Poly-Vinylidene Fluoride (PVDF)/ Span-85 Membrane for Omega-3 PUFA Concentration: Membrane Performance and Fouling Analysis

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Abstract: For increasing the efficiency of the omega-3 production, the effect of span-85 surfactant as a hydrophobic agent (0, 1, 2, 3 and 4 wt%) on the formation of a porous structure in a sub-layer PVDF membrane was studied. Also, the cross-sectional morphology of PVDF membranes was analyzed using scanning electron microscopy (SEM) and the surface property was evaluated using contact angle test. The capacity of the membranes to concentrate omega-3 was examined by the filtration of lantern fish oil. Also, the fouling of the membrane caused by oil filtration was evaluated. All filtration tests were performed in batch mode. Results of the SEM images demonstrated that the addition of span-85 resulted in the forming of a membrane with a denser and more hydrophobic structure. The addition of 2 wt% span-85 increased the omega-3 concentration, while the higher level of span-85 showed a negative effect. The best efficiency of the membrane was 42.64 wt% which was obtained by PVDF/2 wt% span-85. This efficiency was about 11% higher than the control PVDF (0 wt%). Taken together, the presence of span-85 affected the phase inversion process during the membrane formation, which improved omega-3 concentration. Moreover, the more hydrophobic surface of the membrane resulted in higher cake formation.

Keywords: Omega-3 PUFA, Poly-vinyldene fluoride (PVDF), Membranes performance, Span-85, Concentration, Fouling analysis.

1. INTRODUCTION

Eicosapentaenoic acid (EPA) and Docosahexaenoic acid (DHA) are omega-3 fatty acids primarily found in some fish species. ALA (alpha-linolenic acid), another omega-3 fatty acid, is further found in the plant sources suchlike nuts and seeds [1].

EPA and DHA, are omega-3 fatty acids from fish oil, support cardiovascular health, maintain normal blood flow, and support platelet function [2]. They support optimal joint function and skin hydration and are essential components of neuronal, red blood cell membranes and vascular flow to the brain [3]. Thus, developing a proper method to concentrate omega-3 is interesting for the edible oil industry. Different methods, suchlike extraction, vacuum distillation and urea crystallization have been used widely and are known as conventional methods for omega-3 purification [4-6].

However, using these traditional methods for omega-3 purification or concentration has some restrictions suchlike the applying toxic solvents, high level of energy consumption and thermal decomposition of oil during concentration which limits the usage of these methods [6, 7]. Consequently, using a new efficient method which has lower restrictions is necessary.

Membrane separation technology is a new method widely used in the food industry in the last decades [8]. Lower energy consumption, compact structure, and lower investment cost lead to the applying membrane technology in oil and fat industries [9-11]. The implement of membranes was assessed in numerous studies including oil deacidification and degumming, color reduction and solvent recovery [12, 13].

Selective separation of certain compounds is related to the type of membranes. Polymeric membranes are widely used in different membrane separation due to ease of fabrication and increase economic profits [14, 15]. Although many kinds of polymers are available, a polymer should be chosen pursuant to the appropriate characteristics for the intended applications. Moreover, the structures of polymeric membranes and their morphologies may affect the separation process [16-18]. It is known that a denser structure or smaller pore size can enhance the size sieving performance of membranes, resulting in a more selective separation [19]. In particular, poly-

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vinylidene fluoride (PVDF) membranes are used in the fats and oil industry for degumming and deacidification. The higher demands for the application of PVDF can be attributed to the heat and chemical resistance, and hydrophobicity of PVDF than others (such as PVDF/100 – 5000 ppm PVA; polyvinyl alcohol cross-linked PVDF membrane [12].

In our previous study, we investigated the performance of prepared PVDF membranes in terms of the oil permeability and ω3-PUFA rejection through the membrane-based ultrafiltration system. Also, the effect of process pressure and temperature on ω3-PUFA, mechanisms of the membrane fouling and the effect of silica particles on the implement of PVDF composite membranes were studied [23]. Specifically, oil flux and omega-3 PUFA concentration. Moreover, the fouling on the membrane surfaces due to oil filtration was studied [18]. Following the aforementioned research, to investigate the effect of operating parameters (*i.e.*, temperature, pressure and stirring rate) on concentration percentage and permeate flux, Box–Behnken design was used. ANOVA method was performed to analyze the data, and finally, the optimized condition was determined [10].

