ANALYSIS

Vol. 16, 2019

ISSN 2319–7757 EISSN 2319–7765

Indian Journal of Engineering

# Mathematical modeling and verification of pervaporation –assisted esterification reactor

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**Article History**
Received: 22 March 2019
Accepted: 10 May 2019
Published: June 2019

**Citation**
Elham El-Zanati, Heba Abdallah, Eman Farag, Reem Ettouney, Mahmoud El-Rifai. Mathematical modeling and verification of pervaporation –assisted esterification reactor. Indian Journal of Engineering, 2019, 16, 184-196



## ABSTRACT

A mathematical model for esterification coupled with pervaporation is developed to investigate and optimize the effect of water removal from the reaction mixture on the obtainable conversion in a batch reactor fitted with recirculation around a pervaporation membrane. The effects of water removal through pervaporation, temperature, molar ratios of reactants, and catalyst weight percents are compared with experimental data obtained on the laboratory scale. The studied system involves the esterification reaction between acetic acid and methanol with simultaneous pervaporation to produce biofuel (methyl acetate). The model was validated

for different reaction operating conditions and the evolution of water concentration in the reaction mixture is followed throughout the reaction.

**Keywords**: Membrane separation technology, Pervaporation, Esterification, Solid catalysis

## 1. INTRODUCTION

Pervaporation (PV) is an energy efficient technology providing for a high separation efficiency of azeotropic, isomers, and hydrocarbon mixtures. The conversion of a chemical reaction can be enhanced when the reaction is coupled with a pervaporation membrane system (Z. Yun et al., 2010; Q. Liu et al., 2001). In this process, specific compounds diffuse from the liquid mixture through the pervaporation membrane and are then desorbed as vapor at the permeate side of the membrane. The driving force for the transportation through the pervaporation membrane is the partial pressure difference between the two sides of the membrane (H. Abdallaha et al, 2013; A. Hasanoğlu et al., 2011; K.C. Souza Figueiredo et al., 2008).

A typical example for PVMR application in industry is reversible conversion limited reactions such as esterification (P.R. Ajit et al., 2013; F. Xianshe and R.Y.M. Huang, 1996; J. Maa et al., 2009). Conventionally, esterification reactions are carried out under batch conditions using mineral acid catalysts. High temperature is necessary to activate the reaction and long reaction times are usually required to reach maximum conversion. Also, using a strong liquid acid catalyst in the reaction leads to corrosion of the reactor and difficult separation of the spent acid catalyst, which is miscible with the reaction mixture. Some associated problems with this process can be overcome by using heterogeneous catalysts. Heterogeneous solid acid catalysts are preferable because they are easily separated by filtration (E. El-Zanati et al., 2014; R.R. Rohit and A.K. Anton, 2012; X. Su et al., 2016).

Coupling pervaporation with esterification would provide for enhancing the reaction, reducing energy consumption, and would ultimately lead to zero end of pipe waste (P.R. Ajit et al., 2013; F. Xianshe and R.Y.M. Huang, 1996;H. Ayça and D. Salih, 2011). The objective of this work is to develop a mathematical model to describe the recycle batch pervaporation membrane reactor (RBPMR). The experimental data from our previous work will be used to verify and validate the model (Elham ElZanati, 2018). A parametric study of the relevant parameters including molar ratio (MR) of feed mixture, reaction temperature (T), and silica sulfuric acid catalyst weight percent (SSA wt %) will be also conducted.

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## 2. MODEL DEVELOPMENT

**Assumptions**

Figure (1) depicts the recycle batch pervaporation membrane reactor (RBPMR). The following simplifying assumptions have been applied (K.L. Wasewar, 2009; A.Hasanoğlu, 2010; S.Y. Lim, 2002):

1. The pervaporation PV module contains a water selective hydrophilic dense membrane.
2. Isothermal operation is assumed during pervaporation.
3. Constant catalyst activity.

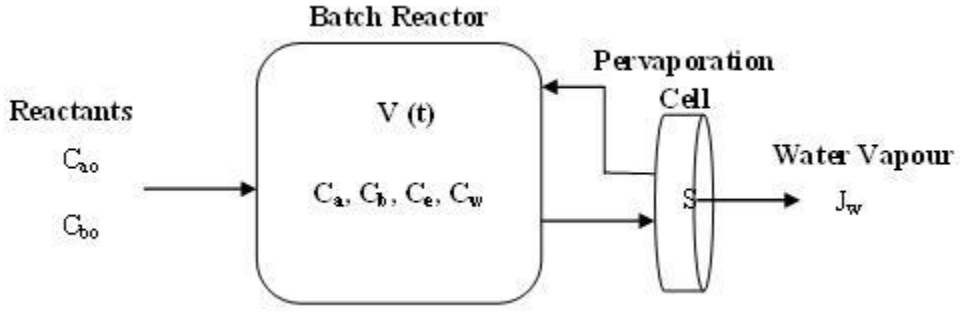


**Figure 1** Schematic Representation of the Pervaporation System

**Reaction Parameters**

The second order reversible esterification reaction between acetic acid(a) and methanol (b) to given methyl acetate (e) and water (w) is

$$a + b \leftrightarrow e + w \quad (1)$$

The esterification reaction rate is given by:

$$r_i = k_f C_a C_b - k_b C_e C_w = k_f (C_a C_b - \frac{1}{K_{eq}} C_e C_w) \quad (2)$$

