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Membrane-based pretreatment to mitigate variations in desalination plants

J. Arevalo, R. Sandin, M. D. Kennedy, S. G. Salinas Rodriguez, F. Rogalla and V. M. Monsalvo

ABSTRACT

The main source of potable water in high water-stress areas is commonly produced in brackish and seawater desalination plants. Owing to the presence of high concentration of suspended solids, organic matter and colloidal particles in raw water, pretreatment processes are needed for a stable operation of desalination plants. A submerged membrane ultrafiltration pilot plant has been operated as pretreatment of complex brackish surface water to study the filtration performance. The results show the membrane performance, chemical reagent requirements, water quality and cleaning procedures efficiency of an ultrafiltration pilot plant used as pretreatment for a reverse osmosis system. Alternative chemical cleaning procedures have been satisfactorily implemented, which maximize permeability recovery and allow a stable operation.

Key words | desalination, drought, fouling, membrane cleaning, ultrafiltration

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INTRODUCTION

Summer on the Mediterranean coast is a hot and dry season when the population in coastal towns increases sharply because of the tourism. Water scarcity issues get worse during the hot season due to the specific characteristics of the Mediterranean climate with low precipitation and high temperatures (Lionello *et al.* 2006). These facts make it difficult to satisfy the increasing water demand and protect the ecological function of the water cycle. Thus, alternative water sources have to be found and exploited, such as seawater and brackish water (Palomar & Losada 2010). In this respect, water sector stakeholders are interested in exploring these alternative sources and creating a regulation framework to favor a sustainable and safe use, as well as promote the use of these alternative water sources (March *et al.* 2014; Gude 2016).

Saline water sources are commonly classified into two salinity categories: seawater, with a high concentration of dissolved salts (30,000–45,000 mg/L total dissolved solids (TDS)) and brackish water, with a lower salinity (1,000–10,000 mg/L TDS) (Greenlee *et al.* 2009). The production of drinking water from low salinity sources is especially

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attractive when available, due to the lower energy demand for the desalination process compared to seawater feed (Dore 2005). In numerous cases, brackish water is collected from groundwater and even rivers to be conveniently treated in brackish water desalination plants (BWDPs) (Mohsen & Al-Javyousi 1999; Christou & Christodoulou 2015). Brackish river water can cause important issues that need to be considered for a correct operation of BWDPs, especially in water-stress areas like the Mediterranean basin where sharp changes in water flow and quality occur, facing even hourly fluctuations in its characteristics (i.e. solids concentration, salinity, natural organic matter (NOM)) which cause severe destabilization episodes in BWDP operation (Bonada & Resh 2013). For this reason, new high performance pretreatment systems are required to ensure a stable operation of BWDPs to guarantee a continuous potable water supply, as well as satisfy the ecological water functions.

Membrane technologies are well established pretreatment processes in desalination facilities where surface brackish water is processed (Fritzmann *et al.* 2007). However, the treatment of fluctuating surface water characterized by a high fouling potential is still a major concern and a critical issue for membrane-based systems implementation (Kimura *et al.* 2004).

It is widely accepted that reverse osmosis (RO) is the most adopted desalination technology worldwide. This membrane-based process uses polymeric membranes to remove molecules and ions from water, obtaining a pure water stream (Greenlee et al. 2009). Figure 1 shows the cumulative production capacity of the 40 largest RO plants (DesalData 2016). The pretreatment capacity is nearly double the plant production capacity, considering 50% recovery in the RO system. It is also important to mention that dissolved air flotation has been coupled to ultrafiltration (UF) and dual media filters in large RO plants as additional pretreatment for coping with difficult feeds and dealing with algal bloom events (Villacorte et al. 2015). Recently, membrane-based pretreatment has been gaining acceptance as a viable alternative to conventional processes, especially in desalination facilities where complex feed water is processed.

