Mass Transport Models in Organic Solvent Nanofiltration: A Review

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ABSTRACT

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Membrane technology has been gradually used as an alternative to the conventional separation and purification method in various industries. In recent years, solvent-stable nanofiltration or organic solvent nanofiltration has becoming practicable through the development of solvent-stable commercial polymeric membranes. Organic solvent nanofiltration has a great potential to replace the conventional energy-demanding process such as distillation due to its ability of separating organic solvents and solutes on a molecular level without phase change and operation at relatively mild temperature. Predicting the performance of such membrane separations is crucial in the process design. Important performance indicator such as the permeate flux and the rejections are strongly related to the fluid dynamics, mass transfer and solute-solvent-membrane interactions. The aim of this paper is to review and assess the transport models of solute and solvent transport relevant to organic solvent nanofiltration. The link between concentration polarization and the hydrodynamics in various configurations are discussed. The effects of process variables on membrane performance and solute-solvent membrane interactions are also reviewed.

Keywords:
Organic solvent nanofiltration; parameter estimation; concentration polarization; transport model

1. Introduction

Towards the efforts of purifying and separating chemical mixtures, various methods have been investigated and discovered. As of current, the techniques employed in the industries to refine chemicals are by extraction, distillation, chromatographic, electrophoresis and crystallization techniques [1]. Although these methods of refining are considered feasible in the current chemical industry, the methods require a significant amount of energy, for example, during distillation process as well as other processes [1]. From the in-depth review by Szekely et al., [2], it was found that the
energy requirement for distillation is at least 5000 times higher as compared to membrane processes for every 10 million kg of solvent generated per year. As the conventional refining method has been found decades ago, membrane separation methods have been proposed in the literatures, which were found to be a viable solution to address the problem of energy expenditure [2]. Membrane separation process (MSP) is currently actively used in purifying water and air[3,4]. Despite the early discovery of this method, MSP is currently still in its development stage for the separation of organic chemicals [5]. In the MSP, there are currently four developed ranges of filtration which are mainly dependent on the pore size of the membrane. These are conventional filtration which ranges from 100 µm to 10 µm pore size, microfiltration (10 µm to 0.1 µm), ultrafiltration (0.1 µm -0.005 µm) and reverse osmosis (0.0003 µm - 0.005 µm) [6]. Nanofiltration was investigated by researchers to be in the range between ultrafiltration and reverse osmosis [6]. The flowrate of solute and solvent through membranes having pore size in the range equal or larger than ultrafiltration are usually described by Darcy’s Law, or commonly known as pore-flow model [7]. Pore-flow model can be defined as fluid velocity proportional to the pressure gradient and inversely proportional to fluid viscosity. The movement of solute and solvent for membranes having a relatively smaller pore size, such as reverse osmosis are commonly described by Fick’s law, or commonly known as solution-diffusion model [7]. Solution-diffusion model relates the diffusive flux to the concentration of solution. As nanofiltration having the pore size in between that of ultrafiltration and reverse osmosis, nanofiltration membranes are usually investigated and described by both pore flow model and solution-diffusion model. In order to obtain the best correlation to the data obtained through nanofiltration process, thermodynamic models were also employed to describe the permeation flux of solute and solvent in nanofiltration membranes [6,7].

The use of membranes for organic solvents is commonly known as organic solvent nanofiltration (OSN) or solvent resistant nanofiltration (SRNF) in the field of membrane separation research. Organic solvent is a carbon-based chemical which is able to dissolve or disperse one or more chemical substance. Aqueous nanofiltration membrane such as cellulose acetate membrane, for example, can be easily dissolved by organic solvent, therefore, render it unusable in the separation process of organic solvents. Therefore, researchers had conducted investigations and created membranes which are more durable and resistant to organic solvent, and are known as organic solvent nanofiltration (OSN) membranes [2,8].