According to our findings mentioned above, there was a dearth of studying the use of PVDF to fabricate an asymmetric membrane with a thin selective layer using phase inversion methods along with dry/wet technique. To enhance the separation performance of the PVDF membrane for omega-3 concentration, span-85 was used as a hydrophobic agent in the membrane preparation process. Scanning electron microscopy was used to study the morphology of the prepared membrane. The surface hydrophobicity of the membrane was evaluated by the contact test method. The capability of the membranes to concentrate omega-3 and the mechanisms of the membrane fouling was evaluated by the filtration of Lantern fish in a batch mode set-up.

2. MATERIALS AND METHODS

2.1. Materials

To fabricate the membrane, poly-vinyldene fluoride (PVDF) with a molecular weight of 275,000 Dalton, supplied by Sigma-Aldrich Chemical Company (St Louis, USA), was used as a polymer material of the membrane. N-methyl-2-pyrrolidone (NMP) with an analytical purity of 99.9% supplied by Merck & Company (Darmstadt, Germany) was used to provide the casting solution for membrane preparation. De-ionized water was used as a non-solvent agent. Span-85 with an HLB of 1.8 and purity of 99% was purchased from Sigma-Aldrich Chemical Company (St Louis, USA) and used as a hydrophobic surfactant to control the mechanism of membrane formation.

2.2. Membrane Preparation

In order to the formation of a porous structure in the sub-layer of PVDF membrane with a dens thin top-layer, the dry-wet technique and phase inversion method were applied. Firstly, span-85 with different weight percentages (1, 2, 3, and 4 wt %) was added to the NMP and stirred adequately to form homogeneous mixtures. Then, 5 g of preheated PVDF at 60 ºC was dissolved in NMP and stirred for 24 h to prepare a uniform solution and was degassed to eliminate bubbles. The sample was subsequently casted on a flat glass plate with a thickness of 300 µm by a film applicator. The casted film was placed in a vacuum oven with a temperature of 150 ºC for 2 min. Then it was moved to a deionized water coagulation bath for a phase inversion process, which resulted in the formation of porous structures in the sub-layer of a PVDF membrane. Finally, the formatted membrane was dried in a vacuum oven for 24 h at 60 ºC [10, 18, 23].

2.3. Membrane Characterization

The cross-sectional morphology of PVDF membranes was evaluated using scanning electron microscopy (SEM) (KYKY-EM3200, KYKY Technology Development Ltd., Beijing, China). All samples were immersed in liquid nitrogen to clean the un-failed cut. Then, the cut surface was coated with a thin golden film by a BAL-TEC SCD 005 sputter coater before analysis (BAL-TECAG, Balzers, Liechtenstein).

Hydrophobicity is the physical property of a molecule. Hydrophobic molecules (oils and fats) tend to be nonpolar and, thus, prefer other neutral molecules and nonpolar solvents. Hydrophobic molecules in water often cluster together, forming micelles. Water on hydrophobic surfaces will exhibit a high contact angle. Surface hydrophilicity/hydrophobicity of the PVDF membranes was specified by the contact angle measurement test using a camera color video contact angle instrument (model no. SSC-DC318P, Tokyo, Japan). Distilled water was dropped manually on the surface by a Gilmont micro-syring and the measurements were done instantly. Ten measurements were carried out at ten different sites and the average of the results was reported [10, 18, 23].

2.4. Omega-3 Poly Unsaturated Fatty Acid (PUFA) Concentration

To evaluate the efficiency of the membrane, the ω3-PUFA concentration process was performed in a batch mode using the flat sheet membrane test cell (stirred ultrafiltration cell; model 8050, Millipore) under a nitrogen atmosphere. The required pressure for oil filtration was supplied by a nitrogen cylinder. All experiments were carried out under stirring, which was adjusted by a magnetic stirrer. The setup vessel was charged with 20 mg of the oil and the filtration was continued until the desired permeated oil quantity was collected.

