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Language Editor: Sajjad Saberi

Art & Design: Fatemeh Hajizadeh

Iranian Association of Chemical Engineers, Unit 11, No. 13 (Block 3), Maad Building, Shahid Akbari Boulevard, Azadi Ave., Tehran - Iran.

Tel: +98 21 6604 2719 Fax: +98 21 6602 2196

# Iranian Journal of Chemical Engineering

Vol. 16, No. 2 (Spring 2019), IChE

<b>Biofouling Behavior in Forward Osmosis Using Vertically Aligned CNT Membrane on Alumina</b>	<b>3-13</b>
M. R. Omidkhah Nasrin, H. Azami, L. Ghaheer	
<b>Prediction of True Critical Temperature and Pressure of Binary Hydrocarbon Mixtures: A Comparison Between the Artificial Neural Networks and the Support Vector Machine</b>	<b>14-40</b>
M. Etebarian, K. Movagharnejad	
<b>Segregation Patterns of an Equidensity TiO<sub>2</sub> Ternary Mixture in a Conical Fluidized Bed: CFD and Experimental Study</b>	<b>41-56</b>
M. Rasteh	
<b>The CFD Providing Data for Adaptive Neuro-Fuzzy to Model the Heat Transfer in Flat and Discontinuous Fins</b>	<b>57-69</b>
R. Beigzadeh	
<b>Interior Modification of Nano-Porous Fillers to Fabricate High-Performance Mixed Matrix Membranes</b>	<b>70-94</b>
H. R. Sanaeepur, A. Ebadi Amooghin, A. Kargari, M. R. Omidkhah, A. Fauzi Ismail, S. Ramakrishna	
<b>Fe<sub>3</sub>O<sub>4</sub> Magnetic Nanoparticles/ Graphene Oxide Nanosheets/Carbon Cloth as an Electrochemical Sensing Platform</b>	<b>95-102</b>
A. Jafarizad, H. Hazrati, A. M. Jabbari Esgandani	
<b>CFD Study of Mixing Concave Turbine in a Stirred Tank</b>	<b>103-118</b>
A. Sinkakarimi, A. Ghadi	
<b>Notes for Authors</b>	<b>119</b>

# Biofouling Behavior in Forward Osmosis Using Vertically Aligned CNT Membrane on Alumina

M. R. Omidkhah Nasrin<sup>1\*</sup>, H. Azami<sup>2</sup>, L. Ghaheer<sup>1</sup>

<sup>1</sup> Chemical Engineering Department, Tarbiat Modares University, Tehran, Iran

<sup>2</sup> Department of Chemical Engineering, Faculty of Engineering, University of Bojnord, Bojnord, Iran

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## ARTICLE INFO

### Article history:

Received: 2016-07-09

Accepted: 2019-02-17

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### Keywords:

Forward Osmosis,  
Membrane,  
Vertically Aligned Carbon  
Nanotube,  
Biofouling,  
Permeate,  
Draw Solution

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## ABSTRACT

Nowadays, forward osmosis (FO) with many advantages in water treatment is so attractive for researchers and investigators that the studies are going to optimize and increase its efficiency. However, one of the most controversial operating malfunctions of FO process is fouling that limits the FO global application. In the following research, vertically aligned carbon nanotube (VACNT) on alumina membrane is introduced with high water permeability and less biofouling potential in forward osmosis for seawater osmotically dilution systems. VACNT membranes were prepared via pyrolysis of polymer into the pores of alumina. The effect of the temperature of pyrolysis process on CNT's structure is assessed, indicating that the crystallinity of the CNTs increases in a higher pyrolysis temperature of 800 °C. A small-scale setup was designed for FO analysis, and measurements of biofouling, flux, and the effect of osmotic pressure were measured. Furthermore, all analyses were compared with commercial TFC membrane, and results demonstrated that VACNT membrane had 40 % less biofouling potential and 2 times better flux results.

