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Deposition of calcium and magnesium from RO concentrate by means of seed crystallization and production of softened water for technical purposes

Alexei Pervov*, Alexei Andrianov

Department of Water Supply, Moscow State University of Civil Engineering. Moscow, Russia, Tel. +7(985) 766 76 44; email: waterlab@yandex.ru (A. Pervov), Tel. +7(499) 183 36 29; email: alexei-waterlab@yandex.ru (A. Andrianov)

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ABSTRACT

Existence of reverse osmosis (RO) concentrate effluents often creates a problem of its handling and utilization. This limits industrial applications of this technique for drinking and industrial water supply. The article presents the results of development and field testing of new proposed technique that removes calcium carbonate and magnesium hydroxide from RO retentate streams using seeding techniques. This new concentrate utilization technique uses RO membrane unit operated in circulation mode. Seed crystals of calcium carbonate and magnesium hydroxide are added to the circulating concentrate flow. Calcium and magnesium deposit on the seed crystals due to their concentrations increase in membrane channel. The test unit is tailored with membrane modules that have “open channels” and eliminate trapping of particles by a spacer and increase of flow resistance. Seed crystals are suspended in circulating concentrate stream. The seed crystallization procedure provides up to 90% removal of hardness and alkalinity from RO concentrate and decrease concentrate total dissolved solids due to calcium and magnesium salts precipitation. RO retentate thus becomes a softened water that can be blended with product water to produce technical water used for cooling or heating, as well as for drinking purposes. The developed process does not require softening reagents that are used in stoichiometric amounts to deposit calcium carbonate and magnesium hydroxide during chemical softening. Sodium hydroxide is used only to produce seed crystals and to maintain pH of feed water at value of 8.8–9.0. Consumption of caustic by new seed crystallization process constitutes 2%–4% of the stoichiometric amount.

Keywords: Concentrate treatment; Reverse osmosis; Seed crystallization; Water softening

1. Introduction

Reverse osmosis (RO) techniques are widely used for different desalination and deionization needs in a lot of industrial applications due to their high purification efficiencies and low chemical consumption. But the existence of retentate often creates serious ecological problems. The existence of concentrate streams in RO and NF systems often limits their application area. Drinking and industrial water stations often have no possibilities to handle large amounts of concentrate that cannot be discharged into surface water sources [1].

Increase of recovery and reduction of retentate flow causes increase of foulant concentration, supersaturation of sparingly soluble salts and their precipitation in membrane channels. High values of concentrate flow are mainly attributed to the “fear” of exceeding the solubility limits of main sparingly soluble constituents such as calcium sulphate and calcium carbonate in RO concentrate. The use of efficient antiscalants prevents scaling and enables us to significantly increase recoveries.

But the use of antiscalants does not exclude existence of retentate streams that cause environment pollution by salts.

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* Corresponding author.

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At present time, a lot of research is devoted to development of tools to radically decrease concentrate flows to utilize them [3–7]. Therefore, to increase recovery, we should reduce concentration values of main components of sparingly soluble salts.

In modern practice, the idea to withdraw excessive calcium from concentrate is being implemented through the use of fluidized reactor [6]. Supersaturated solution (RO concentrate) enters reactor. To ensure high supersaturation and efficient calcium carbonate precipitation, caustic is injected in the feed water entering reactor [5,6]. These tools require the use of special materials (seeds, pellets) and their handling, as well as additional facilities (reactors) that require substantial capital investments and operational costs.

Control of scale formation in membrane channels can be also implemented by means of "seed" precipitation as described by Harries [7]. Addition of seed particles into RO concentrate was implemented by Harries [7] to decrease calcium sulphate supersaturation values in concentrate and to increase recoveries. These techniques described in [7] used tubular RO membrane modules. It is well known that fouling and scaling is dependent on membrane channel geometries [2,8]. Tubular modules are not widely applied due to their high capital costs and low membrane surface per unit volume of the module. Spiral wound membrane configuration is recognized as the most efficient and economically available. But the strict pretreatment requirements and the "fear" to increase recoveries limit the use of these modules in newly developed reagent-free techniques.

Main disadvantages of spiral wound modules are attributed to the presence of separation spacer mesh in the feed channel as it traps fouling particles and increases cross-flow channel resistance [9,10]. The places (spots) where the mesh connects to the membrane surface provide "dead areas" without cross flow that result in high concentration increase at the membrane surface, initiating crystal formation (Fig. 1(a)).

To overcome spiral wound disadvantages, a lot of attempts were undertaken to improve channel geometry [2,8]. Development of an "open channel" modules is a part of long complicated research devoted to solution of scaling control and concentrate utilization [8].

Technical considerations to improve membrane channel geometry and avoid formation of "dead areas" are shown in Fig. 1(c). To withdraw calcium carbonate from retentate and increase recovery, "seeding" technique was developed that consists of addition of "seed" crystals in RO feed water, precipitation of calcium carbonate on the "seeds", sedimentation and removal of excessive seeds as a sludge. This approach is described in previous publications and reports [1,11].]

In a study by Pervov [11], the results of estimation of caustic consumption to reduce calcium in RO concentrate was presented. The research was aimed at investigation of seed growth mass influence on crystal growth rates during their weight increase. The obtained results indicated that caustic consumption to reduce calcium did not exceed 2% of stoichiometric amount required for conventional water softening carried out using pellet reactor or clarifier [6].

