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Effect of alkaline and ozone pretreatment on sludge reduction potential of a membrane bioreactor treating high-strength domestic wastewater

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ABSTRACT

The wide application and utilization of the activated sludge process has resulted in the production of excess sludge, posing a serious disposal problem. Many efforts have been dedicated to reduce the excess sludge by treatments such as digestion and dewatering. In this study, an aerobic submerged membrane bioreactor (MBR) was used to study the effect of alkaline and ozone pretreatment on the efficiency of sludge reduction. For this purpose, two MBRs were fabricated. Among the two MBRs, one acted as a control reactor (CMBR) and the other acted as an experimental reactor (EMBR). The MBRs were operated with mixed liquor suspended solids (MLSS) concentrations in the range of 7,000–7,200 mg/L for a period of 120 d. In the EMBR, part of the MLSS was withdrawn at a ratio of 1.5% of *Q* and was pretreated by alkali-ozone. The sludge pretreatment was carried out at pH 11 and an ozone dosage of 0.09 gO_3/g MLSS. During the pretreatment, 40% COD solubilization and 30% suspended solid reduction were observed. The pretreated sludge was returned to the reactor for further degradation, where it was found to be 37% degraded. During the 120 d of reactor operation, both of the MBRs maintained a relatively constant transmembrane pressure. The sludge digestion does not have any impact on the COD removal efficiency of the reactor.

Keywords: Membrane bioreactor; Pretreatment; Sludge reduction; Domestic wastewater; COD removal

1. Introduction

Membrane bioreactors (MBRs) represent a newly developed wastewater treatment process in which solid–liquid separation happens at aerobic basin itself. As the activated sludge is filtered by the physical barrier of a membrane, effluent does not contain suspended solids and the mixed liquor suspended solids (MLSS) level can be maintained very high (5,000–30,000 mg/L). Consequently, it is possible to operate the reactor with less aeration volume and high sludge retention time (SRT). The high SRT of MBR facilitate effective nitrification by keeping slow

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growing nitrifier in the reactor. In addition, MBR does not rely on sludge settling characteristics, which is a common factor responsible for failure of activated sludge treatment plant. The main advantages of MBR compared to conventional treatment system were as follows (1) smaller area requirement, (2) complete removal of suspended solids and improves tertiary treatment efficiency, and (3) high removal ratios for most contaminants due to increased SRT, reduced sludge yield, and rapid start-up [1]. Most of these features result from the possibility of uncoupling the SRT and the hydraulic retention time (HRT), which reflects the basic concept that solid–liquid (sludge/effluent) separation is provided through a highly efficient membrane filtration rather than the traditional gravity settling [2]. The treatment and disposal of excess sludge produced during aerobic treatment accounts for about 50–60% of the total cost of wastewater treatment [3,4]. Therefore, sludge reduction is very important for economical treatment. MBR have advantages including complete solid removal from the effluent, effluent disinfection, high loading rate capability, low/zero sludge production, rapid start-up, compact size, and lower energy consumption. A combination of sludge disintegration techniques and advanced wastewater treatment processes such as the MBR may also be an interesting approach. The MBR process has been known to have relatively high decay rate and less sludge production due to significantly longer biomass retention in the reactor [5]. However, minimizing the production of sludge by increasing its age is limited as a result of the potential adverse effect of high MLSS on the performance of the MBR. Introduction of sludge disintegration techniques may provide a solution to the problem. A number of sludge disintegration processes including thermal [6], ozonation [7], alkaline [8], fenton [9], mechanical [10], biological [11], ultrasound [12], disperser [13], microwave [14], and biological [15] have been investigated for pretreatment of waste sludge. Recently, pretreatment techniques have been incorporated in MBRs to control excess sludge, including ozone, thermal [1], and cavitation [16] pretreatments. Alkaline pretreatment include usage of chemicals such as NaOH, KOH, $Mg(OH)_2$, and $Ca(OH)_2$. Most of the sludge disintegration investigations are evaluated on the basis of increase in soluble chemical oxygen demand (SCOD) or a decrease in volatile suspended solid (VSS) during sludge pretreatment [17–19]. Among the various alkali agents used for sludge pretreatment, sodium hydroxide is found to be efficient in disintegrating the sludge in terms of increase in SCOD and decrease in VSS [8]. The usage of a single pretreatment method has often resulted in the mineralization of the released soluble organic compound and formation of recalcitrant compounds, which in turn affect subsequent biodegradation [17]. The solution for these problems comes from a combination of pretreatment techniques. In the present study, ozone and alkali pretreatment are combined to control excess sludge production in an MBR. The combination of ozone with alkali has several advantages, such as decrease in the cost of treatment by reducing the ozone dosage, no pH correction required after pretreatment, and saving of released organic carbon [20]. The components for the alkaline-ozone pretreatment were selected based on findings from earlier studies [21,22]. In the present study, a new advanced wastewater treatment process is developed by integrating the MBR process with combined alkaline-ozone sludge pretreatment process for high-strength domestic wastewater. The effect of alkali-ozone sludge pretreatment on the reduction of excess sludge in the system was investigated. The performances of the alkali-ozone pretreatment on the MBR system including membrane fouling and effluent quality were evaluated and compared to a reference system under the same conditions.

