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Induction time measurements in two brackish water reverse osmosis plants for calcium carbonate precipitation

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ABSTRACT

In this research work, the induction time of two brackish water reverse osmosis (BWRO) plants was measured with and without antiscalant (AS) operation with focus on calcium carbonate precipitation. This study focused on two BWRO plants in the Netherlands. The scaling potential of RO concentrate in the plant at recovery of 80% was calculated using the PHREEQC program. Induction times in RO concentrates were measured by collecting RO concentrates directly from the plants, with and without an AS dose in an air-tight glass reactor. The solution was continuously stirred for homogenization and maintained at room temperature using a thermostat. The change in pH of the solution over time was monitored. The time required to change the nucleation phase to just the start of the crystal growth was noted from the pH versus time graph, which was defined as induction time. At the end of the experiments, precipitate with RO concentrate with and without AS dose (plant B) were collected and an X-ray powder diffraction (XRD) and scanning electron microscope (SEM) analysis were performed. The results showed that the measured induction times for RO concentrate with AS dose in the two plants were longer than 100 and 280 h, respectively. The plants were operated at the same recovery (80%) but with different types of feed water and different types of AS. In a plant where the induction time could be measured without AS, the induction time turned out to be short namely about 4 h. The XRD results of this plant with and without an AS dose revealed calcite as the exclusive precipitate. The shapes of the crystals obtained with AS, shown by SEM images, were smaller, less clustered, and had more rounded edges than crystals without AS. The results suggested that the measured induction times of 100–280 h in both plants are much higher since the detention time in RO systems is just 1–1.5 min and even seconds in the last membrane. Therefore, for safe operation, induction time lower than what we measured in two BWRO plants might be needed.

Keywords: Scaling; Induction time; Calcium carbonate; Antiscalant; Brackish water

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1. Introduction

Today, desalination is a solution that is being increasingly applied to water scarcity problems in many regions of the world. For this purpose, different water sources are used, such as seawater, brackish water, and wastewater. Among the various types of desalination technologies, reverse osmosis (RO) is the most common technology for the treatment of brackish

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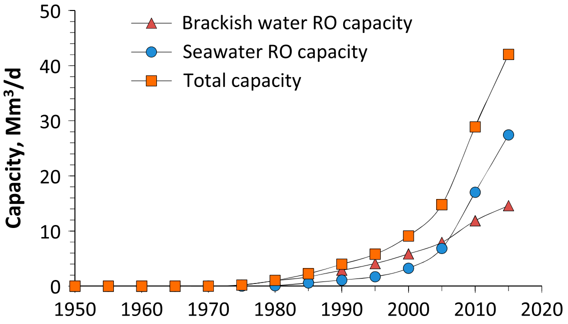


Fig. 1. World seawater and brackish water RO capacity from 1950 to 2013.
Source: Desal data [1].

water and is taking over the lead from seawater RO as well. The growth of reverse osmosis plants throughout the world has been very fast since 2000 and is expected to reach a cumulative capacity of about 45 Mm^3/d by 2015. Both seawater reverse osmosis (SWRO) and brackish water reverse osmosis (BWRO) are growing rapidly. SWRO is growing mainly due to large installations, while BWRO is very substantial as well but with many smaller units, as in Fig. 1 [1].

Both in seawater and brackish water RO, the precipitation of sparingly soluble inorganic compounds plays an important role. In seawater RO the recovery is governed by the osmotic pressure and in brackish water by the scaling potential, due to these compounds. Even at low rate of recoveries, scaling might occur mainly due to calcium carbonate ($CaCO_3$). At higher rate of recoveries in brackish water, compounds such as: barium sulfate ($BaSO_4$), calcium sulfate, strontium sulfate, calcium fluoride, calcium phosphate, and silica (SiO_2) might exceed their solubility. Traditionally, acid is dosed to control scaling due to $CaCO_3$ and antiscalants (ASs) for the other compounds. More recently, ASs able to control $CaCO_3$ scaling are being used on their own or in combination with acid. ASs allow significant super saturation, enabling higher rate of recoveries in particular in brackish water plants. The advantages of higher rate of recoveries in brackish water RO plants are:

- Reduction in the use of raw water.
- Reduction in the capacity of the pre-treatment.
- Lower energy consumption.
- Potentially lower cleaning frequency and smoother operation.

