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Assessment of atomic force and scanning electron microscopes for characterization of commercial and electrospun nylon membranes for coke removal from wastewater

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ABSTRACT

Electrospun polymeric membranes are currently being developed for various applications due to their unique specifications in comparison with the conventional membranes. As electrospun membranes comprise nanostructures (with 3D structure and inter-connected pores) with dimensions lying within the lateral resolution of the microscope, the interpretation of electrospun membrane features is challenging. In this study, scanning electron microscopy and atomic force microscopy were used for characterization of an electrospun (N1) and two commercial membranes (N2 and N3). A self-supported electrospun nanofibrous nylon membrane was fabricated and characterized for pore size, pore size distribution, thickness, nodule size, and pure water permeation flux as well as rejection and flux decline during coke removal from a typical petrochemical wastewater stream. The obtained results show that the electrospun membrane had smoother surface. All membranes showed same separation performance, about 99% rejection, but higher permeation flux achieved for the electrospun membrane.

Keywords: Electrospinning; Nylon membrane; Characterization; SEM and AFM; Coke removal; Wastewater

1. Introduction

Polymer nanofibers are an important class of nanomaterials, which have attracted increasing attention during the last decade. This could be due to the fact that this class of materials have a considerably high surface to mass/volume ratio and also possess special features for advanced applications such as membrane separation [1–3].

The main mechanism involved in the separation process using conventionally-fabricated membranes (flat sheet or hallow-fiber) is sieving [4–6]. Relying solely upon sieving mechanism could decrease the separation efficiency. As a result, attempts have been made to develop membranes in which the separation

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mechanism includes both sieving and depth-filtration such as electrospun membranes [7–9].

Electrospinning is one of the most important, emerging, and versatile techniques currently investigated as an effective method for fabricating micron- to nano-size fibers. It is regarded to hold great promises for membrane/filtration separation purposes [2,7]. Electrospun polymeric membranes have special features compared to commercially-available conventional ones such as higher porosity, higher effective area, higher throughput, 3D interconnected structure, lower pressure drop, longer membrane life, and higher rejection rate [10].

Various operating variables could affect the electrospun membranes' specifications; i.e. fibers' diameter, pore size and pore size distribution, thickness, surface energy, surface roughness, and porosity. These variables are divided into two major groups: First, polymeric dope solution conditions (i.e. concentration, type of solvent, polymer molecular weight, viscosity, surface tension, and conductivity); Table 1 presents some polymer/solvent dopes used in fabrication of electrospun membranes [3]. Second important variable is electrospinning conditions (i.e. applied electrical voltage, needle gage, tip-to-collector distance and angle, environmental temperature and humidity, and injection rate of dope solution). Therefore, characterization of such kind of membranes is highly crucial in order to achieve a clear and more in-depth understanding of the membranes and to predict their performance for various applications.

Beside the well-known and well-developed scanning electron microscopy (SEM), which is widely used for morphology observation of polymeric membranes, atomic force microscopy (AFM) is a probe scanning technique, commonly used for producing 3D images of membranes' surfaces and topographical features. So far, AFM has been intensively applied for characterization of conventional membranes, e.g. microporous membranes, in order to measure the pore size, nodule size, and roughness; and also to study hydrophobicity and to predict fouling phenomenon [11–13]. Although, AFM has been mostly used to study microporous membranes fabricated via conventional techniques [14,15], little attention has been paid to its application for characterizing electrospun; microfibrous or nanofibrous, membranes.

