HETEROGENEOUS ESTERIFICATION OF FREE FATTY ACID TO BIODIESEL

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ABSTRACT

The potential of sulphated zirconia (SZ) as heterogeneous acid catalyst for esterification of free fatty acid (FFA) to biodiesel has been investigated. Experiments using oleic acid as a model compound preceded the esterification of palm oil free fatty acid (POFFA) mixtures. The relation of reaction time, SZ loading and methanol to oil molar ratio on biodiesel yield and conversion was investigated. Optimisation of esterification process via response surface methodology (RSM) for blended POFFA revealed maximum biodiesel yield (FAME) and conversion were 80.2% and 78.4%, respectively. The optimum conditions were operating reaction time = 87min, catalyst loading = 0.98wt% and molar ratio methanol to oil = 11.6:1. The fuel properties of biodiesel from POFFA and oleic acid blends were found to conform to the ASTM standard.

Keywords: *Biodiesel; Free Fatty Acid; Heterogeneous; Response Surface Methodology, Optimisation*

1.0 INTRODUCTION

Petrol and diesel contribute to environmental problems since the exhaust gas from these fuels emits green house gases (GHG) such as carbon monoxide (CO), nitrogen oxide (NOx) and particulate matter [1]. The GHG can be hazardous to human health besides it being a factor to global warning. According to Marchetti *et al*. [2], biodiesel can be defined as a fuel comprised of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats. Biodiesel is green fuel since it is biodegradable and non-toxic, and it significantly reduces toxic and other emissions when burned as fuel. Therefore, biodiesel might become a good alternative for sustainable environment development.

Vegetable oils such as palm, soybean, peanut and olive oil and animal fats such as beef tallow are attractive as raw material for biodiesel production. However, the prices of these oils and fats are relatively high and increase cost for biodiesel production. Therefore, alternative feedstock is being evaluated to identify less expensive materials that could serve as cheaper feedstock for biodiesel. Substantial efforts have been devoted to the development of non-edible oil and waste edible oil such as jatropha oil and waste cooking oil respectively as a biodiesel feedstock [3-8]. Meanwhile, the development of other feedstock such as POFFA is also of interest [9, 10]. POFFA is fatty acids residual based palm oil which is a by-product from distillation and recovery of desired fatty acids. The POFFA is attractive not only to further increase the economic viability of biodiesel, but it is also easier to obtain from the palm oil refineries.

Base-catalysed transesterification is the most common way to produce biodiesel since the reaction proceeds faster than the acid-catalysed reaction. In addition, the alkaline catalysts are less corrosive than acidic ones and therefore are more favourable in industrial processes [2]. The reaction directly converts the triglycerides to form their corresponding fatty acid methyl ester (FAME) in the presence methanol and catalyst. However, POFFA contains high amount of free fatty acid (FFA) (93 wt% or more) [10]. According to Marchetti *et al*. [2], if the amount of FFA in the feedstock exceeds 0.5%, direct esterification will inherently take place. Figure 1 illustrates the reaction path of esterification where **R** and **R'** denotes any hydrocarbon chain.

Esterification can be carried out in the presence of homogenous or heterogeneous catalyst. However, homogeneous catalyst is not recommended in the biodiesel production because of saponification and problems to separate the catalyst from the product[2, 11]. Heterogeneous catalyst seems to be better than homogeneous catalyst for obtaining high biodiesel yield.

Several researchers have studied the performance of zirconia based catalyst for biodiesel production. Jitputti *et al*. [12] studied ZrO_2, ZnO, SO_4^{2-}/SnO_2, SO_4^{2-}/ZrO_2 (SZ), KNO_3/KL zeolite and KNO_3/ZrO_2 for the transesterification of crude palm kernel oil (PKO) and crude coconut oil (CCO). They reported SZ solid acid catalyst exhibited the highest activity for both crude palm kernel oil and CCO transesterification compared to the other catalysts. Meanwhile, Satoshi *et al*. [13] reported three types of solid superacid catalyst, sulphated tin oxide (STO), tungstated zirconia-alumina (TZA) and sulphated zirconia-alumina (SZA) were able to contribute in esterification of n-octanoic fatty acid and transestrification of soybean oil. TZA was quite effective and gave high performance in biodiesel production. Over 90% conversions in both transesterification and esterification were reported. However, STO only showed high activities in the esterification due to its strong acidity compared to the others with 100% conversion.

Kiss *et al*. [14] has investigated the manufacturing of biodiesel via dodecanoic acid esterification using various super acid catalysts such as SZ, $SO_4^{2}–/TiO_2$, $SO_4^{2}–/SnO_4^{2}-$, H-ZSM5 and ion exchange resin. These catalysts were environmentally superior such as low corrosion, easy separation, and produced no acid waste problems. From the results, SZ was found to be a good catalyst for the catalytic biodiesel production. The catalyst possessed strong acid sites, highly selective with high thermal stability under the process conditions.

Therefore, SZ was a potential catalyst for biodiesel production from POFFA since SZ was less expensive and has an economical advantage due to productivity improvement resulting from its good catalytic activity. In addition, SZ has large pores which allow the diffusion of fatty acid molecules. Thus, it can produce high alkyl esters yield [15] and can be easily separated from the product [16]. In this paper, the potential of SZ as heterogeneous acid catalyst for esterification of POFFA to biodiesel is investigated. The relationship of reaction time, SZ loading and methanol to oil molar ratio on biodiesel yield and conversion of oleic acid was investigated using central composite design (CCD). The esterification of oleic acid as model compound was initially optimized via response surface methodology (RSM). Next, the esterification of POFFA and oleic acid mixture at the suggested optimum condition was studied.

$$\underset{\text{Free Fatty Acid}}{\text{R-COOH}} \quad + \quad \underset{\text{Alcohol}}{\text{R'OH}} \quad \longleftrightarrow \quad \underset{\text{Water}}{H_2O} \quad + \quad \underset{\text{Ester}}{\text{R-COOR'}}$$

Figure 1: Esterification reaction

2.0 EXPERIMENTAL

2.1 Material

Pure oleic acid was purchased from Reidel De Haen Company (U.S.A) while POFFA was obtained from Natural Oleochemical Sdn Bhd, Pasir Gudang, Johor, Malaysia. POFFA was received as dark brown liquid with mild odour. The components of POFFA were identified by GC-MS and tabulated in Table 1. Zirconia was purchased from Merck (Germany). Meanwhile, 0.5 N H2SO4, analytical grade methanol (98%) and n-hexane were purchased from Q-Rëc (New Zealand).

