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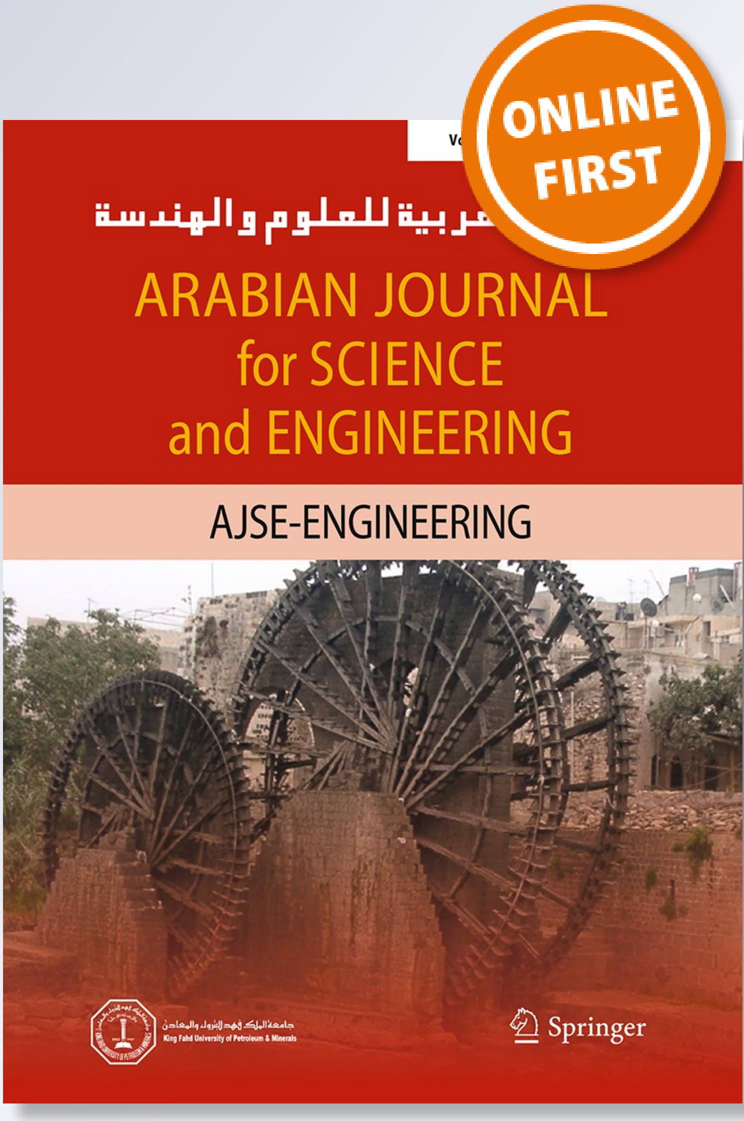

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RESEARCH ARTICLE - CHEMICAL ENGINEERING

Oil Refinery Wastewater Treatment by Using Membrane Bioreactor (MBR)

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Abstract In this work, a new hollow fiber membrane was fabricated for the treatment of industrial wastewater in Al-Daura refinery in Baghdad using the submerged membrane bioreactor (MBR) technique with a new membrane-type module. The hollow fiber characterization seems to be within the range of ultrafiltration membranes, and the fabricated hollow fibers have a skinless porous outer surface. The effects of mixed liquor suspended solids (MLSS) concentration (i.e., 500 and 1000 mg/l) and preheating temperatures of the effluent wastewater (i.e., 25, 45, and 55 °C) at different preheating periods (i.e., 15, 30, and 45 min) on the performance of MBR and removal efficiency of COD, BOD, oil content, phenol, and turbidity were investigated. It was found that the removal efficiency of COD, BOD, oil content, phenol, and turbidity were enhanced by increasing of MLSS concentration. This efficiency was significantly enlarged by applying different preheating temperatures and times. For example, at 45 min preheating time, the removal efficiency of COD, BOD, oil content, and phenol at 1000 mg/l, and 55 °C were measured to be 71, 60 and 100 %, respectively. In fact, the obtained results confirmed that the initial wastewater preheating followed by the submerged MBR can be offered a possibility of alternative process for oily wastewater treatment, especially when reuse of oily wastewater is taken into account.

Keywords Membrane bioreactor · Hollow fiber membrane · Ultrafiltration · Membrane module · MLSS

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1 Introduction

The use of both ultrafiltration (UF) and microfiltration (MF) membranes for separation are seen to be growing rapidly and it extends to a wide range of industrial processes, including biopharmaceutical and biochemical applications [1,2]. In such applications, the use of a membrane bioreactor (MBR) is a new and capable method of treating different wastewaters [3–29]. Examples of membrane bioreactor (MBR) applications are summarized in Table 1. Nowadays, the membrane bioreactor (MBR) is becoming an effective technology for oil refinery wastewater treatment, and there are also a very few employing on the treatment of oil refinery wastewater [30,31]. Veronese et al. [30] used chemical oxygen demand (COD) to evaluate the treatment of oil refinery wastewater by a biological process in combining with submerged UF membranes in order to measure the membrane performance for nine months of operation. The treatment of oil refinery wastewater using submerged membrane bioreactor was also realized by Viero et al. [31]. Their results had proved the potential of the MBR process in treating high-strength feed. Furthermore, Rahman and Al-Malack [32] investigated the use of a cross-flow membrane bioreactor in treating the discharged wastewater from a petroleum refinery. The performance of the MBR process was evaluated at MLSS concentrations of 3000 and 5000 mg/l. It was found that a COD removal efficiency of more than 93 % was obtained at both MLSS values. Scholz and Fuchs [33] had tested the treatment of oily wastewater by MBR process and reported that a high removal rate (about 99.99 %) was achieved for fuel oil and lubricating oil. The average removal efficiency of COD throughout the MBR operation was seen to be 94–96 and 97 % for fuel oil and lubricating oil, respectively.

There are several oil refineries in Iraq, and Al-Daura refinery is one of the biggest refineries, which is located at the

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Table 1 Examples of applications of membrane bioreactor (MBR) on a wide range of industrial processes

