally recommended using an alkaline as well as an acidic cleaner. If not otherwise indicated, e.g. by experiences of previous cleanings it is strongly recommended to perform the alkaline cleaning step first. Acidic cleaning solutions can react with some foulants like humic substances, silica, or biofilm, resulting in a further decline of the membrane performance [4].

Beside the use of alkaline and acidic chemicals a standard cleaning should as well include a final sanitization by a biocide treatment. This is important in order to achieve the best cleaning effect possible and to extend the maintenance intervals by preventing a quick recontamination by micro-organisms.

The detailed procedure of a membrane cleaning program including choice of the cleaner, applied concentration, pH value, temperature during cleaning, contact time, soaking intervals, etc. is very system specific and should be customized for each plant to ensure the best possible cleaning results.

This paper shows the results of studies on the cleaning performance of specialty cleaners in comparison to commodities that were obtained under controlled conditions in pilot membrane plants. Two fouled membranes from different reverse osmosis plants were used for this study.

4. Equipment and material used in this work

4.1. Pilot plant A

This plant is equipped with three test cells set up in serial mode for flat sheet membranes with an active area of 86 cm² (4 × 21.5 cm). The permeate flow of each test cell can be measured and sampled separately. Assuming a recovery per cell of roughly 2%, the increase in concentration for the second and third test cell is negligible. Therefore, the test cells can be as well considered to be set up in parallel design as the feed water quality is nearly the same for all three cells. The high-pressure pump of the plant can deliver up to 60 bar feed pressure. The feed pressure is controlled by an automatic pressure control valve and a pulsation damper. The system volume is 10 L including the feed containment. The data for feed pressure, feed and permeate flow, pH, and conductivity are measured and recorded (Figs. 2 and 3).

4.2. Pilot plant B

This plant is equipped with a single circular test cell for flat sheet membranes with 65 cm² (9.1 cm diameter) active surface. The high-pressure pump can create a feed pressure up to 100 bars. The special design of the pump head creates only a negligible pressure ripple and makes the use of a pulsation damper needless. The data for pH, conductivity, temperature, feed pressure and feed and permeate flow are measured and recorded. The system volume is 10 L including the feed containment (Figs. 4 and 5).

Fig. 2. Pilot plant A with thermostat and control panel.

Fig. 3. Triple test cell of pilot plant A.

Fig. 4. Control panel of pilot plant B.

Fig. 5. Opened test cell of pilot plant B.

4.3. Membranes

Membrane samples from two different spiral wound membrane elements, which have been autopsied in our laboratory, have been used for the cleaning tests (Table 1).

4.3.1. Membrane I

Type: Brackish Water Membrane
Manufacturer: Vontron
Model: ULP32–8040

Chemical composition of fouling layer (analyzed by loss of ignition [LOI] and X-ray fluorescence analysis [XRFA]):

- Mainly organic (>90%).
- Low amounts of silica, iron, phosphorous, and sodium.

Table 1
Specifications for Vontron ULP32-8040 spiral wound element [5]

Operation limits and conditions	
Max. working pressure	41 bar
Max. feed water temperature	45 °C
pH range of feed water during continuous operation	3–10
pH range of feed water during chemical cleaning	2–12
Max. pressure drop of single membrane element	1.0 bar
Max. feed water SDI	5
Residual chlorine concentration of feed water	<0.1 ppm

Optical appearance of membrane surface:

- A slightly reddish color of the surface together with a positive test for iron (total Fe) indicated iron fouling.
- A mucous layer indicated biofouling.

Position of membrane pressure vessel:

- Last element of pressure vessel (brine side)

Type of application:

- Production of water for industrial processes.

Feed water quality:

- City water from local distribution pipelines (pretreatment unknown).

All tests with membrane II have been carried out on pilot plant A.

4.3.2. Membrane II

Type: Sea water membrane
Manufacturer: Dow Filmtec
Model: SW30HR LE-400

Chemical composition of fouling layer (analyzed by LOI + XRFA) (Table 2):

- Organic: >50%.
- Main inorganic compounds: silica (10%), sodium (8%), iron (7%), aluminum (4%), magnesium (3%), sulfate (2%), calcium (2%), and phosphate (2%).

Table 2
Specifications of filmtec SW30HR LE-400 spiral wound element [6]

Operation limits and conditions	
Max. working pressure	83 bar
Max. operating temperature	45°C
pH range, continuous operation	2–11
pH range, short-term cleaning (30 min)	1–13
Maximum element pressure drop	1.0 bar
Maximum feed silt density index (SDI)	5
Free chlorine tolerance	<0.1 ppm

Optical appearance of membrane surface:

- A slightly reddish color of the surface and a positive test for iron (total Fe) indicated iron fouling.