2. Materials and methods

2.1. Synthetic wastewater preparation

Synthetic domestic wastewater was used as the experimental influent (Table 1). It was basically composed of a mixed carbon source, macro-nutrients (N and P), an alkalinity control ($NaHCO_3$), and a microelement solution [23]. The composition contained (per L) 800 mg glucose, 200 mg NH_4Cl, 220 mg $NaHCO_3$, 28 mg KH_2PO_4, and microelement solution (0.19 mg $MnCl_2{\cdot}4H_2O$, 0.0018 mg $ZnCl_2{\cdot}2H_2O$, 0.022 mg $CuCl_2{\cdot}2H_2O$, 5.6 mg $MgSO_4{\cdot}7H_2O$, 0.88 mg $FeCl_3{\cdot}6H_2O$, and 1.3 mg $CaCl_2{\cdot}2H_2O$).

2.2. MBR operation

Schematic diagrams of the MBRs are shown in Fig. 1. Among the two MBRs, one was designated as

Table 1
Characteristics of domestic wastewater

Parameter	Concentration
pH	7.3 ± 0.2
COD	800 ± 20 mg/L
sCOD	750 ± 10 mg/L
TS	2,800 ± 50 mg/L
TN	40 ± 1 mg/L
TP	6.3 ± 1 mg/L

an experimental MBR (EMBR) and was used for experimental purposes, and the other was designated as a control MBR (CMBR) and used as reference system. The working volumes of both the MBRs were 13 L. The HRT of the MBR was 8 h. The synthetic wastewater was fed into the reactor through a solenoid valve, which in turn was connected to a level sensor and implanted inside the MBR. Water level 5 cm above the membrane was set as the lower level. Decrease in water below the lower level is sensed by a level sensor, which sends a signal for the solenoid valve to open. Similarly, the upper level is maintained at 15 cm above the membrane, and a signal to close the valve is sent upon reaching this level. In order to minimize fouling of the membrane through cake layer formation, an aeration system was placed below the membrane sheet, with the cross velocity of uplifting air flow maintained within the range of 20–22 cm/s. The dissolved oxygen (DO) concentration in the aerobic basin was maintained within the range of 3.0–3.5 mg/L using an air regulator, and was monitored continuously through a DO meter. The solid–liquid separation occurs in the aerobic basin with the help of a membrane. A flat sheet membrane made out of polyolefin with a pore size of 0.22 µm was used. The effective surface area of the membrane was 0.1 m^2, with outer dimensions of 25 cm (L) × 35 cm (H) × 1 cm (T).

A suction pump was connected to the membrane, in which provision was made to measure the trans-membrane pressure (TMP) during suction. The flow rate (Q) of the suction pump was set at 36 L/d to maintain the operating flux of 17 LMH. The pump was operated with a sequence of timing which consists of 10 min switch on and 2 min switch off. Both the MBRs were started with return sludge collected from an activated sludge treatment plant in Kerala, India. Both of the MBRs were operated over a period of 120 d. For the first 59 d of the operational period, called Run I, both MBRs were operated under the same operating conditions. From the 60th day onwards, sludge pretreatment was initiated in the EMBR, lasting for 120 d, and called Run II. The sludge production rates (Y_{obs}) of both MBRs were calculated based on Eq. (1) mentioned below.

