

Estimation of Kinetic Parameter using Aspen Plus on The Transesterification of Palm Oil with Methanol to Produce Fatty Acid Methyl Ester

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ABSTRACT

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Fatty acid methyl ester (FAME) is a renewable energy, mainly produced by transesterification of methanol (MeOH) and triglyceride (TG) with the aid of catalyst. The aims of this study are to estimate the kinetic of FAME production via transesterification of TG using Aspen Plus® simulator V10.3. and investigate the influence of process variables, i.e. effect of methanol: triglyceride (MeOH:TG) molar ratio and reaction temperature towards FAME production and TG conversion via sensitivity analysis. The transesterification reaction was modeled in Aspen Plus® V10.3 using stoichiometric reactor (RSTOIC) and continuous stirred tank reactor (RCSTR). In kinetic study, the reaction rate constant was determined using design spec at various reaction temperature. The reaction rate constant, K values that is predicted from the comparison study were 0.0147 s^{-1} , 0.0661 s^{-1} and 0.4674 s^{-1} at reaction temperatures of 433.15 K, 453.15 K and 473.15 K respectively. Thus, the predicted activation energy (E) was 146.833 kJ/mol while pre-exponential factor (k) was 6.83×10^{15} . In sensitivity analysis, the result showed increased MeOH:TG ratio reduced the TG conversion and FAME production. On the other hand, increased temperature caused the FAME production to increase due to high conversion of TG.

Keywords: Kinetics; Aspen Plus; FAME; Heterogeneous catalyst; Transesterification.

1. INTRODUCTION

The scarcity of petroleum reserves and environmental concerns has led to countless endeavours to utilise renewable energy sources [1]. Production of biodiesel or known as fatty acid methyl ester (FAME) plays an important role as alternative for fossil fuels due to its sustainability and eco-friendly characteristics compared to diesel [2]. FAME can burn and release non-toxic clean gases, biodegradable, sulphur-free, low amount of hydrocarbon and particulate matter compared to the diesel fuel [3]. The energy content and physical properties of FAME are closer to petroleum diesel and can be used efficiently in conventional diesel engines without any modification [4]. The property of FAME includes density (0.88 kg/L), viscosity (7.5 mm²/s), cetane number (56), lower heating value (37.1 MJ/kg) and fuel equivalence (0.91) [5].

FAME is mainly produced from reaction of plant oil with methanol via transesterification with the aid of catalyst [6]. The acid or base catalysis reactions is selected in the process by considering the composition of free fatty acids (FFAs) of oil. Base catalysis process can produce

the high yield percentage of FAME with a high purity in a short time within 30 to 60 minutes better than acid catalysis reaction [7]. The transesterification of triglycerides (TG) with alcohol produces FAME and glycerol (G). The TG is found in vegetable oils and animal fats, more generally known as lipids. During transesterification reaction, the highly viscous TG are converted into long chain monoesters, causing the viscosity to become much lower and better combustion properties [8]. The TG is commonly reacted with methanol (MeOH) to produce FAME because MeOH is the shortest chain alcohol [9]. Heterogeneous catalyst is in different phase than reaction system, thus allowing it to be removed easily and prevents excessive washing steps that are commonly used to recover homogeneous catalyst. The spent heterogeneous catalyst can be repeatedly used for the subsequent process [10]. Some of the heterogeneous catalysts used in previous work include alkaline metal catalyst [11], mixed oxide catalyst [12] and zeolite catalyst [13].

In the chemical reaction, understanding the kinetic mechanism is vital in order to improve reaction process and design the reactor systems [14]. There have been several reported kinetic studies for the performance of solid catalyst loaded on the support in transesterification reaction [15-17]. The kinetic studies of the biodiesel process were usually performed by conducting experiment to obtain the value of rate constant, K . Consequently, the kinetic parameter that can affect the yield of FAME can be determined. By using another method, kinetic parameter can be estimated using simulator such as Aspen Plus software. The embedded models of thermodynamic and kinetic Power Law within Aspen facility in developing kinetic models are more suitable with the complex process than kinetic model predicted based on thermal and concentration parameter. Furthermore, Aspen Plus is supported by an effective method such as Wegstein and Newton, to estimate parameter value. Besides Aspen Plus, other platforms such as Aspen HYSYS, VMGsim and SuperPro Designer can be used to develop biodiesel reaction model. Anene and Giwa [18] had designed FAME production model in reactive distillation using Aspen Plus. On the other hand, Aspen Plus was also used in the study on the commercial-scale biodiesel production from algae [19].