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## 1. Introduction

In 1996, it was reported by the U.S. Geological Survey (Gleick) that seas and oceans contained 96.5 % of Earth's water, while 1.7 % of Earth's water was placed in the ice caps and fresh water was approximately 0.8 % of the global water storage [1]. Water applications in various forms ranging from municipal and industrial to agricultural demands are increasing with population growth, while the accessible water sources are not qualified without water treatment systems. Industrialized sea water treatment methods all over the world are divided into two main categories including thermal and membrane

processes. While Multi Effect Distillation (MED) and Multi Stage Flashing (MSF) are the popular methods of thermal desalination systems, reverse osmosis (RO) is the most attractive membrane-based desalination method [2, 3]. In comparison to thermal technologies, nowadays, RO is the most common worldwide technology due to its lower power consumption and reliable aspects [3, 4]. However, some drawbacks in RO processes result in high economic costs, making this attractive method unaffordable for many countries [5]. Therefore, researchers and scientists have investigated inexpensive water reclamation methods with high

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\*Corresponding author: [omidkhah@modares.ac.ir](mailto:omidkhah@modares.ac.ir)

efficiency and lower product cost such as forward osmosis (FO) to be replaced with RO [6]. FO phenomenon is based on water permeation from high osmotic pressure (Donor solution) to low osmotic pressure (Draw Solution); hence, fresh water passes through semi-permeable membrane by an osmotic pressure gradient agent without any hydraulic pressure. Different osmotic agents are used in draw solution preparation such as sodium chloride, magnesium sulfate, glucose, ammonium carbon dioxide, and magnesium chloride [7, 8]. FO technology, due to its natural driving force in comparison to high hydraulic pressure-driven processes, is less subjected to fouling propensity. Facility, low energy utilization, and reliability of FO membrane technique make it an alternative to replace pressure-driven membrane processes such as RO. Despite several advantages of FO process, this technology is not known yet as a sufficient technology for practical and industrial implementation [9]. Internal and external concentration polarizations (CP) are some of the technical factors that affect membrane permeation and efficiency. Generally, CP is one of the significant events relating to membrane structure and depends on the concentration of the osmotic agent on membrane surface, which is associated with the bulk solution. Recently, new researches have shown that sufficient membrane orientation could reduce CP phenomenon and recover the efficiency of FO [5]. As it is described, fouling problems of FO are not as much serious as other membrane-based methods. Nevertheless, bio-fouling among various fouling types is difficult and uneconomical for controlling sea water desalination due to appropriate circumstances of growth, adhesion to membrane surface, and also self-protection property of

microorganisms against chemical cleanings [10, 11]. Recently, the application of nanostructure materials, such as carbon nanotubes (CNTs), has been accepted in water treatment methods. CNT membranes characterized by impressive and important properties can easily overcome membrane processes' obstacles such as low flux, low selectivity, and high fouling tendency (particularly bio fouling) [12]. Doubtless, the application of CNT membrane in FO method can help improve this novel water treatment method in flux recovery and fouling aspects. Frictionless transfer of water molecules through vertically aligned CNT membranes causes less pressure demand for separation. Moreover, CNTs loaded on membrane surface can prevent biofouling by damaging microorganism's cell [13]. Recently, several efforts have been made by researchers to synthesize materials with nanometric dimensions. One of the most important techniques is called "template-assisted synthesis", which is based on synthesis of desired materials within the template channel. A new technique is developed by Kyotani et al. for synthesizing novel carbon materials based on the carbonization of an organic polymer in various inorganic materials [14]. Various organic polymers have been used as carbonaceous bases, including poly (acrylonitrile), poly (*p*-phenylene vinylene), polyimides, poly-vinyl acetate, poly (furfuryl alcohol) (PFA), etc. [15]. This method is called pyrolytic method and is used to produce carbon nano-structure in a template. In this project, pyrolysis of Poly-benzimidazole (PBI) on anodic aluminum oxide (AAO) template for synthesizing VACNT membrane and its performance in forward osmosis processes is investigated.

## **2. Materials and method**

### **2.1. Materials**

A commercially available TFC polyamide RO membrane (Dow Film Tech, BW30) was provided by industrial RO module. Membrane sheets were brought out of module, and membrane sheets without spacers were used. AAO unsupported and autoclavable discs (Whatman, diam. 13 mm, pore size 0.02  $\mu\text{m}$ ) are provided as template for CNT growth in VACNT/AAO membrane preparation. For pyrolysis protocol Polybenzimidazole (PBI) powder (Sigma Aldrich) is used as a carbon source and mounted on the template by deep method. N-Methyl-2-pyrrolidone (NMP) Sigma Aldrich is used as solvent of PBI. Draw Solution (DS) and Feed solution (FS) used in this study are prepared by distilled water. FS is pure without any contaminant, while DS is prepared by dissolving appropriate amount of Sodium Chloride (NaCl), Merck Co.