Where $C_a$, $C_b$, $C_e$, $C_w$ are the mean concentrations of the above species, $r_i$ is the rate of reaction per unit catalyst concentration, and $k_f$ is the forward reaction constant and its temperature dependence is given by the Arrhenius equation:

$$k_f = k_o . exp(-\frac{E_\alpha}{RT}) \quad (3)$$

The parameter $k_b$ is the backward reaction constant, $k_f/K_{eq}$. The equilibrium constant is calculated from the following equations [R.D. Nobel and S.A Stern (2003)]:

$$K_{eq} = K_{eqo} . \exp(- \frac{DH}{RT}) \quad (4)$$

$$K_{eq,T0} = \exp(-\frac{\Delta G_{T0}}{RT}) \quad (5)$$

The values of ΔG and ΔH of products and reactants are given in Table 1.

**Table 1** Gibbs free energy and heat of formation of methyl acetate, water, alcohol and acetic acid

| | Methanol | Acetic acid | Methyl acetate | Water |
|---|---|---|---|---|
| ΔG, J/mol | -166.2 | -389.95 | -316.57 | -237.1 |
| ΔH, J/mol | -239.2 | -484.13 | -445.9 | -285.8 |

Using the numerical values of the parameters in Table (1)

$$K_{eqT} = 6.913 Exp(-922.54/T) \quad (6)$$

**Model Equations**

A schematic outline of the studied RBPMR is shown in Figure (1). The component mass balance equations on the reactor are given by:

$$\frac{d(C_i V)}{dt} = \sigma_i r_i V - \lambda_i J_i S \quad (7)$$

$$V \frac{dC_i}{dt} + C_i \frac{dV}{dt} = \sigma_i r_i V - \lambda_i J_i S \quad (8)$$

Where $C_i$ is the concentration of component i (a,b,e,w) in reactor (mol/cm$^3$), S is the membrane area (cm$^2$), $J_i$ is the permeation flux of component i (mol/cm$^2$.min), $\sigma_i$ is a coefficient equal to (-1) for reactants and (+1) for products, and $\lambda_i$ is a permeability coefficient. The change of volume mixture due to water adsorption is described by:

$$V(t) = V_0 - S\sum_{i=1}^{4}\int_{o}^{t}\frac{\lambda_i J_i}{\rho_i^M}dt \qquad (9)$$

Differentiation of equation (9) gives

$$\frac{dV}{dt} = -\sum_{i=1}^{4}\lambda_i \frac{J_i S}{\rho_i^M} \quad (10)$$

Where$V_o$ is the initial volume mixture and $\rho_i^M$ is the molar density of component i (mol/cm$^3$). For a water selective permeable membrane $\lambda_a = \lambda_b = \lambda_e = 0$ and $\lambda_w = 1$. Substitution into equation (10) gives

$$\frac{dV}{dt} = -\frac{J_w S}{\rho_w^M} \qquad (11)$$

$$J_i = -D_i\frac{\partial C_i}{\partial \delta} \qquad (12)$$

The water permeation flux may be approximated by

$$J_i = K_{p,i}C_i \qquad (13)$$

Where *D* is a Diffusion coefficient, δ is the membrane thickness (cm) and Kp is the pervaporation empirical constant (min/mol).
The water flux through membrane will be given by:

$$J_w = K_{p,w}C_w \qquad (14)$$

Considering the stoichiometry of equation (1) and that the limiting reactant is acetic acid with initial concentration ($C_{ao}$), the concentrations of each component are given by:

$$C_a = C_{a0}\frac{V_0}{V}(1 - x_a) \quad (15)$$

$$C_b = C_{a0}\frac{V_0}{V}(\theta_b - x_a) \quad (16)$$

$$C_e = C_{a0}\frac{V_0}{V}(\theta_e + x_a) \quad (17)$$

$$C_w = \frac{C_{a0}V_0(\theta_w + x_a) - \int SJ_w dt}{V} \qquad (18)$$

Where, $x_a$is the fractional conversion of acetic acid (a) defined as the number of reacted moles of (a) per mole of (a) fed to the reactor, $\Theta_b$, $\Theta_e$ and $\Theta_w$ are the ratios of initial concentrations of species b, e, and w, respectively, to the initial concentration of a.
For a typical water permeable membrane $J_a = J_b = J_e = 0$, and substituting $\sigma_a = -1$, the mass balance for acetic acid is given by:

$$\frac{d(C_a V)}{dt} = -r_a V \qquad (19)$$

Combining with Eqs. (2) and (15) gives:

$$\frac{d(C_{a0}V_0(1 - x_a)}{dt} = -[k_f(C_a C_b - \frac{1}{K_{eq}}C_e C_w)].V \qquad (20)$$

