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Effect of Antiscalant Usage and Air Diffuser Perforation Diameter on Filtration Performance of Submerged Flat Sheet MBR for Treatment of High Salinity and Scaling Propensity Wastewater

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Abstract: Membrane fouling and mineral scaling remain major drawbacks for MBR technology. Membrane fouling reduces the filtration ability in MBR systems by increasing transmembrane pressure (TMP) and thus increases the operational cost. This study focused on the application of commercially available antiscalant in a pilot MBR system and the effect of diffuser perforation diameter for the treatment of high mineral scaling propensity wastewater. Submerged flat sheet membranes (Kubota, nominal pore size: 0.4 µm) were used in the pilot-scale test unit operated in the wastewater treatment plant of ITOB Organized Industrial Zone, Izmir, Turkey. The commercially available antiscalants employed were coded AS-1 and AS-2 for antiscalant study. Long term effect of the two antiscalants employed was investigated under high mixed liquor suspended solid (MLSS) concentration (17–21 g/L) for two months of MBR operation. The effect of low MLSS concentration (10–13 g/L) was also studied without changing the concentration of antiscalant type and concentration. AS-1 was found to be more effective in terms of mineral scale control. The effect of diffuser perforation diameter (1, 3 and 5 mm) on mineral scaling minimization in MBR pilot system was also studied. The best performance with respect to membrane fouling control was found with an air diffuser having 3 mm of diffuser perforated diameter. Some quality analyses of the product water were also carried out to assess the effect of antiscalant addition on microbial activities in the MBR unit. The findings in this study reveal that the use of antiscalants has not affected biological treatment performance of MBR pilot system. The removal ranges obtained during all MBR studies were 98.47–99.9%, 84.62–99.4%, 89.5–98.5%, 86.90–99.9%, 67.01–99.2%, 75.03–93.9%, and 20.36–71.5% for total suspended solid (TSS), color, chemical oxygen demand (COD), NH4-N, PO4-P, NO_2-N, and total nitrogen (TN) respectively.

Keywords: air diffuser; antiscalant; hardness; MBR; membrane fouling; salinity; industrial wastewater treatment

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1. Introduction

Conventional activated sludge (CAS) has been widely used for the treatment of both municipal and industrial wastewater, where wastewater treatment is achieved via biodegradation of organic contaminants in biologically active environment (mainly bacteria and protozoa), while total suspended solids are removed physically in a clarifier [1,2]. Membrane bioreactor (MBR) systems have been widely used due to their numerous advantages over CAS, such as high mixed liquor suspended solid (MLSS), higher solid retention time (SRT), and, hence, higher organic removal efficiency, low footprint, easy automation, lower sludge production, etc. [3–5]. More effective solid–liquid separation is attained via the use of membranes when MBRs are employed for wastewater treatment. Furthermore, ~2–3 and ~6–7 log respective bacteria and virus reduction are achieved when ultrafiltration (UF) membranes are used in MBR systems. Hence, less post treatment is needed, unlike for CAS systems [6–9].

MBR technology is well known for its high quality (in terms of suspended solid and organic contaminants) effluent water. However, membrane fouling remains the bottleneck for the application of MBR in wastewater treatment processes [10]. As a result, permeability decreases, resulting in difficult operational conditions and higher operation costs [11–14]. Membrane fouling can be also categorized as reversible, irreversible, residual, and irrecoverable fouling. Reversible fouling is also known as removable or temporary fouling, which is the loose attachment of foulants on the surface of the membranes, and they are generally removed via physical cleaning, relaxation, or backwash. However, irreversible fouling type of fouling occurs via gel layer formation, adsorption of foulants on the interior of the membrane, pore blocking, and narrowing [3]. Membrane fouling that can only be removed via frequent chemical cleaning will surely reduce the productivity of a treatment plant [15,16]. For example, in the presence of alginate, a severe membrane fouling can be observed when the feed water contains Ca^{2+} ions [17]. The presence of metal ions (Ca^{2+}, Mg^{2+}, Fe^{3+}, and Al^{3+}) and the anions (COO^{-}, $CO_3{}^{2-}$, $PO_4{}^{3-}$, $SO_4{}^{2-}$ and OH^{-}) can cause chemical precipitation and thus inorganic fouling. Some precipitations are formed when the concentrations of these ions exceed their saturation concentrations during water treatment [18]. Biopolymers in high mineral scaling propensity serve as initial nucleation sites for the formation of organic–inorganic complexes [19,20]. Inorganic-enriched biopolymers can enhance the binding of sludge cake layers to the membrane surface and greatly reduce membrane permeability due to charge neutralization and cation bridging [21].

In the last decade, researchers have focused on the possible ways to mitigate membrane fouling. According to Böhm et al. [22], more than 30% of the research papers published today focused on membrane fouling alleviation. A lot of energy has been consumed to minimize membrane fouling during the process, as mentioned by Drews et al. [23], and only 10% of the energy used in MBR is utilized for biodegradation. Furthermore, the traditional ways of membrane fouling mitigation, such as backwashing (in back washable membranes), permeate flux adjustment, and cross-flow velocity are considered not to be sufficient [24].

There are also published works mentioned in the literature for membrane fouling and scaling mitigation, including modification of the surface of the membranes [25]. In a study conducted by Iorhemen et al. [26], it was reported that some adsorbents can be used simultaneously in MBR unit to reduce the toxic effect on the microorganisms responsible for the biodegradation of organic matter in the wastewater. In another study by Aslam et al. [27], it was mentioned that fluidized media were used to alleviate membrane biofouling. Ferric chloride and aluminum sulfate (alum) are the coagulants that are widely used for coagulation in MBR. These coagulants can also be used simultaneously with the biological treatment and the membrane separation at the same time or can be used at pretreatment step prior to MBR unit. Chen et al. [28] employed alum directly into the MBR unit and reported that the concentration of carbohydrate fraction of soluble microbial product (SMPc) is lower than that of the MBR system where alum was not added. In another study by Lee et al. [29], it was reported that the average size of particles in the MBR system has been increased by the aggregation of small particles (from 0.1 to 2 µm) when alum was used into the bioreactor. Gkotsis et al. [30] also mentioned the possibilities of using reagents, electrical field, ultrasound, or ozone to reduce biofouling in MBRs. The emerging technology called quorum quenching was also discussed in detail in a review paper written by Aslam et al. [27]. It was mentioned that the microorganisms responsible for membrane fouling communicate through a medium called quorum sensing. With the help of this innovative technology, the signal can be disrupted, and, hence, membrane biofouling is mitigated. It is also important not to forget the major role played by fluid dynamics. Air scouring is considered as the main method to prevent accumulation of biomass on the surface of the membranes during MBR operations, as mentioned by Böhm et al. [22]. In order to mitigate scaling of Ba, Sr, and Ca scaling, Scholz et al. [31] used phosphate-based antiscalants at a concentration of 16 g/m^3.

Dynamic fluid behavior has a great impact on membrane fouling. Dynamic behavior of fluids during MBR operation is complex, since it consists of both mass and momentum balances, transport of species, multi-phase flow regime, and particulate back transport. Furthermore, dissolved (DO) concentration and membrane scouring in MBR are affected by bubble size and diameter, bioreactor temperature, air flow rate, and biomass concentration [32]. Amini et al. [33] studied the effects of bubble diameter and MLSS concentration on membrane fouling using a computational fluid dynamics (CFD) model [33]. The range of bubble diameter studied was 2–5 mm, and 3 mm of bubble diameter was found to be the optimum bubble size diameter irrespective of MLSS concentration. Drews et al. [23] also studied the effect of bubble size diameter (3–5 mm) on membrane fouling by varying depth channel (3–9 mm). It was found that, as the bubble size diameter increases, air velocity also increases in the riser zone, and less gas enters down comer zone, hence higher drag force is applied to the liquid. Distance between membranes also plays an important role because bubble size was characterized by the space between the membranes [34,35]. In their results, it was shown that, as the space between the membranes decreased (4 mm), the air bubble sizes were fragmented. When the membrane space was increased from 4 to 6 mm, more uniform and better air distribution and, hence, fouling control, were established.