The main interests of the plant operators are to: run the plant smoothly without any problem and to maximize the RO system recovery, enabling the costs related to energy and chemicals to be reduced. Most reverse osmosis membrane plants dose AS to prevent precipitation of sparingly soluble salts. In general, dosing of AS retards the precipitation of sparingly soluble compounds, allowing significant super saturation [2]. The scale inhibition capability of AS depends on its chemical structure, molecular weight, active functional group, and pH [3]. Also, various types of ASs are available in the market, of which the recent bio-based environmentally friendly AS is also gaining popularity in the water industry [4]. It is obvious that suppliers of ASs, as well as operators, do not want to take any risk of scaling to avoid (irreversible) damage to RO membranes.

The actual applied AS doses are based on recommendations by suppliers. These recommendations are most likely based on their own prediction methods: seeded growth experiments, AS prediction software, and experience (pilot testing, full-scale plant testing). Consequently, the prediction for safe recovery is a combination of experience (trial and error) and science. In establishing the required dose, (some) AS suppliers also make use of the "induction time" as a criterion. Although the dosing of AS retards the precipitation of sparingly soluble compounds, but higher dosing might have environmental issues related to the concentrate disposal, economical issues because of high chemical cost and management issue due to the strong environmental regulations.

This paper presents the induction time data measured for the concentrate solution with and without AS dose from the BWRO plant in the Netherlands. Another important aspect of this study is to make some recommendation either to optimize AS dose or to increase the system recovery, based on the induction time experimental results.

1.1. Induction time

Induction time refers to the period preceding the start of "measurable" crystallization in a supersaturated solution [5]. The longer the induction time the lower the probability of scaling. However, no (generally accepted) criteria for safe induction times are available. The concept in the induction time method is to detect the start of crystal growth. Therefore, this method can give an early warning to the reverse osmosis plant operator, that scaling will start to occur. Nevertheless, other methods, such as seeded growth will detect scaling after a growth of crystal. However, for the induction time, detection of scaling depends on the availability of sensitive tools, such as pH, turbidity meter, calcium selective electrode, etc. In this

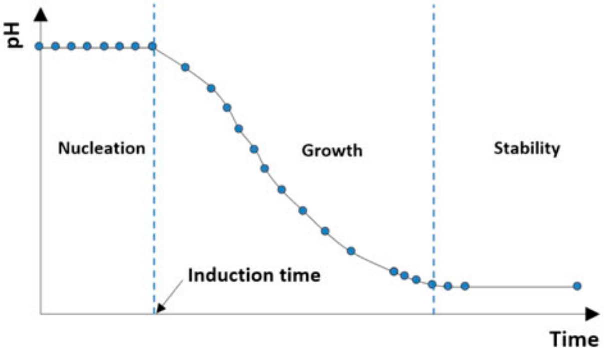


Fig. 2. Concept of induction time to monitor $CaCO_3$ scaling.

study, we focus on the precipitation of $CaCO_3$, thus we decide to consider pH as a tool to determine the induction time. The principle behind the $CaCO_3$ precipitation is expressed as follows: when water is supersaturated with $CaCO_3$ the equilibrium illustrated in Eq. (1) shifts to the right.

$$Ca^{2+} + 2HCO_3^- \leftrightarrow CaCO_3 \downarrow + CO_2 + H_2O \tag{1}$$

It can be noted that 1 mol of Ca^{2+} ion react with 2 mol of HCO_3^- ion forming the precipitation of one mole of $CaCO_3$ and release of one mole of CO_2. To simulate the process in the RO systems, the reactor was completely closed meaning any formed CO_2 did not escape from the reactor. As a result, the concentration of HCO_3^- ion will decrease and CO_2 will increase, resulting in the decrease of pH of the solution, as described by Eq. (2).

$$pH_i = (pK_1)_i + \log\frac{[HCO_3^-]_i}{[CO_2]_i} \tag{2}$$

Fig. 2 illustrates how the induction time can be defined by plotting pH vs. time. It can be clearly noticed by three different stages, such as nucleation, growth, and stable. The induction time is considered as the time required for the change of nucleation phase to the growth phase. Various factors play a role on scaling, such as pH, temperature, growth inhibitors, super saturation, etc. [6].