RO membranes are extremely sensitive to some pollutants and foulant materials, which require a pretreatment where these compounds must be removed to preserve the performance and lifetime of RO membranes. Broadly, a proper selection of pretreatment methods and pre-conditioning configurations can significantly improve the effectiveness and extend the life span of the RO system by preventing or minimizing particulate, colloidal and biological fouling, scaling and membrane plugging, as well as reducing chemical membrane cleaning requirements (Henthorne & Boysen 2015). This makes the selection and operation of the pretreatment a crucial part of the desalination facility (Jamaly *et al.* 2014). The quality of RO feed water, the resulting effluent from the pretreatment, is assessed by means of the silt density index (SDI) whose value should be less than 5 and



Figure 1 | Pretreatment capacity of 40 largest RO plants: ultrafiltration and media filtration.

preferably less than 3 (Greenlee *et al.* 2009). Otherwise, the RO system can be overloaded, membranes get fouled rapidly and frequent cleanings are required to restore productivity and salt rejection (Jamaly *et al.* 2014). Hence, cleaning costs, system performance and stand-still time are very significant.

In the city of Denia, eastern coast of Spain, potable water is produced in desalination plants where brackish surface water and groundwater are the main sources. Owing to the intense touristic activity, the population and water abstraction increase five-fold in summer when water is scarcer, which leads to a significant pressure on water sources. This makes necessary the intensification of drinking water production, the groundwater source being insufficient in the area. Thus, drinking water is also produced from brackish surface water collected from the Racons river in a BWDP. This brackish river suffers hourly fluctuations in its characteristics, which make the steady-state operation of this BWDP very difficult. Thus, new pretreatments are required to ensure a stable operation of this BWDP and guarantee the potable water supply, as well as satisfy other water functions. For this reason, brackish surface water has been pretreated in a membrane-based (submerged UF polymeric) system where process performance, fouling evolution, membrane cleaning and water quality have been evaluated at pilot scale.

MATERIALS AND METHODS

This study was carried out at the Racons BWDP located in Denia, where brackish surface water was collected from the Racons river. This is a short and irregular river, with its spring in the mountain range near the coast. The river drains the mountains and drives the water to the sea, in a short and turbulent path where the water is collected or discharged for different agricultural uses. The occurrence of strong storms and the intense abstraction and/or discharge cause sudden and significant changes in flow and water composition (Table 1). Usually, the water contains high concentration of suspended solids, organic matter, and turbidity levels, generating a muddy ecosystem near the mouth of the river (intake point). The pilot plant was operated over 5 months, from May to October, when water flow and composition suffered severe fluctuations, and water stress was remarkable.

A UF membrane pilot plant equipped with a commercial submerged UF membrane made from polyvinylidene difluoride, with 49 m² of membrane surface and 0.03 μ m nominal pore size, was operated using an out–in filtration configuration

Parameter	Units	Minimum	Maximum	Average	
Calcium	mg/L	56.0	264.0	169.9	
Magnesium	mg/L	55.2	248.0	113.7	
Sulfate	mg/L	152.0	640.0	322.7	
Chloride	mg/L	862.0	3,522.0	1,745.4	
Total hardness	mg/L	480.0	1,680.0	908.5	
Temperature	°C	11.0	29.0	19.5	
pН	-	7.2	8.0	7.7	
Suspended solids	mg/L	0.8	39.6	13	
Turbidity	NTU	2.0	64.7	9.5	
Conductivity	μS/cm	1,490	30,900	6,161	
Organic matter	mg/L	1.9	10.1	4.8	

 Table 1
 Brackish river composition

and dead-end mode. The pilot plant can reach a treatment capacity (permeate production) of $1-4 \text{ m}^3$ /h and a feed/permeate conversion of around 92%. This pilot plant was fully automated, running without continuous supervision and recording all the process (water flows, pressure, transmembrane pressure (TMP), temperature, membrane permeability) and water quality (feed and permeate turbidity) data.

The plant was operated at low permeate flux during the start-up period. Then, permeate flux was gradually increased after some weeks of operation when the permeate quality and membrane performance (in terms of membrane fouling and permeability recovery) reached pseudo steady-state values and the plant ran in a stable manner. Cleaning requirements (frequency, intensity and chemical reagents involved) were also evaluated to optimize reagent consumption, filtration cycle and cleaning process.

The membrane fouling was evaluated through the increase of TMP, in terms of total fouling. Data were continuously saved to determine the fouling kinetics (TMP increase during membrane filtration for a certain time), and monitor how fast the membrane gets fouled. This parameter provides information about how the membrane behaves with this specific water quality treated under certain operation parameters (flux, filtration run length, cleaning procedures).