### **2.2. Synthesis and characterization of VACNT membrane**

As described in the introduction, template method by utilization of pyrolysis technique is used in this project according to the modified template-assisted method [16]. Briefly, AAO templates with an average pore diameter of 13  $\mu\text{m}$  were soaked in 5 % wt. PBI in NMP solution for 60 min and mounted in the oven for 24 hours in order to remove solvent. Discs were sandwiched between two iron discs and placed in the reactor. Pyrolysis process was finalized in furnace at final temperatures of 550  $^{\circ}\text{C}$ , 700  $^{\circ}\text{C}$ , and 800  $^{\circ}\text{C}$  and in the presence of nitrogen atmosphere. The nitrogen flow was adjusted to 170 ml/min. The rate of temperature increment was constant, and the final furnace temperature remained unchanged for 1 hour.

The structural properties of VACNT membranes were evaluated by X-ray Diffraction (XRD), Raman Spectrophotometry, Contact Angle, and Scanning Electron Microscope (SEM). The formation of the carbon phase within the AAO pores was clearly shown by the Raman and XRD spectroscopy of all CNT/AAO. X-ray diffraction (XRD) patterns were taken with a Phillips diffract meter (X<sup>2</sup>-Pert MPD, Philips, Netherland) using Co-K $\alpha$  radiation ( $\lambda = 1.789 \text{ \AA}$ ). The diffract grams were collected at 0.02 $^{\circ}$ /s. The diffraction voltage was 40kV with an electrical current of 30 mA.

$$D = (1 \times \lambda) / (2 \times \sin(\theta)) \quad (1)$$

Equation 1 gives two graphitic peaks  $d_{(002)}$  and  $d_{(100)}$ , which are evaluated in the next section.

The Raman spectra were obtained ( by Senterra, Bruker, Germany) and coupled with optical microscope that focuses the incident radiation down to a 2  $\mu\text{m}$  spot. A laser emission source of 785 nm with a power of 10 mW over the 445-1805  $\text{cm}^{-1}$  region is used. Results are evaluated in the next section.

### **2.3. Evaluation of membrane permeability**

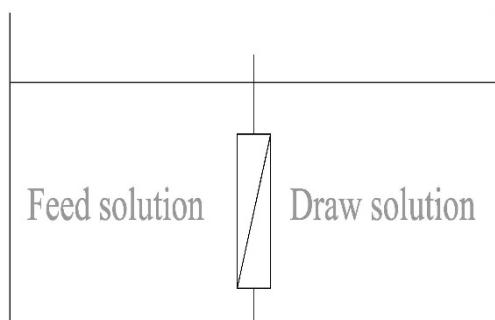
Selected membrane is tested in designed forward osmosis setup. Special setup of plexi-glass designed with two tanks was mounted with dimensions of 15x10x10 cm for draw and donor solutions, which are separated from a membrane module.

The flux performance of VACNT membrane is evaluated at three different draw solution concentrations of 20000-30000-40000 ppm NaCl in distilled water. In all cases, donor solution is considered distilled water. The flux of the membranes was

measured according to Equation 2.

$$F = \frac{V}{A \times t} \quad (2)$$

where V is the volume of permeate, A is the membrane active area, and t is time of the filtration. All experiments were repeated with TFC membrane. In all cases, one layer of membrane sheets is used without any spacer.