Process	Application	Refs.	Process	Application	Refs.
Hybrid membrane bioreactor (HMBR) (i.e., electrocoagulation, biological, and microfiltration processes)	Tannery wastewater	[3]	Anaerobic membrane bioreactor (AnMBR) equipped with submerged flat-sheet microfiltration membranes	Treatment of simulated and actual domestic wastewater (DWW)	[17]
Submerged membrane bioreactor (SMBR)	Municipal wastewater	[4]	Submerged anaerobic membrane bioreactors (SAnMBR)	Domestic wastewater treatment	[18]
Membrane bioreactor hybrid system (MBR-HS)	Municipal wastewater	[5]	Submerged membrane bioreactor (SMBR)	Abattoir wastewater (AW)	[19]
Anaerobic dynamic membrane bioreactor (AnDMBR)	Municipal wastewater	[6]	Electrocoagulation (EC) unit and a submerged membrane bioreactor (SMBR) technology	Grey water treatment	[20]
Membrane bioreactor (MBR)	Municipal wastewater	[7]	Submerged hollow fiber membrane bioreactor (MBR)	Treatment of brackish oil and natural gas field produced water	[21]
Membrane bioreactor using mesh filter for treating low-strength municipal wastewater	Municipal wastewater	[8]	Membrane bioreactor technology (MBR)+RO	Commercial laundry and in a textile factory	[22]
Membrane bioreactor (MBR)	Municipal wastewater	[9]	Submerged anaerobic membrane bioreactors (SAMBRs)	Decolorization of dyeing wastewater	[23]
Submerged membrane bioreactor	Municipal wastewater	[10]	Submerged membrane bioreactor (SMBR) with commercial membrane module	Treatment of model textile dye wastewater (MTDW)	[24]
Nanofiltration membrane bioreactor (NF-MBR) (Ceramic membrane)	Treatment of two pharmaceutical compounds (cyclophosphamide and ciprofloxacin)	[11]	Innovative membrane bioreactor (MBR) system	Removal of effective organic degradation and nutrient (N and P)	[25]
Membrane bioreactor (MBR)	Industrial (pharmaceutical and chemical) wastewater	[12]	Anaerobic membrane photo-bioreactor	Food industry wastewater	[26]
Integrated thermophilic submerged aerobic membrane bioreactor (TSAMBR) and electrochemical oxidation (EO) technology	Thermomechanical pulping pressure treatment	[13]	Granular activated carbon (GAC) and simultaneous application of powdered activated carbon (PAC) with membrane bioreactor (MBR)	Removal of 22 selected trace organic contaminants	[27]
Anaerobic membrane bioreactor (AnMBR) technology	Treatment of sewage sludge	[14]	Anaerobic membrane bioreactor (AnMBR)	Bamboo industry wastewater (BIWW)	[28]
Integrated vertical membrane bioreactor (IVMBR)	Removal of organic matter and nitrogen from synthetic wastewater or domestic sewage	[15]	Aerated and anoxic phases of a full-scale membrane bioreactor (MBR)	Urban wastewater	[29]
Membrane bioreactor system (MBR) with pretreatment of sponge tray bioreactor (STB)	Treatment of primary treated sewage (PTS)	[16]			

southwest of Baghdad on the left side of the Tigris River. However, the proposed MBR in this work may be utilized as an effective wastewater treatment method to avoid the problem of environment pollution, and treated a huge oily wastewater in order to reach the global standards in the environmental regulations.

It can be seen, according to the above-mentioned literature, that the effect of optimal concentration of MLSS and some operating conditions related to the performance of treated oily wastewater by means of MBR process have not been identified yet. Moreover, the details about the characteristics of biomass and their effects on the MBR performance

Table 2 Characteristics of inlet water to the wastewater treatment

Contaminants	COD (ppm)	BOD (ppm)	Oil content (ppm)	Phenol (ppm)	pH	TDS (ppm)
Range	100–1000	24–300	25–1000	1–4	7.3–9	900–2500

Table 3 Characteristics of inlet water to the experimental operation

Contaminants	COD (ppm)	BOD (ppm)	Oil content (ppm)	Phenol (ppm)	pH	TDS (ppm)	Conductivity (μs)
Value	235	46	14	0.7	7.2	1111	1192

have not been extensively mentioned in the literature. Therefore, this work focuses on the impact of MLSS contents and biomass characteristics on the MBR performance. Furthermore, most of the membranes used for such process are commercial and ceramic membranes, whereas in this study a new PVC hollow fiber membrane with porous structure of outer surface was employed. PVC hollow fiber membrane is preferred for the MBR application and was prepared particularly for the purpose of oily wastewater treatment using membrane bioreactor process, and a new membrane module was also applied. The reason behind the using of PVC as a membrane material can be related to the PVC polymer as it is classified as a hydrophobic polymer, which means high contact angle value with water. Thus, this polymer is typically used for the purposes of backwash due to the lower resistance that is provided by membrane surface tension when the hollow fiber membrane brought into contact with water. The aim of this research was to evaluate the separation performance of an immersed PVC hollow fiber membrane bioreactor as a result of using a laboratory-scale experimental unit, which is designed for treating the obtained oil refinery wastewater from Al-Daura refinery with the aim of accomplishment the Iraqi national regulations for the disposal of sewage in rivers. As previously mentioned, the evaluation was carried out at two different MLSS concentrations under various feed temperatures. In addition, the effect of preheating times at different feed temperatures was also investigated. Consequently, a significant objective of this work is also to establish a sufficiently low concentration of MLSS that may have affected the removal efficiency of contaminated compounds in the oily wastewater.

2 Experimental Work

2.1 Oil Refinery Wastewaters

Oily wastewater used in the experiments was brought from Al-Daura refinery in Baghdad. The characteristics of influent wastewater for both a gravity separator tank (API) and a conventional activated sludge tank (CAS) are illustrated in Tables 2 and 3. The oily wastewater treatment in Al-Daura refinery includes many processes such as API separators to the removal of free oil, dissolved air floatation (DAF) for elimination of the emulsified oil from contaminated wastewater, and a biological process to take the dissolved hydrocarbons away by means of conventional activated sludge. The wastewater is finally moved to the sedimentation tank in order to separate the suspended biomass from the effluent water as shown in Fig. 1. The present study is concerned with the treatment of wastewater from the DAF by using MBR instead of CAS or sedimentation route for comparison of the characteristics of water in this method with the ordinary methods. The acceptable limits of all of the contaminants in the discharged water to the river according to the Iraqi Law No. 25 in 2006 are listed in Table 4.

2.2 Experimental Operation

Figure 2 shows a schematic diagram of the membrane bioreactor system. The working volume of the reactor is composed of 18 L of the oily wastewater and 2 L of the activated sludge. The total dimension of the glass reactor is $30 \times 30 \times 50\ \text{cm}^3$. The specifications of the activated sludge employed in the treatment process are shown in Table 5. Hollow fiber membranes were initially placed in a double-module configuration, which is connected with a gas–liquid separator. The allowable vacuum pressure of about −450 mmHg was utilized in the filtration process of oily wastewater. Stone diffusers were immersed in the reactor and also connected to air compressor to facilitate aerate the biomass and sweep the surface of membrane. The process was stopped at each 20 min in order to carry out a physical cleaning for the membrane module via a pressurized tap water under 100 mmHg. The oily wastewater treatment using MBR process was performed under various parameters including two different MLSS concentrations of 500 and 1000 mg/l (same MLSS concentrations as they have already been used in conventional processes for oily wastewater treatment inside AL-Daura refinery) and two different initial preheating temperatures of wastewater (i.e., 45, 55 °C) for a variant periods of time (i.e., 15, 30 and 45 min). The wastewater was preheated to the sug-

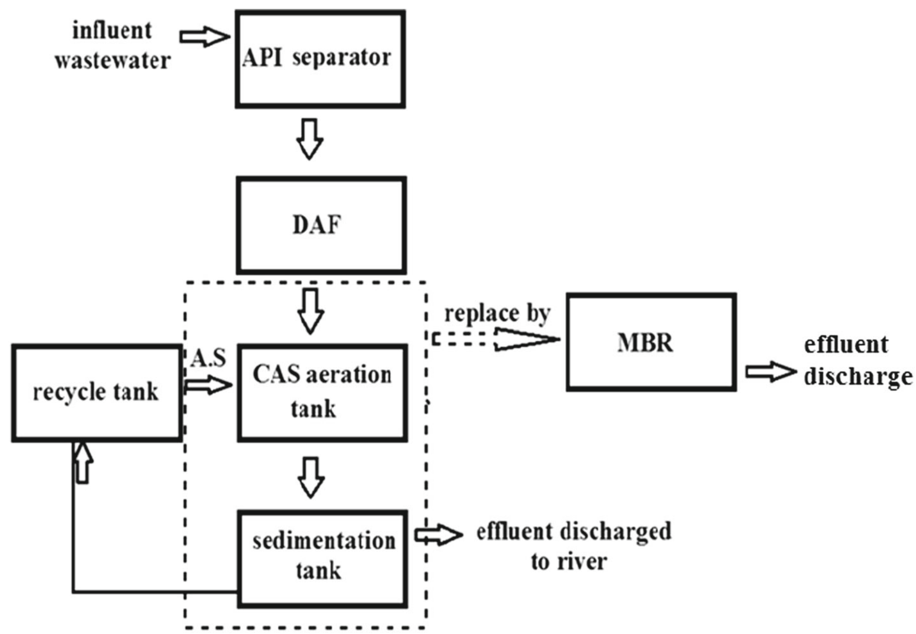

Fig. 1 Process diagram of oily wastewater treatment in AL-Daura refinery

Table 4 Specifications of influent, effluent water of MBR and the allowable limits in Iraq for river discharged water

