



Modeling of catalytic multi bore hollow fiber membrane plug flow reactor

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Article History

Received: 04 July 2019

Accepted: 19 August 2019

Published: August 2019

Citation

El Zanati E, Farg E. Modeling of catalytic multi bore hollow fiber membrane plug flow reactor. *Indian Journal of Engineering*, 2019, 16, 284-293

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General Note



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ABSTRACT

A model of Micro Multi Bore Hollow Fiber catalytic membrane reactor for the esterification process is developed. The catalytic membrane having a functional group is used to prepare 2-ethyl hexanoate from the reaction of methanol and 2-ethyl hexanoic acid. The implanted catalyst into membrane pores is playing a significant role in removing one of the products from the reaction mixture, thus breaks the thermodynamic equilibrium barrier, and increases the reaction conversion. The multi bores membrane reactor behaves as a plug flow reactor, considering the concentration is varying along one of the space coordinates (length), it is described by ordinary differential equations with and without respect of the axial dispersion. The membrane configuration with the perfect microcapillary channels gives regular flow pattern with minimum resistance. Experimental results, of an earlier project, are used to verify and validate the model; this approach gives a reliable tool for design, scaling-up and optimization. The developed model was

solved numerically. It revealed that the considering of the axial dispersion model is a reliable assumption proved appropriate matching with the predicted and calculating reaction conversion.

Keywords: Multibores, plug flow reactor, membrane reactor, modeling, axial dispersion

1. INTRODUCTION

Esterification is a reversible reaction, conducted in the presence of a catalyst to promote the forward direction, the acidic media either homogeneous (liquid) or heterogeneous (solid) acids are usually used (Iachon et al., 2003; Westermann and Melin, 2009), moreover, high temperature and long residence time are necessary to activate the heterogeneous acid catalysts are preferable due to the ease of separation, preventing corrosion reaction to get an optimum conversion. Using heterogeneous catalysts as ion exchange and Nafion resin can overcome some of the homogeneous catalyst problems, and give high reproducibility (Buonomenna et al., 2010; Jeong et al., 2003). The conversion is achieved to exceed the equilibrium conversion by selective removal of one of the products (Le Chatelier principle) (Levenspiel, 1999).

The Membrane Catalysis has great respect comparing with traditional catalysts regarding its easiness to eliminate one of the products (usually water), which enhance the break of the thermodynamic barrier, thus rapidly obtaining high reaction conversion (Thomas et al., 2010; Shelepova et al., 2011; Plazaola et al., 2019; Buonomenna et al., 2010), it operates continuously, hence, the continuous regime gives excellent product quality and quantity.

Polymeric membranes with Micro Multi Bores Hollow Fiber (MMBHF) are a new configuration, which, could combine the characteristics of flat sheet and hollow fiber membranes; therefore, it can tolerate elevated pressures, and it has a small footprint with a large surface area as hollow fibers membranes (ElZanati et al., 2016; Yingbo et al., 2015; Peng, and Wang, 2012; Xiao-Hua et al., 2013). It is considered the appropriate candidate to act as catalyst carriers due to its exceptional mechanical properties, high surface area, high packing density, easy module fabrication, outstanding stability, and reduced fiber breakage (Castanheiro et al., 2006; Klofutar et al., 2010).

According to Le Chatelier's Principle, the reaction conversion can exceed the equilibrium conversion by adding one of the reactants or removing one of the products, in this context, the water molecules are absorbed by a functionalized the grafted sulfonic group (Levenspiel, 1999), inside the membrane pores, where each pore is considered as a micro-reactor. The great contact between reactants and catalytic sites in the membrane reaches to high catalytic activity and minimum resistance (Xiao-Hua Maa et al., 2013).

The objective of this article is to develop a mathematical model of the (MMCF) membrane reactor, conducting an esterification reaction. This model is verified using experimental results generated from a project funded by Science and technological development fund, Ministry of Scientific Research, Cairo, Egypt (ElZanati, 2017).

Reactor Design

Multi Bore Hollow Fiber Membranes

Thirteen Membrane fibers, each one has seven micro bores, were earlier developed (El Zanati, 2017), Figure (1) demonstrates the cross-section of the elaborated fibers.

