

# Aspen Plus Simulation of Saponification of Ethyl Acetate in the Presence of Sodium Hydroxide in a Plug Flow Reactor

U. P. L. Wijayarathne, K. C. Wasalathilake

**Abstract**—This work presents the modelling and simulation of saponification of ethyl acetate in the presence of sodium hydroxide in a plug flow reactor using Aspen Plus simulation software. Plug flow reactors are widely used in the industry due to the non-mixing property. The use of plug flow reactors becomes significant when there is a need for continuous large scale reaction or fast reaction. Plug flow reactors have a high volumetric unit conversion as the occurrence for side reactions is minimum. In this research Aspen Plus V8.0 has been successfully used to simulate the plug flow reactor. In order to simulate the process as accurately as possible HYSYS Peng-Robinson EOS package was used as the property method. The results obtained from the simulation were verified by the experiment carried out in the EDIBON plug flow reactor module. The correlation coefficient ( $r^2$ ) was 0.98 and it proved that simulation results satisfactorily fit for the experimental model. The developed model can be used as a guide for understanding the reaction kinetics of a plug flow reactor.

**Keywords**—Aspen Plus, Modelling, Plug Flow Reactor, Simulation.

## I. INTRODUCTION

**P**LUG flow reactors (PFRs) also known as Continuous Tubular Reactors (CTRs) play a key role in chemical manufacturing facilities where a transformation of chemical substances takes place. In a plug flow reactor, feed is introduced from one end of a cylindrical tube and flows continuously through the length of the reactor as a series of plugs. The product is then collected from the other end of the reactor. In modelling it is assumed that inside a PFR, the fluids flow consistently and there is no radial variation in velocity, concentration, temperature, or reaction rate [1]. The main advantages of PFRs are that they have a high volumetric unit conversion and the capability of running for longer periods without maintenance. For isothermal reactions greater than zeroth order, the PFR volume will usually be lower than the Continuously Stirred Tank Reactor (CSTR) volume for the same conversion and reaction conditions (temperature, flow rate, etc.) [1].

For the industry, PFR can be configured as a single long tube or a bunch of short tubes depending on the application. The diameter of the cylindrical reactor varies from a few centimeters to several meters. Construction cost, pumping cost, residence time and heat transfer requirements are the

main factors which affects the choice of diameter. PFRs are widely used in the industry for gas and liquid phase systems. Gasoline Production, Oil cracking, oxidation of sulfur dioxide to sulfur trioxide, synthesis of ammonia, polymer manufacturing are some the industries where PFRs are being used broadly.

Though they are a number of applications, the controlling of PFR has become a challenging task when it comes to chemical manufacturing. Time varying characteristics of the process have made the operation of the reactor difficult and it needs extensive monitoring. For overcome such issues, modelling and simulation of reactors has become useful when it comes to process optimization and to investigate the effect of various operating parameters towards the process.

Practice of process simulation has evolved over last two decades in the field of engineering. When it comes to process engineering, there is a wide range of process simulation packages which are specifically developed for particular process areas namely, Aspen Plus, Chemcad and Petro-SIM. All these simulators follow the sequential modular approach where the user is able develop a process by combining standard modules in a logical manner. Aspen Plus is one of the industry's leading process simulation software which has the ability to design and simulate a chemical process comprehensively.

Aspen Plus has been used by investigators to simulate and optimize various kinds of process plants and unit operations. Sohail Rasool Lone et al. developed a model to simulate ethyl acetate reactive distillation column using a RADFRAC model [2]. Harjeet Nath et al. developed a model to simulate the abatement of fluorine inside a fluidized bed reactor using RCSTR and RGIBBS models [3]. Rajul Nayak et al. developed a model to simulate coal gasification using RYIELD, RGIBBS and RSTOIC models [4].

The primary objective of this study is to develop a model for a Plug Flow Reactor and to simulate the saponification of ethyl acetate in the presence of sodium hydroxide by varying the reactor temperature and reactant flow rates using Aspen Plus. In order to validate the applicability of the developed model, simulation results were compared with the experimental results collected using EDIBON PFR module [5]. During the laboratory experiment, fractional conversions of the reaction are determined using the conductivity values of the reactants and products under different temperatures.

Wijayarathne U.P.L and Wasalathilake K.C. are with the Department of Chemical & Process Engineering, University of Moratuwa, Sri Lanka (e-mail: udithaw@uom.lk and kimalw@uom.lk, respectively).