Contaminants	Influent	Effluent	Allowable limits
pH			6.5–8.5
COD (ppm)	235	67	100
BOD (ppm)	46	18	40
Oil (ppm)	14	0	10
Phenol (ppm)	0.7	0	0.05
Turbidity (NTU)	21	0.12	5

gested temperatures and subsequently cooled to 25 °C before adding the MLSS. With the aim of increasing the activity of operation, a nutrient solution containing phosphoric acid and urea with ratio 1:5 have been regularly inserted to the bioreactor tank.

2.3 Membrane and Membrane Module

New poly vinyl chloride (PVC) hollow fiber membranes were prepared by dry/wet spinning using the phase inversion method for MBR application. The homogenous dope solution is composed of 16 wt% PVC and 84 wt% DMAC solvent. The dope solution is pressurized from the feed column to the spinneret at nitrogen extrusion pressure of 1.75 bar and then brought into contact with the external coagulation water bath after 3 cm air gap length. The temperature of the external coagulation water is 20 °C. During the flow of the dope solution around the outer surface of the tube inside the spinneret, the bore fluid or internal coagulant was allowed to flow by 3.6 ml/min in the lumen side of the tube inside the spinneret. The internal coagulant is distilled water at 20 °C. The outer and inner diameters of the spinneret are 0.9 and 0.5 mm, respectively. All details about the production method have been illustrated elsewhere [34].

In this work, the proposed submerged membrane module was made by PVC pipes joined with PVC fittings (elbow 90 and diffusers) along with holes on the surface of the pipes as demonstrated in Fig. 3a. The bundle of the hollow fiber membranes consists of 21 fibers with 30 cm in length for each fiber and also connected to the ends of the pipes by epoxy resin as shown in Fig. 3b. The submerged membrane module is carefully prepared to protect the hollow fiber membranes from torsion and pleating during the separation process.

2.4 SEM Measurements

Cross section and outer surface of the PVC hollow fiber membranes were tested by using SEM analysis. The dry hollow fiber was immersed in a liquid nitrogen to fracture the membrane and keep the cross-sectional area without deformation. The hollow fiber was then coated with a thin gold layer using a sputter apparatus before observing them with SEM (TESCAN VEGA3 SB instrument (EO Elektronen Optik-Service GmbH, Germany)).

2.5 AFM Measurements

The atomic force microscopy (AFM), (Angstrom Advanced Inc. (USA), model AA3000) in contact mode with a suitable silicon tip, has been used to study the outer surfaces of the PVC hollow fibers. The surface of hollow fiber membranes has been tested in terms of the mean pore size and mean

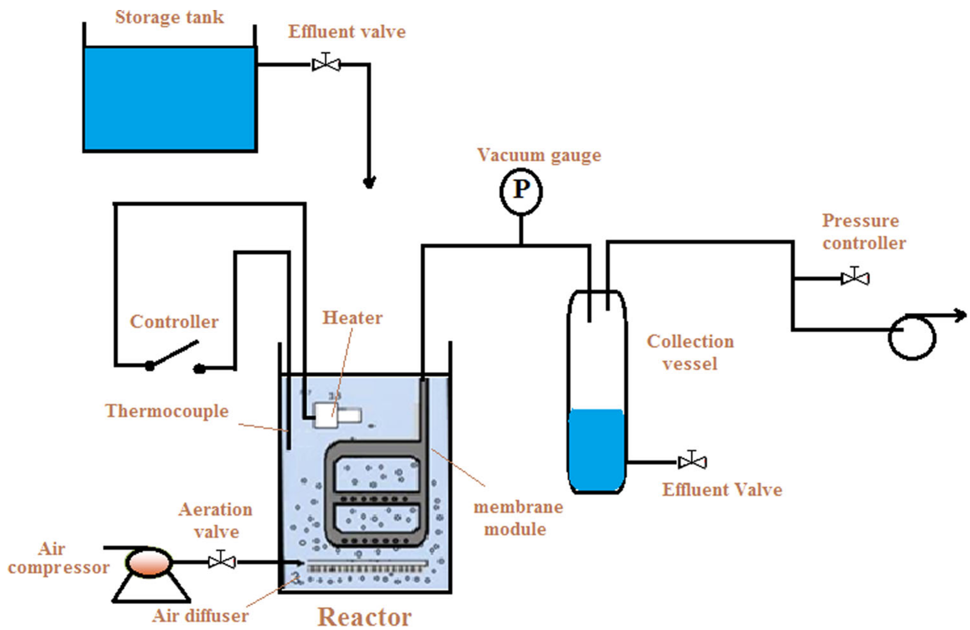

Fig. 2 Schematic diagram of the MBR process system

Table 5 Characteristics of the incoming activated sludge

Contaminants	COD (ppm)	Non-volatile suspended solids (NVSS)	Volatile suspended solids (VSS) (ppm)	pH	TSS (ppm)
Range	3–4	0.15	0.1	7.5	0.2

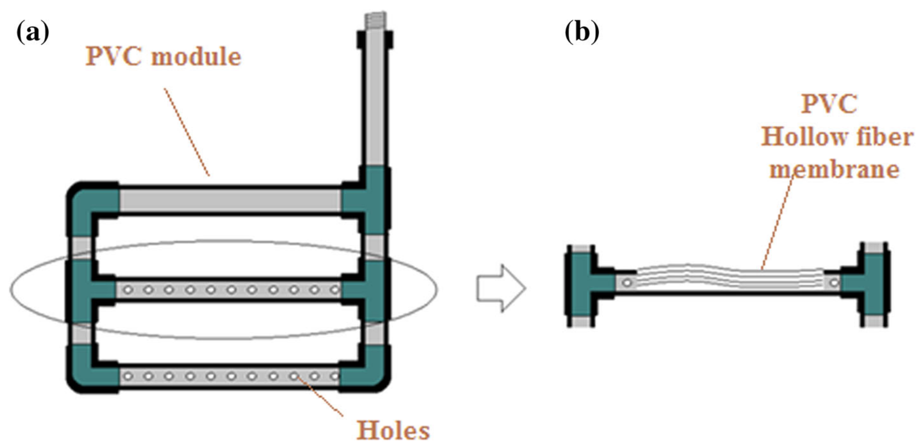

Fig. 3 Schematic diagram of PVC hollow fiber membrane module; *a* membrane module, *b* hollow fiber bundles connected with the module pipe

roughness parameter (Ra) with scan size of (2 µm × 2 µm). By using IMAGER 4.31 software, both the statistical pore size and the distribution were evaluated for the outer surface of PVC hollow fiber.

2.6 Analytical Methods

The chemical oxygen demand (COD), BOD, oil content, phenol, and MLSS of the wastewater were measured in proportion to the described procedures in the standard methods (American Public Health Association (APHA)): methods 5220, 5210, 5520, 5530, and 2540, respectively [35]. Moreover, the turbidity of the wastewater was measured using a turbidity meter (WTW TURB 550, Inolab).

