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# Control of $CaSO_4$ scale formation in membrane distillation by seeded crystallization and in-line filtration

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ABSTRACT

Membrane distillation (MD) is a novel technology that can be used for zero liquid discharge (ZLD) or near-ZLD systems. However, a limiting factor affecting the effectiveness of MD for ZLD applications is membrane fouling due to scale formation. Accordingly, this study intended to develop technologies to alleviate scaling of MD membranes by applying seeded crystallization and in-line filtration. Experiments were carried out using a laboratory-scale direct contact MD (DCMD) system in a batch concentration mode. A saturated solution of $CaSO_4$ was used as a model scale-forming salt and its crystal particles were intermittently added to the feed tank as nuclei to induced seeded crystallization. In addition, a stainless steel mesh filter with the nominal pore size of 2 μm was applied as an in-line filtration to continuously remove suspended crystal particles from the recirculating brine. Results showed that the antiscaling effect of the seeded crystallization depends on the way of crystal particle addition. The in-line filtration was found to be also effective to retard fouling due to scale formation. The combination of seeded crystallization and in-line filtration, however, does not seem to have synergic effect.

*Keywords:* Zero liquid discharge; Membrane distillation; Crystallization; Scale formation; Fouling; Antiscaling; Seeding; In-line filtration

## 1. Introduction

Zero liquid discharge (ZLD) is defined as a treatment system in which no liquid effluent is discharged into natural water bodies, allowing complete elimination of the environmental impacts [1]. ZLD also involves use of treated wastewater, thereby leading to reduced water consumption [1,2]. ZLD can be implemented by reducing the volume of the wastewater effluent using various techniques, including membrane-based separation, evaporation, and

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crystallization [3–6]. ZLD technologies have attracted interest from the industrial sectors over the past decade [3,7–10] because of enforced regulations for wastewater discharge, public perception of environmental impact by industrial activities, and possibilities of contamination of drinking water supply [11].

Among a few technologies, membrane distillation (MD) is a novel approach that enables ZLD or near-ZLD systems [12,13]. MD is a thermal-driven separation process in which a hydrophobic membrane is used as a barrier between hot water and vapor [14]. One of the key features in MD technology is its ability to treat feed water with high salt concentrations [13]. Unlike reverse osmosis (RO), the operation of MD is limited by not osmotic pressure difference but vapor pressure difference [15]. This allows the application of MD for ZLD systems [12,16].

However, one of the limiting factors affecting the efficiency of MD in ZLD applications is fouling due to scale formation [14,15]. Scale formation is a process of formation of solid crystals from a uniform solution inside a process such as boilers, pipes, heat exchanges, water treatment equipment, and membranes [17]. Sparingly soluble salts such as $CaCO_3$, $CaSO_4$, $BaSO_4$, $SrSO_4$, and $SiO_2$ result in scale formation in water treatment systems [6,17,18]. Scale formation in membrane systems results in irreversible reduction in the permeability of membranes and damage of membrane structures [14,15]. Scale formation is a major challenge in the application of MD to ZLD systems because scaling potential of feed waters and brines to the ZLD systems is very high [15,18].

To alleviate the problems of scale formation, membrane crystallization may be considered. Membrane crystallization (MCr) is an innovative process consisting of MD and a crystallizer [19]. In MCr, pure water is produced as permeate from the MD process, while the concentrated solutes can be recovered as solids from the crystallizer [20]. Accordingly, MCr can be used in pharmaceutical industry to recover valuable compounds from liquid streams [21–24]. Moreover, MCr can continuously separate solutes from water, thereby allowing the operation of MD at high recovery without experiencing fouling due to scale formation [25]. However, little information is available on the design and operation of MCr for ZLD applications. The mechanisms of scale control in MCr have not been fully revealed yet.

Therefore, this study investigated the control of MD fouling due to scale formation by introducing the concept for MCr. Seeded crystallization and in-line filtration were attempted to continuously removal crystal particles from recirculating brine. The effect of seeding method and in-line filtration conditions on flux decline in a direct contact MD (MCD) system was experimentally examined and analyzed.

