Combination of Ultrafiltration and Ceramics Adsorption Filter for Pretreatment in Seawater Reverse Osmosis Desalination Process

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To mitigate the fouling of reverse osmosis (RO) membranes, which is one of the main issues in seawater desalination, Hitachi Metals has developed a ceramics adsorption filter (CAF) to remove potential foulants in RO feed water. The entire surface of the ceramic filter facing the water is coated with aluminum oxide, which has a higher isoelectric point (IEP) than seawater. When in seawater, the surface layer is positively charged and therefore adsorbs negatively-charged foulants. In order to promote the use of this product in the water treatment industry, Hitachi Metals and the Nanyang Environment and Water Research Institute (NEWRI) at Nanyang Technological University (NTU) conducted collaborative research to compare the effectiveness of a hybrid ultrafiltration (UF) - CAF pretreatment method with conventional UF pretreatment in reducing RO fouling. The changes in water quality before and after the CAF indicated that the CAF could remove a certain fraction of the dissolved organic matter (DOM) that escaped the UF membrane, thus alleviating RO membrane fouling. In addition, an RO membrane autopsy analysis indicated that a smaller amount of biofilm was formed on the RO membrane when a CAF pretreatment was used prior to RO. This was demonstrated by a slowdown in the decrease of membrane permeability as well as a reduction in the required membrane cleaning frequency by two times or more. This will be likely to reduce the operating costs of seawater desalination plants, and improve capacity utilization, thus resulting in lower water production costs.

● **Key words :** Desalination, Adsorption, Ceramic
● **R&D Stage :** Proto-type

1. Introduction

An increase in water demand has emerged as a global issue due to population growth, economic growth, and climate change. One of the Sustainable Development Goals (SDGs)[1] promoted by the United Nations is to ensure access to water and improve water quality and price.

Desalination is one of the solutions for securing water resources in low-precipitation areas, and many desalination plants rely on reverse osmosis (RO) technology. However, one of the main issues with RO technology is membrane fouling[2]. Fouling of RO membranes results in a decrease in permeability and an increase in water production costs due to an increase in pumping energy, higher chemical usage for membrane cleaning and replacement of RO membranes. Thus, effective pretreatment processes to remove potential foulants in the RO feed water are critical for alleviating the fouling of RO membranes.

Seawater contains suspended solids and dissolved matter. Dissolved matter includes not only ions but also colloids and soluble organic compounds with submicron sizes. These colloids and organic compounds are easily retained and accumulate on the RO membrane surface, thereby reducing the membrane permeability. The organic compounds also act as a food source for bacteria that eventually cause the formation of biofilms on the membrane surface. Therefore, pretreatment is used as the first step to remove suspended solids, organic compounds

and microorganisms from natural seawater prior the RO process, in order to protect the membrane.

Conventional pretreatment processes include (i) filtration by physical separation in various media, and (ii) chemical treatment (e.g., coagulation-flocculation, biocides, antiscalants)[2]. Even though microfiltration (MF) and ultrafiltration (UF) pretreatments are commonly used in seawater reverse osmosis (SWRO) plants, fouling of RO membranes still occurs. Filtration is effective for removing suspended solids but has limited effect on the removal of dissolved organic carbon (DOC) in seawater[3]–[5].

One approach to reduce the amount of organic matter in seawater is through adsorption. Due to the high ionic strength of seawater, metal oxides were found to be a more suitable absorbent for removal of organic matter than activated carbon. Kim et al. proposed the incorporation of powdered iron oxide in the UF system for algal organic matter removal. The study showed that iron oxide had a 50% removal efficiency for DOC, and hence significantly reduced fouling during RO[6]. Alumina is another common absorbent for the removal of contaminants such as toxic metal ions, organic dyes, pesticides and natural organic matter (NOM) in water[7],[8]. However, there has only been a limited amount of research into using alumina as an absorbent for seawater pretreatment applications.

Hitachi Metals published the first use of ceramics adsorption filter (CAF) as an alternative pretreatment approach for RO systems, in IDA World Congress 2017[9]. In order to market this product to water treatment plants, Hitachi Metals engaged with the Nanyang Environment and Water Research Institute (NEWRI) at Nanyang Technological University (NTU), Singapore to investigate the impact of a CAF on RO performance, in December 2017.

In the present study, NEWRI and Hitachi Metals conducted laboratory-scale and pilot-scale SWRO studies, respectively, to investigate the RO performance after CAF treatment. In both tests, two RO systems were operated in parallel. One of the systems consisted of a CAF pretreatment unit after UF pretreatment prior to RO, whereas the other system involved UF and/or dual-media filtration (DMF) followed by RO; the latter system was used as the baseline for comparison. Detailed water analyses were carried out before and after CAF pretreatment to correlate the fouling potential of the RO feed water and RO performance data. Furthermore, the major foulants that were attached to the membrane surface were identified through membrane autopsy.

