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Performance evaluation of membrane bioreactor (MBR) coupled with activated carbon on tannery wastewater treatment

Abolghasem Alighardashi[*1], Mahyar Pakan[1a], Shervin Jamshidi[2b] and Farshid Pajoum Shariati[3c]

[1]Department of Civil, Water and Environmental Engineering, Shahid Beheshti University, Shahid Abbasspour Blvd., Tehranpars, P.O. Box: 167651719, Tehran, Iran
[2]Water and Wastewater Research Center, Water Research Institute (WRI), Shahid Abbasspour Blvd., Tehranpars, P.O. Box: 16765313, Tehran, Iran
[3]Science and Research Branch, Islamic Azad University, Simon Bulivar Blvd., P.O. Box: 14515775, Tehran, Iran

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Abstract. This study evaluates the performance of membrane bioreactor (MBR) coupled with a modified walnut shell granular activated carbon (WSGAC) for tannery wastewater treatment. For this purpose, a pilot with overall volume of 80L and 12 hours hydraulic retention time (HRT) is operated in three scenarios. Here, the chemical oxidation demand (COD) of wastewater is reduced more than 98% in both C:N ratios of 13 (S1) and 6.5 (S2). This performance also remains intact when alkalinity depletes and pH reduces below 6 (S3). The ammonium removal ranges between 99% (S2) and 70% (S3). The reliability of system in different operating conditions is due to high solids retention time and larger flocs formation in MBR. The average breakthrough periods of WSGAC are determined between 15 minutes (S2) and 25 minutes (S1). In this period, the overall nitrate removal of MBR-WSGAC exceeds 95%. It is also realized that adding no chemicals for alkalinity stabilization and consequently pH reduction of MBR effluent (S3) can slightly lengthen the breakthrough from 15 to 20 minutes. Consequently, MBR can successfully remove the organic content of tannery wastewater even in adverse operational conditions and provide proper influent for WSGAC.

Keywords: membrane bioreactor (MBR); granular activated carbon (GAC); Walnut shell; tannery wastewater treatment; nitrification

1. Introduction

Industrial effluents typically contain unusual constituents of pollution for biological treatment in comparison with domestic wastewaters. In order to meet stringent discharge requirements, the application of membrane bioreactors (MBR) have been recently extended. This is because of its

∗Corresponding author, Assistant Professor, E-mail: a_ghardashi@sbu.ac.ir
[a]Ph.D. Student, E-mail: m_pakan@sbu.ac.ir
[b]Ph.D., E-mail: sh.jamshidi@ut.ac.ir
[c]Assistant Professor, E-mail: pajoum@ut.ac.ir

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Table 1 Review of recent applications of MBR on organics removal from ammonia rich wastewaters

Industry	Type of system	COD (mg/L)	COD Removal (%)	BOD (mg/L)	NH_4^+ (mg/L)	NH_4^+ Removal (%)	pH	reference
Dyeing	MBR	1300	-	250	100	-	-	(Feng *et al.* 2010)
Textile	MBR	1500	-	500	50	-	-	(Yigit *et al.* 2009)
Palm oil	MBR	67000	-	34000	50	-	-	(Yuniarto *et al.* 2013)
Pet food	MBR	21000	-	10000	110	-	-	(Acharya *et al.* 2006)
Tannery	Chemical oxidation	2533	-	977	118	-	6-9	(Mandal *et al.* 2010)
Synthetic	MGSBR	300	-	-	80	-	7.4	(Li *et al.* 2005)
Leachate	MBR (FS)	5987	68.5	695	2464	63.4	8.4	(Hashisho *et al.* 2016)
Leachate	MBR (HF)	5987	71.4	695	2464	47.8	8.4	(Hashisho *et al.* 2016)

advantages over the conventional systems like activated sludge. MBR is made of two sequential units and uses membranes as clarification of the aerated microbial consortium. This leads into better effluent quality, more capacity for organic loading, reduced footprint and sludge production, and process flexibility towards influent variations (Judd and Judd 2011, Park *et al.* 2015). In addition, high solid retention time (SRT) provides an opportunity for these systems to be reinforced against shock loads or toxic materials in industrial wastewater treatment (Awan *et al.* 2015, Zhang *et al.* 2015). These advantages have motivated researchers to study MBR for biological treatment of high strength industrial wastewaters (Lin *et al.* 2014, Mutamim *et al.* 2013). Some of recent studies that focused on using MBR for industrial wastewater treatment are summarized in Table 1.