The oil flux (J_o) which permeates through the membrane was characterized using the following equation,

$$J_o = \frac{Q_o}{A \times \Delta t} \quad (1)$$

where Q_o is the volume of the permeated oil (L), A is the membrane area (m^2), and Δt is the sampling time (hr).

The model proposed by Hernia describes the different fouling mechanisms individually [20]. In this work, Hernia's model was used to characterize the most dominant fouling mechanism with an optimization approach for omega-3 PUFA concentration [10, 18, 23].

The Hernia model describes the four separate mechanisms that cause flux decline in membranes under constant pressure. These models are as follows

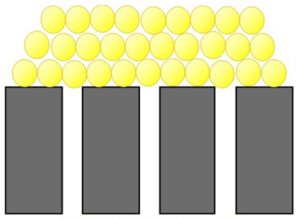
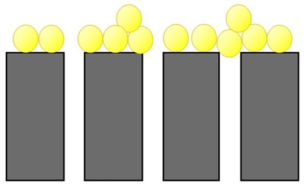
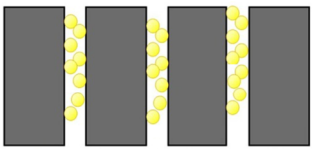
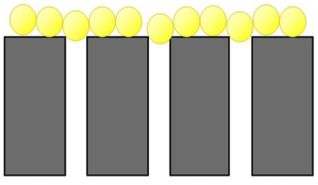
$$\frac{d^2t}{dV^2} = k\left(\frac{dt}{dV}\right)^n \quad (2)$$

K and n values are constant kinetic parameters; four types of fouling can occur in membrane filtration. Internal pore blocking (n = 1.5), complete pore blocking (n = 2), intermediate pore blocking (n = 1) and cake formation (n = 0) are the main fouling mechanisms.

Based on the flux expression (eq. 2), the variation of flux versus time can be written as follow,

$$J = \frac{dV}{A.dt} \quad (3)$$

Table 1: Mechanisms of Membrane Fouling with Corresponding Equation and Schematic Representation

n	Mechanism	Flux eq.	Linear Flux eq.	Schematic Representation
0	Cake formation	$J = \frac{J_o}{\left(2k_{cf}J_o^2 t + 1\right)^{0.5}}$	$\frac{1}{J^2} = \frac{1}{J_o^2} + k_{cf}t$	
1	Intermediate pore blocking	$J = \frac{J_o}{k_{ib}J_o t + 1}$	$\frac{1}{J} = \frac{1}{J_o} + k_{ib}t$	
1.5	Internal pore blocking	$J = \frac{J_o}{\left(k_{sb}J_o^{0.5} t + 2\right)^2}$	$\frac{1}{\sqrt{J}} = \frac{1}{\sqrt{J_o}} + k_{ib}t$	
2	Complete pore blocking	$J = J_o \exp\left(-K_{cb}t\right)$	$\ln\left(\frac{1}{J}\right) = \ln\left(\frac{1}{J_o}\right) + k_{cb}t$	

$$\frac{dJ}{dt} = -kJ\left(AJ\right)^{2-n} \quad (4)$$

According to each *n* value, the analytical expressions of the correspondence model are listed in Table **1**.

3. RESULTS AND DISCUSSIONS

3.1. Membrane Morphology and Hydrophobicity

SEM was used for the qualitative study of the membrane morphology and the results are shown in Figure **1**. SEM images demonstrated that all prepared PVDF membranes consist of a thin dense tap-layer that has the main role in the separation process and also a thick porous sub-layer which acts as a support of the membrane. Due to the immiscibility between water (non-solvent) and polymer, the precipitation phenomena were initiated by immersion of casted films in a water bath. The NMP solvent can be diffused in water and thereby the solvent/non-solvent exchange occurs at several points of the casted film. This incident is also known as dimixing process which its rate can significantly affect the final structure of the membranes. The dimixing continued until the solidification process due to the polymer precipitation was completed [21].

According to Figure **1**, increasing the concentration of span-85 in the membrane solution changed the final structure of the PVDF membranes. It seems that span-85 as a hydrophobic agent, influenced the rate of dimixing process. At the higher rate of dimixing, the final structure of the membrane showed larger micropores as well as higher porosity [22]. Hydrophobic characteristics of span-85 caused a decrease in the rate of water diffusion in the casted film, thereby slowing down the precipitation rate of the film. This resulted in a more compact structure with smaller pores and lower porosity presented in Figure **1**.