## 2. Experimental

### 2.1. Membrane and feed solution

Fat sheet membranes made of PVDF (GVHP, Millipore) were used for the MD experiments. The pore size, thickness, and porosity of the membrane are 0.22 and 125 μm, and porosity is 75%. A laboratory-scale MD module with the effective membrane area of 12 $cm^2$ was prepared prior to the experiments. Saturated $CaSO_4$ solution was used as the feed water, which was prepared using DI water and $CaSO_4$ (2,000 mg/L). The feed water was filtered using GF/C filters prior to the experiments.

### 2.2. Experimental setup

A laboratory-scale system shown in Fig. 1 was developed and used for measuring flux rejection during the MDCr operation. The system consists of a MD module, a crystallizer (feed tank), two recirculation pumps, a heat for feed solution, an electronic balance, a cartridge filter to separate suspended crystals in feed tank, and a cooler for distillate. The feed water was heated using a heater connected to a temperature sensor, allowing the automatic control of feed temperature. The vapor was transported by the pressure difference across the membrane to the permeate side and condensed inside the membrane module. The temperature of the permeate was maintained constant at 20°C using a water bath and a heat exchanger. The electric conductivities of feed water and distillate were continuously measured using a conductivity meter immersed into the feed and distillate tank. An electronic balance connected to a data logger was used to continuously measure the mass of distillate to calculate water flux through the membrane. The permeate flux was then is expressed in terms of time or concentration factor (VCF). The concentration factor, defined as a ratio of the feed volume to concentrate volume, indicates the extent of concentration:

$$\mathrm{VCF} = \frac{V_f}{V_c} = 1 + \frac{V_p}{V_c} \quad (1)$$

where $V_f$, $V_c$, and $V_p$ are the volume of initial feed, concentrate, and distillate, respectively. The operating conditions for MDCr are summarized in Table 1.

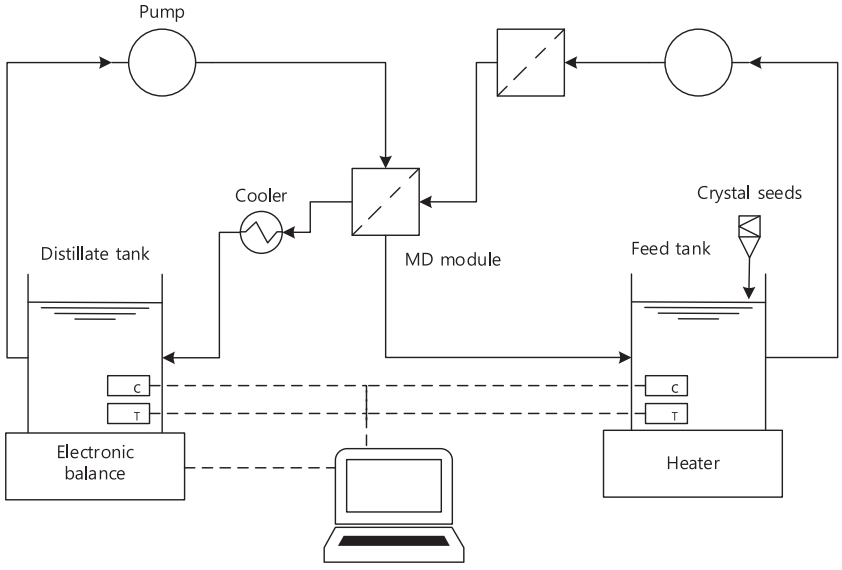


Fig. 1. Schematic diagram of experimental setup for MD.

Table 1
Operation conditions for MD experiments

| Items | Conditions |
|---|---|
| Operation mode | *DCMD* (*Direct Contact Membrane Distillation*) |
| Crossflow velocity | Feed 0.6 L/min–permeate 0.4 L/min |
| Feed | $CaSO_4$ 2,000 mg/L solution |
| Distillate | D.I water |
| Temperature | Feed side (60˚C), permeate side (20˚C) |
| Seeding | Calcium sulfate 200 mg (5 wt% total solute) |
| Cartridge filter | Type: wire mesh, sintered 5 layer wire mesh<br>Pore size: 2 μm |

### 2.3. Control of scale formation

The following methods to control scale formations were attempted in this study:

(1) Seeded crystallization.
(2) In-line filtration.
(3) Seeded crystallization combined with in-line filtration.