3 Results and Discussion

3.1 Hollow Fiber Characterization

The structural morphology of PVC hollow fiber cross section and outer surface can be examined via scanning electron

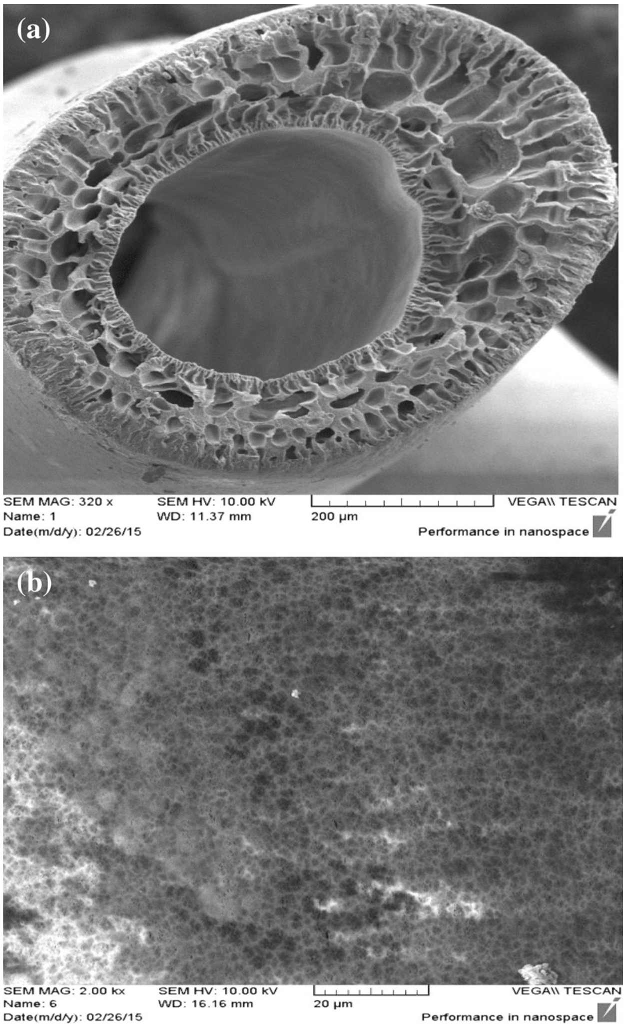

Fig. 4 SEM images of the PVC hollow fibers; **a** cross section; **b** outer surface

micrograph (SEM) and atomic force microscope (AFM). Figure 4a clearly presents the interior core of the PVC hollow fiber membrane. The cross section of the hollow fiber composed of three layers: two finger-like structure located near the inner and outer surface, whereas ellipsoid-like structure located in the middle of the fiber cross section. SEM image of the outer surface of the PVC fiber given in Fig. 4b shows the skinless porous surface, which is preferred for the MBR application due to the high efficiency for the removal of suspended materials along with moderate effect on the membrane permeability. Figure 5 shows the three dimensions (3D) and top-view AFM pictures of the outer surface of the PVC fiber over an area of $2 \times 2\,\mu m^2$. Pore size and pore size distribution were estimated through image analysis of the membranes from AFM data. All the specifications of the PVC hollow fiber membrane are shown in Fig. 4 and Table 6.

3.2 Development of Biomass Growth

Figure 6 shows the effect of MLSS concentration (i.e., 500 and 1000 mg/l) on the biomass growth within 5 days of the process at a temperature of 25 °C and pH = 7.2. It can be noticed that the biomass growth increases with an increase in the MLSS concentration and the time. The growth of biomass in activated sludge throughout the treatment process is due to its nourishment with the organic compounds and nutrient (i.e., urea, phosphorus). This is one of the important factors for increasing the speed and efficiency of the removal of oil and phenol content in wastewater. Rosenberger et al. had reported [36] that the biomass growth seems to be increased with the introduction of microorganisms with the wastewater as the reactor tank had not been vaccinated.

3.3 Effect of MLSS and Preheating Temperatures on COD Removal

The effect of MLSS concentrations (i.e., 500 and 1000 mg/l) on either the COD removal within 5 days of the process at a temperature of 25 °C or trans-membrane pressure (TMP) of about −450 mmHg is shown in Fig. 7. It can be noticed that the COD concentration decreased from 235 to 157 mg/l over 5 days by using 500 mg/l MLSS, whereas a 1000 mg/l MLSS concentration resulted in decreasing in the COD concentration from 235 to 122 mg/l (i.e., 48 % COD removal efficiency). It is worth noting that the increasing of MLSS concentration can be led to an increase in the COD removals. This phenomenon is the result of greater decomposition of hydrocarbons into organic components as well as increasing the nutrition in the sludge within increase in MLSS concentration, which in turn resulted in an increase in the COD removal. Al-Malack [37] demonstrated that the COD removal efficiency from synthetic municipal wastewater is increased due to the increase in MLSS concentration from 3000 to 15,000 mg/l during the realization process. Also, the removal efficiency of COD is improved progressively with time due to the existence of phenol in the oily wastewater as reported by Al-Malack [37]. In fact, as the operation period passes, the system tends to adapt to the phenol, thus its performance improves. It is expected that this effect shall be decreased by either decreasing the content of phenol or increasing the MLSS composition.

Moreover, Rahman and Al-Malack [32] found that the results of the realization of the treated wastewater discharged by a petroleum refinery showed a COD removal efficiency of more than 93 % was obtained at both 3000 and 5000 mg/l MLSS concentrations.

In addition, Fig. 7 shows the effect of preheating times (i.e., 15, 30 and 45 min) at a temperature of 45 °C on the removal of COD from wastewater with 1000 mg/l MLSS concentration. Within 15 min preheating time, the COD con-