Membrane cleanings were performed when the TMP increased notably (over 0.6 bar) or when a different pilot stage was finished. The cleaning strategies were developed following two different mechanisms: mechanical or chemical (described and discussed in the 'Results' section).

Feed and permeate were characterized according to Standard Methods (APHA et al. 2005). Organic substances were further analyzed by liquid chromatography with organic carbon/nitrogen detection (LC-OCD/OND) to characterize and quantify the dissolved organic matter based on their molecular size distribution in water.

Solids particle size distribution was analyzed using an integrated laser diffractor by recirculating suspensions through the optical unit of a Malvern Mastersizer 2000 (Malvern Instrument Ltd, Worcestershire, UK), yielding particle sizes accumulative distribution.

RESULTS AND DISCUSSION

Feed brackish water composition

The pilot plant was operated for 5 months (from 09/05/2017 to 11/10/2017), treating the brackish water from the Racons river. Different flux values and filtration periods were tested to evaluate the influence of different operation parameters in the system performance, cleaning requirements and permeate quality to be subsequently submitted to an RO desalination process (Figure 5).

The feed quality was analyzed extensively to determine its composition over a long-term basis. During the experimental period, the feed showed very significant and sharp changes in flow and composition, which made difficult the plant operation. However, it was an excellent scenario to increase knowledge about membrane-based technologies and the capacity to cope with extreme conditions and facilitate desalination plant operation under challenging scenarios. Table 1 summarizes the river characteristics:

Particle size distribution analysis in brackish river water showed an average particle size of $47.76 \pm 0.51 \,\mu$ m. The cumulative distribution can be observed in Figure 2. The majority of the particles (80%) are distributed between 17 and 83 µm. A small amount of particles, around 2.5% of the particles are below 0.3 µm, are in a range 10 times bigger than the pore size. These particles can cause severe pore blocking problems during the membrane filtration, since the small particles that are close to the pore size, with the pressure as driving force, can get embedded in the pore. However, the presence of bigger particles in sufficient quantity creates a filtration cake layer, which improves membrane filtration (Seminario *et al.* 2002; Hwang *et al.* 2008).

The presence of particles in the feed can cause permeability decline when the membrane gets fouled. During the first minutes in the filtration cycle the particles reach the clean membrane and the smaller particles can reach the pores, causing pore blocking and a more notable permeability decline. After a few minutes bigger particles reach the membrane



Figure 2 | Particle size distribution (µm) in raw brackish river water.

surface and the filtration cake is formed, resulting in a smaller and linear permeability decline (Hwang *et al.* 2008). The filtration cake increases the membrane selectivity and reduces the effective pore size. The formation of the cake layer increases the membrane filtration capacity, which can be easily removed by backwashing (Ho & Zydney 2000; Hwang *et al.* 2007).

The impact of the small particles can be mitigated by coagulant addition. Iron(III) (1 mg/L) was added as coagulant to the feed stream by an in-line injection system with a static mixer in the pipe to maximize turbulence and facilitate the coagulant action. The addition of the coagulant salt aggregates the particles present in the water, making aggregates bigger and favoring the formation of a loose filtration cake layer and therefore the membrane filtration (Matilainen *et al.* 2010).

Permeate quality

Membrane-based filtration systems are very robust and permeate obtained commonly presents good quality in every case, regardless of feed characteristics and operation conditions (Van Hoof *et al.* 1999). Table 2 shows average values of feed and permeate characteristics, as well as membrane removal efficiency. In spite of the aforementioned feed composition fluctuations, high suspended solids and turbidity removal efficiencies (above 99%) were obtained in all cases.

Figure 3 shows the time-course of turbidity levels measured in feed and permeate. Turbidity levels in permeate remained constant during all the experimental procedures, with high quality permeate despite the feed characteristics. These results are in agreement with previous studies where UF membranes have been tested as a pretreatment in desalination plants. Membrane filtration maintained a high permeate quality, even in the worst period when the turbidity increased up to 70 NTU. The SDI values were always below 3.0, suggesting an excellent effluent to be treated with RO membranes (Greenlee *et al.* 2009).