Table 1
Conventional polymer/solvent systems used to prepare spinning dopes for fabrication of electrospun membranes

Polymer	Solvent
Polyacrilonytrile	Dimethyl formaldehyde
Nylon	Formic acid
Polyethylene terftalate	Trifluoroacetic acid/dimethyl chloride
Polystyrene	Toluene/dimethyl formamid/dimethyl acetamide
Polyvinyl alcohol	Water
Polyimides	Phenol

Nylon has been widely used as an important plastic due to its suitable mechanical property. Melt-, wet-, and dry-spinning have been used traditionally for preparation of nylon fibers, typically in the diameter range of 10 to 500 μm. Therefore, fabricating submicron nylon mats are of industrial interest for various applications such as gas transport, barrier layers, and filtration purposes [16]. In the present study, nylon 66 nanofibrous membrane was prepared via electrospinning and characterized for topographical properties (e.g. pore size and pore size distribution, roughness, and nodule size) using AFM and for morphological properties using SEM. Obtained results were compared with two commercially available nylon microfiltration membranes. Comparative analysis of pure water permeation fluxes and wastewater treatment of these membranes was conducted in order to evaluate their performance.

2. Experimental

2.1. Materials

Nylon 66 granules (50,000Mw, Saba Nakh Tayer Co., IRAN) were used for preparation of dope solution with 15 wt.% concentration. Formic acid (Merk, Germany) was used as solvent. All materials were used as received.

2.2. Electrospinning

An electrospinning setup equipped with a Gamma high voltage power supplier (ES60P-5W, Gamma, USA) was used for preparation of electrospun nylon membrane. Constant operating conditions, e.g. 20 kV high voltage, 25 gage stainless steel needle (3 cm length), 9 cm tip-to-collector distance, and 0.14 ml/h injection rate during 4 h spinning time, were used for electrospinning. The properties of dope solution including viscosity, surface tension, and electrical

Table 2
Specifications of dope solution for electrospinning

Conductivity (μS)	Surface tension (dyne/cm)	Viscosity (cP)		
		3 rpm	4 rpm	5 rpm
4.409	36.112	181.5	178.8	175.6

conductivity were also analyzed by using DV-II+Pro viscometer (ROOK FIELD, Germany), tension-meter (Data Physics, Germany), and CC-501 conductivity-meter (ELMETRON, Poland), respectively. Table 2 presents these specifications.

2.3. Commercial membranes

Two commercially available nylon membranes, with 0.22 μm (N1) and 0.45 μm (N2) reported pore sizes (Membrane-Solutions, China), were used for the comparative study.

2.4. Atomic force microscopy analysis

AFM was performed at room temperature (23 ± 1°C), 20 ~ 25% relative humidity, and with non-contact mode on a DUALSCOPE 95-200E equipped with DS95-200 E scanner and DUALSCOPE C-21 controller (DEM, Denmark). Samples were attached to glass slides using a double-sided tape. The scans were performed in air (ambient conditions) medium. The images were scanned using a silicone nitrate prob. The specifications of the applied cantilever and its tip are presented in Table 3. Scanning was performed at a speed of 5 μm/s (1 Hz) and force of 0.15 nN; scan sizes of 1,500, 500, and 200 nm. Phase shift was 215.4 and a sampling resolution of 200 points per line was selected.

2.5. SEM analysis

Morphological observation was carried out using a SEM system (HITACHI S-4160, Japan) with a voltage of 15.0 kV. The SEM images were analyzed using an Image Analyzer Software (Digimizer, version 4.2.0.0).

2.6. Separation efficiency test

A plate and frame cross-flow microfiltration setup, made of Plexiglas™, with 0.2 cm feed channel depth and 5 cm × 10 cm effective area was used for pure water permeation flux measurements of the electrospun membrane (N3) and the commercial ones (N1 and N2). Constant operating conditions (0.4 bar and 600 mL/min inlet pressure and feed flow rate, respectively) were used for pure water flux measurement. The obtained results were indicative of the maximum permeation flux for the fabricated and commercial membranes.

In order to show and compare the separation performance of the commercial and electrospun membranes, a suspension of coke particles (size in range of 2–95 μm) dispersed in distilled water with constant concentration was used as synthetic wastewater for filtration efficiency test.

