Optimization of Biodiesel Production from *Ricinus Communis* (Castor Seed) Oil Using Refluxed Calcined Snail Shell as Catalyst

Muhammad, C.[1], *Jibrin, M.S.[1], Mukhtar, M.[1], Baki, A.S.[2], Sani, U.[3],

[1]Department of Pure and Applied Chemistry, Usmanu Danfodiyo University, P.M.B. 2346, Sokoto, Nigeria

[2]Department of Microbiology, Usmanu Danfodiyo University, P.M.B. 2346, Sokoto, Nigeria

[3]Department of pure and Industrial chemistry, Bayero University, Kano, Nigeria

***Email: muhammadsabiu44@gmail.com**

***Phone Number: 2348032292415, 2348081134025**

ABSTRACT

Depletion of the fossil fuel as well as it environmental problems have attracted the attention of researchers recently, in the process to find the sustainable alternatives. Biodiesel emerges as one of the most energy-efficient and environmentally friendly options in recent times to fulfill the future energy needs. In the current research, the optimization of biodiesel production from castor seed oil using refluxed calcined snail shell was carried out and effect of transesterification variables on biodiesel yield and quality were investigated. The optimization of the biodiesel production was designed using Taguchi array design based on four transesterification variables (Temperature, time, catalyst load and oil to methanol ratio) in four levels. The biodiesel yields obtained ranged 88.02 – 99.68 %. The model shows the optimum yield of 99.68% at optimum condition of 1:6 oil to methanol ratio, 60 °C, 2 hours and 3 w% catalyst load. The response surface regression show that reaction time, catalyst load reaction time*reaction time and reaction temperature*catalyst load significantly affect the biodiesel yield. While, reaction temperature, oil to methanol ratio, reaction temperature*reaction time, reaction temperature*methanol to oil ratio and catalyst load*methanol to oil ratio were found to be statistically insignificant.

Keywords: Optimization refluxed calcined snail shell, biodiesel, transesterification, castor oil.

1.0 INTRODUCTION

Biodiesel emerges as one of the most energy-efficient and environmentally friendly options in recent times to fulfill the future energy needs (Nakarmi and Joshi, 2014; Owolabi *et al.*, 2012). Biodiesel is a renewable diesel substitute that can be obtained by combining chemically any natural oil or fat with alcohol. During the last 15 years, biodiesel has progressed from the research stage to a large scale production in many developing countries (Zhang *et al.,* 2003). In Nigerian context, non-edible oils are emerging as a preferred feedstock and several field trials have also been made for the production of biodiesel (Okechukwu *et al.,* 2015; Owolabi *et al.*, 2012).

Castor oil had its own advantages as one of the promising sources of feedstock for biodiesel production despite having high viscosity compared to other vegetable oils (Ismail *et al.,* 2016). Castor oil does not contain sulfur; it has greater cetane number which indicates a better quality of ignition and more oxygen content which promote complete combustion (Conceicao *et al.,* 2007). Castor oil has improved lubricity over other oils with similar carbon chain length fatty acids. The hydroxylated fatty acids of ricinoleic acid in castor oil impart it better performance as lubricity enhancer than other common vegetable oil esters (Ramezani *et al.*, 2010).

Among the heterogeneous catalysts, calcium oxide has become researchers' favorite because it is cheap and abundantly available in nature as limestone and also from seashells in the form of calcium carbonate, $CaCO_3$. Calcium oxide can be reused up to 3 times in transesterification reaction which makes it an economic catalyst. As the calcium oxide was obtained from the natural source, it is environmentally friendly and causes no harm to the ecosystem. Thus, calcium oxide is suitable to be used in large-scale production of biodiesel for commercial purpose as it needs no post-treatment prior to its disposal to the environment (Kouzu *et al.*, 2008).

Biodiesel production from castor oil has been studied by several researchers. Response surface methodology was used to optimize the transesterification reaction in traditional conditions. The results showed that reaction temperature affected the reaction slightly, however catalyst amount affected significantly (Da Silva *et al.*, 2009). In this research, the optimization of biodiesel production from castor seed oil using refluxed calcined snail shell was carried out to investigate the effect of transesterification variables i.e. oil to methanol ratio, reaction time, reaction temperature and catalyst load on biodiesel yield.

2.0 MATERIAL AND METHOD

2.1Preproduction Process

The castor seed was procured from Yandodo, Kano State, Nigeria. The seed was sundried to reduce the moisture content. The castor seed was de-shelled. The de-shelled seed which was white in color was oven dried at 90 °C for 45 minutes. The dried seeds were grounded using mortar and pestle and weighed. Extraction of castor oil was carried out by soxhlet extraction method as reported by Edison, *et al.,* 2012. The extracted oil had undergone degumming and neutralization as a pretreatment in order to upgrade it physicochemical properties for efficiency in transesterification reaction (Nakarmi and Joshi, 2014).

The calcium oxide catalyst that was used in this study was synthesized from snail shell through the hydrothermal method as reported by Ismail et al., 2016.

2.2 Optimization

Parameters considered for optimization of transesterification include oil to methanol ratio, catalyst load, reaction temperature and reaction time as in Table 1. The experiments were designed using TAGUCHI design on MINITAB 17 Statistical Software. The effects of four factors i.e. oil to methanol ratio, reaction time, reaction temperature and catalyst load at four different levels on biodiesel yield were investigated. The optimum parameters were determined for the highest yield of the biodiesel.