While a major portion of fouling control studies is focused on biofouling and organic fouling, only a limited number of studies reported on inorganic fouling. One of the ways to tackle inorganic precipitation on the surface of the membranes during operation is by lowering the operational pH if $CaCO_3$ is the major soluble mineral scalant. Sanguanpak et al. [36] investigated the effect of pH in the treatment of landfill leachate wastewater. It was clearly shown that, as the pH of the bioreactor decreased, the precipitation of Ca^{2+} and Mg^{2+} ions also decreased as expected. However, as reported by Wu et al. [37], the optimum operation pH for biodegradation is in the range of 6.0–9.0 and further decrease in pH towards pH of 4.0, which will lead to a decline in microbial activity. Zhang et al. [38] also noted that an increase in the pH of the mixed liquor resulted in an increase in cake layer resistance while decreasing the pH below the optimum condition, which resulted in adhesion of flocs on the surface of the membrane. For that reason, decreasing operation pH will decrease scaling formation on the surface of the membranes, but it will also affect microbial responsible for biodegradation in the bioreactor. However, if scaling occurs in the presence of antiscalants (AS), the nucleation and growth of scale crystals can be impeded [39–44]. Optimal AS dosage should be verified experimentally for a given water source, given that AS effectiveness depends on selected type and dosage [39–41,45,46]

Jarma et al. [47] investigated the applicability of antiscalants for the treatment of high mineral scaling propensity wastewater, and their preliminary results showed that antiscalants can be used in MBR operation successfully without affecting the microorganisms responsible for the biodegradation of organic matter in the wastewater. Hence, this study was carried out to evaluate the performance of two commercially available ASs (coded AS1 and AS2) for the treatment of high mineral scaling propensity wastewater in a pilot-scale MBR system. The pilot-scale MBR system was installed at ITOB Wastewater Treatment Plant in Menderes-Izmir, Turkey. Hence, the study was carried out under filed conditions. Likewise, the effect of AS under range of mixed liquor suspended solid (MLSS) and diffuser perforation diameter was investigated. To interrogate the effect of bubble diameter on membrane fouling, three different air diffusers, having perforated hole diameters of 1, 3, and 5 mm, were employed for mineral scale minimization. The effect of AS addition in wastewater treatment performance of pilot-scale MBR was also assessed.

2. Materials and Methods

2.1. ITOB Wastewater Treatment Plant Feed Water Characteristics

The challenge of treating water source of this quality via membrane technology is high Ca^{2+} ion concentration with a Langmuir saturation index (LSI) of 1.04 [48,49]. The LSI was determined via CSMPRO ver6.2.0 (Toray Chemical Korea Inc, Seoul, Korea) using the feed water characteristics in our previous study [48,49]. The treatment plant is responsible for providing water for industrial activities, and the water becomes more concentrated in terms of calcite after been utilized by some industries. ITOB WWTP utilizes full scale MBR for the treatment of both municipal (40%) and industrial wastewater (60%) coming from the Organized Zone. Due to high scaling propensity of this water source, the treatment plant was experiencing severe levels of inorganic fouling (mostly calcite scaling) in the membranes, which necessitates frequent chemical cleaning of the membranes (Table 1). The influent wastewater also changes temporarily in terms of COD, salinity, pH, and concentration of sparingly soluble mineral ($CaCO_3$ in the present study), depending on the production in the Organized Industrial Zone.

Table 1. Physicochemical characteristics of MBR influent.

Parameter	Influent
EC (mS/cm)	2.25–3.58
pH	7.05–7.69
Calcium (mg/L)	140–160
Salinity (‰)	1.52–2.05
TSS (mg/L)	580–1341
NH_4-N	19.7–44.3
PO_4-P (mg/L)	2.1–18.2
TN	35.8–75.3
NO_3-N (mg/L)	0.5–11.0
NO_2-N (mg/L)	0.112–0.921
COD (mg/L)	262–1006
Color (Pt-Co)	1697–2304

EC: electrical conductivity, TN: total nitrogen, COD: chemical oxygen demand, Pt-Co: platinum–cobalt.

2.2. Pilot MBR System Description

A pilot-scale MBR system was installed at ITOB Organized Industrial Zone Wastewater Treatment Plant in Menderes-Izmir, Turkey. The MBR system has a reactor tank volume of 50 L and accommodates Kubota's submerged flat sheet PE-based membrane panels (4 panels), which are hydrophilized with a nominal pore size of 0.4 µm. The active membrane area of each membrane panel is 0.11 m^2. The wastewater coming to ITOB Organized Industrial Zone Wastewater Treatment Plant consists of both domestic and industrial origins (approximately 60% of industrial character and 40% of domestic character). Aeration for fouling control and oxygen demand for microorganisms' aerobic activities are achieved by the use of three Resun ACO-001 model magnetic air pumps (blowers) supplying air at the flow rate of 10–12 L/min to the bioreactor tank. To ensure complete aerobic condition in the MBR system, DO measurements were monitored continuously during the study, and the minimum DO level was set to >2.0 mg/L. Air flow to the bioreactor was controlled manually via air flow meter. Air flow to the pilot MBR was adjusted manually as well. To evaluate the effect of aeration, 3 air diffusers with perforated diameter of 1, 3, and 5 mm were employed (Figure 1). The flow diagram of pilot-scale MBR system was depicted in Figure 1. The properties of the pilot-scale MBR system were summarized in Table 2. The feed to the pilot MBR test unit was taken from the ITOB full-scale MBR treatment plant after passing through pretreatment in the WWTP. Although we employed a single aerobic tank (MBR tank as shown in Figure 1) providing a minimum DO level of 2.0 mg/L (5.59–8.56 mg/L in the present study) in bulk conditions (not in flocs), it is known from many studies and full-scale applications that denitrification may occur in the inner parts of the flocs in aerobic MBRs. Due to higher MLSS levels and different floc structures/morphology in MBRs compared to conventional activated sludge systems, oxygen demand for carbon removal is increased, which results in decreasing DO concentration profile from the outer to the inner parts of MBR flocs.