2.2 Catalyst Preparation

SZ, $SO_4^{2}-/ZrO_2$ catalyst was prepared via wet impregnation method. The zirconia was immersed in 0.5N H_2SO_4 solution and heated to 110ºC to evaporate the water. After the solution turned slurry, it was oven dried at 120°C overnight and calcined at 600°C for 1h.

2.3 Esterification Process

The esterification of model compound, oleic acid and POFFA were conducted in a 250 ml round bottom flask equipped with a reflux condenser and thermometer. The oleic acid, SZ and methanol were mixed at a desired amount. Then, the mixture was stirred at 200rpm and heated at methanol reflux temperature (65ºC). Next, the solution was allowed to cool to room temperature. The catalyst was separated from the product mixture using vacuum filtration at 5mmHg. The product was then poured into a separating funnel and left to settle for 24h to separate the excess methanol.

2.4 Analysis

The compositions of methyl ester was determined by using an Agilent Technologies 6890N Gas Chromatograph- Mass Spectroscopy (GC-MS) with inert mass selective detector 5975. The capillary column was Agilent 19091S-433 HP-5MS (30mm x 250µm x 0.25µm) and helium (2 ml/min) was used as carrier gas. The oven temperature was held at initial temperature 80°C for 0.5 min, and then ramped to a final temperature of 250°C for 5min at a rate of 10°C/min, with the total run time of 42 min. The injector and detector temperatures were 325 oC and 250°C, respectively. The acid value of the oil was determined by titration method using 0.1N potassium hydroxide solution. The optimal conditions of oleic acid and POFFA esterification was analysed by using STATISTICA 6.0 software.

Table 1: FFA profiles and physical properties of residual palm oil

Properties	
Free Fatty Acid Profile (%)	
Lauric acid 12:0	3.39
Stearic acid 18:0	12.68
Oleic acid 18:1	81.44
Others	2.49
Density (g/ml)	67.5
Viscosity (mm^2/s)	0.927

3.0 RESULTS AND DISCUSSION

3.1 Design of Experiments on Esterification Process using CCD

The effect and interaction of reaction time (**X_1**), catalyst loading (**X_2**) and molar ratio methanol to oil (**X_3**) over two observed responses; conversion (**Y_1**) and ester yield (**Y_2**) was analysed using response surface methodology (RSM). Meanwhile, the reaction temperature was maintained at methanol boiling point(65°C) since the methanol would evaporate at higher temperature. A central composite design (CCD) with full 2^3 factorial designs (three factors each at two levels, six star points and two center points) was employed. Table 2 lists the range and level coded of independent variables while the complete design matrix of CCD and results of ester yield and conversion are given in Table 3.

Table 2: Experimental ranges and levels of independent variables

Factors	Symbol	Range and Levels				
		– α (–2)	-1	0	+1	+ α (+2)
Reaction Time, min	X_1	30	45	60	75	90
Catalyst loading, wt%	X_2	0.6	0.8	1.0	1.2	1.4
Molar ratio methanol: oil	X_3	4:1	5:1	6:1	7:1	8:1

Note: *+ α: high star point level; +1: high factorial point level; 0: central point level; -1: low factorial point level; -α: low star point level.*

Table 3: Experiment matrix and experimental results

Manipulated variables			Responses	
X_1	X_2	X_3	Y_1	Y_2
Reaction time (min)	*Catalyst loading (wt%)*	*Molar ratio (alcohol:oil)*	*Conversion (%)*	*Ester yield (%)*
45(-1)	0.8(-1)	5:1(-1)	46.78	35.13
45(-1)	0.8(-1)	7:1(1)	51.74	48.06
45(-1)	1.2(1)	5:1(-1)	50.80	49.80
45(-1)	1.2(1)	7:1(1)	67.94	51.35
75(1)	0.8(-1)	5:1(-1)	46.23	47.27
75(1)	0.8(-1)	7:1(1)	56.62	52.400
75(1)	1.2(1)	5:1(-1)	52.21	43.31
75(1)	1.2(1)	7:1(0)	61.39	59.72
60(0)	1(0)	6:1(0)	63.18	46.99
34(-α)	1(0)	6:1(0)	56.8	40.87
86(+α)	1(0)	6:1(0)	69.83	46.98
60(0)	0.65(-α)	6:1(0)	45.92	38.26
60(0)	1.35(+α)	6:1(0)	56.05	50.49
60(0)	1(0)	4.24:1(-α)	49.48	41.62
60(0)	1(0)	7.76:1(+α)	64.48	55.32
60(0)	1(0)	6:1(0)	57.92	55.8

3.2 FFA Conversion

The empirical mathematical model equation of the FFA conversion is presented in Equation 2:

$$\begin{aligned} Y_1 = &-123.705 + 0.413\, X_1 + 178\, X_2 + 18.866\, X_3 \\ &- 0.002\, X_1^2 - 89.147\, X_2^2 - 1.639\, X_3^2 \\ &- 0.395\, X_1 X_2 - 0.021\, X_1 X_3 + 6.856\, X_2 X_3 \end{aligned} \quad (2)$$

Where, Y_1 is the predicted percentage of FFA conversion. The parity plot in Figure 2 indicates the determination coefficient (R^2) for conversion of methyl ester is 0.8566. The empirical model is adequate to explain most of the variability in the assay reading which should be at least 0.75 [17].