Substitution of components concentration from Eqs. (16), (17), and (18)

$$\frac{dx_a}{dt}.C_{a0}V_0 = k_f\,[\frac{1}{V^2}C^2{}_{a0}V^2{}_0(1-x_a)(\theta_b-x_a)-\frac{1}{K_{eq}}C_{ao}\frac{V_0}{V}(\theta_e+x_a)C_w)].V \quad (21)$$

Rearranging, the rate of change of acetic acid conversion with time may be obtained from:

$$\frac{dx_a}{dt} = k_f[\frac{V_0}{V}C_{a0}\ (1-x_a)(\theta_b-x_a)-\frac{1}{K_{eq}}(\theta_e+x_a)C_w)] \quad (22)$$

The water balance in pervaporation reaction is given by:

$$\frac{d(C_wV)}{dt} = r_wV - J_wS \quad (23)$$

Since $r_w$=-$r_a$, the combination of Eqs. (15), (19), and (23) gives

$$\frac{d(C_wV)}{dt} = \frac{d(C_{a0}V_0(1-x_a)}{dt} - J_wS \quad (24)$$

This may be rewritten as

$$V\frac{dC_w}{dt} + C_w\frac{dV}{dt} = C_{a0}V_0\frac{dx_a}{dt} - J_wS \quad (25)$$

Substitution into Eq. (25) by equation (11), the rate of change in water concentration can be expressed by:

$$\frac{dC_w}{dt} = \frac{1}{V}(C_{a0}V_0\frac{dx_a}{dt} + J_wS(\frac{C_w}{\rho_w^M}-1)) \quad (26)$$

**Model Equations solution**

The governing model equations (9), (22) and (26) were solved using MATLAB Simulink (The Mathworks, Release 2014b) to compare the change in the reaction conversion and water concentration in the reactor with the experimental results. The numerical values used in the simulation corresponded to the experimental conditions of $V_o$ = 111.47 $cm^3$, S= 145.19 $cm^2$.The pre-exponental factor $k_o$ values of Arrhenius equation (3) were estimated using the Matlab M-File based on choosing the best $k_o$value which gave the minimum error between model and experimental data. The Matlab M-File is linked with the Matlab Simulink by $k_o$.

## 3. RESULT AND DISCUSSION

**Verification of the Model**

The developed lumped parameter model of esterification coupled with pervaporation was used to calculate the evolution of the conversion and water concentration in the course of the reaction. The system parameters were adjusted to give the best fit with experimental results by calculating the minimum error between predicted data and experimental readings. The activation energy for the pervaporation – assisted esterification reaction systems has been taken at 6960 J $mol^{-1}$ to evaluate the specific reaction constants (A. Hasanoğlu et al., 2011). There was fair matching between the experimental data and the values predicted by the model considering a second order reaction for both forward and backward reactions.

The experimental results of the esterification reaction catalyzed by SSA and coupled with pervaporation were compared with the model output at the operating conditions: feed temperature 60°C, catalyst weight percentage 2%, ratio between membrane area and volume of mixture S/V 1.3 $cm^{-1}$ and starting molar ratio of methanol to acetic acid 8:1. The good matching between the experimental results and prediction under the same operating conditions both for reaction conversion and water concentration are confirmed as shown in Figure (2) and Figure (3). The values $k_o$; 300000 $cm^3$/mol.min and $K_P$; 4 cm/min gave the best fit between model and experimental results.

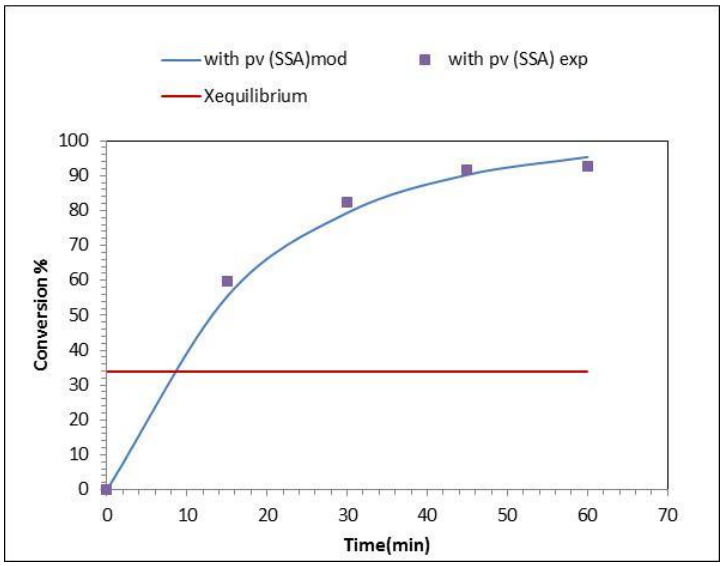


**Figure 2** Comparison between predicted reaction conversion from model and experimental results with PV at $k_o$=3*10$^5$cm3/mol.min, T=60$^o$C, MR=8:1, wt=2%, S/V=1.3cm$^{-1}$