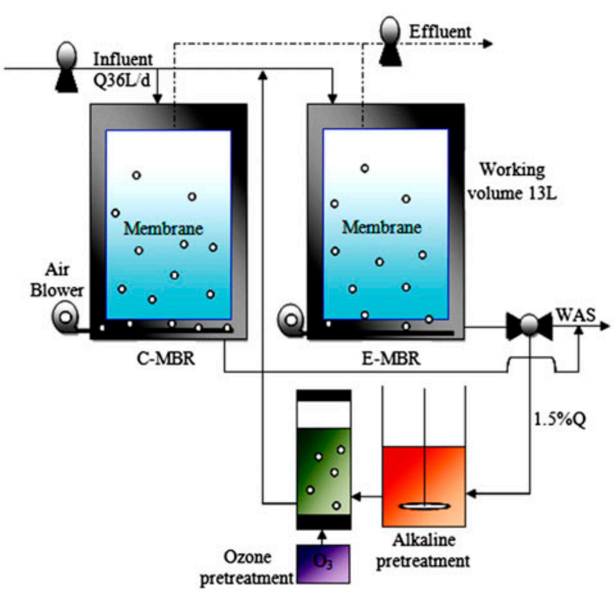

Fig. 1. Schematic representation of MBR with (EMBR) and without (CMBR) sludge pretreatment.

$$Y_{obs} = ((X_o - X_e) + \Delta X)/(S_o - S_e) \quad (1)$$

where X_o, influent suspended solids (g/L); X_e, effluent suspended solids (g/L); ΔX, net solids produced in MBR (g/L); S_o, influent COD (g/L); and S_e, effluent COD (g/L).

Calculation of daily sludge production, and both the MBRs, and comparison of their data will give clear picture about the role of sludge disintegration in controling excess sludge in EMBR. The daily sludge production was calculated based on the Eq. (2).

$$DSP_{day} = (Q_{WAS} \times X_{WAS}) + \Delta X(Q_{eff} \times X_{eff})\,g/L \quad (2)$$

where Q_{WAS}, flow rate of waste activated sludge (L/d); X_{WAS}, SS concentration in waste activated sludge (g/L); ΔX, net solids produced in MBR (g/L); Q_{eff}, flow rate of effluent (L/d); and X_{eff}, SS concentration in effluent (g/L).

2.3. Sludge pretreatment

Sludge pretreatment experiments were carried out once in 7 d. This was done by removing 540 mL of sludge (1.5% Q) every day and pooling the 7 d of sludge together (3.78 L) in a 5 L container. During this process of periodic removing and pooling, the sludge was carefully preserved in a refrigerator at 4˚C to prevent endogenous degradation. A combined treatment of alkali and ozone (O_3) was conducted in the following series, whereby the pooled sludge was treated with alkali for 2 h, followed by ozone pretreatment for 3 h. Alkaline treatment of sludge was carried out in a 5 L batch reactor using 1 N sodium hydroxide. The choice of this alkaline agent and its reaction time were made from different studies, which indicated that sodium hydroxide was more efficient than other alkaline agents in solubilizing the sludge. The first step of alkaline pretreatment was the adjustment of the pH of the solution to 11 by sodium hydroxide with the help of a pH meter. This was followed by keeping the sludge in the reactor in suspension by a slow-speed stirrer for 2 h (Digital Overhead IKA RW 20), to ensure homogeneity during alkaline treatment. After alkaline treatment, the mixed liquor was subjected to ozone pretreatment in 5 L fed batch reactor with an ozone concentration of 0.09 gO_3/g MLSS for 3 h. The ozone was generated from pure oxygen by a generator (Faraday, L10G) and injected into the bottom of the reactor through a thin bubble diffuser. The SS removal and COD solubilization for alkali and ozone were measured individually as well as combinatively. The efficiency of sludge pretreatment was measured in terms of COD solubilization efficiency and was calculated by the following Eq. (3):

$$\propto = (SCOD_p - SCOD_i)/(TCOD_i - SCOD_i) \quad (3)$$

where $\propto$, solubilization efficiency; $SCOD_p$, SCOD concentration of the sludge after disintegration (mg/L); $SCOD_i$, SCOD concentration of the sludge before disintegration (mg/L); and $TCOD_i$, TCOD concentration of the sludge before disintegration (mg/L).