The described seed procedure provides precipitation of calcium carbonate but does not reduce magnesium in RO concentrate and thus does not decrease total hardness. The goal of present study was to investigate possibilities to precipitate not only calcium carbonate but also magnesium hydroxide. It is well known that precipitation of magnesium hydroxide requires high pH values and as described in a previous study by Pervov [11] seeding techniques do not provide suitable conditions for this process. Meanwhile, results of experiments described in our earlier research articles disclosed some interesting results that outlined some ways to reduce magnesium hydroxide. First, production of seed crystals required addition of sodium hydroxide to RO concentrate to initiate calcium carbonate crystallization. SEM analysis of seed crystals showed the presence of magnesium among seeds [11–13]. To withdraw both calcium and magnesium from RO concentrate by means of seeding techniques, seed crystals should contain magnesium hydroxide crystals.

Magnesium hydroxide precipitation greatly depends on pH value. Simultaneous calcium carbonate and magnesium hydroxide scaling occurs, but calcium carbonate growth consumes carbonate ions and changes pH resulting in magnesium hydroxide growth blockage. It seems reasonable that in order to accelerate magnesium hydroxide growth, calcium carbonate deposition should be controlled. This can be done through the use of antiscalants.

Main goals of the conducted research were:

- to study factors that influence seed crystals growth, such as mass of seed crystals, pH and antiscalants presence;
- to study possibilities and conditions of magnesium removal from RO concentrates and
- to determine main industrial characteristics of softening process design, such as recovery, caustic dose, antiscalant dose and seed crystal amounts.

2. Experiments

Seed growth scaling experiments were conducted in the Water treatment laboratory of Department of Water Supply, National Research Moscow State University of Civil Engineering.

Experimental program consisted of three experimental series:

Series 1: Preliminary antiscalant performance comparison tests conducted with Moscow tap water. Selection of the most efficient product basing on scaling rates evaluation. Influence of pH and antiscalant doses.
Series 2: Comparative studies of antiscalant efficiencies during seed crystals growth. Influence of seed amount, antiscalant type and pH on seed crystals growth.
Series 3: Investigation of influence of pH on precipitation of magnesium hydroxide during "seeding" techniques.

All seed crystal growth tests were performed using a laboratory membrane unit shown in Fig. 2. The feed solution (tap water or model solution) is poured in the feed water tank (1) and delivered to membrane module via centrifugal multistage pump (2). The trans-membrane pressure, cross flow and recovery rate are adjusted by valves (10, 11 and 12) and controlled by pressure gauges (6) and flow meters (7 and 9). Seed crystals were added to the feed tank (1) in the beginning of each experiment run.

