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# Performance evaluation of a submerged membrane bioreactor for the treatment of brackish oil and natural gas field produced water

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ABSTRACT

Produced water, which is co-produced during oil and gas manufacturing, represents one of the largest sources of oily wastewaters. Therefore, treatment of this produced water may improve the economic viability and lead to a new source of water for beneficial use. In this study a submerged hollow fiber membrane bioreactor (MBR) has been studied experimentally for the treatment of brackish oil and natural gas field produced water. This type of wastewater is also characterized with relatively moderate to high amount of salt, oil and total petroleum hydrocarbons (TPH). However, the bacteria which are growing in conventional activated sludge and MBR cannot survive at these strict conditions, therefore acclimation of the bacteria is of vital importance. The performance of the biological system, membrane permeability, the rate and extent of TPH biodegradability have been investigated under different sludge age and F/M ratios. The results obtained by gas chromatography analyses showed that the MBR system could be very effective in the removal of TPH from produced water and a significant improvement in the effluent quality was achieved.


## 1. Introduction

Produced water (PW) is described as any water that is present in a reservoir with the hydrocarbon resource and produced with crude oil or natural gas. It contains different substances such as oil, salts, heavy metals, organic acids, radionuclides and is considered as the major source of pollution in the oil and gas fields. Therefore it needs to be properly treated before using for any kinds of purposes [1,2].

Management of PWs has become a major issue in the feasibility of gas field development in order to prevent serious environmental effects and it provides a vital water source for beneficial use [3,4]. Surface discharge of PWs had many drawbacks including salt deposition, stream bank erosion and change in natural vegetation [5]. Currently, there are two main approaches recommended for the management of PW: reinjection to the discharged wells and treatment for reuse. More than 60% of the PW are reinjected into the wells [6]. Besides, reuse of PW has also received more attention due to increasing demand for additional water resources. In this respect, PW management by applying economical and environmentally friendly treatment methods is very important for the protection of the environment. Oil and salt are the main contaminants that should be removed from PW in order to be able to reuse. The ultimate usage of this water is strongly dictated by the removal efficiencies or the final effluent quality [7,8].

Up to now different methods have been applied for the treatment of PW such as dissolved air flotation, gravity separation and skimming, coagulation and flocculation, de-emulsification [9–12]. Since there are many disadvantages associated with these unit operations such as the post-treatment requirement due to the low effluent quality, high footprint need, high chemical consumption and high operation costs, development of a new economical PW treatment option is required. Recently increase in the need for the reuse of wastewater and stringent discharge regulations makes the MBRs attractive for the treatment of PWs. The use of MBR offers many advantages over the conventional treatment methods for the treatment of PWs including high effluent quality, high loading rate capability, low sludge production, no chemical additives need, compactness, and lower energy costs. These advantages highlight the application of MBR in the treatment of produced water.

However, only a few studies in the literature have been carried out to investigate the applicability of the MBR technology for the

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**Table 1**
Wastewater characterization of TPAO basin brackish oil and gas field produced water (the values are given as average ± standard deviation).