2. Materials and methods

2.1. Description of the BWRO plants

The induction time experiments were performed with the RO concentrate collected from the two brackish water plants in the Netherlands. The schematics of the plants are presented in Fig. 3.

The operating data for both plants are presented in Table 1.

2.2. Materials

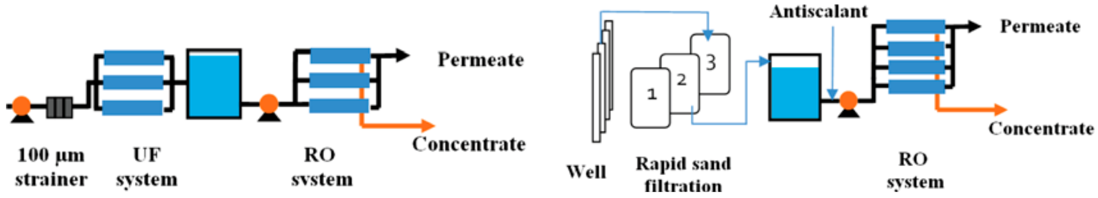
The following materials were used to measure the induction time:

2.2.1. pH meter

The induction time measurement using pH was performed with a highly sensitive pH meter (CyberScan 6000) with nominal accuracy of 0.001. The pH probe was connected in the lid of the experimental reactor. The pH was continuously monitored over

Table 1
Plant operating conditions for treatment plants "A" and "B"

		Plant operating data	
Parameters	Units	Plant A	Plant B
Plant capacity	m^3/h	140	70
Recovery	%	80	80
Feed temperature	°C	6.6	11
Feed conductivity	μS/cm	480	623
Concentrate conductivity	μS/cm	2,100	3,115
Antiscalant		A	B
AS dose	mg/L	1.5	1.25



Plant A Plant B

Fig. 3. Schematics of the treatment plants.

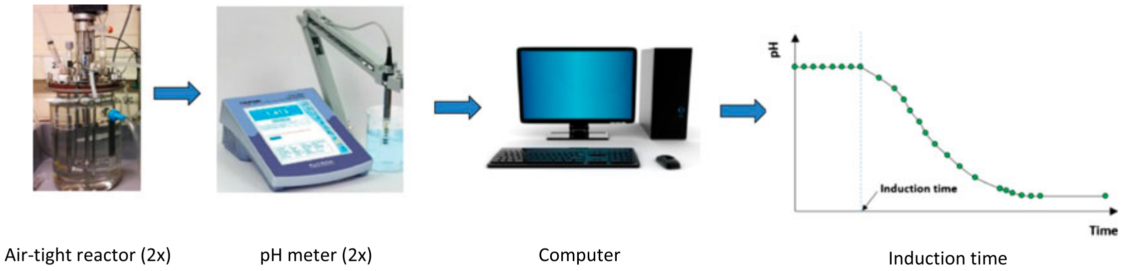


Fig. 4. Experimental setup for induction time measurement.

time offline using the memory of the instrument. The interval of pH recording could be adjusted in the pH meter. Temperature probe was connected to each experimental reactor and the pH meter. It allows correcting the pH at temperature difference since the pH calibration is at 25˚C and the temperature inside the reactor is around 20–23˚C.

2.2.2. Reactors

The "air tight" double-glass reactor with a capacity of 3.1 L (Applikon) was used for the induction time experiment. The reactors were cylindrical in shape with 24 cm in height and 12 cm internal diameters. The reactor was closed with a metallic lid to avoid the escape of CO_2 into air. The lid has the options to insert different probes, such as pH, temperature, conductivity meter, air/overflow, and NaOH addition if necessary. The reactor was equipped with a mechanical stirrer whose rpm could be adjusted. The stirrer allows mixing to ensure the homogenization of the solution. The reactor was connected with feed lines and drainage lines for filling and emptying, respectively.