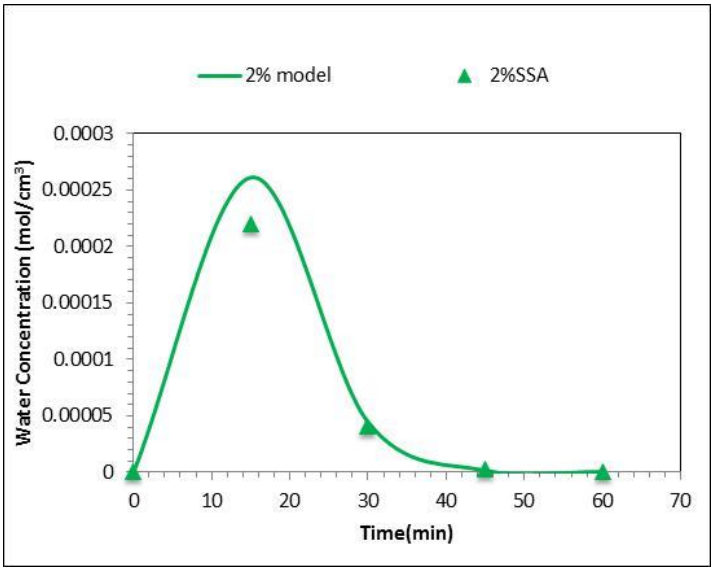


**Figure 3** Comparison between predicted water concentration from model and experimental results with PV at $k_0$=3*10$^5$cm3/mol.min, T=60$^o$C, MR=8:1, wt=2%, S/V=1.3cm$^{-1}$

It is also seen that an equilibrium conversion of 33.9% is reached after 60 min without PV while a conversion of 92.79% is obtained after 60 min with PV. This shift is due to water removal from the reaction medium through the PV selective membrane (E. El-Zanati et al., 2014; W. Zhang et al., 2014; G. Genduso et al., 2015).

Another set of experimental results was compared with model results taking the aforementioned values of $k_o$ and $k_p$ at the operating conditions: feed temperature 60°C, catalyst weight percentage 3%, S/V ratio ; 1.3 cm$^{-1}$ and initial molar ratio of methanol to acetic acid 8:1. Figure (4) shows also a significant increase in conversion from 0.339 (thermodynamic equilibrium conversion) to 0.967. Water diffusion through the PV membrane is the reason for the increase of the thermodynamic equilibrium barrier. There is good agreement between experimental and model output as shown in Figure (4) and Figure (5).

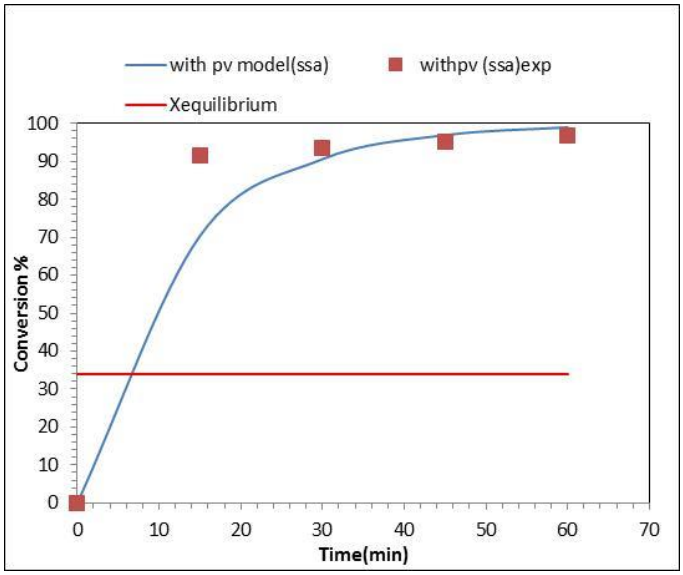


**Figure 4** Comparison between predicted reaction conversion from model and experimental results with PVat $k_0$=3*10$^5$cm3/mol.min, T=60°C, MR=8:1, wt=3%, S/V=1.3cm$^{-1}$

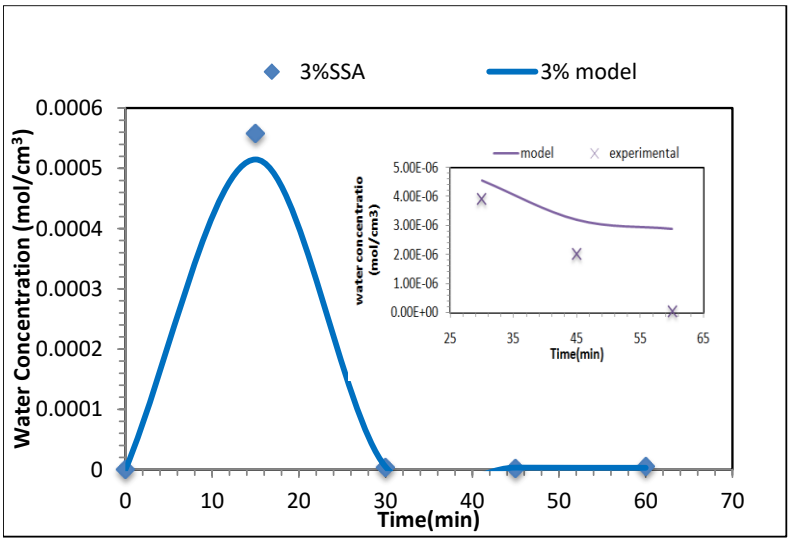


**Figure 5** Comparison between predicted water concentration from model and experimental results with PV at $k_0$=3*10$^5$cm3/mol.min, T=60°C, MR=8:1, wt=3%, S/V=1.3cm$^{-1}$

