Optimization of Coagulation Pretreatment Conditions in a Ceramic Membrane System

AMIR ALANSARI,[1] MERIC SELBES,[2] TANJU KARANFIL,[3] AND JAMES AMBURGEY[1]

[1]Department of Civil and Environmental Engineering, University of North Carolina at Charlotte
[2]Hazen and Sawyer, Fairfax, Va.
[3]Department of Environmental Engineering and Earth Sciences, Clemson University, Anderson, S.C.

The goal of this project was to identify optimal coagulation pretreatment conditions and cleaning protocols in a pilot-scale ceramic membrane system. A matrix of coagulation conditions (coagulant dose and pH) was investigated using aluminum sulfate and ferric chloride at two source waters (river and lake). Increasing backwash frequency to every 30 min and selecting a 30-s interval proved to be critical since it reduced the effects of irreversible fouling during operation. After optimizing the clean-in-place procedure, the membranes were consistently recovered to 4 to 5 psi after the cleaning process. Results showed that optimal pretreatment conditions with aluminum sulfate reduced membrane total fouling rates by at least 80% relative to the control train. Optimal pretreatment with ferric chloride reduced membrane total fouling rates by 95% relative to the control train; however, the pretreatment conditions in which ferric chloride was effective were narrow compared with aluminum sulfate.

Membrane filters are gaining considerable attention for surface water treatment applications in the United States. Membrane systems are relatively easy to install, operate, and maintain while having a significantly smaller footprint compared with conventional treatment processes (Guo & Hu 2012, Li et al. 2011, Huang et al. 2009, Lerch et al. 2005, Young-june Choi & Dempsey 2004, Mo & Huang 2003). Furthermore, the quality of the product water is typically less affected by fluctuations in the source water. Ceramic membranes for drinking water treatment can now be regarded as an alternative to the widely used polymeric membranes (Hofs et al. 2011, Loi-Brügger et al. 2006, Lee & Cho 2004). Ceramic membranes have better structural, chemical, and thermal stability compared with polymeric membranes. Consequently, ceramic membranes can be operated at higher fluxes and extended backwash intervals, and they can tolerate extreme cleaning protocols (Hofs et al. 2011, Mueller et al. 2010, Lehman et al. 2008, Loi-Brügger et al. 2006). Applicable research studies that demonstrate whether ceramic membranes can meet the current US drinking water quality standards are limited. Little is known about optimizing the chemical pretreatment scheme for ceramic membrane systems to minimize the fouling or to enhance the removal of contaminants in US source waters.

BACKGROUND

Membrane fouling. Low-pressure membrane processes such as microfiltration and ultrafiltration rely on particle size exclusion; hence, most particles that are larger than the nominal membrane pore size are rejected and tend to accumulate on the membrane surface or within the membrane pores. The accumulation of these particles can reduce the permeability of the membrane and increase operating pressures. The reduction in membrane permeability, which is an inevitable product of membrane filtration, is called membrane fouling. Fouling agents can completely block membrane pores, constrict the flow through the pores, or decrease the membrane permeability by forming a cake layer (Huang et al. 2009, 2008). Membrane fouling is generally classified operationally as being either reversible or irreversible. If the effects of membrane fouling can be reversed via a hydraulic backwash only, the fouling type is said to be reversible. If a hydraulic backwash is insufficient in reversing the effects of membrane fouling, the fouling is said to be irreversible (Huang et al. 2009). In the case of irreversible fouling, a chemically enhanced backwash (CEB) or a clean-in-place (CIP) process must be used to alter the affinity of the particles to the membrane and reverse the effects of fouling (Hofs et al. 2011; Meyn & Leiknes 2010; Huang et al. 2009, 2008; Meyn et al. 2008).

Coagulation pretreatment. Coagulation pretreatment has been demonstrated as an effective pretreatment solution for microfiltration not only for controlling fouling but also for removing

contaminants smaller than the membrane pores (Meyn & Leiknes 2010, Huang et al. 2009, Meyn et al. 2008, Loi-Brügger et al. 2006, Fiksdal & Leiknes 2006, Matsushita et al. 2005, Matsui et al. 2003, Bérubé et al. 2002, Judd & Hillis 2001). However, understanding coagulation chemistry and the factors that control the coagulation process is important for its successful implementation as a pretreatment stage. Howe and Clark (2006) showed that coagulation pretreatment can have negative impacts on the overall performance of a membrane system when not performed properly. Coagulation pretreatment can improve membrane performance since particles get incorporated into flocs that are larger than the membrane pores; therefore, fewer particles have a chance of blocking the membrane pores (Kennedy et al. 2003).

Pretreatment configuration. Several studies have been performed in which conventional two-stage coagulation/flocculation mixers were compared with inline static mixers. Lehman et al. (2008) observed that with inline flocculation, the decline in specific flux was only 9% from the initial flux, while the specific flux decline was about 22% with the two-stage coagulation/flocculation. Meyn and Leiknes (2010) compared three ceramic coagulant mixing schemes and their impact on dissolved organic carbon (DOC) removal, membrane fouling rate, and residual coagulant concentrations. The first was a conventional rapid mix and flocculation stage with a retention time of 21 min, the second was only a rapid mix stage with a retention time of 7 min, and the third was an inline static mixer with a retention time of 45 s. Using a constant dose of 10 mg/L as iron (Fe) (0.18 mM Fe) at pH 5 for all configurations, the authors observed that the DOC removal was approximately 80% for all configurations. Matsui et al. (2003) observed that for the same coagulant dose, virus removals were similar for both systems that used inline static mixers and conventional rapid mix/flocculation stages. Meyn al. (2012) and Matsushita et al. (2005) also reported that virus removal was similar for both inline and two-stage coagulation/flocculation systems.

OBJECTIVES

The main objective of this research project was to evaluate the impact of coagulant type, coagulant dosage, and coagulation pH on the performance of a pilot-scale low-pressure ceramic membrane system for two surface water sources. This study investigated optimal pretreatment conditions, in terms of membrane fouling rate reduction, for each coagulant. Cleaning protocols (i.e., backwashing, CIP) were optimized to minimize the effects of irreversible fouling.

MATERIALS AND METHODS

Source waters. Raw water was supplied to the mobile pilot-scale coagulation–ceramic membrane treatment system from the raw water reservoirs at two drinking water treatment plants. Both water sources had relatively low DOC concentrations and moderate to low specific ultraviolet absorbance ($SUVA_{254}$) values. The turbidities of the two waters differed significantly. A summary of the raw water quality characteristics during the pilot testing is given in Table 1.