Tannery industry is made of different procedures as soaking, unhearing and liming, bathing and deliming, degreasing, pickling, tannage operation, retaining and finishing. In these processes, wastewater conveys considerable amounts of ammonium and biodegradable products (Wang and Liu 2011). Sabumon (2016) emphasized that a caution should be considered for biological treatment of tannery wastewater as the influent has typically high concentrations of ammonium. Ammonium can be removed from polluted waters by various physicochemical and biological processes. Since biological nitrogen removal is effective and inexpensive, it has been adopted widely rather than physico-chemical processes (Buha *et al.* 2015). Through the biological nitrification process, ammonia is oxidized to nitrite (NO_2^-), and nitrate (NO_3^-), by aerobic autotrophic bacteria (Gerardi 2003). Hence, the aerobic systems need to employ high aeration with low organic loads for proper operation while anaerobic systems would be inhibited by sulphide concentration. Since tannery wastewater consists of high nitrogen content, Ganesh *et al.* (2015) proposed sequencing batch reactor (SBR) as a viable option in comparison with aerobic systems. Munz *et al.* (2008) compared the performance of MBR and conventional activated sludge on tannery wastewater treatment in pilot scale. They concluded that MBR is more effective on COD removal and nitrification. Ammonia oxidizing bacteria (AOB) and nitrifiers are more observed in the bioreactor. Munz *et al.* (2010) also realized that the specific growth rates of AOBs in MBR are in range of 0.45 to 0.49 per day (d^{-1}). Wang and Liu (2011) found that MBR systems can reduce COD and ammonia of tannery wastewater to more than 90%. Ratanatamskul *et al.* (2013) successfully tried the prototype of MBR with 4m3/d for onsite treatment of domestic wastewater.

Here, organic and ammonia removal could exceed 90%. Gallego-Molina *et al.* (2013) focused on nitrogen removal by micro filtration (MF) and using replacement of ammonium salts by carbon dioxide in the deliming process of tannery industry. These could reach 53% of total nitrogen (TN) removal. They found that settling and precipitation of nitrogen has not influence on overall TN removal. De Gisi *et al.* (2009) found that a biological pre-treatment works well for tannery wastewater treatment prior to MBR systems. Ma *et al.* (2012) examined the application of poly aluminum chloride (PAC) combined with MBR to remove pollutants as ammonium from surface waters. They could achieve 93% NH_4 removal in addition to the fouling reduction by PAC. In the present paper, a combination system of aerobic MBR with walnut shell granular activated carbon (WSGAC) is proposed for removal of nitrogenous and carbonaceous pollutants contained in tannery processing wastewater. The objective of this paper is to study the performance of a laboratory scale MBR-WSGAC in three operating conditions. These conditions include low and high N:C ratio of influent in addition to low pH level in final step.

2. Materials and method

2.1 Experimental set-up

This study was carried out in three steps regarding the effluents of tannery industry. In the first step (S1), MBR was fed for two months by real tannery wastewater with low N: C ratio of 0.077. In the second step (S2), the N:C ratio increased synthetically to 0.15 by adding ammonium nitrate (NH_4NO_3) and diammonium phosphate, $(NH_4)_2HPO_4$, resembling to the high nitrogen effluent content of tannery industries. In the third step (S3), adding chemicals for alkalinity adjustments was stopped to evaluate the performance of system in low alkalinity and pH values.

