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Optimization Study of Biodiesel Production from Refined Palm Oil via Base-Catalyzed Transesterification

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ABSTRACT

With the implementation of the Philippine Biofuels Act of 2006, the country continues to search for new biodiesel feedstock and in improving on existing processes related to biodiesel production. In this study, contributions to these two fields of research were done. Palm oil is currently being considered at by the Philippine government as a potential biodiesel feedstock to augment coconut. This study aimed to optimize parameters for biodiesel production using refined palm oil as feedstock by varying the methanol-to-palm oil molar ratio (MeOH:PO) and the palm oil-to-sodium hydroxide molar ratio (PO:NaOH) with the aid of Response Surface Methodology (RSM) at constant temperature (30°C) and reaction time (60 min). The obtained optimum ranges are: 6.5-6.9:1 for MeOH:PO and 1:0.27-0.32 for PO:NaOH. After the optimum ranges for both ratios were determined, the effect of adding various amounts of FAME to the reaction system at the start of transesterification was studied in an attempt to break the immiscibility of methanol and oil in order to increase FAME yield. The increasing the initial amount of FAME added resulted to an increase in the amount of %FAME yield. In the experimental run where 50% by weight (wt%) of FAME was added, %FAME yield was 64%, which is more than double compared to that of the control (29%). These proved that biodiesel could be produced at milder reaction conditions and would require less energy input.

Key words: *biodiesel, methyl ester, oil palm, transesterification*

INTRODUCTION

With concerns regarding global warming and climate change, the search for (and researches on) alternatives for fossil fuel continues. One alternative that has achieved significant attention is biodiesel. The advantages of producing biodiesel include renewability, sustainability, ability to be locally-produced, having properties similar to petroleum diesel, and producing less emission of (certain) pollutants (*Yingying et al 2012; US EPA 2013; Liu et al. 2012*). However, there are still concerns about the production of biodiesel, including its competition with agricultural land and its energy balance (*Mendoza 2007*).

Biodiesel is technically defined as a mixture of mono-alkyl esters of various fatty acids. It is derived either from vegetable oil or animal fat (*National Biofuels Board USA 2013*). However, most commercial Biodiesel are derived from plant-based oils such as soybean, sunflower, canola, rapeseed, and coconut oil (*Yingying et al. 2012*). The process of converting the oil to biodiesel depends on the quality of the oil. For refined oils, transesterification is conducted with different parameters including methanol to oil ratio, catalyst loading, reaction temperature and reaction time (*Yingying et al. 2012; Liu et al. 2012; Al-Hamamre and Yamin 2014*).

Transesterification is a chemical reaction between a short-chain alcohol and vegetable oil (or animal fat) to produce mono-alkyl ester and glycerol (*Al-Hamamre and Yamin 2014*). Stoichiometrically, three moles of alcohol are reacted with one mole of trilyceride to produce three moles of alkyl ester and one mole of glycerol. Since transesterification is a reversible reaction, excess amounts of alcohol is added to drive the reaction towards the production of alkyl esters (*Schuchardt et al. 1998*).

The type of catalyst used is usually based on the quality of the oil, especially its free fatty acid (FFA) content. The higher the FFA content, the less suitable it is for base-catalyzed reaction because it will only favor saponification. Acid catalysts are best suited for oils with FFA concentration of equal to or more than 5% by weight. Disadvantages of using acid catalysts, as compared with base catalysts includes: slower reaction rates; requiring larger amount of methanol; and longer reaction time. Although base-catalyzed reactions have faster reaction rates and needs only mild reaction conditions, these, however, require higher quality of the raw material (i.e., lower FFA and water content) (*Thanh et al. 2013*).

Coconut is the only feedstock used for biodiesel production in the country. To meet the demand for the proposed increase in biodiesel blending as mandated by the Philippine Biofuels Law, the quest for alternative feedstock comes into view. The Philippine Coconut Authority is looking into the proliferation of oil palm to complement cocobiodiesel. Oil palm is attractive mainly because it is the highest oil-yielding plant reported (4.2 t ha^{-1} yr^{-1}) *(Sim et al. 2013*). The recently released palm oil road map is aiming to expand its plantation in the country from the current 56,641 to 300,000 ha. The lands considered for this expansion are said to be idle and unproductive (*Valencia 2014*).

