

SIMULATION OF ETHYL ACETATE HYDROLYSIS IN A CSTR USING ASPEN HYSYS SOFTWARE

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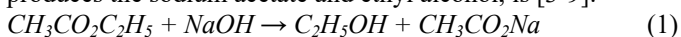
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Abstract— In this paper, Aspen HYSYS V12.0 is used to simulate ethyl acetate hydrolysis with sodium hydroxide in Armfield CEM-MKII CSTR module at steady-state. In order to simulate the process as accurately as possible PRSV package was used as the property method. The results obtained from the simulation were verified by the experimental data. The correlation coefficients (R^2) were 0.998 and 0.999 for reaction conversion (X) and outlet coil heating water temperature (T_{out}), respectively. It proved that simulation results satisfactorily fit for the experimental model.

Keywords— CSTR, Dynamic simulation, Ethyl Acetate Hydrolysis, Aspen HYSYS

I. INTRODUCTION

The continuous stirred tank reactor (CSTR) is a cylindrical vessel, with a height to diameter ratio (H/D) ranged between 2 to 4 for effective mixing and often have baffles to enhance mixing [1]. The baffles are used to ensure the reaction mixture is perfectly mixed. However, it prevents the occurrence of any angular, radial or axial gradients in temperature and concentrations. Therefore, the distribution of temperature and concentrations inside the CSTR are uniform, whereby the reaction rate is uniform at any point inside the CSTR [2]. The hydrolysis of ethyl acetate by sodium hydroxide, which produces the sodium acetate and ethyl alcohol, is [3-9]:



The reaction rate for the aforementioned ethyl acetate hydrolysis reaction is given as [10-12]:

$$-r = -r_{NaOH} = k C_{NaOH} C_{ElAC} \quad (2)$$

The reaction is a first order with respect to the sodium hydroxide and the ethyl acetate and it is a second order overall.

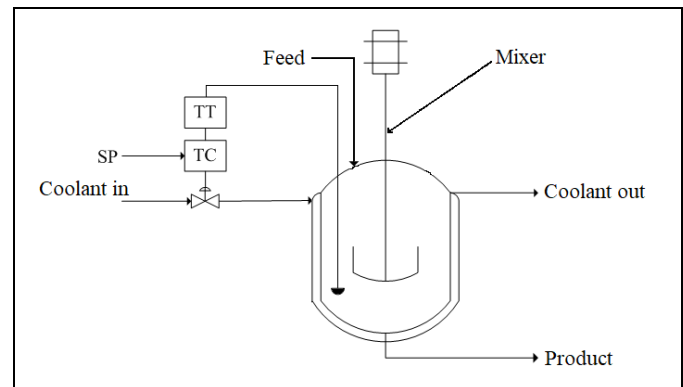


Fig. 1: CSTR Temperature Control using Conventional Feedback System [13].

This reaction is considered to be an equi-molar, non-catalytic, exothermic and an irreversible reaction [14]. Mukhtar et al. [15], studied the kinetics of ethyl acetate hydrolysis with sodium hydroxide at different temperatures and development of mathematical model for holding time in batch reactor. They found the overall reaction order 1.3118 and cannot be expressed satisfactorily as a second order reaction, especially when equimolecular concentrations of both reactants are used. They found the activation energy equals to 4.409 kJ/mole, the heat of reaction ($\Delta H_{R,298}$) -36920 kJ/mol and Gibbs free energy (ΔG_{298}) -24100 kJ. According to Qadir et al. [8], the saponification of ethyl acetate with caustic soda is shifting