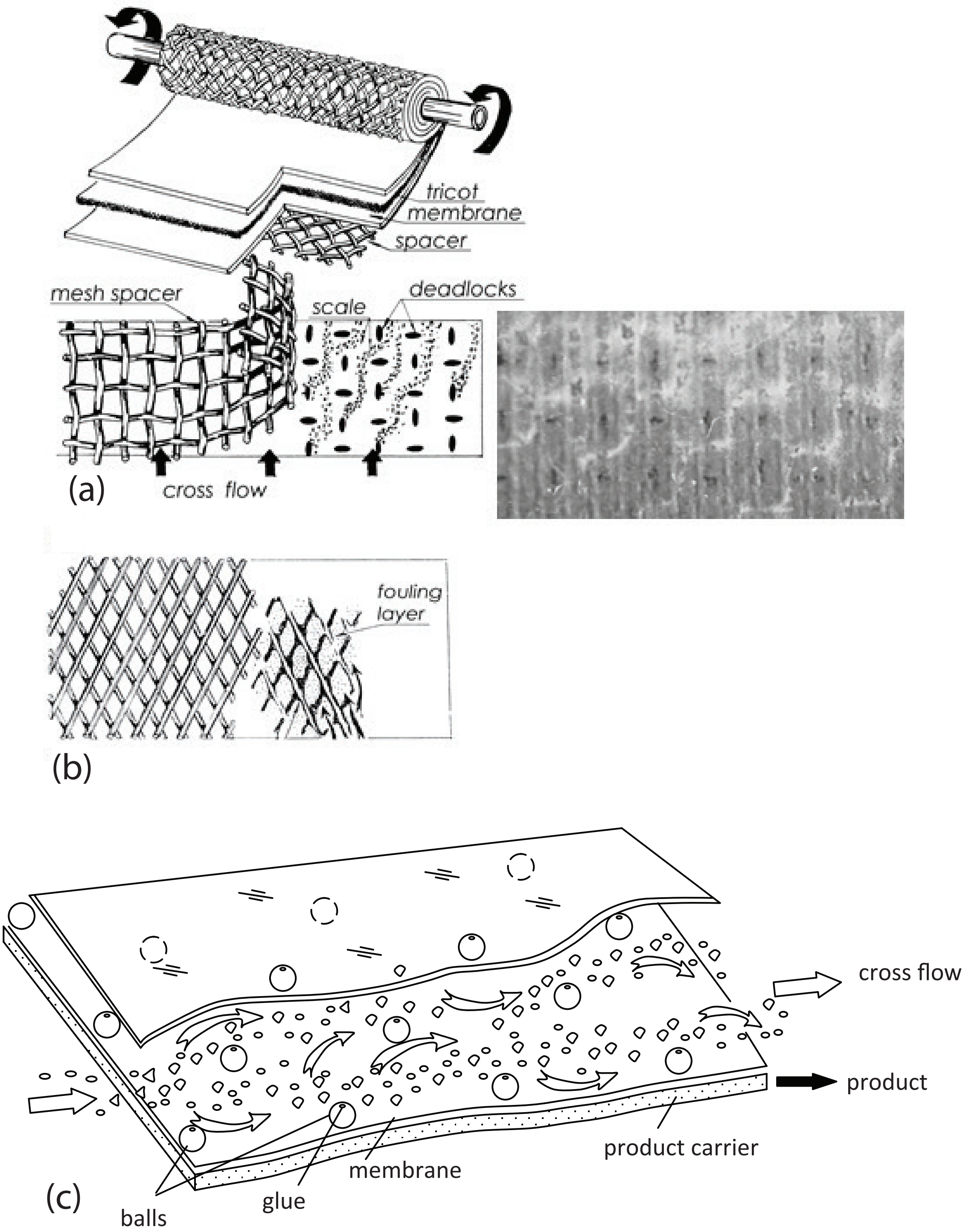


Fig. 1. Fouling and scaling: influence of spacer. Formation of crystals in dead areas. (a) Formation of scale crystals; (b) particle trapping and fouling layer formation; (c) an "open channel" configuration: seed crystals in cross flow.

All seed growth tests were conducted in circulation mode whereby reject flow (concentrate) is returned to the feed water tank (1) and permeate is collected in separate tank (4). The transmembrane pressure was maintained at 7.0 ± 0.2 bar. The product flux, depending on tap water temperature, was varied from 10 to 15 L/h. The volume of feed solution was 50 ± 2 L. Concentrate flow is kept constant at 100 ± 10 L/h. Experiments were carried out with Moscow tap water. During experimental period, the tap water had quite stable quality and total dissolved solids (TDS, ppm) of 246–266 ppm, total hardness of 3.1–3.4 meq/L (155–170 ppm of $CaCO_3$), total alkalinity of 2.5–2.9 meq/L, calcium of 3.2–3.5 meq/L, pH of 7.75–8.2, sulphates of 10–13 mg/L, chlorides of 8–10 mg/L, magnesium of 1.1–1.2 meq/L.

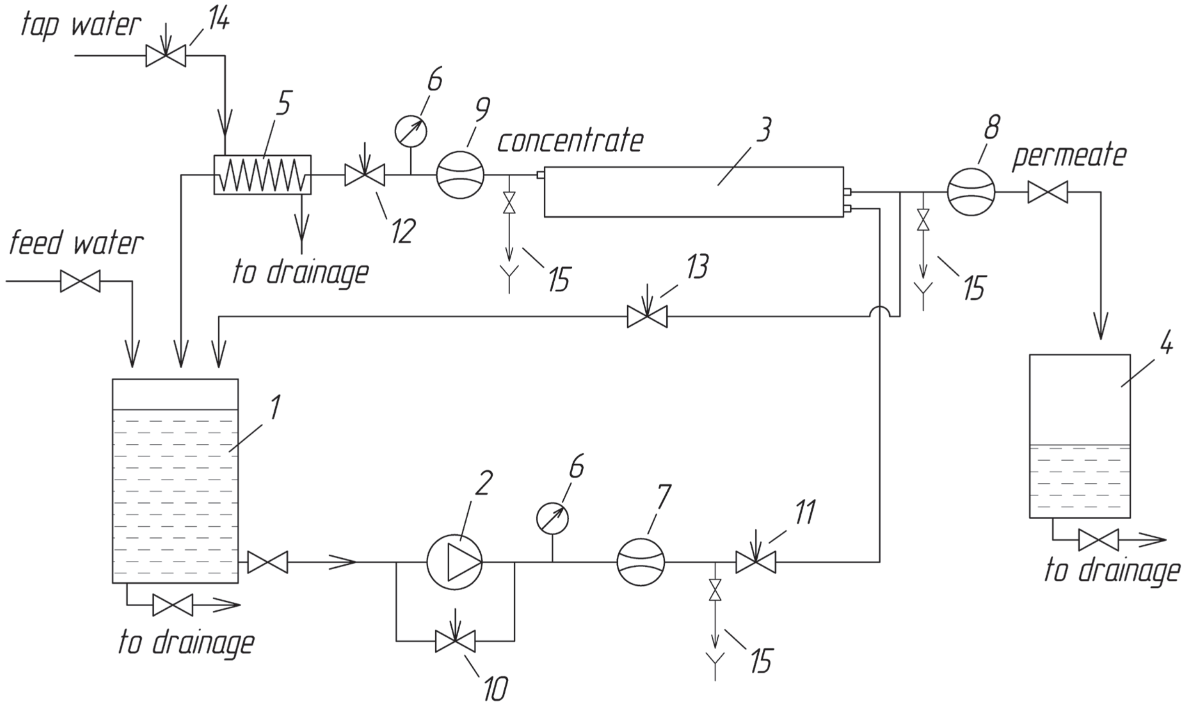


Fig. 2. Schematic diagram of laboratory RO unit for membrane scaling tests: 1 – feed water tank; 2 – pump; 3 – spiral wound membrane module; 4 – permeate tank; 5 – heat exchanger; 6 – pressure gauge; 7 – feed water flow meter; 8 – permeate flow meter; 9 – concentrate flow meter; 10 – by-pass adjusting valve; 11 – feed water adjusting valve; 12 – concentrate adjusting valve; 13 – cooling water adjusting valve; 14 – sampler.

PASP (polyaspartic acid sodium salt) was used in a number of experiments to control calcium carbonate growth. The selection of PASP is explained by the interest in “green” antiscalants that do not contain phosphorous. These antiscalants demonstrate high scale control efficiencies [12,13] and are recommended for use in many desalination applications. PASP has been kindly supplied by Shandong Taihe Water Treatment Technologies Co. Ltd., (Zaozhuang, Shandong Province, P.R. China) and analysed by NMR and dynamic light scattering technique.