Oils and fats are said to be immiscible in methanol (*Al-Hamamre and Yamin 2014*). Due to this, the reaction rate is slower at the beginning of the transesterification reaction. Efforts have been attempted to address this problem including increasing the temperature, intensifying the agitation, and using co-solvents.

This study aimed to optimize the production of methyl ester from palm oil by varying the methanol-to-oil ratio and catalyst-to-oil ratio at constant temperature (30^{o}C) and reaction time (60 min). Also, the effect of adding various amounts of FAME prior to transesterification on the percent FAME yield was also studied.

MATERIALS AND METHODS

Materials

Refined palm oil was obtained from a local supermarket in Los Baños, Laguna, Philippines. Methanol (99.8%, BAKER ANALYZED® A.C.S.) and sodium hydroxide pellets were used for the production of methyl ester while hydrochloric acid was used to stop the transesterification reaction. Ethanol (95%), potassium hydroxide, potassium acid phthalate, phenolphthalein, hexane (99% n-hexane), diethyl ether, formic acid (90%, BAKER ANALYZED®), iodine and silica gel for thin layer chromatography (TLC) were used in the analyses. Sodium chloride was used to prepare the brine solution for methyl ester washing. All of the chemicals used were analytical grade and commercially available.

Methods

All transesterification reactions were performed in 250-ml Erlenmeyer flasks in a water bath (*Libunao et.al. 2015*). Experimental runs and analyses were conducted at the Department of Chemical Engineering, College of Engineering and Agro-industrial Technology, University of the Philippines Los Baños.

The amount of free fatty acid (FFA) present in the refined palm oil was measured following the standard titrimetric method (AOAC Official Method 940.38) detailed by *Madamba* in *1987* (*Libunao et al. 2015*).

Base-catalyzed Transesterification

The required amount of refined palm oil was measured in an Erlenmeyer flask and was heated to the desired temperature. Sodium methoxide solution was prepared by dissolving sodium hydroxide in methanol. The solution was then added to the system with constant stirring at 600 rpm to ensure uniformity. Transesterification reaction were run at varying methanol-to-oil and NaOH-to-oil molar ratios at constant temperature and reaction time.

Washing and Purification

Subsequent to the transesterification reaction, the mixture was allowed to settle in a separatory funnel in order to separate biodiesel, crude oil and other excess reactants by gravity settling. The glycerol at the bottom layer was decanted while the biodiesel layer together with the unreacted methanol were washed several times with distilled water followed by brine washing. This was done to remove unwanted impurities such as soaps and any traces of the excess reactants. After washing, the biodiesel was then dried at 105°C until all the residual water has evaporated.

Addition of FAME

Transesterification reactions were carried out by adding various amounts of FAME (0, 10, 20, 30, 40, 50 % wt FAME/wt sol'n) simultaneously with the methoxide solution.

Response Surface Methodology (RSM)

The transesterification runs were optimized using response surface methodology (RSM) with the help of Design-Expert® Software 8.0 (trial version). The experiments were carried out in a fully randomized Central Composite Design (CCD) considering methanol-to-oil ratio and sodium hydroxide-to-oil ratio as the main factors. The methanol-to-oil ratio varied from 1.96:1 to 9.04:1 while the sodium hydroxide-to-oil ratio tested ranged from 0.01:1 to 0.35:1. Analysis of variance (ANOVA) and the coefficient of determination were used to validate the model generated.

Analysis

Thin-layer chromatography (TLC) was performed to determine the acyl glycerol profile of palm methyl ester. Biodiesel samples were spotted using a Hamilton microsyringe in a 20 x 20 cm silica gel plate. Chromatograms were developed in a chamber with hexane-diethyl ether-formic acid (80:20:2 ratio by volume) solvent-system and were visualized in an iodine chamber. The chromatograms were analyzed and quantified (in terms of percent weight) using Biosoft QuantiScan™.

