Dry degumming of corn-oil for biodiesel using a tubular ceramic membrane

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Abstract

Gum removal from vegetable oils applied for diesel fuel is an essential process, since the presence of gum in direct use to the diesel engine might cause plugging in the engine filters, lines and injectors. In the biodiesel production, the use of crude vegetable oils without degumming might decrease conversion rate and hard to separate glycerol from biodiesel, during and after trans-esterification reaction respectively. Ultrafiltration is potential technology for degumming of crude vegetable oils, however since the oil constituents have nearly close molecular weight, the membrane degumming process is a difficult process. In order to understand the membrane degumming process in corn-oil, both experimental and computational works have been conducted. Dry degumming process of corn-oil has been carried out using a tubular ceramic membrane, and the effects of process conditions (temperature, trans-membrane pressure, cross flow velocity) and concentration polarization were investigated. Computational fluid dynamics simulation based on Galerkin finite element methods (FEM) has been carried out and used to predict the retention layer occurred during inside-out operation of membrane degumming. The numerical results show that the increasing of crossflow velocity lead to increase axial Reynolds number, and on the other hand decrease the thickness of the retention layer. In the x/R=1.0 position, the retention layer thicknesses were $\delta_p = 4.0 \times 10^{-4}$ m and $\delta_p = 1.7 \times 10^{-4}$ m, for Re=1000 and Re=10000, respectively. Moreover, the effect of increasing Schmidt number gives a significant retention layer thickness decline, i.e. from $\delta_p = 4.8 \times 10^{-4}$ m to $\delta_p = 2.2 \times 10^{-4}$ m, by using Sc=4x10^5 to Sc=1.3x10^6, respectively. The Schmidt numbers depends on the diffusion behavior of solute in the solvent, and has been concluded that wet-degumming process was a better method to prevent higher concentration polarization close to the membrane wall.

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1. Introduction

The potency for biomass-derived fuels have enhanced significantly, mostly driven by concerns for energy security and sustainability. Fossil fuel price steeply increase, in vice versa biofuel production cost gradually decrease by technology advancements. In the North and Latin America, ethanol production is dominating, while in Europe, the dominance is for biodiesel with a contribution up to 80% of total biofuel production [1]. Due to agricultural intensification more than 95% of the world biodiesel is produced from edible vegetable oil, especially corn harvested [2].

The possible options in the use of vegetable oils as diesel fuels are: (i) direct use of pure vegetable oils, (ii) esters of vegetable oils, and (iii) blends of oils or esters with diesel fuel. Crude or unblended vegetable oil has been used since the first development of diesel engine [3], however the presence of gumming materials called phospholipids might cause clogging in the engine filter, lines and injectors [4]. Phospholipids' content in the esterified and blended vegetable oil might also cause potential problem in depositing carbon within the engine [5]. In the enzymatic transesterification reaction, phospholipids are able to inactivate Lypozyme, therefore lowering the reaction rate and products [6]. Gum removal therefore becomes an essential process in the usage of vegetable oil in the biofuels. Unlike the conventional degumming process which employs several stages to remove gum from the oil, membrane filtration of crude vegetable oil or its micelle removes undesirable oil impurities in the single stage process [7], however since the oil constituents have nearly close molecular weight, the membrane degumming process is relatively difficult to obtain [8].

The phospholipids dissolved in crude oils typically as amphiphilic (micelle) molecules, which part of the molecule in these compounds is hydrophobic and the rest of the molecule is hydrophilic. In the organic solvent environment, they tend to form inverse micelle, where the lipophilic group is turned outside to the solvent and the lipophobic group is turned inside to form the core of the micelle, therefore called as inverse micelles [9]. Because of their unique ability to form well ordered molecular aggregates and structures, most of membrane-based separation process consider about this surfactant behavior [10].

In this paper, we studied the dry degumming process of corn-oil for biodiesel using a tubular ceramic membrane. Dry degumming was selected to obtain several key characteristics of pure corn-oil and its permeation through ultrafiltration membrane, the data furthermore was used as input in the computational study. The effect of process conditions (temperature, trans membrane pressure, cross flow velocity) and concentration polarization were investigated. Pure corn-oil has been selected as feed, considering the lowest phospholipids’ content of corn oil among other vegetable oils [11]. Computational fluid dynamics (CFD) simulation based on finite element methods (FEM) has been used to solve partial differential equations, combine Navier-Stokes equation in incompressible Newtonian fluid with Darcy’s equations for corn-oil flow models inside the tubular membrane. The computational result therefore use as an analytical method to study the membrane degumming performance.