2.　Overview of CAF

The CAF (**Fig. 1**) is made of cordierite, a ceramic with a composition of $2MgO \cdot 2Al_2O_3 \cdot 5SiO_2$. It has an array of square flow channels whose pitch is a few millimeters. The flow channels have porous walls with a maximum porosity of 70%, and the pores are interconnected. The porous walls are coated with alumina nanoparticles as an adsorbent. The flow channels are arranged to have an alternate pattern of 'feed channel' and 'treated channel'.
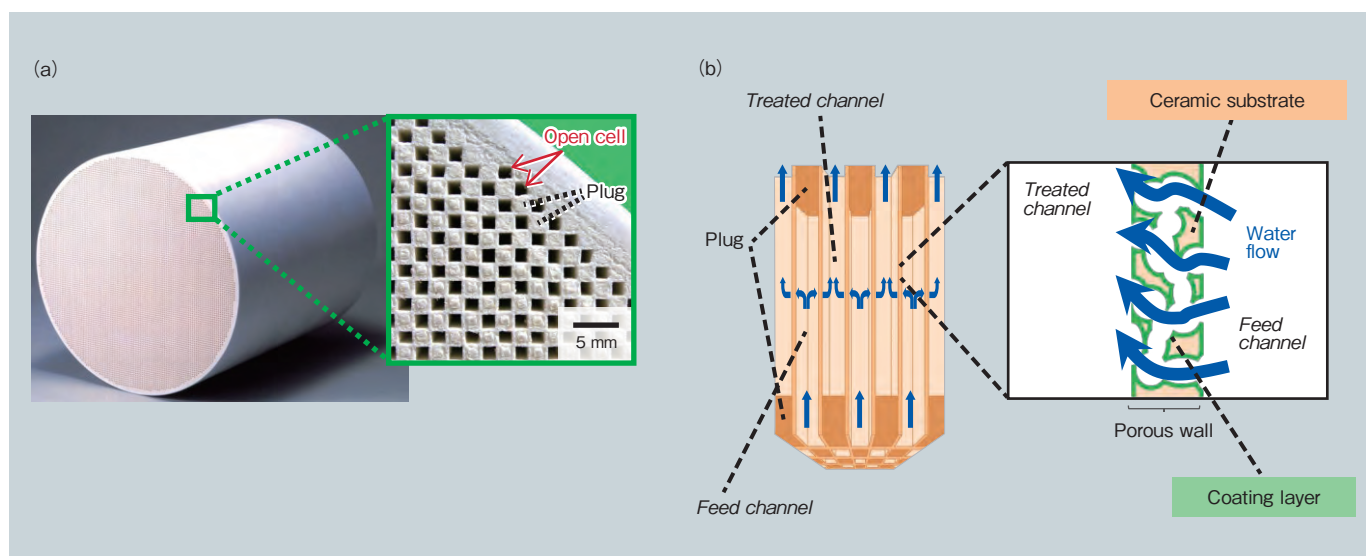

Fig. 1　Structure of CAF (a) photo of CAF, (b) cross-sectional illustration of water flow path

Both types of channel have an open end and a non-porous ceramic plug on the opposite end. The feed water enters the bottom of the CAF via the feed channels, passes through the porous walls where dissolved matter is adsorbed, and then the treated water exits at the top of the CAF via the treated channels.

The CAF is designed to remove material that could cause severe fouling in the RO process. The ceramic filter surface is coated by metal oxide with a higher isoelectric point (IEP) than seawater in order to enhance its adsorption of foulants in seawater, which are typically negatively charged.

The features of the CAF are:

· High chemical resistance, which allows periodic chemical cleaning to recover its adsorption performance
· Comparatively large surface area due to the highly porous walls and micron-size pores.
· Low hydraulic pressure loss (less than 1 kPa).

The main function of the CAF is to adsorb a certain fraction of the soluble organic matter that is typically not retained by the UF membrane. However, the CAF allows the passage of 99% of polystyrene particles (particle diameter of 2 μm)[9]. The removal performance for organic matter with different molecular sizes was compared for the CAF and UF membrane, and the results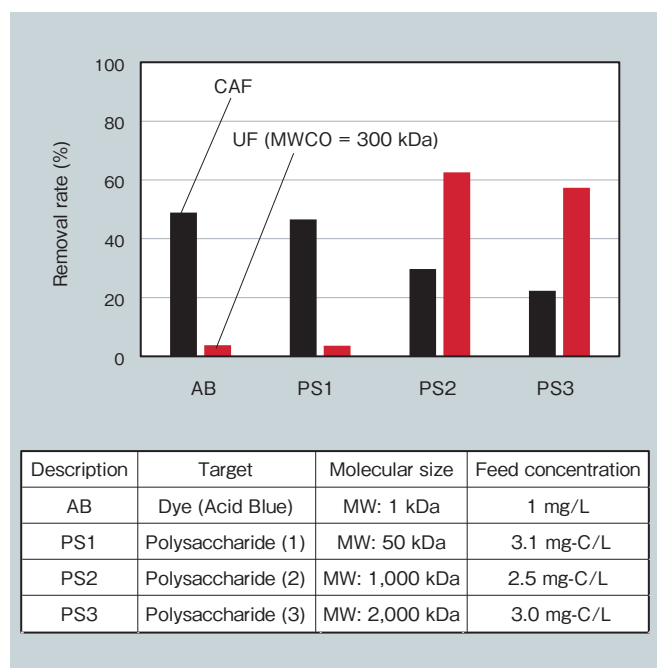 are summarized in **Fig. 2**. The CAF showed higher removal rates for small molecules such as dye (MW: 1,000 Da) and polysaccharide (MW: 50 kDa). In this experiment, the removal rate was defined as the change in the concentration of total organic carbon (TOC) before and after the CAF or UF, except for dyes whose removal rate was calculated based on the reduction in optical absorbance (measured by UV-vis spectroscopy). The results indicated that the CAF has the potential to adsorb low-molecular-weight organic matter.