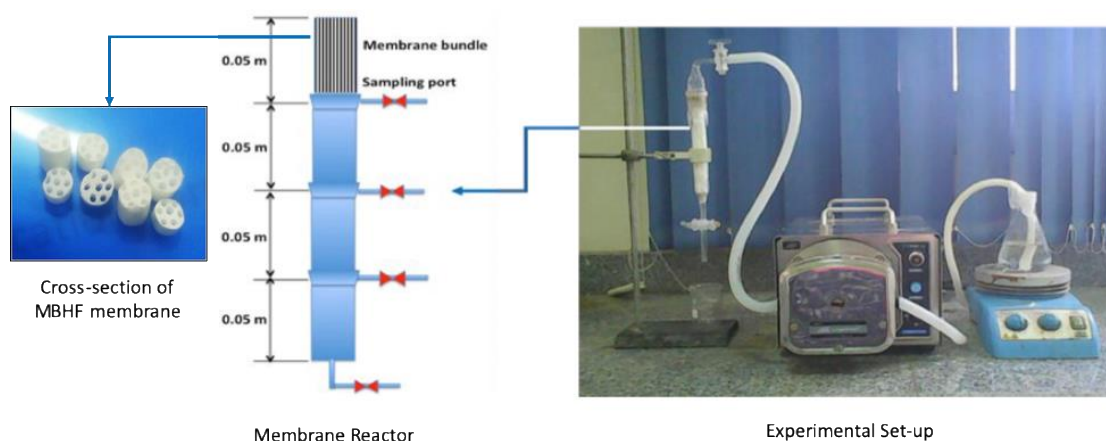


Figure 1 The Seven Bores Hollow Fiber Membrane reactor

Reactor construction

The used catalytic membrane reactor is composed of 13 grafted fibers of 7 bores and 20 cm length contained in a glass tube, has a sample opening every 5 cm to study the kinetics of the esterification process Figure (1).

Grafting of the membrane fibers

A designed spinneret of multi holes is used to cast the fibers, prepared from Poly Ether Sulfone. The functional sulfonic group was grafted into pores of the microbore hollow fiber membrane using styrene as the monomer. The C-S double bond created by the styrene monomer can provide linkages that undergo chain polymerization (Thomas et al., 2010; Kumar et al., 2006). Activation step of the membrane is characterized by implanting a sulfonic group of negative charge on each branch of styrene polymer inside the pores; in which, 0.5N H₂SO₄ is permeated throughout the prepared membrane at 298° K.

2. MODEL DEVELOPMENT

Physical description of the reaction system

The MMBHF catalytic membrane reactor is considered a multi micro plug flow reactors. The developed model relies on the geometry of the membrane, where, the lateral convective diffusion through the membrane wall occurs on internal pores area, which is measured by Brunauer-Emmet-Teller area, meanwhile, the axial dispersion may be affected the reaction propagation Figure (2).

Fick's Law describes the axial dispersion flow through the perfect membrane cylindrical bores with the same radius.