The percent FAME yield was computed using:

$$\text{Percent Actual Yld} = \left(\frac{\text{Actual FAME yield (mL)}}{\text{Theortical FAME yield (mL)}}\right)x100 \qquad \text{(Eq .1)}$$

RESULTS AND DISCUSSION

The FFA content of the palm oil used was already reported in a prior work by *Libunao* (*2014*). To verify this small value for FFA content, the palm oil was analyzed for its glyceride profile using TLC. The FFA content of the palm oil was found to be negligible thus proceeding with base-catalyzed transesterification.

Optimization using RSM

The parameters chosen for optimization were the factors considered to be significant after a previous screening study conducted by *Libunao* (*2014*). Reaction temperature and reaction time were fixed at 30°C and 60 min, respectively. Experimental runs were randomized to minimize errors and unwanted variability as possible.

A second order response surface equation was generated from the results of the CCD experiment (**Table 1**). This equation was then used in predicting the optimal point/range for the methanol:PO and PO:NaOH molar ratios. The coded values are as follows: A - methanol value in the methanol-to-palm oil ratio (A:1), and B - sodium hydroxide in PO:NaOH (1:B).

$$\%\ \text{Yield} = 83.92 + 20.18\,A + 19.43\,B + 11.29\,AB - 10.33\,A^2 - 10.21\,B^2 - 25.18\,A^2B \qquad \text{(Eq. 2)}$$

The terms that have positive coefficients have positive effects on the yield while those that have negative coefficients have negative effects. So for Equation 2, the terms A, B, and AB have positive effects on the yield, but the terms A^2, B^2, and A^2B have negative effects. Thus, increasing the Methanol:PO ratio and the catalyst loading will not necessarily result to an increase in yield. This is because the interaction greatly affects the response. Although percent FAME yield may increase upon increasing the amount of methanol and catalyst loading independently, it is also significantly affected by their interaction.

Table 1. Design matrix and results of two-level-two-factors CCD for optimization of transesterification of palm oil with methanol using sodium hydroxide as catalyst.

A (Methanol: PO)	B (NaOH:PO)	FAME Yield (%)
3.1	0.06:1	63.8489 + 4.10
3.1	0.3:1	31.3692 + 6.35
8.1	0.3:1	87.4887 + 0.24
8.1	0.3:1	87.1435 + 0.24
8.1	0.06:1	69.4277 + 6.43
3.1	0.3:1	22.3868 + 6.35
8.1	0.06:1	78.5224 + 6.43
3.1	0.06:1	58.0559 + 4.10
5.5:1	0.18:1	84.1891 + 1.64
5.5:1	0.18:1	81.0501 + 1.64
5.5:1	0.18:1	84.7472 + 1.64
5.5:1	0.18:1	85.0475 + 1.64
5.5:1	0.18:1	84.5907 + 1.64
9.04:1	0.18:1	93.1697 + 1.34
1.96:1	0.18:1	30.7273 + 3.89
5.5:1	0.01:1	33.8091 + 3.88
9.04:1	0.18:1	95.0674 + 1.34
1.96:1	0.18:1	36.2248 + 3.89
5.5:1	0.35:1	93.6564 + 3.03
5.5:1	0.01:1	39.2988 + 3.88
5.5 : 1	0.35:1	89.3653 + 3.03

The interaction of methanol and sodium hydroxide (AB) will contribute positively as expected since in base-catalyzed transesterification, methanol and sodium hydroxide react with each other to form the alkoxide which will then attack the carbonyl group of the mono-, di- and triglyceride. Increasing the interaction between methanol and sodium hydroxide will result in the increase in amount of available alkoxide to react with the carbonyl groups producing more methyl ester.

Inter-molecular interactions between methanol with itself (A^2 interaction) and NaOH with itself (B^2 interaction), as indicated by their negative coefficients in Equation 2 (-10.33 and -10.21, accordingly), contribute negatively to the response. As mentioned by *Mamilla et.al.* (*2012*), increasing the amount of methanol (being the dispersed phase) will result to an increased interaction with itself causing the formation of bigger globules thus limiting the surface area for reaction between the oil and the alkoxide.