Kinetic constant and kinetic parameter strongly depend on the process parameters, catalyst and feed stocks used in the process. Dhawane *et al.* [20] investigated the kinetic model catalysed by iron supported activated carbon using Power Law kinetic model. The kinetic constant obtained were $0.0372 \text{ mol} \cdot \text{L}^{-1} \cdot \text{min}^{-1}$ and $0.437 \text{ mol} \cdot \text{L}^{-1} \cdot \text{min}^{-1}$, for forward reaction and reverse reaction respectively. Esther Olubunmi *et al.* [21] investigated the kinetic study of FAME production from beef tallow using calcium oxide catalyst. The result showed the reaction followed pseudo first order reaction rate law, with the activation energy and pre-exponential factor of $82.845 \text{ kJ} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$ and $4.805 \times 10^{11} \text{ min}^{-1}$, respectively. The kinetic model is useful to further understand process behaviour and process optimization. The present work aims to estimate the kinetic model parameter using Aspen Plus[®]V10.3 as well as to use parametric study using sensitivity analysis. The design spec tools provided in Aspen Plus[®]V10.3 can manipulate the kinetic parameter to suit with experimental condition. The biodiesel production use in this work is based on the biodiesel process developed by [22].

2. METHODOLOGY

The kinetic model parameter estimation was performed by using build in model in Aspen Plus[®]V10.3 software. Several steps have been conducted which include computing mass balance of the chemical component via simulation of transesterification process using basic RSTOICH

reactor, estimating rate constant in RCSTR reactor, generating the kinetic data, processing validation and finally investigating parametric behaviour through sensitivity analysis. The flow chart of overall activities of this work is shown in Figure 1.

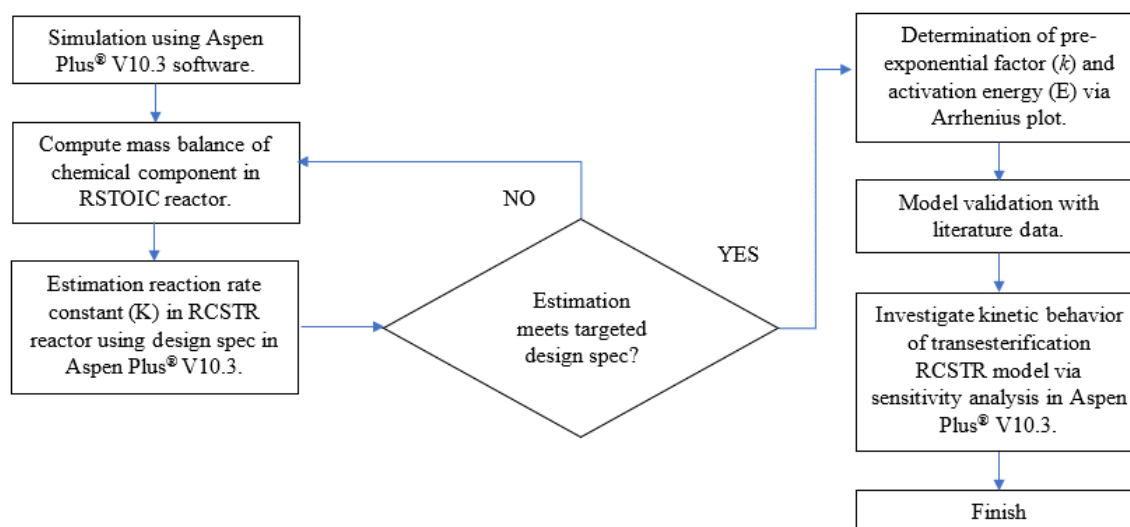


Figure 1: Overall flowchart of simulation activities.