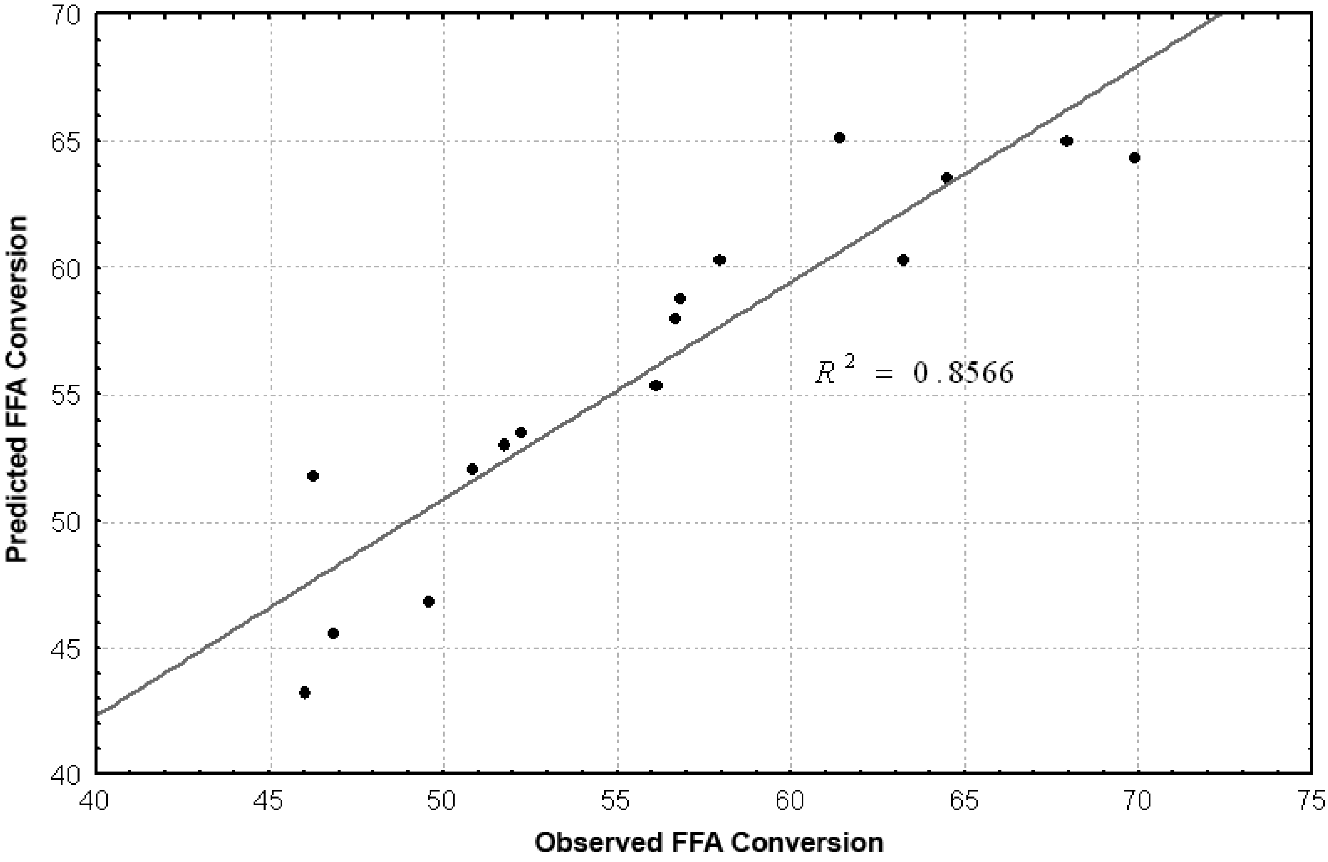


Figure 2: Parity plot for the observed and the predicted conversion

Table 4: Analysis of variance (ANOVA)

Sources	Sum of Squares (SS)	Degree of Freedom (d.f)	Mean Squares (MS)	F-value	Fα
FFA conversion Model					
Regression (SSR)	749.5847	9	83.2872	3.98	$F_{0.10} < 2.96$
Residual	125.4884	6	20.9147		
Total (SST)	875.0732	15			
ME yield model					
Regression (SSR)	535.6392	9	59.5154	2.71	$F_{0.25} < 1.77$
Error (SSE)	131.4633	6	21.9106		
Total (SST)	667.1025	15			

Meanwhile, in the analysis of variance (ANOVA), the ***F***-value of the model (***F***= 3.98) was greater than the tabulated ***F***-value (***F***0.10, 9, 6 = 2.96) (Table 4) indicating that the model was statistically significant at 90% confidence level. Figure 3 illustrates the Pareto chart and the corresponding ***p***-value of variable to check the significance of regression coefficients of the conversion model. As shown, the largest effect on conversion of methyl ester was linear term of methanol to oil molar ratio (X_3) which implied a higher ***t***-value (3.9501) and lowest ***p***-value (0.0075) at 99% significant level. Catalyst loading in linear(X_2) and quadratic (X_2^2) term could also be regarded as significant variables in FFA conversion at 97% and 95% significant level, respectively.

The three dimensional surface and contour plots of conversion over reaction time, catalyst loading and molar ratio are represented in Figure 4. These figures exhibit that the catalyst loading (SZ) and methanol to oil molar ratio have the most significant effect on the methyl ester conversion compared to reaction time. As illustrated in Figure 4a, the conversion of methyl ester started at 0.6wt% catalyst loading and reached maximum around 1.0wt% and became constant which may be due to catalyst deactivation. On the other hand, Figure 4b exhibits the conversion increased when excess methanol was applied in the reaction. Meanwhile, reaction time has a slight significant effect on the conversion (Figures 4a and 4b). The relationship between catalyst loading and molar ratio of methanol to oil is depicted in Figure 4c.