However, in the course of enabling MSP in refining chemicals, various factors which impede its implementation in the industry have been thoroughly considered. Membrane fouling is one of the issues that have been investigated by researchers in the recent decade [9-12]. Membrane fouling is caused by a phenomenon known as concentration polarization, where solutes accumulate at the membrane interface, forming a layer of solutes during MSP [13]. From a study by Shi et al., [14], the effects of membrane fouling on organic solvent nanofiltration were investigated. Membrane fouling has been considered to reduce the permeance especially at high solute concentrations. A higher concentration of solute results in a higher osmotic pressure needed for the nanofiltration process. Subsequently, it reduces the effective driving force across the membrane.

Besides that, the effect of charges on organic solvent nanofiltration also has been investigated. In a study by Yao et al., [15], it was found that charge repulsion effect promotes a higher rejection of positively charged membrane towards positively charged organic dyes. But, the effect of charge interaction was not as significant as in aqueous system due to the low dissociation of ions in organic solvent [15]. Other issues that have been studied are the low volumetric flux of permeate, the instability of membranes towards organic chemicals, and the selectivity of the membrane towards chemical compounds[2].
In order to provide solutions to the problems, the prediction of the membrane separation performances is essential [2,16]. The prediction and quantification of the membrane separation performance will enlighten the researchers on the approaches that should be taken to improve the membranes. In this paper, the current researches on the use of different transport models to predict mass transfer in organic solvent systems were reviewed. Besides that, the effects of concentration polarization on the transport processes were also briefly discussed.

2. Membranes for OSN

The typical materials for the construction of OSN membranes are polymers and inorganic compounds [4]. These materials are usually prepared in the form of dope polymer solution. The dope solution can be used to synthesize two main types of polymeric membranes; namely integrally skinned asymmetric (ISA) membranes, and thin film composite (TFC) membranes. ISA membranes are prepared through phase inversion technique [4] and TFC membranes are by interfacial polymerization or dip-coating method [17].

For ISA membranes, the synthesis process involves the precipitation of casting solution into two phases; one which is rich in polymer that constitutes to the matrix structure of the membrane, and another is polymer-poor, where the liquid forms the membrane pores [4]. The thermodynamic condition of the system, solvent and non-solvent exchange during the formation the polymeric membranes deeply affects the membrane morphology. The skin layer of the membrane structure as can be seen from Figure 1. (a), for example, is thicker and denser when a dope solution with a high concentration of polymer is used. This results in a higher selectivity but a lower permeability of the feed solvent [18]. The polymers for ISA membranes are polypropylene, polyimide, polysulfone, polyethersulfone, and polyvinylidene fluoride [19].

TFC membrane consists of a separating layer cast on a porous support ultrafiltration (UF) membrane. The mechanical stability of the membrane is dependent on the type of porous polymer support [20]. The types of polymer for the porous supports are the same as for ISA membrane. The type of top layer on the other hand affects the permeability of apolar solvents [2]. Some of the examples of polymers casted on the porous support are acyl chlorides [21,22], polyethylene imine [23], diamine [24], polydimethylsiloxane [25], and polyvinyl alcohol [26]. The type of polymers used for synthesis of membrane affects the permeability and selectivity of a chosen feed solvent differently, whether it is a protic, aprotic, polar or apolar solvent. The interaction between the feed solute-solvent and the membrane polymer during the nanofiltration process directly impacts the permeate flux and rejection. The interaction can be due to the sorption or solubility of the solute-solvent and polymer [27]. In order to describe the interactions during membrane nanofiltration in the form of equation, various models have been introduced. The diffusivity of the solute solvent in the polymer has been investigated in the form of parameter known as diffusion coefficient [27]. The
solubility on the other hand are commonly calculated by solubility parameter [28]. The product of both solubility parameter and diffusion coefficient yields permeability.