**Model Validation for different conditions**

The model was validated at the following set of conditions: 60°C, 5% SSA, MR 4:1, and a membrane area to the mixture volume of 1.3 cm$^{-1}$. The reaction conversion fitted that predicted from the developed model under the same conditions and the same model and design variables:

- Pre-exponential factor in Arrhenius equation, $k_o$; 300000 cm$^3$/mol.min.
- Pervaporation empirical constant, $K_P$; 4 cm/min.
- Catalyst concentration, Cc; 0.00027 mol/cm$^3$.
- Activation energy , E; 6906 J/mol

Figure (6) illustrates that the predicted reaction conversion after 30 min was 92.5% compared to 79.5% at the same conditions. The model was tested using the aforementioned design parameters. It is obvious that the reaction conversion calculated from experimental data and that predicted by the model are in agreement, which implies that the model is valid and applicable for different conditions. The obtained results explain that the amount of water in the reactor decreased using PV due to water adsorption through PV hydrophilic membrane. Figure (7) shows the clear difference in the amount of remaining water using PV.

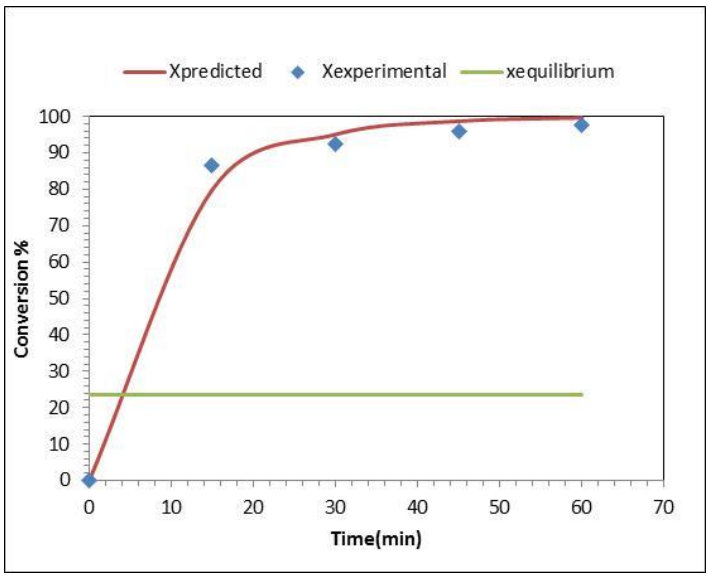


**Figure 6** Comparison between predicted reaction conversion from model and experimental results with PVat $k_0$=3*10$^5$cm3/mol.min, T=60$^o$C, MR=4:1, wt=5%, S/V=1.3cm$^{-1}$

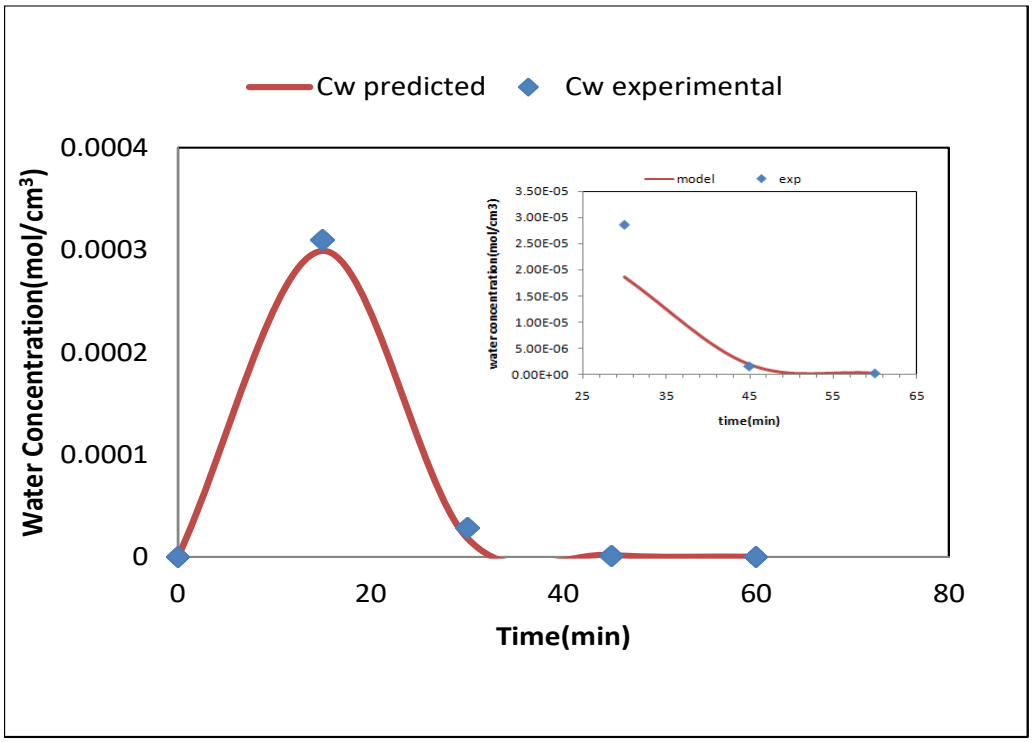


**Figure 7** Comparison between predicted water concentration from model and experimental results with PV at $k_0$=3*10$^5$cm3/mol.min, T=60$^o$C, MR=4:1, wt=5%, S/V=1.3cm$^{-1}$