The adsorption performance of the CAF relies on the contact probability between the molecules and the adsorbent on the surface of the CAF pores. The contact probability is determined by the contact time, i.e., the time taken to pass through the porous wall, assuming a fixed channel structure. Therefore, the space velocity (SV) was used to design the flow rate for the CAF.

3. Laboratory Testing at NEWRI (Singapore)

In the laboratory test at NEWRI, to clarify the different substances removed by the UF membrane and the CAF, two RO systems were operated in parallel, and fed with UF filtrated seawater de-chlorinated with sodium bisulfite (i.e., a mixture of $Na_2S_2O_5$ and $NaHSO_3$), one without and another with CAF treatment prior to RO, as shown in **Fig. 3**. The evaluation test was performed twice with different CAF cleaning frequencies, i.e., once a week in the first test and once a day in the second test.

The seawater was pumped through the CAF using a peristaltic pump at a constant flow rate of 70 mL/min, equivalent to a SV of 120 /h. A total volume of 15 L of seawater was treated by the CAF daily, and 50 mL of seawater samples were collected before and after CAF

Description	Target	Molecular size	Feed concentration
AB	Dye (Acid Blue)	MW: 1 kDa	1 mg/L
PS1	Polysaccharide (1)	MW: 50 kDa	3.1 mg-C/L
PS2	Polysaccharide (2)	MW: 1,000 kDa	2.5 mg-C/L
PS3	Polysaccharide (3)	MW: 2,000 kDa	3.0 mg-C/L

Fig. 2 Comparison of removal rates by CAF and UF for organic matter with different molecular sizes
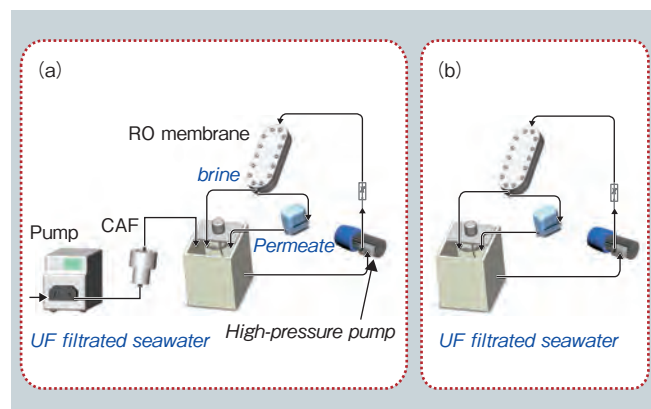

Fig. 3 Schematic diagram of experimental RO system (NEWRI)
(a) RO 1 with CAF pretreatment, (b) RO 2 without CAF pretreatment

filtration for analysis.

A high-pressure pump was used to pump the RO feed from a feed tank to the crossflow RO cell at a flow rate of 0.5 L/min (corresponding to a crossflow velocity of 0.1 m/s). The feed pressure of 4.5 MPa was adjusted by a pressure regulator located after the RO cell. The RO membrane used was a Nitto Hydranautics SWC5-4040. The membrane flux was calculated based on the permeate flow rate that was measured using a digital mass flowmeter. The permeate and concentrate were recycled to the feed tank to maintain constant volume. The conductivities of feed and permeate were monitored with conductivity transmitters. Data for the feed pressure, permeate pressure, conductivity, and permeate flow rate were recorded every minute by a data acquisition logger. The temperature of the RO feed solution in the feed tank was controlled at 28ºC using a chiller.

Water samples were collected daily before and after the CAF for measurements, i.e., analysis of the total dissolved organic carbon concentration by a Shimadzu TOC analyzer, characterization of the organic fraction by a fluorescence spectrophotometer (Agilent Technologies) and liquid chromatograph-organic carbon detection (LC-OCD; DOC-Labor Dr. Huber)[10]. In this study, a statistical modelling method, namely PARAFAC, was applied to further decompose the excitation emission matrix (EEM) into individual components with unique fluorescence features using MATLAB® R2018b software ("MATLAB" is a registered trademark of The MathWorks, Inc. in the USA.) with the DOMFluor toolbox following the protocols described by Stedmon and Bro[11]. This allows effective tracking of the fate of individual organic components in seawater systems. The fluorescence spectra corresponding to the blank (pure water) was subtracted from each measured EEM, and the fluorescence intensities were further normalized by the Raman peak for pure water at an excitation wavelength of 350 nm, and were calibrated against the quinine sulfate dilution series, producing EEMs in units of quinine sulfate equivalent (µg/L QSE).