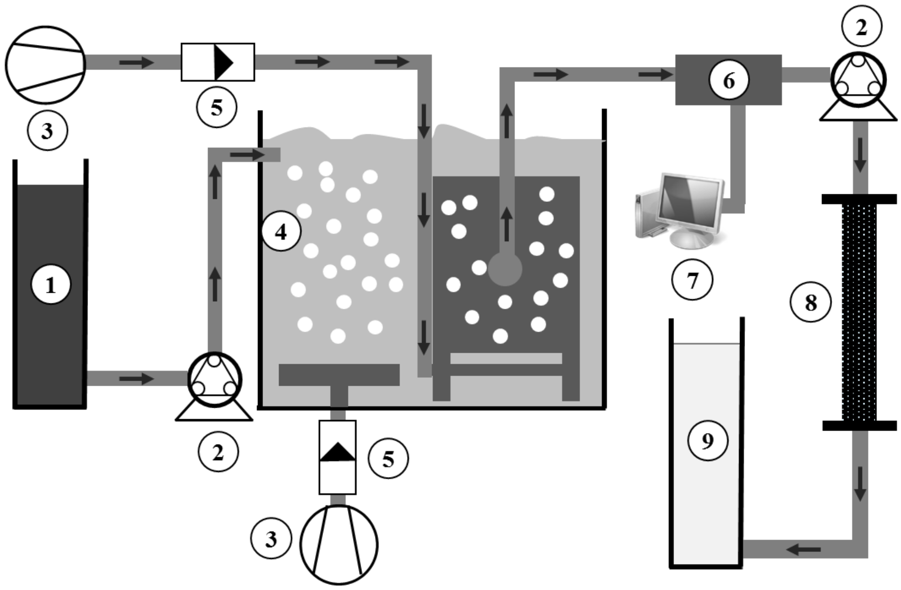

Fig. 1 Schematic digram of pilot: 1-feeding tank, 2- peristaltic pump, 3-blower, 4-MBR, 5-air flow meter, 6-trans membrane pressure, 7-PC (data recorder), 8-GAC, 9-effluent tank

Table 2 Parameters of the membrane modules used in this experiment

Parameter	Characteristics
Membrane module	Microdyn-Nadir Bio cell
Pore size	0.04 µm
Frame type	PVC
Maximum pressure	0.4 Bar
Surface area	0.35 m^2
Membrane type	PES
pH	2-11

Table 3 Physical-chemical characteristics of activated carbons

	Walnut shell (This study)	Almond shell*	Hazelnut shell*
C%	88.7	50.3	51.4
H%	0	6.05	5.95
Ash%	2.6	0.31	0.49
Moisture (%)	9	8.7	7.7
Iodine number ($mg.g^{-1}$)	905	638	965
BET surface area (m^2g^{-1})	1434.6	736	793

* derived from Aygun *et al.* (2003)

2.2 Pilot specifications

The aeration tank of MBR was made of Plexiglas having 64 cm length, 34 cm width, and 63 cm height with the net volume of 80 L (Fig. 1).The peristaltic pump fed the reactor with an average flow rate of 160 L/day as it equally discharged through the membrane module. The membrane module (Microdyn-Nadir GmbH, Germany) was installed vertically in the aerobic zone within a PVC frame as diffusers were located at the bottom of membrane and the aeration tank. The characteristics of membrane are described in Table 2.

2.3 WSGAC specifications

The granular activated carbon used in this research was originated by the commercial walnut shell obtained from Tūyserkān, Iran. For preparation of modified WSGAC, the physical walnut shell (carbon source) was mixed with H_3PO_4 (5%) with constant magnetic stirring of 30 minutes. The chemo-physical activated carbon was washed with hot ultra-pure water, until the pH of the filtered solution became neutral. The samples were dried overnight at 120℃ and placed in dry place and stored in plastic containers. The overall characteristics of WSGAC are listed in Table 3.