On the other hand, increasing the amount of catalyst have the same interaction effects for both methanol (AB and A^2B interactions) and itself (B^2 interaction). Generally, an increase in amount of NaOH would hasten the reaction and give better methyl ester yield. However, upon reaching the optimum catalyst concentration, excessive catalyst will drive the reaction towards saponification. NaOH will react with triglyceride to form soap and water thus reducing the biodiesel yield.

To determine the validity of the model generated, several tests were conducted. First of these tests was the Analysis of Variance (ANOVA) using the F- and p- tests. The p- and F- values of the model were checked to determine if the model derived is significant or not. Since the p-value for the model obtained is <0.0001 and its F-value (94.11) is higher than the theoretical F0.05(6,14) value (2.8477), this means that the model is significant (*Noshadi et al. 2012*).

Aside from checking the overall model, each term was also tested for their significance. The p-value should be less than 0.05 for the term to be significant. From the results of ANOVA, all of the terms in the model equation has a p-value of <0.0001 which means they are all significant. A p-value of <0.001 signifies that "there is only a 0.01% chance a model F-value this large is the product of noise in the experiment" (*Noshadi et al. 2012*).

Both the p-value for the Lack of Fit of 0.2630 (p-value>0.05 is not significant) and its F-value of 1.50 show that lack of fit is insignificant. This is highly desirable since a significant lack of fit may mean that there is an unaccounted for regressor-response relationship by the model. In short, the model fits to the experimental results (*Anderson and Whitcomb 2005*).

Also from doing ANOVA, the values of R-squared, Adjusted R-squared and Predicted R-squared were obtained (0.9881, 0.9830 and 0.9703, respectively). Since the R-squared and its adjusted values were near each other, it can be concluded that there is no non-significant term involved in the model. Aside from being in agreement with the raw R-squared, the value of Adj R^2 is high (near 1) indicating that the model is of high significance (*Noshadi et al. 2012*).

Since the model and its individual terms are statistically valid, the contour plot and response surface were then generated (**Figures 1** and **2**). From the said figures, it could be noted that as the amount of methanol is increased from 3 to 6.5:1, FAME yield also increased but a decrease in yield was observed when the amount of methanol was further increased from 7.1 to 8:1. This decline in yield, as seen in **Figure 2**(**a**), can be attributed to the dilution effect that high amount of methanol gives. A dilute solution means less interaction between the reactants and the catalyst. Moreover, high molar ratios of MeOH:PO shifts the reaction backwards due to the increased solubility of glycerine in the solution resulting to a lower methyl ester yield. On the other hand, increasing the amount of NaOH will not cause an outright decrease or increase in the yield. It also depends on the amount of methanol used (*Lakshimini et al. 2011*).