order and cannot be expressed as second order reaction kinetics, also it has been found that the reaction is exothermic in nature and low reaction temperature favors the high conversion and high reaction rate. Ghobashy et al. [16], studied the production parameters ethyl acetate hydrolysis process in a PFR using an anion ion-exchange as a catalyst and acid-base titrations. They determined the activation energy as 32669.86 kJ/mol and rate constant as 331.7893. They found the reaction order as a second-order reaction. Mesfer et al. [17], studied the batch reactor performance optimization using multiple regression analysis for hydrolysis of ethyl acetate by sodium hydroxide. They specified agitation rate and reactants concentration as significant operating parameters. They found the maximum conversion 99.5% was obtained under optimum operating conditions of agitation rate 70 rpm and reactants concentration 0.05 M. Borovinskaya et al. [9], investigated ethyl acetate hydrolysis in T-shaped, interdigital and the chicane microreactors. They studied volumetric flow rate effect on reactor performance, and reported that T-shaped reactor shows good performance only at high flow rates.

The utilization of Aspen HYSYS to simulate ethyl acetate hydrolysis with sodium hydroxide in a CSTR at steady-state and dynamic simulation offers all state variables and parameters in order to model ethyl acetate hydrolysis.

II. METHODOLOGY

A. Experimental Setup

The procedure of ethyl acetate hydrolysis using sodium hydroxide was done in an experimental Armfield CEM-MKII CSTR connected to water bath as heating water utility source is as follow:

- The water bath was connected to a centrifugal pump (NOP35) with flow meter to measure the heating water flow rate that enters the CSTR heating coil.
- The reaction volume inside a CSTR was adjusted as 1750 ml.
- A 0.1 M of sodium hydroxide was prepared by dissolved 16.3 gm into 200 ml of distilled water in a flask, and then diluted into 4 litres.
- A 0.1 M of ethyl acetate was prepared by measured 40 ml of ethyl acetate using burette, and then diluted into 4 litres of distilled water.
- The flow rates of ethyl acetate and sodium hydroxide were adjusted at 66.67 ml/min for both, and the feed temperatures were measured by a thermometer.
- The flow rate of water bath was 66.67 ml/sec.
- The agitator speed was adjusted at 7.0 rps.
- The sodium hydroxide concentration was measured by conductivity meter prop.
- Two thermometers were used to measure the CSTR temperature and the outlet heating water from the heating coil.

- The sodium hydroxide electrical conductivity, the reactor temperature and the outlet heating water temperature were measured at steady-state.

The dimensions of Armfield CSTR obtained from Armfield CSTR CEM MkII manual [18], and the heating coil dimensions were measured manually as shown in table (1).

Table-1: Geometrical parameters of Armfield CSTR CEM MkII.

Design Parameter	Value
CSTR diameter, (m)	0.150
CSTR reaction volume, (liter)	1.750
Coil length, (m)	1.500
Coil inside diameter, (m)	0.050
Coil outside diameter, (m)	0.064

Sodium acetate Density and molecular weight were obtained from Haynes et al. [19] as 1.528 g/cm³ and 82.034 g/mol respectively. By pressing “Estimate Unknown Props” in Critical tab, Aspen HYSYS will estimate normal boiling point and critical properties for sodium acetate.

B. Steady-state Simulation

The reacting components ethyl acetate, sodium hydroxide, ethanol and water were selected from HYSYS databank, while sodium acetate was hypothesized by means of its molecular weight, normal boiling point and density. Based on considerations that mentioned in property package selection, the property model Peng-Robinson-Stryjek-Vera (PRSV) model catering to real and highly non-ideal (non-electrolytic) chemical systems was selected. The aforementioned reaction was defined in HYSYS by adding a reaction set. The kinetic data were selected from Wijayarathne and Wasalathilake (2014), which the activation energy (E_a) and frequency factor (k_o) were found to be 41400 kJ/mol and 2194760. First of all a steady state reaction was conducted. Both reactants entered the CSTR at 34 °C, 1 atm and 0.1 M. The feed flow rates for the ethyl acetate and sodium hydroxide were entered as 56.0 and 52.0 ml/min respectively. After clicking on the simulation environment tab, the material streams, energy stream and CSTR were installed in the simulation environment. The reactants and product streams conditions and the reactants streams compositions were entered. The simulated reaction system consisted of 1750 ml reaction volume of cylindrical reactor fabricated with glass with heating water system. The energy stream was connected to the CSTR, which represented the heating water coil that heats up the reaction mixture in experimental CSTR. Besides that, the logical unit (SET-100) was added to adjust the mass flow rate of stream (S-2) by means of stream (S-1) using multiplier equals 1.00. The logical unit (SET-100) is used to avoid fluctuations that occurs when the simulation transfer