For seeded crystallization, 200 mg of $CaSO_4$ particles were directly added to the feed tank during the MD experiments. The purpose of seeded crystallization was to accelerate the growth of bulk crystals to reduce the growth of surface crystals on the MD membrane. The particles were added at different concentration factors: 1.2, 1.25, and 1.3. A cartridge filter with a pore size of is 2 μm was applied to examine the effect of in-line filtration, which aimed at the removal of suspended crystals from the solution to prevent fouling due to cake formation. Moreover, the combination of seeded crystallization and in-line filtration was applied. In this case, the particles were added at two concentration factors: 1.15 and 1.25.

### 2.4. Liquid entry pressure (LEP)

The liquid entry pressure (LEP) is an important property for MD membranes because it is related to the resistance against the pore wetting [14]. If the feed water exceeds the LEP of the membrane, it penetrates into the pores, leading to pore wetting and poor rejection for solutes. In this study, the LEP of the membranes were experimentally measured using a device shown in Fig. 4. The system consists of a high-pressure nitrogen cylinder, a pressure regulator, a pressure sensor, a gas chamber, a water chamber, a, diagram, and a membrane holder. The pressure applied to the membrane increases stepwise until the

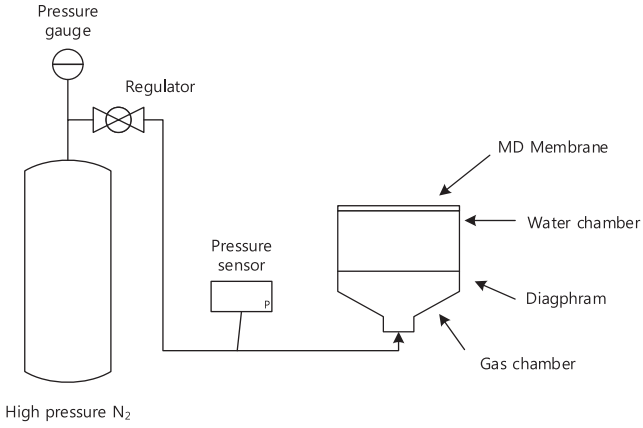


Fig. 2. Schematic diagram of experimental setup for liquid entry pressure measurement (LEP).

water penetrates the membrane. The measurements were triplicated to check the reproducibility of the results (Fig. 2).

## 3. Results and discussion

### 3.1. Effect of seeded crystallization on scale formation

To begin, experiments were conducted to examine the effect of scale formation on flux in MD. The flow rates of feed and distillate were 600 and 400 mL/min, respectively. The temperature difference between feed and distillate was 40˚C. A saturated $CaSO_4$ solution was used as feed solution. The changes in MD flux with VCF or operation time are shown in Fig. 3. The black symbols indicate the results of MD experiments without any antiscaling techniques (control). Initially, the flux maintained constant at approximately 19 kg/$m^2$ h. At VCF of 1.29, however, the flux began to decrease, which is attributed to the fouling due to scale formation of $CaSO_4$. The flux decline occurred after 16 h of MD operation. It should be noted that the scale formation is a function of concentration (or VCF) and time. Although the solution is supersaturated, it takes time to start scale formation by rapid crystal growth [17]. Accordingly, the VCF and time in which flux decline occurs are important to characterize the scaling potential of the feed water, which are defined as critical VCF ($VCF_c$) and critical time ($T_c$). In Fig. 3, the $VCF_c$ and $T_c$ for the control experiment were 1.29 and 16 h.

In Fig. 3, the results of the seeded crystallization during the MD operation are also presented. Three MD runs were carried out and the crystal particles were added to the feed solution at VCFs of 1.2, 1.25, and 1.3, respectively. The amount of crystals added to