3. Transport Model

The transport models that are currently being actively applied in the prediction of membrane performance are irreversible thermodynamics models, solution-diffusion models, and pore-flow models [2,16]. From the literatures, the irreversible thermodynamics transport models can be used for the description of solute and solvent transport of nanofiltration membranes. The irreversible thermodynamics models are namely Kedem-Katchalsky model [29]

\[
J_v = L_p(\Delta P - \sigma_i \Delta \pi)
\]

(1)

\[
J_s = P_i \Delta c_i + (1 - \sigma_i)J_v \tilde{c}_i
\]

(2)

where \(J_v\) is the volumetric flux of solvent, \(J_s\) is the volumetric flux of solute, \(P_i\) is the permeability coefficient of the solute \(i\), \(\sigma_i\) is reflective coefficient, \(L_p\) is the mechanical filtration coefficient and \(\tilde{c}_i\) is the average solute concentration in the membrane, and Spiegler-Kedem model [30]

\[
J_v = L_p(\Delta P - \sigma_i \Delta \pi)
\]

(3)

\[
J_s = -P_i \Delta x \frac{d}{dx} c_i + (1 - \sigma_i)J_v \tilde{c}_i
\]

(4)

However, the models do not consider the transport processes within the membranes [2]. On the other hand, solution-diffusion models are suitable to describe the solute and solvent transport when the mechanism of transport processes within the membrane is known[31,32]. Eq. (5) and Eq. (6) shows the solution diffusion model.

Solvent flux

\[
J_i = \frac{D_j K_j}{l} \left( c_{io} - c_{il} e^{\left( -\frac{\nu (\rho_o - \rho_l) \Delta p}{RT} \right)} \right)
\]

(5)

Solute flux

\[
J_j = \frac{D_j K_j}{l} \left( c_{jo} - c_{jl} \right)
\]

(6)

where the subscript \(i\) and \(j\) indicates component \(i\) (solvent) and component \(j\) (solute) respectively. The product of diffusion coefficient, \(D\) and sorption coefficient, \(K\); \(DK\) is also usually referred as permeability coefficient, \(P\). \(l\) is the thickness of the membrane. \(c_{io}\) is the concentration of component \(i\) in the feed side, and \(c_{il}\) is the concentration of component \(j\) in the permeate side of the membrane. By including the effect of viscous flow, solution-diffusion with imperfections model [33] was formed

\[
J_i = \frac{D_j K_j c_{io} v_i}{l \nu_l R_T} (\Delta p - \Delta \pi) + \frac{c_{io} B_0}{\eta l} \Delta p
\]

(7)
where \( B_0 \) is the specific permeability of the membrane (\( m^2 \)), \( \eta \) is the viscosity of the permeate mixture (\( Pa.s \)), \( R_g \) is the gas constant (\( J \ mol^{-1} K^{-1} \)), \( v_i \) is the molar volume of component i (\( m^3 \ mol^{-1} \)).

Meanwhile, pore-flow model (Eq. (8)) is suitable for the description of transport processes in porous membranes such as microfiltration membranes and ultrafiltration membranes [2,16].

\[
J = \frac{k(p_o - p_l)}{l} \tag{8}
\]

where \( k \) is the permeability coefficient, \( p_o \) is the pressure at the feed side, \( p_l \) is the pressure at the permeate side, \( l \) is the thickness of the membrane. From Eq. (8), a more popular derivative of pore-flow model, Hagen-Poiseuille model was derived

\[
J = \frac{\varepsilon r^2 \Delta P}{8 \mu \tau \Delta x} \tag{9}
\]

where \( r, \varepsilon, \tau \) and \( \Delta x \) represents pore size, membrane porosity, membrane tortuosity and membrane thickness respectively.

4. Membrane Performance Prediction by Transport Models

In the development of membrane processes, several stages of modeling were involved starting from lab scale till the implementation in the actual industrial scale [2]. Parameter estimation is performed in the membrane scale, through non-linear regression of transport models against the experimental data in solute and solvent system. The regression is performed by a certain selected algorithm which minimizes the error between the expected data and the model output, which results in the estimation of model parameters [16]. When the model parameters are available, modeling can be performed to predict the performance of the membrane, as shown in the process flow in Figure 2.

![Fig. 2. A typical process flow for a modeling process [2,16,34]](image-url)
The selection of models for the estimation and prediction depends on the type of membrane as well as the operating conditions [34]. Some researchers compare different types of models for a selected solute-solvent system, in order to obtain the best model to describe the separation process [16,35].