The schematic diagram of the experimental setup is shown in Fig. 4.

In this experimental setup, the pH is continuously monitored. Induction time was considered as the time required for clear change in slope between the nucleation and growth phase.

2.3. Experiment

From plant A, RO concentrate with AS dose was only collected and from plant B, RO concentrates with and without AS dose were collected in an air-tight double wall reactor, and was equipped with a double-paddled shaft mechanical stirrer. Plant A operator did not allow the AS dose to be interrupted, thus it was not possible to collect RO concentrate without AS dose. The AS at plant "B" was shut down at one pressure vessel for about 7 min to flush the AS system prior to collect RO concentrate without AS dose.

The collected concentrate solution was transported to UNESCO IHE for continuous monitoring to determine the induction time. Initial pH of the concentrate solution was recorded at the plant location and then continuously monitored during the transportation to the laboratory of UNESCO-IHE. During sample transportation, the temperature of RO concentrate inside the reactor increases from 6.6 to 23˚C for plant A and from 11 to 23˚C for plant B. However, in the laboratory, the temperature was controlled to around 20–23˚C using a thermostat. The solution inside the reactor was continuously stirred at 150 rpm to ensure the homogeneity of the solution. The change in the pH of the solution was continuously recorded using the pH meter.

Batch tests are only simulating the behavior of the concentrate in the RO system. Hence, measured induction time will be a function of feed water characteristics only, such as pH, ionic strength, etc. Nevertheless, real-field conditions, such as the presence of the membrane, membrane spacer, flow rate, and the detention time will greatly influence scaling in the RO system and cannot be simulated by such experiments. Yet, in practice, if the average residence time of given water within a module is less than the induction period obtained from batch experiments for a specific salt e.g. $CaCO_3$, it is considered that the scale would not have time to form [7].

2.4. Calculation of saturation of $CaCO_3$

PHREEQC (US Geological Survey), a computer program written in the C programming language, was used in this research to calculate the saturation index (SI). This program is based on an ion-association aqueous model and has capabilities for speciation and saturation-index calculations and batch-reaction.

2.5. Experimental conditions XRD

X-ray powder diffraction (XRD) patterns were recorded in Bragg-Brentano geometry in a Bruker

Table 2
Feed and concentrate concentration at 80% recovery

	Plant A		Plant B	
Parameters	Feed water, concentration, mg/L	Concentrate concentration at 80% recovery, mg/L	Feed water, concentration, mg/L	Concentrate concentration at 80% recovery, mg/L
Ca	47−54	235−270	107−116	535−580
Mg	7	35	9–10	45−50
Na	31−36	155−180	26−30	130−150
Cl	45−50	225−250	33−37	165−185
HCO_3^-	106−118	530−590	246−263	1,230–1,315
SO_4^{2-}	54−60	270−300	100	500
C	<1−1.5	<5−7.5		
F	0.22−0.24	1.1−1.2	0.074−0.079	0.37−0.39
K	6.4−7.3	32−36.5	4.17−4.76	20.85−23.8
P (PO_4)	0.015−0.016	0.076−0.079		
N	2.9−3.5	14.5−17.5	0.88−2.3	4.4−11.5
Sr			0.36−0.38	1.8−1.9
Ba			0.08−0.09	0.4−0.45
Color (Pt)	11–14	55−70	0. 8−3.6	3.9−18.1
Si (SiO_2)	0−0.033	0−0.16	2.5−2.6	12.4−12.7

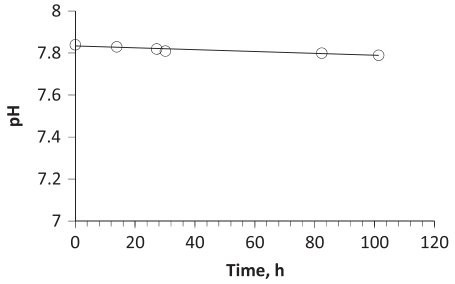


Fig. 5. pH changes over time for the RO concentrate (plant A) measured with AS dose (1.5 mg/L) and $SI_{calcite} = 1.2$.