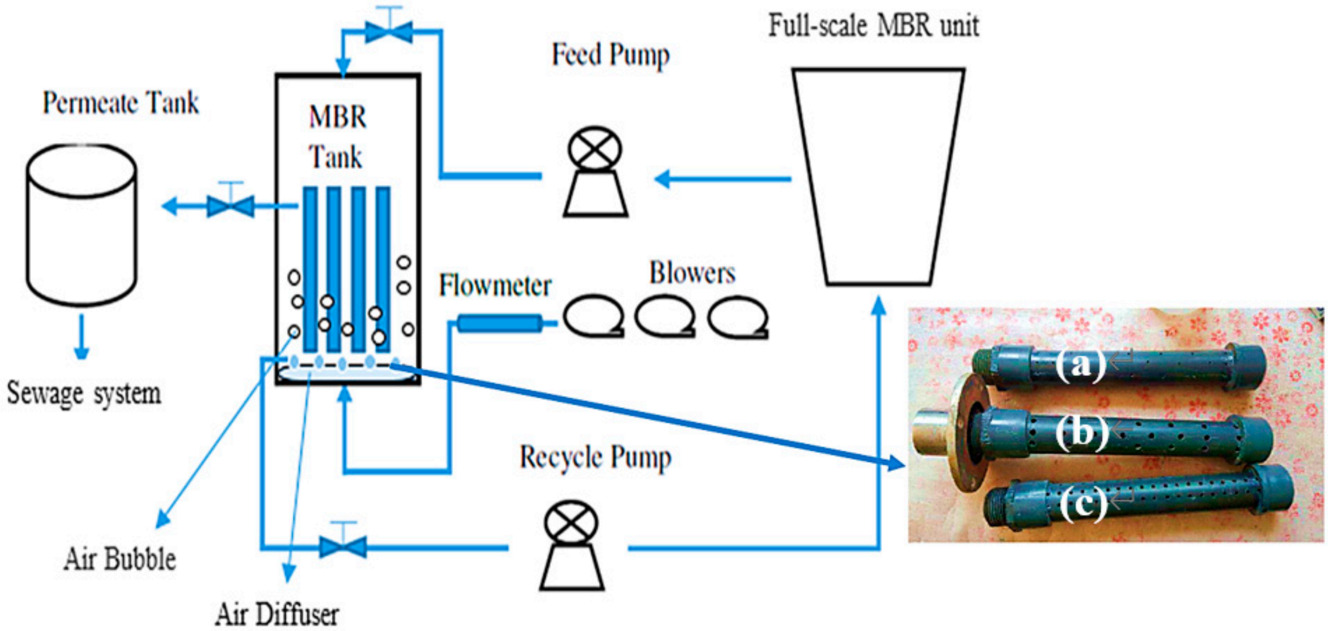

Figure 1. Flow diagram of pilot-scale MBR unit system, which utilizes main WWTP MBR as feed water source via feed pump, the pilot system used three Kubota plate type 0.4 µm membranes, aeration was achieved via three mini-blowers, excess sludge was taken from the reactor to WWTP MBR via recycle pump. Air diffusers with 1, 5, and 3 mm of perforated hole diameters ((**a**): 1 mm, (**b**): 5 mm and (**c**): 3 mm).

Table 2. Properties of the pilot MBR test unit.

Property	Value
Reactor capacity (L)	50
Membrane active area (m^2)	0.11
Nominal pore size of membrane panel (micron)	0.4
Membrane type	Plate and frame
Membrane material	Kubota-PE
Diffuser diameter (mm)	1, 3, 5
MLSS (g/L)	10–22
HRT (h)	3–4
SRT (day)	37–45

MLSS: mixed liquor suspended solid, HRT: hydraulic retention time (h), SRT: solid retention time.

2.3. MBR Tests

To maintain the MLSS level at a desired concentration and to fix the SRT value in the pilot-scale MBR system, the excess waste sludge was fed back to full-scale MBR unit of ITOB WWTP. The driving force for permeation of the treated water in the pilot MBR was the hydrodynamic pressure of the wastewater inside the reactor due to head difference between the membranes and the level of the wastewater inside the reactor. MBR effluent was collected in the 200-L of permeate tank. The pilot-scale MBR system was equipped with a Supervisory Control and Data Acquisition (SCADA) program, where the level of the wastewater in the reactor can be adjusted. Conductivity, pH, temperature, and DO level were monitored at any time from the control panel. The hydraulic retention time (HRT) and solid retention time (SRT) of the pilot-scale MBR system were kept the same throughout the study with that of full-scale MBR as 12 h and 35–40 days, respectively.

To evaluate the effect of AS addition on MBR treatment performance, raw wastewater samples were taken from the equalization unit of ITOB Wastewater Treatment Plant every week, while the permeate samples were taken from the MBR effluent stream at the exit of the pilot-scale MBR system.

During this study, the following parameters and conditions were investigated using pilot-scale MBR system:

1. **MLSS concentration in the bioreactor:**

- Low MLSS concentration (10–13 g/L)
- High MLSS concentration (17–21 g/L)

Firstly, the low MLSS concentration study was conducted, followed by a study with higher MLSS levels. Low MLSS levels were achieved by withdrawing excess sludge from the pilot scale MBR via recycle pump, while high MLSS levels were attained by not withdrawing sludge from the study at low MLSS concentration until 17–21 g/L of MLSS level was achieved.

2. **Type and dosage of antiscalant:**

The two candidate antiscalants employed in this study were commercially available antiscalants used in high mineral scaling propensity water treatment [50–53].

- A (40 mg antiscalant/L wastewater)
- B (40 mg antiscalant/L wastewater)

Successful application of the antiscalants used in this study for scaling prevention (especially $CaCO_3$) was experienced in our previous studies [38,40].

3. **Perforated hole diameter of air diffuser**

- 1 mm
- 3 mm
- 5 mm

The aim of the first test was to find the best MLSS level. Antiscalant concentration was doubled (40 mg/L solution) this time, while it was 20 mg/L in the study conducted by Jarma et al. [47]. HRT and SRT values were kept constant in all the studies. A summary of the experimental conditions is given in Table 3.

Table 3. Experimental test conditions employed in the pilot-scale MBR operation.

	Effect of MLSS Concentration		**Effect of Type of Antiscalant**	**Effect of Air Diffuser Hole Diameter**	
Operational Parameter	Study 1	Study 2	Study 3	Study 4	Study 5
MLSS (g/L)	10–13	17–21	9–13	15–18	18–22
AS type	AS-1	AS-1	AS-2	AS-1	AS-1
AS concentration (mg/L)			40		
AS Dosage (mL/min)			5		
Diffuser hole diameter (mm)		3		5	1
Operation time (month)	1	2	1	1	1

MLSS: Mixed liquor suspended solid.

2.4. MBR Pilot System Performance Evaluation

To evaluate the performance of the pilot scale MBR permeate flux was monitored over time, and permeate flux in this study was calculated using the equation below (Equation (1)).

$$J = \frac{Q_p}{A} \tag{1}$$

where Q_p is permeate flow rate (L/h), A is membrane active area (m^2).

The ambient temperature of the field changes with time. Hence, permeate flux must be corrected by temperature correcting factor. The permeate flux changes with temperature, since both water viscosity (higher water permeation through the membranes) and DO varies with temperature [54]. For that reason, the temperature-corrected permeate flux was calculated using Equation (2).

$$J(20\ ^\circ\mathrm{C}) = \frac{Q_p \times exp^{-0.0239\times(T-20)}}{A} \tag{2}$$

where J (20 °C) is the temperature corrected flux at 20 °C, Q_p (L/h) is the flow rate of the permeate measured at temperature T (°C), and A is the total active membrane area (m^2).

Another aspect of this study was to evaluate chemical cleaning of the membrane. To accomplish this, permeate flux of the membrane was measured prior to each experiment. The membranes were subjected to citric acid cleaning after terminating each experiment.

During the operation of pilot-scale MBR system, the hydraulic head from the wastewater feed was the driving force for water permeation, which was set at 0.6 m (~0.6 atm) above the membranes. Membranes were cleaned before starting every experiment. The cleaning protocol used in this study was adopted from Jarma et al. [47], where physical cleaning was applied first to remove removable foulants, then NaOCl (3000 mg/L) soaking for a period of 1 h, washing and rinsing with tap water, citric acid soaking (2000 mg/L) for a period of 1 h, and finally washing and rinsing the membranes with tap water. Chemically enhanced backwash cleaning protocol was adopted from the review conducted by Wang et al. [25]. Equation (3) was used to calculate cleaning efficiency of the membranes after chemical cleaning.

$$Flux\ Recovery = \frac{J}{J_o} \times 100 \tag{3}$$

where J and J_o are the fluxes of cleaned and virgin membranes, respectively.