Table 3
The specifications of cantilever and tip of the applied AFM

	Value
Cantilever	
Length (μm)	160
Width (μm)	45
Thickness (μm)	4.6
Spring/force constant (N/m)	42
Resonance frequency (kHz)	285
Slope (°)	10
Tip	
Material	Silicone nitrate
Height (μm)	10 ~ 15
Tip curvature radius (nm)	10>

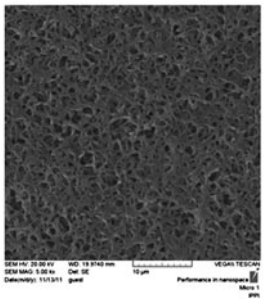
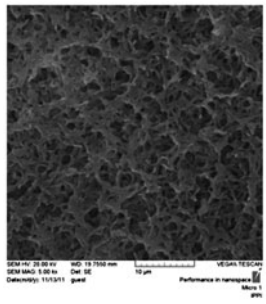
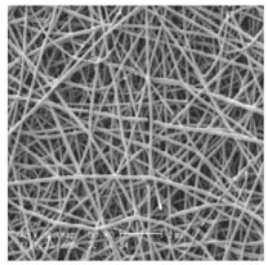
3. Results and discussion

3.1. Morphological observation (SEM analysis)

Uniform pore size and pore size distribution are two important specifications which could directly affect the microporous membranes' performance. Fabricating techniques and preparing conditions could change the pore size, pore size distribution, and their uniformity. Commercial microporous nylon membranes are usually fabricated using solution-casting method. As could be observed in Table 4, the reported pore size for N1 and N2 membranes were 0.22 and 0.45 μm. The average pore size for the electrospun membrane (N3) developed in this study was measured at 0.49 μm using SEM image analysis. It is worth quoting that the pore size values measured for the commercial membranes via SEM images analysis (0.35 and 0.42 μm for N1 and N2 membranes, respectively), were not completely in agreement with the values reported by the manufacturer (0.22 and 0.45 μm for N1 and N2 membranes, respectively). Fig. 1 shows the pore size distribution for N1, N2, and N3 membranes. It was observed that the N3 membrane had a uniform pore size distribution in comparison with N2. More than 48% of the analyzed pores were in the range of 0.4–0.6 μm, revealing a uniform pore size distribution. It should be noted that uniform pore size distribution could potentially increase rejection efficiency.

The reported thicknesses values for N1 and N2, and the measured value for N3 are presented in Table 4. As could be observed, the reported thickness values for the N1 and N2 membranes were 110 μm. The parameter was also investigated for both the commercial and electrospun membranes using a digital micro-meter in at least 5 points. The thickness values for N1 and N2 were measured at 125 and 127 μm, respectively. The electrospun membrane was found

Table 4
Specifications of commercial and electrospun nylon membranes

Membrane	N1	N2	N3
Illustrations			
Pore size (μm)[a]	0.22	0.45	0.53
Thickness (μm)	∼110	∼110	∼150

[a]Pore size (μm) measured based on SEM image processing.

thicker in comparison with the commercial ones. Given the nanofibrous structure of this membrane, its higher thickness could result in self-supporting characteristic. On the other hand, one of the most important shortcomings of the commercial membranes investigated herein and the commercial membranes in general is the necessity of implementing a support layer which may introduce a number of weak-points such as increased membrane resistance and decreased permeation flux [29]. The prepared electrospun nanofibrous membrane in this work was completely self-supported and was shown to possess a durable structure through the pure water permeation flux efficiency tests (see section 3.3).

It has been well documented that fibers' diameter could be considered as a key parameter for performance evaluation of electrospun membranes [2,7–10]. The average fibers' diameter for the fabricated electrospun membrane; N3, was measured at about 102 nm confirming its nanofibrous nature. Fig. 2 presents the