**Figure 1.** Schematic diagram of the FO system.

## 2.4. Evaluation of VACNT membrane biofouling

### 2.4.1. Anti-bacterial analysis

VACNT, AAO, and TFC membranes were analyzed for bacterial growth potential. E. coli suspension at about  $10^8$ - $10^9$  (cfu/mL) was prepared. 10 ml E. coli suspension was induced into the surface of membrane discs (with a diameter of 1 cm) and placed in an incubator at 37 °C. After 24 h period of contact, the whole E. coli suspension was collected with normal saline (0.9 %wt. NaCl) and serially diluted 2-10 times with a dilution ratio of 3 by normal saline. The dilutions were plated on Luria-Bertani (LB) agar plates. Viable bacterial colonies were counted after incubation at 37 °C for 48-72 h. The reduction rate in the number of bacteria was calculated using the following expression [17].

$$R = \frac{(B-A) \times 100}{B} \% \quad (3)$$

Equation 3 introduces antibacterial percentage, where R is the reduction rate, A is

the number of bacteria recovered from the inoculated membrane samples over the constant contact time, and B is the number of bacteria recovered from the inoculated membrane sample at no contact time.

### 2.4.2. Biofouling analysis

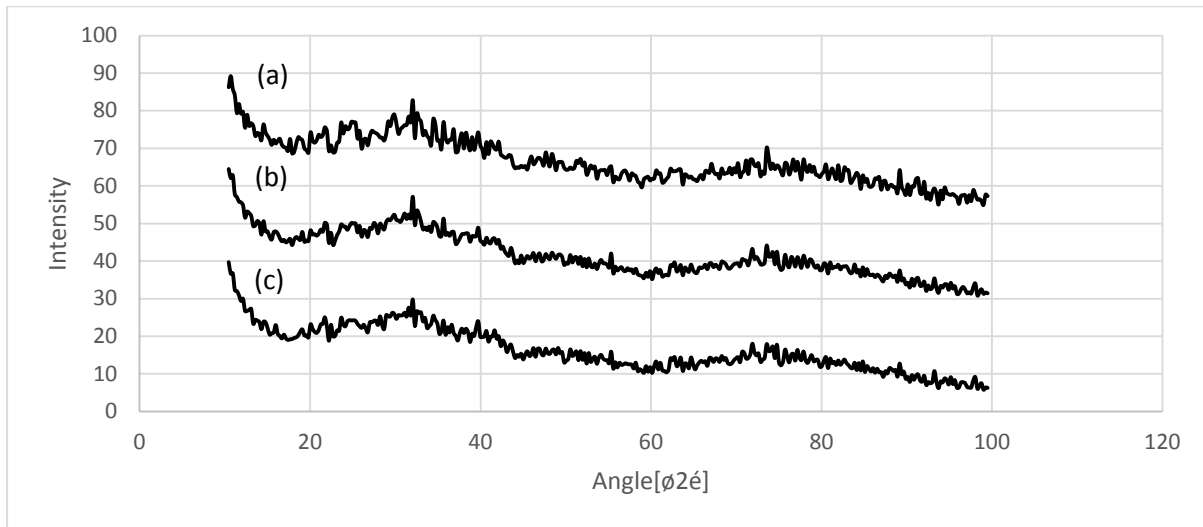
Autoclaved membranes after inducing 10 ml of E.coli bacteria suspension were incubated at 37 °C for 18 h. Then, all membranes were tested in the similar procedure of the clean membrane performance analysis [17-19].

