A Novel Photobiological Process for Reverse Osmosis Concentrate Treatment Using Brackish Water Diatoms

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Abstract: A unique aqueous silica removal process using naturally occurring diatoms for water reuse and desalination is described. Several strains of brackish water diatoms have been isolated and tested. Among them *Pseudostaurosira* and *Nitzschia* species showed promise. Reverse osmosis (RO) concentrate samples from two full-scale advanced water purification facilities and one brackish groundwater RO plant in Southern California have been successfully treated by this process. This new photobiological process could remove aqueous silica, as well as phosphate, ammonia, nitrate, calcium, iron and manganese very effectively. Under non-optimized conditions, 95% of 78 $mg \cdot L^{-1}$ reactive silica in an RO concentrate sample could be removed within 72 hours. In most cases, addition of nutrients was not necessary because the RO concentrate typically contains sufficient concentrations of macronutrients derived from the source water (i.e., treated wastewater and brackish groundwater). Preliminary characterization of organics indicated that there was no major generation of dissolved organics, which could potentially foul membranes in the subsequent RO process. This new algal process has a strong potential for its application in desalination and water reuse in the United States and around the world.

Keywords: Advanced water purification, bacillariophyta (diatoms), concentrate management, potable reuse, reverse osmosis

Introduction

The drought in California is an unprecedented crisis and has made the state's water supply more vulnerable than it has ever been. Not only in California, but other arid and semi-arid states and countries are facing an urgent need for alternative water resources as well. In recent years, more and more water utilities in the southwestern United States and around the world have begun exploring water from unconventional water resources, such as reclaimed water and brackish groundwater, using reverse osmosis (RO) (Greenlee *et al.*, 2009; Pérez-González, *et al.*, 2012). Brine (concentrate) management and minimization has become a critical issue in RO-based water reuse and desalination projects, especially in inland areas where the means of brine disposal are limited. In order to minimize the volume of RO concentrate further, many advanced water treatment facilities are considering adding an additional stage of RO process to recover another 10 to 15% of usable water, although serious scaling due to the presence of inorganic scalants, including silica, calcium, and phosphate is a major obstacle (Asano *et al.*, 2007). In order to solve this challenge, a unique photobiological process utilizing selectively cultured diatoms has been developed to efficiently remove these inorganic scalants from RO concentrate so that additional RO can be employed to recover more fresh water (Ikehata *et al.*, 2017). This approach will help reduce the environmental impacts of water reuse and brackish water desalination by harnessing the natural power of microalgae that has been known for decades, but largely overlooked in water and wastewater treatment.

Previously, rapid removal of reactive silica and orthophosphate was observed in a silica-rich brackish agricultural drainage water and an RO concentrate sample from the Groundwater Replenishment System (GWRS), Orange County Water District (OCWD) using a mixed diatom culture obtained from an evaporation pond in the Central Valley of California (Ikehata *et al.*, 2017). Silica was likely utilized by the diatoms in the silicification process (Lewin, 1954; Martin-Jezequel *et al.*, 2000). One strain of diatom, *Pseudostaurosira trainorii* PEWL001, was isolated from the mixed culture, and additional three strains, including

Nitzschia communis PEWL002, *Anomoeoneis sphaerophora* PEWL003, and *Halamphora sydowii* PEWL004, were isolated from another water-sediment sample from the evaporation pond. In this study, these isolated strains, in particular *P. trainorii* PEWL001 and *N. communis* PEWL002 (Figure 1), were used to treat RO concentrate samples from different full-scale RO facilities in Southern California. The impacts of this algal treatment on dissolved organic matter (DOM) in the selected ROC were also studied.

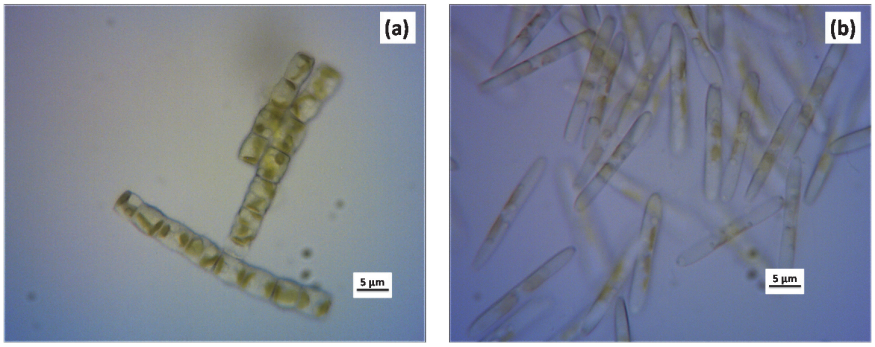

Figure 1 Photomicrograph of (a) *P. trainorii* PEWL001, and (b) *N. communis* PEWL002