In the pilot (Fig. 1), the WSGAC column was made of plexiglas with internal diameter of 5 cm. This was packed with the adsorbent between two supporting layers of sand and glass wool with total height of 30 cm and average pore diameter of 2 nm. The observed pore volume was 0.747 cm^3/gr. The WSGAC has the chemical composition of 88.7% carbon, 5.2% nitrogen, 4.7% oxygen, and 1.2%s ulfur. Table 3 shows the characterization of walnut shell comparing with almond and hazelnut shells (Aygun *et al.* 2003). It is obvious that the WSGAC has more surface area and carbon content while its iodine number is rather high. According to the previous laboratory scale experiments, the adsorbent follows Langmuir isotherm on nitrate removal (Fazeli *et al*. 2016).

2.4 Pilot operation and start-up

The pilot was inoculated by municipal sludge with an average mixed liquor suspended solids (MLSS) of 6.5 gr/L. It was derived from a wastewater treatment plant with MBR system in Tehran. The HRT was set 24hr in the start-up and decreased to 12 hrs in the main test. In the whole experiment, the local temperature was between 19 and 26℃. Air was continuously diffused directly below the membrane module for biomass growth and scouring the membrane surface and also for turbulence in the bioreactor. The DO was maintained at 3.5±0.5 mg/L. In addition, the average MLSS content of MBR maintained between 5.7 nd 8.7 gr/L by discharging the excess sludge by the sampling valves beneath the reactor. Here, the ratio of mixed liquor volatile suspended solids (MLVSS) to MLSS was 0.89 in average.

In order to control the fouling in MBR, the pilot used physical relaxations periodically. As shown in Fig. 2, four times of relaxations were used in 7 months of operation. The first period attributes to S1 while the second is in duration of S2. It is obvious that the diffusers were properly in line and trans-membrane pressure (TMP) did not exceed 80 mBar. However, in long run after the experiments, the membrane module encountered fouling after 4 months in which TMP reached 250 mBar.

2.5 Sampling and tests

In order to study the performance of MBR, in addition to the operating parameters, the analytical factors were measured according to the standard methods (APHA, 2003) as Table 4. Here, the analysis of N components and COD were carried out by the photometric cell test (Hach method) and DR5000 Spectrophotometer for these parameters

2.6 Wastewater specifications

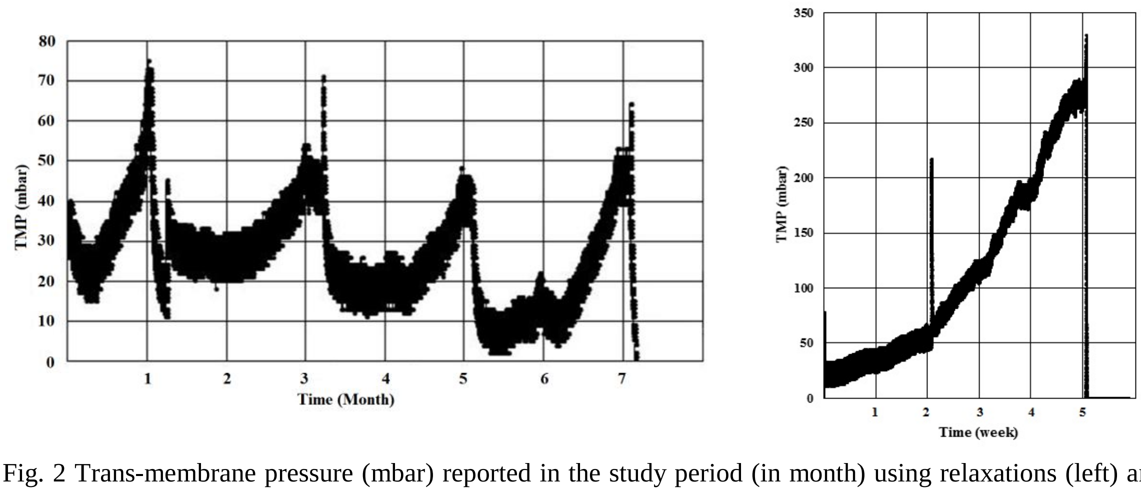

Fig. 2 Trans-membrane pressure (mbar) reported in the study period (in month) using relaxations (left) and without relaxations in start-up period (right)