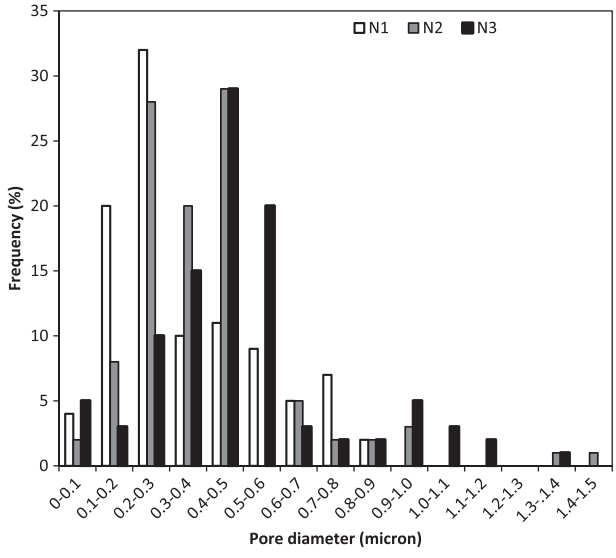


Fig. 1. Pore size distribution of the commercial (N1, N2) and the electrospun (N3) membranes, based on the image processing of the obtained SEM images.

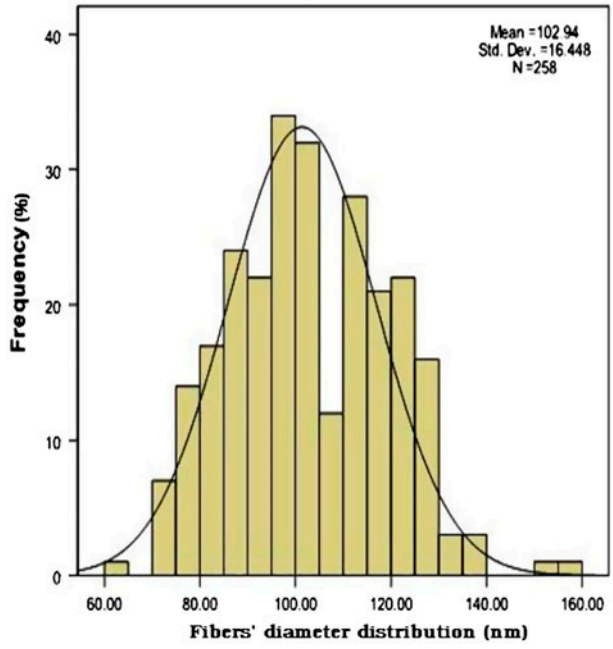


Fig. 2. The fibers' diameter distribution (based on image processing).

fibers' diameter distribution of the N3 membrane. Moreover, these results indicated that the N3 membrane had a uniform fiber distribution, and to the best of our knowledge, in the open literature this was the lowest fibers' range reported for electrospun nylon 66 membranes so far. Table 5 tabulates a literature review of various electrospun nylon membranes reported so far.

3.2. Topographical observation (AFM analysis)

Fig. 3 presents the three-dimensional (3D) AFM images, which representing the topographical observation of the investigated membranes; N1, N2, and N3. Surface roughness is also an important structural property of polymeric membranes and could be presented as average roughness (R_a), root-mean-square roughness (R_q), and/or peak-to-valley height (R_z). Surface skewness (R_{sk}) is a measure of symmetry of the height distribution. Negative skew values correspond to dominance of valleys, associated with porous-like surface, while positive skew values suggest that peaks dominate the surface [13]. Surface kurtosis (R_{ku}) describes sharpness of the height distributions. Kurtosis values lower than 3, (the Gaussian distribution corresponds to a kurtosis of 3) [26] indicate a flat and repetitive surface, while values greater than 3 suggest a sharper height distribution. These roughness parameters were estimated from images scanned over an area of $2 \times 2\,\mu m$ from each sample and are presented in Table 6.