## 3. Results and discussion

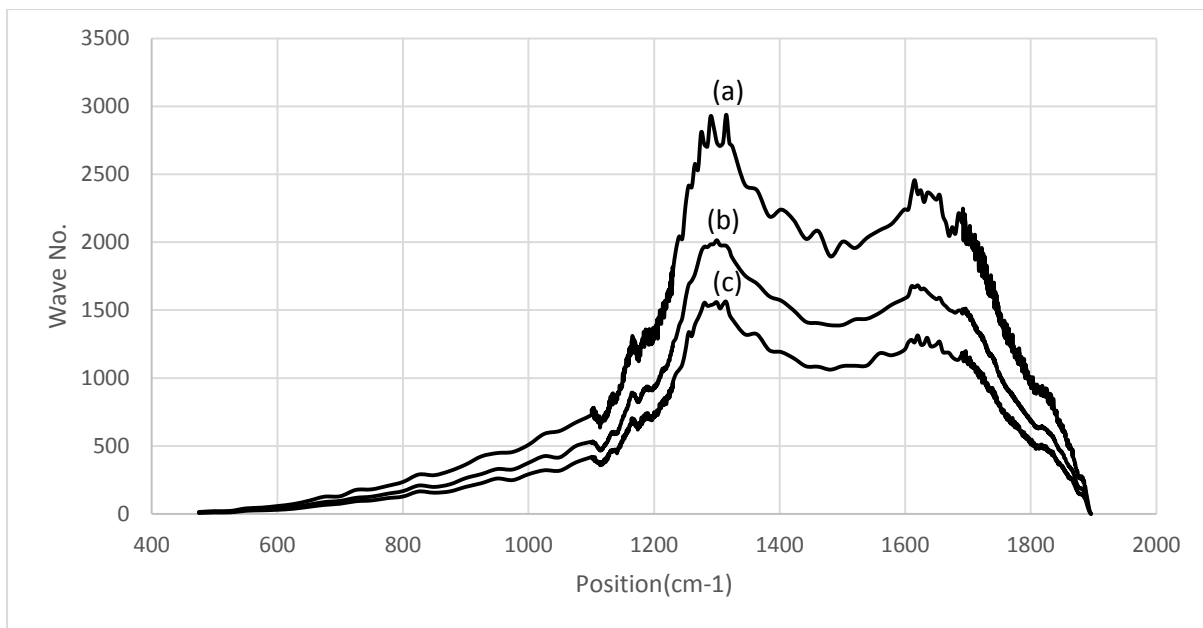
XRD data of the CNT/AAO are present in Fig. 2, showing two main graphitic peaks: a broad diffraction peak at about 30° (002) that represents the distance of the different layers of multi-wall carbon nanotubes; a low-intensity peak almost at 55° (100) reflecting the carbon-carbon bond in a hexagonal structure of graphitic carbon layers [20].

Raman spectrophotometry results are shown in Fig. 3. Two peaks are observed in 1300 and 1600; the first one is related to disordered nano carbon layers and the second one related to graphitic layers. The  $I_G/I_D$  ratio is the parameter that indicates the crystallinity of CNTs structure [15]. As shown in Fig. 3, the quality of the CNTs was modified in high pyrolysis temperature. The increase of compactness of MWCNTs in higher synthesis temperature was reported by Maniwa et al. [21].

As shown in Figure 3, an increase in the temperature of the pyrolysis step leads to more ordered CNTs, which is in agreement with XRD results. The relative intensity of the IG and ID bands, as an indication of the quality of the carbon nanostructures, varies from 0.73 to 0.88, thereby revealing some degree of disorder in the CNT/AAO.



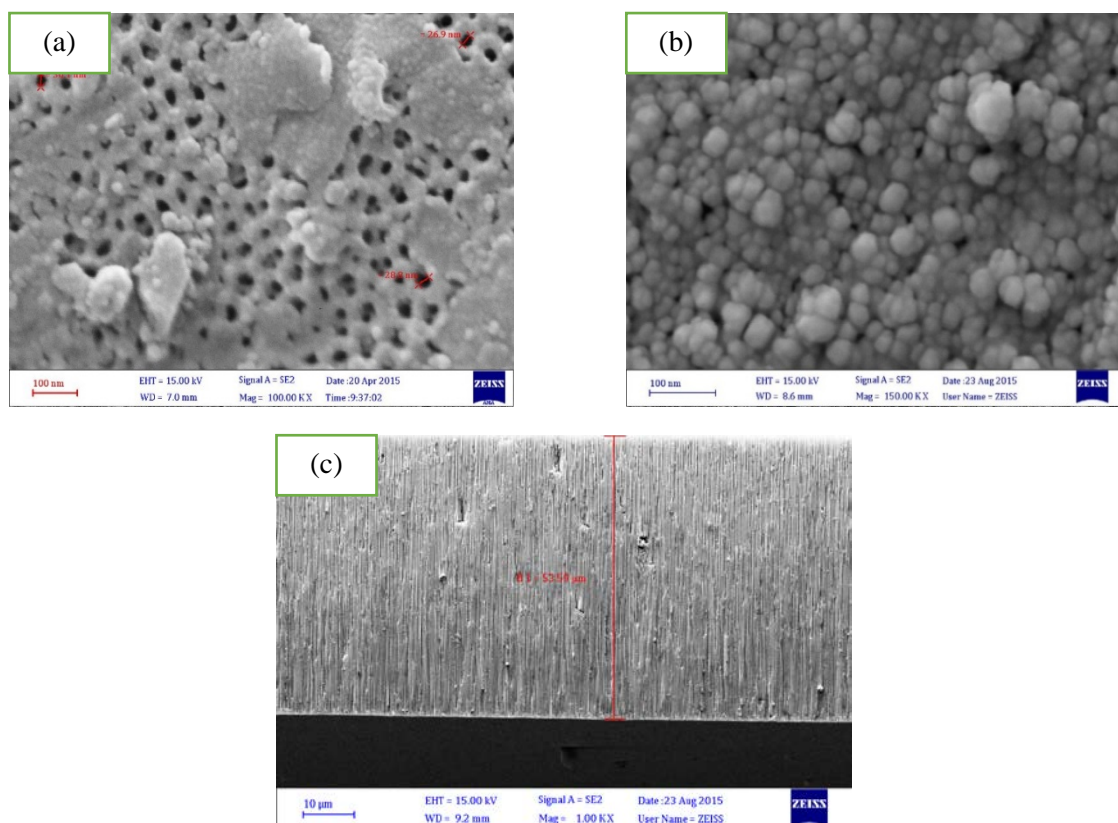
**Figure 2.** XRD patterns of the CNTs/AAO with different final pyrolysis temperatures: (A) 550 °C, (B) 700 °C, and (C) 800 °C.