Temperature, TDS (conductivity), pH values, total hardness, total alkalinity and calcium concentrations were determined in all samples. Conductivity and temperature were controlled by a laboratory conductivity meter Cond 730 (WTW inoLab®); pH values were determined – using laboratory pH meter HI 2215 (Hanna Instruments, Vohringen); total alkalinity was determined – by titration with HCl; total hardness and calcium was determined – by EDTA titration. Magnesium concentrations were determined as a difference between total hardness and calcium concentrations.

The amounts of scales of $CaCO_3$ or $Mg(OH)_2$ (expressed as Ca^{2+} and Mg^{2+} in meq) accumulated on seed crystal surface were calculated as differences between initial amounts of calcium in feed solutions and sums of calcium amounts in concentrates (circulating solution) and permeates [8]. Seed crystals growth rates were determined according to the technique described in the literature [8–13].

To perform “seeding” experiments, new “open channel” spiral wound modules of 1812 standard (12″ length and 1.8″ in diameter) were manufactured using the BLN-type flat sheet membranes, manufactured by CSM (Korea). Modules were manufactured in accordance with configuration shown on Fig. 1 using rolling machine “Model no. RS 4040A” supplied by “Hydrocomponents and Technologies”, CA. Membrane surface in test modules was 0.5 m^2.

The idea to utilize concentrate in RO applications is presented in Fig. 3. The seed material can be produced by sedimentation of fresh crystals after addition of caustic solution to RO concentrate (Fig. 3(a)). Figs. 3(a) and (b) show flow diagrams of the process. After seed crystals were added to the feed water tank (1), feed water was pumped to membrane module (3). Product water was withdrawn from the system into product collect tank (5) [11].

Concentrate was returned back to the feed water tank (1). Calcium and carbonate concentration values were constantly growing during the test run. This provided driving force for crystal growth. After feed water volume was decreased by 6–8 times the test unit was stopped and crystals were sediment in the feed water tank. Calcium carbonate was withdrawn from concentrate and concentrate salinity was reduced by the value of precipitated calcium carbonate (in meq). Concentrate after sedimentation can be mixed (blended) with product water. As a result, softened water with reduced salinity, hardness and alkalinity values is obtained. These “seeding” techniques using spiral wound “open channel” modules were developed by us earlier and already described in the literature [11].

In our experiments, feed water (well water, tap water) volume was reduced by six times and stoichiometric amount of 1 N caustic solution equivalent to total hardness was added to concentrate collected in flush water tank (5) to

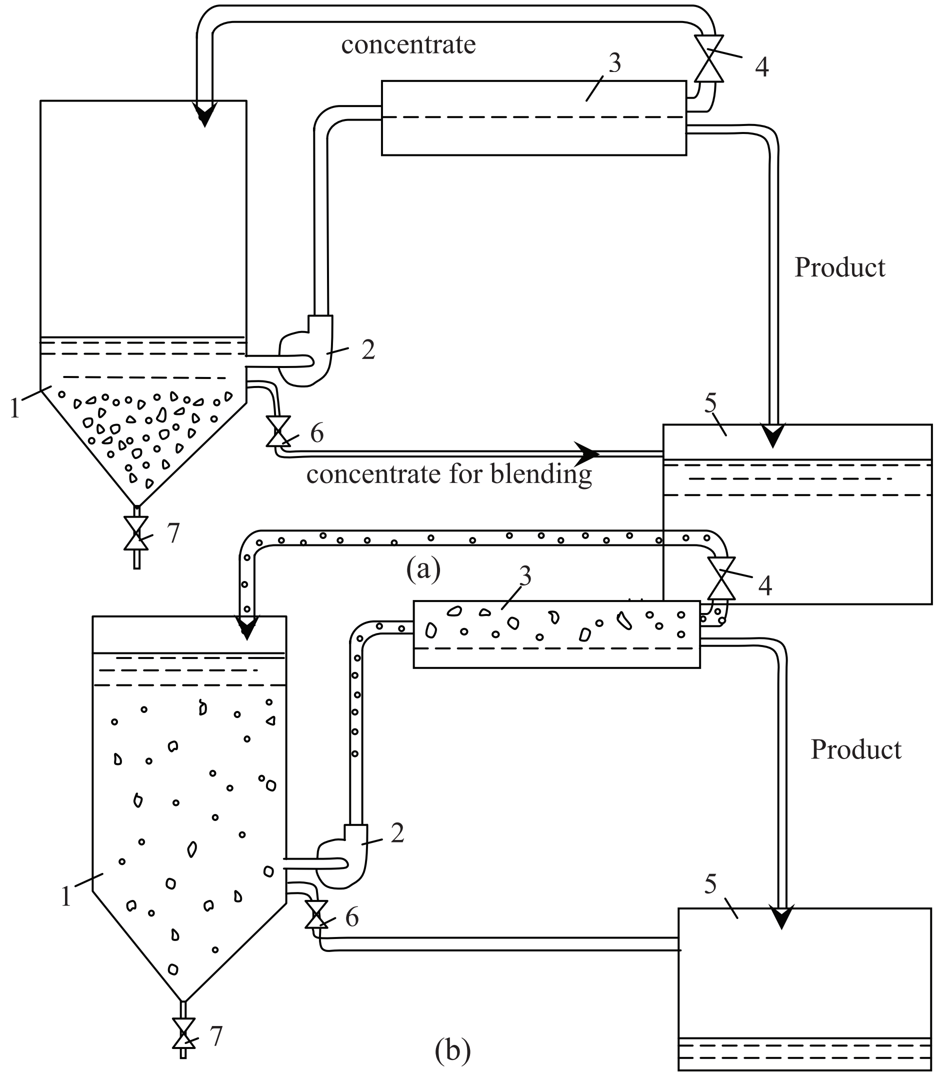


Fig. 3. The idea to utilize concentrate in RO and a flow diagram of the test process to withdraw calcium carbonate from RO concentrate: (a) "seed" production; (b) "seed" circulation. 1 – feed water tank; 2 – feedwater pump; 3 – RO membrane "open channel" module; 4 – pressure regulation valve; 5 – product water tank; 6 – concentrate valve.