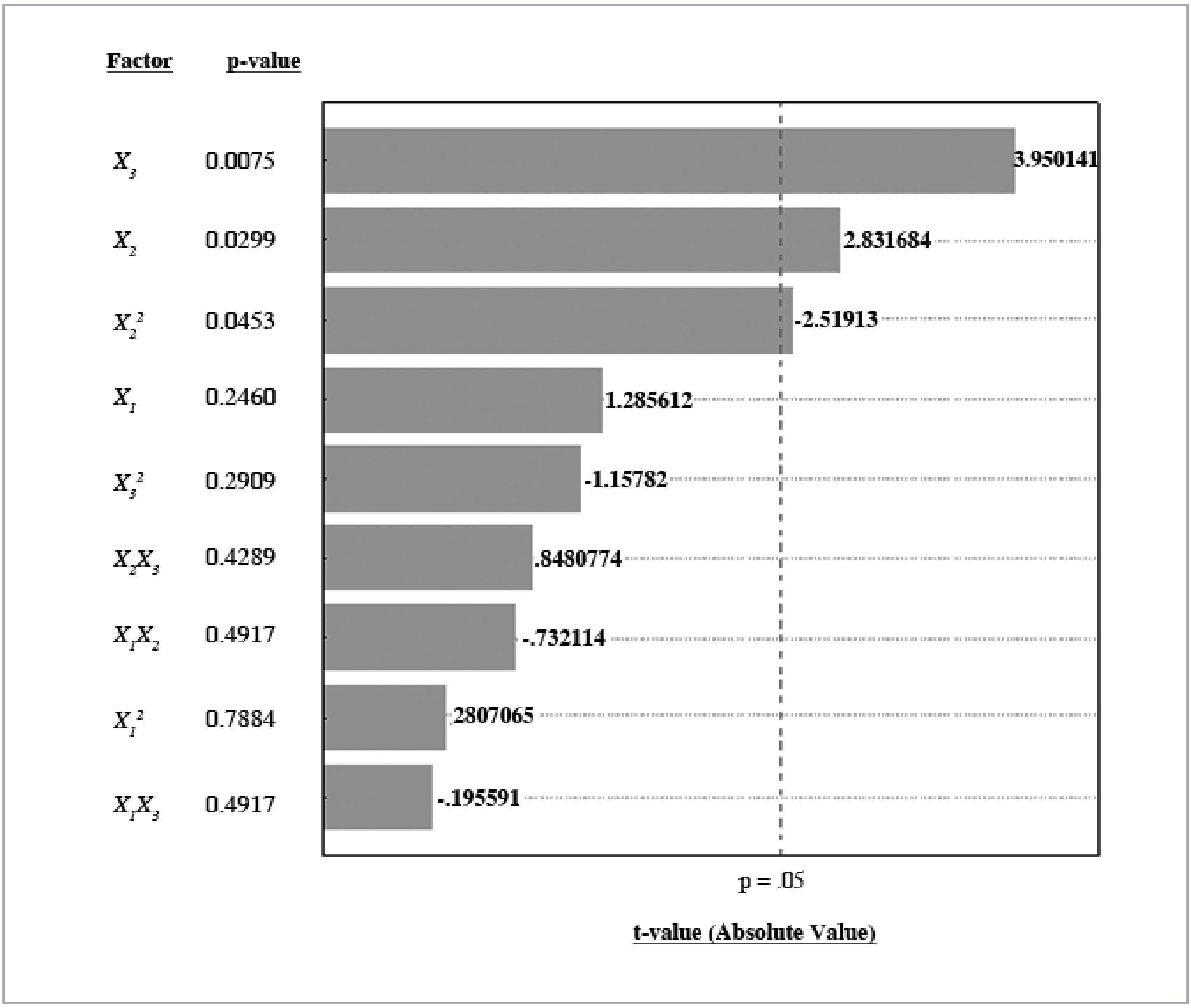
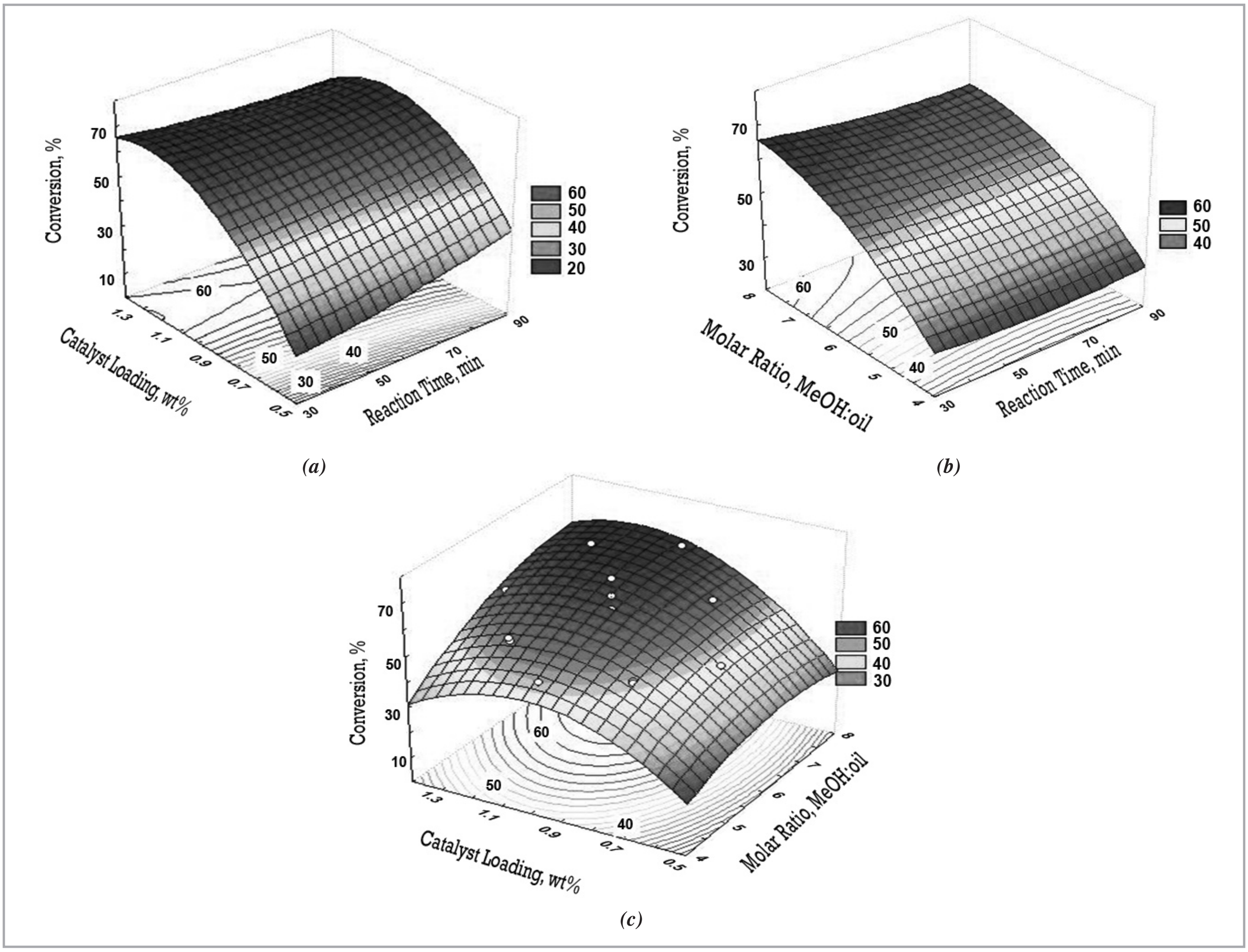