Table **2** also lists the contact angle test results for all prepared membranes. The contact angle of the membranes increased by increasing span-85 concentration. Increase of the contact angle showed that span-80 has a positive effect on the membrane performance which leads to the higher separation of ω3-PUFA from fish oil [10, 18, 23].

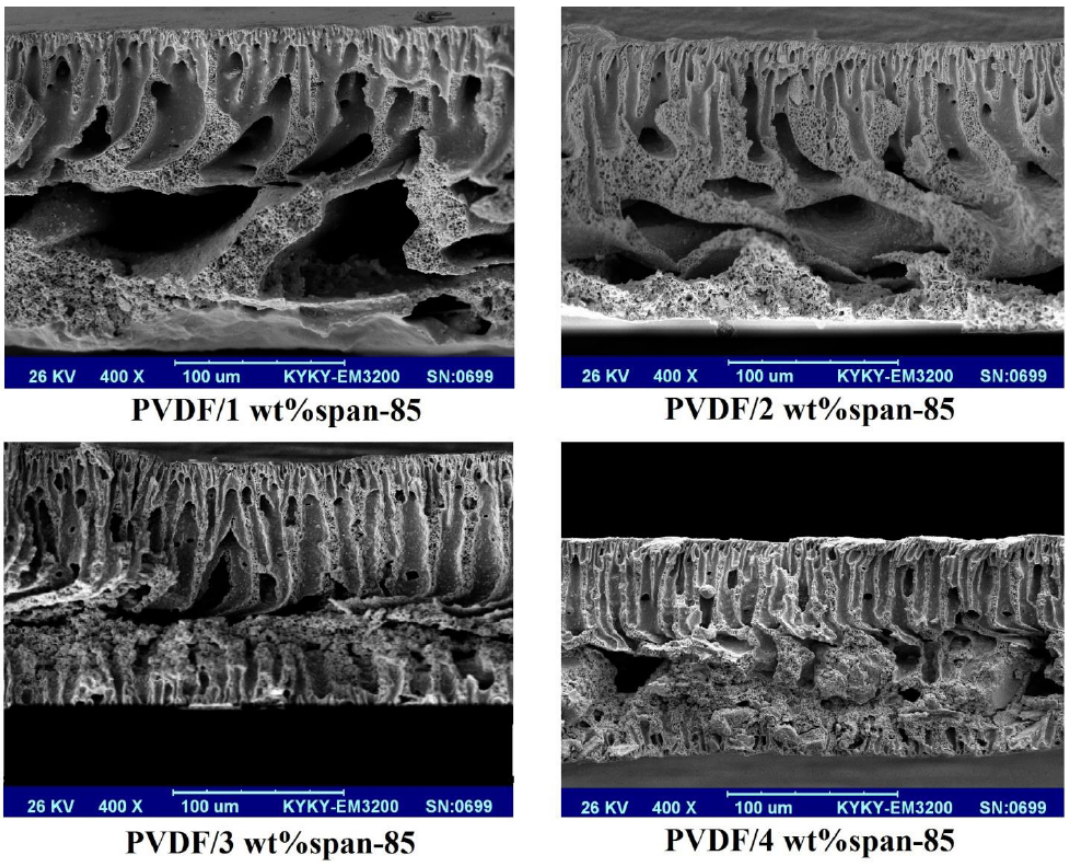


Figure 1: SEM images of PVDF membranes containing specific amount of span-85.

Table 2: Results of Contact Angle Measurement for Each Synthesized Membrane

	Neat PVDF	PVDF/1 wt% span-85	PVDF/2 wt% span-85	PVDF/3 wt% span-85	PVDF/4 wt% span-85
Contact angle	102.4	108.2	113.8	116.6	119.3

3.2. Omega-3 Concentration

The effect of span-85 contents on omega-3 concentration using PVDF membrane is listed in Table **3**. Results showed that the addition of span-85 to the PVDF matrix has a trade-off effect on omega-3 concentration. Increasing span-85 content up to 2 wt% improved the omega-3 concentration. The best membrane performance was obtained by PVDF/2 wt% span-85, which concentrated omega-3 by 42.64 wt%. In contrast, the higher level of span-85 (*i.e.*, 3 and 4 wt %) indicated a negative effect on membrane performance and resulted in a lower concentration rate of 37.54 wt%.