As can be observed in Figure 3, turbidity levels in the feed spiked several times during the experimental period, such as in June, July, September and October. There are several factors affecting the changes of flow and composition in this river. The river showed the special characteristics commonly found in the Mediterranean basin, with very short course, and great variations in water flow and water composition due to storms in the springs, causing the drag of suspended solids, organic matter and other compounds (Bonada & Resh 2013). Due to the intense agriculture activity in the

Table 2 Average feed and permeate composition during long-term UF experimentation

	SS (mg/L)	Turbidity (NTU)	рН	Conductiv. (mS/cm)	COD (mg/L)	Alkalinity (mg/L)	Hardness (mg/L)	Sulfate (mg/L)	Silica (mg/L)	SDI
Feed	13.2	15.7	7.4	6.29	29.6	264.8	858.7	281.8		
Perm.	0.1	0.1			21.1			263.3	8.6	2.3
Rem.	99.7	99.4			26.9			6.6		

SS: suspended solids; Conductiv.: conductivity; COD: chemical oxygen demand; SDI: silt density index; Perm.: permeate; Rem.: removal (%).



Figure 3 | Time-course of feed and permeate turbidity in the experimental period.

Mediterranean regions almost all the rivers are used for agriculture irrigation. In this case, the Racons river is intensively used for almond trees and rice crop irrigation. This causes changes as well in flow and composition due to opening/closing of lock-gates in the irrigation periods.

Membrane performance

Figure 4 shows membrane permeability values along the experimentation period, which greatly depend on turbidity of the feed. Initially, low turbidity levels in the feed led to high permeability values. After membrane adaption to the process conditions, a slight pressure decrease was observed. Then, a high suspended solids concentration caused a sudden increase of turbidity in the feed, which reduced the permeability significantly. These unstable conditions remained during all the experimentation, but membrane permeability was stabilized in the range 200–300 L/m²·h·bar (LMH/bar), which demonstrates that it is possible to operate this submerged technology membrane in a complex scenario and maintain an excellent membrane performance and permeate quality.

Different permeate fluxes were tested in order to check the influence of the flux over the membrane fouling kinetics and membrane recovery capacity upon mechanical cleaning procedures (Figure 5). For this purpose, permeate fluxes between 30 and 70 L/m²·h (LMH) were tested, treating flow rates between 1.5 and 3.4 m³/h. Membrane fouling kinetics was studied to determine the maximum flux allowed for a stable operation of UF membranes treating this specific water feed. In principle, permeate flux was expected to be the main factor causing membrane fouling, and therefore the increase of fouling kinetics. Higher flux drives more particles to the membrane, but sudden fluctuations of the feed composition was a more impacting



Figure 4 | Time-course of permeability and turbidity levels in the feed.

factor than flux in this work. Fouling kinetics is shown in Figure 6 where turbidity values (average value for each filtration cycle) are also depicted. Fouling kinetics is measured as the mbar increase in TMP per unit of time filtering feed water, in mbar/min. With this unit it is possible to compare the effect of changes in feed water composition on membrane fouling, by measuring how fast the membrane gets fouled.

It can be observed how changes in membrane flux impact on membrane fouling kinetics. Theoretically higher fluxes bring more fouling particles to the membrane surface, which gets fouled faster. The fouling kinetics remained almost constant during the first tests, and showed higher and unstable values at increasing fluxes. Nevertheless, significant changes in fouling kinetics were observed during the operation at a given permeate flux value, which indicates that feed characteristics are a relevant factor, which was critical treating a feed water characterized by a fluctuating composition (Figure 6).

Changes in turbidity impacted the membrane fouling kinetics, following a similar pattern along experimentation. In fact, sudden turbidity increases (in May and June, mainly) led to significant increases in the membrane fouling kinetics.

Turbidity is commonly related to suspended solids concentration in river water. High suspended solids concentration makes difficult the UF plant operation, especially under dead-end mode. Thus, membrane systems without air scouring during filtration periods and working in deadend strategy lead to high fouling rates, which hinder their feasible operation at suspended solids concentration higher than 100 mg/L in the membrane tank. This concentration was reached in the worst case scenario when the turbidity was over 30 NTU, when the suspended solids in the membrane tank reached more than 100 mg/L and the fouling rate had values above 6 mbar/min. With these



Figure 5 | Time-course of fouling kinetics and membrane flux during pilot plant operation.

fouling rate values the operation of the plant is severely limited and operation changes are needed.