Figure 3 : Pareto chart and p-value of conversion



(a) *(b)*

(c)

Figure 4: The response surface plot of conversion as a function of (a) catalyst loading and reaction time at methanol to oil molar ratio = 8:1 (b) methanol to oil molar ratio and reaction time at catalyst loading = 1.4wt% (c) methanol to oil molar ratio and catalyst loading at reaction time = 60min

3.3 Ester Yield

The model equation for ester yield with coefficients in coded units of factors is given in Equation (3).

$$\begin{aligned} Y_2 = {} & -108.93 + 1.45\, X_1 + 143.65\, X_2 + 6.975\, X_3 \\ & - 0.009\, X_1^2 - 45.845\, X_2^2 - 0.518\, X_3^2 \\ & - 0.608\, X_1 X_2 + 0.059\, X_1 X_3 - 0.062\, X_2 X_3 \end{aligned} \tag{3}$$

where, Y_2 is the predicted percentage of ester yield.

The determination coefficient, $\mathbf{R}^2$ obtained was 0.8493 as shown in the parity plot in Figure 5. Therefore, it can be implied that the 84.93% of variability in the data fitted to the model. In the analysis of variance (ANOVA) (Table 4), ***F***-value for experimental (F =2.71) was lower compared to the tabulated ***F***-value ($F_{0.25,\,9,\,6}$ = 1.77). This indicated that the fitted model exhibited no lack of fit at the 75 % confidence level. Meanwhile, the Pareto chart and corresponding p-value in Figure 6 display the linear term of molar ratio methanol to oil (X_3) in the model have the largest effect on ester yield as the factor variable have larger ***t***-value (3.4093) and smaller ***p***-value (0.0143). This indicated that coefficients of the factor variable affect over ester yield at 99% significant level while other variables could be considered less significant to affect ester yield. Meanwhile, linear term of catalyst loading (X_2) could also affect ester yield at 95% significant level.

The empirical model is plotted as a three-dimensional surface representing the response (ester yield) as a function of two factors for experimental range considered (Figure 7). The surface response of operating reaction time and catalyst loading on ester yield is confined in the smallest ellipse as illustrated in Figure 7a. The ester yield increased when the reaction time and catalyst loading increased and decreased slightly after the optimum value is achieved. Meanwhile, Figure 7b exhibits high molar ratio of methanol to oil produced high ester yield. The same trend for methanol to oil molar ratio can be observed in Figure 7c where the interaction with catalyst loading denoted the largest effect on ester yield.