2. Experimentation
2.1 Materials

Pure corn oil (Chang Chi Foodstuff Factory Co. Ltd., Taiwan) was used as fluid, and lecithin (Sigma Chemical Co.) was used as solute representation, added in a certain concentration by heated and stirred. Ethyl alcohol 95% (Echochemical, Taiwan), n-hexane (Seedchem Company Pty. Ltd., Australia), sodium hydroxide pellets (J.T. Baker Company, Sweden), and nitric acid 68-71% (Fisher ChemAlert Guide, USA) were used for membrane pre-treatment and re-conditioning.

A Filtanium™ tubular ultrafiltration inorganic membrane (Tami Industries, France) made from the ATZ (alumina/titania/zirconia) support layer and titania as filtrate layer was used for corn-oil degumming. The membrane
module is a single channel module, with length 600 mm, the outer diameter is 10 mm and the inner diameter is 6 mm. Membrane surface area is 0.011 m$^2$ with the pore size of 300 kDa.

2.2 Methods

2.2.1 Experimental set-up

Experimental work has been carried out using a tubular membrane system as shown in the Figure 1. The major components of the system are a 2.5 L feed tank, a circulating pump (GE Commercial Motors, ½ HP, AC motor, thermally protected), a tubular membrane module, and a concentrated recirculation line. Both permeate and concentrated streams were recycled back to 2.5 L open feed tank, equipped with a stirrer. This resulted in a stable feed concentration reading and allowed the unit to be operated continuously for long periods. A flowmeter was fitted to retentate line to measure the flow rate of this stream. The feed, permeate and retentate pressures were determined by pressure gauges. The feed line temperatures were controlled by a digital thermocouple device, and the water jacket temperature circulation temperature controlled by a batch circulator. The digital mass balance (Precisa Gravimetric AG, Switzerland) used to measure permeate flux and simultaneously connected to the PC to display a real time flux graphic in a worksheet program.

![Figure 1. Schematic diagram of experimental set-up.](image)

2.2.2 Membrane pre-treatment and regeneration

The pre-treatment for the new ceramic membrane was needed, used a solvent of high polarity to a non-polar solvent [12], using pure water, water + ethyl alcohol, pure ethyl alcohol, ethyl alcohol + n-hexane and pure n-hexane. Binary solutions of these solvents were used at concentration of 30%, 50% and 70% (v/v). The membrane was kept immersed in each of these nine solutions at room temperature, each for 12 hours (108 hours totally), with the following ratios: (1) pure water, (2) 70:30 of water : ethyl alcohols, (3) 50:50 of water : ethyl alcohols, (4) 30:70 of water : ethyl alcohols, (5) pure ethyl alcohol, (6) 70:30 of ethyl alcohols : n-hexane, (7) 50:50 of ethyl alcohols : n-hexane, (8) 30:70 of ethyl alcohols : n-hexane, and (9) pure n-hexane. Ethyl alcohol and n-hexane formed homogeneous solutions in all of the proportions used in the pre-treatment.
The membrane regeneration is a treatment for used membrane to generate properties as a new membrane or an alike new membrane. The acid-alkaline cleaning methods for membrane regeneration were proposed elsewhere [14, 15]. The procedure as follows: (1) immersed membrane in base solution (1% NaOH) at 60°C for 2 hours, (2) washed membrane using pure water, (3) immersed membrane in acid solution (1% HNO₃) at 60°C for 2 hours, and (4) washed membrane using pure water. After this procedure, the membrane is a new-like membrane, then a membrane pre-treatment before use it for filtration was needed.

2.3 Properties of fluid solutions

The physical properties of the vegetable oils may obtain from literature data [16, 17], however for better approximation, the properties of fluids and membrane were first defined by experimental data as a base value for simulation. The kinematic viscosity of solutions was measured using viscometer (Koehler Instrument Company Inc.). The quantities parameters data obtained from experiment, i.e.: fluid viscosity, fluid density, diffusion coefficient, Reynolds number, Schmidt number, concentration of phospholipids in the oil, pressure and flow rate of operations, were used as input variables in the mathematical model. The phospholipids concentrations were tested at 1-2wt%, the temperatures ranged from 40-60 °C, cross-flow velocities ranged from 4.6-5.2 m/s, and TMP was set at 105 kPa.