Postel et al., [36] had found that the changes in the solvent properties resulted from the mixture of different solvents had affected the interactions between membrane, solvent and solute. The interaction causes a different flux and retention trends. In their study, Maxwell-Stefan model which includes effect from the interactions was able to describe the solvent and solute fluxes successfully [36]. Campbell et al., [37], on the other hand, had emphasized on the effect of pore size on the separation process due to the use of hybrid metal organic/polyimide ultrafiltration membranes in their experiments. In order to include the effect of pore size in the calculations, Nernst-Planck equation was described as a function of pore size to estimate the rejection of solutes. In another study by Shi et al., [38], classical solution-diffusion model and film theory were used to describe the effect of mass transfer. Based on the model implementation by using OSN Designer software tool, the necessary parameters such as pressure drops and mass transfer were successfully obtained through experimental data regression [38]. See Table 1.

### Table 1

| Model                                  | Membrane          | Solute/solvent                                      | Reference |
|----------------------------------------|-------------------|----------------------------------------------------|-----------|
| Generalized Maxwell-Stefan model       | Polyimide         | Toluene, isopropanol, methanol, decane, dodecane, tetradeacne, hexadecane, octadecane, docosane, tetracosane | [36]      |
| Nernst-Planck pore flow equation       | Polyimide         | Acetone toluene                                    | [37]      |
| Classical solution-diffusion with film theory | Polyimide      | Ethyl acetate, sucrose octaacetate                | [38]      |
| Nernst-Planck equation with Hagen-Poiseuille model | TiO₂/ZrO₂ | Polystyrene, THF                                    | [39]      |
| Solution-diffusion, pore-flow, reduced solution-diffusion imperfection models | Polyimide, composite PDMS | n-hexane, ethanol, rapeseed oil, olive oil, babassu oil, oleic acid, lauric acid | [35]      |
| Solution-diffusion                     | Composite PDMS    | Dodecene, polyethylene glycol ether                | [40]      |
| Maxwell-Stefan with Flory-Huggins model | Composite PDMS/PAN | p-cymene, d-limonene, α-pinene, canola oil, n-hexane, n-heptane | [41]      |

In attempt to implement an existing nanofiltration model to OSN, a model which was developed by Bowen and Welfoot [42] to describe the transport of uncharged solute in aqueous application was studied by Blumenschein et al., [39]. The effects of pore size dependent viscosity and pore size distribution of ceramic membrane were included for the model adaptation to OSN. From the results obtained from their study [39], it was found the transport model based on the model of Bowen and Welfoot [42] was able to predict the rejections of tetrahydrofuran, n-heptane and ethanol in ceramic OSN membranes.

In the recent paper by Werth et al., [35], they had investigated the applicability of different models to describe the mass transfer in oil and solvent mixtures. From their investigation, a reduced form of solution-diffusion imperfection model was able to describe the mass transfer most accurately, as compared to the solution-diffusion model and pore-flow model. They had concluded that the small compounds (free fatty acid and solvent) contributes to mass transfer by convection and alternatively, triglycerides can be described by diffusion [35]. In a different study, the feasibility of OSN for the recovery of organic compound (terpenes) from oil was investigated [41]. Another
equation, Flory-Huggins model were used to describe the interactions between polymer, solvent and oil [41]. From the ternary Flory-Huggins model, volume fractions of oil and solvent were obtained, which were then used to simulate mass transfer by a linearized Maxwell-Stefan model [41]. They had found that sorption level of oil and solvents can be predicted by Flory-Huggins model, and both the fluxes of solvent and oil can be calculated by a linearized Maxwell-Stefan model [41].

5. Concentration Polarization

When a membrane is subjected to a filtration process of fluids with impurities or mixtures, a phenomenon known as concentration polarization will occur. Concentration polarization can be simply explained by the formation of a composition gradient at the feed-membrane interface (as shown in Figure 2). It is crucial to decrease the effect of concentration polarization, as it will increase the possibility of membrane fouling [13].