form calcium carbonate and magnesium hydroxide. After nucleation occurred and crystal formation phase was finished, crystals were sedimented during 3–4 h, collected and added to the feed water tank. Throughout the test run, feed water was constantly concentrated and samples were collected that correspond to concentration factor values 2, 3, 4, 6 and 8. During seed crystals sedimentation no evidence of crystal growth was noticed: growth of calcium carbonate and magnesium hydroxide is dependent greatly on pH values. Consumption of carbonate and hydroxyl ions decreases alkalinity and pH values that in turn decreases supersaturation and blocks further crystal growth. These crystals grow only under supersaturation conditions that are provided by increase of concentrations of all species in reverse osmosis membrane channel [11].

Small crystals that sediment on membrane surface during membrane operation can be easily flushed from the membrane surface by applying "flushings" carried out by opening of pressure regulation valve (13) (Fig. 2). In some test runs, antiscalant was added to the feed water tank (1). Antiscalant dose was 10 ppm. Caustic solution was added to the feed water to increase pH value. 0.7–1.5 meq of NaOH were added to reach 8.9–10.5 pH values.

Fig. 4 shows results of experimental test runs. Fig. 4(a) shows concentration values of calcium and magnesium (in the feed water tank (1), Fig. 3) vs. concentration factor (initial feed water volume/volume in the tank (1)). The amount of calcium carbonate seed crystals added to the feed water tank (1) was 5 g (150 meq). To produce magnesium hydroxide seed crystals, 200 meq of caustic were added to concentrate to precipitate 200 meq of seed crystals (150 meq of calcium carbonate and 50 meq of magnesium hydroxide).

To investigate influence of antiscalant on seed crystal formation and growth, SEM microphotos of "fresh" seeds and seed crystals on different steps of seed growth were made throughout each test run. Scale crystals were "flushed" off membrane surface after opening pressure control valve (12) (Fig. 2) and further pressure drop.

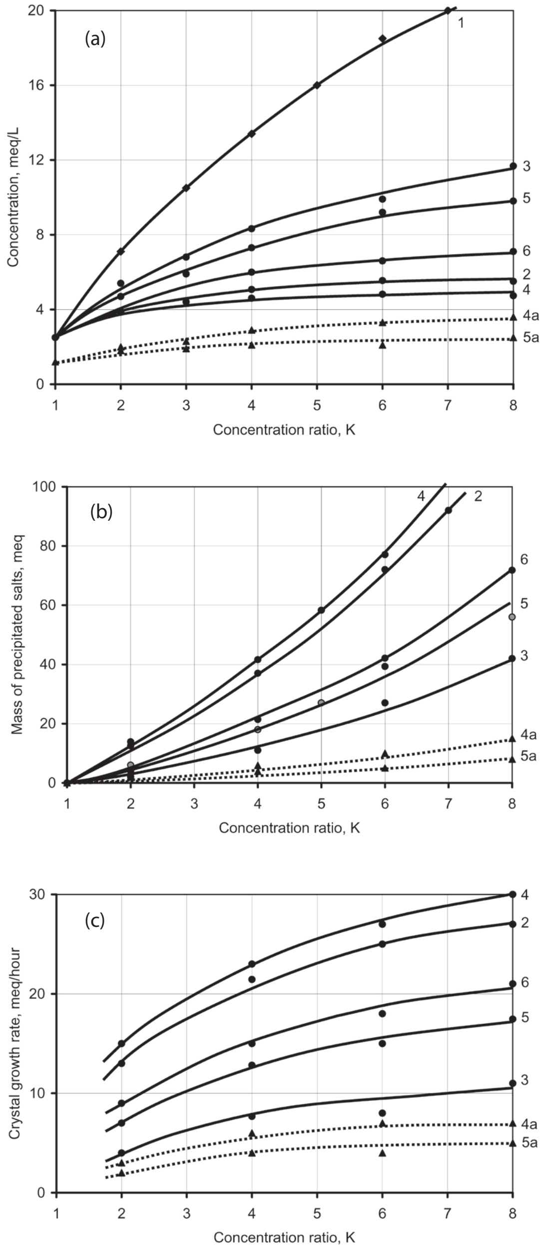


Fig. 4. “Seed” crystals growth experiments. Influence of pH and added seed mass on scaling rate: (a) calcium and magnesium ion concentration values vs. concentration coefficient (*K*); (b) calcium carbonate and magnesium hydroxide precipitated amount vs. concentration coefficient (*K*); (c) calcium carbonate and magnesium hydroxide precipitation rate vs. concentration coefficient (*K*); 1 – Moscow tap water without “seed” crystals; 2 – “seed” dose 150 meq, without antiscalant addition, pH = 7.7, seed crystals: calcium carbonate; 3 – “seed” dose 150 meq, PASP dose is 10 ppm, pH = 7.7, seed crystals: calcium carbonate; 4 – “seed” dose 200 meq, without antiscalant addition, pH = 8.9, seed crystals: calcium carbonate and magnesium hydroxide; 5 – “seed” dose 200 meq, PASP dose is 10 ppm, pH = 8.9, seed crystals: calcium carbonate and magnesium hydroxide; 6 – “seed” dose 150 meq, PASP dose is 10 ppm, pH = 8.9; 4a – “seed” dose 200 meq, PASP dose is 10 ppm, pH = 8.9; magnesium hydroxide growth; 5a – “seed” dose 200 meq, without antiscalant addition; pH = 8.9; magnesium hydroxide growth.