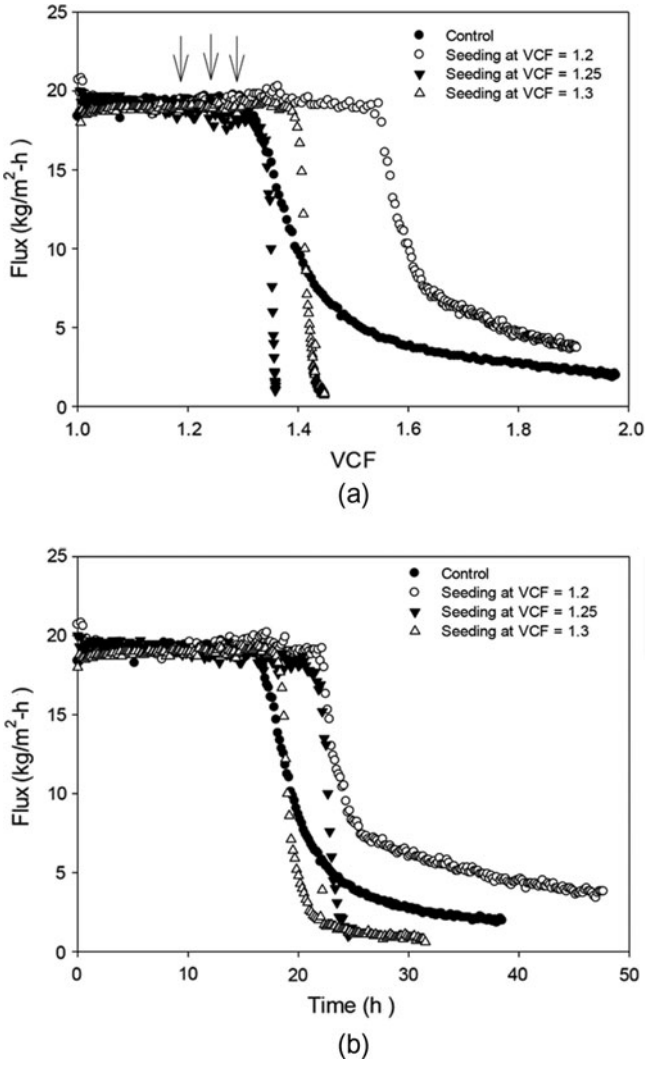


Fig. 3. Effect of crystal seeding on MD flux declines due to scale formation: (a) flux vs. VCF and (b) flux vs. time.

the solution was 200 mg, which corresponds to 5% of total amount of $CaSO_4$ in the solution. As shown in Fig. 3, the addition of crystals at VCF = 1.2 was effective to retard flux decline due to scale formation. The flux was constant until the VCF reached at 1.54 (or $VCF_c$ = 1.77). The time before the onset of flux decline increased to 22.4 h. This suggests that the seeded crystallization was effective to alleviate fouling by scale formation.

However, the effect of the seeded crystallization appears to be sensitive to the point of the crystal addition. When the crystals were added at VCF = 1.25, the antiscaling effect was reduced. The flux decreased beyond the VCF of 1.54 and the time of 20.4 h. This is clearly less efficient than the case with the crystal addition at VCF = 1.2. The antiscalant effect was even

Table 2
Effect of crystal seeding on feed turbidity and LEP

| | No seeding | Seeding at VCF 1.2 | Seeding at VCF 1.25 | Seeding at VCF 1.3 |
|---|---|---|---|---|
| Turbidity (NTU) | 12.89 | 124.25 | 257.89 | 407.1 |
| LEP (bar) | 2.17 | 2.06 | 2.04 | 2.21 |

lower when the crystals were added at VCF = 1.3. In this case, the flux decreased beyond the VCF of 1.31 and the time of 18.3 h. These results suggest that the crystals should be added at an appropriate VCF or time to effectively induce bulk crystallization. Once the process of fouling begins on the membrane surface, it seems to be less efficient to use crystal particles for seeded crystallization. In other words, the seeded crystallization should be carried out before scale formation on MD membrane occurs.

Since the particles are added to the solution, they may affect the integrity of the MD membrane surface, leading to a loss of functionality of the membrane. Accordingly, the LEP values of the MD membranes were measured after the experiments. As shown in Table 2, the turbidity of the final solution increased by adding the seed particles. For instance, it increased up to 407 NTU by the seeding at VCF = 1.3. However, the LEP was not changed by the seeding of particles. Under all conditions, the LEP values range from 2.04 to 2.17. Accordingly, it can be concluded that the addition of seed particles did not affect the integrity of the membrane. Of course, a longer operation of MD may result in different results. However, under the conditions considered here, the adverse impact of seeding seems to be negligible.