2.1 Process Modelling and Simulation

The transesterification process of fatty acid methyl ester (FAME) from cooking palm oil (CPO) developed by [22] was simulated using conversion reactor (RSTOIC) and rigorous reactor (RCSTR) in Aspen Plus®V10.3 simulator with some modification and limitation as follows:

1. The actual methanol to triglyceride ratio and operating parameter such as temperature and pressure were according to process in experimental work by [22].
2. The TG conversion used in the simulation was assumed to derive from conversion of triolein to methyl oleate which represents the FAME.
3. The simulation in RSTOIC was based on TG conversion reflecting the effect of reaction time, methanol to oil ratio, reaction temperature and amount of catalyst. Thus, the conversion was normalized according to minimum and maximum of conversion compute from experimental work at same experimental design.
4. The mass balance of RSTOIC was computed based on the conversion of TG while RCSTR was temperature dependence in Power Law model.
5. The kinetic in RCSTR was estimated based on comparison method in two reactors (RSTOIC and RCSTR).
6. The reaction was assumed to have forward reaction K_1 only, with excess methanol in the reaction.

2.1.1 Simulation in RSTOIC

The main feedstock used in transesterification process was triglyceride (TG) which represents CPO and excess methanol (MeOH) to produce FAME and glycerol (G). The reaction involved during transesterification process is presented by Equation 1.



According to stoichiometric chemical Equation, 1 mole of TG in this case is triolein reacted with 3 moles of MeOH to produce 3 moles of FAME and 1 mole of G. Fresh excess of MeOH was mixed with TG in 12:1 mole ratio at different operating temperature ranging from 433K-473K. The simulation was conducted using RSTOIC, while thermodynamic properties were represented by NRTL model. The simulation was setup according to variables tabulated in Table 1 and the conversion values of TG was estimated according to the finding in experimental work by [22]. The estimated conversion was used to compute mass balance of FAME production in RSTOIC.

Table 1: Operating condition used in RSTOIC and RCSTR simulation of FAME production.

Variable	Value
TG feed flowrate	0.01 kmol/h
MeOH feed flowrate	0.12 kmol/h
Inlet stream pressure	15 bars
Inlet stream temperature	433.15-473.15 K
Reaction temperature (RCSTR)	433.15-473.15 K
Initial rate constant (K)	0.3 (s ⁻¹)

2.1.2 Simulation in RCSTR

RCSTR model is a more complex model than conversion reactor of RSTOIC. The simulation in RCSTR involved kinetic model to estimate the reaction products. The reaction in RCSTR was modeled by Power Law and the reaction rate was expressed by Arrhenius equation as shown in Equation 2.

$$r = k(T)^n \exp \left[-\frac{E}{RT} \right] \prod_{i=1}^N (C_i)^{\alpha_i} \quad (2)$$

The kinetic rate (r) is expressed in kmol/(s·m³), and the first term of equation is expressed by Arrhenius equation with k as pre-exponential factor, E as activation energy, R as gas constant (8.314 J/mol), and T as temperature in kelvin. While second term is concentration dependent term. The reaction was assumed to follow first order reaction.

Since the kinetic constant and kinetic parameters for kinetic model were unknown, thus the estimation of reaction products in RCSTR were determined by comparing with the mass balance obtained from RSTOIC. The detail explanation estimation of kinetic constant based on comparison method is further explained in Section 2.2. The arrangement of RSTOIC and RCSTR in Aspen Plus is shown in Figure 2.