| Parameter | Average value |
|---|---|
| COD (mg/L) | 2371 ± 521 |
| TSS (mg/L) | 58 ± 50 |
| TDS (mg/L) | 8367 ± 1679 |
| TKN (mg/L) | 25 ± 4.9 |
| $NO_3$-N (mg/L) | 0.07 ± 0.01 |
| $NO_2$-N (mg/L) | n.d |
| $NH_4$-N (mg/L) | 18 ± 3.5 |
| TP (mg/L) | 0.4 ± 0.3 |
| TPH (mg/L) | 2301 ± 4.4 |
| $CN^-$ (mg/L) | 0.1 ± 0.01 |
| Oil and Grease (mg/L) | 140 ± 118 |
| Salinity (‰) | 8.7 ± 1.7 |
| Temperature | 20.1 ± 4.9 |
| pH | 7.95 ± 0.2 |
| Conductivity (μS/cm) | 13,460 ± 2541 |
| $Na^+$(mg/L) | 2976 ± 520 |
| $Cl^-$ (mg/L) | 3861 ± 785 |
| $SO_4^{2-}$(mg/L) | 204 ± 63 |
| $Br^-$(mg/L) | 16.1 ± 1.7 |
| $Ca^{2+}$(mg/L) | 72.4 ± 41 |
| $Mg^{2+}$(mg/L) | 34.4 ± 10.8 |
| $S^{2-}$ (mg/L) | 376.4 ± 331.2 |
| $Cr^{6+}$(mg/L) | 0.015 ± 0.01 |
| $Cu^{2+}$(mg/L) | 0.027 ± 0.02 |
| $Fe^{2+}$(mg/L) | 1.0 ± 0.1 |
| $K^+$ (mg/L) | 481.7 ± 190.4 |
| Sr (mg/L) | 4.4 ± 1.2 |
| $Ba^{2+}$(mg/L) | 0.6 ± 0.2 |
| $Ni^{2+}$(mg/L) | 0.018 ± 0.015 |
| Zn(mg/L) | 1.74 |
| $Cd^{2+}$(mg/L) | <0.001 |
| $Hg^{2+}$(mg/L) | <0.01 |
| $Pb^{2+}$(mg/L) | <0.01 |

treatment of wastewater from the petrochemical industry [13,14] and oil-contaminated wastewaters [15–18]. The aim of this study was to investigate the operational stability of MBRs for the removal of organics under moderate salinity conditions from real oil and gas production wells. MBR was operated in two different sludge retention times (SRT) (30 days — during 230 days and infinite —during 50 days) and four different F/M ratios (0.45, 0.25, 0.55 and 0.30 gCOD/gMLVSS.day). COD, MLSS, MLVSS and TPH analyses were performed both in MBR system and the effluent in a regular basis.

## 2. Materials and methods

### 2.1. Oil and gas field PW

PW is a very complex mixture of dissolved and dispersed oil compounds, production chemical compounds, dissolved formation minerals, production solids (including bacteria, asphaltenes, waxes, formation solids, corrosion and scale products) and dissolved gases [19]. PW used in the study was taken from the TPAO (The Turkish Petroleum Company) Basin (Trakya, Turkey), was transported (2 h period) to the laboratory under refrigeration and stored at 4 °C before utilization. During the experimental study, several samples were supplied by TPAO and the inherent variability of the oil and gas field PW characterization is illustrated in Table 1.

### 2.2. Experimental set-up and membrane properties

The laboratory scale continuous-flow submerged MBR system was operated at room temperature for 297 days under two different SRTs of 30 days and infinite. Fig. 1 shows a simplified drawing of the experimental MBR with its major components. The effective volume of the MBR was about 5.1 L. Aeration was provided by diffusers and aeration rate was applied over 6–8 L/min in order to obtain a desired dissolved oxygen concentration. Dissolved oxygen concentration was continuously monitored in the aerobic tank using a Hach Lange dissolved oxygen meter. A Proportional Integral Derivative (PID) control of blower speed was provided by an Allen Bradley SLC 500 programmable controller (Milwaukee, WI). Produced water was fed to the MBR by a peristaltic pump (Watson Marlow 300 series) and the permeate was taken from the submerged membrane unit by a vacuum pump (Watson Marflow 300 series). MBR system was operated by full automated systems. Detailed information and short term operational results could be found in the study of Atay et al. [20]. The seed sludge used in the MBR system was obtained from a laboratory scale MBR treating leachate. The vertically mounted hollow fiber membranes used in the study were supplied from Zena Membranes and had a total area of 0.188 $m^2$ and nominal pore size of 0.1 μm. The hollow fiber membrane properties are given in Table 2. Membranes were cleaned by both physical backwashing and applying chemical agents including hydrochloric acid (HCl),