Position of membrane pressure vessel:

- Third element of pressure vessel (from feed side).

Type of application:

- Sea water desalination for drinking water production.

Feed water quality:

- Sea water pretreated by coagulation and sand filtration.

All tests with membrane II have been carried out on pilot plant B.

4.4. Applied Cleaning chemicals

See Table 3.

4.5. Test conditions and procedure

Sheets of membranes have been sampled from the autopsied elements and mounted in the relevant pilot plant. As the membrane elements were not in use for a longer period, the samples needed some time to be "re-wetted." Running under normal operating conditions, it took up to 72 h to achieve constant values of permeate flow and salt rejection.

The test sequence for each membrane sample followed the sequence given below:

Step 1: Re-wetting of membrane samples.
Step 2: Flux test ($Flux_{start}$).
Step 3: First cleaning.
Step 4: Flux test (only in pilot plant A).
Step 5: Second cleaning.
Step 6: Flux test ($Flux_{end}$).

For the re-wetting phase and the flux tests, a standard solution with 2,000 mg/l sodium chloride (NaCl) was used. The pressure during these steps was 30 bars (Pilot Plant A), respectively, 10 bars (Pilot Plant B). A biocide was applied to rule out the influence of biofouling during the tests. Between each step, the plant was rinsed carefully with demineralized water until the pH and conductivity values indicated that the cleaning solution had been sufficiently removed from the system.

The cleaning steps have been performed at atmospheric pressure and at a temperature of 23 ± 1°C. The applied product concentration in the cleaning solution was 1% for all Osmotech products. The pH during the cleaning was set to 2.0 (acidic), respectively, 12.0 (alkaline) to minimize the effect of a varying pH between the tests. The pH has been controlled and kept constant during the cleaning steps. The duration

Table 3
Names and main active ingredients of applied cleaning chemicals

Name/Chemistry		Active Ingredients			
		Alkalis	Acids	Surfactants	Complexants
Osmotech	2230	X		X	X
Osmotech	2632	X		X	X
Osmotech	2691	X		X	X
Osmotech	2610	X		X	X
Osmotech	2212	X		X	
Sodium Hydroxide[a]		X			
Osmotech	2575		X		
Osmotech	2173		X	X	
Hydrochloric Acid[a]			X		

[a] Analytical grade quality.

of the cleaning steps was 2 h for "plant A" (60 min recirculation and 60 min soaking) and 1 h for "plant B" (30 min recirculation and 30 min soaking). For each test, a separate membrane sheet was used.

4.6. Normalization and evaluation of data

Although the conditions during the tests, regarding feed temperature, pressure, etc. have been quite stable due to the fact that the pilot plants were running in a laboratory environment, all data have been normalized prior to evaluation. The normalization has been performed by use of the "FTNORM" software provided by Dow Filmtec.

Membrane sheets with small surface areas, like the ones used in this work, often show a high variation in their permeate flux. Even new membrane samples cut from the same sheet can easily show differences of 10% and more compared to each other. Knowing this, the absolute permeate flux of the samples could not be used for the evaluation of the cleaning tests because the fluctuations of the samples would have been too high to make a reliable evaluation possible. Thus, the values for the permeate flow after each cleaning step have been compared to the initial permeate flow before the first cleaning step (Step 2 of the test sequence) and the increase in permeate flux has been expressed in percent, following the simple formula:

$$\text{Flux increase} = \frac{\text{Flux}_{\text{end}} - \text{Flux}_{\text{start}}}{\text{Flux}_{\text{start}}} \times 100\%$$

5. Results

5.1. Evaluation of cleaning sequence

To investigate the effects of the sequence of a 2-step cleaning using acidic and alkaline cleaning solutions, two tests have been repeated in reversed order using the same types of cleaning solutions. After each step, a flux test has been done to evaluate the effect of the cleaning. The two cleaning steps of each test have been carried out with the same membrane sample. In tests 1 and 2, hydrochloric acid was used for the acidic and sodium hydroxide for the alkaline cleaning step. The same procedure has been repeated using Osmotech 2575 (acidic) and Osmotech 2691 (alkaline).

The results for tests 1–4 can be seen in Fig. 6. The values in brackets indicate the flux increase achieved by the single cleaning step.