2.4. Analytical methods

Samples for analysis of soluble constituents were filtered through a 0.45-μm membrane filter (GD/X PVDF, Whatman). Mixed liquid suspended solids (MLSS), mixed liquid volatile suspended solids (VSS), sludge volume index, and chemical oxygen demand (COD) were then determined on the filtrate obtained in accordance with standard methods [24]. pH and DO of the samples were measured with a Horiba pH/DO meter (Model D-55E, Japan).

3. Results and discussion

Fig. 2 presents data on the solids profile of both the MBRs. One of the advantages of the MBR over a conventional reactor is the fact that it can be operable with a high MLSS concentration. After the start-up, the MLSS concentration starts to increase steadily with an increase in the period of reactor operation, and reached a value of 7,500 mg/L on day 10 for the EMBR and day 9 for the CMBR. Subsequently, the MLSS concentration was maintained in the range of 7,000–7,200 mg/L for both MBRs by withdrawing the excess activated sludge. In the present study, sludge reduction experiments were carried out at a fixed MLSS range of 7,000–7,200 mg/L. It was clear that the sludge reduction could be increased by operating the MBR at relatively high MLSS concentrations [4]. At high MLSS concentrations, the sludge yield was comparatively lower than at low MLSS concentrations, and the designed pretreatment Q also carried more solids for disintegration. However, it was reported that an increase in solid concentration in the MBR causes cake fouling, and this requires frequent cleaning [22,25].

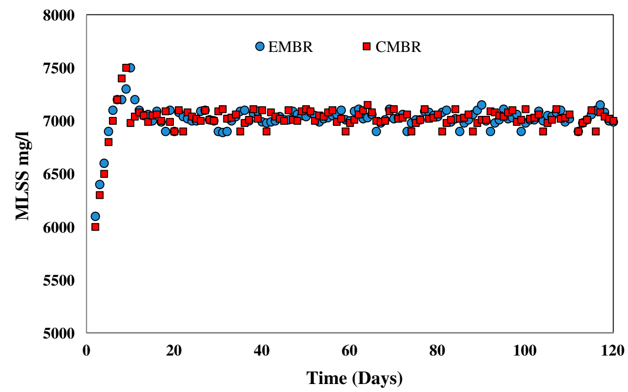

Fig. 2. MLSS profile of MBRs during the study period.

Consequently, the present study uses an MLSS of 7,200 mg/L, which was found to be optimum for the performance of MBRs. From the figure it is clearly evident that the volatile solids of the both MBRs are almost identical during the period of study. It was expected that integration of sludge pretreatment system in EMBR may cause decrease in volatile fraction of suspended solids. However, the inorganic fraction of the degraded cells did not accumulate in the EMBR and presumably permeated through the membrane as ionic species. Similar to our study, while working on integration of sludge disintegration system in A_2O treating domestic wastewater, Banu et al. [4] have reported, that there is no change in volatile fraction of the mixed liquor before and after the sludge pretreatment.

3.1. Sludge pretreatment

Pretreatment was done to improve the bioavailability of sludge particulate material. SCOD calculations were considered the main parameter for evaluation of sludge particulate material, and this enables an evaluation of the maximum level of sludge solubilization. Increased SCOD is determined as the substance that can be readily biodegradable [26]. In the present study, sludge pretreatment was carried out by subjecting a mixed liquor of EMBR to 1.5% of Q. Understandably; an increase in pretreatment Q of over 1.5% increases the percentage of sludge reduction.