Material and Methods

A brackish water diatom *P. trainorii* E. Morales PEWL001 was isolated from agricultural drainage water collected in the Central Valley of California, USA during the summer of 2010 as described earlier (Ikehata et al., 2017). First, the drainage water sample was incubated at room temperature (~ 25°C) under continuous illumination over a period of time (~10 days) until visible algal colonies became visible. Strains were then isolated from the colonies by a combination of serial dilution, agar plate, and micropipette techniques (Andersen and Kawachi 2005). Another brackish water diatom *N. communis* Rabenhorst PEWL002 was isolated from a drainage water sample collected from the same area in November 2015. The diatom seed cultures were maintained in 15-mL or 50-mL VWR clear polypropylene centrifuge tubes containing 0.2-µm filtered diluted synthetic seawater containing Guillard's F/2 medium (Guillard, 1975) or 0.2-µm filtered RO concentrate sample from the GWRS (see below). The concentration of total dissolved solids (TDS) in the F/2 medium was 7 $g \cdot L^{-1}$, which is similar to that of the RO concentrate samples treated in this study.

RO concentrate samples were obtained from three full-scale RO facilities, including the GWRS of the OCWD in Fountain Valley, CA, USA, the Leo J. Vander Lans Advanced Water Treatment Facility (LVL AWTF) of the Water Replenishment District of Southern California (WRD) in Long Beach, CA, USA, and the Chino I Desalter of Chino Basin Desalter Authority/Inland Empire Utilities Agency (IEUA) on April 22nd, 2016, November 21st, 2013, and August 25th, 2016, respectively. The collected RO concentrate samples were characterized for basic water quality (Table 1) and kept refrigerated until use. The analytical methods used are also listed in Table 1.

A HACH DR-2800 spectrophotometer and a HACH 2100N turbidimeter (Loveland, CO, USA) were used for the colorimetric and turbidity analyses, respectively. A HACH ISENA38101 combined with an HQ40d portable meter was used for sodium analysis. Boron analysis was performed by TestAmerica (Irvine, CA, USA). An Oakton pHTestr2 and a TDSTestr2 (Vernon Hills, IL, USA) were used for the pH, TDS, and temperature measurement. UV-Vis and fluorescence analyses were conducted with a Varian Cary 100 Bio

UV-Vis spectrophotometer (Agilent Technologies, Santa Clara, CA, USA) and a Horiba FluoroMax-4 spectrofluorometer (Horiba Scientific, Edison, NJ, USA) in the Urban Water Research Center at the University of California, Irvine, CA, USA.

Table 1 Basic water quality of RO concentrate samples collected from three full-scale RO facilities in Southern California