3. Computational method

3.1 CFD governing equations

The representation of inside-out process in tubular ceramic membranes with cylindrical coordinate systems is modelled and shown in Figure 1. The flow system was governed by equations conserving mass and momentum balance using finite different methods (FDM) [18] and improvement of FDM using control volume (SIMPLE algorithm based on Finite Volume Methods) [19], both use these equations to solve the reverse osmosis (RO) system of removing salt from water. In this paper, the 2D Galerkin finite element method (FEM) has been used for the equation given, as a representative model of removing phosphatides from vegetable oil.

![Figure 2. Cross section view of flow in a tubular permeable membrane.](image)

The governing equations are described as follow:

**Continuity equation:**

\[
\frac{\partial V}{\partial r} + \frac{V}{r} + \frac{\partial U}{\partial x} = 0
\]  

(1)

**Axial momentum equation:**

\[
\rho \left( V \frac{\partial U}{\partial r} + U \frac{\partial U}{\partial x} \right) = - \frac{\partial P}{\partial x} + \mu \left[ \frac{\partial}{\partial r} \left( \frac{1}{r} \frac{\partial}{\partial r} \left( rU \right) \right) + \frac{\partial^2 U}{\partial x^2} \right]
\]  

(2)

**Radial momentum equation:**

\[
\rho \left( V \frac{\partial V}{\partial r} + U \frac{\partial V}{\partial x} \right) = - \frac{\partial P}{\partial r} + \mu \left[ \frac{\partial}{\partial r} \left( \frac{1}{r} \frac{\partial}{\partial r} \left( rV \right) \right) + \frac{\partial^2 V}{\partial x^2} - \frac{V}{r^2} \right]
\]  

(3)

**Mass transfer or solute transport equation:**
\[ U \frac{\partial C}{\partial x} + V \frac{\partial C}{\partial r} = D \left( \frac{1}{r} \frac{\partial}{\partial r} \left( r \frac{\partial C}{\partial r} \right) + \frac{\partial^2 C}{\partial x^2} \right) \]  
(4)

3.2 Boundary conditions

The above model requires the implementation of boundary conditions given below:

**Inlet boundary condition**, \( x = 0 \)

\[ U(0, r) = 2U_{\text{max}} \left( 1 - \left( \frac{r}{R} \right)^2 \right), \text{ where } U_{\text{max}} = \frac{2}{3} U_{\text{ave}} \]  
(5)

\[ V(0, r) = 0, \]  
(6)

\[ C(0, r) = C_0 \]  
(7)

**Outlet boundary condition**, \( x = L \)

\[ \frac{\partial U(L, r)}{\partial x} = 0; \frac{\partial V(L, r)}{\partial x} = 0; \frac{\partial C(L, r)}{\partial x} = 0 \]  
(8)

**At axisymmetric axis**, \( r = 0 \)

\[ \frac{\partial U(x, 0)}{\partial r} = 0; V(x, 0) = 0; \frac{\partial C(x, 0)}{\partial r} = 0 \]  
(9)

**At membrane surface or permeable wall**, \( r = R \)

\[ \frac{\partial U(x, R)}{\partial r} = 0; V(x, R) = V_w(x) \]  
(10)

\[ V_w(x)C(x, R) = D \frac{\partial C(x, R)}{\partial r} \bigg|_{\text{wall}} \]  
(11)

\( V_w(x) \) is determined by Darcy equation, using resistance-in-series model:

\[ V_w(x) = \frac{\Delta P}{\mu (R_m + R_p)} \]  
(12)

where \( \Delta P = P - P_e \), the resistances \( R_p \) obtained using the following equation:

\[ R_p = \int_{R-\delta_p}^{R} r_p \, d\delta = r_p \delta_p \]  
(13)

when \( \delta_p \) is similar to the distance from the membrane surface where the value of concentration is similar to the inlet value of concentration, so the equilibrium between convective and diffusive fluxes is reached when \( \frac{C - C_0}{C_0} < 0.001 \), adapted from [19].