NOM was identified as the main foulant present in raw brackish water, which caused the membranes to suffer organic and biological fouling. Figure 7 shows the LC-OCD results obtained in raw river water and permeate, concluding that UF membranes retained foulants present in river water efficiently. More than 66% of NOM was removed, reaching a removal efficiency of biopolymers and humic substances up to 86.3 and 70.4%, respectively. The removal of organics with a lower molecular was about 56% (building blocks), 51% (acids with low molecular weight, LMW) and 45% (neutral compounds with LMW). Thus, UF removal was mainly determined by size exclusion and surface charge phenomena.

These results are in agreement with those observed with UF membranes in others applications (Shon *et al.* 2004),

where the use of UF membranes with coagulant dosing, like Fe salts, can remove a substantial fraction of the organic matter present in the influent.

The foulant materials are mainly removed by size exclusion, as can be observed by the higher removal percentages in the bigger particle sizes. The addition of coagulant salts (1 mg/L Fe^{3+}) makes the particles increase their size and be more easily removed by membranes (Matilainen *et al.* 2010; Schurer *et al.* 2013). However, the nature of the particles, their chemical composition and rheological properties cannot be determined by LC-OCD. Some of the particles in all the sizes show specific rheological properties, with a flexible or compressible behavior with pressure. These specific characteristics can make these particles difficult to retain even if they are bigger than the pore size in the membrane (Villacorte *et al.* 2009) and they can cross the membrane, being found in UF permeate.



Figure 6 | Time-course of fouling rate and turbidity of the feed.



Figure 7 | Concentration of substances quantified by LC-OCD in river and UF permeate samples.

Membrane cleaning procedures

Cleaning strategies are required to restore the membrane permeability by removing foulants adhered to the membrane surface. Conventional cleaning strategies are based in mechanical and chemical cleanings, depending if chemical reagents are used or not.

Mechanical cleanings used in the present study comprise an initial air scouring followed by backwash with permeate at 1.3 times the filtration flow, and partial drainage of the membrane tank. This mechanical process is able to remove most of the foulants during normal operation, being effective enough to maintain acceptable permeability values with the help of chemical cleanings (Gao *et al.* 2011).

Chemical cleaning used in the plant follows two strategies, working with UF permeate to prepare the chemical solutions. A less aggressive chemical cleaning is performed regularly for maintenance; this more frequent and less aggressive cleaning procedure is the chemical enhanced backwash (CEB), performed with 100 ppm of sodium hypochlorite for 30 minutes contact time. A CEB every 2 days was necessary to maintain a sustainable plant performance. Otherwise, permeability started to decline notably. CEB cleanings are useful to control the fouling adhered to the membrane on a regular basis, and control the biofilm generation in the pipes and clean the face of the membrane. CEB cleaning is used to remove the foulants before the generation of a dense layer and dense biofilms, and with low chemical reagent concentration and low contact time can reduce the TMP and the fouling not removed by mechanical cleanings. With good water quality (below 10 NTU) a CEB can be performed every 4 days and the permeability can be maintained. But in the worst case scenario, when turbidity spiked up to 70 NTU, CEB must be performed much more often, even every day, to keep stable the membrane performance. CEB frequency, chemical concentration and contact time are the main working parameters for a stable operation of UF plants. Nevertheless it is necessary to reach a balance between cleaning requirements, chemical reagent consumption and chemical dosage (Porcelli & Judd 2010).

If the membrane suffers more severe fouling episodes, a more intense cleaning procedure, cleaning in place (CIP), might be necessary, by using higher concentration of chemical reagents and longer contact time. In this case two strategies were followed, an oxidant chemical cleaning with two different compounds (sodium hypochlorite and hydrogen peroxide) and acid chemical cleaning. The oxidant chemical cleaning was performed when the organic matter fraction was predominant (Porcelli & Judd 2010). Oxidation degrades the NOM functional groups to carboxyl, ketonic and aldehyde groups, which makes them more susceptible to hydrolysis and increases hydrophilicity of their parent compounds. Therefore, oxidation reduces the adhesion of fouling materials to membranes (Thurman 1985; Liu et al. 2001). In this study two oxidant chemical reagents were used: sodium hypochlorite and hydrogen peroxide. Sodium hypochlorite is a more common reagent used in chemical cleanings and the most effective (Porcelli & Judd 2010). In our case, the concentration of 500 ppm and 2 h contact time was enough to restore the permeability values and allow a sustainable performance. But hypochlorite presents a problem with water with high salt concentration, as we have with brackish water, because the addition of this reagent causes an increase in pH, which causes the formation of calcium carbonate particles in the cleaning solution, creating an extra fouling source. To avoid this formation it is possible to work with lower hypochlorite concentration to keep the pH values lower than those favoring carbonate generation or prepare chemical cleaning solutions with softened water, if available.