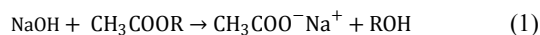
## II. NOMENCLATURE

T	- Absolute Temperature
C	- Concentration
x	- Fractional Conversion
$k_0$	- Frequency Factor
$\lambda_m$	- Molar Conductivity of an ion
k	- Rate Constant
r	- Rate of Reaction
v	- Reactor Volume
$T_{\text{reference}}$	- Reference Temperature
$\kappa_T$	- Solution Conductivity
$\kappa_{T \text{ reference}}$	- Solution Conductivity at $T_{\text{reference}}$
$\theta$	- Temperature Coefficient
R	- Universal Gas Constant
F	- Volumetric Flow Rate

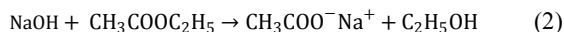
## III. THEORY

## A. Saponification

Saponification is the hydrolysis of a carboxylic acid ester in a basic medium (1). Saponification is essentially a soap-manufacturing process that takes place by reacting the fatty acid with alkali, especially triglycerides producing glycerol and carboxylate salts (soap).



In this experiment, saponification of ethyl acetate in the presence of sodium hydroxide is used to demonstrate the saponification reaction. The saponification of ethyl acetate reaction is as follows.



when it comes to chemical kinetics, this reaction is a prominent second order reaction identified in literature [6], [7].

In a PFR, reactant fluids are pumped through a tubular section where the reaction is carried out. At the inlet of the PFR, reactant concentration is very high, hence the rate of reaction is also high. But, when it passes through the reactor, reactants get consumed and products are formed. Therefore rate of reaction drops through the PFR with respect to reactor length.

## B. Conductivity Theory

Conductivity of a solution is given by,  $\kappa = \lambda_m C(3)$  [8]. Where,  $\lambda_m$  is the molar conductivity of an ion and C is the ion concentration in the solution. Feed stream for the PFR consists with equal volumetric flow rates of  $\text{NaOH}_{(\text{aq})}$  and  $\text{CH}_3\text{COOC}_2\text{H}_5_{(\text{aq})}$ . Since the conductivity value for  $\text{CH}_3\text{COOC}_2\text{H}_5_{(\text{aq})}$  is negligible with reference to  $\text{NaOH}_{(\text{aq})}$ , conductivity of the feed solution can be estimated as, [8]

$$\kappa_{\text{feed}} = [\lambda_{\text{Na}^+} \times C_{\text{Na}^+}] + [\lambda_{\text{OH}^-} \times C_{\text{OH}^-}] \quad (4)$$

Similarly for the product stream, since it consists with both

the products  $\text{CH}_3\text{COO}^- \text{Na}^+ + \text{C}_2\text{H}_5\text{OH}$  as well as the unreacted NaOH, conductivity of the product stream could be expressed as, [8]

$$\kappa_{\text{product}} = [\lambda_{\text{Na}^+} \times C_{\text{Na}^+}] + [\lambda_{\text{OH}^-} \times C_{\text{OH}^-}] + [\lambda_{\text{CH}_3\text{COO}^-} \times C_{\text{CH}_3\text{COO}^-}] \quad (5)$$

Furthermore, online conductivity meter displays the conductivity value for the solution as at 25°C. Therefore, it should be corrected to the operating temperature using the expression, [9]

$$\kappa_{T \text{ reference}} = \frac{100}{100 + \theta(T - T_{\text{reference}})} \times \kappa_T \quad (6)$$

Hydrolysis reaction of ethyl acetate with sodium hydroxide is in the form of  $A + B \rightarrow C + D$ . Moreover, the concentration of both the NaOH and  $\text{CH}_3\text{COOC}_2\text{H}_5$  are similar, the concentration of components at initially and any time t, can be expressed as follows.

TABLE I  
CONCENTRATION OF STREAMS WITH TIME

Time	$C_A$	$C_B$	$C_A$	$C_B$
t=0	$C_{A_0}$	$C_{A_0}$	0	0
t=t	$\frac{C_{A_0}(1-x_A)}{2}$	$\frac{C_{A_0}(1-x_A)}{2}$	$\frac{C_{A_0}x_A}{2}$	$\frac{C_{A_0}x_A}{2}$

By replacing the concentration terms in above (4) and (5),

$$\kappa_{\text{feed}} = [\lambda_{\text{Na}^+} \times C_{A_0}] + [\lambda_{\text{OH}^-} \times C_{A_0}]$$

and

$$\kappa_{\text{product}} = [\lambda_{\text{Na}^+} \times C_{A_0}] + [\lambda_{\text{OH}^-} \times \frac{C_{A_0}(1-x_A)}{2}] + [\lambda_{\text{CH}_3\text{COO}^-} \times \frac{C_{A_0}x_A}{2}]$$

Fractional conversion could be determined by solving for x.