Table 4 Methods and standards used for wastewater analysis and tests

Parameter	Standard	Model
pH	APHA 2003,4500-H	HQ40d, HACH ®
EC	APHA 2003, 2510	HQ40d, HACH ®
TDS	ASTM 5907	HQ40d, HACH ®
DO	APHA 2003,4500-O	HQ40d, HACH ®
Temperature	APHA 2003, 2550C	HQ40d, HACH ®
COD	HACH method 8000	DR5000, HACH ®
BOD	APHA 2003	BODTrakTM, HACH ®
MLSS	APHA 2003, 2540D	-
MLVSS	APHA 2003, 2540-E	-
Alkalinity	APHA 2003, 2320	-
$N\text{-}NH_3$	HACH method 8038	DR5000, HACH ®
$N\text{-}NO_2$	HACH method 8507	DR5000, HACH ®
$N\text{-}NO_3$	HACH method 8039	DR5000, HACH ®

Table 5 Average influent characteristics in three scenarios

Parameters	unit	S1	S2	S3
$N\text{-}NO_2$	mg/L	<0.05	<0.05	<0.05
$N\text{-}NO_3$	mg/L	35	80	80
$N\text{-}NH_4$	mg/L	62	118	118
TN	mg/L	97	198	198
NH_4/TN	-	0.64	0.6	0.6
NaOH	mg/L	30	30	-
Na_2CO_3	mg/L	10	10	-
COD	mg/L	2500	2500	2500
BOD	mg/L	1100-1430	1100-1430	1100-1430
TN/BOD	-	0.077	0.156	0.156
Alkalinity	$mgCaCO_3/L$	>500	>500	150-200

The leather industry wastewater was derived from Charmshahr industrial city, Tehran province, Iran. Here, the screened, grit and grease removed influent was pumped directly to the feeding tank. In the two initial steps of experiments, sodium carbonate and sodium hydroxide were added to influent to increase the alkalinity for nitrification while it was stopped in the third condition (S3). The overall characteristics of wastewater are shown in Table 5.

3. Results and discussion

Regarding the experimental results in three steps, it is realized that the aerated membrane bioreactor is capable of removing organic contaminants of wastewater. As shown in Fig. 3, ammonium removal increases to nearly 99% in S1 as this performance continues and remains