Parameter	Analytical Method	OCWD GWRS	WRD LVL AWTF†	Chino I Desalter
Sodium ($mg \cdot L^{-1}$)	HACH ISENA38101	1,167	667	337
Potassium ($mg \cdot L^{-1}$)	HACH 8049	171	71	11
Calcium ($mg \cdot L^{-1}$)	HACH 8204	456	416	1,264
Magnesium ($mg \cdot L^{-1}$)	Calculated	139	99	118
Iron ($mg \cdot L^{-1}$)	HACH 8008	<0.02	0.24	0.03
Copper ($\mu g \cdot L^{-1}$)	HACH 8143	<1	4	5
Manganese ($mg \cdot L^{-1}$)	HACH 8149	0.396	0.358	0.375
Ammonia-N ($mg \cdot L^{-1}$)	HACH 10023/10031	5.2	4.1	<0.02
Boron ($mg \cdot L^{-1}$)	EPA 200.7 Rev 4.4	0.9	Not tested	Not tested
Chloride ($mg \cdot L^{-1}$)	HACH 8207	1,900	810	760
Sulfate ($mg \cdot L^{-1}$)	HACH 8051	980	800	570
Bicarbonate ($mg \cdot L^{-1}$)	HACH 8203	1,077	1,318	1,732
Nitrate-N ($mg \cdot L^{-1}$)	HACH 10206	25	23	248
Reactive silica ($mg \cdot L^{-1}$)	HACH 8185	133	78	146
Orthophosphate ($mg \cdot L^{-1}$)	HACH 8048	5.6	8.5	1.04
Total dissolved solids ($mg \cdot L^{-1}$)	Oakton TDSTestr2	6,690	3,880	4,260
Turbidity (NTU)	EPA 180.1	1.16	2.07	0.623
Total hardness ($mg \cdot L^{-1}$ as $CaCO_3$)	HACH 8213	1,720	1,453	3,650
Alkalinity ($mg \cdot L^{-1}$ as $CaCO_3$)	HACH 8203	883	1,080	1,420
Total chemical oxygen demand ($mg \cdot L^{-1}$)	HACH 8000	245	154	129
Dissolved chemical oxygen demand ($mg \cdot L^{-1}$)*	HACH 8000	217	104	53
Temperature (°C)	Oakton TDSTestr2	20.2	Not tested	30.6
pH	Oakton pHTestr 2	7.98	8.2	7.3
Color at 455 nm (PtCo unit)	HACH 8025	271	96	7

Note: *Filtered through 0.2 μm membrane filter, †This sample was collected before the recent facility expansion, which involved the addition of third stage RO and completed in 2014.

A series of RO concentrate treatment experiments were conducted in a bench-scale semi-batch mode using 500-mL polyethylene terephthalate (PETE) bottles (Φ = 65 mm) and VWR SuperClear 50-mL polypropylene centrifuge tubes with screw caps (Φ = 29 mm, VWR International, USA). These containers were placed in an illuminating reflective incubator with 9-W light-emitting diode (LED) bulbs (light temperature 5000 K, 800 lm each; Cree, Inc., Durham, NC, USA). The LED bulbs emitted visible light radiation ranging from 400 to 750 nm with a sharp peak at 450 nm and a broader peak at 550 nm. The relative radiant power was two times higher at the former peak than at the latter one. The photosynthetically active radiation was measured as 1.6 $\mu E \cdot s^{-1} \cdot m^{-2}$ using an International Light Technologies ILT 1400 Portable radiometer with an attenuated PAR sensor (Peabody, MA, USA). The incubation temperature was at 25±2 °C. Prior to the diatom inoculation, RO concentrate samples were filtered through 0.2-μm membrane filters. No chloramine residual was detected in the RO concentrate samples at the time of the treatment experiment. Pre-cultured diatom suspension (500 μL or 5 mL, respectively) was added to the 50- or 500-mL containers to initiate the photobiological treatment. The seed culture was pre-grown in the GWRS ROC or Guillard's F/2 medium as described above. The initial biomass concentration in each container was about 0.15 g dry weight L^{-1}. Aliquots of samples were withdrawn periodically from the containers to measure color, reactive silica and orthophosphate concentrations during the treatment. Once reactive silica concentration was reduced below 1 $mg \cdot L^{-1}$, supernatant was removed from the containers by decantation while a majority of algal biomass was kept in the

container. Fresh RO concentrate was added to the container for another semi-batch cycle. The supernatant was further analyzed for water quality. At the end of the last cycle of semi-batch experiment, the dry weight of biomass was determined using the method described earlier (Ikehata *et al.*, 2017). In the case of brackish groundwater RO concentrate treatment, sodium phosphate monobasic (ACS reagent; Sigma-Aldrich, St. Louis, MO) or F/2 medium concentrate (no silica, F/2 Algae Food; Fritz Aquatics, Mesquite, TX) was added to adjust the initial orthophosphate concentration.