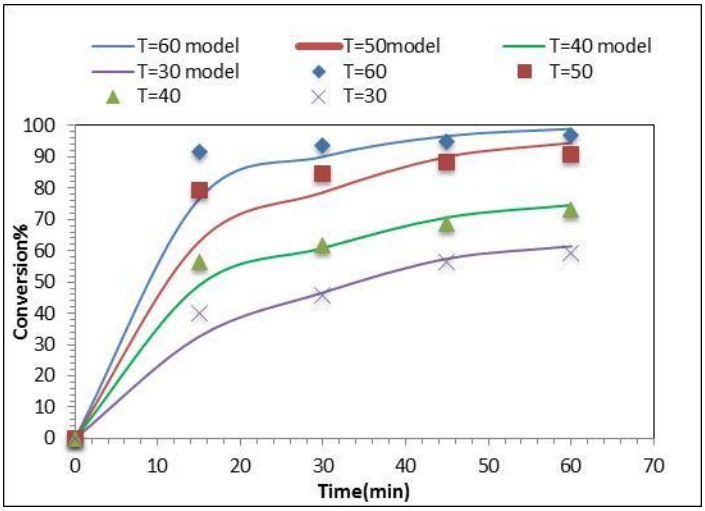


**Figure 8** Comparison between predicted reaction conversion from model and experimental results with PV catalyzed by SSA at different temperatures

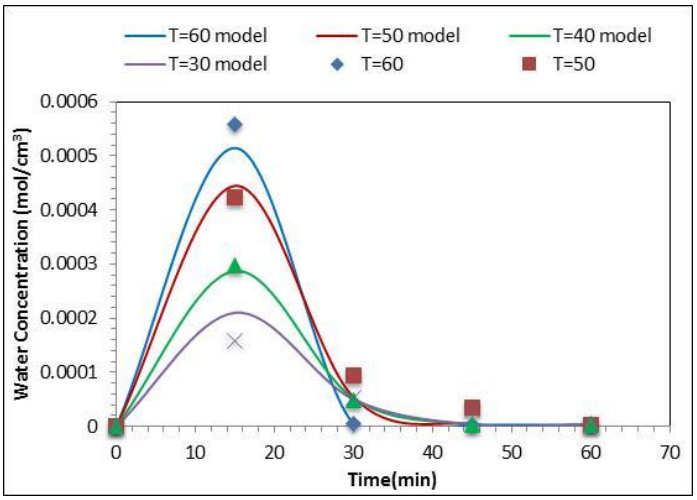


**Figure 9** Comparison between predicted results from model and experimental results for produced water concentration with PV catalyzed by SSA at different temperatures

**Parametric investigation**

Batch experiments were carried out under different conditions of molar ratio of methanol to acetic acid, temperatures, and catalyst weight percent. The experimental results of batch reaction coupled with pervaporation process were compared with the model output**.**

**Effect of reaction temperature**

Figures (8) and (9) show respectively the effect of temperature on the reaction conversion of acetic acid and water content in the reactor. The temperature was changed from 40 to 60˚C at a fixed percent value of SSA catalyst weight (wt=3 %), reactants ratio (MR=8:1 methanol to acetic acid respectively) and S/V ratio =1.3 $cm^{-1}$. The predicted data are in fair agreement with experimental results. The highest conversion obtained 96.76% was at 60˚C after 60 min. The rate of water production also increased with temperature. The results show that, at all operating temperatures, the rate of water production increased in the early stage of reaction, it then decreased gradually with time as shown in Figure (9). It is obvious that the reaction rate constant increases with temperature and that the equilibrium conversion also increases due to breaking of the thermodynamic barrier by water removal by PV membrane (J. Maa et al., 2009, S. Korkmaz et al., 2009, C.S.M. Pereira et al., 2010).

**Effect of reactant molar ratio (Methanol/ acetic acid, MR)**

Figures (10) and (11) show the effect of initial reactant molar ratio on the performance of pervaporation–esterification. The studied reactant ratios ranged from 2:1 to 8:1 methanol to acetic acid at fixed values of other parameters: temperature (60°C), SSA catalyst weight (wt%=3 %)and S/V ratio =1.3 $cm^{-1}$. The predicted reaction conversions were in conformity with the experimental results as shown in Figure (11). The highest conversion obtained after 60 min (96.76%) was observed at the high molar ratio (8:1 MR).