Membrane specifications. Ceramic membrane hollow fibers[1] with a nominal pore size of 0.1 μm were used in this study. Each

TABLE 1	Source water characteristics	
Parameter	**River Average (Min–Max)**	**Lake Average (Min–Max)**
Temperature—°C	19 (10–34)	27 (15–37)
Turbidity—ntu	8 (2–46)	2 (1–3)
Color—cu	28 (8–99)	22 (16–31)
pH	6.9 (6.7–8.9)	7.1 (6.9–7.3)
Alkalinity[a]—ppm	26 (13–32)	18 (15 –22)
Hardness[a]—ppm	28 (22–33)	20 (16–23)
Conductivity—μS/cm	143 (79–161)	71 (23–82)
UV_{254}—m^{-1}	6.7 (5.8–7.9)	2.7 (2.2–3.2)
DOC—mg/L	2.7 (2.6–2.8)	1.7 (1.7–1.8)
$SUVA_{254}$—L/mg-m	2.4 (2.1–3.0)	1.6 (1.3–1.9)

$CaCO_3$—calcium carbonate, DOC—dissolved organic carbon, $SUVA_{254}$—specific ultraviolet absorbance, UV—ultraviolet

[a]Concentrations reported as $CaCO_3$ equivalents

membrane element contained approximately 300 fibers and a total surface area of 0.8 m². Each fiber had an inner diameter of 3 mm and an outer diameter of 4 mm, and was 439 mm long. The membrane material was made from and coated with alpha-alumina (α-Al_2O_3). The structure was asymmetric and designed for inside to outside flows. The membrane could be backwashed (without air scouring) with pressures up to 3 bar (43.5 psi). The operating transmembrane pressure (TMP) can be up to 6 bar (87 psi). The ceramic membrane modules used could tolerate pH from 0 to 14 and temperatures from –5 to 120°C.

Pilot configuration. The pilot system was equipped with two identical process trains (Figure 1). Raw water entered the pilot facility through a 1-in.-diameter polyvinyl chloride (PVC) supply line and emptied into a 30-gal (114-L) circular high-density polyethylene (HDPE) tank[2] equipped with an overflow. Water then passed through a high-capacity 100-mesh (150-μm) pleated stainless steel strainer. The pH of each train was independently adjusted by acid/base addition via two digital peristaltic pumps[3] until the target pH on the permeate side was reached. Coagulant was added, coagulated train only, via a peristaltic pump[4] upstream of a 24-element static mixer with a 0.25-in. (6.4-mm) inner diameter (ID).[5] Filtered water quality was independently monitored from each treatment train by online pH meters[6,7] and turbidimeters.[8] Permeate flow rates were measured by magnetic flow meters.[9,10] Membrane feed and backwash pressures were measured with pressure transducers.[11] A computer with development environment software[12] using analog to digital converters[13] was used to record data and control automatic operations of the pilot plant. The software also served to halt system operation should the TMP exceed 30 psi (2.07 bar). For each train TMP, turbidity, pH, and flow rate were logged using the development environment software at 10-s intervals. The data logged at each consecutive time interval were the result of signal averaging over a certain period; therefore, minor differences can be found between the online instruments and the recorded values in the software.

Air-actuated three-way stainless steel ball valves[14] were used with an air manifold to switch between normal operation and backwash flows. A gear pump[15] was used to backwash the system at an initial pressure of 25 psi (1.72 bar). Water was pumped through the membranes in dead-end mode at a constant flux of 100 L/m^2-h (59 gfd) with gear pumps mounted on 56C face motors[16] controlled by variable frequency drives.[17] The flux was held constant at all times via the variable frequency drive's (VFD's) built-in proportional-integral-derivative (PID) controller. The VFDs used the output from the flow meters to match the user-specified set points by increasing or decreasing the motor speed. The membranes were hydraulically backwashed with the permeate water at an initial pressure of 25 psi (1.72 bar) and flow rate of approximately 3.4 L/min (0.9 gpm). A CIP was performed in order to recover the membranes to a common baseline pressure before the start of each experiment. Table 2 summarizes the coagulants and conditions selected on the basis of values typically reported in the literature. Each coagulant dose was investigated at three pH levels, thereby creating a 3 × 3 matrix of investigated conditions. Stock solutions of aluminum sulfate[18] and ferric chloride[19] were prepared by dissolving 10 g of the chemical in 10 L of distilled deionized water (DDW). Ferric chloride doses were selected on the basis of equimolar concentrations as aluminum. Sodium hydroxide pellets[20] were dissolved in 20 L DDW to achieve concentrations ranging from

TABLE 2	Summary of coagulants investigated		
Coagulant Type	**Investigated Site**	**Doses**	**pH**
Aluminum sulfate (Al$_2$(SO$_4$)$_3$ • 18 H$_2$O)	River and lake	0.5, 1.3, 3.5 mg Al/L[a] (0.019, 0.05, 0.13 mM Al)	5.5, 6.0, 7.0
Ferric chloride (FeCl$_3$ • 6 H$_2$O)	Lake	1.0, 2.6, 7.0 mg Fe/L[b] (0.018, 0.05, 0.13 mM Fe)	4.5, 5.5, 7.0

Al—aluminum, Al$_2$(SO$_4$)$_3$—aluminum sulfate, Fe—iron, FeCl$_3$—ferric chloride

[a]1 mg/L as Al ≈ 12.3 mg/L as aluminum sulfate
[b]1 mg/L as Fe ≈ 4.84 mg/L as ferric chloride