Results and Discussion

As shown in Figure 2 the photobiological treatment using isolated diatoms was very effective in removing reactive silica and orthophosphate from RO concentrate samples obtained from two full-scale advanced water purification facilities, namely LVL AWTF and GWRS. Three semi-batch cycles were successfully performed in both cases, although the silica removal was apparently faster in the former RO concentrate sample (up to 35 $mg \cdot L^{-1} \cdot day^{-1}$) than the latter (up to 8 $mg \cdot L^{-1} \cdot day^{-1}$). The diatom growth and silica uptake might be inhibited by certain dissolved inorganic constituents, such as ammonia (Natarajan, 1970; Azov and Goldman, 1982) and copper (Florence and Stauber, 1986), as well as organics such as herbicides (Debenest *et al.*, 2009). In addition, the color of the latter RO concentrate sample was almost three times higher than the former sample (Table 1) and might have reduced the light available for photosynthesis. The rate of silica removal by the purified *N. communis* PEWL002 from GWRS RO concentrate was similar to that observed during the RO concentrate treatment using a mixed diatom culture (Ikehata *et al.*, 2017). The silica removal accelerated in the second and third cycles, which implies that the diatom biomass concentration is an important factor. At the end of the third cycle, the biomass concentration was 2.1 g dry weight L^{-1}.

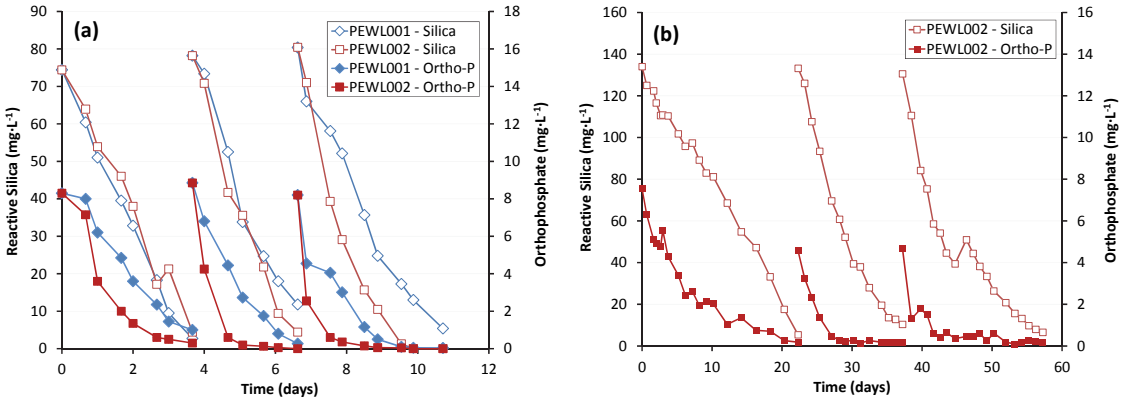

Figure 2 Removal of reactive silica and orthophosphate from (a) LVL AWTF and (b) GWRS RO concentrate samples by the photobiological treatment using *P. trainorii* PEWL001 and *N. communis* PEWL002

The rates of silica removal by two diatom species were almost identical in LVL AWTF RO concentrate in the first and second cycles. However, the silica removal by *P. trainorii* PEWL001 slowed down significantly in the third cycle, likely due to contamination by green algal cells (Ikehata *et al.*, 2017). No contamination was observed during the LVL AWTF RO concentrate treatment with *N. communis* PEWL002, whereas a very similar contamination issue occurred in the case of the GWRS RO concentrate treatment with *P. trainorii* PEWL001,

157 which implied that further purification of the latter diatom strain would be required. At the end
158 of the third cycle, the biomass concentrations of *P. trainorii* PEWL001 and *N. communis*
159 PEWL002 were 0.61 and 1.5 g dry weight L^{-1}, respectively.

160 Figure 3 shows the removal of nutrients and RO scaling constituents by the photobiological
161 treatment of LVL AWTF and GWRS RO concentrate samples using *N. communis* PEWL002.
162 A similar result was obtained with *P. trainorii* PEWL001 (data not shown). A majority (>70%)
163 of iron and manganese were removed by the photobiological treatment. In addition, two other
164 major RO scaling factors, calcium and bicarbonate, were removed by more than 60%. The
165 precipitation of calcium carbonate as calcite or aragonite was speculated (Borowitzka, 1987).

166 In those RO concentrates from the advanced water reclamation facilities, phosphorus was
167 apparently the limiting nutrient. While ammonia was the preferred nitrogen source and was
168 completely removed in the case of GWRS RO concentrate treatment (Figure 3b), both nitrate
169 and ammonia were consumed simultaneously in the case of LVL AWTF RO concentrate
170 treatment (Figure 3a). The reason for this difference is unclear because these RO concentrate
171 samples contained fairly similar levels of phosphorus and nitrogen compounds (Table 1).
172 Additional experiments are currently being conducted to explore this issue.