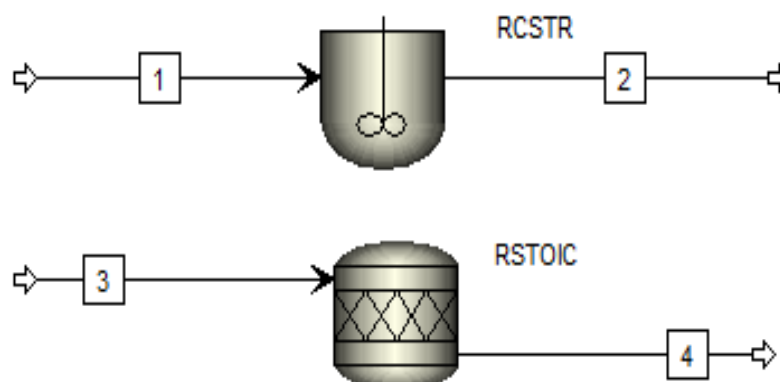


Figure 2: Flowsheeting of RCSTR and RSTOIC in Aspen Plus environment.

2.2 Estimation of Kinetic Constant

The kinetic constant and kinetic parameter, i.e., pre-exponential factor (k) and activation energy (E) in Equation 2 were determined by comparing the mass balance of TG at stream 2 and 4. The design spec tool in Aspen Plus® V10.3 is designed to target mole flowrate of TG at stream 2 (TGCOUT) to attain the TG mole flowrate at stream 4 (TGSOUT) when varying pre-exponential factor in Equation 2. The k of the reaction was manipulated to make rate RCSTR equivalent to rate RSTOIC as written in Fortran statements as in Equation 3 and 4. TGSIN and TGCIN are TG mole flowrate at stream 3 and stream 1 respectively.

$$\text{RSTOIC} = \text{TGSIN} - \text{TGSOUT} \quad (3)$$

$$\text{RCSTR} = \text{TGCIN} - \text{TGCOUT} \quad (4)$$

Thus, the kinetic constant can be estimated by manipulating k within 0-1 limits at temperature (433.15 K - 473.15 K). Then, the k and E values were calculated using estimated k obtained from simulation. Based on Arrhenius equation, $\ln K$ vs $1/T$ was plotted to determine the linear equation as Equation 5.

$$\ln K = \left[-\frac{E}{R} \right] \frac{1}{T} + \ln k \quad (5)$$

The calculated E and k were used as kinetic parameter in RCSTR for model validation. The TG conversion was validated with conversion used in RSTOIC.

2.3 Sensitivity analysis

The RCSTR was further used to analyse the effect of MeOH:TG ratio between 4:1-20:1 and the effect of reaction temperature (393.15 K – 483.15 K). The effect of MeOH:TG ratio and reaction temperature were analysed using sensitivity model analysis tool in Aspen Plus® V10.3 to observe the response of FAME and TG mole flowrate at stream 2 and TG conversion.

3. RESULTS AND DISCUSSION

3.1 Simulation and determination of reaction kinetic constant, K

The FAME production of transesterification process was simulated using RSTOIC and RCSTR to estimate rate constant of the reaction at different temperature. Table 2 tabulates the flowrate at stream 4 (TGSOUT) and stream 2 (TCGOUT). The similar mole flowrate was obtained from simulation using RCSTR and RSTOIC as indicated by TGCOUT and TGSOUT respectively. Thus, the kinetic constant is valid to be obtained using RCSTR. At the beginning of simulation with the combination RSTOIC and RCSTR, the initial k value of 0.3 was used in power law kinetic rate to iterate the most suitable k value to ensure the transesterification reaction that simulated in the RSTOIC can be obtained with RCSTR. Table 2 shows the value obtained for kinetic constant, K . During simulation in RCSTR, it was assumed no activation energy implied in the reaction, thus kinetic constant is equivalent to pre-exponential factor determined from simulation.

Table 2: Estimation reaction rate constant in RCSTR

Reaction temperature (Kelvin)	TGSOUT (kmol/h)	TGCOUT (kmol/h)	Rate constant (s^{-1})
433.15	0.002605	0.002605	0.0148
453.15	0.00069	0.0007	0.0661
473.15	0.0001	0.000106	0.4674

Based on the obtained K value at different temperature, value of K increased when the reaction temperature increased. It shows the kinetic rate is faster at higher temperature due to enhanced frequency of collisions among molecules at higher temperature. To estimate kinetic parameter for transesterification reaction, the K values and temperature were rearranged to plot $\ln K$ over $1/T$ for first order reaction and overall value of k and E were obtained. Figure 3 shows the linear Arrhenius plot with mean and interception values are -17661 and 36.46 respectively. Therefore, the calculated value for E and k were -146.8336 KJ/mol and $6.83 \times 10^{15} s^{-1}$ respectively.