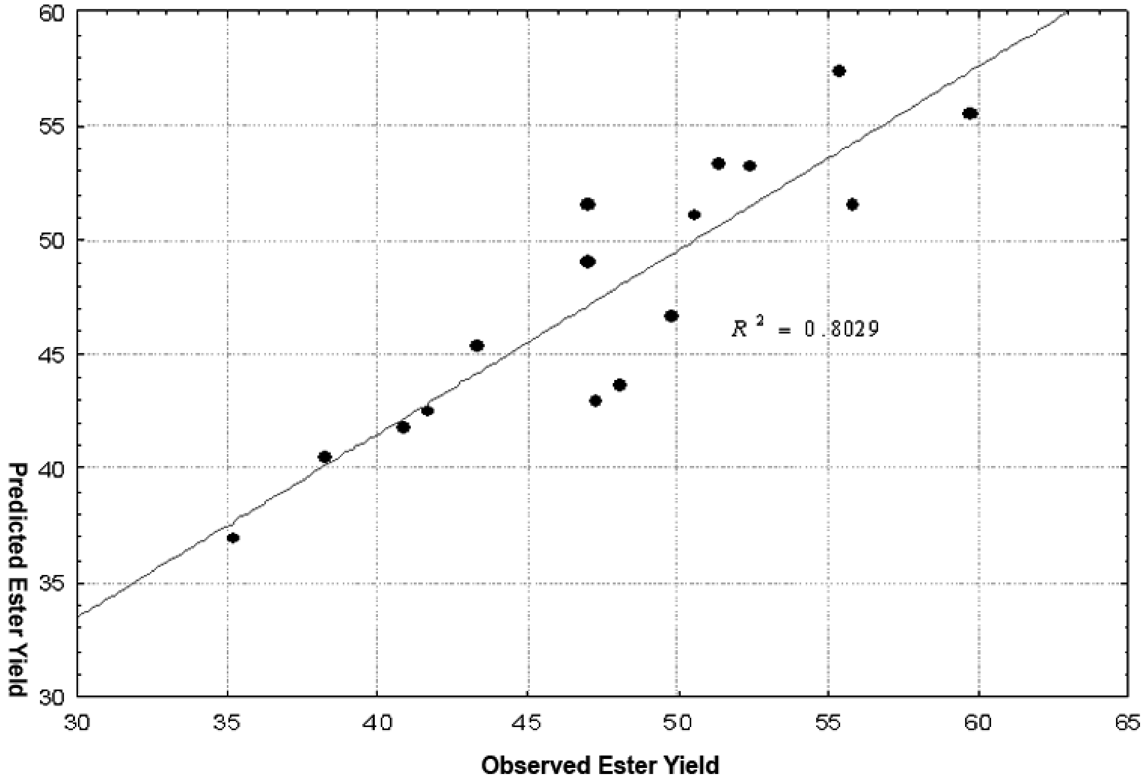


Figure 5: Parity plot for the observed and the predicted ester yield

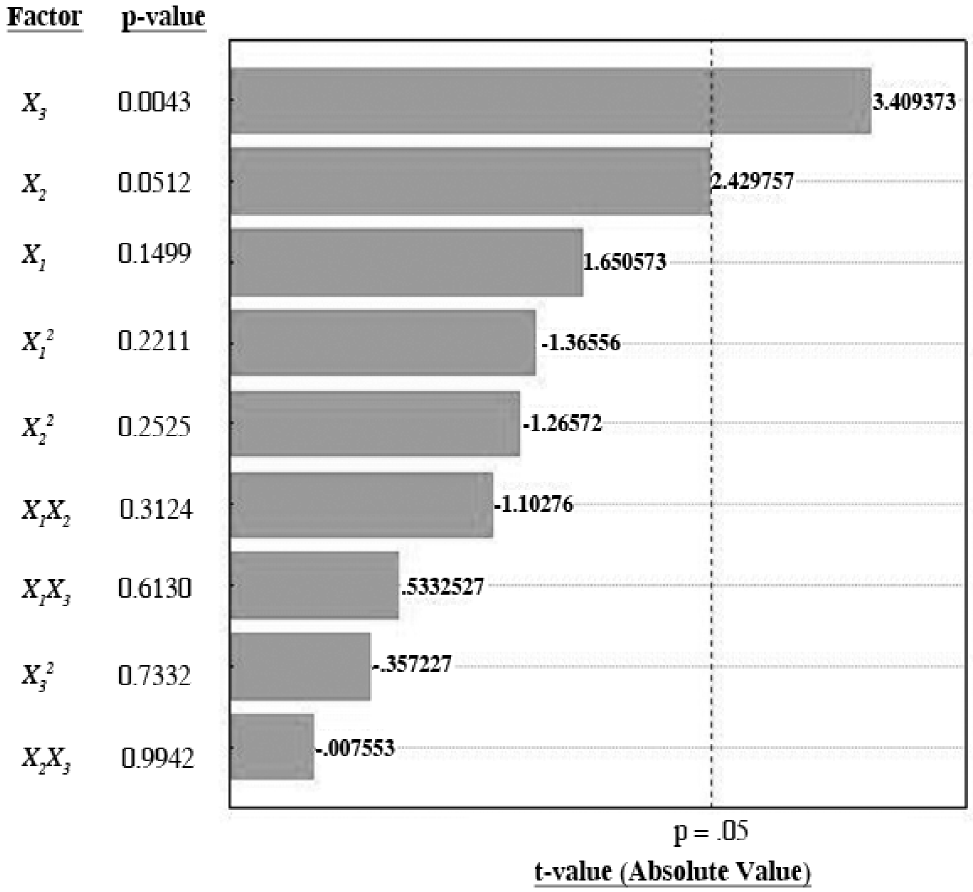


Figure 6: Pareto chart and p-value of ester yield

3.4 Optimisation Ester Yield of Esterification Oleic Acid

In this work, the model compound, oleic acid was optimized since the major component in POFFA is oleic acid. The response surface analysis using Statistica 6.0 software indicated that the predicted optimum ester yield of oleic acid esterification is 65.2% at operating reaction time = 87.3 minutes, 0.98wt% catalyst loading (SZ) and molar ratio methanol to oil = 11.6:1 as tabulated in Table 5. Although reaction time was found to have no statistically significant effect on either yields or conversion, in any of the terms (linear, interaction or quadratic) of the regression models at 95% confident level, the extended reaction time is considered since the ME yield model was accepted at 75% confident level for the ANOVA. Additional experiment was carried out to validate the optimisation result obtained by the response surface analysis. The experimental yield and differences between the predicted and observed values were reported as 64.60 % and 0.95%, (Table 5), respectively. The errors were considered small as the observed values are within the 5% level of significance. Therefore, the optimization condition of ester yield of oleic acid with RSM was at operating reaction time = 87.3 minutes, 0.98wt% catalyst loading (SZ) and molar ratio methanol to oil is 11.6:1.