0.01 to 0.1 N. Hydrochloric acid[21] was diluted in 20 L DDW to achieve concentrations ranging from 0.01 to 0.1 N. The dispense rate of the pH adjusting chemical was manually set until the target pH was reached and stabilized.
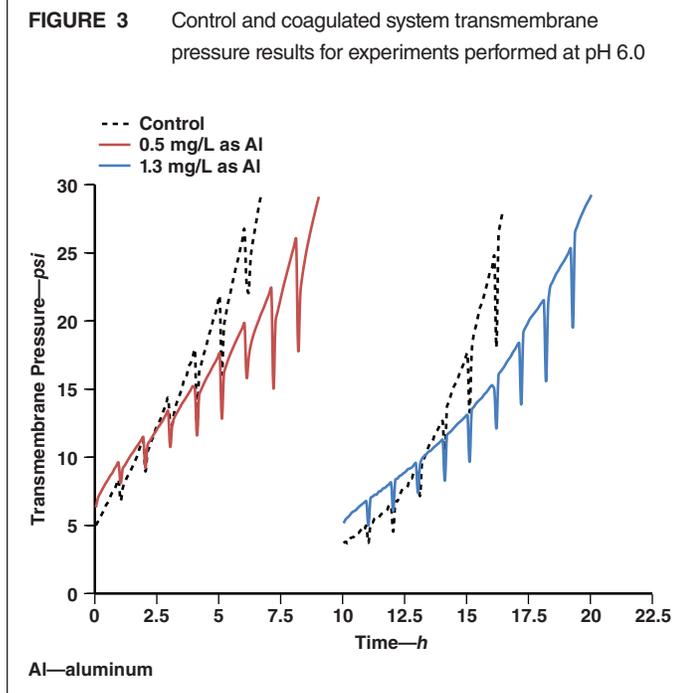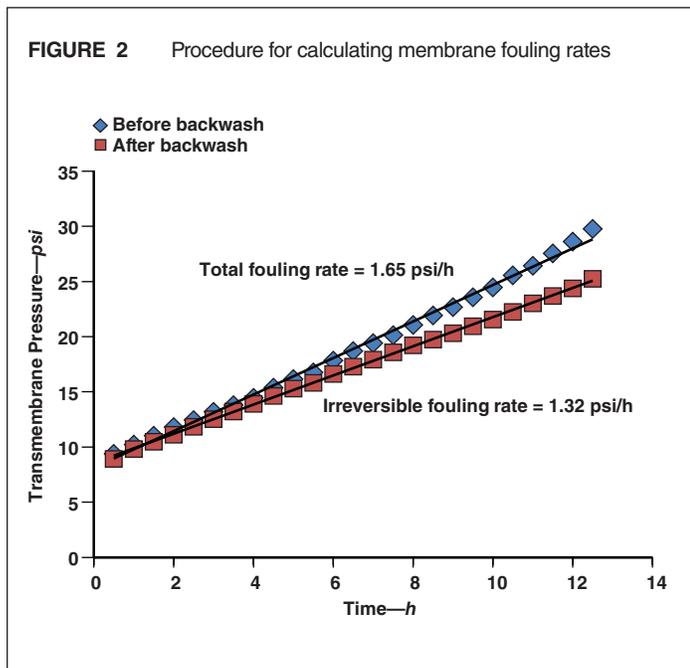
Membrane fouling rate. Membrane fouling was quantified in this study as the rate of transmembrane pressure gain per hour. TMP data before and after each backwash cycle were extracted from the unfiltered logged pressure data by following procedures described by Meyn and Leiknes (2010) and Zhu et al. (2012). This was the pressure immediately before the membrane feed

FIGURE 1   Schematic of the hybrid coagulation–ceramic membrane system (coagulated train)
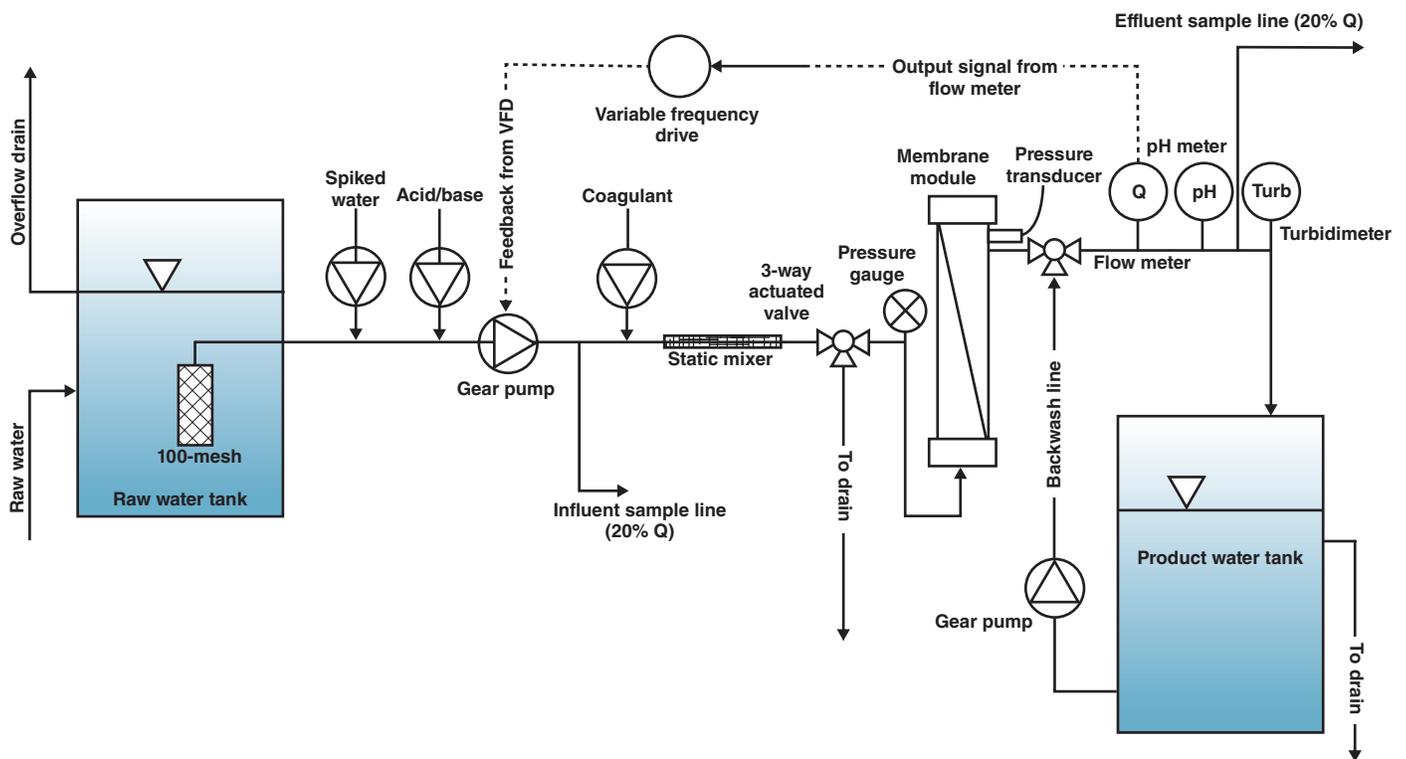
Q—flow rate that is measured by the flow meter, VFD—variable frequency drive

pumps were stopped for the backwash sequence and 1 min after backwash to account for the pump ramp-up period. An illustration of the raw data extraction and membrane fouling rate computation is shown in Figure 2. The slope of the "after backwash" plot represents the rate of irreversible fouling, while the slope of the "before backwash" plot represents the rate of total fouling, the sum of reversible and irreversible fouling rates. Based on r^2 values, it was determined that either a linear regression or an exponential transformation could be used to calculate membrane fouling rates; however, for purposes of consistency and simplicity, only the linear regression method was used in this study.