## C. PFR Theory

Since the saponification of ethyl acetate with presence of sodium hydroxide is a second order reaction [6], rate of the reaction can be expressed as follows.

$$r = kC_{\text{NaOH}}C_{\text{CH}_3\text{COOC}_2\text{H}_5} \quad (7)$$

According to the Arrhenius equation

$$k = k_0 \exp\left(\frac{-E}{RT}\right) \quad (8)$$

Tubular reactors falls under the category of flow reactors where the key assumption is that the fluid is perfectly mixed in the radial direction but not in the axial direction (zero back mix). Each plug of differential volume is considered as a batch reactor where the volume tends to be zero.

The PFR model is well applied for many fluids. Ideal PFR consist with a constant residence time which is same as the space time if other conditions such as pressure and temperature are same throughout the reactor [10]-[12].

## D. Plug Flow Reactor Design Equation

General assumptions made for mathematically model an

ideal plug flow reactor,

- There is no mixing in the axial direction (direction of flow)
- Complete mixing in the radial direction
- Uniform velocity profile across the radius

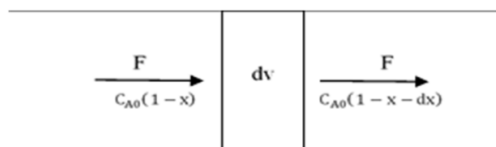


Fig. 1 Elemental material balance for a PFR

Applying material balance for component A (NaOH) over small elemental volume  $dv$  under the steady state operation results,

$$\begin{aligned} C_{A0}(1-x)F - (-r_A dv) &= C_{A0}(1-x-dx)F + 0 \\ -r_A dv &= C_{A0}F(dx) \\ \frac{V}{F} &= C_{A0} \int_0^x \frac{dx}{-r_A} \end{aligned} \quad (9)$$

Residence time is same as the space time and given by

$$\tau = \frac{V}{F} \quad (10)$$

Therefore,

$$\tau = C_{A0} \int_0^x \frac{dx}{kC_{A0}^2(1-x)^2} \quad (11)$$

#### IV. METHODOLOGY

##### A. Materials

All chemicals used for this work are of analytical reagent grade. The stock solutions of NaOH (~0.1 M) and ethyl acetate (~0.1 M) were prepared using distilled water. Ethyl acetate from Sigma-Aldrich Corporation, Spruce St., St. Louis, Missouri United States with 99% assay (density:  $0.902 \text{ g cm}^{-3}$ ) was directly used as stock solution. 99.8% assay NaOH pellets from Merck Specialties Private Limited, Mumbai, India was used and the stock solution of NaOH was standardized against freshly prepared standard hydro chloric acid.

##### B. Experimental Setup

###### 1. Reactor Module

Plug Flow Reactor in EDIBON Computer Controlled Chemical Reactors Training System was used to carry out the hydrolysis reaction. The experimental setup was mainly equipped with following components.

- Two computer controlled feed pumps coupled with flow meters to measure the flow of liquids in the range of 0.7-7 l/h and 0.54-5.4 l/h
- Thermostatic bath of 9 liters capacity that regulates the temperature between ambient temperature and  $70^\circ\text{C}$  with a level switch and a pump which are computer controlled
- An online feed pre-heater prior to introduce the reactants

to the tubular section where the reaction is carried out

- Tubular section is having a diameter of 5.8mm and a length of 16.5m
- Type “J” temperature sensor to get the temperature of the reactor in a continuous way
- An online conductivity meter (CRISON CM38)
- Three Pyrex-glass tanks of 10 liters capacity, two of them for the reagents and the other one for the accumulation of product.

###### 2. Conductivity Monitoring Instrument

CRISON CM38, an online conductivity and temperature meter available with EDIBON PFR module was used to measure the conductivity and the temperature of the product stream. According to the product specifications, this online device is capable of measuring the conductivity of a solution in the range of  $0.01 \mu\text{S/cm}$  to  $199.9 \text{ mS/cm}$  with error percentage  $\leq 1\%$  and also it is capable of measuring the temperature of a solution in the range of  $-20^\circ\text{C}$  to  $150^\circ\text{C}$  with error  $\leq 0.5^\circ\text{C}$ . Reference temperature for the measuring device is  $25^\circ\text{C}$  and all the conductivity values display as at reference temperature [13].