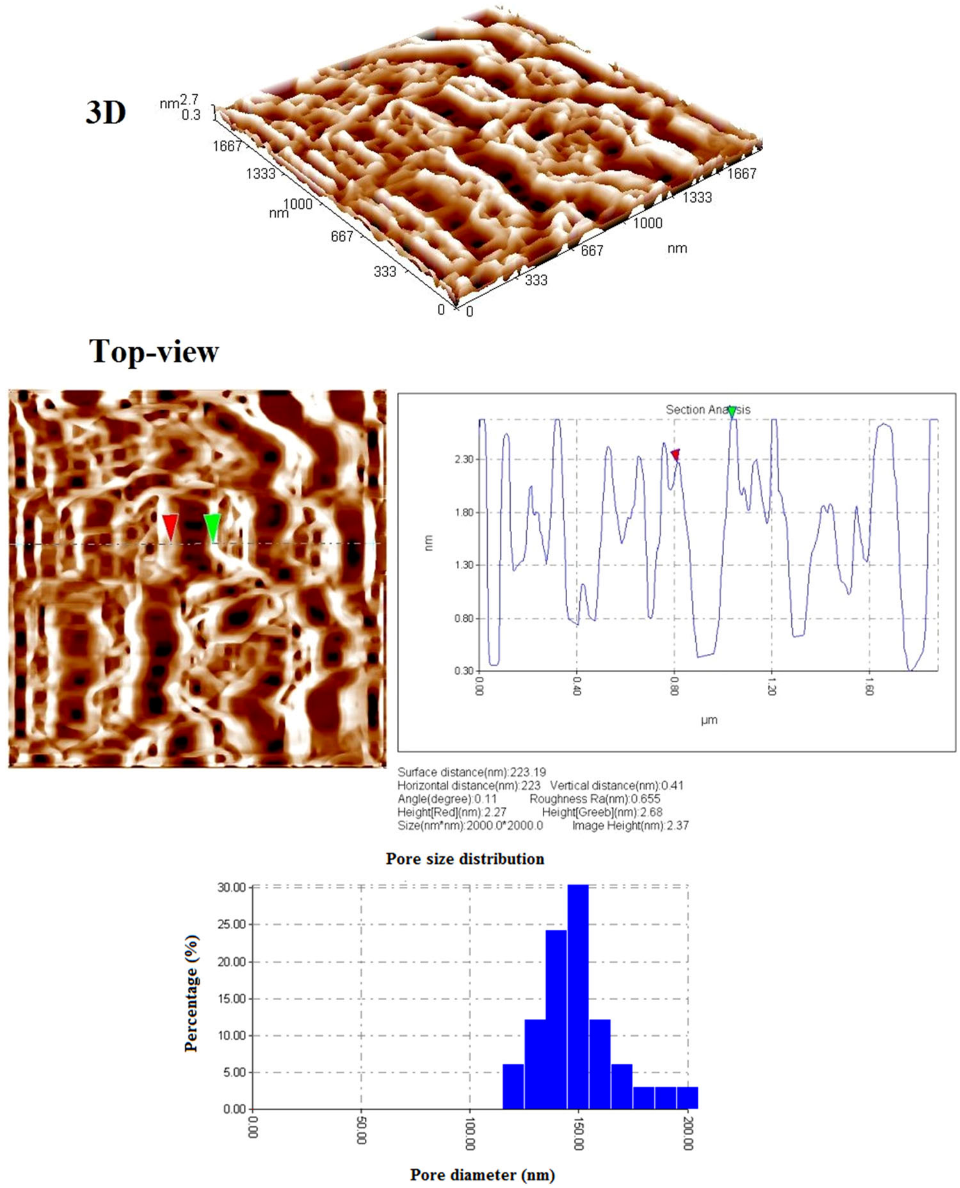

Fig. 5 Three dimensions and top-view AFM images with pore size distribution of the PVC hollow fiber membrane

Table 6 Properties of the prepared hollow fiber membrane

Membrane characteristics	Outer dia.	Inner dia.	Porosity	Roughness average	Mean pore size	Length of fiber	Surface area
Value	1.2 mm	1.0 mm	77 %	0.602 nm	120 nm	30 cm	11.3 cm^2

centration was seen to be decreased from 235 mg/l at the first day to 117 mg/l at the fifth day with 50 % removal efficiency. Meanwhile, by using 30 and 45 min preheating times, the COD concentration was reduced from 235 mg/l at the first day to 97 and 85 mg/l at the fifth with 58 and 64 % removal efficiencies, respectively. COD removal was drastically increased with preheating temperature of about 55 °C by using 15, 30, and 45 min and as shown in Fig. 8. The COD concentration was also decreased from 235 mg/l at the first day to 111, 86, and 67 mg/l at the fifth day with 52, 63, and 71 % removal efficiencies for 15, 30, and 45 min, respectively.

It is worth mentioning here that the removal efficiency is significantly affected by preheating time. This phenomenon can be attributed to the degradation of the hydrocarbon molecules as a result of the preheating temperature, and further increasing in preheating temperature results in an increase in the degradation. Increasing the preheating temperature

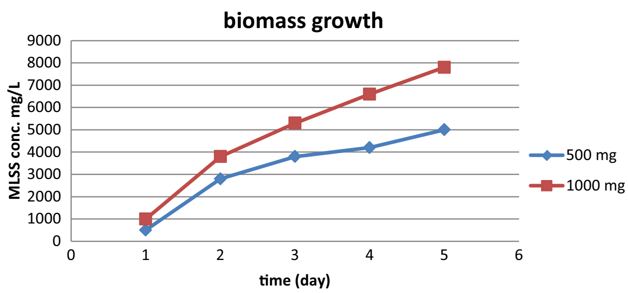

Fig. 6 MLSS growth in the reactor at initial MLSS concentration of 500 and 1000 mg/l, temperature of 25 °C, pH = 7.2, and HRT of 5 days

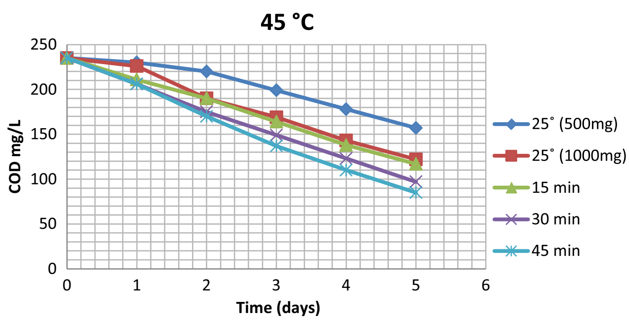

Fig. 7 COD removal with different preheating periods, temperature of 45 °C, initial MLSS concentration of 1000 mg/l, initial COD = 235 mg/l, TMP = −450 mmHg and HRT = 5 days

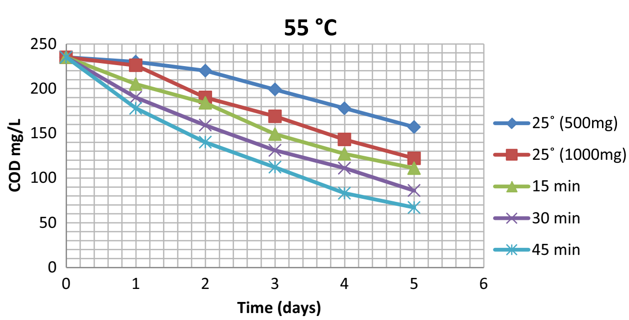

Fig. 8 COD removal with different preheating periods, temperature of 55 °C, initial MLSS concentration of 1000 mg/l, initial COD = 235 mg/l, TMP = −450 mmHg and HRT = 5 days

and time also can lead to increase in the diffusion of the organic compounds from the surface of the wastewater into the atmosphere. This result agrees with that reported by Al-Malack and Chu and Li [37,38]. The specifications of the treated wastewater after 45 min and 55 °C initial preheating is within the allowable limits for COD of discharged water in Iraq as confirmed by Table 4.

3.4 Effect of MLSS and Preheating Temperatures on BOD Removal

Figure 9 shows the effect of MLSS concentrations on BOD removal after 5 days of the process at a temperature of 25 °C, TMP of −450 mmHg and with an initial BOD concentration of 46 mg/l. By using 500 mg/l of MLSS concentration, the BOD concentration is decreased from 46 to 31 mg/l during 5 days of the process (32 % removal efficiency), as shown in Fig. 9. Using MLSS concentration of 1000 mg/l leads to a decrease in the BOD concentration from 46 to 28 mg/l (BOD removal efficiency of about 40 %). This result means that the membrane system has a moderate success in removing of BOD. Moreover, Fig. 9 shows the removal of BOD from wastewater with the preheating temperature of 45 °C at different preheating times (i.e., 15, 30 and 45 min) with 1000 mg/l initial MLSS concentration. Within 15 min preheating time, BOD concentration seems to be decreased from 46 mg/l at the first day to 27 mg/l at the fifth day (41 % removal efficiency). While using 30 and 45 min preheating times, the BOD concentration is decreased from 46 to 23 and 21 mg/l during 5 days (50 and 54 % removal efficiencies), respectively. BOD removal is drastically increased with the preheating temperature of 55 °C within 15, 30, and 45 min, as demonstrated in Fig. 10. The BOD concentration decreased from 46 to 26, 21, and 18 mg/l with 43, 54, and 60 % removal efficiencies within preheating times of 15, 30, and 45 min, respectively. This phenomenon may be attributed to the increase in biodegradation of the organic compounds because of increasing the biomass (MLSS). Noor et al. [39] had reported that to ensure all the organics are completely biodegraded, high concentration of biomass (MLSS) needs to used. Furthermore, in order to obtain the optimal performance in treating high-strength wastewater, the concentration of MLSS should be high in order to enhance the biodegradation process of the organic compounds, but should be taken into consideration that the increase in MLSS may accelerate the fouling on the membrane owing to the high suspended solid [40]. Besides, the increase in BOD removal efficiency with a two different preheating temperatures and times is attributable to the enhancement of the solubility of organic compounds, which is usually executed within a wide range of temperatures (i.e., 40–180 °C). However, the volatile organic compounds in oily