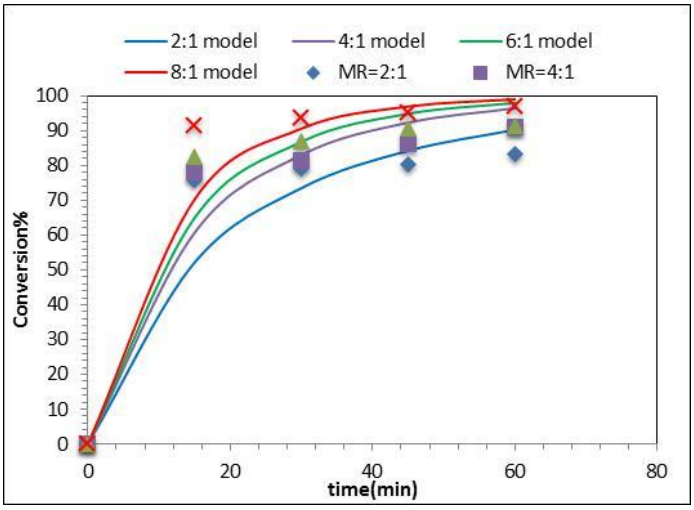


**Figure 10** Comparison between predicted reaction conversion from model and experimental results with PV catalyzed by SSA at different molar ratios

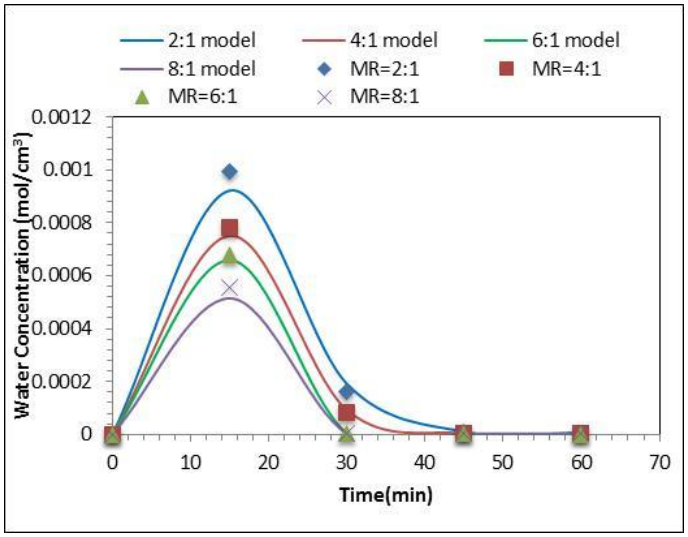


**Figure 11** Comparison between predicted results from model and experimental results for produced water concentration with PV catalyzed by SSA at different molar ratios

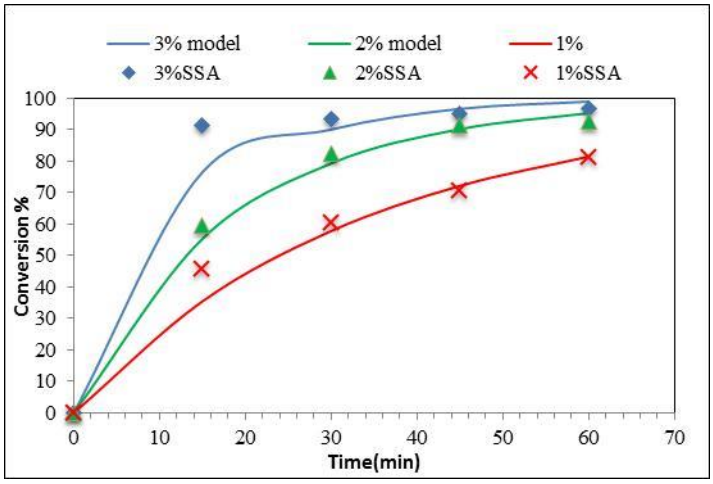


**Figure 12** Comparison between predicted reaction conversion from model and experimental results with PV catalyzed by SSA at different catalyst weight percent

**Effect of catalyst weight percent (wt %)**

The heterogeneous catalyst (silica sulfuric acid SSA) has a significant effect in accelerating the rate of ester production. It was used to study the effect of catalyst weight percent on the reaction conversion. Figure (12) and Figure (13)depict respectively the effect of SSA catalyst weight percent on the progress of the reaction pervaporation unit. There is fair matching between experimental and predicted data. The SSA weight percent was varied from 1% to 3% for a temperature of 60˚C, reactants molar ratio (MR=8:1) and S/V ratio =1.3 $cm^{-1}$. It can be seen that, increasing the SSA weight % increases reaction conversion. The conversion reached 96.76% after 60 min at 3% SSA wt%.

Increasing the catalyst weight percent increases the rate of water formation in the early stages of the reaction. This is because the rate of water production associated with the catalytic effect is higher than the rate of water removal by pervaporation. For quasi – complete conversion the amount of water in the reactor will be quasi –completely extracted by PV unit. Thus, the water remaining in the reactor in the final reaction stages is higher at lower SSA wt%.

The reaction conversion results under all experimental conditions are plotted versus their model output values in Figure (14). The results show that the kinetic model is in good agreement with experimental data.