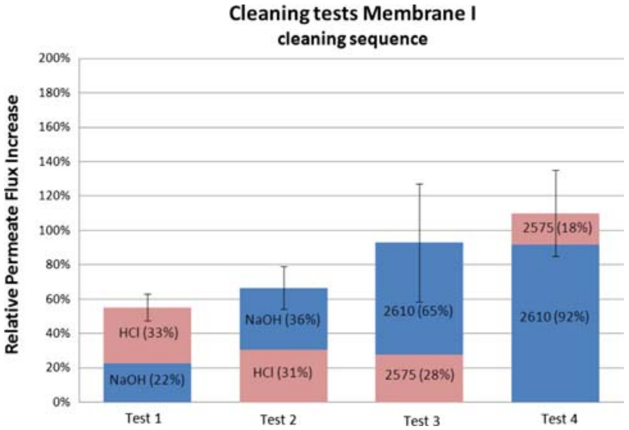

Fig. 6. Evaluation of cleaning sequence of different cleaning programs (the lower part of the bar shows the first cleaning step and the upper part shows the second one); dark gray indicates alkaline cleaning and light gray indicates acidic cleaning.

A definite conclusion which cleaning step to perform first cannot be drawn by the results of this test. While the order "acidic followed by alkaline" seems to give a slight advantage regarding tests 1 and 2, it is just the opposite in tests 3 and 4. The overall differences in flux increase regarding the cleaning sequence are not very significant. However, a negative effect on the permeate flux if the acidic cleaning step is performed first could not be observed in this test run.

What is more noticeable is the difference between the use of standard chemicals (tests 1 and 2) and specialty cleaners (tests 3 and 4). Using the Osmotech products—no matter in which order—the flux increase is significantly higher. Looking at the single cleaning steps, this is mainly the benefit of the alkaline cleaner Osmotech 2610 which gives the highest contribution to flux increase.

5.2. Evaluation of cleaner combinations—Membrane I

In order to find out the best cleaning program for this specific type of fouling and to evaluate the differences in cleaning performance, further tests with different combinations of cleaners and chemicals have been done. In these tests, the standard procedure beginning with the alkaline cleaning step followed by the acidic one has been applied. The only exception is test 5. In this case, the membrane has been cleaned twice using the same type of alkaline cleaner. Tests 5–9 in Fig. 7 show the results. The shown standard deviation is the total one for the complete test including both cleaning steps.

Looking at the results of tests 1–9, the following trends can be identified:

- The alkaline cleanings using Osmotech cleaners show by far the highest efficiency regarding the single cleaning steps. Only Osmotech 2212 performs significantly lower.
- The acidic cleanings do not show a similar variation in effect regarding the use of Osmotech cleaners or HCl.
- The average efficiency of the acidic cleanings is highest if performed as first cleaning step or if the previous alkaline cleaning was less effective (tests 1, 2, 3, and 8). The better the previous alkaline cleaning step, the lower the performance of the acidic cleaning (tests 4, 5, 6, 7, and 9). It can be assumed that the alkaline Osmotech cleaners remove to a certain extend some of the substances that are typically dealt with by acidic cleanings, (e.g. iron and calcium).

Considering the high portion of organic components in the fouling layer (probably mainly due to biofouling) and the low fraction of inorganic material, the result is not surprising. Alkaline cleanings are well known to have the better performance against (bio-) organic deposits, where the effect of acidic cleaners is clearly limited.

5.3. Evaluation of cleaner combinations—Membrane II

An equivalent test series has been carried out on a set of samples from membrane II in pilot plant B. In contrast to the conditions of the previous test scenario, the contact time of the cleaning solutions was lower (1 h instead of 2) and no flux test was performed between the single cleaning steps. The alkaline cleaning step was always performed first. The bar graphs in Fig. 8 show the results of the tests.

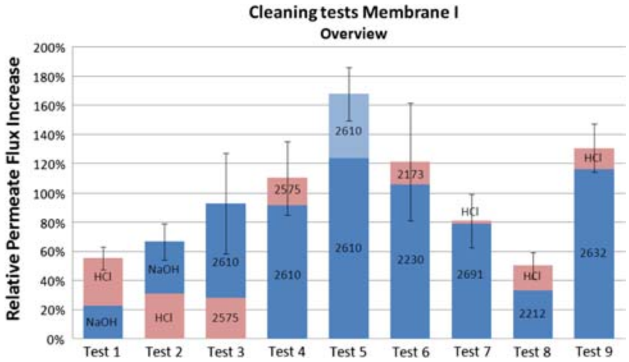

Fig. 7. Cleaning tests on membrane I—overview about different combinations of Osmotech cleaners. For comparison the results obtained with commodities are also shown (dark gray indicates alkaline cleaning and light gray indicates acidic cleaning).