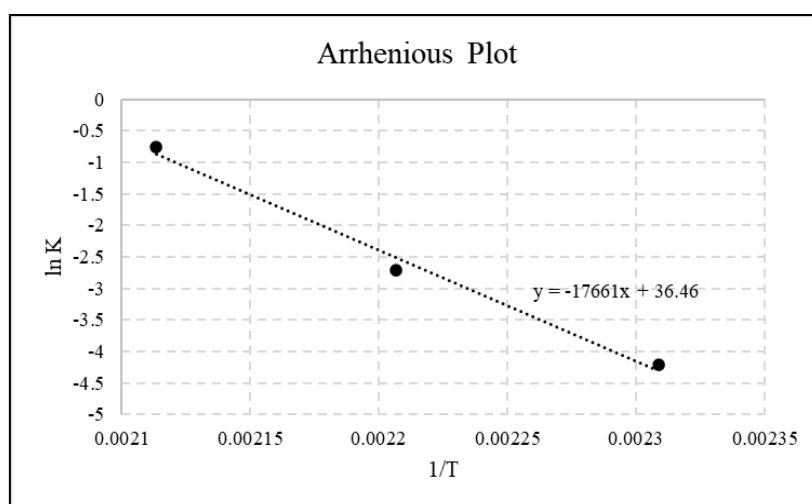


Figure 3: Linear Arrhenius plot

After determining overall kinetic parameter value, the transesterification reaction was simulated using RCSTR. The result obtained from the RCSTR model simulation was validated with literature data in terms of TG conversion from [22]. Table 3 shows the effect of temperature on TG conversion, and it shows the trend on TG conversion varied at different temperature. The

% error was calculated based on Equation 6 and it shows the acceptable % error were obtained except for % error at temperature 453.15 K which is 33.4 %.

The estimated kinetic parameter values in RCSTR predicted TG product slightly close to literature at temperature 433.15 K and 473.15 K. However, at temperature 453.15 K the prediction of TG production is lower than what was observed from past literature. In modelling, the prediction of reaction products is highly dependence to the selection of thermodynamic model and kinetic model assumptions made prior to simulation. Thus, the variation might be related to the assumptions when developing the model. Furthermore, during the experiment, there were other effects that could influence the reaction during FAME production such as mixing speed, and reaction time other than temperature and MeOH:TG molar ratio [23] which were not considered during simulation.

$$\% \text{ Error} = \left| \frac{\text{Conversion}_A - \text{Conversion}_B}{\text{Conversion}_A} \right| \times 100\% \quad (6)$$

where:

Conversion_A=TG conversion from literature data

Conversion_B=TG conversion from Aspen Plus

Table 3: Model validation on TG conversion simulated in RCSTR and literature data.

Reaction temperature (Kelvin)	TG conversion (RCSTR)	TG conversion (Literature)	% Error
433.15	0.7183	0.7395	2.9
453.15	0.6195	0.931	33.4
473.15	0.8832	0.99	10.8

3.2 Sensitivity Analysis

3.2.1 Effect of MeOH:TG molar ratio in RCSTR

The parametric study on feedstock ratio (MeOH:TG) and reaction temperature was conducted using sensitivity analysis in Aspen Plus®V10.3. The response of TG conversion and FAME production over varying MeOH:TG ratio from 4:1 to 20:1 at different temperature (393.15 K - 483.15 K), are shown in Figure 4a and 4b respectively. The molar flowrate of TG was 0.01 kmol/h while MeOH was varied according to the feed stock ratio.