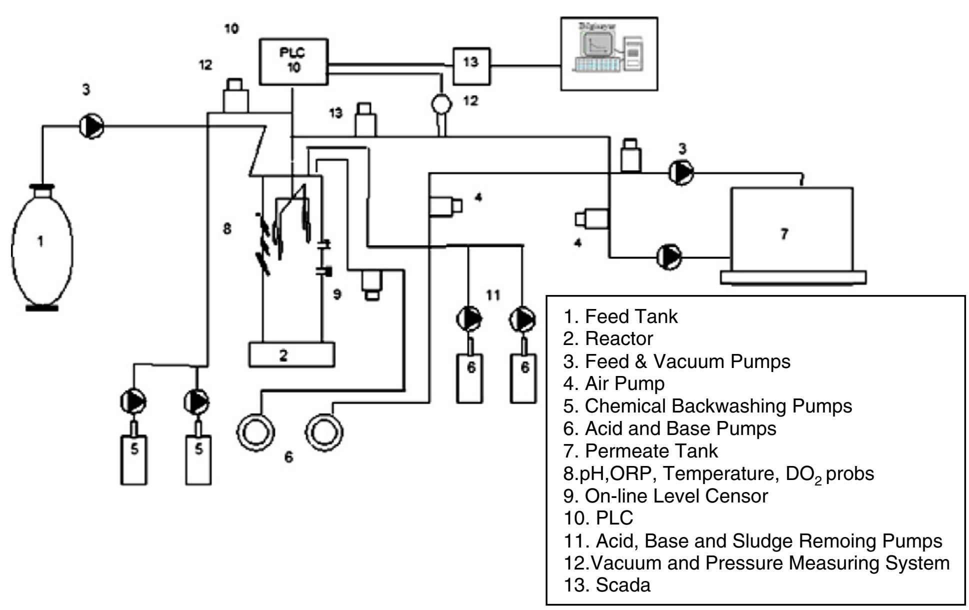


**Fig. 1.** Schematic diagram of the MBR system.

**Table 2**
Hollow fiber membrane properties.

| Properties | Unit | Nominal value | Minimum value | Maximum value |
|---|---|---|---|---|
| Inner diameter of fiber | mm | 0.24 | 0.22 | 0.26 |
| Outer diameter of fiber | mm | 0.31 | 0.29 | 0.32 |
| Wall thickness of fiber | mm | 0.03 | 0.03 | 0.04 |
| Length of fiber | cm | 150 | 145 | 155 |
| Size of fiber pores | µm | 0.1 × 0.7 | | 0.2 × 0.9 |
| Volumetric porosity of fiber | % | 50 | 43 | 57 |
| Flux of tap water | $L/m^2/h$ (1.0 bar. 25 °C) | 300 | 250 | 400 |
| Strength | N/fiber | 2 | 1.7 | |

sodium hydroxide (NaOH) and sodium hypochloride (NaOCl). Filtration lasted for 4 min and treated water was used for backwashing for 6 s.

### *2.3. Analysis methods*

Mixed liquor suspended solids (MLSS), mixed liquor volatile suspended solids (MLVSS), chemical oxygen demand (COD), total dissolved solids (TDS), ammonia, phosphorus, sulfide, total Kjeldahl nitrogen (TKN), ammonia and oil and grease parameters were measured according to the standard methods [21]. The turbidity was measured with a portable turbidity meter (Hanna 93703: Hanna HI, Portugal). All metals were analyzed by inductively coupled plasma-optical emission spectrometry (ICP-OES, Perkin-Elmer Optima 3300 DV, USA). All gas chromatography analyses were conducted using a Hewlett Packard 5890 Series II Plus Gas Chromatograph (Agilent Technologies Canada Inc., Mississauga, ON) equipped with an electron capture detector and a DB 5.625 capillary column 30 m × 0.25 mm × 0.25 µm (Agilent Technologies Canada Inc., Mississauga, ON). Ion chromatography (IC) was used for the analysis of nitrite ($NO_2^-$), nitrate ($NO_3^-$), chlorite ($ClO_2^-$), bromide ($Br^-$) and fluoride ($F^-$) following the method described in Standard Method 4110 B (APHA, 1998) and using a Dionex 500 Ion Chromatograph (Sunnyvale, CA) equipped with a Dionex ICS-3000 brand, ion chromatography. SEM analysis was performed at JEOL brand with JSM-840A model and EDX analysis was performed at XRF Systems brand with 500 Digital Processing model. Before the analysis, samples were coated with gold.