Water after flushing was collected in a separate tank and further filtered using laboratory membrane 0.8 micron microfilter cell. Microfilters with rejected seed crystals were rinsed with distilled water and dried at 50°C. Then microfilters were tested using “Quanta 250 FEI” microscope. Microscope testings were conducted under low-vacuum conditions, voltage was 12.5 and 15 kV, large field detector was applied and magnitude was ×500 – ×5,000. Seed crystal mass composition was investigated using energy disperse X-ray spectroscopy techniques “GENESISAPEX 2 EDS System with APOLLO X SDD EDAX”.

3. Discussion of results

Results of seed growth investigations are shown in Figs. 4 and 5. Fig. 4(a) shows dependencies of calcium and magnesium ions concentration values in the circulating feed water solution (in the tank (1)) vs. the concentration factor (*K*) value, which is determined as a ratio of feed water volume to concentrate volume, during a test run. Fig. 4(b) shows mass of accumulated calcium carbonate vs. *K*. Mass of accumulated calcium carbonate and magnesium hydroxide was calculated as differences between total calcium and magnesium in the feed water in the beginning of experiment and total calcium and magnesium in water concentrate. Each concentration factor value corresponds to certain time period elapsed from the beginning of experiment. Calcium carbonate growth rates were calculated as tangents of slopes of accumulated mass vs. time curves (Fig. 5(b)). Addition of caustic to increase pH value also accelerates crystal growth. During seed growth experiments magnesium hydroxide precipitation was detected at high pH values. Fig. 4(a) shows magnesium ion concentration vs. *K* (curve 4) and magnesium hydroxide growth rate vs. *K* value is presented in Fig. 6(c). The driving force for magnesium hydroxide was supersaturation reached due to magnesium concentration increase and high pH value. To study magnesium hydroxide crystal growth together with calcium carbonate growth and its dependence on pH values and antiscalant presence, another experimental series was conducted. pH values in the feed water were 8.3 and 9.5. Intensive calcium carbonate growth reduces pH value in concentrate and magnesium hydroxide crystal growth. To control calcium carbonate crystal growth and reduce its influence on magnesium hydroxide formation as well as to facilitate magnesium hydroxide growth, we added antiscalant to the feed water tank. To test influence of antiscalant presence on magnesium hydroxide precipitation, PASP antiscalant was added to the feed water in certain test runs. PASP dose was 10 ppm. Results of testings are shown in Fig. 5. Fig. 5(a) shows dependencies of calcium and magnesium concentrations on concentration ratio *K*. Curves 1 and 2 describe calcium (1) and magnesium (2) ions concentration increase in the tap water in the presence of antiscalant and feed water pH value 9.5. Curves 3 and 4 describe calcium carbonate (3) and magnesium hydroxide (4) growth in the feed water after addition of seed crystals, antiscalant and caustic. pH of the feed water was 8.2. Curve 5 describes calcium concentration vs. *K* dependence for the case when feed water pH was 7.7, antiscalant was not added. In this case no evidence of magnesium hydroxide growth was observed (Fig. 5(a)). Seed crystals in all cases contained both calcium carbonate and