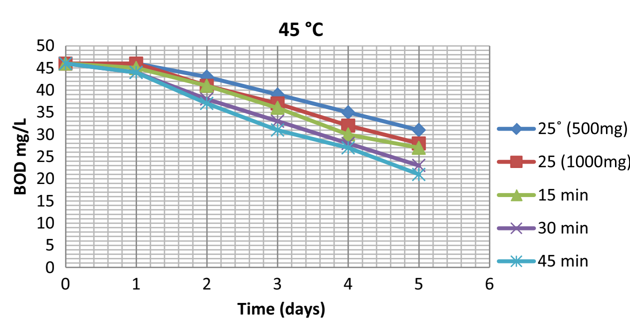

Fig. 9 BOD removal with different preheating periods, temperature of 45 °C, initial MLSS concentration of 1000 mg/l, initial BOD = 46 mg/l, TMP = −450 mmHg and HRT of 5 days

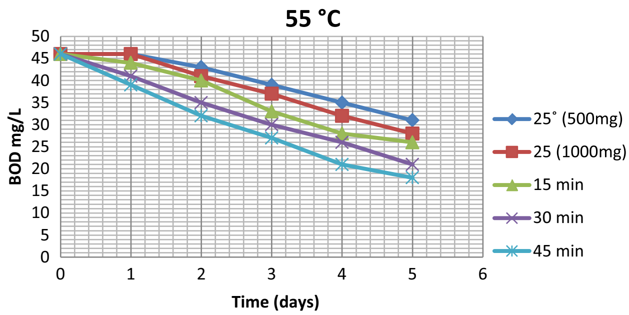

Fig. 10 BOD removal with different preheating periods, temperature of 55 °C, initial MLSS concentration of 1000 mg/l, initial BOD = 46 mg/l, TMP = −450 mmHg and HRT of 5 days

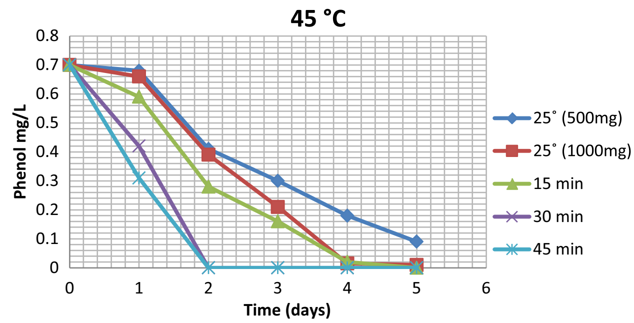

Fig. 11 Phenol removal with different preheating periods, temperature of 45 °C, initial MLSS concentration of 1000 mg/l, initial phenol concentration = 0.7 mg/l, TMP = −450 mmHg and HRT of 5 days

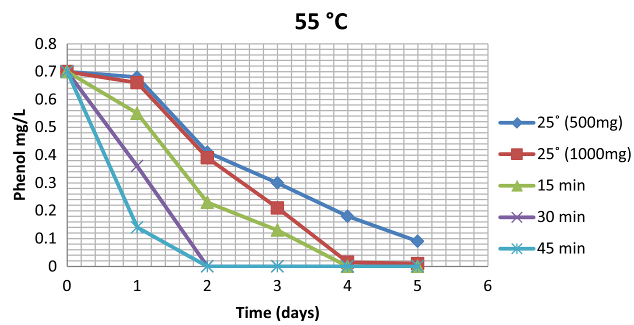

Fig. 12 Phenol removal with different preheating periods, temperature of 55 °C, initial MLSS concentration of 1000 mg/l, initial phenol concentration = 0.7 mg/l, TMP = −450 mmHg and HRT of 5 days

wastewater are evaporated at high temperatures, which is in turn results to enhance of the BOD removal efficiency.

Finally, it can evidently be said that water out the MBR at different preheating periods is within the condition limits for BOD of discharged water in Iraq as shown in Table 4.

3.5 Effect of MLSS and Preheating Temperatures on Phenol Removal

It is well known that the major sources of phenolic compounds in oil refinery are crude oil distillation and the finishing product. Therefore, these compounds must be removed from the wastewater prior to discharge. The concentration of phenol in contaminated wastewater in Al-Daura refinery is typically about 0.7 mg/l, and the toxic effect of phenol on the environment gives important to remove it before the wastewater has been discharged.

Figure 11 shows the effect of MLSS concentrations (i.e., 500 and 1000 mg/l) on phenol removal efficiency with a process of investigation time of 5 days at 25 °C, TMP −450 mmHg. The initial phenol concentration in wastewater was found to be 0.7 mg/l, and by using 500 mg/l MLSS, the phenol concentration was seen to be reduced from 0.7 to 0.09 mg/l over 5 days. Additionally, by using 1000 mg/l MLSS, the results showed an excellent removal efficiency for phenol (as a result of reducing the phenol concentration from 0.7 to 0.01 mg/l with 98 % removal efficiency). In Sect. 3.1, it has been mentioned that the increase in MLSS concentration led to an acceleration of biomass growth, where the biomass is acclimatized to phenol with time, and in sequence it results in the increasing of the removal of phenol. Yamagishi et al. [41] inspected phenol removal using cross-flow membrane filtration and had found that at an MLSS concentration of 8000 mg/l, 99.1 % of phenol was removed. Al-Malack [37] had also found that at an MLSS concentration of 15,000 mg/l, the phenol removal efficiency was arrived at 70 %. It is worth mentioning that the MLSS concentration has a significant effect on the removal of phenol from wastewater, and the phenol removal efficiency present in this process is very convincing with reference to that present in the literature [37,41]. These results support the hypothesis that a relatively low concentration of MLSS has a strong effect on the removal efficiency of the compounds in oily wastewater.

Figure 11 shows the removal of phenol from wastewater with a preheating temperature of 45 °C at different preheating times such as 15, 30 and 45 min using a 1000 mg/l MLSS concentration. It can be seen within the preheating time of 15 min that the phenol concentration decreased from 0.7 mg/l at the first day to 0 mg/l at the fifth day, meaning that the removal efficiency of the phenol has reached 100 %. With a further increase in the preheating times (up to 30 and 45 min), the phenol concentration is decreased from 0.7 to 0 mg/l at the second day (100 % phenol removal efficiency). Likewise, increasing the preheating temperature to 55 °C resulted in a rapid reduction in the phenol concentration within 15, 30, and 45 min preheating times, as presented in Fig. 12. It can also be observed that the phenol concentration is decreased from 0.7 to 0 mg/l over 5 days using a 15 min preheating time, whereas with 30 and 40 min preheating times the removal efficiency of phenol is found to be 100 % during the first 2 days. This is attributed to the conversion of insoluble phenolic compounds in water to soluble compounds,

which facilitates the biodegradation process. This results are in agreement with that reported by Barrios-Martinez et al. [42], and the final concentration of phenol in the permeate stream for the suggested procedure was within the specification limits for phenol of discharged water in Iraq as shown in Table 4.