D8 Advance diffractometer equipped with a Vantec position sensitive detector and graphite monochromator. Data collection was carried out at room temperature using monochromatic Co Kα radiation ($\lambda = 0.179026$ nm) in the 2θ region between 10° and 110°, step size 0.041° 2θ. Step time 2 s. The sample was placed on a Si {5 1 0} substrate and rotated during measurement. Data evaluation was done with the Bruker program EVA.

3. Results

3.1. Scaling potential

The maximum and minimum feed and concentrate concentration at 80% recovery for both plants A and B are presented in Table 2, respectively. The concentration for Fe and Mn was below the limit of detection i.e., < 0.01 (Mn) and < 0.05 (Fe).

Quality parameters were analyzed in the feed water and calculated for the concentrates by taking into account 80% recovery and assuming 100% rejection of the ions by the RO membranes.

The potential for scaling of calcite, aragonite, and dolomite, when operating at a recovery of 80%, was calculated using the PHREEQC computer program. The saturation Index (SI) was used as a criterion for

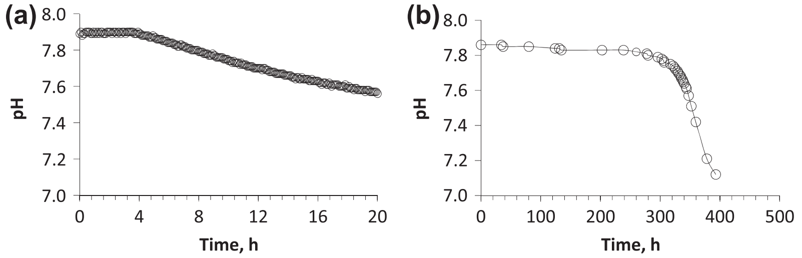


Fig. 6. pH changes over time of RO concentrate for plant B (a) without AS and (b) with AS (1.25 mg/L and $SI_{calcite} = 1.94$).

the scaling potential. In the calculation maximum Ca^{2+}, Mg^{2+}, and HCO_3^- concentrations were used based on the available data for each of the plants. The measured pH values in the concentrates were used as well (see Figs. 5 and 6). Furthermore, SI was calculated for various temperatures:

- For plant A: 2.5°C (min. temperature in the plant), 6.6°C (max. temperature in the plant), and 23°C (laboratory temperature).
- For plant B: 11°C (max. temperature in the plant) and 23°C (laboratory temperature).

The following conclusions can be drawn from the results presented in Tables 3 and 4:

- The concentrates show super saturation with calcium-based compounds, such as calcite, aragonite, and dolomite for both plants A and B.
- The saturation index (SI) for calcite at laboratory temperature 23°C was found to be almost the same for plant B (1.94) and for plant A (1.20).
- The scaling potential increases with the temperature due to the increase in the super saturation level. As the temperature increases, the nucleation rate increases and thus increases the scaling potential [8]. Therefore, the variation on the temperature greatly influenced the stability of the membrane concentrate.

Table 3
Plant A: SI for potential scaling phases calculated at initial pH 7.84, max. Ca^{2+}, Mg^{2+}, and HCO_3^- and at various temperatures

Phase	Symbol	Temperature		
		2.5 ℃	6.6 ℃	23 ℃
Aragonite	$CaCO_3$	0.98	1.01	1.05
Calcite	$CaCO_3$	1.14	1.17	1.20
Dolomite	$CaM_g(CO_3)_2$	1.25	1.32	1.5

Table 4
Plant B: SI for potential scaling phases calculated at initial pH = 7.86, max. Ca^{2+}, Mg^{2+}, and HCO_3^- and at various temperatures

Phase	Symbol	Temperature	
		11 ℃	23 ℃
Aragonite	$CaCO_3$	1.64	1.8
Barite	$BaSO_4$	1.07	0.81
Calcite	$CaCO_3$	1.79	1.94
Dolomite	$CaMg(CO_3)_2$	2.67	3.17

3.2. Induction time for brackish water RO concentrate

As expected, the induction times measured for the RO concentrates in both plants turned out to be strongly affected by dosing of AS. The results from batch test experiments for real RO concentrate with and without AS dose are shown in Figs. 5 and 6 for plant A and B, respectively.