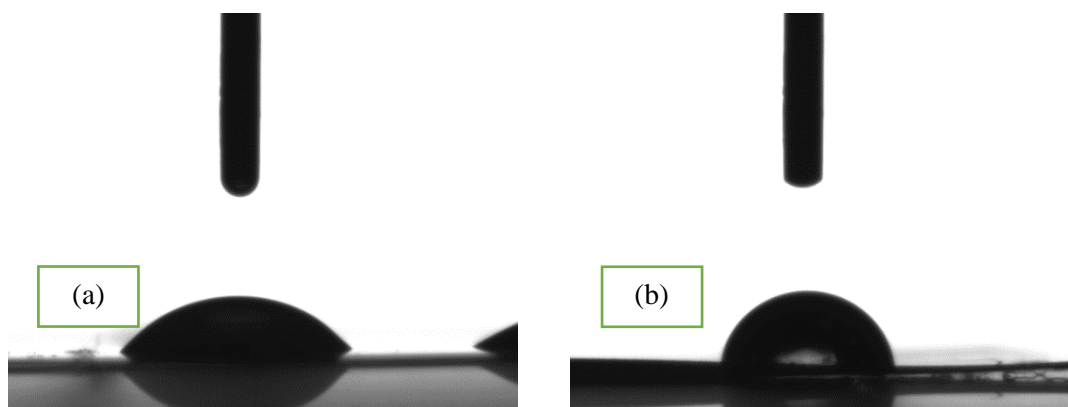
**Figure 3.** Raman spectra of CNTs/AAO pyrolyzed in (a) 550 °C, (B) 700 °C and (C) 800 °C.

Raman and XRD spectra analysis showed that optimized pyrolysis temperature was 800 °C; therefore, the rest of VACNT/AAO membranes were prepared at this temperature and applied in the next steps of the experiments. Fig. 4 shows SEM images from AAO and VACNT/AAO membranes. Pore size of VACNT/AAO membrane was obtained by image J software and was about 2

nm. Porosity of VACNT/AAO membrane is 5 %. According to cross-section SEM results, the CNTs were made with a high alignment level. Fig. 5 indicates the hydrophobicity of the VACNT membranes' surface. Contact angle is evaluated by sessile drop analysis. As is obvious, the surface of the VACNTs was found hydrophobic.