from steady-state mode to dynamic mode. In order to define the specific heat capacity of cooling water utility, the inlet heating water temperature was inserted to HYSYS as 42 °C by clicking on “Process Utility Manager” tab. The streams were connected to the CSTR, and the reaction set was attached to the simulation environment. Stryjek and Vera (1986), were modified Peng–Robinson equation of state. They significantly improved the model's accuracy by introducing an adjustable pure component parameter and by modifying the polynomial fit of the acentric factor. The modification also gives improved fitting of the vapor pressure of normal fluids [20]:

$$P = \frac{RT}{V-b} - \frac{a_i a}{V^2 + 2bV - b^2}$$

where

$$a = (0.457235R^2 T_c^2 / P_c) a_i$$

$$b = 0.077796RT_c / P_c$$

$$a_i = [1 + \kappa(1 - T_r^{0.5})]^2$$

$$\kappa_0 = 0.378893 + 1.4897153\omega_{ac} - 0.17131848\omega_{ac}^2 + 0.0196554\omega_{ac}^3$$

$$\kappa = \kappa_0 + \kappa_1(1 + T_r^{0.5})(0.7 - T_r)$$

$$T_r = \frac{T}{T_c}$$

Stryjek and Vera (1986), were recommended to use for reduced temperature above 0.7 for all components. Eq. (3.6) becomes:

$$\kappa = \kappa_0$$

For individual user-added hypothetical components in Aspen HYSYS, the term (κ) can either be entered or they are automatically regressed against the Lee-Kesler correlation [21]. The kappa value, κ , is calculated using the estimated acentric factor (ω_{ac}) and critical temperature (T_r) for sodium acetate by Aspen HYSYS. On Aspen HYSYS Fluid Package tap, the value of kappa (κ) for sodium acetate is inserted on “PRSV Component Parameters”. The average jacket temperature is [22]:

$$T_j = \frac{T_{in} + T_{out}}{2} \quad (3)$$

Taking outlet temperature as a subject:

$$T_{out} = 2T_j - T_{in} \quad (4)$$

However, the overall heat transfer coefficient (U) is obtained by Luyben [1]:

$$\frac{1}{U} = \frac{1}{h_i} + \frac{A_i}{A_o h_o} + \frac{e}{A_i k_m} \quad (5)$$

Where e is the thickness of the reactor wall. The inside film coefficient, h_i , of an agitated vessel is [1]:

$$\frac{h_i D_o}{k} = a \left(\frac{D_{Agitator}^2 N \rho}{\mu} \right)^b \left(\frac{c_p \mu}{k} \right)^{1/3} \left(\frac{\mu}{\mu_w} \right)^m \quad (6)$$

Where D_T is diameter of tank, k is thermal conductivity of the process liquid, D_A is diameter of agitator, N is revolutions per second of agitator, ρ is density of process liquid, μ is viscosity of process liquid at temperature in vessel, μ_w is viscosity of process liquid at wall temperature, and c_p is heat capacity of process liquid. The heat transfer coefficient (h_o) for vertical helical coil is [23]:

$$h_o = 0.17 \left(\frac{k}{d_i} \right) \left(\frac{D_A^2 N \rho}{\mu} \right)^{0.67} \left(\frac{c_p \mu}{k} \right)^{0.37} \left(\frac{D_A}{D_T} \right)^{0.1} \left(\frac{d_i}{D_T} \right)^{0.5} \left(\frac{\mu}{\mu_w} \right)^m \quad (7)$$