$$N_i = -D_i \frac{dC_i}{dl} \quad (1)$$

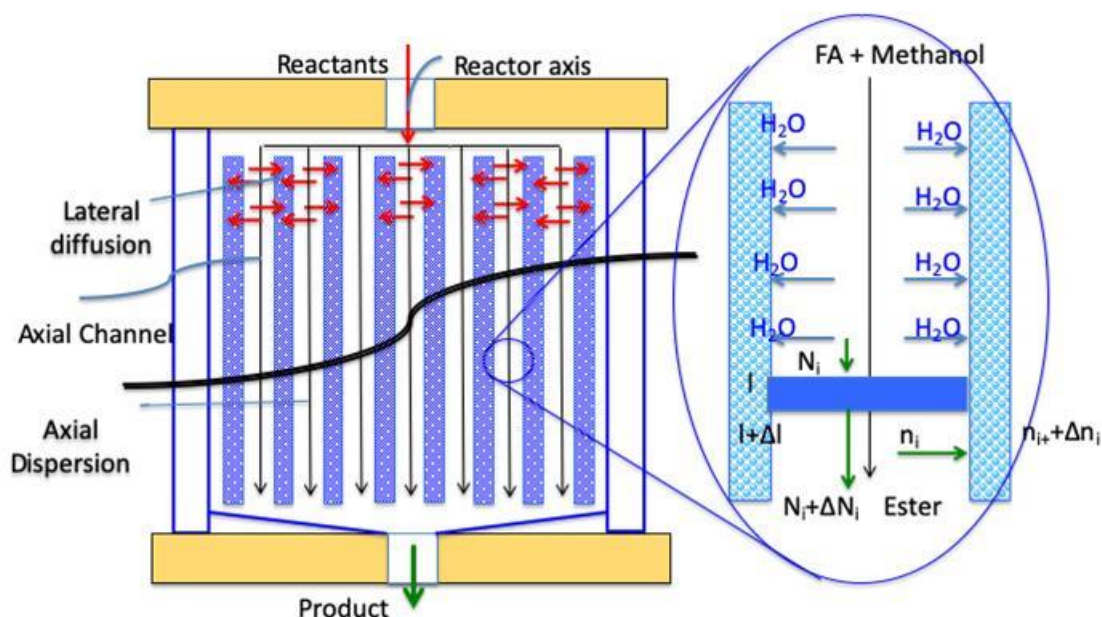


Figure 2 The geometry of MMBHF catalytic membrane reactor and flow pattern

Model assumption

The following assumptions are adopted to develop the model.

- 1- The MMBHF membrane reactor can be considered a plug flow reactor of radial homogeneities till exit, the fluid moves through the channel with uniform velocity.
- 2- The flow inside the micro bores is considered perfectly mixed.
- 3- In the plug flow, the convective flow is dominating besides of the dispersion of mass and heat-driven concentration and temperature gradients.
- 4- Plug flow reactor is a steady-state reactor.
- 5- Constant density.

6- Single reaction occurring in the bulk of the fluid (homogeneously), one of the products (water) is linked and adsorbed on the active functional sulfonic group, which was grafted inside the pores of the membrane walls.

7- The reaction is isothermal and isobaric.

8- The perfectness of the cylindrical micro bores is satisfactory to neglect the resistance.

9- Mass transport of the reactants is axial and lateral-dispersed flow.

10- Diffusion coefficients independent on the concentration, that is constant throughout the membrane thickness.

11- The magnitude of dispersion is sufficient to account for the joint effect of all dispersive phenomena. The model is known as the dispersion model (T. Westermann et al., 2009).

12- The active sites, in the membrane pores (SO_3^- groups), have an affinity only towards the water, the removal of water molecules shifts the esterification reaction to the forward direction according to the Le Chatelier principle.

13- The unreacted alcohol, 2-ethyl hexanoic acid, and ester are not adsorbed on the membrane.

14- The driving force of water transfer from the pore bulk to the pore surface was the difference between their concentrations at equilibrium.

15- Fick's law describes the axial dispersion of mass flow (equation 1).

Model derivation considering the axial dispersion

Figure (2) demonstrates a longitudinal capillary section of the membrane and the grafted functional sulfonic group. The equation (2) describes the reaction.



- The reaction rate constant can be written as (equation 3):

$$r = k_1 C_a C_b - k_2 C_e C_w \quad (\text{mol/cm}^2 \cdot \text{s}) \quad (3)$$

The forward reaction rate constant k_1 is a function of the reaction temperature; it is determined using the Arrhenius equation:

$$k_1 = k_0 e^{-E/RT} \quad (4)$$

The backward reaction rate constant k_2 is equal to k_1/K . The equilibrium constant (K) is calculated using the following equations (Ichikawa et al., 2010):

$$\ln K_{298} = \frac{-\Delta G_{298}}{RT} \quad (5)$$

$$K = K_0 e^{-\Delta H/RT} \quad (6)$$

The values of ΔG and ΔH Table (1) are the difference of Gibbs free energy of products and reactants, and the difference of the heat of formation of products and reactants respectively.