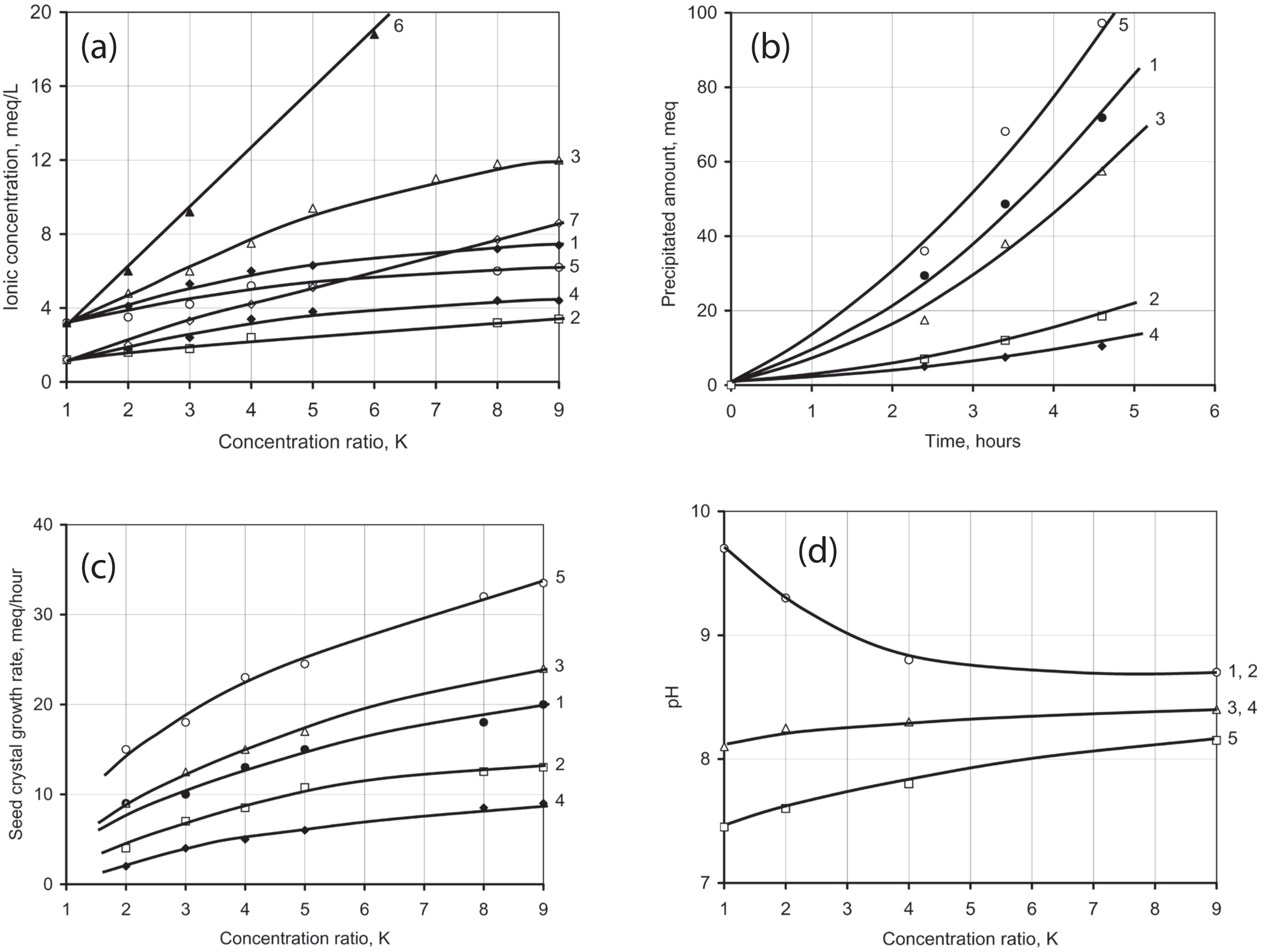
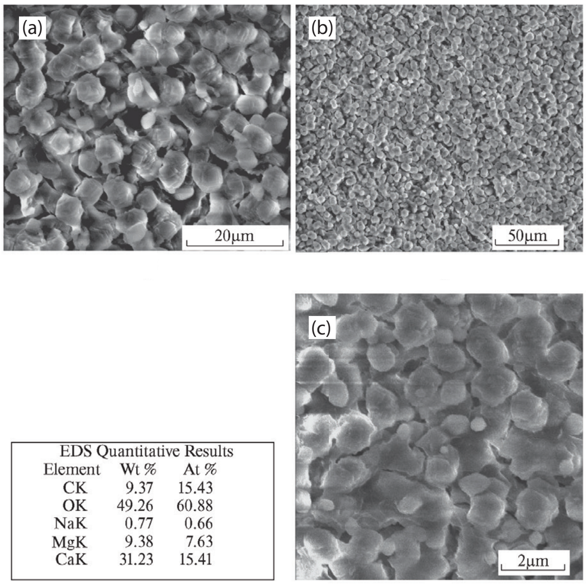


Fig. 5. Results of experimental study of pH and antiscalant influence on seed crystals growth: (a) calcium and magnesium concentration values vs. concentration ratio *K*; (b) amount of deposited calcium and magnesium (meq) vs. time (h); (c) calcium carbonate and magnesium hydroxide growth rate values vs. concentration factor, *K*; (d) pH values in the feed tank vs. concentration ratio, *K*; 1 – calcium, Moscow tap water, seed crystals amount: 200 meq, pH = 9.5; PASP dose – 10 ppm; 2 – magnesium, Moscow tap water, seed crystals amount: 200 meq, pH = 9.5, PASP dose – 10 ppm; 3 – calcium, Moscow tap water, seed crystals amount: 200 meq, pH = 8.2, PASP dose – 10 ppm; 4 – magnesium, Moscow tap water, seed crystals amount: 200 meq, pH = 8.2, PASP dose – 10 ppm; 5 – calcium, Moscow tap water, seed crystals amount: 200 meq, pH = 7.7, without antiscalant addition.

magnesium hydroxide. Fig. 5(b) shows the calculated values of amounts of precipitated calcium carbonate and magnesium hydroxide. Fig. 5(c) describes results of scaling rates calculations vs. *K*. It is obvious that magnesium hydroxide growth rate is higher in the presence of PASP. Antiscalant addition decreases calcium carbonate growth rate, but facilitates formation of magnesium hydroxide. This can be explained by reduction of pH value during calcium carbonate growth. This pH raise decreases magnesium hydroxide growth. Fig. 5(d) shows dependencies of pH of concentrate on *K* throughout test runs. There are certain doubts about the nature of magnesium deposits. Conventionally magnesium hydroxide is produced at high pH values (higher than 11). Magnesium can also be deposited as magnetite. But mass balance analysis has indicated that bicarbonate ions (alkalinity) have been spent to form only calcium carbonate. Formation of magnesium hydroxide is also confirmed by relationships shown in Fig. 5.

SEM photos were made to investigate influence of antiscalants presence and pH values on seed crystal growth. SEM photos of crystal are shown in Figs. 6 and 7, where presence of magnesium hydroxide is detected.