3.6 Effect of MLSS and Preheating Temperatures on Oil Removal

The concentration of oil in contaminated wastewater from Al-Daura refinery is usually about 14 mg/l. The removal of oil compounds from the wastewater is therefore studied in the present work. Figure 13 shows the effect of MLSS concentrations (i.e., 500 and 1000 mg/l) on oil removal from the wastewater at 25 °C and TMP of −450 mmHg. It can be noticed that the concentration of the oil is decreased from 14 to 2.3 mg/l by using 500 mg/l MLSS concentration during 3 days of the hydraulic retention time. For further increase in MLSS concentration up to 1000 mg/l, the oil concentration seems to be decreased from 14 to 1.3 mg/l (90.7 % oil removal efficiency). This phenomenon is descriptive of greater decomposition of hydrocarbons to organic components with an increase in MLSS concentration and also improved nutrition by sludge, which consecutively led to superior removal of oil. Hence, it is worth noting that a relatively low MLSS concentration such as 500 or 1000 mg/l is sufficient enough to obtain a significant impact on the degradation of oily compounds in wastewater.

Figure 13 shows the removal of oil from wastewater with a preheating temperature of 45 °C at different preheating times (i.e., 15, 30, and 45 min) using 1000 mg/l MLSS concentration. It can be seen that within 15 min preheating time, oil concentration is decreased from 14 mg/l at the first day to 0.72 mg/l at the third with 95 % removal efficiency. While using 30 and 45 min preheating times, oil concentration is reduced from 14 mg/l at the first day to 0 mg/l within 3 days of the hydraulic retention time with a removal efficiency

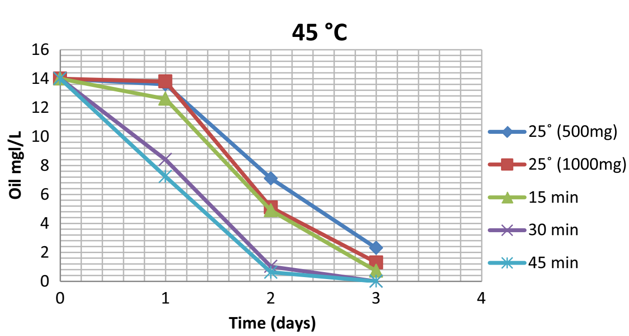

Fig. 13 Removal of oil at different preheating periods, temperature of 45 °C, initial MLSS concentration of 1000 mg/l, initial oil concentration = 14 mg/l and TMP = −450 mmHg

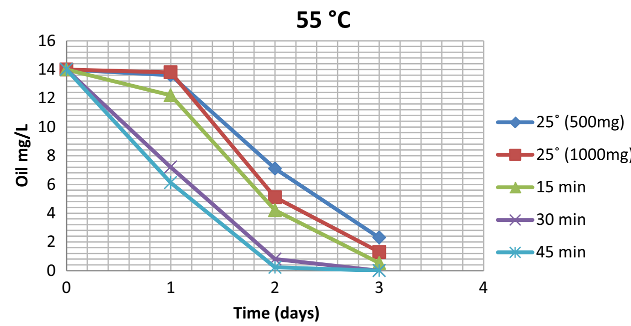

Fig. 14 Removal of oil at different preheating periods, temperature of 55 °C, initial MLSS concentration of 1000 mg/l, initial oil concentration = 14 mg/l and TMP = −450 mmHg

of 100 %. Moreover, increasing the preheating temperature to 55 °C leads to increase in the oil removal rapidly within the preheating times of 15, 30, and 45 min, as illustrated in Fig. 14. The oil concentration is also decreased from 14 mg/l within 3 days to 0.53 at 15 min preheating time, but the oil concentration is decreased to 0 mg/l within 3 days for both 30 and 45 min preheating times, and the removal efficiency was recorded to be 100 %.

There are several environmental factors that can affect hydrocarbon biodegradation in wastewater, such as temperature, concentration of nutrients and oxygen. Therefore, the enhancement of oil removal efficiency may be accredited to the solubilization of organic particles after thermal pretreatment, which is normally achieved in a temperature range from 40 to 180 °C when the carbohydrates and the lipids of the sludge are easily degradable [43]. These results are within the specification limits for oil of discharged water in Iraq as illustrated in Table 4.

3.7 Effect of MLSS and Preheating Temperatures on Turbidity Removal

Figure 15 shows the effect of MLSS concentrations (i.e., 500 and 1000 mg/l) on the removal of turbidity within a hydraulic retention time of 5 days at 25 °C and TMP of −450 mmHg. The initial turbidity of the oily wastewater is 21.1 NTU. It can be seen from Fig. 15 that 500 mg/l MLSS concentration results in a decrease in the turbidity of the oily wastewater from 21.1 to 1.1 NTU throughout 5 days of the hydraulic retention time (94.8 % removal efficiency). Using a 1000 mg/l MLSS concentration can decrease the turbidity concentration from 21.1 to 0.5 NTU and give excellent removal efficiency of 97.6 %. Figure 15 shows the removal of the turbidity from oily wastewater with a preheating temperature of 45 °C at different preheating times (i.e., 15, 30, 45 min) by using a 1000 mg/l MLSS concentration. It can accordingly be distinguished that at a preheating time of 15 min, the turbidity of the wastewater is decreased from 21.1 mg/l to 0.46 NTU

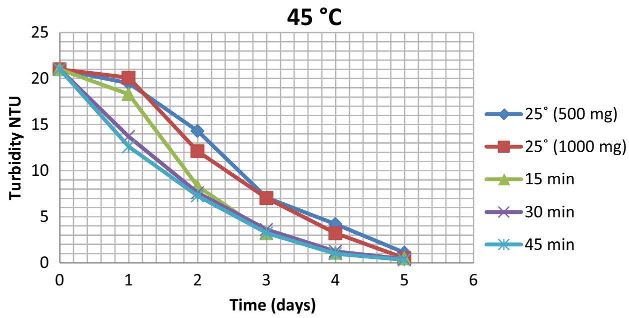

Fig. 15 Removal of turbidity at different preheating periods, temperature of 45 °C, initial MLSS concentration of 1000 mg/l, initial turbidity = 21.1 NTU, TMP = −450 mmHg and HRT = 5 days

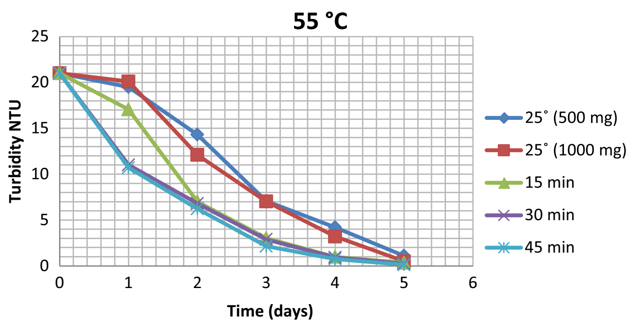

Fig. 16 Removal of turbidity at different preheating periods, temperature of 55 °C, initial MLSS concentration of 1000 mg/l, initial turbidity =21.1 NTU, TMP = −450 mmHg and HRT = 5 days

within 5 days of the hydraulic retention time and that the removal efficiency is about 97.8 %. Using 30- and 45-min preheating periods, turbidity reduces from 21.1 to 0.4 and 0.31 NTU during 5 days with 98 and 98.5 % removal efficiencies, respectively. It can be concluded that there is no significant effect of preheating time at temperatures of more than 30 °C, as it is clearly shown in Fig. 15. The removal of the turbidity is drastically increased with a preheating temperature of 55 ° Cat 15, 30, and 45 min, as shown in Fig. 16. It can be seen that the turbidity of the wastewater decreases from 21.1 NTU to 0.34, 0.24, and 0.12 NTU with 98.3, 98.8, and 99.4 % removal efficiencies at 15, 30, and 45 min preheating times, respectively. It can be concluded that the preheating temperature and time significantly enhanced the removal efficiency of the compounds in parallel to the effect of MLSS concentration. This result is in a good agreement with that reported by Choi et al. [44]. From Table 4, it can be observed that the removal efficiency of the oily wastewater turbidity is within the specification limits for turbidity of discharged water in Iraq.