Table 1 Gibbs free energy and the heat of formation of products and reactants

Item	Methanol	2-ethylhexanoic acid	Water	Meth. Eth. Hexanoate
ΔG , kJ/mol	-166.4	-251.7	-228.61	-371
ΔH , kJ/mol	-238.4	-333.0	-285.8	-399.3

Component mass balance:

$$n_{if} + \sigma_i r S + \gamma_i k_1 S (C_i - k_d C_{is}) = n_{io} + \frac{dn_{is}}{dl} + A_c \frac{dN_i}{dl} \quad (7)$$

Considering that:

$$n_i = q_f C_i \text{ and } N_i = -D_i \frac{dC_i}{dl}$$

$$q_f C_{if} + \sigma_i r S + \gamma_i k_1 S (C_i - k_d C_{is}) = q_o C_i + \frac{d(V C_i)}{dl} - A_c D_i \frac{d^2 C}{dl^2} \quad (8)$$

By rearrangement and dimensional correction:

$$q_f C_{If} + \sigma_i r S + \gamma_i k_l S (C_i - k_d C_{is}) = q_o C_i + \left(\frac{q_f}{A_c}\right) V_p \frac{dC_i}{dl} - x \left(\frac{q_f}{A_c}\right) C_i \frac{dA_s}{dl} - d_h A_c D_i \frac{d^2 C_i}{dl^2} \quad (9)$$

$$S = S_o - A_s, \text{ The internal pore surface area (cm}^2\text{) at any time t.} \quad (10)$$

$$ds = -dA_s \quad (11)$$

$$V_p = V_{po} - A_s x \quad (12)$$

$$dV_p = -x dA_s \quad (13)$$

Water adsorption, n_{ws} :

The change of water adsorbed on pore surface with channel length is:

$$\frac{dn_{ws}}{dl} = \frac{d(C_{ws} A_s)}{dl} = -\gamma_w k_{lw} \left(\frac{A_c}{q_f}\right) (C_w - k_d C_{ws}) S \quad (14)$$

$$C_{ws} \frac{dA_s}{dl} + A_s \frac{dC_{ws}}{dl} = k_{lw} \left(\frac{A_c}{q_f}\right) (C_w - k_d C_{ws}) S \quad (15)$$

The change of the surface area covered with water (cm²) with length of channel is equal the change of water volume inside the pores divided by the water layer thickness;

$$\frac{dA_s}{dl} = \frac{k_{lw} \left(\frac{A_c}{q_f}\right) (C_w - k_d C_{ws}) S}{x \rho_M} \quad (16)$$

The change of water adsorbed on membrane surface with capillary length:

$$\frac{dC_{ws}}{dl} = \frac{1}{A_s} \left[k_{lw} \left(\frac{A_c}{q_f}\right) (C_w - k_d C_{ws}) S - \frac{k_{lw} \left(\frac{A_c}{q_f}\right) C_{ws} (C_w - k_d C_{ws}) S}{x \rho_M} \right] \quad (17)$$

$$q_o = q_f - \frac{k_{lw} (C_w - k_d C_{ws}) S}{\rho_M} \quad (18)$$

Rewriting equation (9) for each component (a, b, e and w) gives:

$$\frac{dC_a}{dl} = \frac{1}{\left(\frac{q_f}{A_c}\right) V_p} \left[q_f C_{If} - r S - q_o C_a + x \left(\frac{q_f}{A_c}\right) C_a \frac{dA_s}{dl} + d_h A_c D_a \frac{d^2 C_a}{dl^2} \right] \quad (19)$$

$$\frac{dC_b}{dl} = \frac{1}{\left(\frac{q_f}{A_c}\right) V_p} \left[q_f C_{If} - r S - q_o C_b + x \left(\frac{q_f}{A_c}\right) C_b \frac{dA_s}{dl} + d_h A_c D_b \frac{d^2 C_b}{dl^2} \right] \quad (20)$$

$$\frac{dC_e}{dl} = \frac{1}{\left(\frac{q_f}{A_c}\right) V_p} \left[q_f C_{If} + r S - q_o C_e + x \left(\frac{q_f}{A_c}\right) C_e \frac{dA_s}{dl} + d_h A_c D_e \frac{d^2 C_e}{dl^2} \right] \quad (21)$$

$$\frac{dC_w}{dl} = \frac{1}{\left(\frac{q_f}{A_c}\right) V_p} \left[q_f C_{wf} + r S - k_l S (C_w - k_d C_{ws}) - q_o C_w + x \left(\frac{q_{wf}}{A_c}\right) C_w \frac{dA_s}{dl} + d_h A_c D_w \frac{d^2 C_w}{dl^2} \right] \quad (22)$$