At the end of the experiments, autopsies of the fouled RO membrane and used CAF were performed. The samples were first soaked in a sterilized 0.85% sodium chloride solution, then sonicated, followed by vortexing to detach the foulants. The extracted solutions were used for the organic characterization described above and for microbial analysis.

Extracellular polymeric substances (EPSs) were measured as the sum of the polysaccharide and protein content. The polysaccharide was measured using the colorimetric method (phenolic sulfuric acid method). Quantification of the protein content in EPS was conducted using a micro bicinchoninic acid (BCA) protein assay kit (Pierce, #23235).

The cell viability was quantified using flow cytometry (BD, USA). Specifically, 1 mL of samples was stained with 1 µL of both SYTO® ("SYTO" is a registered trademark of Molecular Probes, Inc. in the USA.) 9 and propidium iodide (Molecular Probes, USA). The sample was transferred to a flat-bottomed well plate for flow cytometry analysis (with an unstained sample as a control). Counts in a defined region of the density plot were converted to live and dead cells.

4. Pilot-scale Testing at Hitachi Metals (Japan)

Hitachi Metals set up a pilot-scale RO system at Water Plaza Kitakyushu on the shore of the Japan Sea. A schematic view of the system is shown in **Fig. 4**. The
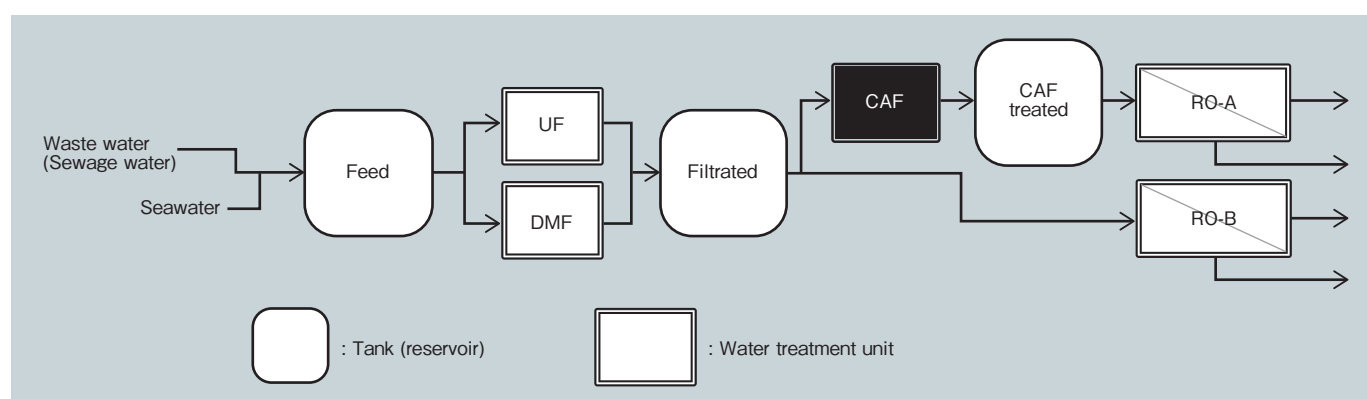

Fig. 4　Schematic diagram of pilot-scale RO system (Hitachi Metals)

system had parallel RO desalination lines with different pretreatment methods. The seawater was first filtered by UF and a dual-media filter (DMF). UF filtrated seawater and DMF filtrated seawater were mixed in a tank and split into two lines, i.e., with and without CAF treatment prior to RO. The volume of seawater fed into the system was 50-60 m³/day. The operating conditions for each unit are summarized in **Table 1**. The permeability and salt rejection rate were standardized by the method described in the document issued by the American Society for Testing and Materials (ASTM)[12], using the measured values of water temperature, feed pressure, flux and conductivity.

The pilot-scale SWRO systems were operated for a

month, but the reduction rates for the permeability for the two RO systems were the same. This could be due to the low concentration of organic matter in the seawater. In order to investigate the CAF effect, wastewater was added to the seawater in a small percentage (< 2%) in order to increase the DOC in the water during the final week of the evaluation test.

The water samples from the seawater tank (collected weekly), UF+DMF filtrate tank and CAF treated tank were measured by a Shimadzu TOC analyzer and a quartz crystal microbalance (QCM) (Biolin Scientific - qsense). Hitachi performed QCM measurements using a unique method to estimate the amount of organic matter that attached easily to the RO membrane[13]. The QCM instrument measures the weight change based on the shift in the resonance frequency of the sensor crystal[14]. The frequency shift is proportional to the weight of deposited components per unit area of the sensor surface. Using a sensor crystal coated with polyamide similar to the RO membrane surface, the frequency shift indicates the weight of the components that have high affinity for the RO membrane.