Figure **2** illustrates the variation of the oil flux in terms of time for each membrane. The oil flux decreased as the filtration time increased up to 180 min. The final steady-state flux for each membrane was much lower than the initial flux. The main flux decline occurred in the first 30 min and the intensity of flux variation decreased until it reached a steady amount.

Increasing the concentration of span-85 in the membrane decreased the flux at each time. The initial flux for the control PVDF membrane was 8.22 lit/m^2.h and decreased to 7.24 lit/m^2.h for the membrane that contained 4 wt % span-85. The presence of span-85 in the casting solution resulted in the formation of a membrane with a more compact structure and lower porosity.

Figure **3** depicts the experimental data and the theoretical way of the four mechanisms. The dominant mechanism for each membrane was determined based on the root mean square difference. Internal pore blocking (n = 1.5) occurs when the penetrant sizes are smaller than membrane pore diameters. This leads to the adsorption of particles on the inner pore walls that can decrease the membrane flux significantly. When

Table 3: Omega-3 Concentration Values (wt%) by Means of Membranes at 4 Bar, 30 ºC ad 100 rpm

Fatty acid	Neat PVDF	PVDF/1 wt% span-85	PVDF/2 wt% span-85	PVDF/3 wt% span-85	PVDF/4 wt% span-85
$\sum\omega_3$	38.21±0.76	40.56±0.78	42.64±0.84	41.31±0.63	37.54±0.71

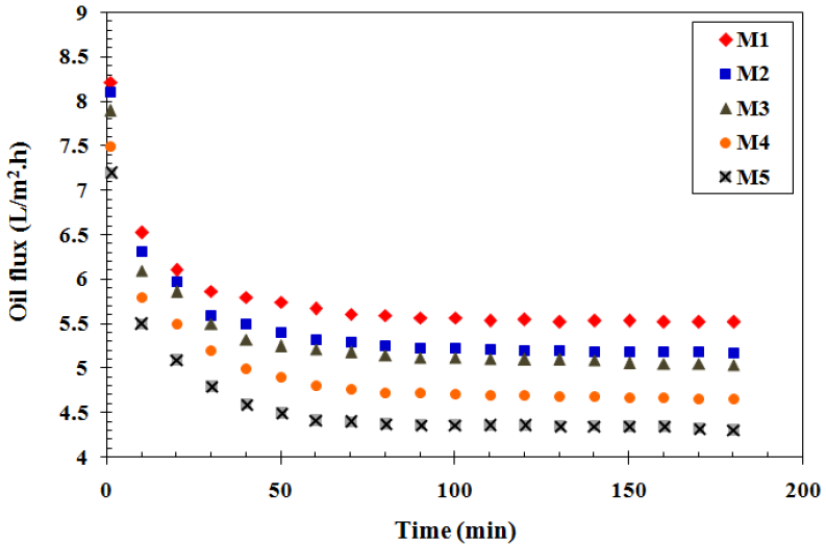


Figure 2: The variation of oil flux versus filtration time for each synthesized membrane.