### 3.2. Effect of in-line filtration on scale formation

Another approach to decrease the effect of scale formation on fouling is the in-line filtration. During the MD operation, the crystals are formed not only on the membrane, but also in the bulk solution. Moreover, the crystals formed on the membrane surface may be re-suspended by the cross-flow effect. According, the application of in-line filtration may reduce the amount of total crystals formed inside the MD system. In this study, a wire mesh-type metal filter prepared by five (5) sintered layers was used for continuous filtration of recirculating concentrate inside the MD system. The nominal pore size was 2 μm. Since the crystals suspended in the bulk solution are larger than this size, it was believed that most particles were removed by this filter.

As shown in Fig. 4, the in-line filtration appears to be effective to lessen flux decline due to scale formation. The flux began to decrease after VCF of 1.77, which corresponds to 30.4 h. Compared with the case without in-line filtration (control), the point of the startup of scale formation is retarded. Moreover, it seems that the in-line filtration is better than the seeded crystallization (Fig. 3) in terms of antiscaling effect. These results suggest that the continuous removal of crystals in the bulk solution is effective to reduce the fouling propensity by the scale formation. In other words, the crystals exist in the bulk solution

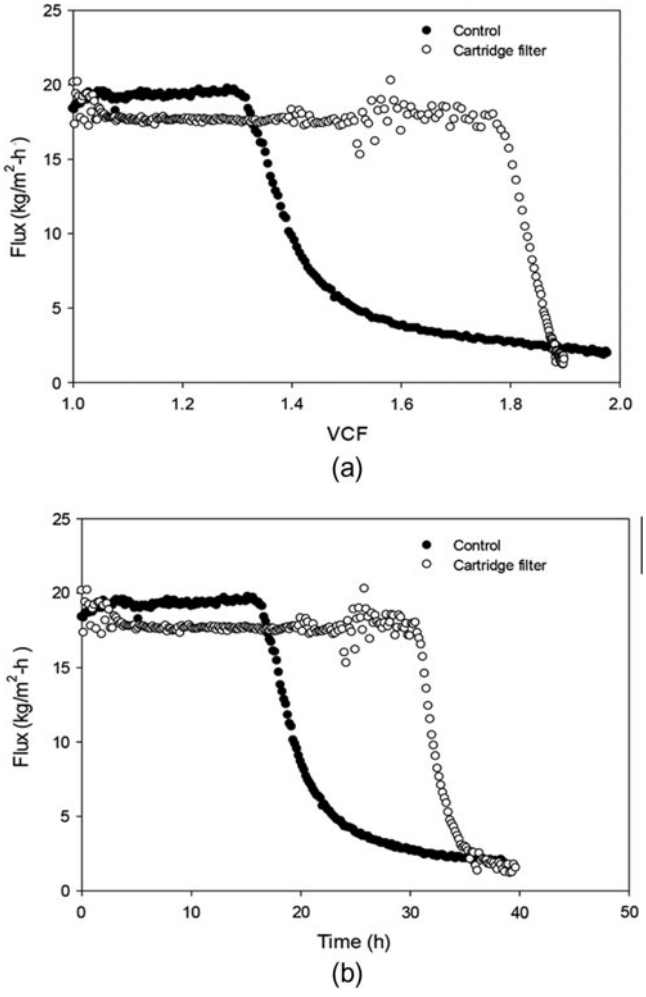


Fig. 4. Effect of in-line filtration on MD flux decline due to scale formation: (a) flux vs. VCF and (b) flux vs. time.