However, it was reported that an increase in the pretreatment of *Q* of over 1.5% was not an economically viable option [27]. Consequently, it was decided that the pretreatment of *Q* would be maintained at 1.5%. Among the various pretreatment techniques used for controling excess sludge production in wastewater treatment systems, hybrid techniques such as thermo-chemical and alkali-ozone were found to be more effective [28]. During the pretreatment, SCOD concentration was found to be increased, and is shown in Fig. 3. With alkaline treatment, the SCOD increases in the range of 900–1,400 mg/L, and subsequent ozone treatment produces an additional SCOD increase of 2,400–3,000 mg/L. Combining alkaline with ozone treatment resulted in SCOD release in the range of 3,500–4,300 mg/L. The average SCOD solubilization efficiency by alkaline treatment was about 15%, and the corresponding average suspended solid (SS) reduction was about 11%. The combination of ozone with alkali treatment leads to an increase in SCOD solubilization from 15 to 40%, and an SS reduction from 11 to 30%.

From Fig. 4, it is clearly evident that the combination of these two technologies proved to be a promising choice of sludge pretreatment. Alkaline treatment destroys floc structures and cell walls by hydroxyl anions. Extremely high pH (10–12) causes natural shape losing of proteins, saponification of lipid, and hydrolysis of RNA [17]. The combination of alkali with ozone treatment not only reduces the ozone dosage significantly, but also decreases the cost of the sludge pretreatment. In the present study, the combination of pretreatment techniques of ozone (0.09 gO_3/g MLSS) and alkali (pH 11) resulted in the COD solubilization of 40%. The integration of pretreatment techniques individually in MBR resulted in very low COD solubilization (10–12%) at high alkali (11.4–12) and ozone dosage (0.1 gO_3/g SS) [1]. In

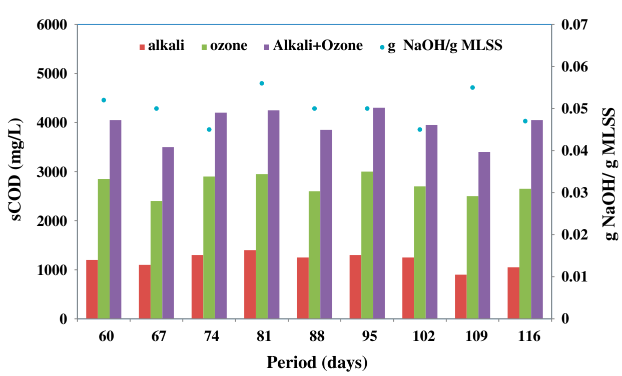

Fig. 3. Effect of alkali-ozone pretreatment on SCOD release during the study period.

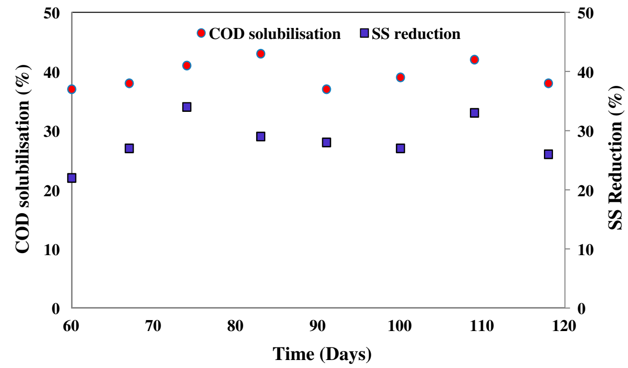

Fig. 4. Effect of alkali-ozone pretreatment on COD solubilization and SS reduction.

addition, the alkali acts as a buffering agent to resist the drop in pH during ozone treatment [21]. The recycling of this slightly alkaline (pH 9.2–9.6) pretreated sludge at 1.5% of *Q* into the EMBR did not cause any significant change in the pH profile of the reactor. During Run II, the pH profile of the EMBR and CMBR were in the range of 7.4–7.6 for former and 7.0–7.2 for latter, respectively. The slight increase in effluent pH of EMBR was attributed to the recirculation of alkaline ozone pretreated sludge. However, the recirculation did not cause any abrupt increase in pH, which subsequently decreases biological degradation in aerobic system. During sludge recirculation, heavy dilution of pretreated sludge with wastewater happens at aerobic basin and was responsible for neutralization of pH.