However, the heating coil energy balance is [22]:

$$\dot{m}_{H_2O} c_p_{H_2O} (T_{j,in} - T_j) + U_{coil} A_{o,coil} (T_j - T_R) = 0 \quad (8)$$

Hence, the outlet coil temperature is:

$$T_{out} = 2 \left(\frac{U_{coil} A_{o,coil} T_R - \dot{m}_{H_2O} c_p_{H_2O} T_{in}}{U_{coil} A_{o,coil} - \dot{m}_{H_2O} c_p_{H_2O}} \right) - T_{in} \quad (9)$$

C. Validation of Simulation Model

Using the experimental data, the mean relative error (MRE), correlation coefficient (r) and root mean square error ($RMSE$), the absolute fraction of variance (R^2) and Root-Mean-Square of Relative Error ($RMSRE$) were used as criteria to validate the simulation model. These statistical methods were calculated by equations (10) - (14), respectively.

$$r = \frac{\sum_{i=1}^n (a_i - a_m)(p_i - p_m)}{\sqrt{\sum_{i=1}^n (a_i - a_m)^2 \sum_{i=1}^n (p_i - p_m)^2}} \quad (10)$$

$$MRE(\%) = \frac{1}{N} \sum_{i=1}^n \left| \frac{a_i - p_i}{a_i} \times 100 \right| \quad (11)$$

$$RMSE = \sqrt{\frac{1}{N} \sum_{i=1}^n (a_i - p_i)^2} \quad (12)$$

$$R^2 = 1 - \left(\frac{\sum_{i=1}^n (a_i - p_i)^2}{\sum_{i=1}^n p_i^2} \right) \quad (13)$$

$$RMSRE = \sqrt{\frac{1}{N} \sum_{i=1}^n \left(\frac{a_i - p_i}{a_i} \right)^2} \quad (14)$$

Where a_i is the experimental value; p_i is the simulated value; N is the number of data points. The correlation coefficient lies between -1.0 to +1.0. According to Ratner [24], a strong relationship when the correlation coefficient lies between ± 1.0 to ± 0.7 . The range of absolute fraction of variance between 0 to 1.0, with goodness fitting near 1.0 and weakness fitting near 0.

III. RESULTS AND DISCUSSION

1. Experimental Results

The feed temperature (T_{Feed}), reactor temperatures ($T_{Reactor}$), heating water flow rate (F_{coil}), reaction mixture conductivity (A), reaction conversion (X), inlet (T_{in}) and outlet (T_{out}) temperatures of heating water inside the coil were measured as shown in table (2). The temperature dependence of the electrical conductivity of reaction mixture is showed in fig. 2. The electrical conductivity of reaction mixture increases with increasing reactor temperature. From fig. 3, it is clear from the results, the fractional conversion increases as the reactor temperature increases.

Generally, the outlet coil temperatures are slightly decrease with increasing heating water flow rates, and that due to the mass flow rate is inversely proportional to difference between inlet and outlet heating temperatures. Such as experiments number (19) and (20), the inlet heating temperature at 39.4 °C

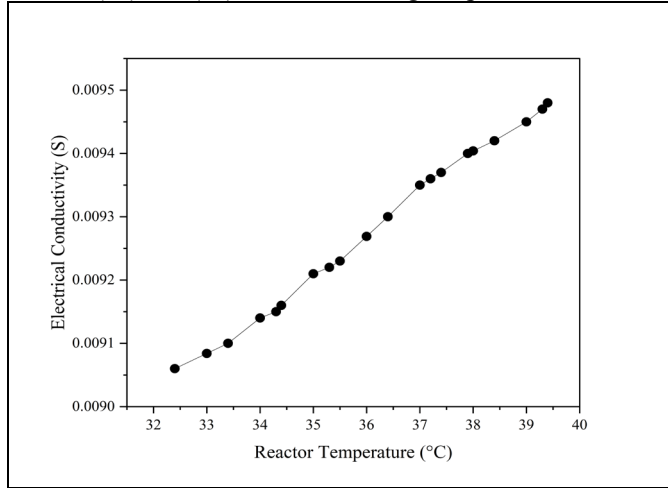


Fig. 2: Temperature Dependence of Electrical Conductivity for Ethyl Acetate Hydrolysis.