### *2.4. Activated sludge process assessment*

The sludge age ($\theta_c$) and the food to microorganism ratio (F/M) are two common parameters that can provide insight into the design and control of an activated sludge process [22]. A high mean cell residence time and a low F/M ratio will produce a lower sludge yield [23]. Mean cell residence time for the MBR system was calculated (Eq. (1)) based upon Stephenson et al. (2000) as follows:

$$\theta c = \frac{Vr.X}{Qw \cdot Xw + Qe \cdot Xe}$$

where $\theta_c$ is the sludge age or mean cell residence within the MBR system (days), $V_r$ the MBR system volume ($m^3$/day), X is the concentration of volatile suspended solids in the MBR system (mg/L), $Q_w$ is the waste sludge removed (kg/day), $X_w$ is the concentration of suspended solids in the waste sludge (mg/L), $Q_e$ is the permeate flowrate ($m^3$/day) and $X_e$ is the concentration of suspended solids in the permeate (mg/L).

The food to microorganism ratio was calculated according to Metcalf and Eddy [22] (Eq. (2)) as follows:

$$F:M = \frac{So}{\theta \cdot X}$$

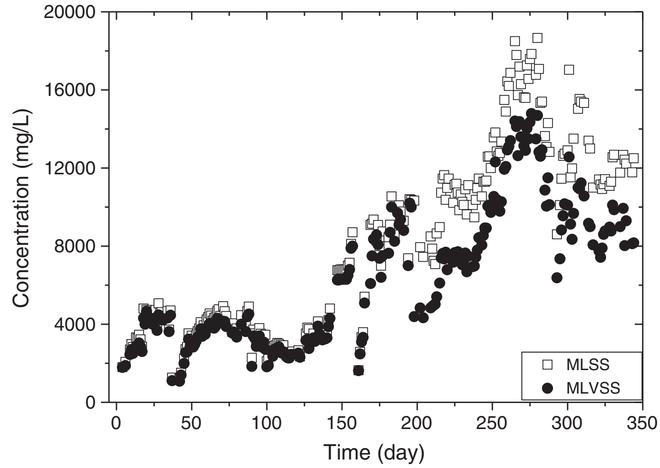


**Fig. 2.** Variation of MLSS and MLVSS throughout the experimental study.

where F/M is the food to microorganism ratio ($day^{-1}$), $S_o$ is the inlet $cBOD_5$ (mg/L), θ is the hydraulic detention time based on the MBR system volume = $V_r/Q_e$ (days), and X is the concentration of suspended solids in the MBR system (mg/L).

## **3. Results and discussion**

### *3.1. Solid concentrations in the MBR system*

Fig. 2 presents the MLSS and MLVSS concentrations in the MBR during the whole experimental period of 297 days. It can be seen that the MLSS concentration increases from 2000 mg/L low concentration to about 12,000 mg/L. During the first 160 days of operation, the increase in MLSS concentration was relatively constant, but after that, suddenly MLSS and MLVSS concentrations decreased suddenly. This might be attributed to the variations in the substrate quality in terms of $BOD_5$:N:P ratio, the suspended solid content in the inlet and also some operational problems with aeration system inside the bioreactor. Additionally, the accumulation of salinity might cause a harmful effect on microorganisms. The average MLVSS/MLSS ratio was 0.89 and 0.98 for the SRT values of 30 days and infinite, respectively. The suspended solid concentration (SS) of the permeate was also investigated during the operation and found to be less than 1 mg/L. No significant increase in the SS content of the permeate was observed throughout the operation of 297 days.