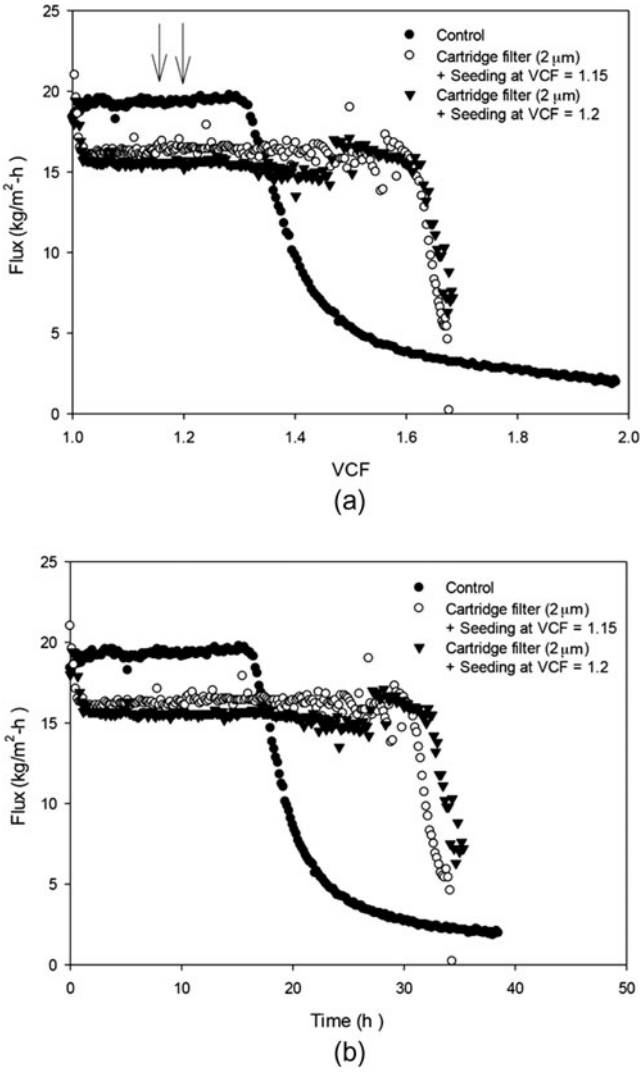


Fig. 5. Effect of crystal seeding followed by in-line filtration on MD flux decline due to scale formation: (a) flux vs. VCF and (b) flux vs. time.

result in fouling to a certain degree. Of course, the fouling could not be completely prevented by the in-line filtration, implying that not only bulk crystals but also surface crystals cause MD fouling.

### 3.3. Combination of seeded crystallization with in-line filtration

Since both seeded crystallization and in-line filtration were effective to reduce fouling potential of the $CaSO_4$ saturated solution in the MD system, a set of experiments were attempted to combine the two methods at the same time. In these cases, the MD system was operated with the in-line filter (the metal cartridge filter) and then the crystal particles were added at a certain VCF. The results are shown in Fig. 5. As expected, the combination of the seeded crystallization and in-line filtration was also effective to decrease fouling due to scale formation. Although the initial flux was lower, the flux decline did not occur until the VCF reached 1.58, which corresponds to about 30 h. Moreover, unlike the case of the seeded crystallization without the in-line filtration, the point of the crystal addition did not affect the antiscaling effect.

It should be noted that the combination of two methods is not much more effective than the in-line filtration. As shown in Fig. 4, the $VCF_c$ and $T_c$ were 1.77 and 30.4, respectively, for in-line filtration. However, the $VCF_c$ and $T_c$ for the combined method were 1.6 and 30. This suggests that there is no synergic effect of combining two methods. This may be attributed to the mechanisms of these two methods: The seeded crystallization preferentially induces bulk crystallization, leading to decreased rate of surface crystal formation on the membrane surface. On the other hand, the in-line filtration removes suspended crystal particles from the solution, resulting in net reduction in the amount of

Table 3
Comparison of $T_c$ and $VCF_c$ for different antiscaling methods

| Antiscaling methods | $T_c$ (h)[a] | $VCF_c$[b] | $T_c \times VCF_c$ |
|---|---|---|---|
| Control | 16.4 | 1.29 | 21.1 |
| Cartridge filter (2 μm) | 30.38 | 1.77 | 53.8 |
| Seeding at VCF = 1.2 | 22.4 | 1.54 | 34.6 |
| Seeding at VCF = 1.25 | 20.4 | 1.31 | 26.7 |
| Seeding at VCF = 1.3 | 18.3 | 1.39 | 25.4 |
| Cartridge filter (2 μm) + seeding at VCF = 1.15 | 29.4 | 1.58 | 46.5 |
| Cartridge filter (2 μm) + seeding at VCF = 1.2 | 31.9 | 1.6 | 51.0 |

[a]$T_c$: time required before the onset of flux decline due to scale formation.
[b]$VCF_c$: VCF required before the onset of flux decline due to scale formation.