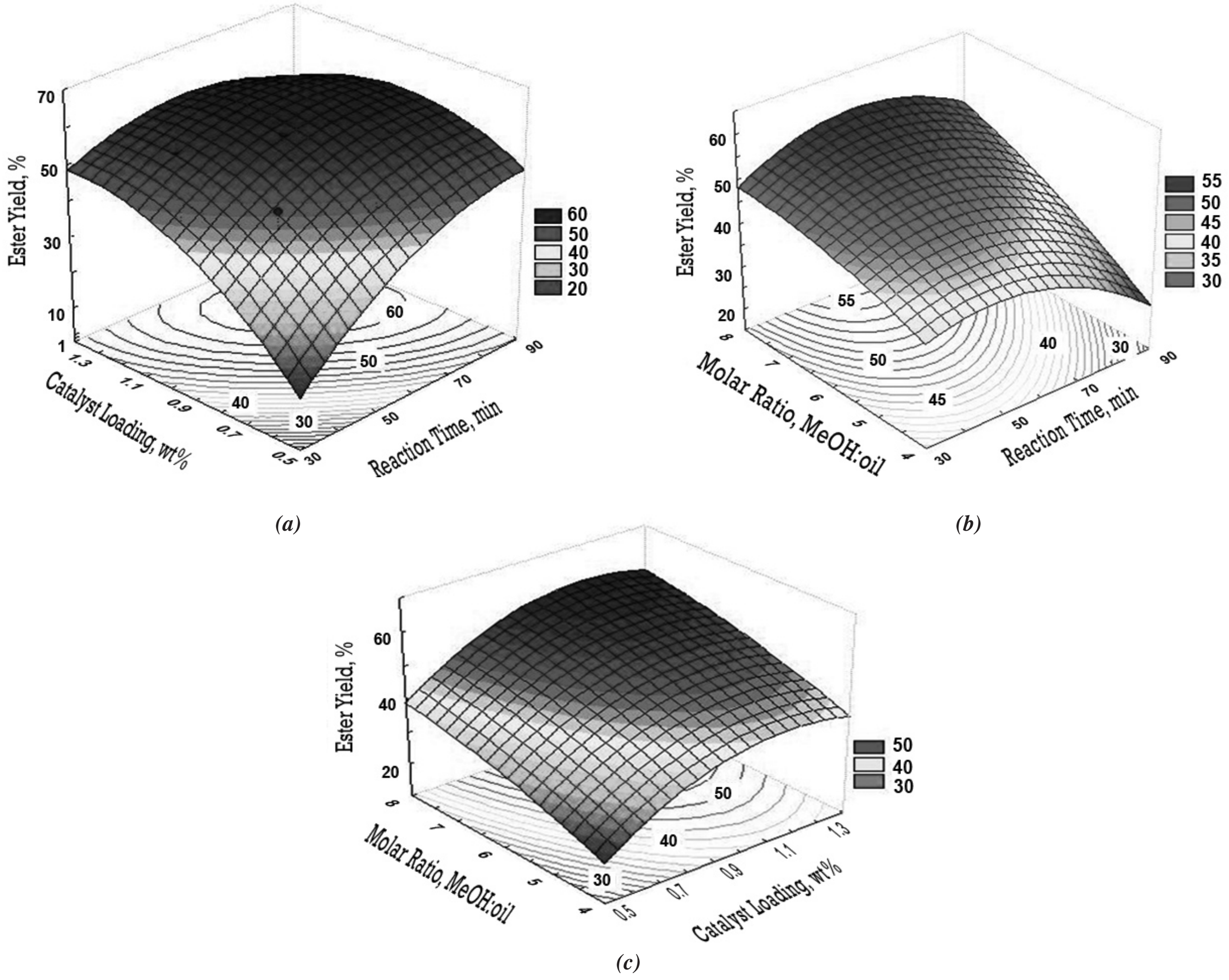


Figure 7: The response surface plot of ester yield as a function of (a)catalyst loading and reaction time at methanol to oil molar ratio =8:1(b) methanol to oil molar ratio and reaction time at catalyst loading = 1.4wt% (c) methanol to oil molar ratio and catalyst loading at reaction time = 60min

3.5 Heterogeneous Esterification of POFFA at Optimum Condition

Heterogeneous esterification of POFFA did not produce any FAME as the final product since the kinematic viscosity of POFFA was considerably high at 67.53 mm2s-1. As an alternative, the viscosity of POFFA has to be reduced to allow the reaction to occur at a lower temperature . Thus, POFFA was blended with 75% of free fatty oleic acid (kinematic viscosity = 21.1mm2s-1) in order to reduce the kinematic viscosity of POFFA. Table 6 presents the experimental results at optimum condition by using model compound (*i.e* oleic acid) and blend POFFA (25% POFFA + 75% oleic acid) as a sample.

The model compound gave a good yield as predicted in the response surface analysis indicating that the statistical approach is valuable in the optimisation of process. Therefore, the blended POFFA which demonstrated an excellent ester yield and conversion of 80.2% and 78.4%, respectively can be accepted as an alternative source to produce biodiesel.

In addition, Fig. 8 depicts the methyl ester yields for catalytic SZ and non catalytic esterification of oleic acid and blended POFFA at optimum condition. Esterification of oleic acid and blended POFFA using SZ as heterogeneous catalyst produced a high yield of methyl ester compared to non catalytic esterification. At optimum condition, 64.6% and 80.2% of methyl ester yield was produced from oleic acid and blended POFFA, respectively while non-catalytic reaction gave methyl ester yield of only 4.37% and 3.80%, respectively. This demonstrates that the SZ significantly enhanced the rate of esterification as reported in previous studies [12, 14].

3.6 Properties of Methyl Ester

The kinematic viscosity and density of oleic acid and blend POFFA as well as biodiesel produced from oleic acid and blend POFFA are summarised in Table 7. As for the standard, the value is given by ASTM D6751[18]. Density for oleic methyl ester (0.858) is slightly lower than the ASTM D6751 limit. However, it is still in good agreement with the standard because the biodiesel produced is lighter than the water. Meanwhile, biodiesel from blend POFFA was comparable with the limit.