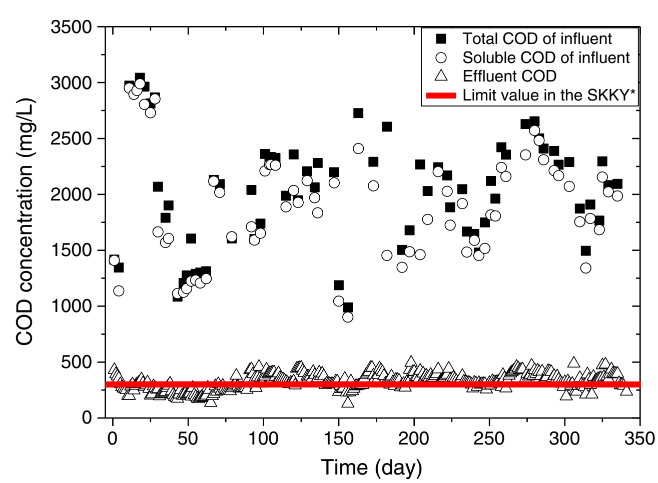


**Fig. 3.** Variation of COD in the influent and effluent of the system.

**Table 3**
Oil and grease concentrations in the influent and effluent of the system including the removal efficiencies.

| Day | Influent (mg/L) | Effluent (mg/L) | Removal efficiency (%) |
|---|---|---|---|
| 110 | 36 | 11 | 69 |
| 113 | 31 | 13 | 59 |
| 121 | 47 | 7 | 85 |
| 160 | 39 | 12 | 69 |
| 188 | 50 | 13 | 74 |
| 223 | 42 | 15 | 63 |
| 248 | 40 | 12 | 69 |

### 3.2. Performance of the MBR

Throughout the experimental study, different parameters representing the permeate quality such as oil and grease, COD and TPH were examined. The system showed a stable performance in spite of the variations in the feed for both SRTs.

**Table 4**
TPH concentrations in the influent and effluent of the system including the removal efficiencies.

| Day | Influent (ppm) | Effluent (ppm) | Removal efficiency(%) |
|---|---|---|---|
| 225 | 1030 | 185 | 82 |
| 233 | | <8 | 99 |
| 237 | | <8 | 99 |
| 245 | 2210 | <8 | 99 |

#### 3.2.1. COD removal

The membrane separation plays a significant role in maintaining stable COD removal in submerged MBRs. Moreover, MBR system acts as an additional purification phase to reject the remaining particulate COD and to produce an improved permeate quality. The variation of COD in the influent and effluent is shown in Fig. 3. It is apparent from Fig. 3 that despite the variations in the influent between 1500 and 3000 mg/L, the effluent quality is stabilized approximately below 500 mg/L. The system attained pseudo-steady-state within a short time. The pore size of the membrane which is lower than 0.10 µm