Table 4
Comparison of total water production and average flux for different antiscaling methods

| Antiscaling methods | Total water production (L) | Average flux (kg/m$^2$ h) |
|---|---|---|
| Control | 0.516 | 11.2 |
| Cartridge filter (2 μm) | 0.70 | 14.8 |
| Seeding at VCF = 1.2 | 0.693 | 12.1 |
| Seeding at VCF = 1.25 | 0.519 | 17.2 |
| Seeding at VCF = 1.3 | 0.450 | 12.2 |
| Cartridge filter (2 μm) + seeding at VCF = 1.15 | 0.637 | 15.4 |
| Cartridge filter (2 μm) + seeding at VCF = 1.2 | 0.645 | 15.1 |

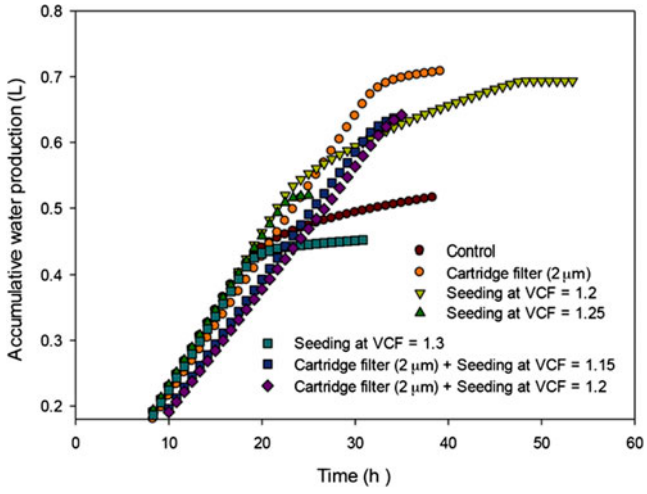


Fig. 6. Comparison of accumulative water production in MD operation for different antiscaling methods.

crystals in the MD system. Accordingly, combination of seeded crystallization with in-line filtration simply results in the removal of crystal particles without inducing the bulk crystallization.

Table 3 compares the effectiveness of different antscaling methods in MD systems. The $T_c$ and $VCF_c$ determined from Figs. 3–5 are presented. Moreover, the products of $T_c$ and $VCF_c$ are also shown. Since the crystallization is a function of time and concentration, the products of $T_c$ and $VCF_c$ indicates the driving force of the scale formation. If this value is larger, the antiscaling method is more effective to retard the onset of scale formation. As can be seen in the table, the in-line filtration increases the products of $T_c$ and $VCF_c$ from 21.1 to 53.8, which is 254% increase. The seeded crystallization also increases this up to 34.6, which is 164% increase. The combined methods results in 220–241% increase in the product of products of $T_c$ and $VCF_c$.

The total water production and average flux over the MD operation was compared for different antscaling methods in MD systems. As listed in Table 4, the in-line filtration increased the total water production from 0.52 to 0.70 L. Moreover, the average flux increased from 11.2 to 14.8 kg/m$^2$ h. By applying other antiscaling methods, the total water production and average flux increased. As shown in Fig. 6, the accumulative water production increased by applying these antiscaling methods.

## 4. Conclusions

In this study, control of fouling by scale formation in MD systems was attempted by applying seeded crystallization and in-line filtration. The following conclusions were withdrawn:

(1) The seeded crystallization could reduce fouling due to scale formation in MD system. However, its antiscaling effect seems to depend on the point of crystal particle addition.
(2) The in-line filtration using 2-μm metal mesh filter was found to be also effective to retard fouling due to scale formation by continuously removing crystals from the system.
(3) The combination of seeded crystallization and in-line filtration was also effective to control MD fouling by scale formation. However, it does not seem to have synergic effect because its effect was similar to that of the in-line filtration.
(4) By applying the antiscaling methods the product of $VCF_c$ and $T_c$ increased, indicating that the rate of scale formation was significantly reduced. Moreover, the total water production and average flux increased up to 1.35 times by the antiscaling methods.

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