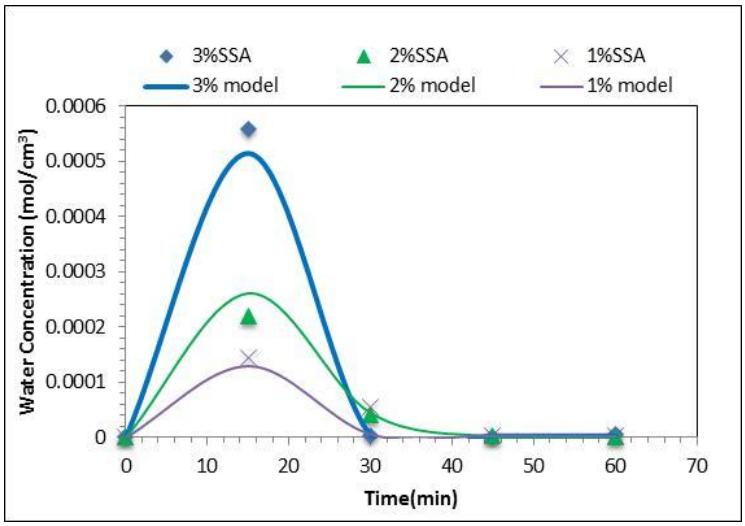


**Figure 13** Comparison between predicted results from model and experimental results for produced water concentration with PV at different SSA weight percent

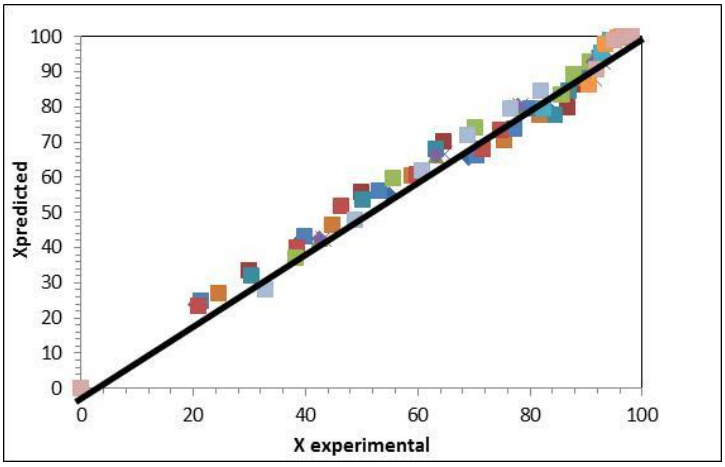


**Figure 14** Model output versus experimental results for different operating conditions

## 4. CONCLUSION

Coupling of pervaporation with esterification of methanol and acetic acid is an ideal alternative method for enhancing the esterification reaction. This reduces reaction time with low energy consumption. It is a cleaner operation compared to distillation since it has no end of pipe waste. A lumped parameter model was developed for esterification in a RBPMR. The model was verified, adjusted and validated using experimental results. The initial reaction constant was adjusted to provide good agreement between the reaction conversions determined from the model and experimental results. The model optimum reaction conditions obtained are a MR of 8:1 (Methanol: acetic acid), a temperature of 60˚C, and a catalyst wt% of 3%. The results indicate that the kinetic model is in good agreement with experimental results.

**Nomenclature**

$C_a$ : Concentration of acetic acid in reactor (mol/cm$^3$)
$C_{a0}$ : Initial concentration of acetic acid ( mol / cm$^3$)
$C_b$ : Concentration of methanol in reactor (mol/cm$^3$)
$C_e$ : Concentration of methyl acetate in reactor (mol/cm$^3$)
$C_w$: Concentration of water in reactor (mol/cm$^3$)
D : Diffusion coefficient (cm$^2$/min)
E : activation energy (J/mol)
$\Delta G$ : Gibbs free energy of reaction (J / mol)
$\Delta H$ : Heat of reaction (J / mol)
$J_i$ : flux (mol / cm$^2$. min)
$k_f$ : Forward reaction rate constant (cm$^3$/mol min)
$k_b$ : Backward reaction rate constant (cm$^3$/mol min)
$k_o$ : Pre-exponential factor in Arrhenius equation (cm$^3$/mol min)
$K_{eq}$ : Equilibrium constant
$K_p$ :Pervaporation empirical constant (min / mol)
r : rate of reaction (mol/cm$^3$ min)
R : the universal gas constant (8.314 J / mol.k)
S : Membrane area (cm$^2$)
T : temperature (K)
t : reaction time (min)
V : Volume (cm$^3$)
$x_i$ : the mole of component in the liquid phase (feed)

**Greek symbols**

$\rho_i^M$ : The molar density of component i (mol/cm$^3$)
$\theta_i$ : Ratio of initial concentration of component i to initial concentration of the limiting reactants
$\sigma_i$ : coefficient equal (-1) for reactants & (+1) for products

$\lambda_i$ : permeability coefficient equal (0) for component a,b and e, while it equal (+1) for component w
$\delta$ : membrane thickness (cm)

**Funding:** This research received no external funding.

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

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