2.5. Methods

Electrical conductivity (EC), total dissolved solids (TDS), salinity, pH, temperature, and dissolved oxygen (DO) of the samples were measured by portable EC meter (Hach Lange HQ14D), pH meter (HQ11D Hach Lange), and DO probe (Hach LDO, Hach Lange) respectively. TSS, MLSS, and color analyses were made by the help of Hach Lange DR3900 spectrophotometer. Some other quality parameters, such as total nitrogen (TN), chemical oxygen demand (COD), PO_4–P, NH_4-N, NO_2-N, and NO_3-N were also performed with Hach Lange test kits. The Hach Lange LT200 thermo-reactor was used for heating purposes during COD and TN analysis. The results of the quality analyses for the MBR influent used in this study are presented in Table 1.

3. Results

Antiscalant application in mineral scale mitigation is not new for sure, but the mineral scale in membrane desalination is traditionally assessed by monitoring either transmembrane pressure or permeate flux decline, followed by freshwater flushing and, sometimes, with chemical cleaning when the scaling is severe. The effectiveness of two commercially available antiscalants (AS-1 and AS-2), as well as the effect of MLSS concentration and diffuser perforated diameter, were assessed in this study. The optimum antiscalant dose for mineral scale is generally determined experimentally. However, the recommended dose by various antiscalants is generally in the range of 2–10 mg/L, depending on the feedwater characteristics [55]. Nonetheless, the study by Trukhina et al. [56] shows that the presence of the suspended particles (size range of 4–40 nm, source water gypsum saturation indext (SI_{gypsum}) of 3.9) inhibits the mode of action of the antiscalant employed during desalting process. Due to the fact that some of the antiscalants meant to inhibit nucleation of sparingly mineral scalants ($CaCO_3$ in the present study) in the feedwater will be adsorb by the flocs in the MBR unit. Hence, antiscalant concentration in the present study (20–40 mg/L) was employed more than the recommended dose in the literature (2–10 mg/L) [56,57]. Though, most of the studies conducted in the literature were employed to suppress mineral nucleation in either brackish or seawater desalting process.

3.1. Effect of MLSS Concentration

3.1.1. Study 1, Using Low MLSS Levels (10–13 g/L)

To evaluate the effect of AS-1 performance at low MLSS concentration, Study 1 was initiated after the membranes were cleaned out of place (COP). The test with low MLSS concentration (10–13 g/L) was initiated using AS-1 at a concentration of 40 mg/L. The air diffuser perforated hole diameter used in this study was 3 mm. Temperature corrected permeate flux with wastewater at the beginning of the test was 40.9 $Lm^{-2}h^{-1}$, but it suddenly decreased to about 15 $Lm^{-2}h^{-1}$ due to rapid accumulation of biomass on the surface of the membrane at the second day of the operation. After the 15th day of operation, permeate flux started to decrease below 10 $Lm^{-2}h^{-1}$, and cleaning was performed using NaOCl solution (3000 mg/L) at the 24th day. As explained by Yiğit et al. [58], the particle size distributions were larger at lower MLSS concentration compared to the study at higher MLSS concentration. This was the reason why the system operated for a longer time without chemical cleaning. Chemical enhanced backwash (CEB) was also applied using NaOCl solution for 1 h in order to restore permeate flux, and 59.0% of the initial membrane flux was recovered after CEB (Figure 2).

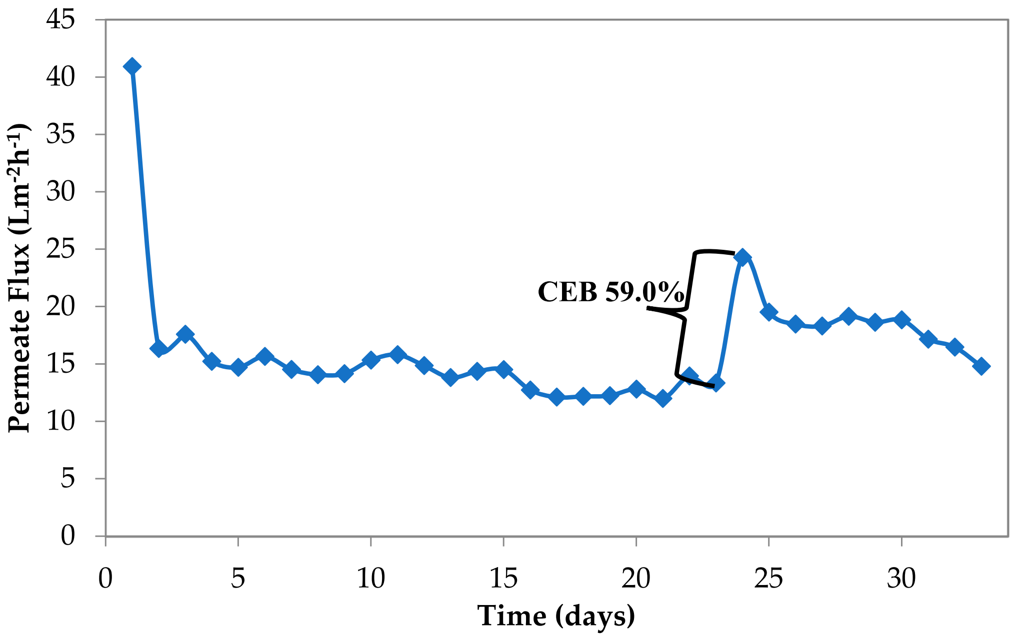

Figure 2. Temperature corrected flux versus time plot obtained at high AS concentration (40 mg/L AS-1 and low MLSS of 10–13 g/L).

Some quality analysis of the permeate obtained from the pilot MBR was carried out to assess the effect of AS dosage on biodegradation of the wastewater. The water quality analysis showed similar trend with the previous study [47]. TSS and color were highly removed, with a removal efficiency of more than 99.9% for TSS and 99.4% for color. The removal efficiency for COD was 98.5%, while PO_4-P was also highly removed with a removal efficiency of 99.1%, and TN removal efficiency was 59.1%. The change in denitrification efficiency can be attributed to the change in the MLSS concentration and, hence, decrease in the number of the denitrifying bacteria. The performance of the pilot-scale MBR system at low MLSS concentration is presented in Table 4.

Table 4. Summary of the quality analyses of completed tests with pilot MBR system.

	Effect of MLSS Concentration		**Effect of Antiscalant Type**	**Effect of PHD of Air Diffuser**	
Operation parameters	Study 1	Study 2	Study 3	Study 4	Study 5
Feed MLSS (g/L)	17–21	10–13	9–13	15–18	18–22
EC (mS/cm)	3.62–4.59	3.65–3.95	3.38–3.99	3.18–3.94	3.92–3.98
Salinity (‰)	1.91–2.68	1.94–2.09	1.77–2.10	1.98–2.09	2.08–2.12
TDS (mg/L)	1870–2620	1890–2050	1740–2059	1938–2043	2034–2074
pH	7.46–8.29	6.73–7.07	7.12–7.44	7.64–8.02	7.93–8.21
DO (mg/L)	5.59–6.94	7.95–8.56	6.75–7.41	7.45–8.14	7.32–7.88
Color (Pt-Co)	26–53	17–44	44–92	105–122	105–123
TSS (mg/L)	1–8	0	0	5–13	7–14
AS type	1	1	2	1	1
PHD (mm)	3	3	3	5	1
Time (month)	2	1	1	1	1

EC: electrical conductivity, TDS: total dissolved solid, Pt-Co: platinum–cobalt, DO: dissolved oxygen, TSS: total suspended solid, MLSS: mixed liquor suspended solid, PHD: diffuser perforated hole diameter.