The TG conversion and FAME production showed similar trend at different MeOH:TG ratio. When the MeOH:TG ratio was increased consistently, the result showed decreasing trend for TG conversion and FAME production. The MeOH:TG ratio can affect the conversion ability of TG where the higher molar ratio can lower down the yield of FAME [24]. Similar trend was observed by [25] and [26], the lower production of FAME was observed at higher MeOH:TG ratio. There is a possibility that the excessive use of MeOH could probably dilute the reactants [26]. The increased MeOH:TG ratio at different temperature also showed similar decreasing trend. The highest conversion (99.1%) and FAME produced (0.0297 kmol/h) were observed at 4:1 MeOH at temperature of 473.15 K. On the other hand, the lowest conversion (14.6%) and

lowest FAME production (0.0044 kmol/h) were achieved at 20:1 MeOH: TG ratio and temperature of 433.15 K.

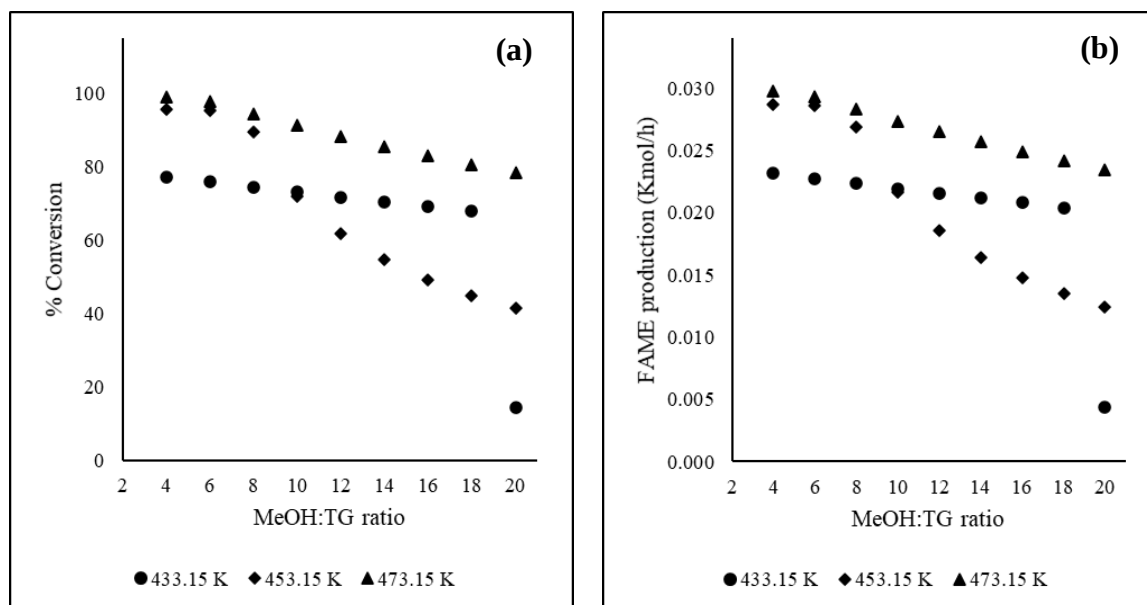


Figure 4: (a) TG Conversion and (b) FAME production at various MeOH: TG ratio

3.2.2 Effect of reaction temperature in RCSTR

Reaction temperature is a very important parameter that can influence the production of FAME. Figure 5a and 5b show the TG conversion and FAME production depend greatly on the temperature ranging from 393.15 K - 483.15 K. The significant increment was observed for both TG conversion and FAME production. However, the developed kinetic model showed that at 443.15 K, the conversion dropped to 44% as well as FAME only produced about 0.0132 kmol/h.