The non-dimensional quantities are:

\[ \text{Re} = \frac{\rho U_{\text{ave}} 2R}{\mu} \]  
(14)

\[ \text{Re}_w = \frac{\rho V_{w0} 2R}{\mu} \]  
(15)

\[ Sc = \frac{\mu}{\rho D} \]  
(16)
In this work, the Reynolds numbers were tested at 1000, 3000, 7000 and 10000. The Schmidt numbers were tested at 400,000; 700,000; 1,000,000; and, 1,300,000.

4. Results and discussion

4.1 Corn-oil permeation

Figure 3 shows permeation of pure corn-oil and corn-oil with the addition of 1 wt% lecithin under TMP of 105 kPa, T=50-60 °C, and CFV of 4.6 – 5.2 m/s. It has been shown that permeate flux decline due to the presence of oil impurities, the effect was larger in the higher concentration of phospholipids.

![Figure 3](image)

Figure 3. Flux decline due to the presence of higher phospholipids content in corn-oil (TMP of 105 kPa, T=50-60 °C, and CFV = 4.6 – 5.2 m/s): pure corn oil + 1wt% lecithin (·) and pure corn oil (○).

As mentioned, the properties of solutions were obtained from experimental data and used as variables in the CFD modelling. Table 1 summarize the density and viscosity of fluids measured.

<table>
<thead>
<tr>
<th>Fluids</th>
<th>T (°C)</th>
<th>Kinematic viscosity (ν) cSt</th>
<th>Dynamic viscosity (μ) cP</th>
<th>Density (ρ) g/mL</th>
<th>kg/m³</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hexane</td>
<td>40</td>
<td>0.3661</td>
<td>3.661x10⁻³</td>
<td>0.2518</td>
<td>0.6878</td>
</tr>
<tr>
<td>Pure corn oil</td>
<td>40</td>
<td>27.091</td>
<td>2.709x10⁻⁵</td>
<td>25.294</td>
<td>0.9334</td>
</tr>
<tr>
<td>Corn oil + 2wt% lecithin</td>
<td>40</td>
<td>27.873</td>
<td>2.787x10⁻⁵</td>
<td>28.409</td>
<td>1.0193</td>
</tr>
<tr>
<td>Pure corn oil</td>
<td>50</td>
<td>19.086</td>
<td>1.909x10⁻⁵</td>
<td>18.006</td>
<td>0.9435</td>
</tr>
<tr>
<td>Corn oil + 2wt% lecithin</td>
<td>50</td>
<td>21.236</td>
<td>2.124x10⁻⁵</td>
<td>25.500</td>
<td>0.9183</td>
</tr>
<tr>
<td>Corn oil + 1wt% lecithin</td>
<td>60</td>
<td>14.351</td>
<td>1.435x10⁻⁵</td>
<td>12.972</td>
<td>0.9039</td>
</tr>
</tbody>
</table>
4.2 Membrane resistances

The resistance in series concept that shows the whole pressure flux behavior observed during ultrafiltration membrane was used as mass transport calculation through the membranes [20]. For a real feed in actual operation, the flux ($J$), in the term of volumetric flux (dividing by permeate and fluid density, respectively), can be written as the applied transmembrane pressure ($\Delta p$) divided by total resistance of the membrane in a certain fluid viscosity ($\mu$), i.e. intrinsic membrane resistance ($R_m$), resistance caused by fouling ($R_f$) and resistance due to the concentration polarization and polarized boundary layer ($R_p$) as follows:

$$ J = \frac{\Delta p}{\mu (R_m + R_f + R_p)} $$  \hspace{1cm} (18)

A ceramic membrane resistance ($R_m$) in this experiment can be calculated from Equation 18 using pure hexane as the feed through the clean membrane using the equation found elsewhere [21]. The equation can be rewritten as:

$$ J_h = \frac{\Delta p}{\mu_h R_m} $$  \hspace{1cm} (19)

in which $J_h$ is the hexane flux through the clean membrane, and $\mu_h$ is the viscosity of the hexane. The calculation of hexane flux is the flux at steady state. Table 2 summarized the calculated membrane resistance, that comparable with the values provided by literature.