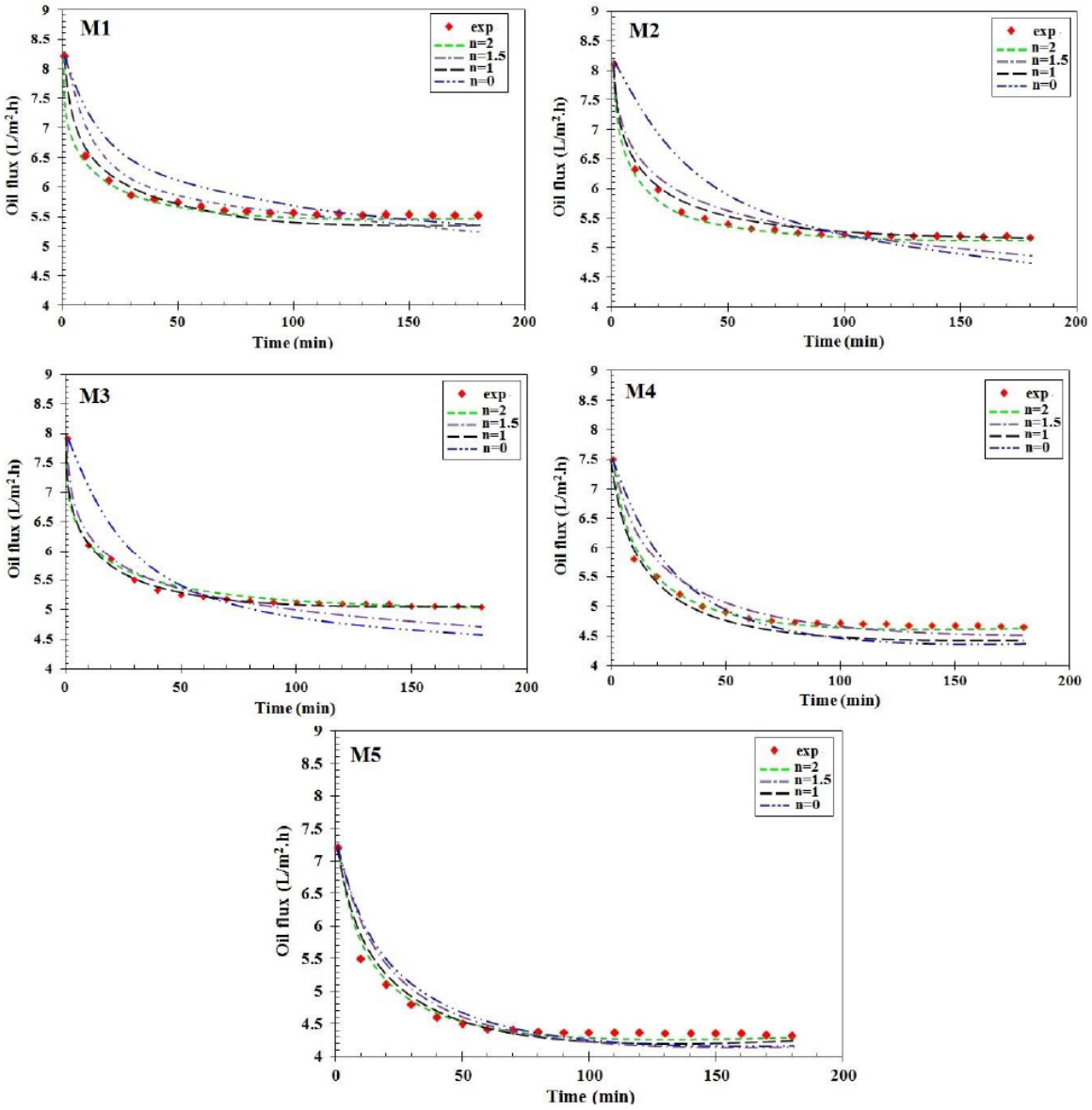


Figure 3: Predicted oil flux value compared to experimental data by means of each fouling model for all membranes.

the particle sizes are close to the surface pore size of the membrane, intermediate pore blocking (n = 1) occurs. Complete pore blocking (n = 2) happens when the penetrate size is larger than the surface pore size. In this case, particles blocked the membrane pores leading to the exponential flux decline. The last mechanism is cake formation (n = 0), which occurs when a layer of the particles is accumulated on the surface of the membrane. It seems that the more hydrophobic surface of the membrane increased the attraction force between the particles and resulted in higher cake formation.

As shown in Figure **3**, all mechanisms cause oil flux reduction. Table **4** lists the root-mean-square deviation (RMSD) for each mechanism in all synthesis membranes.