The used CAF was cut out after 9 weeks of continuous operation and soaked overnight in dilute hydrochloric acid (0.1 mol/L), and sodium hydroxide solution (0.1 mol/L) to extract the adsorbed matter. The extracted solutions were neutralized and the organic components present were characterized by LC-OCD.

5. Results and Discussion

Figs. 5-7 depict the profiles of the normalized permeability of the RO membranes in the 1st and 2nd laboratory-scale tests performed by NEWRI and the pilot-scale test performed by Hitachi Metals (HML). Two points should be noted from these results. First, the normalized permeability showed a decreasing trend with time, indicating the occurrence of membrane fouling. Secondly, UF-CAF-RO showed higher permeability (i.e., less membrane fouling) compared to UF-RO. Comparing the 1st and 2nd tests at NEWRI, the difference increased from 10% to 30% when a higher-frequency CAF backwash was adopted. This indicates that higher adsorption performance can be achieved by more frequent CAF chemical backwashing. In the pilot-scale test

Table 1　Operating conditions for pilot-scale test

Ceramics adsorption filter (CAF)	
Volume	10 L
Size	250 mm diameter, 185 mm length
Number	1
Feed mixture ratio	DMF: UF = 1: 2
Space velocity	90 – 150 /h
Pressure drop	< 5 kPa
Cleaning chemical	sodium hydroxide (0.4 wt%)
Cleaning frequency	daily
Dual media filter (DMF)	
Media	sand / anthracite
Filtration area	0.08 m³
Linear velocity	150 m/d
Pressure drop	< 10 kPa
Backwashing frequency	daily
UF	
Type	Dow SFP-2660 PVDF, 0.03 μm pore
Flux	0.7 – 1.0 m/d
Pressure drop	40 kPa
Backwashing frequency	once / 30 – 45 min.
CIP frequency	once every 3 months for seawater, weekly for wastewater-added seawater
RO	
Type	Hydranautics SWC5-4040
Size	100 mm diameter, 1,016 mm length
Number	1
Feed water	900 L/h
Permeate water	72 L/h (recovery rate: 8%) *const*.
Flux	0.2 m/d
Operating pressure	3.5 – 4.5 MPa

conducted by Hitachi Metals in Japan, an accelerated permeability drop was observed for the RO system without CAF after addition of wastewater into both systems. The weights of the components deposited on the RO sensor of the QCM from seawater with and without wastewater are shown in **Fig. 8**. The components that

have a high affinity for the RO membrane passed through the UF membrane, and the potential to foul the RO membrane became higher after wastewater addition. The increase in the organic matter content in the feed water caused a difference in the fouling rate for the two pretreatment processes.

Generally, chemical cleaning is conducted to recover the performance of a RO system when its permeability is reduced by 10-15% of its initial value. The downtime associated with membrane cleaning is a factor that decreases the utilization capacity of seawater desalination equipment. As such, the performance indicator used in both the studies by NEWRI and Hitachi Metals was the operating time required for the RO permeability to drop by 10% from the initial value. Our results showed that the RO membrane cleaning frequency was reduced because of adding the CAF after the UF as an RO pretreatment. As shown in **Fig. 9**, the time taken for the permeate flow rate to decrease by 10% was
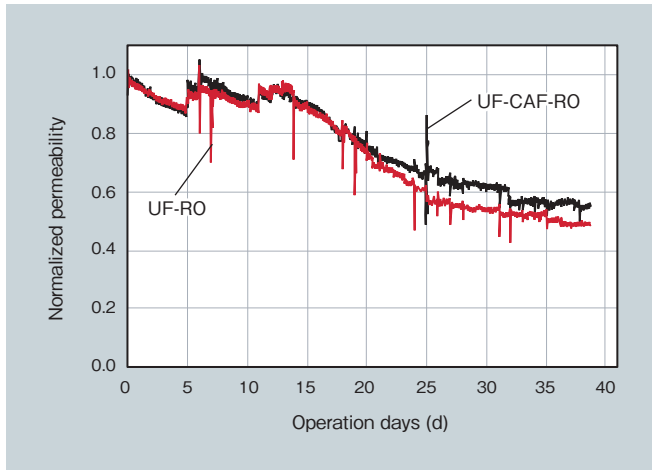

Fig. 5   RO permeability degradation in first test by NEWRI
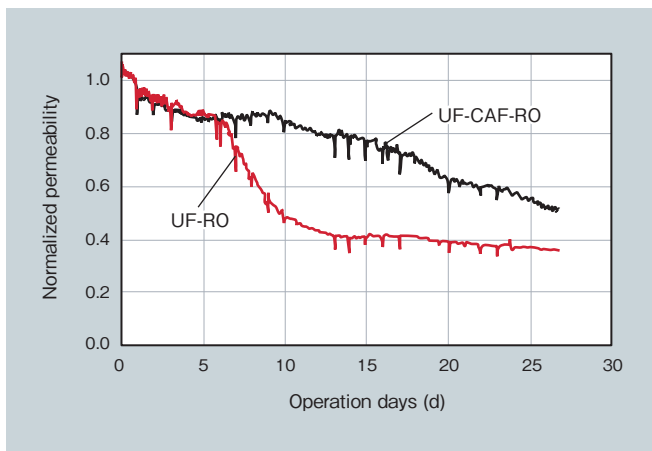