3.2.1. Plant A

For plant A, the measured induction time with AS dose was found to be longer than 100 h as shown in Fig. 5. In this plant, we could not measure the induction time for RO concentrate without AS dose, as the plant operator did not allow the AS dose to be interrupted. The experiment was stopped after 100 h due to the interruption in recording pH.

3.2.2. Plant B

In this plant, the induction time was measured for both scenarios i.e., with and without AS dose as in Fig. 6. By measuring the induction time with and without AS, the factor between the two measured induction times could be calculated. The summary of the preliminary induction time measurement is presented in Table 5. The results revealed that the ratio of induction time measured for the RO concentrate with and without AS was about 70.

In both plants, no scaling occurred, which indicates that an induction time of 280 h (plant B) and more than 100 h (plant A) coincides with no scaling of $CaCO_3$ at the existing temperatures. The two applied ASs are quite different, the one used in plant A is a phosphonate-based and in the plant B the AS is bio-based. The results indicate that induction time measurements in the RO concentrate might be a useful tool in verifying predictions of safe recovery (no scaling occurring) as a function of AS dose.

The measured induction time with AS dose for plant "B" was longer than plant "A" despite the higher saturation index for different precipitating phases and even lower AS doses. The two ASs are quite different, the one used in plant A is a phosphonate and in plant B is bio-based. The induction time was measured at laboratory condition where there was higher temperature than at the real plants. For instance, the temperature of RO sample inside the

Table 5
Results of induction time measurements for plant B

Plant	AS dose (mg/L)	$SI_{calcite}$	Induction time, h	Ratio for induction time with and without AS dose
Without AS	0	1.94	~ 4	~70
With AS	1.25	1.94	~280	

laboratory reactor was increased during transportation from 6.6 to 23˚C for plant A and from 11 to 23˚C for plant B. This increase in temperature has increased the saturation index of calcite from 1.17 to 1.20 (plant A) and from 1.79 to 1.94 (plant B). Therefore, the induction time when measured in a real plant could be even longer than that measured at laboratory conditions. It is because nucleation kinetics increases with temperature. Likewise, the nucleation rate of specific crystal morphology depends on the super saturation and nucleation energy. And, super saturation is also a function of temperature i.e., as the temperature increases the super-saturation also increases.

Furthermore, the operating conditions at two different plants possibly could also affect the induction time which can be further explained as below:

- The flow conditions i.e., the cross flow velocities in each of the plants and other operational parameters might have a direct impact on the concentration polarization and agglomeration of particles either with each other or the membrane [9].
- The types of AS used in both the plants were quite different which might have influenced the induction time in each plant. In general, AS can be categorized into two groups: nucleation inhibitors and growth inhibitors. Therefore, based on the type of AS used, measured induction time will also be affected which needs to be further investigated as part of the research.
- Increase in ionic strength at constant super saturation decreases the induction time [10].
- Difference in feed water quality for instance, the presence of sulfate, magnesium, and natural organic matter could also play a role in inhibiting $CaCO_3$ scaling [11–14] as they alter the kinetics of precipitation of $CaCO_3$ as these may act as a natural AS.

Similarly, the measured induction time without AS dose at plant B shows that the RO concentrate was stable for about 4 h even at a higher temperature at around 23˚C. Therefore, it raises the question about how high safety factors on induction time are needed for preventing $CaCO_3$ scaling in RO systems. As a whole, the detention time of the bulk concentrate is most likely in the order of only 1–1.5 min and in the last elements not more than several seconds [7].