FIGURE 2   Procedure for calculating membrane fouling rates

FIGURE 3   Control and coagulated system transmembrane pressure results for experiments performed at pH 6.0

Preliminary experiments. Preliminary experiments, using the 3 × 3 aluminum sulfate matrix (Table 2), were performed at the river source location. The backwash frequency and duration were set to 1 h and 30 s, respectively. The CIP protocol involved soaking the membranes in a 6,400-ppm phosphoric acid solution for 1 h. Another set of preliminary experiments was performed to optimize backwash parameters such as duration and frequency. These parameters were systematically adjusted to improve the overall performance of the system by maximizing the volume of product water recovery, extending filtration times, and reducing irreversible fouling rates. The backwash durations selected were 100, 55, 30, 20, and 10 s. The backwash modification experiments were performed at a coagulant dose of 1.3 mg/L as aluminum (Al), pH 5.5, 30 min backwash intervals, initial backwash pressure of 25 psi (1.72 bar), and backwash flow rate of approximately 3.4 L/min.

RESULTS AND DISCUSSION

Preliminary experiments. Preliminary experiments at the river source location were performed using aluminum sulfate at the conditions described in Table 2. At the conditions investigated, both the control and coagulated trains reached the 30-psi cutoff limit within 10 h or less. Very little or no TMP recovery was observed after each hydraulic backwash. Figure 3 shows this for two experiments performed at pH 6.0 using aluminum sulfate (coagulated train only) at concentrations of 0.5 and 1.3 mg/L as Al, respectively. Similar trends were observed at all of the other coagulation conditions, which were published in a report by Amburgey and colleagues (2014). This was an indication that backwash parameters needed to be modified. A 1-h backwash frequency was originally used for the preliminary experiments; therefore, in an attempt to improve the hydraulic backwash TMP recovery, the frequency was reduced to 30 min. Additionally, the effect of backwash duration on the total membrane fouling rate at the same conditions was investigated (Figure 4). The lowest fouling rates were observed at

FIGURE 4   The effect of backwash duration on membrane fouling rate
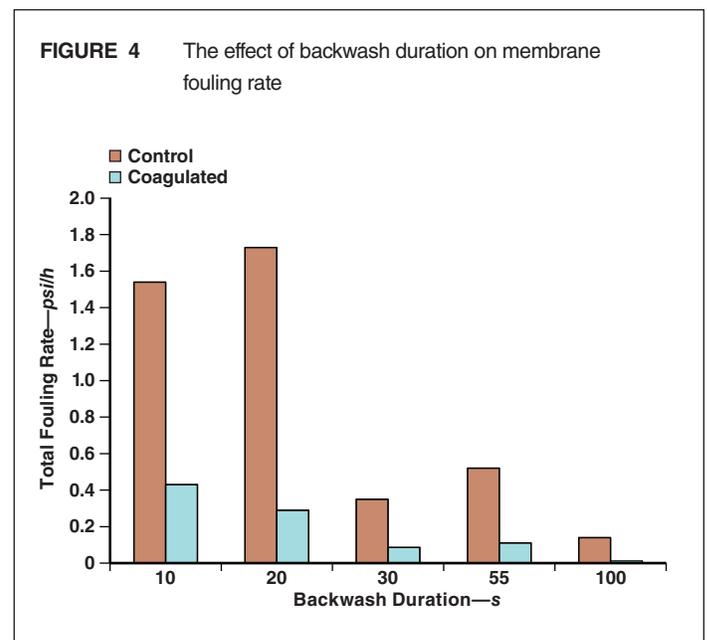
the 100-s backwash duration in both the control and coagulated systems. At a constant flux of 100 L/m²-h and a 30-min backwash frequency, approximately 10% of the filtered water would be needed for backwashing alone if the 100-s duration was selected. The authors regarded this as being operationally impractical and likely not economical due to the high demand of product water; therefore, this option was not selected. No significant differences were observed between the 55- and 30-s backwash durations in either system. At 30 s, only 3% of the filtered water would be needed for backwashing compared with 5% at 55 s. As a result, the backwash protocol was modified by setting the duration to 30 s and the frequency to 30 min.

The preliminary experiments also showed that the CIP procedure using phosphoric acid (6,400 mg/L) alone was inconsistent and sometimes insufficient in recovering the TMP after the membranes were fouled (Figure 5). The pressure range after CIP was 4–8 psi and 5–25 psi for the control and coagulated trains, respectively.

Several authors have reported that sodium hypochlorite (i.e., bleach) is an effective cleaning agent for recovering fouled membranes (Porcelli & Judd 2010, Strugholtz et al. 2005). As a result, the CIP procedure was modified to include a 30-min soak in bleach (350 mg/L) followed by a 10-min soak in phosphoric acid (1,300 mg/L). With the modified CIP procedure, the TMP was effectively recovered to 3.5–5 psi in both the control and coagulated train after each experiment (Amburgey et al. 2014).

Figure 6 compares the coagulated train TMP results for experiments performed before and after optimization of the backwash and cleaning protocols under the same coagulation pretreatment conditions for the coagulated train. It should be noted that the experiments being compared were not performed on the same day. It was observed that reducing the frequency to 30 min was effective at recovering the TMP after each filtration cycle. Also, the TMP reached a steady state after an initial increase in which the effects of irreversible fouling became negligible. The starting