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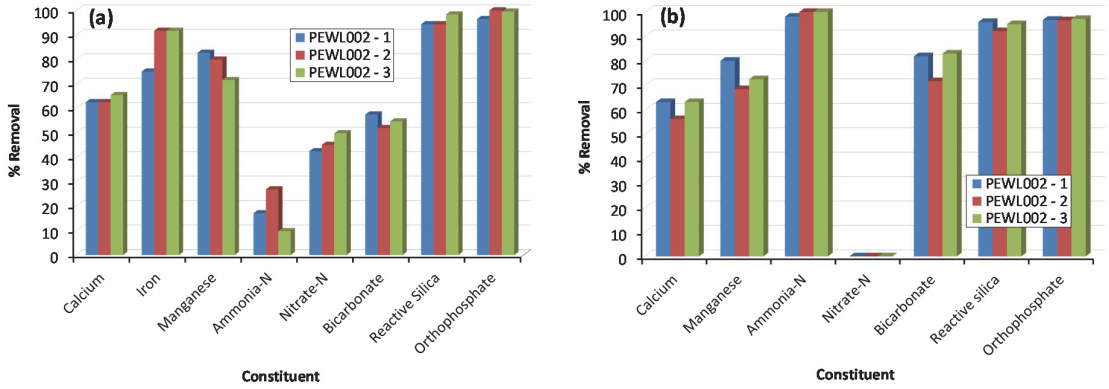

174

175 **Figure 3** Removal of nutrients and scaling constituents from (a) LVL AWTF and (b) GWRS
176 RO concentrate samples by the photobiological treatment using *N. communis* PEWL002

177

178 In addition to the RO concentrate samples from the two advanced water reclamation
179 facilities, another sample from Chino I Desalter, which is a brackish groundwater desalination
180 facility, was treated by the photobiological treatment. It was found that phosphorus in the RO
181 concentrate sample was not enough (1.0 $mg \cdot L^{-1}$ as orthophosphate) to complete the silica
182 removal (Figure 4; blue diamonds). Therefore, phosphate was added as sodium phosphate or
183 F/2 medium component. It was found that 5 $mg \cdot L^{-1}$ of orthophosphate was enough to
184 completely remove 146 $mg \cdot L^{-1}$ of silica. The silica removal rate was 18 $mg \cdot L^{-1} \cdot day^{-1}$, although
185 it accelerated in the second and third cycles (data not shown). Also, pure sodium phosphate
186 was less effective than F/2 medium to facilitate silica removal (Figure 4). Trace minerals
187 and/or vitamins in the F/2 medium (Guillard, 1975) might have enhanced the diatom growth
188 and silica uptake.

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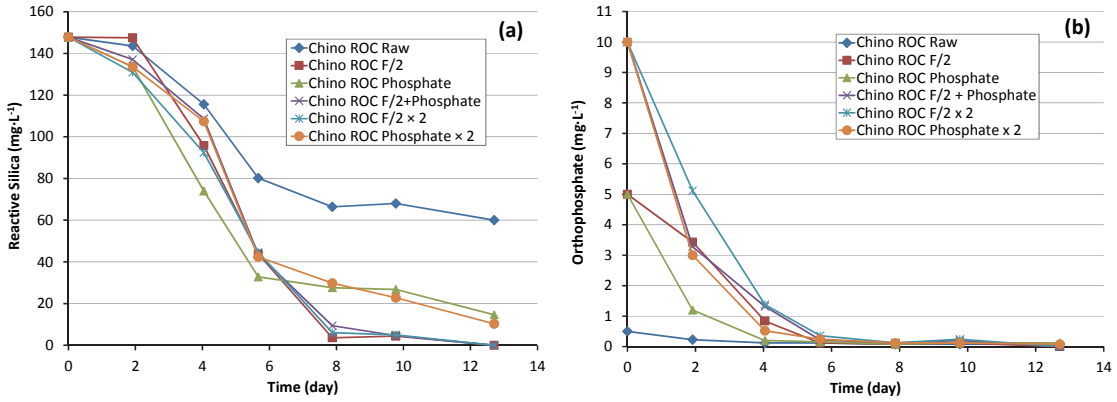