**Figure 4.** SEM images of a) AAO membrane, b) front VACNT/AAO-800, and c) cross VACNT/AAO-800.



**Figure 5.** Contact Angle results for a) AAO and b) VACNT/AAO.

### 3.2. Membrane permeability and selectivity

The water flux and salt rejection of VACNT/AAO and TFC membranes were evaluated by osmotic dilution using NaCl draw solution under three different osmotic pressures at 25 °C. The permeability results are shown in Fig. 6, and rejection results are shown in Fig. 7. It is observed that VACNT/AAO membrane provides higher

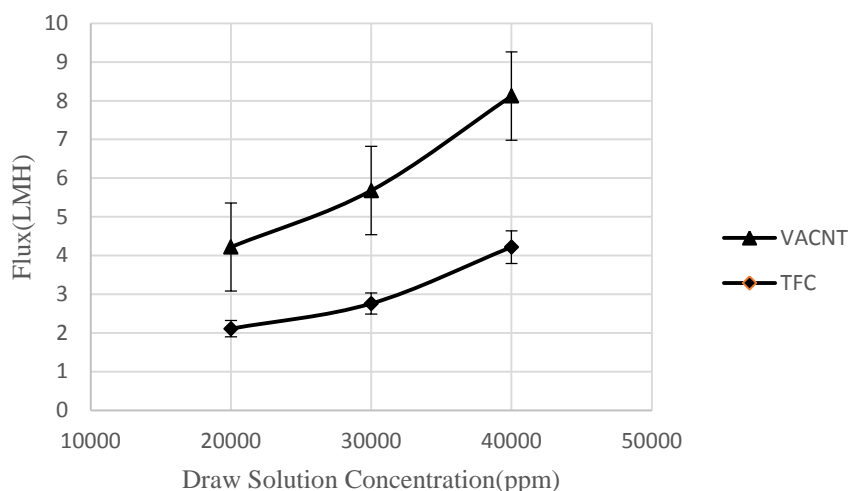
flux; however, TFC membranes had higher salt rejection. Results showed that, at higher osmotic pressures, higher flux was achieved. Fig. 7 shows that, with the passage of time, TFC membrane had about 100 % rejection performance.

Fast water flux is described by the statement in which hydrophilic water quickly passes through the hydrophobic CNT's inner wall

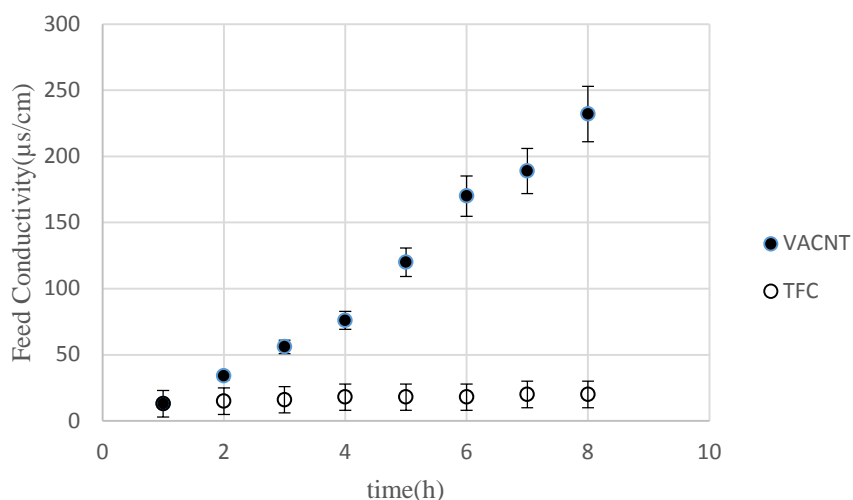


due to low hydrophilic-hydrophobic interaction [22], which was previously revealed by many researchers [23, 24]. Furthermore, it appears that water molecules spontaneously flow into the internal CNTs by

forming a strong hydrogen-bonding network, which is thermodynamically stable arrangement of water molecules within the interior of CNTs [25, 26].



**Figure 6.** Permeability of VACNT/AAO and TFC membranes in different draw solution concentration.



**Figure 7.** Salt rejection vs. time for VCNT/AAO and TFC membrane for draw solution concentration of 40000 ppm.

### 3.3. Membrane anti-bacterial and anti-biofouling ability

As described before, VACNTs have antibacterial properties. AAO, VACNT/AAO, and TFC membranes in the same shape and diameter were autoclaved before antibacterial analysis. Table 1 shows the reduction rate of bacteria colonies on the surface of membranes. The method used for

measurement is a plate count method. R (%) is calculated by Equation 3.