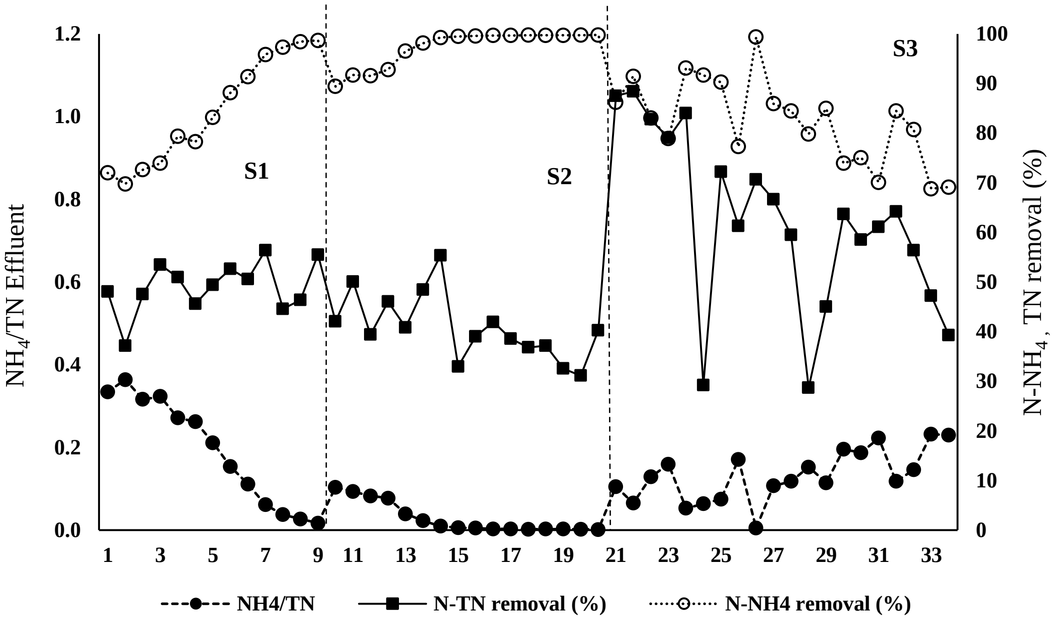

Fig. 3 The performance of MBR and GAC on ammonium and TN removal

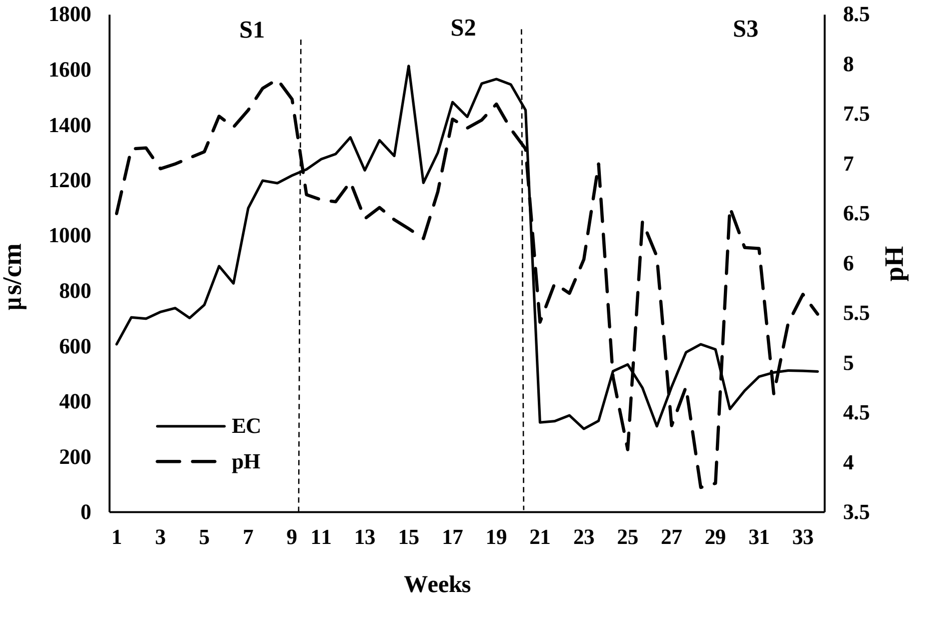

Fig. 4 pH and EC content of effluent in different sampling and experimental steps

steady in S2. It shows that enhancement of initial NH_4 of wastewater may not limit the potential of nitrification unless the alkalinity reduces (S3). In S1 and S2, the reactor shows almost a limited variation in TN removal, mostly due to the performance of WSGAC. This ranges in average between 30 to 55%. The mean values of TN removal are respectively 49 and 40% for S1 and S2. Here, pH varies between 6.5 and 7.5 (Fig. 4).

As illustrated in Fig. 4 for S3, pH significantly decreases below 6. This is directly due to the stop in adding chemicals for alkalinity adjustments that correlates with the electro-conductivity of the effluent. Since *Nitrosomonas* and *Nitrobacter* respectively grow well at pH 7.9 and 7.4, it is expected that low pH slows nitrification down (Gerardi 2003, Tarre and Green 2004). However, MBR performance in S3 shows that acid phase cannot significantly inhibit nitrification and only slows it down. NH_4 removal ranges between 70 to 90% with an average of 82% (Fig. 3) while the ratio of NH4: TN increases to 0.23. This performance is rather significant in comparison with previous studies over MF (Gallego-Molina *et al.* 2013). This implies that MBR can provide proper environment for biomass to be active in unstable condition of low pH. By comparing pH with NH_4 removal in Figs. 3 and 4, it can be concluded that nitrification gradually decreases in pH below 6.5 for twelve weeks in MBR system. This can be due to high residence time of microbial consortium that increases the operational reliability against alkalinity reduction. It can provide an opportunity for operators to rehabilitate their system by pH adjustments chemically (Gieseke *et al.* 2006). Yet, it may be steepened in pH lower than 4. In contrary to the reduction of NH_4 removal, TN removal of MBR-WSGAC is developed to 62% in average. This can be due to the fact that WSGAC better adsorbs nitrate and nitrite in lower pH and EC values of MBR effluent.