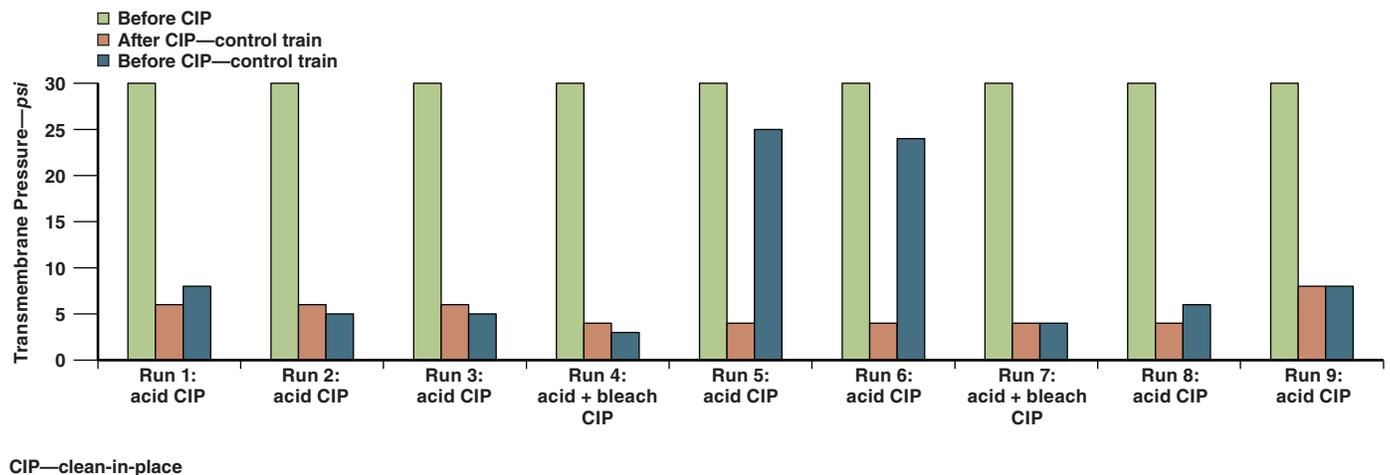**FIGURE 5**   Preliminary experiments of transmembrane pressure results before and after clean-in-place protocols

CIP—clean-in-place

FIGURE 6   Results of optimizing backwash and clean-in-place protocols under the same coagulation pretreatment conditions

Al—aluminum

FIGURE 7   Aluminum sulfate pretreatment transmembrane pressure results at the river source

AI—aluminum
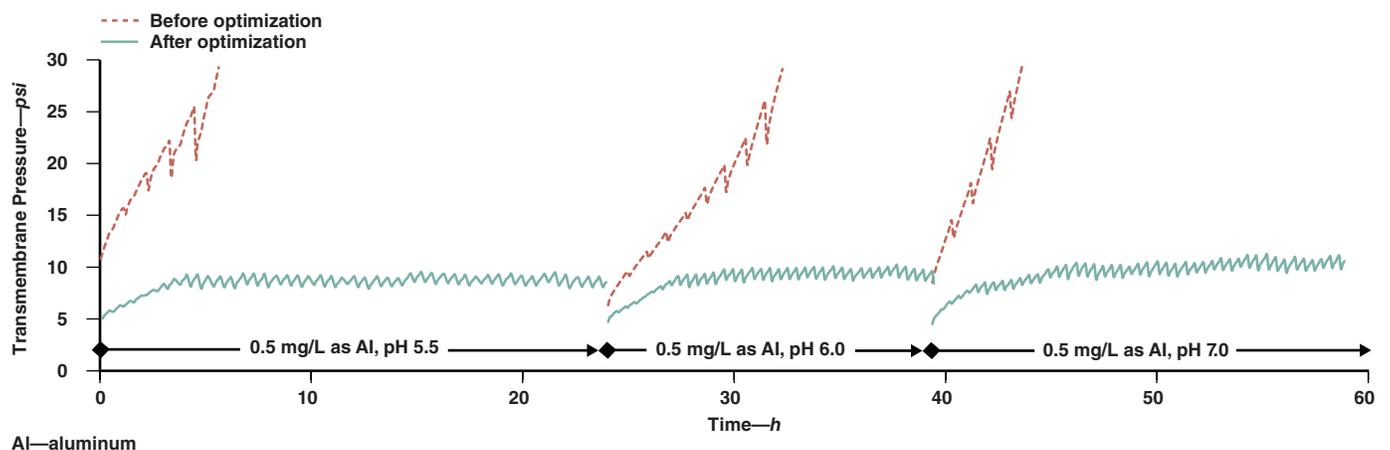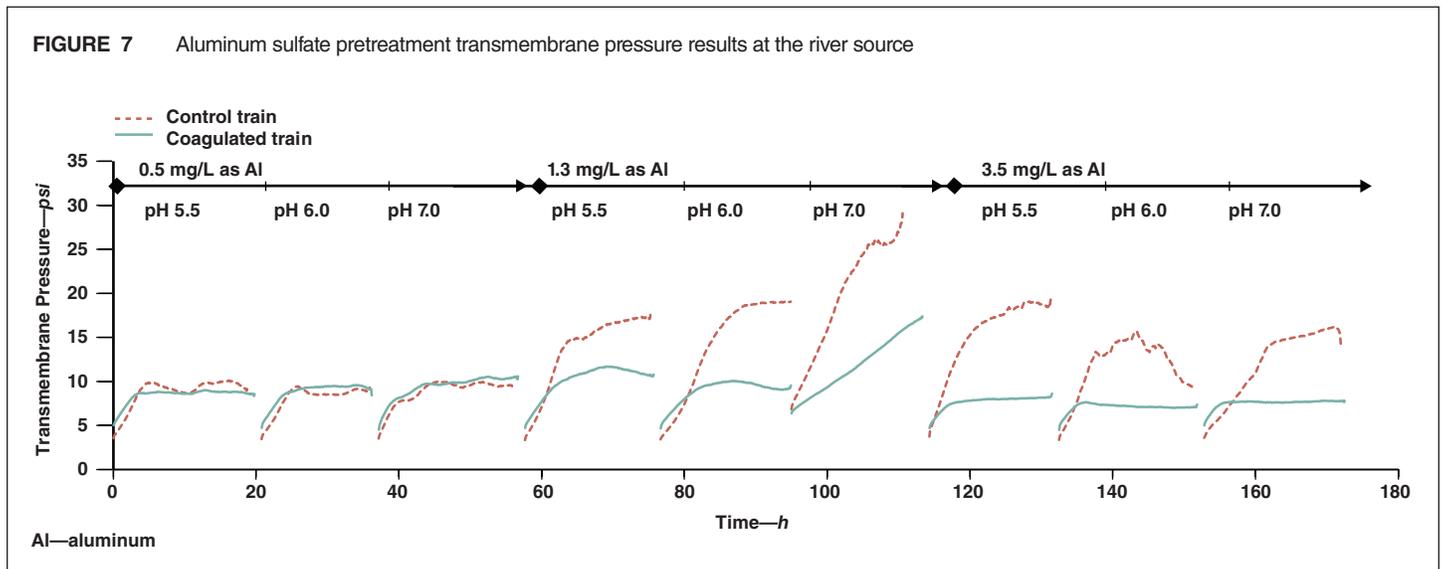
TMP was more consistent after optimization as a result of the new CIP protocol. These results highlight the importance of optimizing backwash and cleaning protocols in order to minimize the effects of irreversible fouling, maximize the volume of product water recovery, and extend filtration times.