As could be observed the average roughness (R_a), which shows the deviation in height, was higher for N2 and lower for the N3, 120 and 53.5 nm, respectively. Similar to R_a, the R_q; which represents the standard deviation of surface heights, as well as the third roughness value; R_z, were also higher for N2 and lower for N3. This means that the electrospun nylon membrane had smoother surface compared to the two commercial nylon membranes. On this basis, it could be concluded that lower surface roughness leads to lower fouling risk and higher surface hydro-

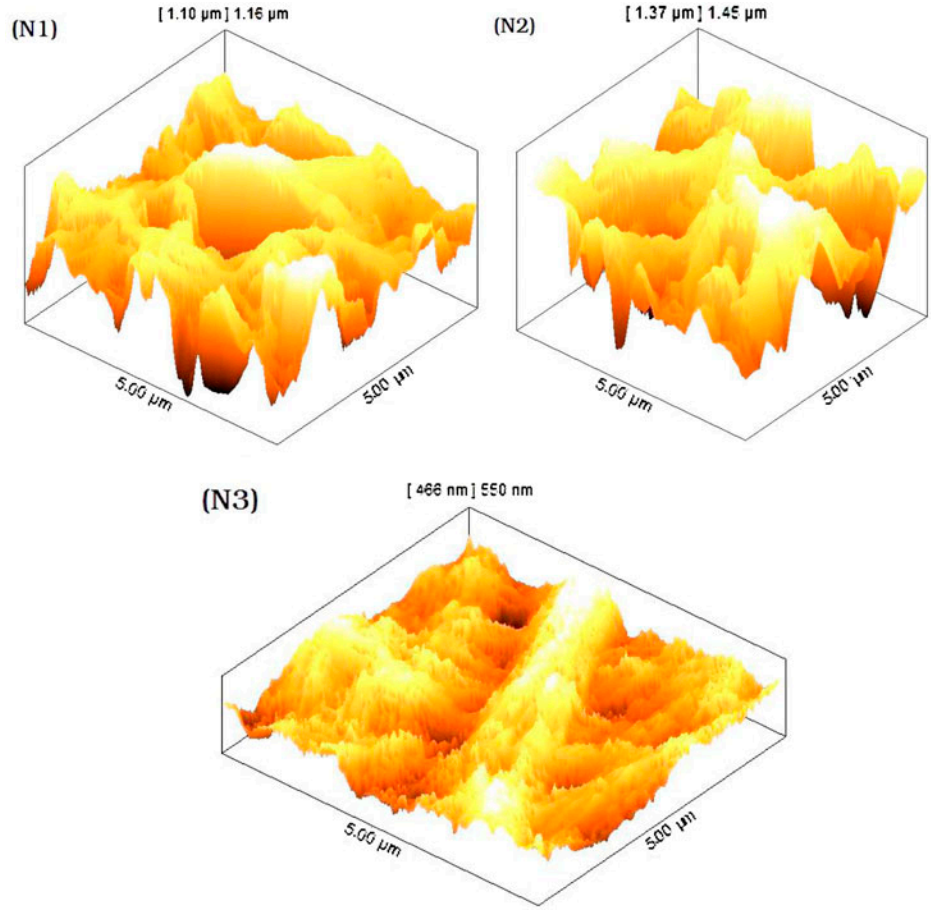


Fig. 3. Closed view of the electrospun nylon 66 fibers (via SEM image analysis), before (A) and after (B) pure water permeation test.

Table 5
The fibers' diameter of the electrospun nylon 6 and nylon 66, as reported so far in literature

No.	Polymer	Concentration (wt.%)	Fibers' diameter (nm)	Application	Ref
1	Nylon 6	10 and 15	65∼107	–	[16]
2	Nylon 66	10∼15	150∼200	Conductive nanofibers	[17]
3	Nylon 66	–	∼148	Sensor	[18]
4	Polyamide 66	15∼17	131∼176	–	[19]
5	Nylon 6	20	∼200	Filter	[20]
6	Nylon 11	10 ∼ 20	∼200	–	[21]
7	Nylon 6	20	30 ∼ 110	Membrane	[22]
8	Nylon 6	25	150 ∼ 250	Filter	[23]
9	Nylon 6	10	220	–	[24]
10	Nylon 66	20	550	–	[25]

Table 6
A quantitative summery of the roughness parameters determined for various membranes studied

Membrane	Roughness parameters				
	R_a (nm)	R_q (nm)	R_z (nm)	R_{sk}	R_{ku}
N1	86.9	114	474	0.13	5.22
N2	120	146	666	−0.32	2.80
N3	53.5	68.3	412	−0.034	3.15

Table 7
Nodule size (min., max., and mean) of the commercial and electrospun nylon membranes

Membranes	Nodule size (nm)		
	Min.	Max.	Mean
N1	24.7	159	118
N2	9.23	476	243
N3	14.5	86.3	53.2

philicity. These could be attractive findings for a number of applications such as biological solution microfiltration in which the applied microporous membrane should be as hydrophilic and smooth as possible.