Figure 4 Removal of reactive silica from Chino I Desalter RO concentrate sample by the photobiological treatment using *P. trainorii* PEWL001: (a) reactive silica removal, and (b) orthophosphate uptake

Since the goal of this photobiological RO concentrate treatment is to enable the secondary RO without fouling and scaling, it is very important to characterize the organic matter after the photobiological treatment. Besides, it is well known that phytoplankton, including diatoms, excrete dissolved and particulate organic matter (Bjørrisen, 1988; Biddanda and Benner, 1997) and that seawater RO desalination is often affected by harmful algal brooms and organic particulate matter called transparent exopolymer particles associated with them (Caron *et al.*, 2010; Villacorte *et al.*, 2013). The preliminary analysis appeared to be very encouraging.

After the photobiological treatment of LVL AWTF RO concentrate sample using brackish water diatoms *P. trainorii* PEWL001 and *N. communis* PEWL002, filtered color (not shown), UV absorbance at 254 nm (not shown), and chemical oxidation demand (COD; Figure 5) were not significantly increased. A similar result was obtained when GWRS RO concentrate was treated in the same way.

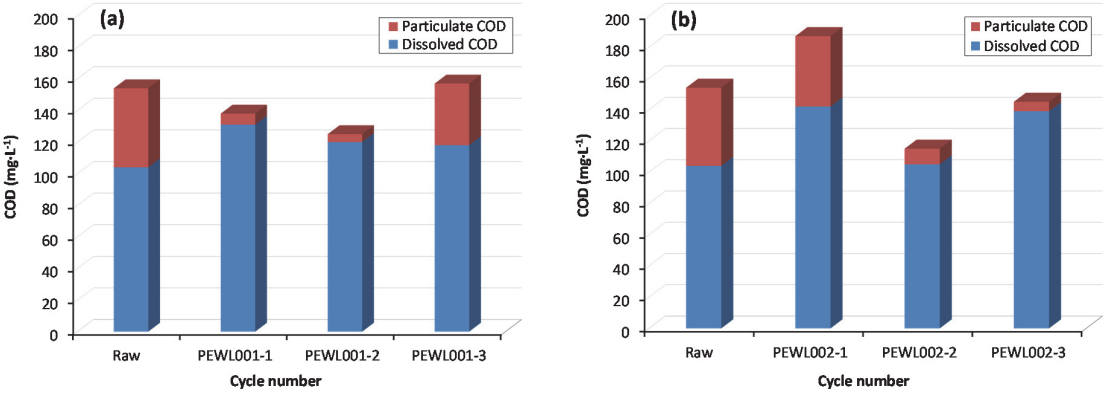

Figure 5 Changes in dissolved and particulate chemical oxygen demand (COD) before and after the photobiological treatment of LVL AWTF RO concentrate sample using (a) *P. trainorii* PEWL001, and (b) *N. communis* PEWL002

Preliminary analysis of DOM was attempted using the fluorescence spectrometry. As shown in Figure 6, the strong fluorescence peak due to UV humic-like component (A peak), as

well as weaker peaks due to visible humic-like component (C peak), marine humic-like component (M peak), and protein-like component (T peak), was present the excitation-emission matrix (EEM) of raw (untreated) LVL AWTF RO concentrate sample, which is similar to that of raw GWRS RO concentrate sample (not shown), as well as the reported EEM of RO concentrate from another RO facility (Bagastyo *et al.*, 2011). The appearance of EEM of photobiologically treated LVL AWTF RO concentrate was very similar to that of untreated ROC even after three semi-batch cycles (Figure 2a). The peak integrals and fluorescence were compared before and after the treatment as shown in Figure 7. Overall peak integral was decreased by the photobiological treatment using both *P. trainorii* PEWL001 and *N. communis* PEWL002. Peak A intensity decreased significantly (about 21%), especially with *P. trainorii*, indicating the humic-like component was degraded by the photobiological treatment. While the intensities of Peaks C and M were also slightly decreased (14% of Peak C, 18% of Peak M in the case of the treatment with *P. trainorii*), the intensity of Peak T did not change significantly in the RO concentrate samples after the photobiological treatment with the both diatom species. More detailed analysis of DOM with EEM and size exclusion chromatography is currently underway. The impact of the photobiological treatment on trace organic compounds, such as pharmaceuticals and personal care products, and disinfection byproducts, in the RO concentrate samples is also being investigated.