3.2. *Effect of pretreatment on sludge yield*

The sludge yield of the system depends on the organic strength of the wastewater. Based on the organic content, domestic wastewater was classified into three types, including low strength (COD = 250 mg/L), medium strength (COD = 430 mg/L), and high strength (COD = 800 mg/L) [29]. The present study uses domestic wastewater with 800 ± 20 mg/L of COD, falling under the category of "high-strength." The sludge production rate was calculated based on Eq. (1) and is presented in Fig. 5. The Y_{obs} value for both the MBRs was found to be 0.38 kg MLSS/kg COD. The presently observed Y_{obs} value was considerably higher than the values reported for sludge reduction in MBR-treated domestic wastewater [21,27]. However, the wastewater used by researchers [21,27] falls under the category of "low-strength." It was well known that high-strength wastewater produces more biomass than low-strength wastewater, and this may be the reason for high biomass production observed in the present study. In Run

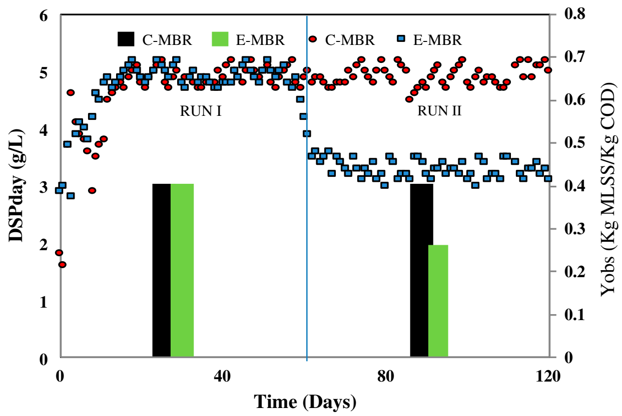

Fig. 5. Effect of alkali-ozone pretreatment on COD solubilization and SS reduction.

II, the average Y_{obs} values for the EMBR and CMBR were found to be 0.24 kg MLSS/kg COD and 0.38 kg MLSS/kg COD, respectively. Upon comparing the Y_{obs} values of the EMBR with CMBR, it was clearly evident that sludge pretreatment plays an important role in excess sludge reduction, accounting for 37%. Excess sludge removal was a regular procedure in the aerobic treatment system and was carried out to maintain the biomass balance inside the reactor. The quantification of the daily sludge production (DSP_{day}) is based on Eq. (2). This mass balance equation takes into account the quantity of waste sludge, the accumulation within the biological reactor and the sludge loss with treated effluent. However, it was known that the quantity of sludge passing the membrane permeation was zero and the accumulation of sludge in the reactor could be negligible as MLSS was maintained at a relatively constant level. A graphic representation of DSP_{day} calculated by Eq. (2) vs. time shows the dynamic evolution of sludge production over the experimental period, and is presented in Fig. 5. From Fig. 5, it was clearly evident that during Run I, the solids concentrations in the DSP_{day} of both the MBRS were similar and varied in the range of 5 g/d. During Run II, there was a significant reduction in solid concentration in the DSP_{day} of 3.1 g/d, and this indicates the role of sludge pretreatment. From these observations, it was clearly evident that the alkali-ozone sludge pretreatment in the EMBR accounted for 37% of the reduction in DSP_{day}. The observed excess sludge reduction was comparable with the values of 33 [27] and 42% [4] reported for MBRs, integrated with pretreatment techniques. By considering the economics, proposed method of sludge reduction was feasible than other conventional anaerobic digestion, where it demands high capital cost.

In addition, hydrolysis is the rate limiting step in anaerobic digestion and sludge without pretreatment normally results in very poor solids reduction [15]. In the present study biodegradation of solids happens at free of cost along with wastewater treatment and it did not demand additional energy as well as capital investment.