Ignoring the effect of axial dispersion

A reforming of the previously developed model considering the ignoring of the axial dispersion effect on the change of reaction concentration along the length of the reactor. Equation (20) demonstrates the simplified model:

$$q_f C_{If} + \sigma_i (S_o - A_s) (k_1 C_a C_b - k_2 C_e C_w) + \gamma_i k_l (S_o - A_s) (C_i - k_d C_{is}) = q_o C_{io} + v V \frac{dC_i}{dl} - v V C_i \frac{dA_s}{dl} \quad (23)$$

3. RESULTS AND DISCUSSION

The nonlinear model of esterification reaction accomplished in micro reactor of multi bore hollow fiber membrane is presented. The reactants are entered inside the micro bore hollow fiber lumen, and then dispersed axially.

Model solution

The axial dispersion plug flow model for micro multi bores membrane reactor is developed assuming that each bore is functioning as a plug flow reactor. The model was solved for the change reaction conversion versus the independent spatial longitudinal variable (length, l) at specific simulated state variables C_{i0} (C_{a0} , C_{b0} , C_{e0} , and C_{w0}), C_{ws} , q_0 , S_0 and A_c , using Simulink, Matlab b2014. The earlier experimental results produced and published from the project titled "Novel Membranes with Multiple Hollow Holes (MMHH) for Reaction Catalysis to produce biofuel", ID 9185, funded by Science and Technology Development Fund, Ministry of Scientific Research, Cairo, Egypt (El Zanati, 2017).

The activation energy (E) for the esterification reaction of methanol with 2-ethyl hexanoic acid has been reported in the range from 42 to 53.7 kJ mol⁻¹ (Moya-Leon et al., 2006); accordingly, a value of 50 kJ mol⁻¹ (average) is appropriate and used to estimate the reaction constants.

Design parameters

- Membrane bore diameter: 0.02 cm (ElZanati et al., 2015).
- Total internal pore surface area, S_o (of 13 fibers of 7 bores), 114.296 cm².
- Membrane pore volume, V_o ; 0.57148 cm³.
- The cross-sectional surface area; 0.028574 cm².
- q_f ; 0.2105 cm³ s⁻¹; reaction temperature, 25°C (298 K); catalyst loading capacity based on the IEC corresponding to styrene mass fraction 0.12 during grafting formation [18].
- $MR(n_{af}/n_{bf})$, 1:1.

Model verification

The governing equations (9-22) were solved and verified using the experimental results obtained from the mentioned previous work (El Zanati et al., 2015). The sum of residual squares errors (SRS) (or ΣError^2 for N readings) was used to achieve the best fitting of results, Figure (3).

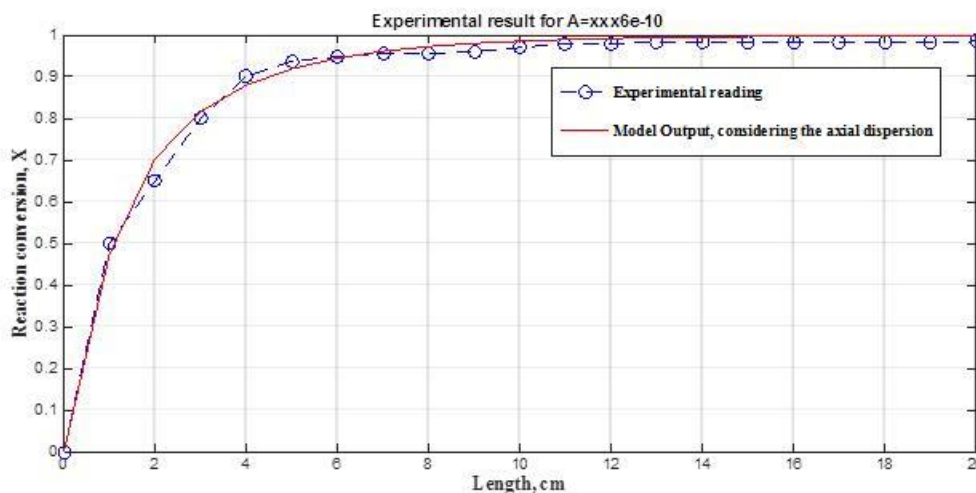


Figure 3 Curve fitting of reaction conversion calculated experimentally and that predicted using the model concerning the axial dispersion