Skewness is the third moment of the profile amplitude probability density function and is used to measure the profile symmetry about the mean line. When the height distribution is symmetrical, R_{sk} is zero. None of the investigated membranes in this work had zero skewness value, meaning that no symmetrical profile was observed for any of the membranes investigated. In fact, if the height distribution is asymmetrical, and the surface has more peaks than valleys, then the skewness moment is positive, as could be observed in case of N1 membrane. On the other hand, if the surface is more planar and valleys are predominant, therefore the skewness is negative, as was the case for N2 and N3 membranes. Moreover, it could be concluded that negative skewness values for N2 and N3 membranes was due to their higher porosity (valleys were predominant on the membrane surface) (Table 4).

Kurtosis moment is the fourth moment of the profile amplitude probability function and corresponds to a measure of surface sharpness. R_{ku} of 3 represents the Gaussian amplitude distribution, and the surface is called Mesokurtic. Smaller values of R_{ku} represent the flat surface, as observed for N2 and the surface is called Platykurtic; while higher values than 3 represent more peaks than valleys. The R_{ku} value for N1 and N3 membranes were measured at 5.22 and 3.15, respectively. It is worth noting that the R_{ku} value of electrospun nylon membrane developed herein was closer to Gaussian (Mesokurtic) distribution than those of the commercial membranes, revealing the uniform structure of the electrospun membrane.

AFM with non-contact mode has the potential to provide additional resolution allowing measurement of the nodule size [30]. This analysis could yield more in-depth understanding of the topographical architectures resulting from electrospinning process. Therefore, the electrospun and commercial membranes were analyzed for their nodule sizes. The membranes' nodule sizes were determined by AFM based on the method described by Khulbe and Matsuura [27]. In order to measure the nodule sizes, cross-sectional line profiles were selected to traverse micron ($5 \times 5\,\mu m$) scan surface areas of the AFM images. The diameter of the nodules (i.e. height peaks) were measured by a pair of cursors along the reference lines. The horizontal distance between each pair of cursors was taken as the diameter of the nodule. The AFM software (Dualscope™/Rasterscope™ SPM, Version: 2.1.1.2; in this work) used allowed quantitative determination of