Moreover, in periods between 15 and 20 weeks, partial granulation of sludge is observed in which denser microbial structure with better settling ability is formed (Liu and Tay 2004). The particle size distribution analysis shows a shift to larger flocs from S1 to S2 (Fig. 5). The diagram illustrates that less than 30% of sludge particles in S2 are 1.5-2.5 times bigger in size than S1. This justifies why nitrification (Fig. 3) and COD removal (Fig. 6) can reach to their maximum level in S2.

Fig. 6 shows that the COD concentration in MBR effluent does not significantly differ in three operating conditions and all three are below 40 mg/L. For S1, S2 and S3, the overall COD removal in the aeration tank are 87.3, 81.9 and 88.8%, respectively. Yet, membrane system respectively

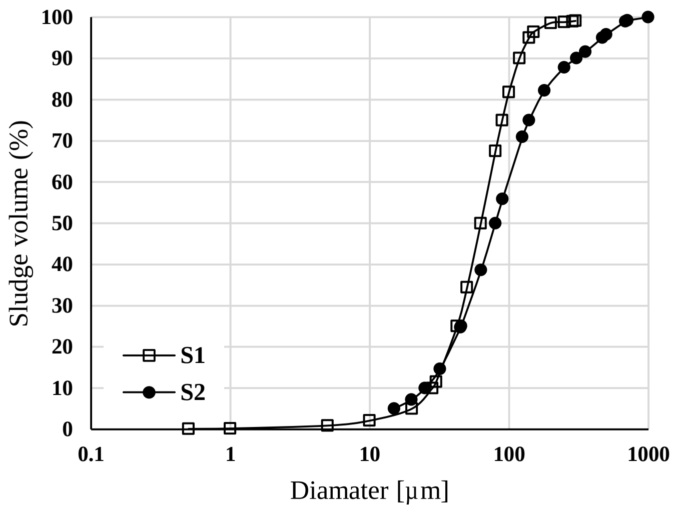

Fig. 5 Particle size distributions of sludge in MBR system in two scenarios

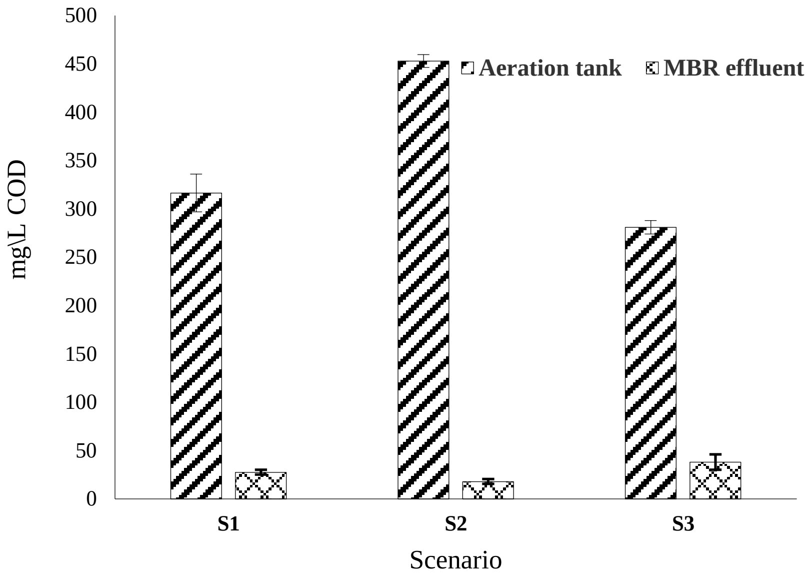

Fig. 6 Soluble COD (mg/L) in the aeration tank and effluent of MBR in three scenarios