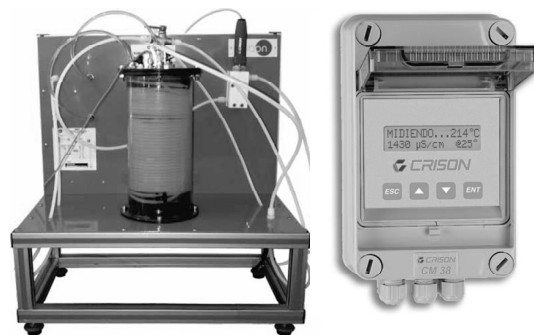


Fig. 2 PFR and Conductivity meter

###### C. Method

10 liter of 0.1M NaOH and 0.1M  $\text{CH}_3\text{COOC}_2\text{H}_5$  (ethyl acetate) solutions were prepared and stored in feed vessels. Plug Flow Reactor module was switched on and the water bath temperature was set to  $30^\circ\text{C}$ . Circulation pump was switched on in order to circulate the hot water to the shell side of the PFR. Flow rate of 0.1M NaOH was set to 2.4 l/h and allowed the system to reach steady state. Once the conductivity value was stable, it was recorded from the online conductivity meter. Water bath temperature was gradually increased to  $35^\circ\text{C}$ ,  $40^\circ\text{C}$  and  $45^\circ\text{C}$  while maintaining the flow rate of NaOH solution at 2.4l/h. Conductivity values corresponding to these temperatures were recorded for latter calculation requirements. Then the PFR was thoroughly washed using distilled water while allowing the water bath to cool down.

Once the temperature of the PFR reached to  $30^\circ\text{C}$ , flow rates of NaOH& ethyl acetate was set as 2.4 l/h and both the feed pumps were switched on simultaneously to feed the reactants to PFR.

Hydrolysis reaction was carried out inside the PFR

according to the reaction (2).

System was allowed to reach steady state operation and the conductivity of the product stream was recorded using the online conductivity meter. Water bath temperature was again gradually increased to 35°C, 40°C and 45°C while maintaining the flow rates of the reactants at 2.4 l/h. Conductivity values of the product stream corresponding to these temperatures were also recorded.

Fractional conversion for the reaction (2) was calculated using the conductivity values recorded from the product stream at different temperatures. These results were compared with the simulation results from Aspen Plus V8.0.

#### D. Method Aspen Simulation

PFR was modeled in Aspen Plus according to the dimensions and the operating conditions of the EDIBON reactor module. Then it was compared with the experimental results. HYSR was used as the property method for the Aspen model.

There are various physical property methods available in Aspen Plus V8.0 which can be used for the estimation of physical properties. HYSR (HYSYS Peng-Robinson EOS package) property model was used in this study [14].

Two inbuilt models of a Mixer and a PFR were used to model the system (Fig. 3). In the experimental setup two reactants were mixed together before introducing to the PFR. Therefore a model of a Mixer was used in the aspen simulation to represent this operation. Two feed streams, Ethyl acetate and Sodium hydroxide were defined as ESTER and NAOH respectively. Concentration of the feed solutions were set as 0.1 mol/dm<sup>3</sup> where, the solvent was defined as water since both the reactants were in aqueous medium and the flow rate for each feed was set as 2.4 l/h. Temperature and pressure for feed streams were set as 30°C and 1 atm respectively.

PFR model in the aspen simulation represent the tubular reactor in the experimental module and the parameters were set accordingly. Length of the reactor was 16.5m and the diameter of the tube was 5.8mm. Phase of operation for the experimental model was only liquid and therefore valid phase the PFR model in the Aspen was selected as liquid- only (Fig. 4). Operating condition for the PFR was set as 30°C and 1atm.

After setting up the physical parameters, reaction kinetic parameters were introduced to the Aspen model. According to the literature [8], the saponification reaction follows the power law (7) and hence it was selected as the reaction class in the Aspen model. Driving force was also set as 'exponent' of one for both the reactants since the reaction rate is proportional to the concentration of each of the reactant. Activation energy ( $E_a$ ) and frequency factor ( $k_0$ ) were found to be 41400 kJ/kmol and 2194760 respectively [8] and those values were fed to the Aspen model (Fig. 5). The results that were obtained upon the simulation of the model are given in Table III.