Coagulation pretreatment optimization. Nine experiments were performed with each coagulant. Each experiment lasted 24 h or until the pressure limit of 30 psi was reached. Only aluminum sulfate was investigated at the river water source location. The aluminum sulfate dose ranged from 0.5 to 3.5 mg/L as Al, and the pH ranged from 5.5 to 7.0. It was observed that for most experiments, the system experienced an initial linear increase in TMP in which most of the fouling was irreversible before reaching a stable pressure when fouling became mostly reversible (Figure 7). It should be noted that the data shown in Figure 7 were averaged and smoothed for the general purposes of presenting the TMP trends only.

TMP results of both the control and coagulated trains were analyzed from the logged data to obtain the total membrane fouling rates shown in Figure 8. There did not appear to be a fouling rate trend that was dependent on pH for the control train. The variability in the control train fouling rates was due to changes in the source water quality characteristics between experiments. At a dose of 0.5 mg/L as Al, the coagulant was effective only at a pH level of 5.5 (Figure 8). There was improvement in fouling rate reduction when the coagulant dose was increased to 1.3 and 3.5 mg/L as Al with respect to the control train. The highest reduction in fouling rate (97%) was observed at a coagulant dose of 3.5 mg/L as Al and pH 6.0. However, the improvement when more than doubling the coagulant dose from 1.3 to 3.5 mg/L as Al was minor. Not only would increasing the coagulant dose increase chemical costs, but the higher coagulant dose would lead to an increase in the amount of solids generated. As a result, the optimal dose in terms of fouling rate reduction was selected at 1.3 mg/L as Al and pH of 6.0 for the ceramic membranes being used at the selected water sources.

For the lake water, both aluminum sulfate and ferric chloride were investigated. Figure 9 shows the general TMP trend of both trains using aluminum sulfate pretreatment (coagulated train only). Figure 10 shows the results of the total fouling rates of the control and coagulated trains for aluminum sulfate. The control train results had no distinguishable pH-based/fouling trends. None of the control train experiments achieved a stable TMP. Most control train experiments lasted between 8 and 15 h before reaching the 30 psi pressure limit. On the other hand, coagulated
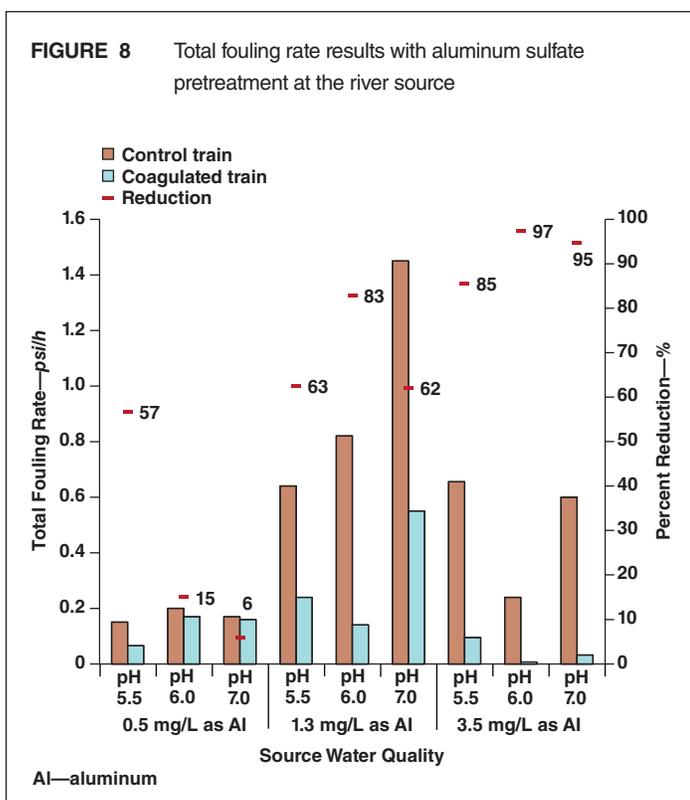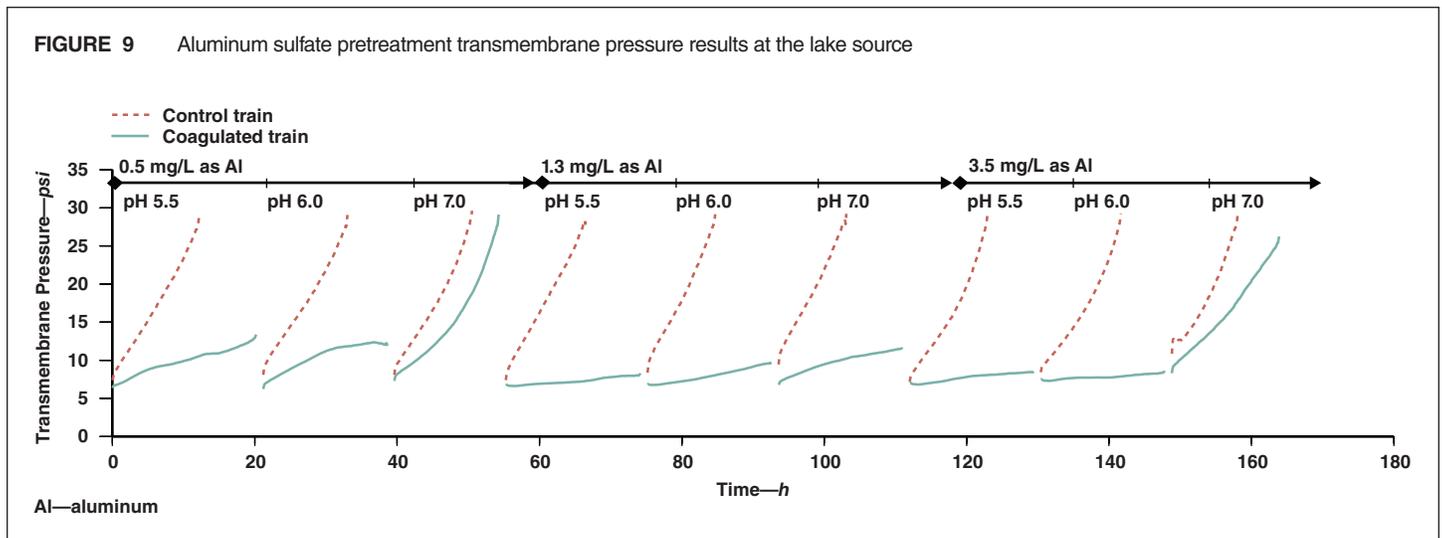