Fig. 6   RO permeability degradation in the second test by NEWRI
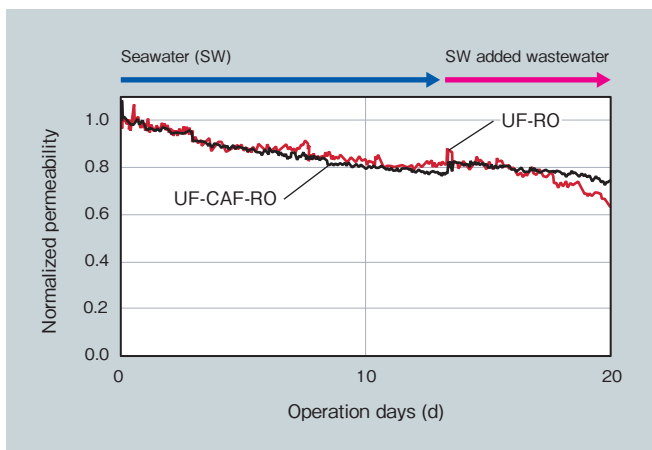

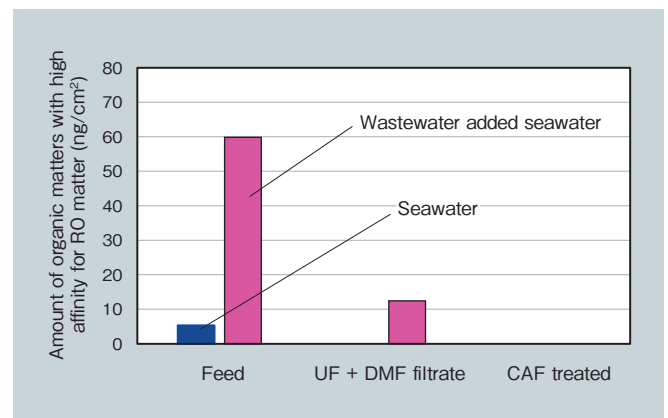Fig. 7   RO permeability degradation in pilot-scale test by Hitachi Metals

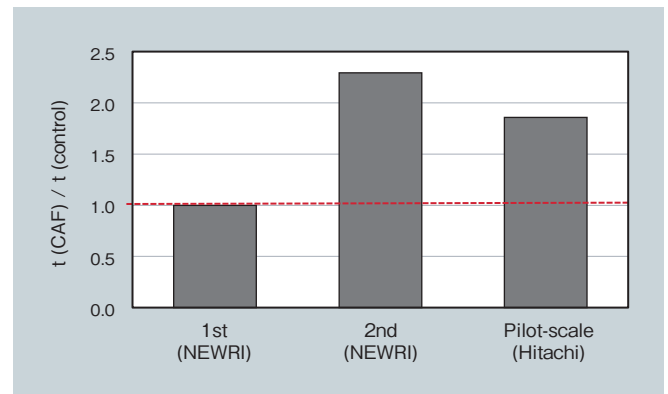Fig. 8   Amount of organic matter attached to RO membrane measured by QCM

Fig. 9   Ratio of time span required for chemical cleaning of RO membrane (" t " in y-axis stands for time taken for 10% decline in permeability for each pretreatment)

extended by up to 2.3 times relative to that without the CAF. This is likely to reduce the operating costs of seawater desalination plants and improve the utilization capacity, resulting in lower water production costs.

In order to determine the type of components that the CAF can capture, the water samples taken before the CAF (UF filtrate) and after the CAF were examined by LC-OCD in NEWRI's test. The total amount of DOC in the seawater ranged from 1.2 to 1.4 mg-C/L in both tests. From the LC-OCD analysis, four components were quantitatively measured, namely, biopolymer (BP), humic acid (HA), building blocks (BBs), and low-molecular weight matter (LMW). Among the organic components, the CAF tended to remove BP and HA as shown in **Table 2**, but showed limited removal of BB and LMW. Based on the Hitachi Metals pilot-scale test, the total organic matter in Japanese seawater was about 30% lower than that in Singapore, but the order of the composition ratio for CAF feed water was similar for all samples, i.e., HA > LMW > BB > BP. The values in the two rightmost columns in **Table 2** are the concentrations of the components in the extract from the used CAF in dilute hydrochloric acid and sodium hydroxide solution. They indicated the types of substances that were adsorbed by the CAF. The amount of BP in extracts was higher than that in the feed water for the CAF. BPs have a high molecular weight and consist of polysaccharides and proteins, which may cause fouling of RO membranes or are produced in the biofilm [15].