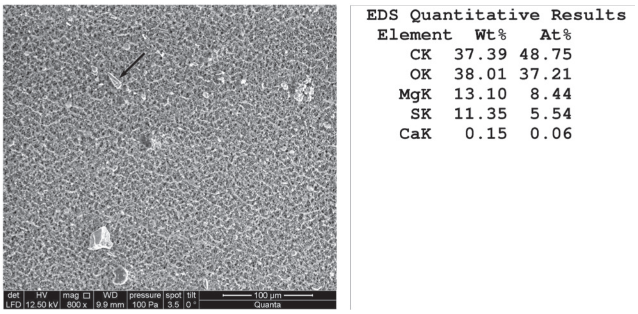
The consumption of sodium hydroxide and required amount of calcium carbonate and magnesium hydroxide seed mass can be determined using calculation tools described by Pervov in [11]. As calcium carbonate and magnesium hydroxide scaling rate values depend on pH, antiscalant dose and seed mass, it seems reasonable to control seed crystal growth and to maintain calcium carbonate growth rate to magnesium hydroxide growth rate ratio corresponding to [Ca]/[Mg] concentrations ratio in the feed water. This makes softening process efficient to reduce calcium and magnesium in proportion. To control this ratio, we should maintain pH value on certain level that corresponds to required Ca/Mg ratio in the feed water. According to Table 1, [Ca]/[Mg] ratio in the feed water equals 2.2. Fig. 5 shows that at pH value 9.5,



EDS Quantitative Results

Element	Wt %	At %
CK	9.37	15.43
OK	49.26	60.88
NaK	0.77	0.66
MgK	9.38	7.63
CaK	31.23	15.41

Fig. 6. SEM microphotographs of seed crystal after nucleation with different magnitudes ((a), (b), (c)) and results of X-ray spectroscopic analysis.



EDS Quantitative Results

Element	Wt%	At%
CK	37.39	48.75
OK	38.01	37.21
MgK	13.10	8.44
SK	11.35	5.54
CaK	0.15	0.06

Fig. 7. Microphotographs of magnesium hydroxide crystals flushed off RO module and results of X-ray spectroscopic analysis.

the ratio of calcium carbonate growth rate value to magnesium hydroxide growth rate value is 4. At pH = 8.2, this ratio value is 2.2. Fig. 8 shows the approach to determine pH value of the feed water to reach required [Ca]/[Mg] in the softened water.

Blending of product and retentate streams after "seed" growth enables us to produce water with lower content of magnesium, calcium and bicarbonate ions as well as lower TDS value than existing RO retentate. Table 1 shows an example of Moscow Tap water composition, product and concentrate, corresponding to recovery value 0.9, concentrate chemical composition after "seed" treatment, as well as examples of blending product water and retentate at different ratios.

4. Conclusions

(1) A new technique is described that enables us to increase recoveries and utilize RO concentrate that consists of calcium carbonate and magnesium hydroxide precipitation on the "seed" crystals and further blending of softened concentrate with RO product water. As a result, softened water is obtained with lower TDS and hardness than of the initial feed water;

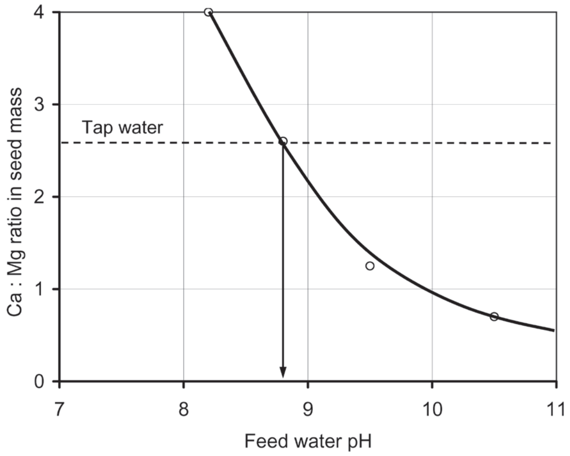


Fig. 8. Recommendation to select feed water pH to reach required [Ca]/[Mg] concentrations ratio in the softened water.

Table 1
Chemical composition of Moscow tap water, RO product water, RO concentrate after seed treatment, results of product and concentrate blending at different ratios

Component	Feed water	RO concentrate after seed treatment	RO product	Blending results, product : concentrate		
				10:1	2:1	1:1
Hardness, meq/L	3.2	9.7	0.12	0.99	3.25	4.9
Ca^{2+}, meq/L	2.3	7.0	0.05	0.7	2.5	3.5
Mg^{2+}, meq/L	0.9	2.7	0.04	0.27	0.9	1.35
$Na^+ + K^+$, meq/L	1.0	9.9	0.1	1.0	3.4	5.0
Alkalinity, meq/L	2.9	9.08	0.1	0.9	3.03	5.1
Cl^-, meq/L	1.0	9.3	0.07	0.94	3.2	4.7
SO_4^{2-}, meq/L	0.3	2.9	0.02	0.3	0.95	1.46
pH	8.48	8.5	7.1	–	–	–
TDS, ppm	260	1,492	18	151	497	756
Flow, %	100	10	90	100	60	20

(2) To remove magnesium from RO concentrate, seed crystals should contain magnesium hydroxide and feed water pH value during seed treatment should be 8–9.5;
(3) Intensive calcium carbonate growth on the seed crystals reduces pH value in concentrate flow and decreases magnesium hydroxide deposition;
(4) The presence of antiscalants in RO retentate significantly decreases rate of calcium carbonate precipitation on the “seed” surface and facilitates magnesium deposition on the seed crystals.
(5) Experimental relationships enable us to determine optimum values of feed water pH, recovery and “seed” doses to remove calcium carbonate and magnesium hydroxide from RO retentate.

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