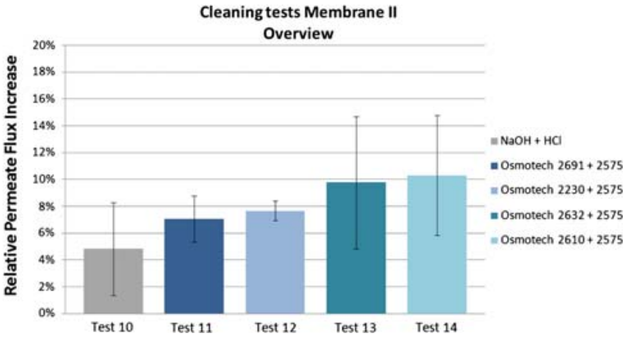

Fig. 8. Cleaning tests on membrane II—overview about different combinations of Osmotech cleaners. For comparision, the results obtained with commodities are also shown.

Unlike the previous results in chapter 5, the overall increase in permeate flux was significantly lower. Possible explanations for this observation could be the shorter cleaning time and the overall lower amount of foulants on the membrane surface, which perhaps limited the overall range for improvement. The evaluation of this test series by comparing the flux increase of tests 10–14 to each other, however, draws a similar picture than observed in Fig. 8. The flux increase achieved in the tests based on Osmotech cleaners again was significantly as higher as by the use of standard chemicals. The cleaner combinations used in tests 13 and 14 increased the permeate flux around twice as much as the standard chemicals used in test 10.

However, the conclusions from this test series should be drawn more cautiously because the standard deviation in most tests is significantly higher than in the previous tests shown in Fig. 7.

6. Conclusion

The better performance of Osmotech cleaners reported in this paper compared to the commodities (sodium hydroxide and hydrochloric acid) is most probably caused by the additional active ingredients contained in the specialty cleaners. On the other hand, this does not necessarily mean that a cleaning using commodities cannot give satisfying results. A successful cleaning always depends on several factors which must be in balance to give the desired efficiency. The most important of these factors are:

- The choice of the cleaning chemicals (depending on the composition of the foulants).

- The concentration of the cleaning chemicals (influencing also the pH).
- The temperature (influencing the speed of the chemical reactions).
- The exposition time (the chemical reactions need a certain time to complete).
- The flow speed (influencing the shear force which helps to remove deposits by kinetic energy).

Three of these factors are limited due to technical parameters regarding the plant design and the membrane specifications:

- A temperature of more than 40˚C during cleanings is usually not recommended as a standard procedure. Frequent cleanings at higher temperatures under harsh chemical conditions can result in membrane damages or a significantly shorter membrane lifetime (for details on the exact limits for each specific type of element, the membrane manufacturer should be consulted),
- The applicable pH value during cleanings for a wide range of reverse osmosis membranes varies between pH 1–3 for acidic cleanings and pH 11–13 for alkaline cleanings. The pH limits are furthermore depending on the applied temperature, especially in the alkaline range.
- The flow speed is limited primarily by the design of the membrane elements and the applied equipment (pumps) during cleanings and can as well only be chosen within certain limits.

This leaves only two parameters which can be influenced directly during cleanings: the choice of chemicals (and in certain ranges the concentration) and the cleaning time. A longer exposition time combined with several soaking intervals is a well known and widely applied method to increase the cleaning performance significantly. However, in real applications this comes at the cost of longer system downtimes which decreases the overall permeate production—and thereby the profitability—of the plant. Regarding this it becomes clear that the choice of the cleaning program is not only a matter of technical issues but also of economical ones.

The use of specialty cleaners must be evaluated also from this point of view. The higher price for the cleaners compared to commodities will pay back quickly due to the reduced system downtime. Another possible advantage by use of specialty cleaners is a lower cleaning frequency in the long-term view. This would be the result of a better cleaning performance and a slower recontamination in consequence. The effect is system specific and will be visible within long-term field application. A lower cleaning frequency would not only have the effect of reduced system down time, but furthermore a smaller number of cleanings most probably improves the overall membrane lifetime—offering possible savings again by means of postponed replacement of membrane elements.

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