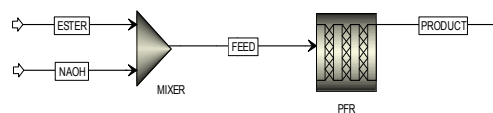


Fig. 3 Aspen Flow Sheet

Fig. 4 PFR Configuration

Fig. 5 PFR Reaction Kinetics Data

## V. RESULTS AND DISCUSSION

### A. Conductivity vs. Temperature

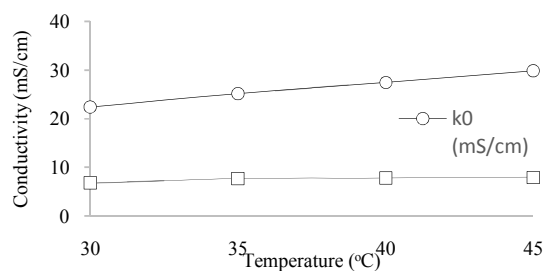


Fig. 6 Solution Conductivity vs. Temperature

Conductivity values of both NaOH stream and product stream were adjusted to the operating temperature of the reactor according to (6) and were plotted against the reactor temperature (Fig. 6). The conductivity values of the solution show linear dependence on the reactor temperature.

### B. Comparison of Simulated Results and Experimental Results

Table II shows the calculated results for the fractional conversion of the saponification reaction at different reactor temperatures for both the experiment and the simulation. It is clear from the results that in both the cases, the fractional conversion increases as the reactor temperature increases.

The experimental values obtained for fractional conversion were correlated with the results obtained from the simulation (Fig. 7). The correlation coefficient ( $r^2$ ) was 0.98 and it proved that simulation results show a good fit for the experimental model.

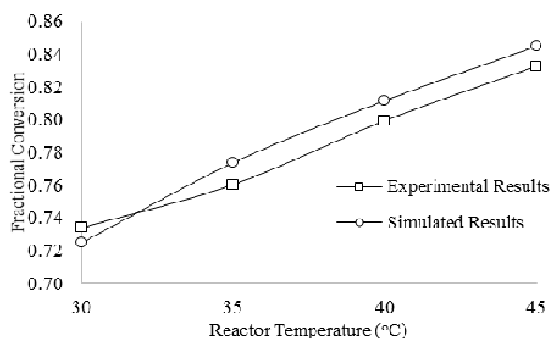


Fig. 7 Fractional conversion vs. reactor temperature

### C. Effect of Temperature on the Reaction

TABLE II  
FRACTIONAL CONVERSION AT DIFFERENT REACTOR TEMPERATURES

Reactor Temperature (°C)	30	35	40	45
Experimental results	0.73	0.76	0.80	0.83
Simulated results	0.72	0.77	0.81	0.84

Figs. 8-11 show the variation of molar fractions of ethyl acetate and sodium acetate at the reactor temperatures of 30°C, 35°C, 40°C and 45°C respectively. It is clear from these figures that the molar fractions of reactants decrease with the increment of reactor temperature. This is due to the fact that the reaction rate increases with the reactor temperature and hence, the molar fractions of products increase (7&8).

Fig. 13 shows the curve for molar flow of sodium acetate obtained under different reactor temperatures (From 25°C to 200°C). It can be seen that the sodium acetate molar flow rate increases with the reactor temperature and it reaches a maximum value of 0.24 mol/h when the reactor temperature is 150°C.

It is also evident that the molar fractions of products increase with increment of length of the plug flow reactor. This is because the residence time of reactants increases with the length of the reactor, where it allows sufficient time for the reaction to take place (11).

TABLE III  
RESULTS SUMMARY

	Caustic	Ester	Product at 30°C	Product at 35°C	Product at 40°C	Product at 45°C
H <sub>2</sub> O kmol/h	1.7055E-01	1.6950E-01	3.4005E-01	3.4005E-01	3.4005E-01	3.4005E-01
CH <sub>3</sub> COOC <sub>2</sub> H <sub>5</sub> kmol/h	-	2.4000E-04	6.6052E-05	5.4319E-05	4.5229E-05	3.7255E-05
NaOH kmol/h	2.4000E-04	-	6.6052E-05	5.4319E-05	4.5229E-05	3.7255E-05
CH <sub>3</sub> COO <sup>-</sup> Na <sup>+</sup> kmol/h	-	-	1.7395E-04	1.8568E-04	1.9477E-04	2.0275E-04
C <sub>2</sub> H <sub>5</sub> OH kmol/h	-	-	1.7395E-04	1.8568E-04	1.9477E-04	2.0275E-04
Total Flow kmol/h	1.7079E-01	1.6974E-01	3.4053E-01	3.4053E-01	3.4053E-01	3.4053E-01
Total Flow kg/h	3.08	3.07	6.16	6.16	6.16	6.16
Total Flow l/min	0.04	0.04	0.08	0.08	0.08	0.08