<table>
<thead>
<tr>
<th>$\mu_h$ (Pa.s)</th>
<th>$J_h$ at steady state (LMH)</th>
<th>Per membrane area (m/s)</th>
<th>$\Delta p$ (kPa)</th>
<th>$R_m$ (1/m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.518x10^-4</td>
<td>48.9</td>
<td>1.5x10^-4</td>
<td>70</td>
<td>1.85x10^-12</td>
</tr>
<tr>
<td>2.518x10^-4</td>
<td>55.6</td>
<td>1.7x10^-4</td>
<td>105</td>
<td>2.45x10^-12</td>
</tr>
</tbody>
</table>

Average $R_m = 2.15x10^{12}$ m^-1

Compare with values from literatures

<table>
<thead>
<tr>
<th>$R_m$ (1/m)</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.98x10^{11} – 3.93x10^{11} m^-1</td>
<td>[21]</td>
</tr>
<tr>
<td>3x10^{12} – 5x10^{12} m^-1</td>
<td>[22]</td>
</tr>
</tbody>
</table>

Resistance due to fouling ($R_f$) is important to be known to understand fouling occurred in the membrane, calculated from the pure hexane flux through the membrane after processing and rinsing with hexane, as this latter procedure removes the reversible barriers (concentration polarization and polarized layer).

The equation can be written as:

$$ J_{h'} = \frac{\Delta p}{\mu_h (R_m + R_f)} $$  \hspace{1cm} (20)

in which $J_{h'}$ is the hexane flux through the fouled membrane.

Then, from the term of total resistance:

$$ R = R_m + R_f + R_p $$  \hspace{1cm} (21)

the resistance due to the concentration polarization and polarized layer ($R_p$) can be defined. The resistances calculated from Equation 18-21, are presented in the Table 3.

<table>
<thead>
<tr>
<th>TMP (kPa)</th>
<th>$R_m$ (m^-1)</th>
<th>$R_f$ (m^-1)</th>
<th>$R_p$ (m^-1)</th>
<th>$R$ (m^-1)</th>
</tr>
</thead>
<tbody>
<tr>
<td>105</td>
<td>2.45x10^-12</td>
<td>12.95x10^-12</td>
<td>6.4x10^-11</td>
<td>16.04x10^-12</td>
</tr>
</tbody>
</table>

Other variables needed as input in computational work summarizes in Table 4 were measured from experiments. Here, an assumption was taken for diffusivity coefficient of phospholipids, this number was taken from the aggregate diffusivity of soybean lecithin micelle in the experiment [23]. Then, from this diffusivity coefficient, the
Schmidt number was calculated (Equation 16).

<table>
<thead>
<tr>
<th>Quantity</th>
<th>Value</th>
<th>Note</th>
</tr>
</thead>
<tbody>
<tr>
<td>$R_m$</td>
<td>$2.15 \times 10^{12} \text{ m}^{-1}$</td>
<td>Intrinsic membrane resistance</td>
</tr>
<tr>
<td>$U_{ave}$</td>
<td>5.2 m/s</td>
<td>Cross flow velocity, at flow rate 8.9 L/min (membrane cross section area is 28.27 mm$^2$)</td>
</tr>
<tr>
<td>$V_w(x)$</td>
<td>$1.34 \times 10^6 \text{ m/s}$</td>
<td>Local permeation flux, at 0.44 LMH</td>
</tr>
<tr>
<td>$C_0$</td>
<td>1 wt%</td>
<td>Concentration of phospholipids</td>
</tr>
<tr>
<td>$e$</td>
<td>$4 \times 10^{-3} \text{ m}$</td>
<td>Membrane thickness</td>
</tr>
<tr>
<td>$R$</td>
<td>$6 \times 10^{-3} \text{ m}$</td>
<td>Radius of the membrane</td>
</tr>
<tr>
<td>$L$</td>
<td>0.6 m</td>
<td>Membrane length</td>
</tr>
<tr>
<td>$\mu$</td>
<td>$0.012972 \text{ kg m}^{-1} \text{s}^{-2}$</td>
<td>Dynamic viscosity of the oil</td>
</tr>
<tr>
<td>$\rho$</td>
<td>903.9 kg m$^{-3}$</td>
<td>Density of the oil</td>
</tr>
<tr>
<td>$Re$</td>
<td>4,350</td>
<td>Reynolds number (Eq. 14)</td>
</tr>
<tr>
<td>$Re_w$</td>
<td>0.1</td>
<td>Reynolds number near membrane surface (assumption)</td>
</tr>
<tr>
<td>$D$</td>
<td>$3.4 \times 10^{-11} \text{ m}^2 \text{s}^{-1}$</td>
<td>Diffusivity coefficient of lecithin [23]</td>
</tr>
<tr>
<td>$Sc$</td>
<td>422,058</td>
<td>Schmidt number (Eq. 16)</td>
</tr>
</tbody>
</table>