FIGURE 8   Total fouling rate results with aluminum sulfate pretreatment at the river source

AI—aluminum

FIGURE 9   Aluminum sulfate pretreatment transmembrane pressure results at the lake source

Al—aluminum

train results showed that coagulation pretreatment was effective in reducing membrane fouling in most cases and stabilized the TMP. At the coagulant doses of 0.5 and 1.3 mg/L as Al, it appeared that the total fouling rate increased as the pH was increased from 5.5 to 7.0. At pH 7.0, a relatively stable TMP could only be achieved at a dose of 1.3 mg/L as Al; however, the rates of membrane fouling were still lower with coagulation pretreatment compared with the control experiments. The quality of raw water was not a contributing factor for the relatively poorer performance of the experiments conducted at pH 7.0; thus, it is most likely related to the coagulation pretreatment conditions (i.e., coagulant dose and pH). Coagulation pretreatment was at least 80% effective

FIGURE 10   Total fouling rate results with aluminum sulfate pretreatment at the lake source

Al—aluminum

in reducing membrane fouling, except at a dose of 0.5 and 3.5 mg/L as Al at pH 7.0. The greatest reduction (96%) was achieved at a coagulant dose of 3.5 mg/L as Al at a pH of 6.0. Approximately 93% of total fouling was reduced at a dose of 1.3 mg/L as Al at a pH of 5.5. The addition of the coagulant consumes alkalinity, which depresses the pH of the product water; therefore, a base had to be added to maintain the pH for most experiments. It was determined that a dose of 1.3 mg/L as Al was feasible not only because it required a lower coagulant dose, but also because no pH adjustment was necessary for coagulation pretreatment. As a result, a coagulant dose of 1.3 mg/L as Al and pH of 6.0 were selected as optimal for the lake source as well as for the river source.
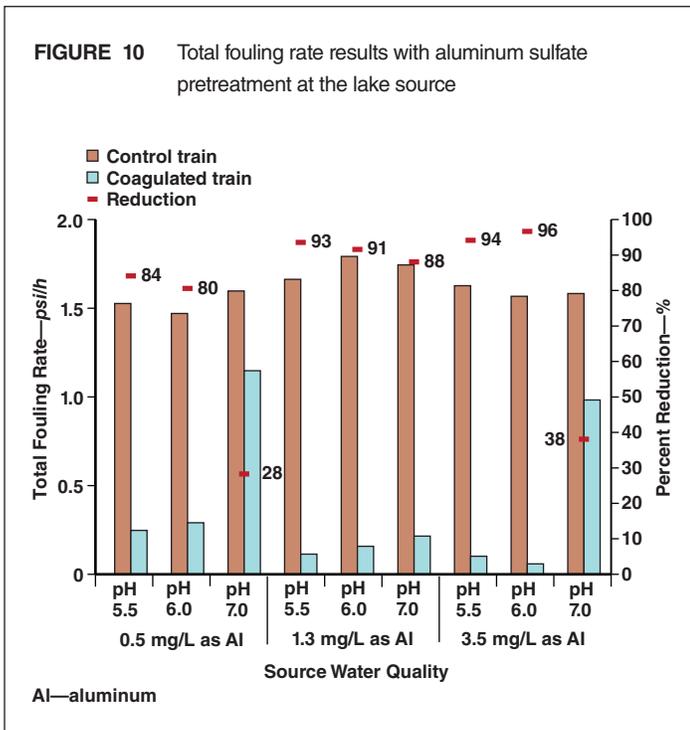
Figure 11 presents the general TMP trend for the experiments performed using ferric chloride pretreatment (coagulated train only) at the lake source. Fouling of the control train was typical, with most experiments lasting only between 8 and 15 h before reaching the pressure limit. The pressure gain was linear, and fouling was mostly irreversible. Total fouling rates for the control and coagulated train experiments with ferric chloride are shown in Figure 12. Coagulated train results indicated that at the low coagulant dose (1.0 mg/L as Fe), no reduction in membrane fouling was observed at any investigated pH level. Further, no significant improvement was observed at a pH of 7.0 at any of the investigated coagulant doses. On the other hand, membrane fouling was effectively reduced at a dose of 2.6 and 7.0 mg/L as Fe at pH 4.5 and 5.5. Only the conditions implemented at the coagulant dose of 2.6 mg/L as Fe were adequate to stabilize the TMP and minimize the effects of irreversible fouling. Most coagulated train experiments fouled irreversibly and linearly without ever reaching a stable TMP level. The highest reduction (95%) in fouling was achieved at a low pH of 5.5 and a dose of 2.6 mg/L as Fe. On a molar basis, equal concentrations of both coagulants resulted in optimal levels of fouling reduction; however, the optimal pH was 5.5 for ferric chloride and 6.0 for aluminum sulfate. Comparing the ferric chloride optimization results with aluminum sulfate optimization results, Figures 8 and 10