3.1.2. Study 2 with High MLSS Concentration (17–21 g/L)

To assess the effect of antiscalant performance at high MLSS concentration, the pilot MBR was run at MLSS concentration of 17–21 g/L. Chemical cleanings of MBR membranes prior and after each study were performed outside of the system (COP). After physical cleaning of membranes (sludge removal from membrane surfaces by the help of tap water), membranes were soaked into a 3000 ppm of NaOCl solution for 1 h. Afterwards, membranes were flushed with tap water, followed by subsequent soaking in 2000 mg/L citric acid solution for 1 h. Finally, clean membranes were flushed with tap water again and assembled in the system, and Study 2 was initiated.

After membranes were cleaned, the permeate flux with wastewater was measured as 37.23 $Lm^{-2}h^{-1}$. The diffuser perforated diameter (3 mm) used in this study was maintained constant, as in the case of low MLSS concentration (Section 3.1.1). The AS dose was maintained as constant, as in the case of Section 3.1.1. AS-1 was employed at a concentration of 40 mg/L. There was a high flux drop in the first six days due to rapid accumulation of biomass on the surface of the membranes, and this was similar to the findings by Jarma et al. [47] and Skouteris [59]. Temperature-corrected permeate flux was partially stable around 10 $Lm^{-2}h^{-1}$ between the sixth and thirteenth days of the study, as shown in Figure 3. However, the permeate flux decreased below 10 $Lm^{-2}h^{-1}$ on the fourteenth day of the test.

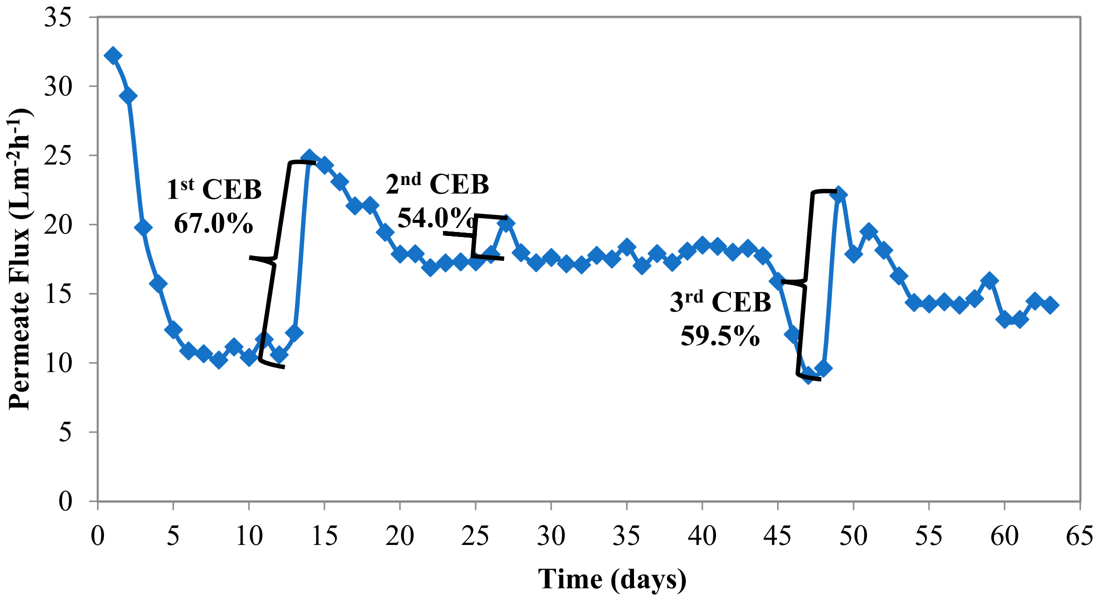

Figure 3. Temperature corrected flux versus time plot obtained at high antiscalant concentration (40 mg/L), AS-1 and high MLSS (17–21 g/L). MLSS: mixed liquor suspended solid.

In the study by Jarma et al. [47], the concentration of antiscalant was half of the one used in this study (MLSS concentration in the previous study was 12–19 g/L). It took only three days before the permeate flux dropped below 10 $Lm^{-2}h^{-1}$ in the present study. However, after the first CEB cleaning, the system operated longer than the study conducted by Jarma et al [38]. This can be attributed to the increase in antiscalant concentration by 50%. The sudden drop in permeate flux can be attributed due to the size of the flocs in the reactor. This was explained by Yiğit et al [58], where the size of the flocs in the reactor decreased as the MLSS increased. In their study, it was found that the average particle size of the flocs in the MBR was larger than 120 µm when the MLSS was 4600 mg/L. However, the average particle size distribution decreased when the MLSS concentration increased from 6600 mg/L to 12,600 mg/L [54]. For that reason, membranes were fouled due to pore blocking by the fine particles.

MBR membranes were cleaned by CEB for 1 h. Temperature corrected permeate flux during wastewater treatment was 24.82 $Lm^{-2}h^{-1}$ (after cleaning), which equals 67% of the initial flux. The system operated until the second CEB on the 27th day of operation was conducted. Flux recovery of second CEB (by maintaining CEB cleaning protocol explained in this section) was 54% this time. It was considered that decrease in permeate flux recovery was caused by inorganic salts accumulation on membranes with time.

The third CEB was performed on the 49th day of operation, and membrane flux recovery was found as 59.5%, which was greater than the membrane flux recovery of the second CEB. The reason behind this was probably due physical damage from physical cleaning of MBR membranes, though the cleaning was carried out using a soft brush to clean the removable foulants form the surface of the fouled membranes. Hence, one must be careful during physical cleaning of the fouled membranes. This test was continued for two months, and it was concluded that the system could be operated for a long period of time with chemical cleanings without scaling the membranes.

To assess the effect of antiscalant addition on microbial activity in the in MBR, some quality analysis of the produced permeate were performed. Water quality analysis showed that 99.7% of TSS removal was achieved during this study (test with high MLSS concentration). Color removal was also high, with an average rejection of 98.9%. The removal of color from wastewater approached 100%. In terms of COD removal, the system showed a good performance, with an average COD removal of 98.4%. This result was in accordance with the data reported in the literature [60–64]. However, Di Trapani et al. [64]

reported a negative effect of high COD value in wastewater for MBR process because of high fouling propensity of the wastewater in their study. The removal of PO_4-P was 99.2%, which is slightly higher than the study reported by Jarma et al. [47]. The reason for high PO_4-P removal was attributed to its precipitation due to high concentration of Ca^{2+} ions in wastewater or its removal with adsorption by the biomass in the reactor [37,65]. An amount of 71.5% of TN removal was obtained during this study, and the findings in this study demonstrated that increasing the concentration of antiscalant in the MBR did not affect the denitrification in the MBR system. Details of quality analysis of the pilot-scale MBR system are summarized in Table 4.

3.2. Type and Dosage of Antiscalant

Study 3-Effect of Antiscalant Usage

High concentrations of multivalent ions in wastewater result in serious scaling problem due to precipitation of insoluble salts, such as $CaCO_3$ on MBR membranes. Failure to optimize either operational condition, such as MLSS concentration (Section 3.1), diffuser perforated hole diameter (Section 3.3), and antiscalant concentration dose and type will necessitate frequent membrane cleaning (intermittent operation). In this study, use of antiscalants during MBR operation was investigated to minimize membrane fouling by increasing the saturation index of the feedwater during MBR permeate production.