3.3. *Effect of pretreatment on effluent quality and performances of MBR*

Fig. 6 shows the variation in COD removal efficiency of both the MBRs during the study period. The COD removal efficiencies of the EMBR before and after the introduction of sludge pretreatment were found to be in the ranges of 97–99% and 98–99%, respectively (calculated from Fig. 6). From the results, it was evident that the COD removal efficiency of the EMBR remains unaffected before and after the introduction of sludge reduction practices. The COD removal efficiency of the CMBR during the study period was found to be in the range of 96–99% (calculated from Fig. 6). A *t*-test analysis showed that the differences between the EMBR and CMBR were not statistically significant. However, it has been reported that in wastewater treatment processes including disintegration-induced sludge degradation, the effluent water quality was slightly deteriorated due to the release of non-degradable substances such as soluble microbial products [30]. The COD removal increased with an increase in time during the initial phases of the reactor operation. It attains a steady state on day 19. From then onwards, the COD removal was in the range of 96–98% (calculated from the graph). During the stable operational period, the SCOD concentration in the aerobic basin of MBR was found to be 22–40 mg/L for the EMBR and 24–45 mg/L for the

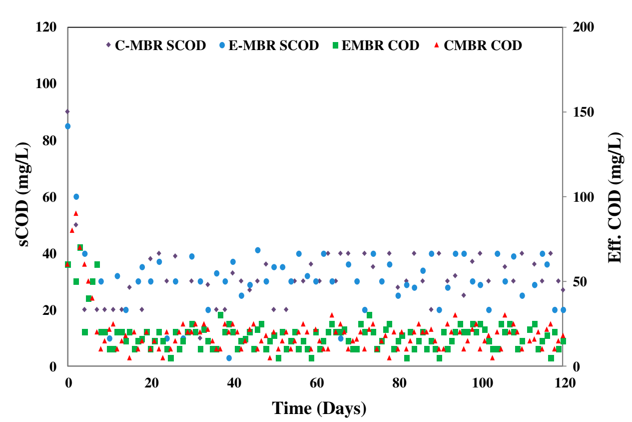

Fig. 6. Efficiency of MBR treating domestic wastewater.

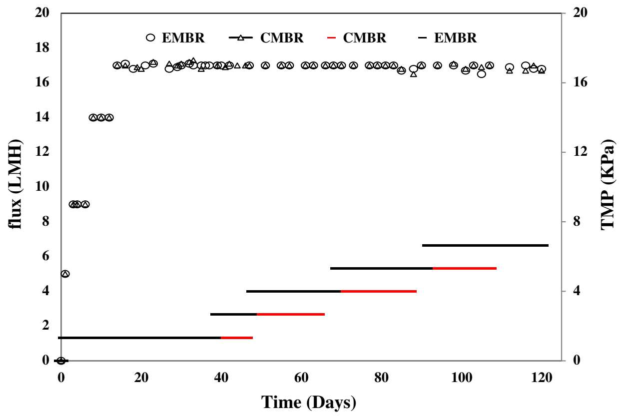

Fig. 7. Variation of MBRs TMP profile during the study period.

CMBR. The corresponding organic concentration in the effluent varied from 8 to 22 mg/L for the EMBR and 12–26 mg/L for the CMBR. From this, it can be concluded that the membrane separation played an important role in providing excellent and stable effluent quality.

The suction pump was started after the first week of seeding and was based on the SCOD concentration of the aerobic basin. The pump was started when the SCOD in the aerobic basin was less than 45 mg/L. The designed flux for the membrane was 17 LMH. This was achieved by a stepwise increase of flux from 25 to 100% over a period of three weeks. Fig. 7 shows the TMP variation during the operational period, indicating that the TMP increased slowly over a period of 120 d. At the end of 120 d of reactor operation, the TMP was found to be 7 kPa.

4. Conclusion

The stable operation of the MBR process was possible without significant accumulation of biomass, when part of the biological solids was disintegrated with alkali at pH 11 and an ozone dose of 0.09 gO_3/g MLSS. The combination of ozone with alkali achieved 40% COD solubilization. Recycling of the pretreated sludge at 1.5% *Q* in the EMBR for the subsequent biodegradation causes an excess sludge reduction of 37%. The recycling of pretreated sludge in the EMBR did not cause any significant increase in TMP. In Run II, the EMBR sludge yield decreases compared to that in the CMBR. The COD removal efficiency of both MBR reactors was achieved up to 97%. The excess sludge production in MBRs was constrained by the combined pretreatment method without any deterioration in the treated water quality and membrane performances.

Acknowledgments

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