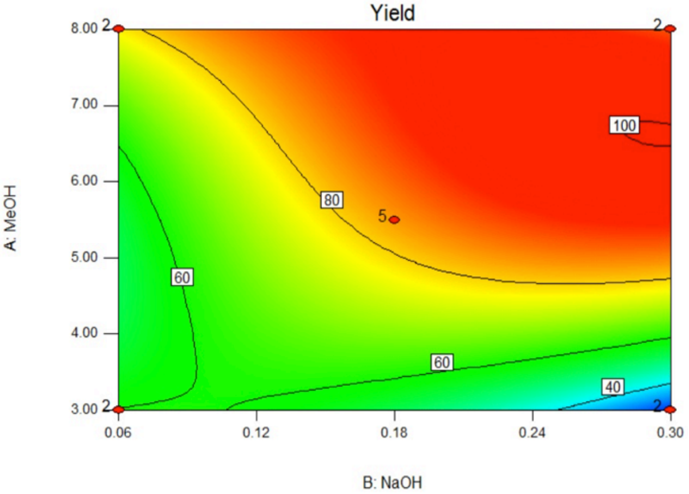


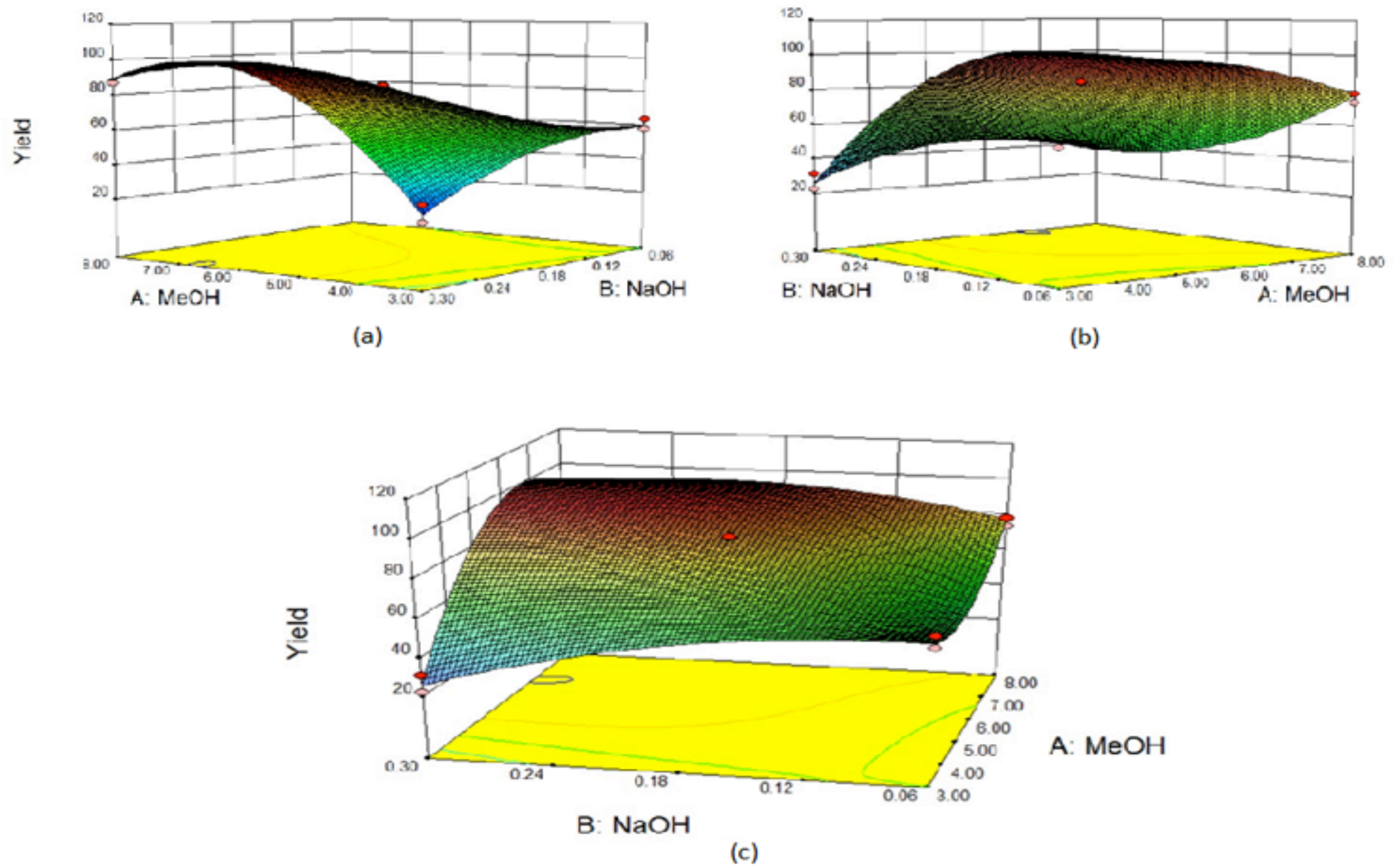
Figure 1. Contour plot of the regression model obtained for methanolysis of palm oil using sodium hydroxide as catalyst.