Water chemistry and operational parameters play a vital role in the selection of an appropriate antiscalant [66,67]. Hence, different commercially available antiscalants (AS-1 and AS-2) were employed. The concentration of the antiscalant was maintained constant as 40 mg/L of wastewater within the scope of the study. The diffuser-perforated hole diameter of the air diffuser used in this study was kept constant at 3 mm. The MBR system was initiated with a temperature corrected permeate flux of 37.2 $Lm^{-2}h^{-1}$ in the presence of wastewater (Figure 4) right after cleaning COP of the membranes. After the 9th day of this study, permeate flux decreased below 10 $Lm^{-2}h^{-1}$, and CEB was applied on the 14th day of the study, and it took 25 days before the membranes were cleaned when AS-1 was employed. This clearly shows that, AS-1 inhibits $CaCO_3$ scaling during MBR wastewater treatment compared to AS-2. The antiscalant prevents mineral scaling of sparingly soluble ions via dispersion, crystal lattice distortion, and chelation effect. Hence, the backbone or major ingredient content of an antiscalant plays a vital role in mineral scale inhibition. The major ingredients of AS-1 were organic polyacrylates and phosphonates [68], while the main ingredients of AS-2 were reported as organophosphonate and polyacrylates [69–71], and the mode of action of AS-1 is via dispersing of the sparingly mineral soluble ions [68], while the mode of action of AS-2 is via threshold effect [52]. However, AS-2 technical details were not provided by the manufacturer, and the mode of action of AS-2 seems to be similar to that of AS-1 based on the flux declined found in this study (Figures 3 and 4). According to classical nucleation theory, heterogeneous nucleation is more thermodynamically favorable than homogeneous nucleation due to a reduced energy barrier [72]. Hence, the presence of suspended particles in the MBR has accelerated heterogeneous mineral nucleation of $CaCO_3$ in the MBR unit, and suspended particles act as epicenter of mineral nucleation [73–76]. It has been proposed that polymorphic phases that develop on various active sites of the foreign particulate matter do so via physisorption [77,78]. The presence of suspended particles in a high mineral scaling propensity feedwater also affects the compactness and physical properties of the scale formed. The temperature-corrected permeate flux was 24.9 $Lm^{-2}h^{-1}$ in wastewater after CEB cleaning. However, the percentage of the permeate flux recovered using AS-2 was a little bit higher (67.0%) than that of AS-1 (59.0%) right after chemical cleaning. This clearly shows that most of the scaling on the surface of the membranes was cleaned when physical, NaOCL and citric acid cleaning was introduced.

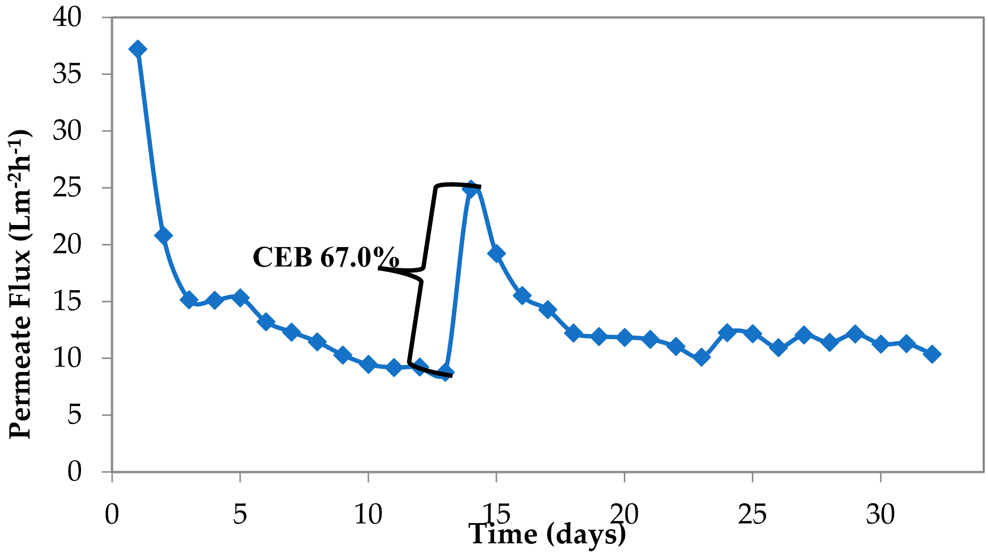

Figure 4. Temperature corrected flux versus time plot obtained using AS-2 at a concentration of 40 mg/L and a MLSS of 9–13 g/L.

It is important to keep in mind that, depending on the active ingredient of the antisclant, overdose of antiscalant above the optimum dose can lead to the absorption of residual antiscalant to membrane surface and increase the risk of biofouling. For example, phosphates in phosphate-based antiscalant can easily be hydrolyzed into orthophosphate ions and subsequently result in the formation of calcium phosphate and elevate the risk of biofouling on the surface of the membranes [79]. Consequently, phosphonate-based antiscalants are more often used to avoid the inactivity of phosphate-based antiscalants due to their hydrolysis [79]. The above reasons suggest that choosing of an appropriate antiscalant at an optimum dose is critical for the treatment of high mineral scaling propensity source water.

The performance of pilot-scale MBR system was not influenced by the type of antiscalant employed, as can be seen in Table 4. According to permeate water quality analyses of Study 3 (using AS-2), TSS and color were highly rejected in a similar trend with the previous study (Section 3.1.2). The removal efficiencies of TSS and color were 99.9 and 98.3%, respectively. COD was also highly removed with a removal efficiency of 97.6, while 99.2% of PO_4-P was also removed from the wastewater. However, TN removal was lower compared to the previous study (Section 3.1.2), with a removal efficiency of 47.8%. It was considered that AS-2 might have inhibited the denitrifying bacteria, which resulted in a partial denitrification.

3.3. Effect of Air Diffuser Perforated Hole Diameter

One of the solutions to mitigate membrane fouling in MBR process is the optimization of aeration intensity, as stated Krzeminski et al. [49]. Air was supplied to the MBR tank for two purposes: (i) the supply required oxygen for the microorganisms in the MBR unit for microbial activities and (ii) for membrane fouling minimization. Considering that, the air flow regime in MBR tank can be changed by manipulating the average hole size diameters of the air diffuser. In the study by Jarma et al. [38], an air diffuser with hole diameter of 3 mm was tested in pilot scale for fouling mitigation. In this case, different air diffusers with perforated hole diameter of 5 and 1 mm were employed.

3.3.1. Study 4 Using an Air Diffuser with 5 mm of Perforated Hole Diameter

In Study 4, an air diffuser with hole diameter of 5 mm was used, while AS-1 was employed at a concentration of 40 mg/L wastewater. The MLSS concentration during the study was maintained at 15–18 g/L. The temperature corrected flux of 32.4 $Lm^{-2}h^{-1}$ was recorded right after initiating the experiment (after COP cleaning). Chemical cleaning in this study was employed when the temperature corrected flux of the system approached 10 $Lm^{-2}h^{-1}$.