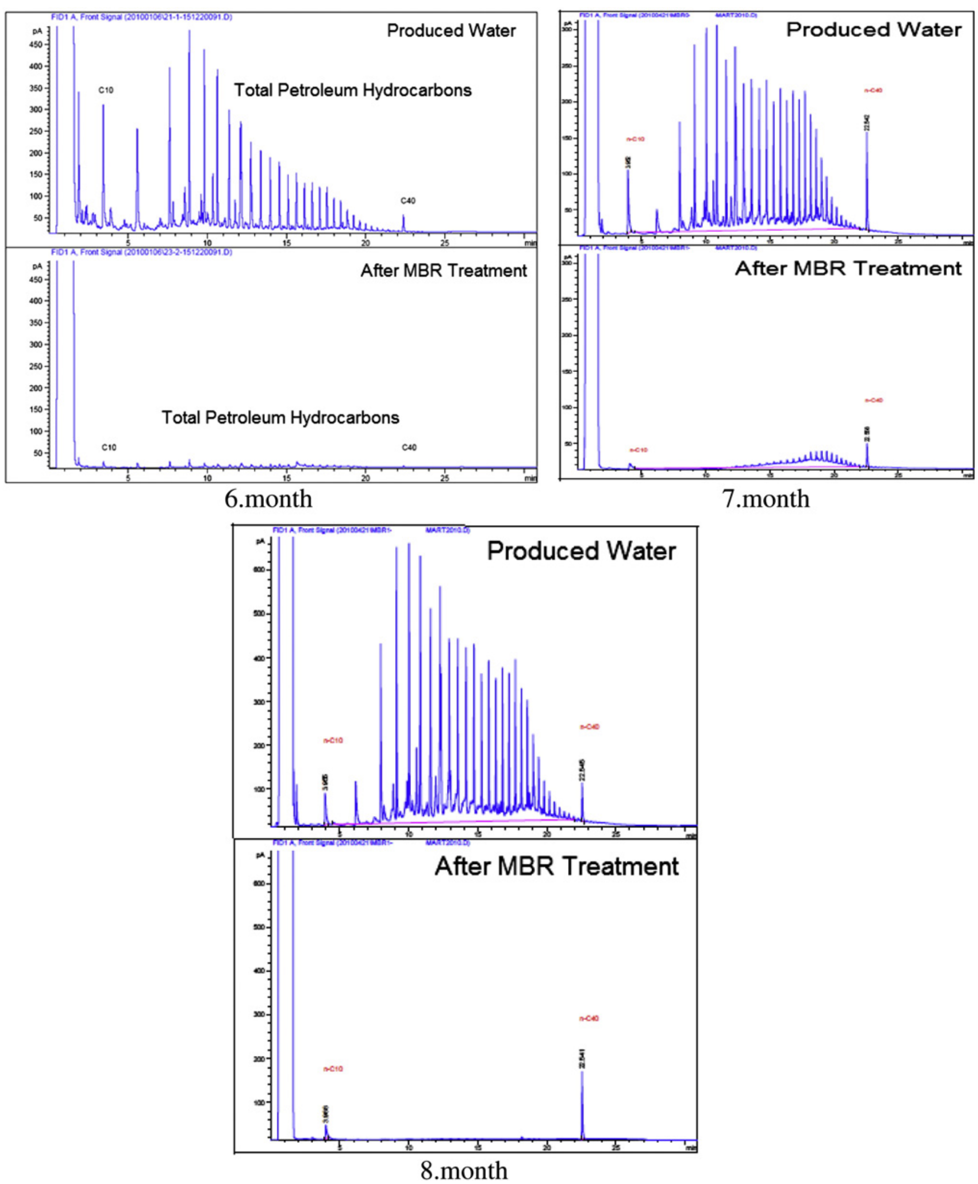


**Fig. 4.** Comparison of TPH values of influent and permeate of MBR treatment.

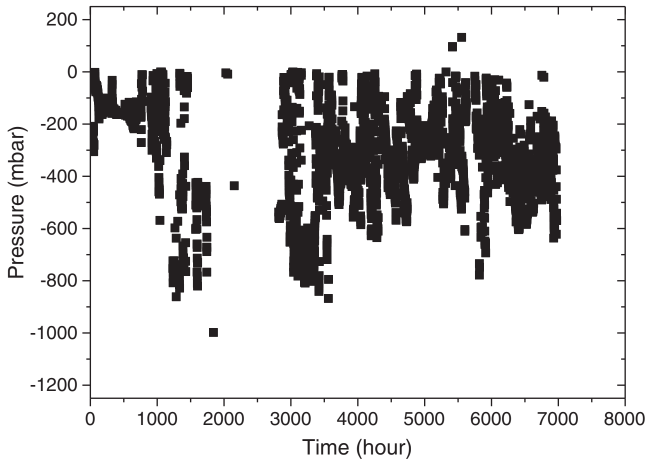


**Fig. 5.** Pressure values measured online in the MBR system.

resulted in the further removal of soluble contaminants. Consistent removal of soluble COD (sCOD) by the membrane was observed throughout the operational period at two sludge ages. Although COD removal in the bioreactor slightly increased with shortened SRT (30 days), the total COD removal efficiency of the MBR process could be kept over 80–85% independent from the SRT. COD removal rate slightly increased with SRT due to the higher concentration of biomass which may decompose organic compounds. It was seen that membrane separation played an important role in maintaining stable COD removal used in produced water treatment. Stable values of COD in the effluent seemed to be one of the major advantages of submerged MBR that made it a promising alternative to the conventional treatment process for the treatment of PW.