### 4.3 Prediction of retention layer thickness

Retention layer within the flow channel only occurs in a small vicinity near the membrane surface. The retention developed shortly after the feed flow flew into the fluid flow channel. The flow in an axisymmetric tubular membrane of length $L=2*R$ was simulated. The simulation has been done for axial Reynolds number between 1,000 and 10,000, for Schmidt number between 400,000 and 1,300,000 and using fixed value of wall Reynolds number of 0.1.

Figure 4 shows the variation of concentration polarization growth along the tube length. As it is shown in the figures, increasing Reynolds number by increasing inlet axial velocity can decrease the retention layer growth. Higher Reynolds number can improve the performance of a tubular membrane. It is also shown that the retention layer growth was lower with increasing of Re number. For example, in the $x/R=1.0$ position (middle of the membrane channel), the retention layer is about $4.0 \times 10^{-4} \text{ m}$ for flow velocity $Re=1000$, and by increasing flow velocity to $Re=10000$, the retention layer was decreased to approximately $1.7 \times 10^{-4} \text{ m}$.

Figure 5 shows that increasing Schmidt number might also decrease the retention layer thickness. In the case of flow velocity $Re=1000$ for instance, using solution liquid with $Sc=4 \times 10^5$, produced retention layer of about $4.8 \times 10^{-4} \text{ m}$. By increasing $Sc$ number of the feed to $Sc=1.3 \times 10^6$, the retention layer was decreased to approximately $2.2 \times 10^{-4} \text{ m}$. $Sc$ number depends on the diffusion of the particle in the solvent, hence it is important to choose a suitable solvent to improve the membrane degumming process. The computational result shows that wet degumming using solvent might transform the phospholipids in the aggregate micelle form, therefore easy to reject and less concentration polarization layer might increase flux through the membrane.
Figure 4. Effect of Reynolds number on the concentration boundary layer thickness in the function of the axial dimensionless position (x/R) next to membrane surface (R=0.999) for Sc=400,000, Re=0.1.

Figure 5. The prediction of retention layer thickness occurred in certain Reynolds number and Schmidt numbers in x/R=2.

5. Conclusions
Mathematical model of membrane degumming inside a tubular ceramic membrane was developed and used to predict corn-oil velocity profiles and phospholipids concentration boundary layer on the membrane wall. The solute equation is mathematically expressed using 2D convective diffusion equation in cylindrical coordinate system. The inside-out process ultrafiltration membrane was modeled, and supporting experimental work has been done to obtain initial variables in a certain operating condition, membrane and fluid properties. The FORTRAN
codes were developed using PDE2D program, employing finite element scheme with Galerkin method. The developed numerical model successfully predicts the fundamental mechanisms involved in permeate flux decline during crossflow filtration.

The numerical results show the increasing crossflow velocity enhances axial Reynolds number, and leads to decrease the thickness of the retention layer, for instance the retention layer was decreased from $\delta p = 4.0 \times 10^{-4}$ m to $\delta p = 1.7 \times 10^{-4}$ m, while increasing flow velocity of $Re = 1000$ to $Re = 10000$ ($x/R = 1.0$). The effect of Schmidt number increase also contributes to significant retention layer thickness decline. By increasing Sc number of the feed solution from $Sc = 4 \times 10^5$ to $Sc = 1.3 \times 10^6$, the retention layer was decreased from $\delta p = 4.8 \times 10^{-4}$ m to $\delta p = 2.2 \times 10^{-4}$ m. The Schmidt numbers depends on the diffusion behavior of solute in the solvent, and has been concluded that wet degumming process employing solvent is better to prevent higher concentration polarization close to the membrane wall than dry degumming.

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References