When an air diffuser with perforated hole diameter of 5 mm was employed, it took only three days for temperature corrected flux of the system to drop below 10 $Lm^{-2}h^{-1}$ (Figure 5). The temperature-corrected flux further dropped below 4 $Lm^{-2}h^{-1}$ on the 14th day of operation, despite CEB employment. An amount of 35.0% of the initial temperature corrected flux was recovered when CEB was applied. It was mentioned in the literature elsewhere [50] that the coarser the diameter of the air diffuser used in MBR, the higher the cleaning efficiency that was obtained. It was also reported by Culfaz et al. [51] that large air diffuser diameter showed a better membrane cleaning performance in HF membranes. Yamanoi and Kageyama [52] reported that better fouling mitigation was observed in their study when an air diffuser with hole diameter of 11–21 mm was utilized compared with a diffuser having a perforated hole diameter of 3.7–10 mm. However, this contradicts the result found in this study, where better performance was obtained with an air diffuser of perforated hole diameter of 3 mm instead of an air diffuser with perforated hole diameter of 5 mm. It was evident based on the findings in this study that a diffuser with optimum smaller diameter will generate more air bubbles compared to a diffuser with higher diameter. The higher the number of air bubbles generated in the MBR tank, the more mass transfer area is available to prevent particles from clogging the active surface of the membranes. This might be due to unequal numbers of perforated holes on the air diffusers. There were more perforated holes on the diffuser with hole diameter of 3 mm compared to that of 5 mm, as can be seen in Figure 1. The findings in the present study agree with the findings in the study conducted by Sofia et al. [80]. Based on the results found in their study, it was found that an air diffuser with 0.5 mm of perforated hole diameter was more effective in membrane scouring than an air diffuser with perforated hole diameter of 2 mm for a flat sheet membrane [80]. Larger air bubbles are expected to be produced when a diffuser with a 5 mm perforated hole is employed compared to 2 mm perforated hole air diffuser. In the present study, larger air bubbles produced with 5 mm perforated hole diffuser were more effective for distancing flocs away from the membranes. Diffuser hole diameter plays a vital role in fouling minimization during wastewater treatment by MBR technology. Hence, it is of paramount importance to take diffuser hole diameter coupled with location, geometry, and bubble distribution, not only the size of the bubbles, when evaluating the membrane fouling strategies of fouling minimization.

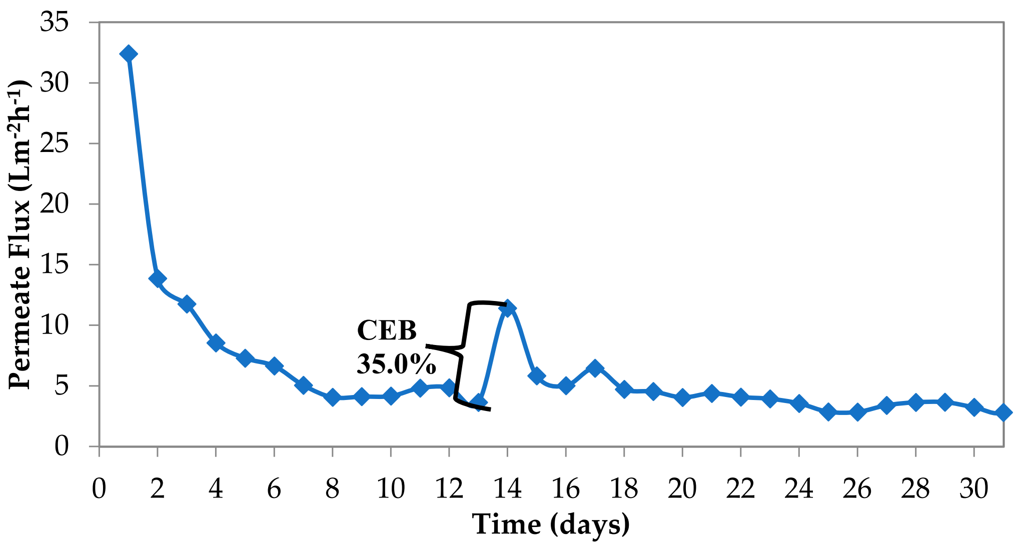

Figure 5. Temperature corrected flux versus time plot of a diffuser with 5 mm of hole diameter (40 mg/L AS-1 and 15–18 g/L of MLSS).

During our test with an air diffuser having perforated hole diameter of 5 mm, MLSS in the bioreactor was changing between 15–18 g/L. Looking at the quality analysis of the MBR effluent, TSS and color were removed with a removal efficiency of 98.5 and 90.5%, respectively. It can be clearly seen that the perforated hole diameter of air diffuser has affected the quality of the treated water negatively due to inefficient aeration. The removals of COD and PO_4-P were also less than the removals obtained with the air diffuser, having a 3 mm of perforated hole diameter. The removal efficiencies were 95.3 and 67% for COD and PO_4-P, respectively. On the other hand, TN removal obtained using the air diffuser with perforated hole diameter of 5 mm was far less (20.4%) than the removal obtained with the air diffuser having perforated hole diameter of 3 mm, even though the MLSS concentrations were similar in both cases.

3.3.2. Effect of an Air Diffuser with 1 mm of Perforated Hole Diameter (Study 5)

An air diffuser of hole diameter of 1 mm was used to investigate the effect of aeration regime on membrane fouling minimization in MBR wastewater treatment. During this study, AS-1 was employed at a concentration 40 mg/L wastewater. The membranes were cleaned by COP prior to the start of the experiment. Temperature-corrected flux of 34.7 $Lm^{-2}h^{-1}$ was observed at the starting hour of the experiment. However, it took only five days for the temperature-corrected flux to fall below 10 $Lm^{-2}h^{-1}$, as shown in Figure 6. CEB was applied on the 18th day of the study due to the decrease in temperature-corrected flux below 4 $Lm^{-2}h^{-1}$ during wastewater treatment. An amount of 47.0% of flux recovery was attained via cleaning COP and CEB. The pilot MBR was shut down early on the 24th day of the test due to MBR tant level controller failure. The feed pump continuously worked independently from the water level in the MBR tank, resulting in an increase in the feed MLSS with a concentration of 18 and 22 g/L. Based on the results obtained, it was clearly seen that permeate flux dropped within a short period of time compared to the study using an air diffuser with hole diameter of 3 mm. However, membrane fouling behavior was similar to the study using an air diffuser with hole diameter of 5 mm, even though the air diffuser hole distribution was not the same at each case, because the air diffuser with hole diameter of 1 mm has more perforated hole and hence more air bubbles for fouling control compared to that of 5 mm diffuser.

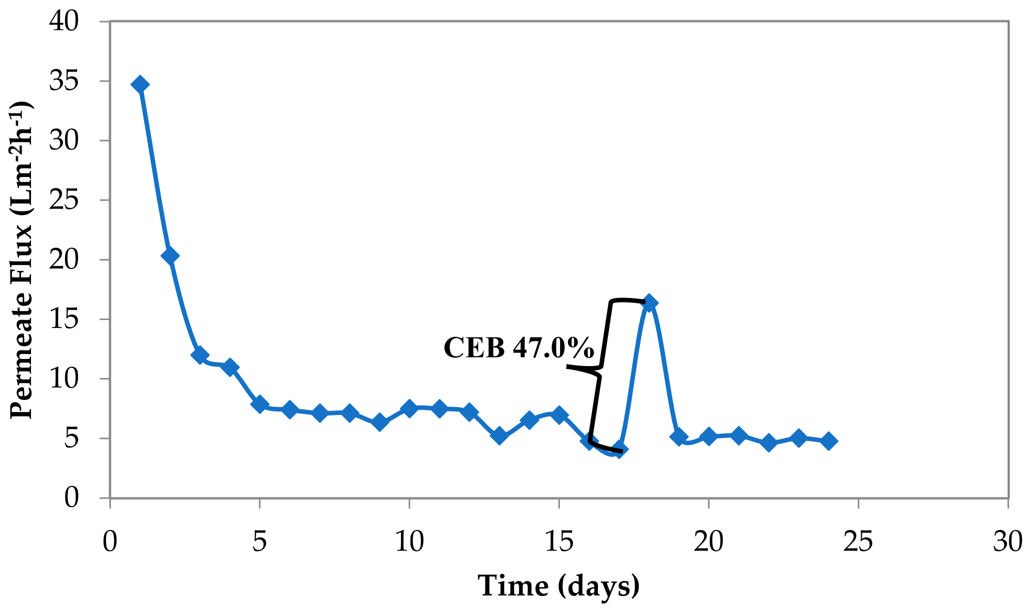

Figure 6. Temperature corrected flux versus time plot when an air diffuser with 1 mm of perforated hole diameter was used.