Table 5: Critical values results for ester yield

Factor	**Observed Minimum**	**Critical Values**	**Observed Maximum**
Reaction time (min)	33.5	87.4	86.5
Catalyst loading (wt %)	0.65	0.98	1.35
Molar ratio (methanol: oil)	4.24:1	11.6:1	7.76:1
Predicted ester yield (%)		65.22	
Experimental ester yield (%)	64.60		
Error (%)	0.95		

Table 6: Experimental results at optimum conditions

Sample	**Ester Yield (%)**	**Conversion (%)**	**Observation (Colour)**
Free fatty oleic acid	64.6	100	Light yellow
POFFA 25% + Oleic acid 75%	80.2	78.4	Light Brown

Kinematic viscosity is an important property for fuel as it is an indication of the ability of a material to flow. The higher viscosity value will increase the tendency of the fuel to cause problem in the combustion engine and oil line. For biodiesel, the kinematic viscosity at 40ºC should be between 1.9 and 6.0mm^2/s [19]. From the specification analysis, both methyl ester from oleic acid and blended POFFA met the requirements with values being 3.51mm^2/s and 3.53mm^2/s, respectively. Hence, no modifications are required for handling the biodiesel from oleic acid and blend POFFA in the existing engine.

Table 7: Properties of sample and FAME

Properties	**Feedstocks**		**Biodiesel**			
	Oleic acid	***Blend POFFA***	***B100 (Malaysia)***	***ASTM D6751 (USA)***	***Oleic acid***	***Blended POFFA***
Density (20ºC) (g/ml)	0.868	0.927	0.878	0.87-0.9	0.858	0.878
Kinematic viscosity, 40ºC (mm^2/s)	21.2	25.6	4.4	1.9 - 6.0	3.51	3.53

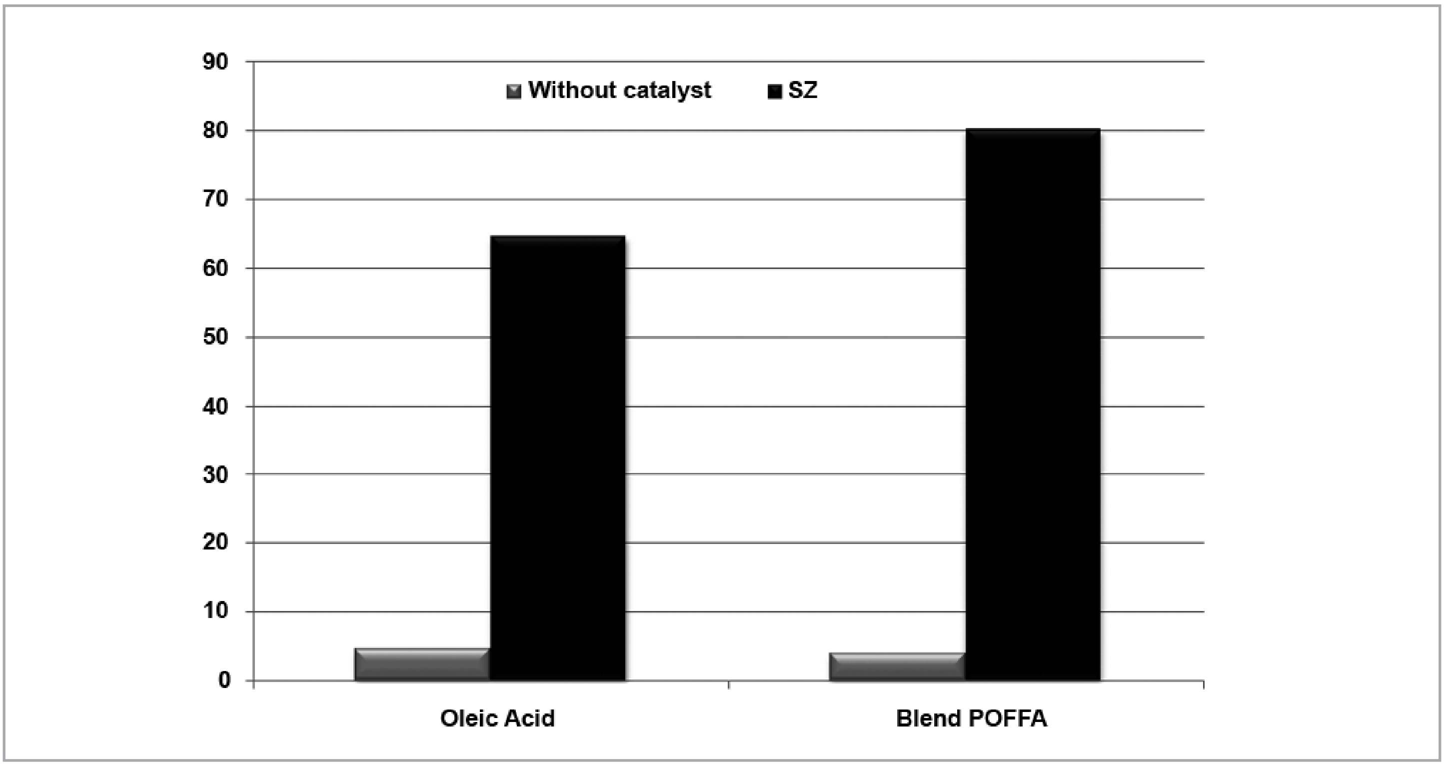


Figure 8: Methyl ester yields for catalytic and non catalytic esterification of oleic acid and blended POFFA at operating reaction time = 87.38 minutes, 0.98wt% catalyst loading (SZ) and methanol to oil molar ratio = 11.6:1

4.0 CONCLUSION

All the parameters such as reaction time, catalyst loading and methanol to oil molar ratio gave a significant effect on the heterogeneous esterification of oleic acid and blended POFFA. Longer reaction time increased the biodiesel production. Apart from that, excess alcohol led to complete reaction. The maximum biodiesel yield from blended residual palm oil was 80.2% at optimum conditions 87 min of reaction time, 0.98wt% of SZ catalyst and methanol to oil molar ratio = 11.6:1. The results indicated that blended POFFA has the potential to be used as a source for biodiesel production. From the cost perspective, biodiesel from blended POFFA is able to compete with the conventional petroleum diesel since POFFA is a by-product of palm oil refinery. In addition, the fuel properties of biodiesel from blended POFFA were within the standard requirement of ASTM D6751 for density and kinematic viscosity.

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