3.3. Induction time at varying AS dose and fixed recovery

The induction times measured in both the plants with AS dose were too long. It raises the question for optimization of either AS dose or increase the system recovery. Therefore, induction time experiment at varying AS doses and fixed recovery were performed by simulating the synthetic brackish water in the laboratory. The concentrated synthetic brackish water solution at 90% recovery (TDS of 4,690 mg/L) was prepared using inorganic salts. The AS tested were phosfonate-based AS which was applied at various doses from 5 to 20 mg/L at concentrate which corresponds to 0.5–2 mg/L to feed stream. All experiments were performed for a short duration in a closed glass reactor with an initial pH of the solution about 8.5 ± 0.1 and SI for $CaCO_3$ ranges from 2.55 to 2.65. The initial pH of the solution was adjusted by adding 1 M NaOH solution.

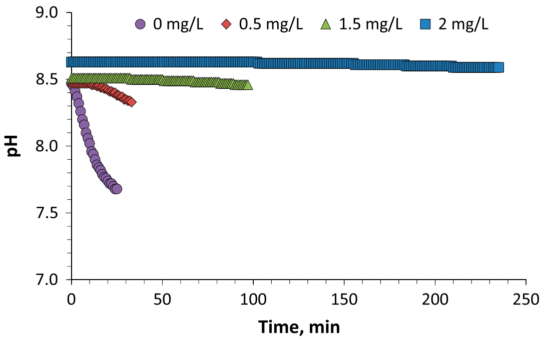


Fig. 7. pH changes over time of synthetic BWRO concentrate at 90% recovery with various AS doses 0, 0.5, 1.5, and 2 mg/L at feed solution which corresponds to 0, 5, 15, and 20 mg/L applied at concentrate solution at 90% recovery.

The effect of various AS doses on suppression of $CaCO_3$ scale can be clearly visualized in Fig. 7. For instance, the measured induction time for concentrated solution at a recovery of 90% was very short at no AS dose and 0.5 mg/L AS dose at feed. While, at higher dose of 1.5 and 2 mg/L, the solutions were stable for more than 100 and 250 min, respectively. The result showed that dose of applied AS is an important factor for increasing the induction time for $CaCO_3$. The significance of the result is that it provides an input on the optimization of the recovery and the AS dose. However, a substantial amount of research is still needed on the optimization of the plant operation e.g. longer test on varying the AS dose at a fixed recovery and monitoring whether scaling occurs or not, varying at different RO recovery rates, testing at different locations with different compositions, and temperatures of the feed water, etc.

3.4. XRD and SEM analysis

The induction time experiments for plant B were left to run until precipitation occurred. The precipitate was collected from the bottom of the reactor for two analyses: XRD which gives information about the structure of the crystalline materials, and scanning electron microscope (SEM) which was used to produce images by scanning the precipitate with a focused beam of electrons. The XRD and SEM results for both samples are presented in Figs. 8(a) and (b), respectively.

In Figs. 8(a) and (b), the measured XRD patterns are shown in black, while the colored lines give the peak positions and intensities of the identified phases. All the patterns are background-subtracted, meaning the contribution of air scatter, possible fluorescence radiation, and filter material is subtracted.

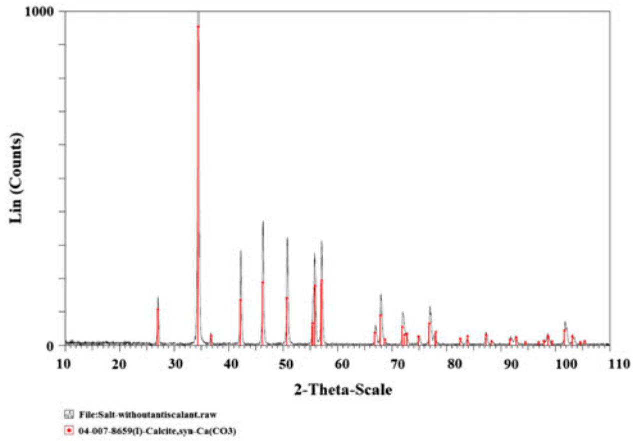


Fig. 8(a). XRD analysis for the precipitated salt without AS dose for plant B.