Hydrogen peroxide was selected as the second oxidation agent to be tested. This chemical reagent is a strong oxidant able to remove NOM, and despite being less used than hypochlorite, previous results are promising. In this study, hydrogen peroxide was dissolved in ultrafiltrated brackish water, which definitely avoided the precipitation of carbonate species. In this case 1,000 ppm of hydrogen peroxide was added for 1 hour of contact time. Figure 8 shows the performance of the chemical cleaning. It can be seen how, during the two filtration cycles prior to the chemical cleaning, the membrane permeability is low, and then the chemical cleaning is performed and the permeability is



Figure 8 | Time-course of permeability values during recovery tests using hydrogen peroxide chemical cleanings.

recovered, as can be observed in the last two filtration cycles, where the permeability is clearly recovered. The permeability is recovered after chemical cleaning with hydrogen peroxide, this compound at this concentration and contact time being effective enough to remove the foulant adhered to the membrane.

In addition, another chemical cleaning with acid was evaluated, using citric acid (1.5 g/L) and hydrochloric acid at pH 2.3, for 2 h. This cleaning method was used alone and after the oxidant CIP. In both cases the efficiency of this cleaning was limited or ineffective, which reinforces the aforementioned hypothesis that NOM is the main fouling source in the brackish river feed studied in the present work.

Acid cleanings are very effective when inorganic fouling is the prevailing fouling form (Porcelli & Judd 2010), or when it is required to remove the excess of iron salts from coagulant adhered to the membrane. If organic and biological fouling are the main component in membrane fouling the addition of acid can be counter-productive and worsens the fouling problems because of the reaction of the components. Biofilms generated in the membrane are formed from many different components, mainly polysaccharides and proteins (Flemming & Wingender 2010). Some acids have a mildly oxidative action with organic matter, forming soluble aromatic aldehydes and acids at NOM functional groups (Thurman 1985). But if the biofilms have a strong presence of proteins the interaction of the acids with proteins presents in the biofilm can produce the denaturing of the protein and the generation of a gel-like structure which make worse the fouling problems (Totosaus et al. 2002; Mohammadi et al. 2003).

CONCLUSIONS

The ultrafiltration technology is a viable option in extremely complex brackish natural waters treatment as a pretreatment for RO desalination plants. The operation of UF plants in fluctuating environments is a feasible option, assuring the quality of the permeate in all cases. Permeate obtained is characterized by a very good quality as feed for RO and stable in time. With turbidity values in feed water up to 80 NTU, the permeate turbidity was stable around 0.1 NTU. The UF membranes achieved a significant removal of organic matter, mainly of the bigger particle fractions, with up to 80.6% removal efficiencies of biopolymer and 70.4% of humic acids.

Mediterranean rivers used as source for fresh water production present a high complexity in treatment due to the intense abstraction, water scarcity and climate characteristics, suffering sudden and intense changes in composition. Turbidity of the feed was mainly associated with the suspended solids concentration, which clearly impacted membrane fouling rate, being more relevant than membrane flux in the specific tested conditions. Nevertheless, the plant was able to be operated up to 70 LMH in the worst conditions with the addition of 1 mg Fe³⁺/L to increase particle size and facilitate membrane filtration.

The main foulant agent present in the river water was NOM, the oxidant chemical cleaning being the most effective in permeability recovery. Both sodium hypochlorite and hydrogen peroxide were effective to keep the membrane performance, hydrogen peroxide being a better solution if softened water is not available. Acid cleanings were not effective and can be counter-productive if biofilm is developed on the membrane surface. CEB can be performed every 4 days if the water presents turbidities below 10 NTU. In the worst case scenario, with high turbidity episodes, it is necessary to perform a 100 ppm CEB with sodium hypochlorite every day.

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