the heating water flow rate was as 0.066 and 0.0233 kg/sec respectively at constant feed and reactor temperatures and inlet coil temperatures varies 1°C, the outlet water temperature as 40°C in experiment (19) and 44°C in experiment (20), which increases 4°C when the flow rate decreases about 64.7%.

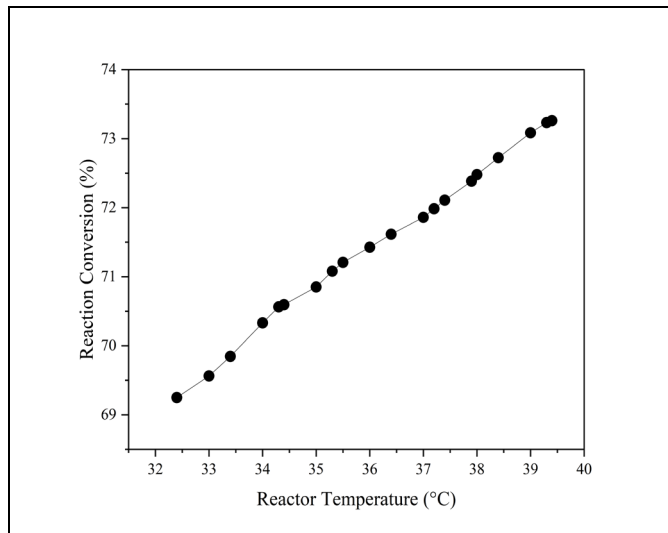


Fig. 3: Effect of Reactor Temperature on the Reaction Conversion.

The ethyl acetate hydrolysis with sodium hydroxide was simulated in a CSTR using Aspen HYSYS. Two feed streams, sodium hydroxide material stream (S-1) and ethyl acetate material stream (S-2) were defined as shown in fig. 4. The heating utility was defined as energy stream (Q-100).

Table -2: Operating Conditions and Reaction Conversion in Armfield CSTR CEM MkII.

No	T_{Feed} [°C]	F_{coil} [$\times 10^{-3}$ kg/sec]	$T_{Reactor}$ [°C]	A_t [mS]	[°C]	[°C]	X (%)
1	34.0	50.667	32.40	9.06	37.0	36.0	69.251
2	34.0	50.667	33.00	9.084	37.0	40.0	69.563
3	34.0	50.667	33.40	9.100	37.0	40.0	69.846
4	34.0	66.667	34.00	9.140	37.0	40.0	70.331
5	34.0	66.667	34.30	9.150	37.0	39.0	70.563
6	34.0	50.667	34.40	9.160	38.0	40.0	70.595
7	35.0	66.667	35.00	9.210	39.0	40.0	70.851
8	34.0	66.667	35.30	9.220	39.0	40.0	71.080
9	36.0	50.667	35.50	9.230	38.0	40.0	71.209
10	32.0	66.667	36.00	9.269	40.0	41.0	71.428
11	32.0	20.004	36.40	9.300	38.0	41.0	71.615
12	32.0	66.667	37.00	9.350	41.0	41.0	71.860
13	32.0	66.667	37.20	9.360	41.0	42.0	71.985
14	32.0	20.004	37.40	9.370	38.0	42.0	72.109
15	32.0	66.667	37.90	9.400	42.0	41.0	72.385
16	32.0	27.70	38.00	9.404	39.0	43.0	72.479
17	32.0	23.338	38.40	9.420	39.0	43.0	72.723
18	32.0	23.338	39.00	9.450	40.0	45.0	73.086
19	32.0	66.667	39.30	9.470	43.0	40.0	73.233
20	32.0	23.33	39.40	9.480	42.0	44.0	73.261