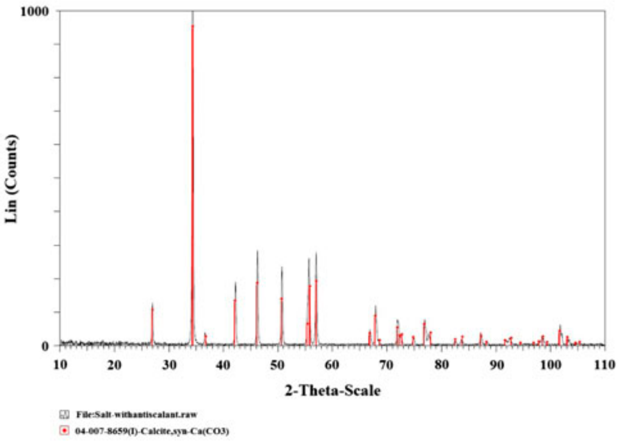


Fig. 8(b). XRD analysis for the precipitated salt with AS dose for plant B.

The result from XRD analysis shows that calcite was only the form of precipitation found in samples from plant "B" for both cases i.e. with and without AS dose. No other forms of $CaCO_3$ and other compounds were detected.

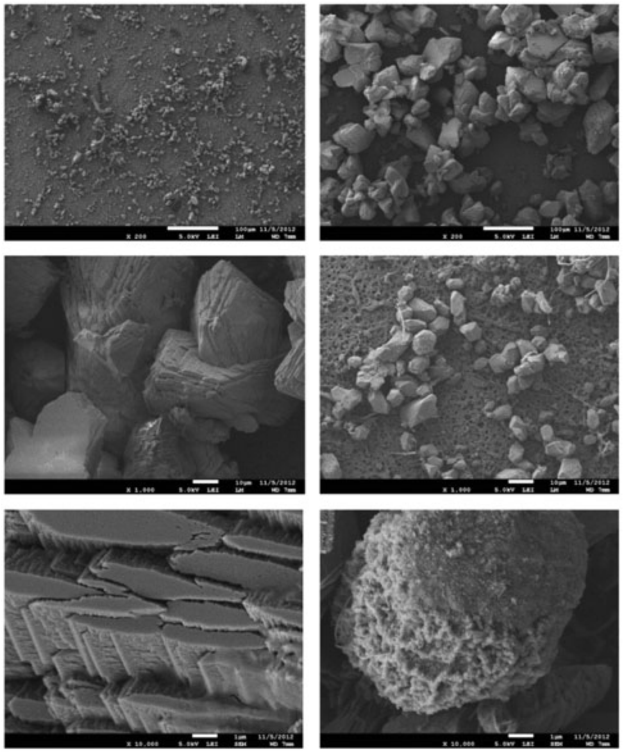


Fig. 9. SEM pictures for the final crystal phases with AS (right) and with no AS (left) for plant B (top 200× magnification, middle 1,000× magnification and bottom 10,000× magnification).

The SEM results from Fig. 9 showed that the crystals formed without ASs are found to be more clustered and larger (left figure) than with AS (right figure). In addition, the shape of the crystals formed with AS has more rounded edges than without AS.

4. Conclusions

The main objective of this study was to measure the induction time of RO concentrate from two different RO plants in the Netherlands with and without the presence of AS. The following conclusions can be drawn from the research:

- The induction time values were the following: for plant A were more than 100 h (sample with AS) and for plant B were 4 h (sample without AS) and 280 h (sample with AS).
- The RO concentrate from plant B was stable almost for about 4 h even without AS dose. This is much higher than the required detention time for the bulk RO concentrate i.e. about 1–1.5 min. Therefore, it raises the question about how high safety factors on induction time do we need for RO systems? This also requires further research aiming to support on optimizing the AS dose that RO plants are currently dosing.
- The result from XRD analysis shows that calcite was only the form of precipitation found in samples from plant "B" for both cases i.e. with and without AS dose. No other forms of $CaCO_3$ and other compounds were detected.
- Concentrate samples of both plants showed scaling of calcite only after exceeding the measured induction time.

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