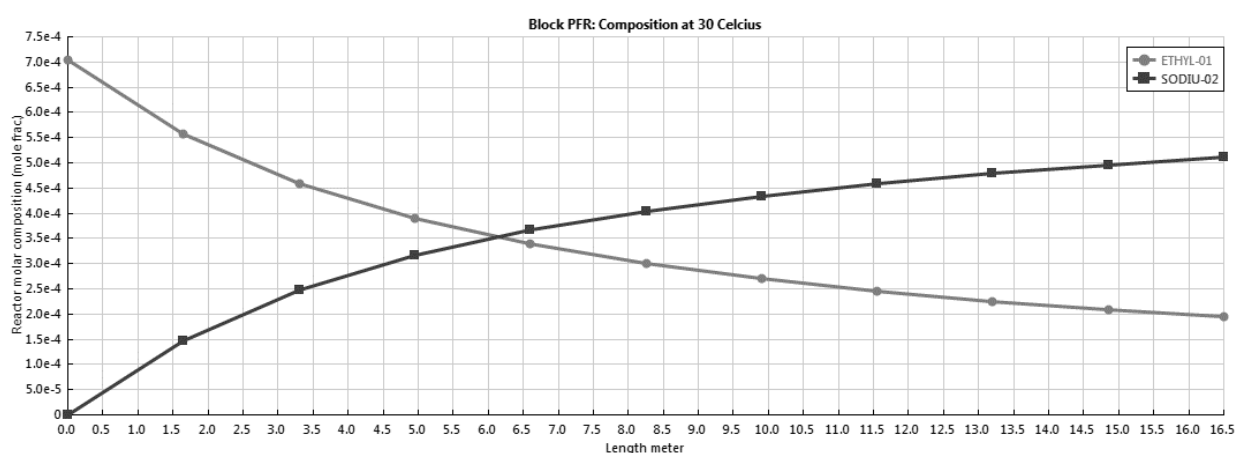


Fig. 8 Ethyl acetate mole fraction vs. PFR length at 30°C

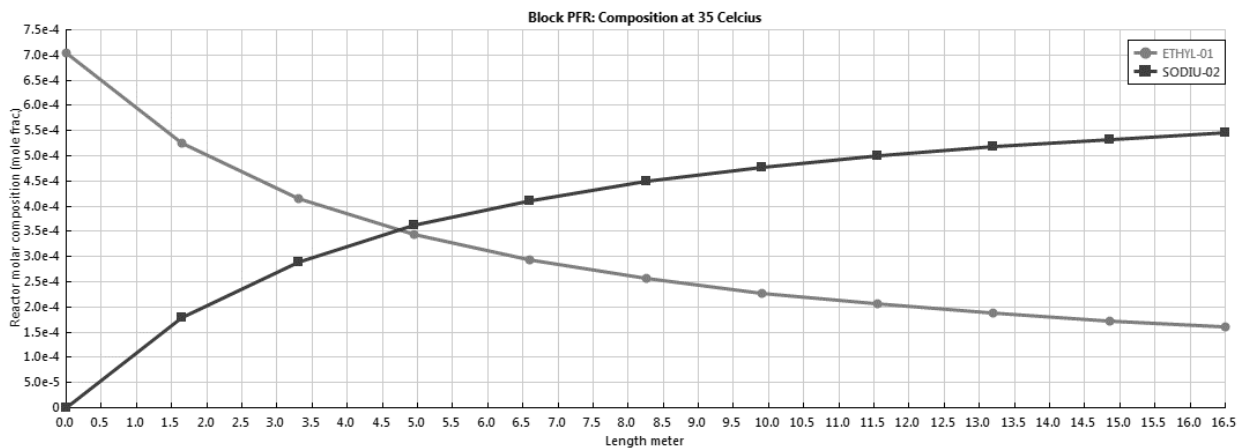


Fig. 9 Ethyl acetate mole fraction vs. PFR length at 35°C

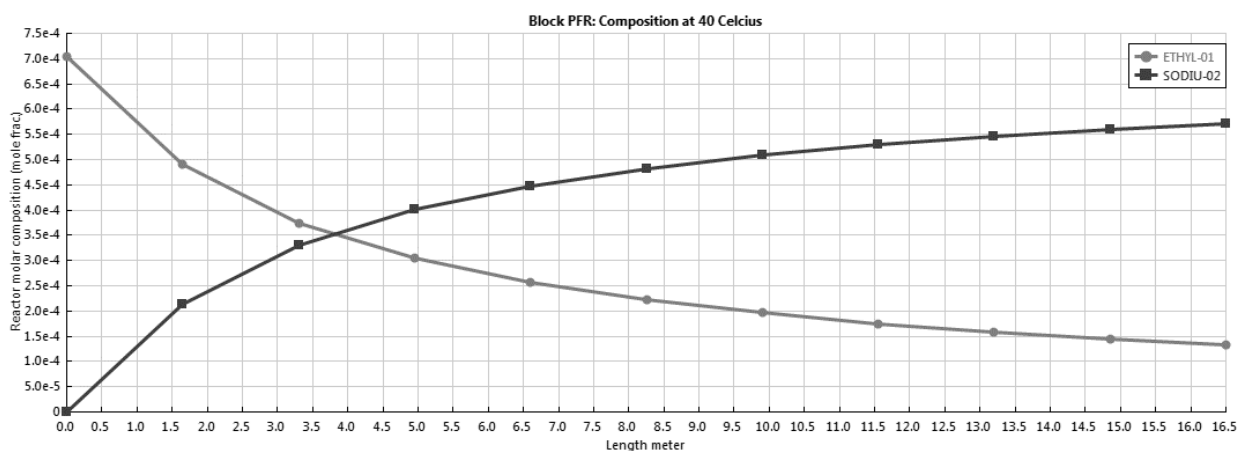


Fig. 10 Ethyl acetate mole fraction vs. PFR length at 40°C

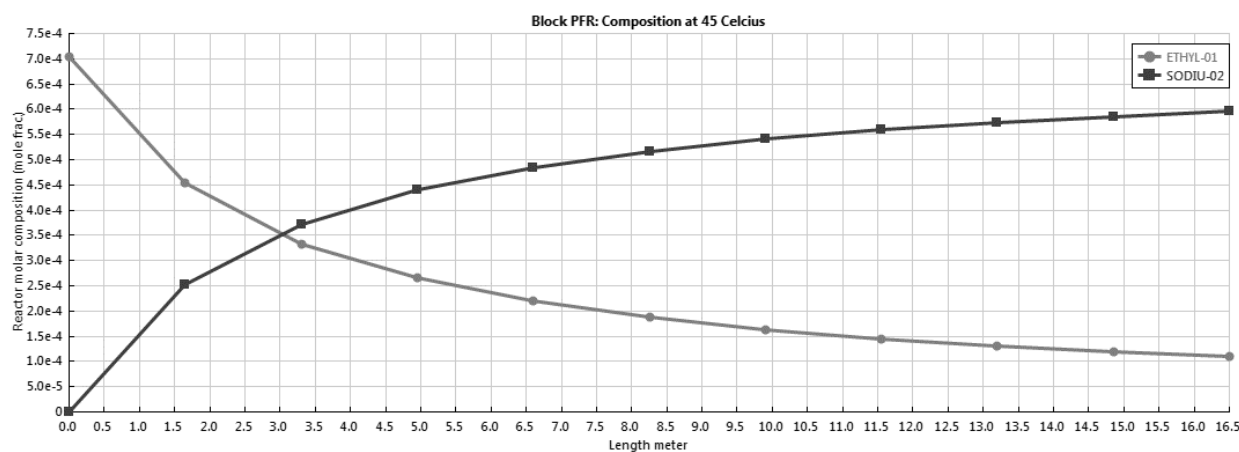


Fig. 11 Ethyl acetate mole fraction vs. PFR length at 45°C

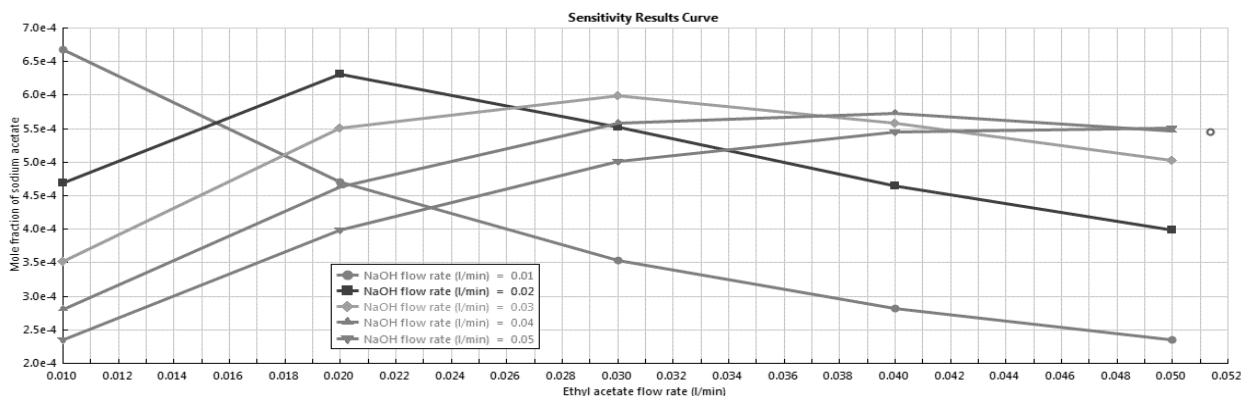


Fig. 12 Variation of ethyl acetate mole fraction with acetate and NaOH flow rates

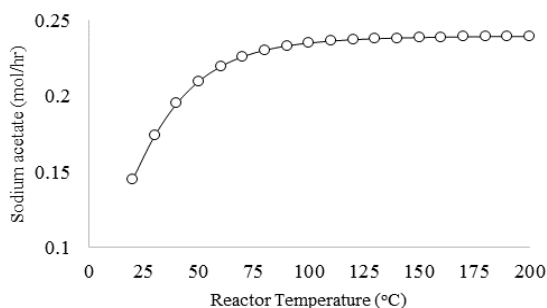


Fig. 13 Variation of sodium acetate flow rate with reactor temperature

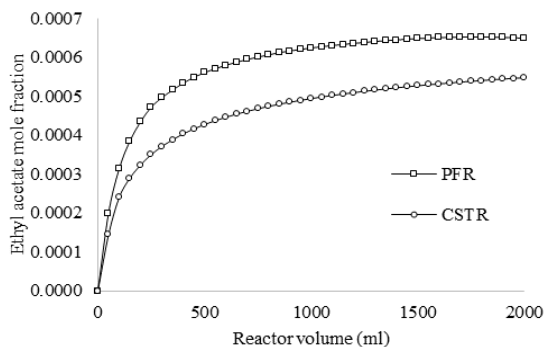


Fig. 14 Ethyl acetate mole fraction vs. reactor volume

#### D. Effect of Reactant Flow Rates on the Reaction

A sensitivity analysis was done in Aspen Plus model to investigate the effect of reactant flow rates towards the reaction.