Quality analysis of MBR effluent was evaluated to evaluate in the influence of both antiscalant dosing and air aeration via 1 mm hole diameter aerator. The results obtained during 24 days of the test revealed 99.5% of TSS removal, 84.6% of color removal, 96.6% of COD removal, 72% of PO_4-P, and 71.5% of TN removals. The treatment performance of the pilot-scale MBR system with an air diffuser having hole diameter of 1 mm showed a better performance compared to the study with an air diffuser having 5 mm of perforated hole diameter due to the different distributions of perforated hole in both air diffusers (Table 4). It is hypothesized that the diffuser with a hole diameter of 1 mm helped in providing perfect conditions for microbial activities in the MBR tank.

Figure 7 shows the comparison of flux values with time of the studies conducted with air diffusers having different hole diameters. All three studies (study-1, 2 and 3) were conducted using AS-1 and air diffusers having hole diameters of 1, 3 and 5 mm. The respective MLSS concentration ranges in these studies were in the range of 15–18, 17–21, and 18–21 g/L.

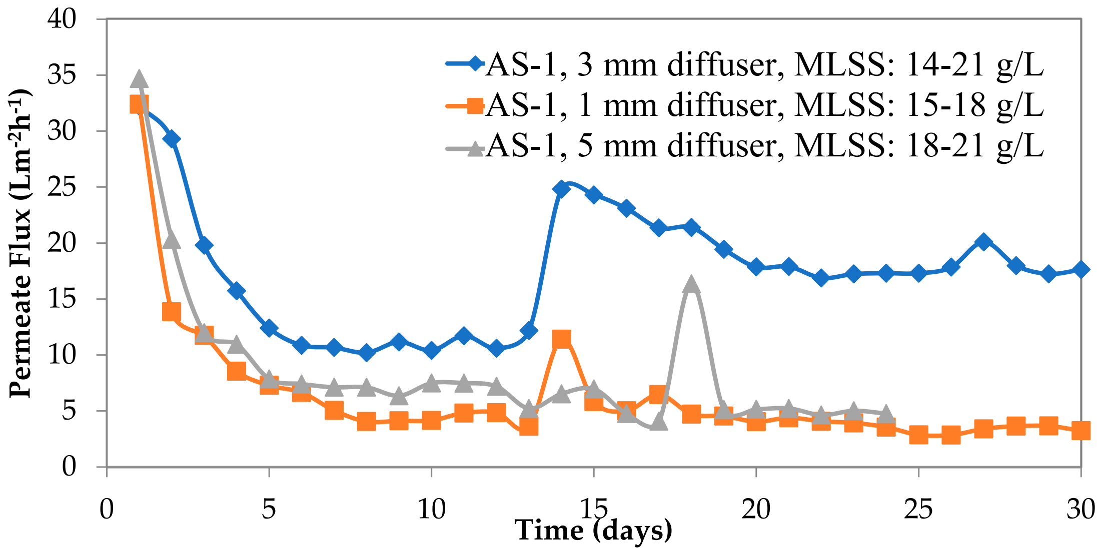

Figure 7. Comparison of the tests with different diffuser hole diameters (AS-1, MLSS: 15–21 g/L).

Tests run with an air diffuser having a hole diameter of 5 mm and 1 mm were similar in terms of flux decline with time. The fluxes of these tests were lower than the test with an air diffuser having hole diameter of 3 mm, as shown in Figure 7. Jarma et al. [38] also used an air diffuser with 3 mm of hole diameter, and their results showed a better performance in terms of sustainable flux during MBR wastewater treatment. However, their MLSS concentration was in the range of 12–19 g/L, while the MLSS in this study using an air diffuser with 3 mm of hole diameter was 17–21 g/L. That might be the reason for having better fouling control compared to the one in this study.

In Tables 4 and 5, water quality data of MBR effluents and rejections were summarized according to various process parameters employed. The EC values of MBR effluents were variable in the range of 3.18 and 4.59 mS/cm during the pilot-scale MBR studies, while TDS was in the range of 1740 and 2620 mg/L. The salinity range was as high as 1.77–2.68‰ pH values measured were generally around 6.73–8.29. The variability of MBR feedwater was dependent on wastewater produced by industries in the Organized Zone.

High DO (from 5.59 mg/L to 8.56 mg/L) showed the success of the aeration system, which is very important for biological degradation of organic waste present in the wastewater. High COD removal (>95%) was observed in all tests performed to reduce membrane fouling. MBR systems were reported to be successful for high COD and TSS rejection [55]. Especially, in Study-5 (summary of the experimental conditions are given in Table 3), permeate TSS was found to be increased up to 14 mg/L, which could be due to the variability of the raw wastewater. In parallel to this, there was an increase in the color up to

123 mg/L Pt-Co (Table 4). As seen in Table 5, when the biological treatment performance of our pilot-scale MBR system was compared with the study reported by Melin et al. [56], it can be said that there was no significant negative effect of using antiscalant when treating wastewater of high scaling propensity via MBR technology.

Table 5. Summary of the rejection values of completed tests with pilot MBR system.

	Studies	TSS	Color	COD	NH_4-N	PO_4-P	NO_2-N	TN
Effect of MLSS Concentration	Study 1	99.7	98.9	98.4	99.9	99.2	93.9	71.5
	Study 2	>99.9	99.4	98.5	99.8	99.1	86.0	59.1
Effect of AS Type	Study 3	>99.9	98.3	97.6	99.5	99.2	88.9	47.8
Effect of Diffuser Pore Diameter	Study 4	98.47	90.25	95.34	86.90	67.01	75.03	20.36
	Study 5	99.14	84.62	96.63	99.94	72.02	89.17	71.51
Literature (Melin et al. [56])		>99	NG	89–98	80–90	62–97	NG	36–80

NG: not given, MLSS: mixed liquor suspended solid, COD: chemical oxygen demand, TSS: total suspended solid, TN: total nitrogen.

4. Conclusions

Effects of MLSS concentrations, antiscalant type, and dose and diffuser perforated diameter were investigated for the treatment of high mineral scaling propensity water in a MBR pilot system. According to the results obtained in this study, the biological treatment performance of the MBR system was not adversely affected in all tests performed using antiscalants to reduce nucleation of sparingly mineral ions. Another approach to reduce membrane fouling was using an air diffuser in the MBR tank. In this study, three different air diffusers with perforated hole diameters of 1, 3, and 5 mm were used to investigate the effect of aeration intensity on membrane fouling mitigation. The results found in this study showed that using an air diffuser with 3 mm of perforated hole diameter was more effective on fouling mitigation in terms of permeate decline, while permeate flux with time changes in the studies performed using air diffusers with 1 and 5 mm of perforated hole diameters were similar. After the 13th day of operation, the flux values were 3.6, 5.2, and 12.2 $Lm^{-2}h^{-1}$ when air diffusers of 1, 5, and 3 mm of perforated hole diameters were employed, respectively. It was therefore concluded that selection and optimization of antiscalant and diffuser perforation diameter are crucial to prevent both biofouling and mineral scaling for the treatment of high mineral scaling propensity wastewater.

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