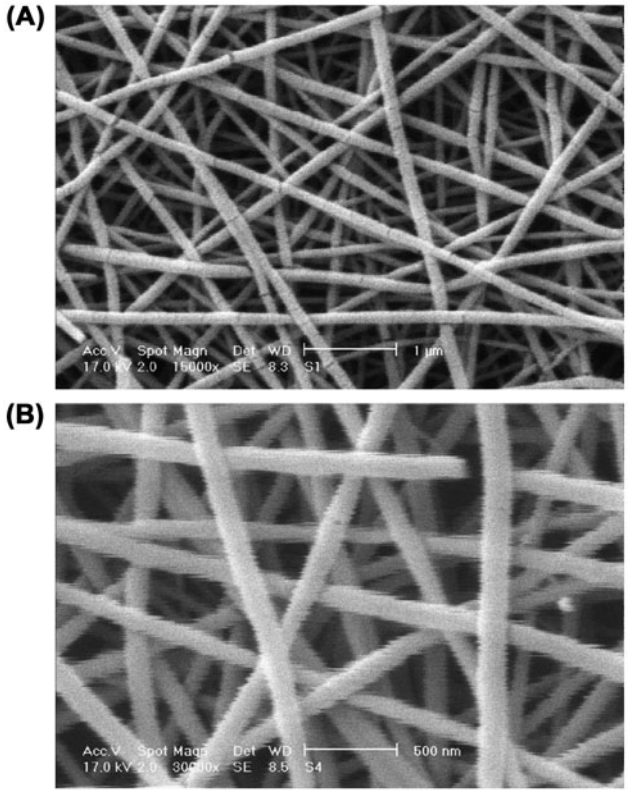


Fig. 4. 3D AFM images of the investigated nylon membranes, commercial membranes with (N1) 0.22 μm, (N2) 0.45 μm pore sizes, and (N3) electrospun membrane with 0.53 μm pore size.

nodules by use of the images. Nodule sizes were determined for at least 30 points on each membrane sample.

Table 7 presents the mean, maximum, and minimum values of nodules sizes based on the topographical (AFM) analysis. As could be observed, the nodule size was significantly lower for the electrospun membrane; N3, compared to the two commercial nylon membranes. The mean value of the nodule size was measured at 118 and 243 nm for N1 and N2 membranes, respectively; while this value was measured at 53.2 nm for the N3 membrane. This nodule size difference could be described based on the following explanation.

The commercial membranes (such as N1 and N2 in the present study), are usually fabricated through solution-casting/phase-inversion method, and have two-dimensional (2D) composite structures consisting of a thin microporous active (selective) layer on a porous support layer. This 2D structure may limit the membrane's performance. In contrast, the N3 membrane had a 3D structure with interconnected pores as could be observed in Fig. 4. This specification is one the major advantages of the electrospun membranes compared to the conventional ones. In fact, randomly structured fibers, either in nanosize or microsize, lead to fabrication of a highly porous membrane with highly selective separation capacity. This specification could be obviously interesting in aqueous solutions filtration (suspensions or emulsions) and oily wastewaters treatment [7,28].

3.3. Separation performance evaluation test

The pure water permeation flux (mass; kg, or volume; L, of the collected permeate per the membrane effective area (m^2) and operating time (h)) for the three investigated membranes was measured using a microfiltration setup via constant operating conditions of 0.4 bar and 600 mL/min inlet pressure and flow rate, respectively. Fig. 5(A) shows the variation of the permeation flux vs. time for the commercial (N1 and N2) and electrospun (N3) nylon membranes. The obtained values refer to the maximum permeation fluxes which could be reached for these membranes. As nylon is a hydrophilic polymer, high permeation flux is expectable for these kinds of membranes, especially for the electrospun nylon membrane. Moreover, the higher pore size and porosity of the N3 membrane led to higher permeation flux in comparison with the N1 and N2 membranes. In order to investigate the durability of the electrospun membrane, SEM image was provided after pure water permeation test. As could be observed in Fig. 4(B), no change was observed in the structure of the electrospun membrane, even after using 0.4 bar feed pressure. Obtained result could support this hypothesis that the prepared self-supported electrospun nylon membrane has durable structure and could be used for filtration purposes.

The main purpose of this study was the comparison the characteristics of commercial and electrospun nylon membranes using microscopic methods (AFM and SEM). However, comparative study of the separation performance of these membranes could supports hypothesizes concluded in the previous sections. Therefore, coke removal from an aqueous stream, as a synthesized wastewater [4] with feed concentration of 0.1% was investigated for evaluation of separation performance of applied membranes. The feed temperature, applied pressure, and feed flow rate were 25°C, 0.4 bar, and 600 mL/min, respectively. Fig. 5(B) represents the flux variation vs. operating time for the three applied membranes. At the beginning time, the permeation flux rapidly decreases for two commercial membranes but more slightly for N3 membrane. It was due to precipitation of coke particles on the