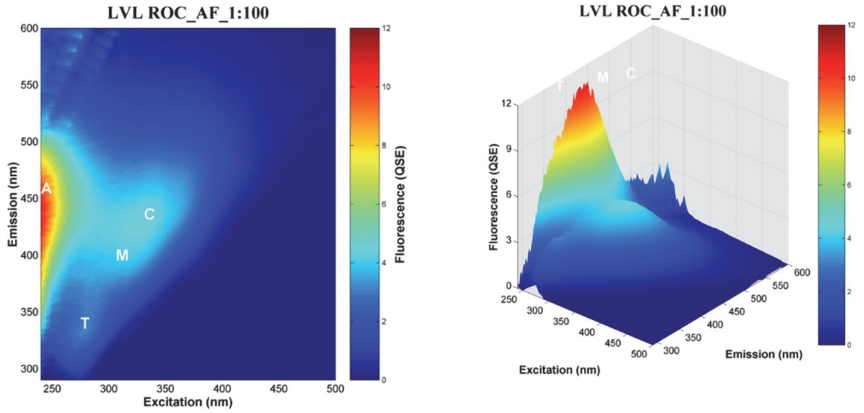

Figure 6 Excitation-emission matrix (EEM) spectra of untreated LVL AWTF RO concentrate sample

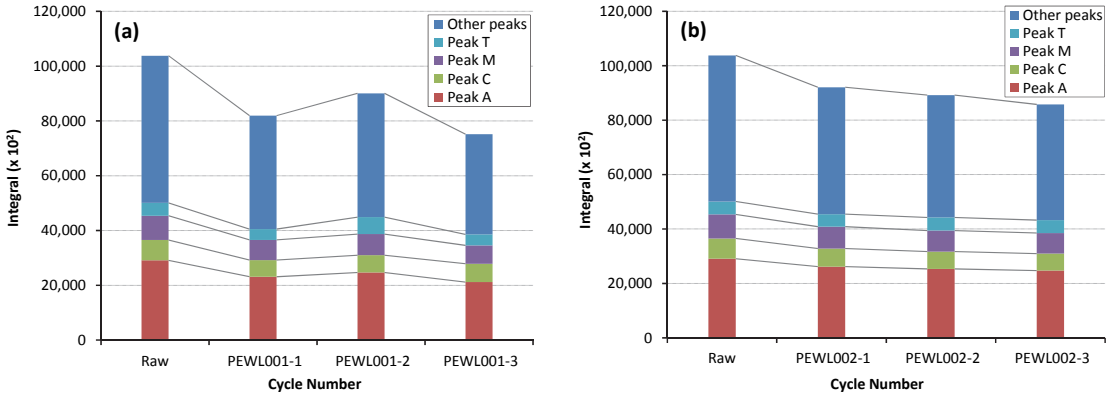

Figure 7 Impact of the photobiological treatment on the LVL AWTF RO concentrate EEM

peak integrals: (a) *P. trainorii* PEWL001, and (b) *N. communis* PEWL002

Conclusions

Three RO concentrate samples from three full-scale RO facilities in Southern California have been successfully treated by the photobiological treatment using isolated brackish water diatoms, *P. trainorii* PEWL001 and *N. communis* PEWL002, in laboratory-scale photo-bioreactors. The photobiological treatment could be performed at least three cycles in a semi-batch mode. The rate of silica removal varied in the different RO concentrate samples, which indicated the presence of some inhibitory components in certain samples. Nutrient addition was not needed when the RO concentrate samples from advanced water treatment facilities (LVL AWTF and GWRS) were treated. However, the brackish groundwater RO concentrate tested in this study (Chino I Desalter) did not contain enough phosphorus to complete silica removal and required its supplementation. In addition to silica, orthophosphate, calcium, iron, manganese, bicarbonate, ammonia, and nitrate were effectively removed by the photobiological treatment. Since many of them are responsible for RO scaling, there is a potential to use this technology as a pretreatment of RO concentrate from the primary RO to make the secondary RO more feasible, cost effective and environmentally friendly. Preliminary analysis of DOM showed no significant increase in organic matter that could cause RO membrane fouling.

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