3.8 Effect of Nutrients on pH

The effect of MLSS concentration on the pH values of the oily wastewater over hydraulic retention times of 5 days

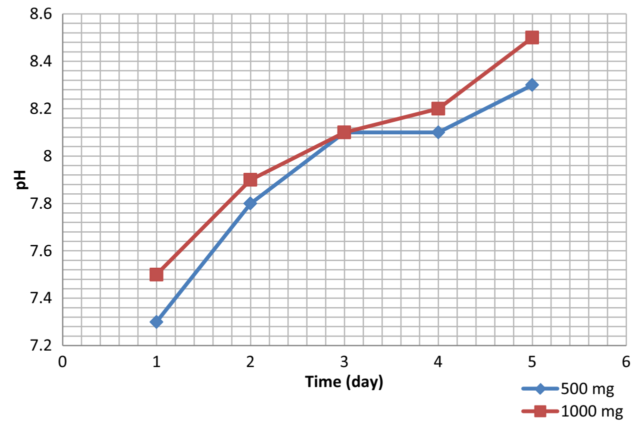

Fig. 17 pH behavior as a function of time at temperature of 25 °C and HRT of 5 days

is presented in Fig. 17. It can be seen that the pH values are increased from 7.3 to 8.3 with the addition of a 500 mg/l MLSS concentration, whereas the pH values are changed from 7.5 to 8.5 by using a 1000 mg/l MLSS concentration. In the biodegradation process, the biomass needs to receive a continuous supply from nutrients in order to increase its activity and growth. The nutrient contains urea and phosphorus, which may be changed with time to alkaline compounds, indicating an increase in the pH values. However, all pH values can be conformed to Iraqi domestic water standards. Furthermore, Mohammed et al. [45] had also reported that the increment in pH values of municipal wastewater may be caused by the degradation of volatile fatty acids.

3.9 PVC Hollow Fiber Membrane Performance

The permeation flux of the hollow fiber membrane is a function of time at 1000 mg/l MLSS concentration, as explained in Fig. 18. It can be noticed that the permeation flux is 12.64 (l/m^2 h) or 300 (ml/h) in the first 20 min of the process, by decreasing to 10.58 (l/m^2 h) or 250 (ml/h) throughout 20 min within the first day. After 5 days, the permeation flux of the hollow fiber membrane is 1.05(l/m^2 h) or 25 (ml/h) [the permeated water was collected each 20 min per day, 100 min for 5 days]. Finally, the permeation flux of the PVC MBR after 25 days is 0.63 (l/m^2 h) or 15 (ml/h). Therefore and in order to reactivate the efficiency of the hollow fiber membrane, backwash process was carried out with pure water circulation. The permeation flux of the hollow fiber membrane after backwash is found to be 11.6 (l/m^2 h) for the first 5 days as shown in Fig. 18. This decline in permeation flux is attributed to the fouling phenomenon due to the accumulation of flocs as well as the dissolved and the suspended solids on the surface of the hollow fiber membrane. Chu and Li [38] found that with an increase in the MLSS con-

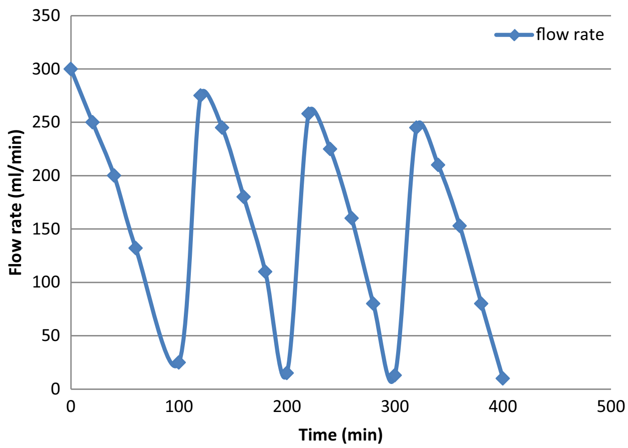

Fig. 18 Permeation flux of PVC hollow fiber membrane at TMP of −450 mmHg

centration, the permeation flux had decreased due to the suspension rheological properties. Similar behavior for permeation flux of the membrane was also observed by Zhang et al. [46].

However, using high MLSS concentration in handling the high-strength wastewater can be enhanced by the membrane performance by speeding up the degradation process, but at the same time high concentrations of MLSS can be accelerated by the fouling of the membrane [47]. In addition, high MLSS concentration may lead to clogging and low efficiency of aeration, as well as the bioreactor tank should be large enough and consequently lead to increasing the capital cost of the process [48]. Besides, the result reported by Katayon et al. [49] had showed a decrease in permeates flux by using high MLSS concentration. They also found that at low MLSS concentration, good removal efficiency of TSS and turbidity was observed. Therefore, it is important to mention here that a good MBR performance obtained by this method is due to high efficiencies in the removal of oil, all organic compounds and phenol and can be achieved by using a relatively low concentration of MLSS.

Actually, it can be said that the decline in research and development of MBR is attributed to the getting of suitable membranes and the high capital cost of the process (including the cost of the chemical and biological compounds). Furthermore, membrane fouling is another major factor that may have impacted the performance as well as the limitations of operating conditions such as pressure and temperature. For those reasons, the real tangible outcomes from this work which is benefit for the industry are by using hydrophobic polymer for fabrication of suitable membrane for MBR application such as PVC in order to overcome the difficulties in getting the membranes and its cost, in addition to the use of temperature and MLSS concentration that are present in this study as optimal operating conditions.

4 Conclusions

A new PVC hollow fiber membrane was prepared with a new configuration module for the treatment of oily wastewater in the Al-Daura refinery by using a submerged membrane bioreactor (MBR) technique. The results showed that the COD, BOD, oil content, and phenol removal efficiency were influenced by the concentration of MLSS. The removal efficiency increases with an increase in the MLSS concentration and other operating conditions that are studied in this work, such as feed temperature and preheating time. The removal efficiency of COD, BOD, oil content, and phenol at 1000 mg/l and 55 °C with 45 min preheating time were 71, 60, 100, and 100 %, respectively. The pH values of the effluent were increased from 7.5 to 8.5 using 1000 mg/l MLSS concentration due to the conversion of urea and phosphorus to alkaline compounds over time. Moreover, it can be recorded that a relatively low concentration of MLSS is resulted an insufficient removal efficiency of contaminated compounds in oily wastewater. From the results of this process, it can be concluded that the submerged membrane bioreactor (MBR) offers a potential alternative process for oily wastewater treatment and supplies effluent of sufficient quality for water reuse.

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