The theoretical optimum %FAME yield that could be obtained is indicated by the value 100 (**Figure 1**). The range of optimum methanol-to-palm oil molar ratio is from 6.5-6.9:1 and for palm oil-to-sodium hydroxide molar ratio, it is from 1:0.27-0.32. Experimental runs were conducted to confirm the optimum ranges obtained using RSM. The combination of parameters used were 6.75:1:0.295 MeOH:PO:NaOH molar ratio (30°C, 60min, refined palm oil). The average %FAME yield obtained was 97.35% which is lower than the values predicted by the model. The possible explanations for this include side reactions (i.e. saponification), errors (human, experimental and instrumental) and incomplete transesterification. Saponification is the chemical reaction between oil and NaOH to form soap and water (*De Boni and da Silva 2011; Zumdahl 1998*). As mentioned earlier, this side reaction competes with transesterification reaction thus reducing the biodiesel yield.

Compared with the best MeOH:PO:NaOH ratios and reaction times of the previous studies done by *Bataller* (*2007*), (8:1:0.2 and 1.75 hr) and *Patena* (*2009*), (9:1:0.3 and 2 hr) with *Jatropha*, the obtained MeOH:PO:NaOH ratio and reaction time are lower for this study. However, the reaction temperature used that resulted to more than 99% FAME content is higher than the room temperature used in Bataller's study and to the 30°C used in Patena's (*Bataller 2007; Patena 2009*).

Methyl Ester as Co-solvent

One economic and more environment-friendly way



(a) (b) (c)

Figure 2. The 3D surface of the actual and predicted experimental yields using the equation model developed for methanolysis of palm oil using sodium hydroxide as catalyst.

that this study suggests in addressing the immiscibility issue of methanol and palm oil is to add methyl ester itself at the beginning of the transesterification reaction. Methanol and triglycerides are both miscible with the methyl ester; hence, it could also serve as a co-solvent. The amount of FAME added were 0 (control), 10, 20, 30, 40 and 50%wt FAME (**Figure 3**). There were increasing trends in all the responses (%FAME content, amount of FAME recovered and %FAME yield). This is expected since methyl ester is known to break the immiscibility between the reactants. The highest %FAME yield obtained after 30 min of reaction (without agitation) was observed with the reaction system with 50wt% FAME at the start of the reaction (64%). This value is more than twice the %FAME yield of the control (29%). This means that using FAME as co-solvent effectively breaks the immiscibility and thus increasing the rate of reaction. The reaction mixture at the start of transesterification is a heterogeneous mixture before the presence of the initial FAME. The mixture then becomes pseudo-homogeneous upon agitation and at this point, controlled by both mass transfer and chemical reaction. As the amount of FAME increases, the ternary system ofmethanol-FAME-oil shifts from heterogeneous to a homogenous phase as the solubility of methanol in the oil-FAME phase increases (*Cheng et al. 2009; Zhou et al. 2006*). The addition of FAME at the start of the reaction, thus, speeds up the transition from heterogeneous phase of the reaction mixture to a homogenous one.

Several means were done to address this immiscibility issue; two of them were compared in the next part of thestudy. Agitation was contrasted with the addition of co-solvent (FAME) in terms of methyl ester production. The plots for 1 (with agitation only), 3 (with co-solvent only)

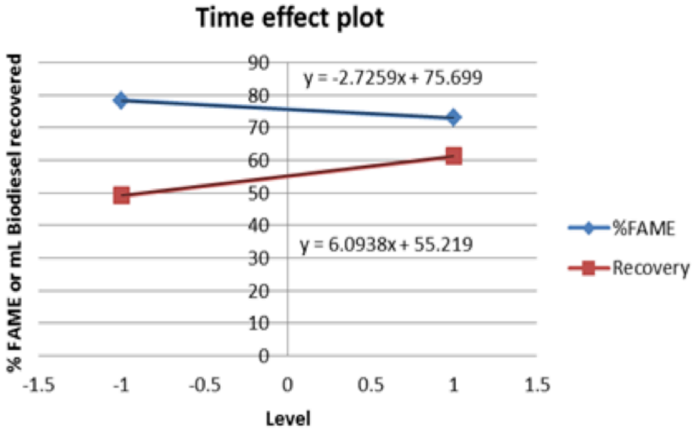


Figure 3. Main effect plot showing the influence of time (factor C) on FAME concentration and recovery of the Biodiesel layer.