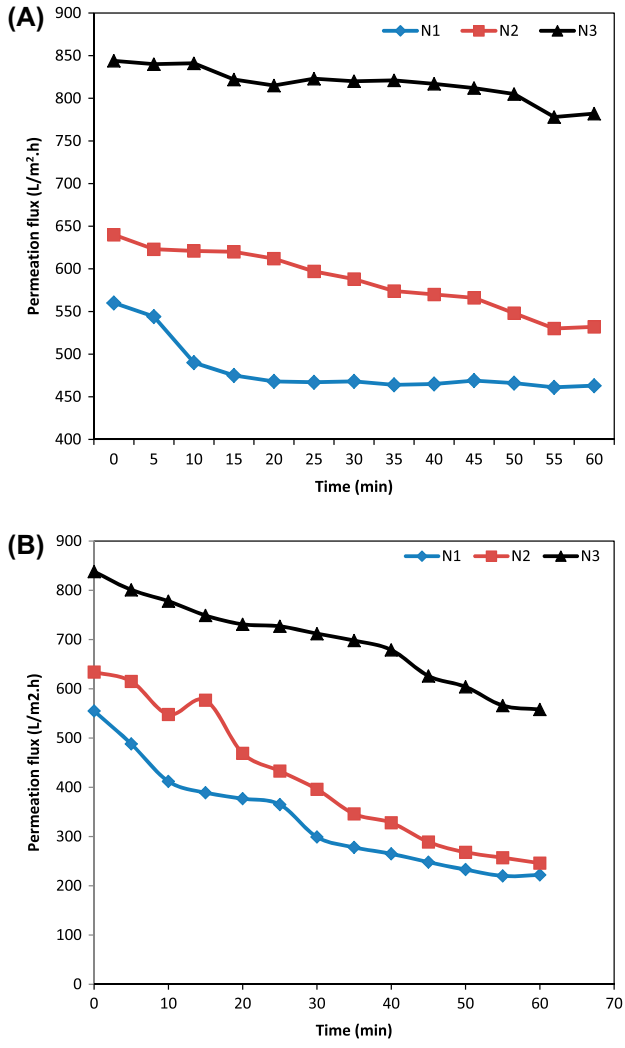


Fig. 5. The variation of pure water permeation flux (A) and synthesized wastewater (B) versus time among the commercial (N1, N2) and the electrospun (N3) membranes (Pressure: 0.5 bar; Flow rate: 600 mL/min, coke concentration: 0.1%).

membranes' surface. As time goes by, the permeation flux is decreased for all membranes. The main importance of this experiment was that, for all membranes, no coke particle passed through the membranes and the permeate samples were the same as those for pure water. It means that the investigated membranes, N1, N2, and N3, have suitable pore size range for this separation. It is worth quoting that the permeate samples were analyzed using particle size analyzing (CILAS-1064 particle size analyzer) test for coke particle detection for at least 5 times.

It is worth noting that, however, the coke particle rejection of all membranes was 99%, the permeation flux of the electrospun membrane was significantly higher than that of commercial membranes. This result can support all hypothesis discussed in previous sections.

4. Conclusions

In the present study, the application of SEM and AFM for characterization of electrospun microporous membranes has been assessed. The objective was to understand whether AFM could provide more in-depth information to increase our understanding of the electrospun structure compared to the SEM method. This understanding could also be utilized to evaluate the fabrication method used. Two commercial and one electrospun nylon membranes were analyzed using morphological and topographical images obtained by SEM and AFM, respectively. Pore size and pore size distribution, nodule size, and roughness parameters were analyzed. It was found out that the electrospun membrane was in general of superior specifications compared to the two commercial nylon membranes studied. More specifically, the electrospun membrane not only had a 3D structure, but also had a smoother surface in comparison with the 2D commercial membranes. Fabrication method could effectively influence the membranes' characterization. Both SEM and AFM method could provide valuable, but almost different results when used for membranes' characterization. Overall, the AFM was found as a powerful and practical analysis method for characterization of electrospun microporous membranes.

The obtained results in this work could be considered as a reference for researchers who are interested in the fabrication and application of electrospinning in order to prepare microporous membranes for various applications, especially nylon 66 electrospun membranes for microfiltrations of either suspensions or emulsions solutions.

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