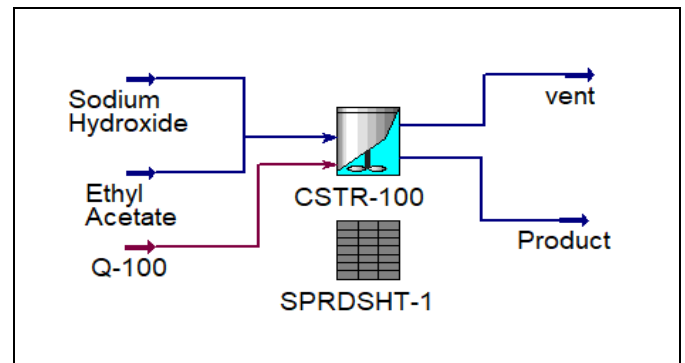


Fig. 4: CSTR Flow Sheet in Aspen HYSYS Simulation Environment.

2. Simulation Results

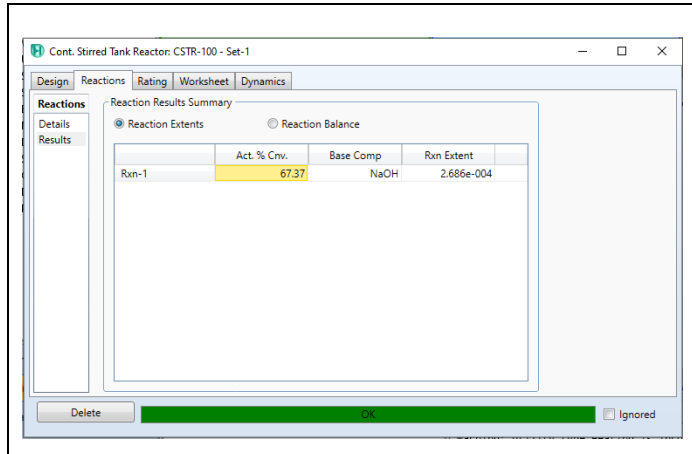


Fig. 5: Reaction Conversion of Sodium Hydroxide.

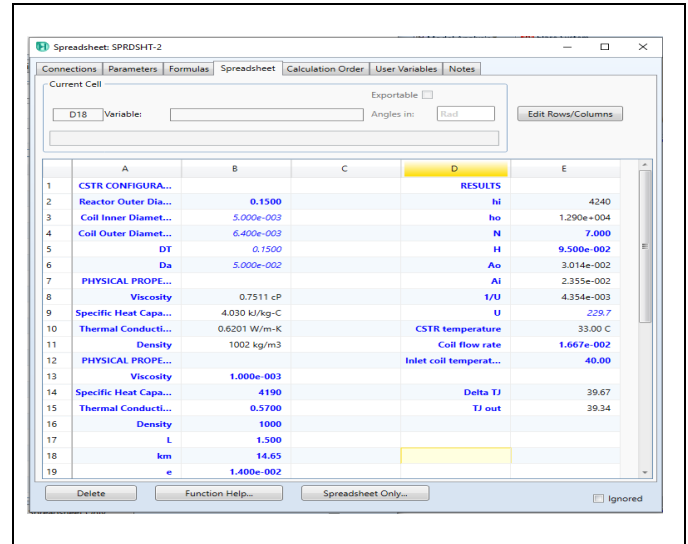


Fig. 6: Spreadsheet Cells for Calculating Outlet Heating Water Temperature inside the Coil.

Table-3: Percentage Error between Experimental and Simulated Results for Reaction Conversion and Outlet Heating Water.