The results of the axial dispersion model were reasonably compatible with the experimental data simulated at the same reaction conditions at the model parameters gives the best are summarized as follows:

- The initial esterification forward reaction constant, k_o (frequency factor), is equal to $2.92\text{E-}5 \text{ m}^3 \cdot \text{mol}^{-1} \cdot \text{s}^{-1}$.
- The mass transfer coefficient of liquid water, k_l : $1.764\text{E-}7 \text{ m} \cdot \text{s}^{-1}$.
- The adsorption coefficient of water, k_d : $6\text{E-}8 \text{ m}^{-1}$.
- The water adsorption thickness, X , was $5.8\text{E-}12 \text{ m}$.

The maximum predicted reaction conversion X_p obtained is 0.97606 with minimum error of 0.02 at equal $2.92\text{E-}5$ as demonstrated in Figure (4).

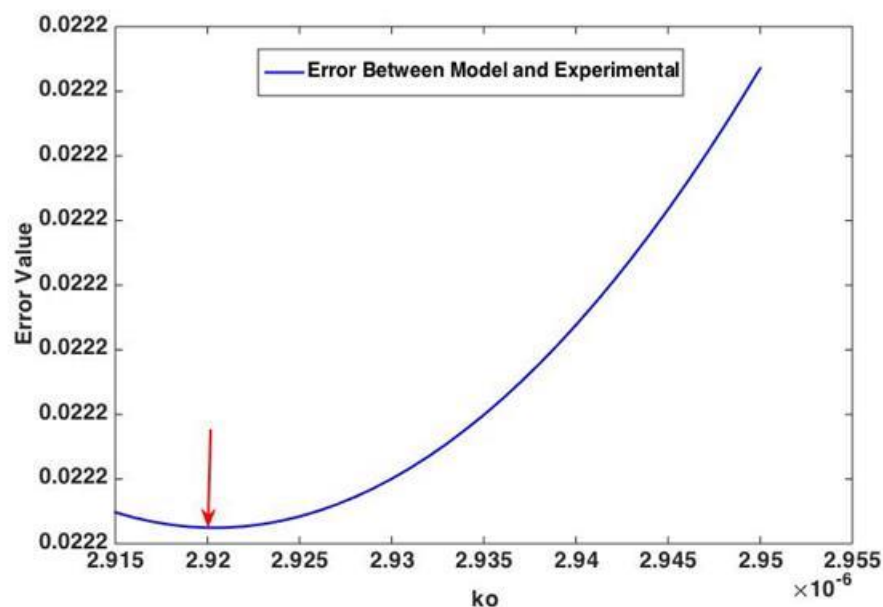


Figure 4 minimum errors for best fitting of all parameters

The solution of the second model approach (ignoring the axial dispersion) simulated at the same model parameters; does not fit the experimental results; where the maximum X_p is 0.796 (18.45%) decrease compared with that obtained when axial dispersion is considered. Therefore, the supposed axial dispersion in the plug flow reactor is excellent approach, Figure (5). It is, also, noticed from Figure (3) that the reaction conversion is approached more than 90% at reactor height 5cm, then X_p gives an asymptotic value; therefore, the length of the reactor can be designed in this case; shorter than 20cm, this behavior might be attributed to the large inner surface area of the membrane which reveals high number of pores (contain the active sulfonic group) in the micro-multi channels hollow fiber membrane.