### *3.2.2. Oil and grease removal*

Characterization of PWs is needed for regulatory compliance and for the selection of suitable management/disposal options such as reuse and disposal. Oil and grease are the main constituents of produced water. Besides, salt content (known as salinity, conductivity, or TDS) is one of the other primary constituents of concern. Oil and grease is an important discharge contaminant as it results in a potentially toxic effect near the discharge point. Contamination and accumulation of oil on ocean sediments may occur if the dispersed oil contacts the ocean floor. Dispersed oils can also rise to the surface and spread, causing an increased biological oxygen demand near the mixing zone [24]. In this study more than 60% of the oil and grease was successfully retained in the MBR. Increase in the SRT

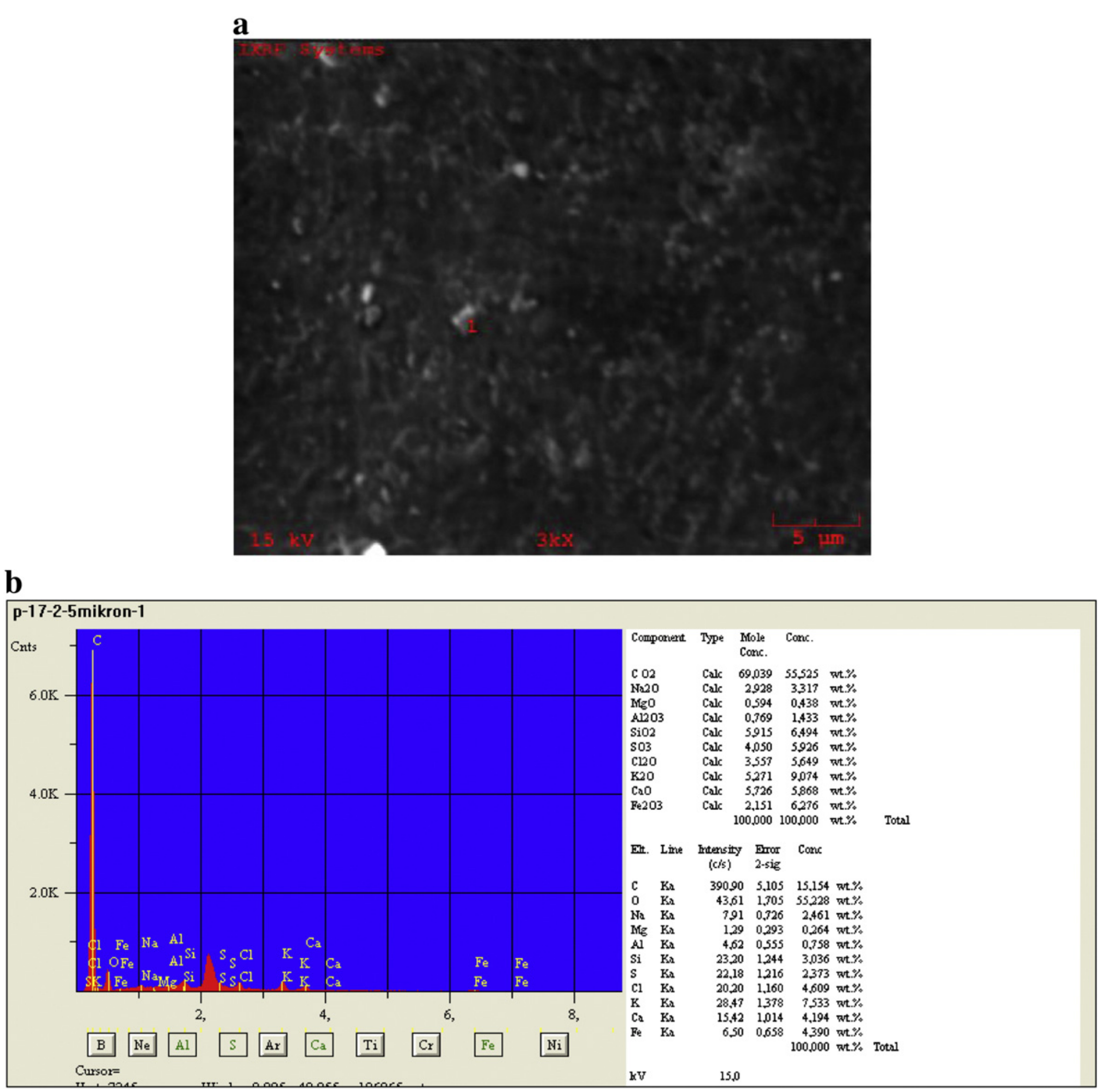


**Fig. 6.** EDX analysis of hollow fiber membrane ((a) scanning image; (b) spectrum graphics of observed particles).

from 30 days to infinite increased the removal efficiency of oil and grease marginally from 60% to 85% corresponding to the effluent concentrations of 15 mg/L and 12 mg/L, respectively. Data in Table 3 indicate that the increase in SRT resulted in an increase in the removal efficiency of the oil and grease.

#### 3.2.3. TPH removal

In general, petroleum hydrocarbons are divided into four groups: the saturates, the aromatics, the asphaltenes (phenols, ketones, esters, fatty acids, and porphyrins), and the resins (pyridines, quinolines, carbazoles, sulfoxides, and amides) [25]. The produced water used in this study is a complex mixture of hydrocarbons and various organic compounds (aromatics, phenols, and alcohols). Fig. 4 shows that alkanes are the most abundant constituents in the produced water.

Table 3 summarizes the quality of the permeate. Removal efficiencies of 97–99% for TPH were achieved in the MBR process. After startup, the inoculated oil-degrading microorganisms started to digest the crude oil and hydrocarbons. In this study, the removal of >99% hydrocarbons in the bioreactor sludge was observed.

Chromatographic profiles of initial hydrocarbons, as well as residual hydrocarbons in the MBR (Fig. 4 a, b) demonstrated that the MBR removed almost all of the light hydrocarbons from n-C9 to n-C13 after 6 months. In addition, an important reduction of hydrocarbons ranged between C13 and C40 was observed. The biodegradation efficiencies for TPH compounds were also calculated based on the residual masses found at the end of the inoculated assays (Table 4). The biodegradation efficiencies of the individual PAHs analyzed ranged between 82% and 99% (Table 4). This result showed that the well acclimated natural inoculum was active in degrading crude oil in moderate conditions [26].