Based on the results of Table **4**, as span-85 content increased in the membrane, the cake formation mechanism became more serious. As mentioned in morphology studies, the porosity of the formatted membrane decreased as span-85 concentration increased. The pores with much smaller sizes can lead to the accumulation of larger particles on the surface of the membrane. Moreover, the AFM image from the surface of membranes showed that the intensity of fouling on the surface of the membranes was increased. Figure **4** showed that the thickness of the fouling on the membrane increased the span-85

Table 4: Root-Mean-Square Deviation (RMSD) between Calculated and Experimental Flux Data for Each Fouling Model

Membrane	Fouling model			
	Cake formation (n = 0)	Intermediate pore blocking (n = 1)	Internal pore blocking (n = 1.5)	Complete pore blocking (n = 2)
M1	2.453	0.801	1.241	0.120
M2	2.113	0.918	1.026	0.228
M3	1.718	0.713	0.918	0.212
M4	1.312	0.978	1.144	0.142
M5	0.803	0.927	0.889	0.158

M1 – M5 = fabricated membrane with different weight percent of span-85 (0, 1, 2, 3, and 4 wt %)

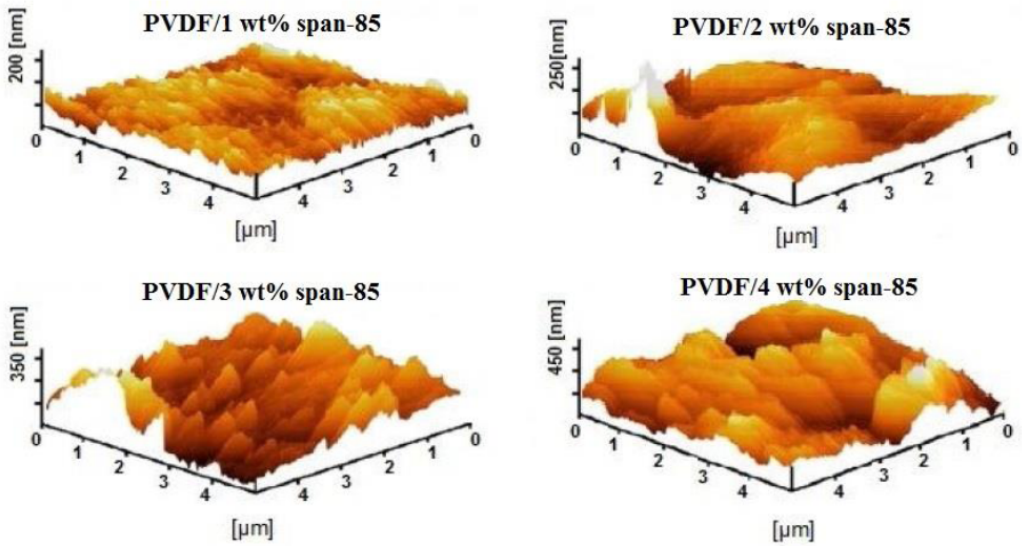


Figure 4: AFM image of fouled surface of each membrane containing span-85.

concentration. As mentioned above, a higher concentration of span-85 resulted in the formation of the membrane with higher surface hydrophobicity enhancing the PUFA concentration on the feed side. But, the higher hydrophobicity of the membrane caused more fouling on the surface.

The present results were consistent with our previous results reported in the supplementary [10, 18, 23].

4. CONCLUSION

The effect of span-85 on the performance of PVDF asymmetric membrane for omega-3 concentration from Lantern fish oil was evaluated in the batch mode system. Each membrane was prepared using the phase inversion method in the water bath at 0 ºC. The SEM images showed a porous morphology for each membrane with a thin selective top-layer. The porosity and the pore size of the membrane decreased as span-85 content increased in the PVDF matrix. Moreover, the results of the contact angle test revealed that the surface hydrophobicity of the membrane increased, and the highest omega-3 concentration was obtained with PVDF containing 2 wt % span-85. The extra addition of span-85 to the membrane matrix led to a lower omega-3 concentration. The fouling analysis of each membrane showed complete pore blocking as the main fouling mechanism that occurred in all membranes. It was shown that the more hydrophobic surface of the membrane resulted in higher cake formation.

For future studies, it can be suggested to evaluate the concentration of ω3-PUFA in this type of process by designing and fabricating a continuous system.

CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

ABBRIVATIONS

J_o	oil flux
Q_o	volume of the permeate oil (L)
A	the membrane area (m^2)
Δt	sampling time (hr)
t	Time (hr)
V	cumulative volume of filtrate (L)
k	constant
J	flux

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