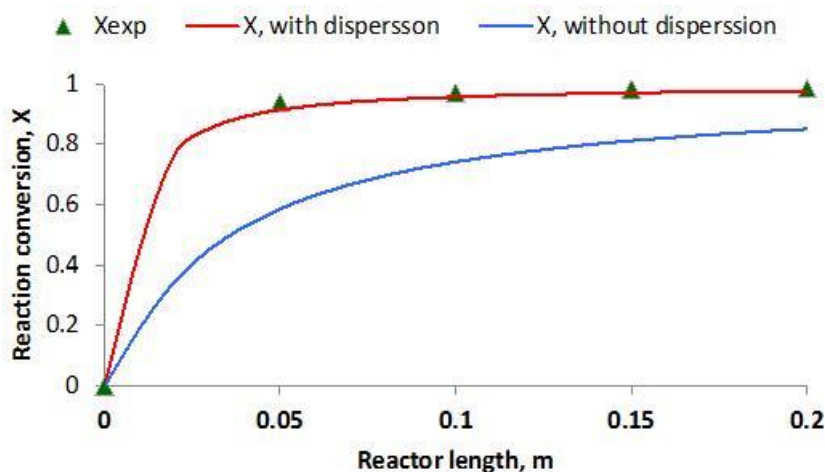


Figure 5 Conversion versus channel length for the two models

The developed model with axial dispersion is clearly showed that it was valid and could be applied at other design/operating conditions with reasonable reliability and accuracy. The model is therefore suitable for design, replication, scaling-up, optimization, and parametric investigation.

The model considering the axial dispersion is used to check and verify the concentrations of methanol and 2-ethylhexanoate (ester) concentrations with length (C_a and C_e), Figure (6).

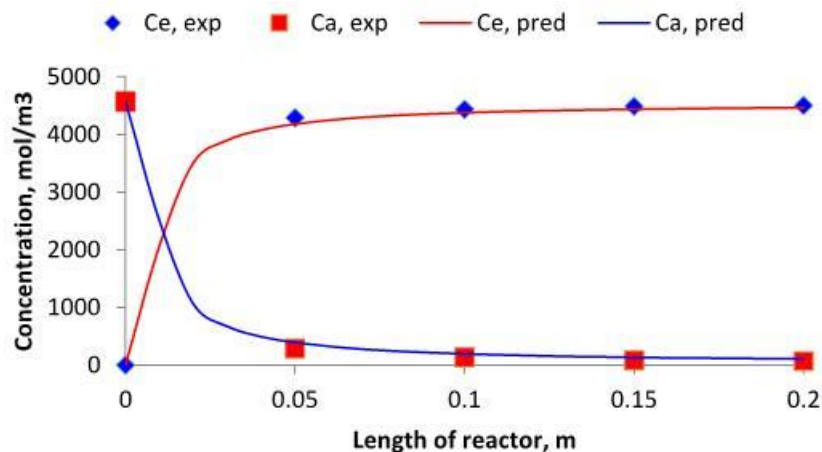


Figure 6 Best fitting of concentration of experimental results and the predicted by the model concerning the axial dispersion (C_a/C_b and C_e versus length)

The verified model was also used to detect the change of the water concentration in product stream with length and compared with that obtained from experimental data. It is clear that the most of produced water is adsorbed on the membrane pores, while a trace percentage (7.27E-9%) of water released in output stream, Figure (7).

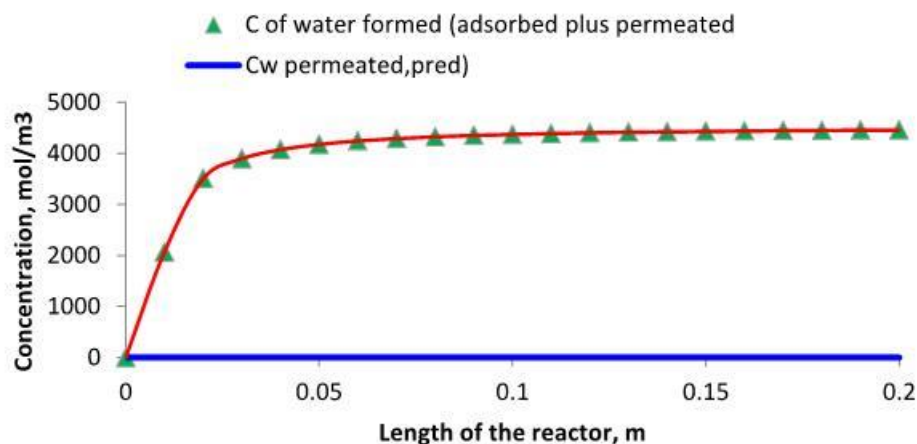


Figure 7 Change of concentration of Water in Permeate and concentration of water adsorbed on membrane