### 3.3. Membrane performance

Membrane flux was maintained at 10 $L/m^2/h$ with a constant aeration intensity during the 297 days operation. Cleaning was conducted once when trans-membrane pressure (TMP) reached 800 mbar (Fig. 5). The corresponding permeability after physical cleaning was restored to 60% and to 95% after subsequent chemical cleaning. Therefore, the TMP increase was caused by both the physically reversible cake layer and irremovable (chemically reversible) fouling.

Element analysis was also performed on the hollow fiber membrane surface layer in order to identify the chemical components of the layer by EDX analysis. In the lab-scale MBR, membranes exhibited inhomogeneous fouling, depending on the position of fibers and module in the filtration line and on the age of the membrane. Fig. 6 shows an example of a hollow fiber membrane surface with obvious deposits that contain organic and inorganic foulants. The elements of C, O, Na, Mg, Al, Si, S, Cl, K, Fe, and Ca were detected. The C element came from organic matter in the mixed liquor adsorbed on membrane surface. The other elements were due to some inorganic precipitates originating from produced water.

## 4. Conclusions

After one year operation of lab-scale MBR system for the treatment of produced water, a stable performance is obtained in spite of the variations in the influent at different SRTs. Although the COD removal in the bioreactor slightly increased with shortened SRT, the total COD removal efficiency of the MBR process could be kept over 80–85% independent of SRT. The COD removal rate slightly increased with SRT due to the higher concentration of biomass which may decompose organic compounds. The increase of sludge age increased the removal efficiency of oil and grease dramatically from 60% to 85%. The hydrocarbons removal efficiency of 99% was achieved. The MBR removed almost all of the light hydrocarbons from n-C9 to n-C13 and an important reduction of hydrocarbons ranged between C13 and C40 was also observed. The corresponding permeability after physical cleaning was restored to 60% and to 95% after subsequent chemical cleaning.

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