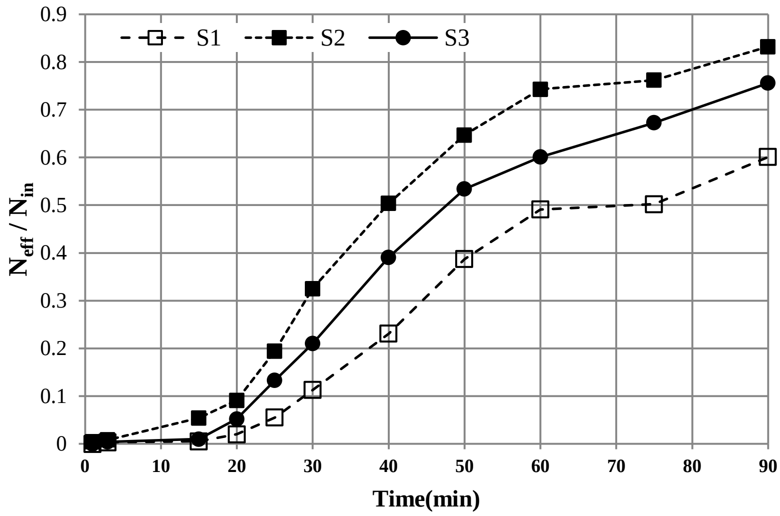

Fig. 7 Comparative nitrate adsorption of WSGAC in three operational scenarios

increases these efficiencies to 98.9, 99.3 and 98.5%. Mannucci *et al.* (2014) could reach into 90% COD removal of tannery wastewater by upflow anaerobic filters (UAF). It points to several conclusions. First, the aerated MBR is enough for organics removal of tannery wastewater. Second, this system can efficiently work in higher N: C ratio of influent. Third, high nitrification and low chemical adjustments for alkalinity and pH reduction may not have considerable impact on COD removal. Therefore, these systems can provide a rather carbon and suspended solids free effluents for WSGAC as the next step. Here, it is expected that nitrogen removal increases using

WSGAC to compensate TN removal.

Fig. 7 shows that in the operating condition with the highest TN influent content (S2), the columns of WSGAC encounter breakthrough by 15 minutes in average. This is the duration in which 5% of nitrate concentrations pass through the GAC column. It means that by that time, the effluent quality regarding nitrate concentration degrades and the potential of WSGAC adsorbent reduces significantly. However, in S1 with lower TN concentrations, the breakthrough duration exceeds 25 minutes as expected. Here, the interesting point is that in S3, in which the TN content of influent is as high as S2, the breakthrough is calculated between S1 and S2 conditions. The duration is nearly 20 minutes. This can be due to the fact that in lower pH and EC content of MBR effluent, the potential of nitrate adsorption increases. This implies that in tannery wastewater treatment by MBR, there is an operational alternative that WSGAC increases its adsorption efficiency whenever the alkalinity is not fixed by chemical additives. The experimental results revealed that MBR can well resist against low pH of influent in couple of weeks in which the WSGAC can increase TN removal efficiency. In addition, in spite of low operational duration of WSGAC, TN and COD removal of this system is comparable with even modified constructed wetlands in same HRT with much higher footprint area (Jamshidi *et al.* 2014).

4. Conclusions

This study evaluated the performance of MBR-WSGAC for tannery wastewater treatment. Here, the nitrogen content of influent is high and MBR showed well performance on COD removal and NH_4 nitrification to more than 98%. In addition, the experimental results revealed that MBR can well maintain its performance even in low alkalinity and therefore pH level in the aeration tank. This can ensure some reliability for operation in times of high nitrogen content of influent, high nitrification rate and low alkalinity. Moreover, MBR showed TN removal between 30 to 45%. This efficiency on TN removal exceeds 70% using WSGAC. However, the breakthrough of column was reported between 15 to 25 minutes as low pH and dissolved solids of MBR effluent could increase its efficiency and life time. This duration shows that MBR can successfully reduce the impurities of tannery wastewater and provide proper influent for WSGAC. Here, the modified WSGAC can be used as natural additive to reduce TN level of wastewater.

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