4. CONCLUSION

A plug flow with axial dispersion model was developed for esterification reaction of methanol and 2-ethylhexanoic acid in micro-multi channels hollow fiber catalytic membrane reactor. Another approach of ignoring the axial dispersion was developed. The two developed models are solved and verified using published experimental results. The initial reaction constant was adjusted to obtain good agreement between the reaction conversions determined from the model and experimental results. The simulation process is executed at the reaction conditions: MR of 1:1 (Methanol: 2-ethylhexanoic acid), T of 298°K, and IEC corresponding to 0.12 styrene mass fraction, and S_o 114.296 cm^2 . The model solution revealed that the consideration of axial dispersion in the micro channels plug flow reactor is convenient and satisfactory. The model will be used to study scale-up of the reactor to semi-commercial design size, and accomplish a preliminary feasibility.

Acknowledgement

The author gratefully acknowledges the total financial support Science and technology Development Fund (STDF), Ministry of scientific Research and Technology, Egypt.

Funding: This research was funded by the Science and technology Development Fund (STDF), Ministry of Scientific Research and Technology, Egypt.

Conflicts of Interest: The authors declare no conflict of interest.

Author contribution

E. El Zanati: The author has suggested the research point, and she developed the mathematical model describing the Multi Bore Hollow Fiber membrane reactor, which was used to prepare an ester, and also, she verified and validated the model using the published experimental data of a project entitled "Novel Membranes with Multiple Hollow Holes (MMHH) for Reaction Catalysis to produce biofuel", funded by the Science and technology Development Fund (STDF), Ministry of Scientific Research and Technology, Egypt.

E. Farg: She was responsible on the performance of the experimental work, data analysis and discussion.

Nomenclatures

a	: Alcohol (methyl)
b	: Acid (2-ethylhexanoic acid.)
e	: Ester of 2-ethylhexanoic acid
w	: Water
i	: Reaction components (alcohol, acid, ester and water),
C _i	: Bulk concentration of component i, molcm ⁻³
C _{is}	: Surface concentration of component i on pores surface, molm ⁻²
C _{ws}	: Concentration of adsorbed water molecules on pore surface, molcm ⁻²
D _i	: Diffusivity of component i, cm ² s ⁻¹
E	: Activation energy, kJmol ⁻¹
ΔG	: Difference of Gibbs free energy of products and reactants, kJmol ⁻¹
ΔH	: Difference of heat of formation of products and reactants, kJ mol ⁻¹
k ₁	: Forward reaction constant, cm ⁴ mol ⁻¹ s ⁻¹
k ₂	: Backward reaction constant, cm ⁴ mol ⁻¹ s ⁻¹
kl	: Mass transfer coefficient of water in the bulk solution, cms ⁻¹
kd	: Adsorption coefficient of water inside the pores, cm ⁻¹
K	: Equilibrium constant, dimensionless
l	: Length of channels, cm
N _i	: Mass flux of component, molcm ⁻² s ⁻¹
n _i	: Number of components i, mol
n _{ws}	: Number of adsorbed water molecules on pore surface, mol
r	: Rate of reaction, mols ⁻¹ cm ⁻²
σ _i	: -1 for reactants and +1 for products.
γ _i	: 0 for i: a, b and e, while it equal -1 for i: w.
q _i	: Flow rate, cm ³ s ⁻¹
ρ _M	: Molar water density, gmolcm ⁻³ .
q _f	: Feed flow rate, cm ³ s ⁻¹
q _o	: Output flow rate, cm ³ s ⁻¹
SRS	: Sum of residual squares
R	: Reaction gas constant: 0.008314, kJmol ⁻¹ K ⁻¹
T	: Temperature degree, K
X _p	: Predicted reaction conversion

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