The higher conversion was noted at temperature more than 423.15 K which indicates more than 50% conversion. At lower temperature, the energy was not enough to shift the reaction forward, thus less TG was converted to FAME. However, the conversion at temperature 433.15 K was observed to be higher than 443.15 K and 453.15 K which were 71.8%, 44% and 61.9 % respectively. Moreover, two trends were observed at lower temperature (393.15 K – 433.15 K) and higher temperature (443.15 K – 483.15 K). From the literature, lower temperature may result in lower TG conversion, but higher temperature poses higher conversion which is greater than 90 %. Probowoputra *et al.* [27] investigated the effect of temperature on bio-oil yield from castor oil at different pressure using Aspen HYSIS simulator. From the findings, it was observed that the bio-oil yield was maximum at highest temperature (358 K). The FAME yield from castor oil was able to reach 93.8 % at pressure 707 kPa. Other researchers [28] evaluated on the temperature effect ranging from 313 K - 353 K on FAME production using transesterification reaction of *Chlorella vulgaris* with the presence of zeolite catalyst. The maximum biodiesel yield produced (36.8 %) at 333 K, while at 313 K, the yield was at lowest (13.1 %). Thus, it can be concluded that temperature has a strong effect towards the FAME yield. Higher temperature activates the catalyst activity, accelerates movement of molecules and the reaction will go even faster [28].

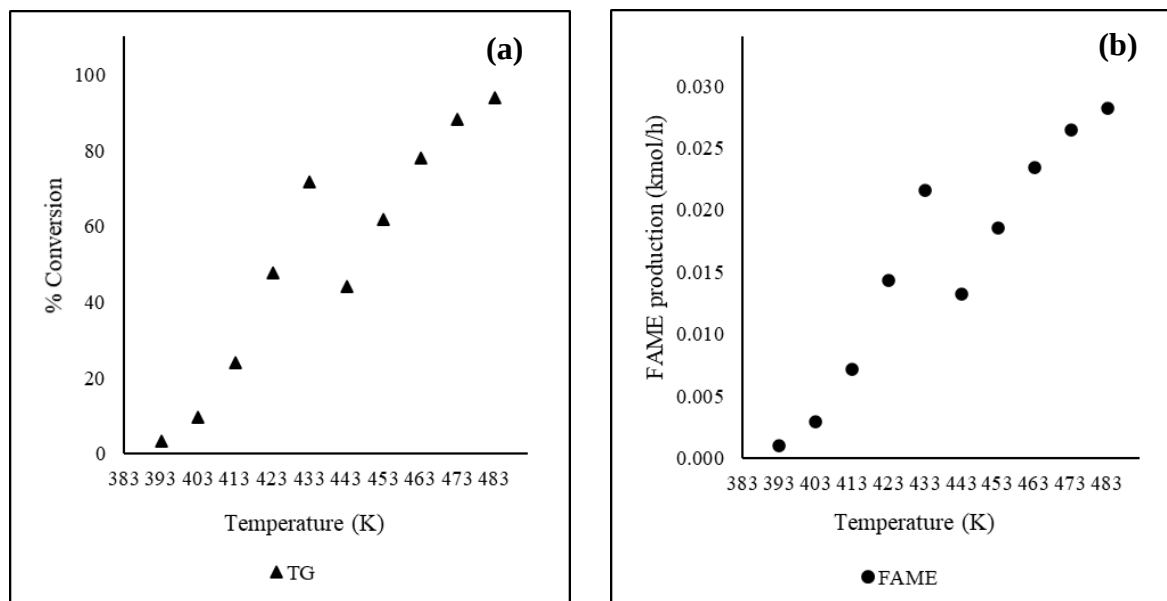


Figure 5: (a) TG conversion and (b) FAME production at various temperature

4. CONCLUSION

This study has determined the kinetic constant, K values that is predicted from the comparison method by using design spec analysis tool in Aspen Plus[®] V10.3, which are 0.0148 s^{-1} , 0.0661 s^{-1} and 0.4674 s^{-1} at reaction temperature of 433.15 K, 453.15 K and 473.15 K respectively. The activation energy (E) and pre-exponential factor (k) are equal to -146.8336 kJ/mol and $6.83 \times 10^{15} \text{ s}^{-1}$ respectively. The model validation agrees to literature data with % error is less than 11 % except for 33.4 % at 453.15 K. The MeOH:TG ratio and temperature are found to have major impact on TG conversion. Increased temperature may increase the FAME production, while increased MeOH:TG ratio may decrease the FAME production.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interest regarding the publication of this paper.

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