The effect of concentration polarization can be described by several ways. Some of the earlier publication on the effect of concentration polarization were presented in the late 80’s in the case of protein ultrafiltration [43, 44]. Years afterward, Peeva et al., [9] published the first work to consider concentration polarization in OSN [9]. Peeva et al., [9] had listed seven non-linear algebraic equations (Eq. (10) to Eq. (16)) which allows the prediction of permeate flux and solute rejection.

\[
\frac{J}{k_1} = \ln \left( \frac{c_{1,A2} - c_{1,A3}}{c_{1,A1} - c_{1,A3}} \right) 
\]

\[
\frac{J}{k_2} = \ln \left( \frac{c_{2,A3} - c_{2,A2}}{c_{2,A3} - c_{2,A1}} \right) 
\]

\[
J = J_1 v_1 + J_2 v_2 
\]

\[
J_1 = P_{1,M} \left( x_{1,A2} - \frac{y_{1,A3} J_1}{y_{1,A2} J_1 + J_2} \right) \exp \left( - \frac{v_1 p}{RT} \right) 
\]

\[
J_2 = P_{2,M} \left( x_{2,A2} - \frac{y_{2,A3} J_2}{y_{2,A2} J_2 + J_1} \right) \exp \left( - \frac{v_2 p}{RT} \right) 
\]

\[
\gamma_1 = f(x_1), \gamma_2 = f(x_2) \quad \text{or} \quad \gamma_1 = \gamma_2 = 1 
\]

\[
\text{Rej}_{calc} = 1 - \frac{J_1}{J_{c1,A1}} 
\]

where \(\gamma\) is the activity coefficient, \(k\) is the mass transfer coefficient, \(p\) is the applied pressure. Subscript \(A1, A2,\) and \(A3\) are component \(A\) at interface 1 (feed), interface 2 (membrane) and interface 3 (permeate) as can be seen in Figure 3. Subscript 1 and 2 are component 1 and component 2 respectively.

Baker [4] proposed another equation (Eq. (17)) to estimate concentration polarization which is based on Peclet number. In comparison with the equations proposed by Peeva et al., [9], the equation by Baker [4] only requires the estimation of mass transfer coefficient, and the experimental data of flux, permeate and retentate concentration. Zwijnenberg et al., [45] had published an article on the application of the equation by Baker [4] for the nanofiltration of organic solvents. For the equation by Baker [4], concentration polarization is described by concentration polarization modulus which is written as \(E_0/E\).
whereby \( E_0 \) is enrichment factor, which can be defined as \( C_{A3}/C_{A1} \); \( E \) is true enrichment factor, also written as \( C_{A3}/C_{A2} \). The ratio of the convective transport, \( J_v \), and diffusive transport, \( k \), is known as Peclet number [46].

![Fig. 3. A schematic showing the concentration polarization effect [13,46](17)]

The mass transfer coefficient can be determined by several ways. Peeva et al., [9] had proposed the use of Eq. (18), paired with Chilton-Colburn correlation (Eq. (19)) to obtain the mass transfer coefficient. Eq. (18) was used for the calculation of mass transfer coefficient, which can be determined from the independent measurements of dissolution of benzoic acid plate in water at two cross-flow rates.

\[
\ln \left( \frac{c_{b}^*}{c_b} \right) = \frac{kA}{V} t
\]  

(18)

where \( c_b \) is the molar concentration of benzoic acid (mol/m\(^3\)), \( c_{b}^* \) is the solubility of benzoic acid in water at 30°C (mol/m\(^3\)), \( A \) is the active membrane area (m\(^2\)), volume of water (m\(^3\)), \( t \) is the time duration for the dissolution of benzoic acid and \( k \) is the mass transfer coefficient (m/s).