The components identified from the EEM-PARAFAC analysis in three components, on their excitation wave length (Ex) and emission wave length (Em) in the unit of nm. The three components described C1, C2 and C3 in this paper were:

C1: Ex/Em ≤ 250 (310)/402; Marine humic materials
C2: Ex/Em = 280/314; Amino acids, free or bound in proteins
C3: Ex/Em = 260 (370)/466; Terrestrial humic-like substances

The concentrations of each component are summarized in **Table 3**. The EEM-PARAFAC analysis in the second evaluation test also indicated that the CAF showed a

Table 2　LC-OCD analysis of organic components in water before and after CAF

	1st test			2nd test			Pilot-scale test		
	Concentration (μg/L) (ratio in DOC (%))		Removal rate (%)	Concentration (μg/L) (ratio in DOC (%))		Removal rate (%)	Concentration (μg/L) (ratio in DOC (%))		
	CAF feed	CAF treated		CAF feed	CAF treated		CAF feed (w/o WW)	Extract by acid	Extract by base
DOC	1,296	1,277	1.4	1,409	1,370	2.7	948	91	169
BP	117 (9.7)	116 (9.1)	1.0	91 (7.9)	82 (6.0)	8.9	58 (6.1)	20 (22.0)	17 (10.1)
HA	572 (47.4)	519 (40.6)	9.3	497 (43.4)	476 (34.7)	4.2	412 (43.4)	n.d.	56 (33.3)
BB	122 (10.1)	130 (10.2)	—	181 (15.8)	186 (13.6)	—	183 (19.3)	24 (26.4)	40 (23.8)
LMW	395 (32.8)	372 (29.1)	5.8	377 (32.9)	397 (29.0)	—	296 (31.2)	47 (51.6)	55 (32.7)

'-' in the columns for removal rate means the value is negative.
n.d.: under the detection limit

Table 3　Amount of organic components in RO feed water, foulants extracted from CAF and RO membrane in second evaluation test by NEWRI

	RO feed water		CAF	RO membrane	
	UF-CAF (μgQSE/L)	UF (μgQSE/L)	UF-CAF (ngQSE/gCAF)	UF-CAF-RO (ngQSE/cm²)	UF-RO (ngQSE/cm²)
C1	7.4	6.6	7	6	6
C2	4.8	4.1	26	50	35
C3	3.4	3.3	2	3	2

greater tendency to capture proteins from the feed water. The concentration of C1 was higher than those of C2 and C3 in the RO feed water. On the other hand, the concentration of C2 was the highest in the foulants of the CAF and RO membranes.

Microbial analysis of RO foulants was also performed. The EPS content of the fouled RO membranes is shown in **Fig.10**. The EPS was mainly composed of proteins rather than polysaccharides. The amount of EPS on the

UF-CAF-RO fouled membrane was 20% and 45% lower than that for the UF-RO fouled membrane in the weekly and daily CAF backwash tests, respectively. The cell count for CAF-UF-RO was similar to that for the UF-RO fouled membrane in the first evaluation test, but was 76% lower in the second evaluation test, as shown in **Fig. 11**. The presence of the CAF is able to suppress bio-film accumulation on the RO membrane.
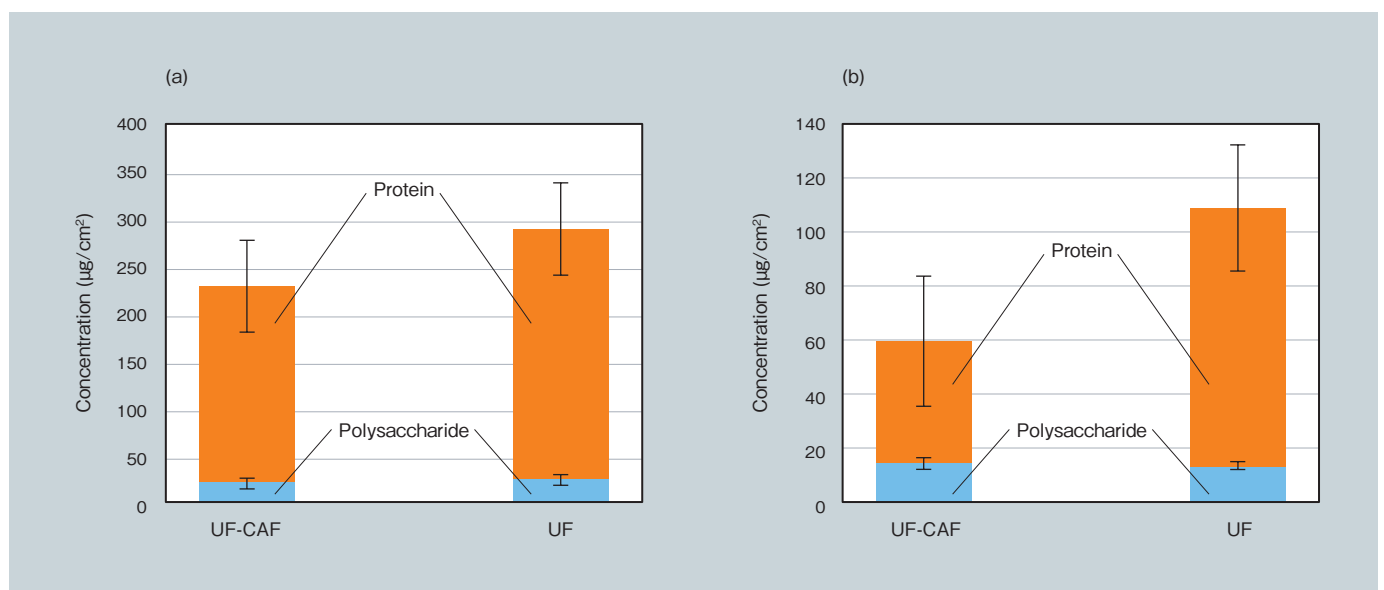