FIGURE 11   Ferric chloride pretreatment and transmembrane pressure results at the lake source
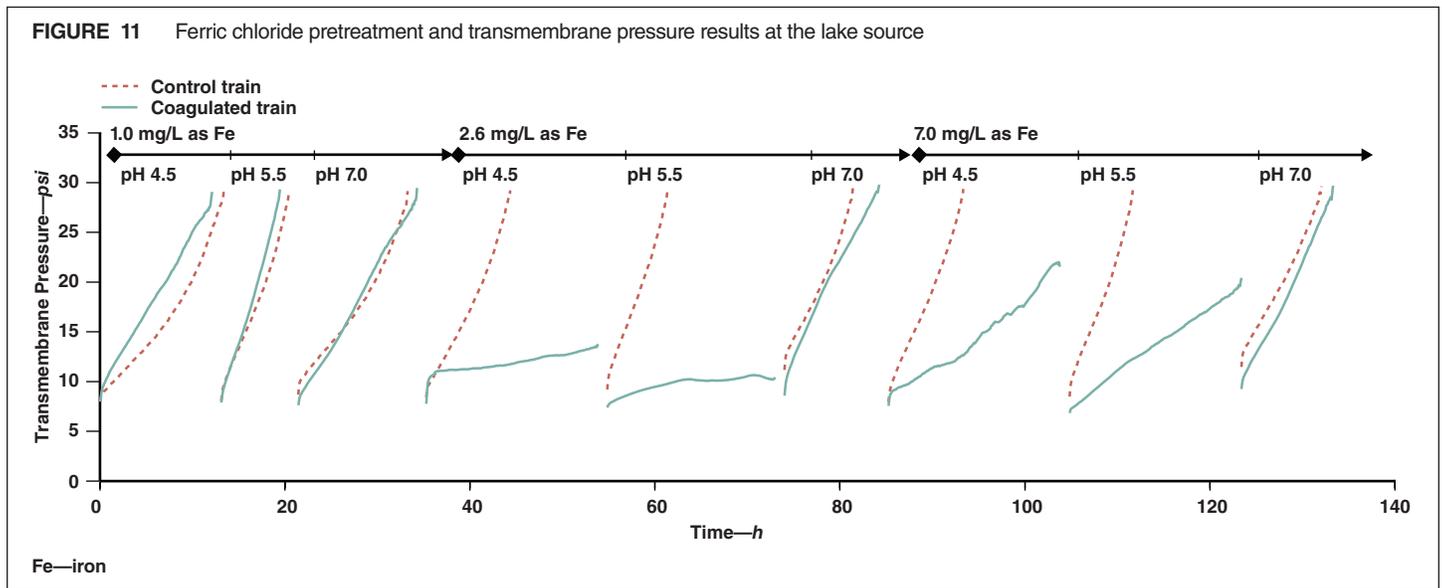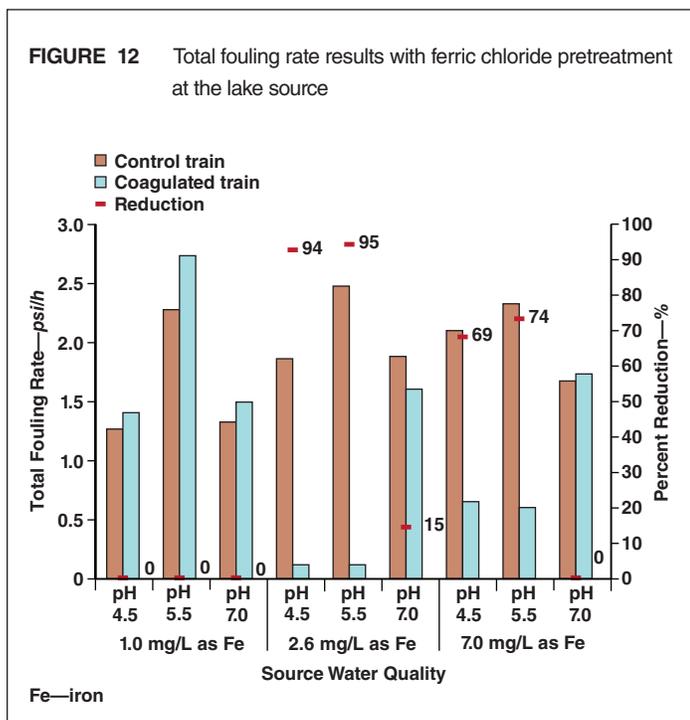
clearly show that aluminum sulfate performed more consistently across the nine coagulated train experiments with very little variation between them. Ferric chloride (Figure 12) was found to be effective only in the mid-high investigated dose range and low pH levels.

CONCLUSIONS

The overall objective of this study was to identify optimal coagulation pretreatment conditions for minimizing the rate of membrane fouling. Preliminary experiments at the river source revealed the importance of optimizing backwash and membrane

FIGURE 12   Total fouling rate results with ferric chloride pretreatment at the lake source

cleaning protocols. Coagulation pretreatment was ineffective when backwashing was done every hour, but substantial improvements were observed when the frequency was reduced to every half hour. An acid soak alone was found to be inconsistent and ineffective in recovering membrane TMP to a constant starting pressure; however, a combination of bleach followed by an acid soak was found to be successful in recovering the TMP between experiments. Aluminum sulfate optimization experiments using river water showed that with a dose of 1.3 mg/L as Al (0.048 mM Al; ~14 mg/L as aluminum sulfate) at pH 6.0, total membrane fouling rate was reduced by 83% compared with the control train. At this condition, the total fouling rate was 0.15 psi/h with coagulation pretreatment and 0.81 psi/h without coagulation pretreatment. Aluminum sulfate optimization was performed at the lake source to determine the flexibility and consistency of the coagulant in improving the performance of the membrane system. The results showed that an 80% reduction of membrane fouling rate was achieved by almost all conditions. Ferric chloride optimization results showed that it was not effective at either the highest pH (7.0) or the lowest dose (1.0 mg/L as Fe) conditions. On the other hand, 95% of the membrane fouling rate was reduced at a dose of 2.6 mg/L as Fe (0.047 mM Fe; ~7.6 mg/L as ferric chloride) at pH 5.5. The pressure stabilized with aluminum sulfate at almost all conditions investigated; however, with ferric chloride this was observed only at a dose of 2.6 mg/L as Fe at pH 4.5 and 5.5.

ENDNOTES

[1]Hyflux® InoCep®, Hyflux Ltd., Singapore
[2]Nalgene™ Heavy-Duty Tank, Thermo Scientific, Waltham, Mass.
[3]Masterflex Peristaltic Pump, Cole-Parmer®, Vernon Hills, Ill.
[4]Dynamax RP-1 Peristaltic Pump, Rainin Rainin Instrument Co., Emeryville, Cal.

ABOUT THE AUTHORS

Amir Alansari (to whom correspondence may be addressed) is a graduate research assistant in the Department of Civil and Environmental Engineering at the University of North Carolina at Charlotte, 9201 University City Blvd., Charlotte, NC 28223 USA; amiralansari89@ gmail.com. He obtained his MS and BS degrees in civil engineering from the University of North Carolina at Charlotte. Meric Selbes is an assistant engineer at Hazen and Sawyer, Fairfax, Va. Tanju Karanfil is the associate dean for research and graduate studies at Clemson University, Clemson, S.C. James Amburgey is an associate professor at the University of North Carolina at Charlotte.

PEER REVIEW

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