The mass transfer coefficient of benzoic acid obtained from Eq.(18) can then be used to obtain the mass transfer coefficient of selected solute through the correlation with the following empirical formula

\[
Sh = \frac{k d_h}{D} = a Re^b S^c \left( \frac{d_h}{L} \right)^d
\]  

(19)

where \( k \) is the mass transfer coefficient (m/s), \( d_h \) is the hydraulic diameter (m), \( D \) is the diffusivity (m\(^2\)/s), \( L \) is the channel length (m) and constants \( a, b, c, d \) which depends on the system geometry, flow, and type of fluid.
Eq. (19) can then be simplified to Eq. (20) where the system conditions are assumed to be constant.

\[ k \propto \eta^{(c-b)} D^{(1-c)} \]  \hspace{1cm} (20)

Eq. (21) can then be obtained from Eq. (20).

\[ \frac{k_{\text{solute}}}{k_{\text{benzoic acid}}} \propto \left( \frac{\eta_{\text{solute}}}{\eta_{\text{benzoic acid}}} \right)^{(c-b)} \left( \frac{D_{\text{solute}}}{D_{\text{benzoic acid}}} \right)^{(1-c)} \]  \hspace{1cm} (21)

Besides that, mass transfer coefficient can also be obtained through non-linear parameter estimation method. This method is usually performed through the use of computer equipped with software such as MATLAB, CAPE OPEN and Aspen One as suggested by Peshev et al., [34]. The procedure of the parameter estimation is as shown in Figure 3, whereby, a set of experimental data of permeation flux and rejection is required, coupled with a selected membrane transport model.

In the paper by Murthy and Gupta [47], they have suggested a simplified graphical method for the estimation of mass transfer coefficient. A combined equation of solution diffusion and film theory was employed in their study

\[ \ln \left[ \frac{(1-R_0) \times J_v}{R_0} \right] = \ln \left( \frac{D_{AM}}{K \delta} \right) + \frac{J_v}{k} \]  \hspace{1cm} (22)

whereby through the plot of \( \ln \left[ (1-R_0) \times J_v / R_0 \right] \) against \( J_v \) will yield a straight line, and the gradient of the plot will be \( (1/k) \) and the \( y \)-intercept will be \( \ln(D_{AM}/K\delta) \) [47].

As concentration polarization will definitely occur during membrane filtration process, steps need to be taken to reduce its effects (Table 2). One of the steps is by using a higher feed velocity for cross-flow module. The high velocity of feed will obstruct the formation of stagnant layer on the membrane. For dead-end module, a stirrer should be employed to prevent the settling of fluid mixtures, as well as to create turbulence for an improved mass transfer [4, 13].

| Table 2 |
| --- |
| **Comparison between concentration polarization equation by Peeva et al., [9] and Baker [4]** |

| Peeva et al., [9] | Baker [4] |
| --- | --- |
| **Equation parameters** | **Parameters which requires estimation** |
| \( J_i, k_i, \gamma_i, \chi_i \) | \( J_i, k, \chi_i \) |
| **Methods of estimation** | **k - nonlinear parameter estimation by using regression method [46]** |
| \( k_i - \) Chilton-Colburn correlation [48] \( \gamma_i - \) modified UNIFAC method [50] \( v_i - \) Fedors method [49] | |
| **Number of components** | **Not stated** |
| Two | |
| **Description of concentration polarization** | **By the term concentration polarization modulus** |
| Rejection and permeate flux which includes the effect of concentration polarization | |
| **Membrane module** | **Cross-flow** |
| Cross-flow | |

6. Conclusions

The use of transport model to predict the performance of a transport process is undeniably important, considering its ability to forecast the flux and mass transfer in a membrane separation process. As there can be various systems of organic solvents, the model to best describe each system can be different. The model which is suitable to describe the transport process is greatly dependent
on the interaction between solute, solvent and membrane. From the brief review, solution-diffusion imperfection model is a good model to describe the mass transport of both small and large solutes in an organic solvent system, as it considers the effects of both convection and diffusion. However, the description of solute and solvent fluxes can be best described by the Flory-Huggins model paired with linearized Maxwell-Stefan model, where the ternary interactions between solute, solvent and membrane polymer can be taken into account. As for the pore-flow model, it is best to be used when a membrane having large pores, such as ultrafiltration membrane is involved.

For the investigation of the effect of concentration polarization, there are various ways to describe its effects. The variation in the description of concentration polarization is mostly due to the method of mass transfer coefficient estimation. From the review, the selection of concentration polarization model is based on the assumption made for the organic solvent system. The description of concentration polarization can be solely based on the diffusion or convection depending on the type of transport model selected for the mass transfer estimation.

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