Table 1 shows that there is no antibacterial potential on AAO membrane surface, while, after CNT growth on AAO template, it is clear that surface structure can damage cell membranes of bacteria and prevent microorganisms growth.

Previous reports discovered that CNTs

acted as numerous moving nano darts, attacked and penetrated into the membrane of the cells, reduced their metabolic activities, and killed the bacteria [27, 28]. The penetration of the CNTs into the cells' membrane is the first step of the cells' disruption. The high-order MWCNTs could penetrate into the cells membrane rapidly due to the more vertically aligned structures with smoother surface wall.

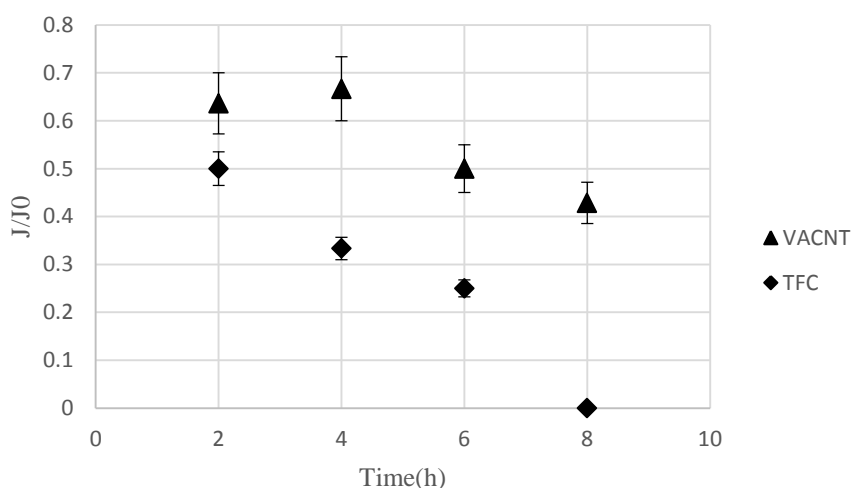
**Table 1**  
reduction ratio of bacteria colonies induced on membrane surfaces.

No.	Membrane sample	R(%)
1	TFC	20
2	AAO	13
3	VACNT/AAO	92

For anti-biofouling tests, after injection bacteria suspension on membranes and incubation, membranes were smoothly washed, and the permeability of the membranes was measured in the same condition of draw solution concentration of 40000 ppm.

Fig. 8 shows that bacteria adhesion on the surface of VACNT membrane is less than that on TFC membrane, and pore bucking is about 40 % lower for VACNT/AAO membrane.

After *E. coli* culturing, the water flux of fouled membranes and also their salt rejection decreased significantly, compared to pristine membranes. Once the biofilm formed during the biofouling phenomena, the new barrier layer against the water permeation across the membrane was generated. Additionally, the biofilm induced concentration polarization, resulting in inhibited back diffusion of salt ions on the membrane surface [29, 30]. The greater ionic activity in the boundary layer caused an increase in salt transport through the membrane and, thus, the salt rejection decreased after the biofilm formation. In addition, the polymeric membranes are vulnerable to biodegradation or biodeterioration via enzymes, localized pH or redox potential changes [31]. It could be another reason for the fall of salt rejection in TFC membrane.



**Figure 8.**  $J/J_0$  after biofouling induced on VACNT/AAO and TFC membranes for draw solution concentration of 40000ppm.

#### 4. Conclusions

Results showed that VACNT membranes could be used in forward osmosis dilution,

especially in pretreatment systems of RO or other water purification systems. The permeability of VACNT membranes was two

times that of the commercial RO membrane. Moreover, VACNT membrane had an antibacterial surface that limits microorganism's growth. The less adhesion of dead microorganism and antibacterial potential of the VACNT membranes caused VACNT to have about 40 % lower biofouling tendencies in comparison to RO TFC membrane. However, less rejection of this membrane was the obstacle that should be mitigated in the future.

### **Acknowledgement**

This study was supported by Iranian National Science Foundation (INSF) (Grant no: 92000914). The authors gratefully acknowledge this support.

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