Fig. 12 shows how the mole fractions of sodium acetate vary for different flow rates of NaOH and ethyl acetate. It shows that in each case the highest mole fraction of sodium acetate can be achieved when equal flow rates of NaOH and ethyl acetate are pumped to the plug flow reactor. According to the stoichiometry of (2) it is clear that equal amounts of reactants are needed to obtain the highest yield.

When the flow rate of ethyl acetate exceeds the flow rate of NaOH as there is no more NaOH left in the reactor to react with ethyl acetate, the excess ethyl acetate remains in the

product stream. Therefore though same amount of sodium acetate is produced by the reaction, mole fraction of sodium acetate is decreased because of the presence of unreacted ethyl acetate in the product stream.

#### E. Volume Comparison of CSTR and PFR

Volume required for a PFR is relatively less when compared to the volume required for a Continuous Stirred Tank Reactor (CSTR) to achieve the same conversion for a reaction while maintaining the other parameters as constant [15]. This theory is well applied for the saponification reaction of ethyl acetate with sodium hydroxide. During this study it has also proven with using Aspen simulation that the volume of CSTR to achieve the same conversion is about 3.5 times that of a PFR (Fig. 14). Therefore, it is economically feasible to use a PFR to carry out this saponification reaction in industrial scale even when producing with large quantities.

#### VI. CONCLUSION

In this work, a simulation model was developed for the reaction of saponification of ethyl acetate in the presence of sodium hydroxide inside a plug flow reactor using Aspen Plus. The model was validated by the experimental results obtained from EDIBON Plug Flow Reactor module. The experimental results showed a good fit to the Aspen Plus model.

Aspen Plus model proved that the rate of reaction increases as the reactor temperature and the length of the plug flow reactor increase. The highest yield of the reaction was obtained at a reactor temperature of 150°C.

These results improve the understanding of reaction kinetics with reference to the saponification reaction and would be very useful in the design of plug flow reactors.

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