Fig. 10 EPS content of fouled RO membranes with and without CAF pretreatment (a) 1st evaluation with weekly backwash, (b) 2nd evaluation with daily backwash
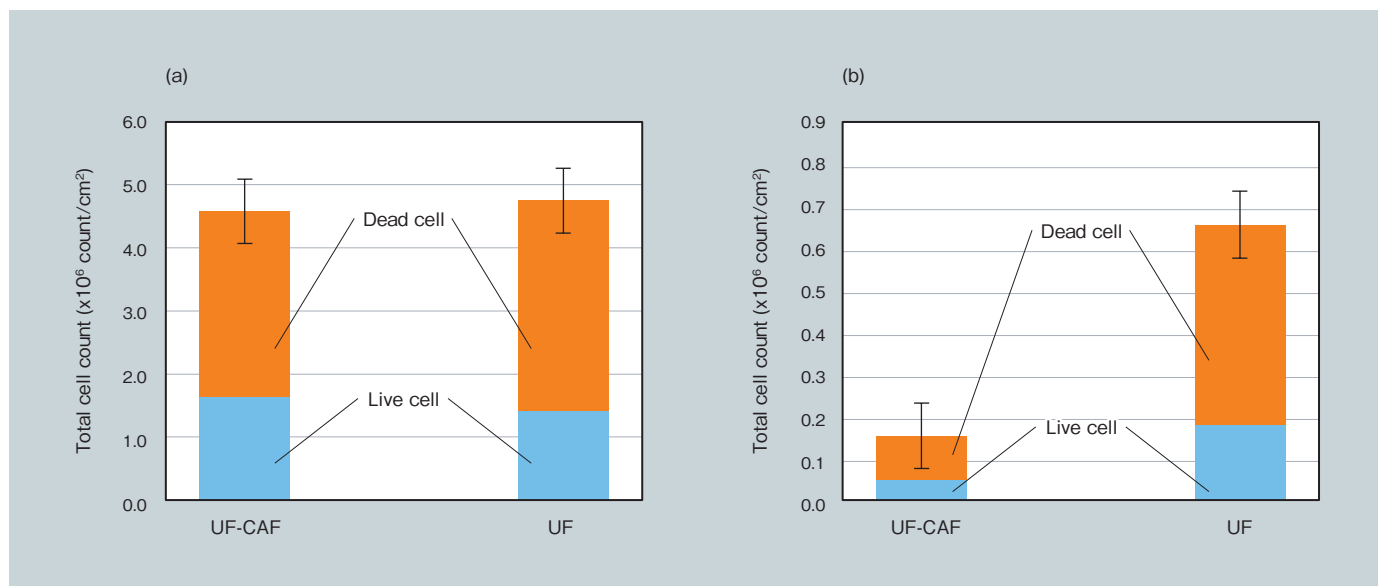

Fig. 11 Live/dead cell count on fouled RO membranes with and without CAF pretreatment (a) 1st evaluation with weekly backwash, (b) 2nd evaluation with daily backwash

6. Conclusion

Hitachi Metals has developed a ceramics adsorption filter (CAF) with porous walls coated with an adsorbent material that suppresses the fouling of RO membranes by selectively removing the dissolved organic matter in the RO feed water. The ceramic filter surface is coated by a metal oxide with a higher isoelectric point than that for seawater in order to increase its ability to adsorb negatively charged organics. NEWRI and Hitachi Metals conducted laboratory-scale and pilot-scale SWRO studies, respectively, to investigate the RO performance after CAF treatment. In both tests, two RO systems were operated in parallel, by feeding UF (+DMF) filtrated seawater without and with CAF treatment prior to RO.

The UF-CAF-RO showed less RO membrane fouling compared to UF-RO even though the removal rate for dissolved organic matter was negligible. Additionally, the total organic matter derived from the seawater on the UF-CAF-RO membrane was about the same or slightly more than that on the UF-RO membrane. It is worth noting that the CAF appeared to be able to suppress biofouling on the RO membrane. Furthermore, it was observed that the components (total amount and composition of ion species) adsorbed on the surface of the CAF changed depending on the conditions of the water. Hitachi metals and NEWRI are currently conducting further study to determine an effective combination of pretreatments for different types of feed water for desalination and reuse systems with RO membranes.

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