and 4 (with both agitation and co-solvent) were similar with each other (**Figure 4**). Since the amount of methyl ester produced were almost the same, the addition of co-solvent could replace agitation in the transesterification reaction. Also, there was no significant difference in employing both agitation and the addition of FAME at the same instance in order to address the concern on immiscibility. In the same figure, it could also be observed that the addition of co-solvent attained the Philippine National Standard for the percent FAME content of the biodiesel (96.5%) faster compared with the set-up with agitation (1 and 3). This implies that addition of a co-solvent could accelerate the production of methyl ester in accordance with the Philippine National Standard. For plot 2 (without agitation, without FAME), it attained around 95% FAME content after an hour without any attempt of breaking the immiscibility. The possible explanation for this could be the fatty acid profile of palm oil. Palm oil have lauric, myristic and palmitic fatty acids, with higher amount of palmitic. These lower-numbered fatty acids are less immiscible to methanol than the higher ones (like linolenic) (*Zumdahl 1998*). This slight partial miscibility could be the reason for the production of FAME despite no agitation employed or co-solvent added.

CONCLUSION AND RECOMMENDATIONS

The results of the study indicated that increasing the methanol content and catalyst loading will only increase the %FAME yield at a certain level only.. These two major factors, together with their interaction with one another, dictate whether the % FAME yield will increase or decrease. The optimum methanol-to-palm oil molar ratio was determined to be 6.5-6.9:1 while for palm oil-to-sodium hydroxide molar ratio, it was 1:0.27-0.32. Using the ratio 6.75:1:0.295 (MeOH:PO:NaOH) resulted to 97.35%FAME yield. The obtained yield is lower than the predicted values by the model possibly because of side reactions, errors and incomplete transesterification reaction. Aside from determining the optimum MeOH:PO:NaOH ratio, this study also conducted experimental runs using methyl ester as co-solvent. Various amounts of FAME were added at the start of transesterification (6.75:1:0.295 MeOH:PO:NaOH) to determine the effect on biodiesel yield. Adding 50% by weight FAME initially to the transesterification reaction could double the amount of FAME produced (64%) compared to the reaction system with no additional FAME (29%). With these results, the need to agitate the reaction mixture in transesteriification could be eliminated with the addition of FAME as co-solvent. It could also shorten reaction time needed to achieve the Philippine National Standard for FAME content of biodiesel.

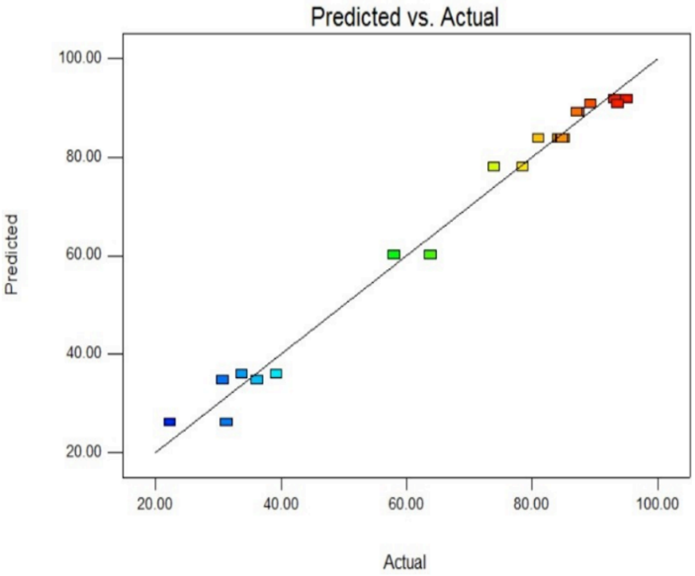


Figure 6. Plot of predicted values versus the actual values of the methanolysis of palm oil.

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