X [%]			T_{out} [°C]		
Experimental	Simulated	E (%)	experimental	simulated	E (%)
69.251	67.049	3.179	36.0	35.7	-0.746
69.563	67.241	3.338	40.0	43.1	7.730
69.846	67.368	3.549	40.0	42.1	5.259
70.331	67.555	3.947	40.0	40.8	2.090
70.563	67.648	4.132	39.0	40.6	4.191
70.595	67.678	4.132	40.0	41.7	4.270
70.851	67.861	4.221	40.0	40.2	0.411
71.080	67.951	4.402	40.0	41.6	4.087
71.209	68.011	4.491	40.0	42.6	6.523
71.428	68.159	4.577	41.0	41.2	0.401
71.615	68.276	4.662	41.0	43.3	5.717
71.860	68.450	4.745	41.0	42.2	2.840
71.985	68.507	4.831	42.0	43.7	4.052
72.109	68.564	4.916	42.0	44.3	5.581
72.385	68.706	5.083	41.0	41.6	1.366
72.479	68.734	5.168	43.0	42.2	-1.831
72.723	68.845	5.333	43.0	42.2	-1.847
73.086	69.011	5.576	45.0	45.2	0.540
73.233	69.092	5.654	40.0	38.9	-2.629
73.261	69.119	5.653	44.0	43.2	-1.796

The exiting stream from the CSTR (S-4), is carried out the ethanol, sodium acetate, and the unreacted ethyl acetate, sodium hydroxide and water. Furthermore, a CSTR, spreadsheet, material streams and energy stream were defined as shown in fig. 5. The reaction conversion calculated by HYSYS on “Reaction” tab on CSTR, as shown in fig. 5. However, Fig. 6 shows energy balance calculations for CSTR on Spreadsheet “SPRDSHT” by importing physical properties of reaction mixture, tap water, and CSTR configuration. The percentage error of experimental and simulated reaction conversion, outlet heating water temperature is shown in table 3. The percentage error of conversion increases with reactor temperature increase at specified reactor volume. The variations between the calculated and simulated results for reaction conversion are due to the hypothetical component (sodium acetate); because of binary coefficients for solubility data is missing in Aspen HYSYS software. Whereby, accuracy of the results was affected. The differences between experimental and simulated results for outlet heating temperature due to tap water properties, which affects in coil heat transfer coefficient (h_o). In addition, the properties of hypothetical sodium acetate in reaction mixture properties, those affects in inside-film coefficient (h_i) for reaction mixture side.

From table 4, Aspen HYSYS simulation yields a correlation coefficient with a strong relationship between the experimental and simulated results, with a very good fit representing by R^2 above 0.9 for all parameters, providing an acceptable accuracy ($RMSRE < 0.1$) for conversion and outlet heating coil water temperature. The deviations between experimental and simulated results represented by $RMSE$ are under 3.329% for conversion, 1.557 °C for outlet heating temperature. MRE not exceeds 5% for both conversion and



outlet heating temperature respectively. These statistical methods represented excellent agreement between the experimental and simulated results which demonstrate good model verification.

Table-4: Performance Measurements for Simulated Results.

Performance Measures	Parameter	
	X	T_{out}
R	0.998	0.795
MRE (%)	4.579	3.073
$RMSE$	3.329	1.557
R^2	0.998	0.999
$RMSRE$	0.046	0.037

IV. CONCLUSION

In this paper, a simulation model was developed for ethyl acetate hydrolysis with sodium hydroxide inside a CSTR using Aspen HYSYS. The simulation model was validated by the experimental data obtained from Armfield CEM-MKII CSTR module. The experimental data showed a good fit to the HYSYS model.

Aspen HYSYS model showed that the reaction conversion increases as the reactor temperature and outlet heating coil temperature increase. Whereby, these simulation results improve the understanding of kinetics of ethyl acetate hydrolysis and would be very useful in the design of CSTRs.

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