Table 1: Transesterification factors and their levels

Factor	Levels
Oil to methanol ratio	1:6, 1:8, 1:12, and 1:18
Reaction temperature (°C)	50, 55, 60 and 70
Reaction time (Hour(s))	1, 1.5, 2, 2.5 and 2.5
Catalyst load (w/w%)	0.5, 1, 3 and 7

2.3 Transesterification of Castor Seed Oil

The transesterification was carried out in 500 cm^3 two necks round bottom flask as reactor equipped with a condenser, thermometer, and hotplate magnetic stirrer. The 45 g of refined castor oil was initially charged into the reactor, and then preheated to 50 °C. In order to maintain the catalytic activity, the solution of 0.225 g (0.5 wt%) of CaO (refluxed calcined snail shell) in 9.169 g (1:6 oil to methanol ratio) methanol was freshly prepared so that prolonged contact with the air would not diminish the effectiveness of the catalyst through interaction with moisture and carbon dioxide. The solution was preheated to 50 °C in the water bath and then added to the preheated oil after which the reaction was timed at 1 hour and the agitation was kept at 300 rpm. After the reaction time, the mixture was allowed to settle under gravity for 24 hours in the separating funnel. Two layers were formed: the upper layer consisted of methyl ester, methanol traces, residual catalyst, and other impurities, whereas the lower layer consisted of glycerol, excess methanol, catalyst, and other impurities. After separation from the glycerin layer, the methyl ester layer was centrifuged and then purified by washing with hot distilled water at 60°C until the washing water had a pH value similar to that of distilled water (Nakarmi and Joshi, 2014). The hot distilled water-to-crude methyl ester ratio was 1:1. To prevent the possibility of losing the methyl ester due to emulsion formation, the washing was done gently. After then the weight of biodiesel was taken and yield was determined. The same method was adopted for the transesterification of each run based on the design of the experiment. The percentage yield of biodiesel was calculated by using equation 1:

$$\text{Biodiesel yield} = \frac{weight\ of\ biodiesel}{weight\ of\ oil}\, x\ 100 \quad (1)$$

3.0 RESULTS AND DISCUSSION

The optimization of biodiesel production from castor seed oil using refluxed calcined snail shell was carried out based on Taguchi orthogonal array design and yield obtained is presented in Table 2. The specific gravity and kinematic viscosity of each run were studied and presented in Table 3.

Table 2: Taguchi Orthogonal Array Design and the yield obtained

Oil To Methanol Ratio	Reaction Temperature (°C)	Reaction Time (H)	Catalyst Load (wt. %)	Yield (%)
1:6	50	1	0.5	93.77
1:6	55	1.3	1	98.26
1:6	60	2	3	99.68
1:6	70	2.3	7	93.51
1:8	50	1.3	3	99.22
1:8	55	1	7	88.20
1:8	60	2.3	0.5	88.02
1:8	70	2	1	96.87
1:12	50	2	7	90.72
1:12	55	2.3	3	86.99
1:12	60	1	1	91.06
1:12	70	1.3	0.5	94.77
1:18	50	2.3	1	95.10
1:18	55	2	0.5	98.46
1:18	60	1.3	7	97.23
1:18	70	1	3	97.53

Table 3: Taguchi Orthogonal Array Design and properties of biodiesel in each run

Oil To Methanol Ratio	Reaction Temperature (°C)	Reaction Time (H)	Catalyst Load (wt. %)	Specific Gravity	Viscosity At 40°C (mm^2/S)
1:6	50	1	0.5	0.8685	13.00
1:6	55	1.3	1	0.8586	5.50
1:6	60	2	3	0.9202	7.32
1:6	70	2.3	7	0.9194	120.00
1:8	50	1.3	3	0.8936	7.90
1:8	55	1	7	0.9669	11.90
1:8	60	2.3	0.5	0.9430	12.80
1:8	70	2	1	0.9879	60.00
1:12	50	2	7	0.8983	109.00
1:12	55	2.3	3	0.9044	8.25
1:12	60	1	1	0.9566	29.12
1:12	70	1.3	0.5	0.9887	50.10
1:18	50	2.3	1	0.9262	40.00
1:18	55	2	0.5	0.9025	15.10
1:18	60	1.3	7	0.9661	50.51
1:18	70	1	3	0.9218	28.40

3.1 Optimization Process

In the optimization process, the Taguchi array design in Minitab Statistical tool was able to function as an optimal design for the desired response based on the input criteria and model obtained. The general linear model analysis was carried out to fit the response variable and to investigate the variable that is significant (Table 4). The "P" value less than 0.05 indicated the particular term was statistically significant. The correlation coefficient (R^2) of the analysis is 95.85% which shows the variables fit the model. The analysis showed that reaction time (at 1.30 H) and oil to methanol ratio (at 1:12) were found to be significant while the reaction temperature (P= 0.441) and catalyst load (P= 0.234) were found to be statistically insignificant as shown in Table 4.

Table 4: General Linear Model: Yield (%) versus Reaction Temperature, Reaction Time, Catalyst Load and Oil to Methanol Ratio.

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	94.57	1.72	55.00	0.000	
REACTION TEMPERATURE (°C)					
55	-1.72	1.35	-1.28	0.291	1.50
60	-0.70	1.35	-0.52	0.637	1.50
70	0.73	1.35	0.54	0.625	1.50
REACTION TIME (H)					
1.3	4.73	1.35	3.51	0.039	1.50
2.0	3.56	1.35	2.64	0.078	1.50
2.3	-1.74	1.35	-1.29	0.289	1.50
CATALYST LOAD (wt. %)					
1.0	1.33	1.35	0.99	0.396	1.50
3.0	2.10	1.35	1.56	0.217	1.50
7.0	-1.34	1.35	-0.99	0.394	1.50
OIL TO METHANOL RATIO					
1:08	-3.46	1.35	-2.57	0.083	1.50
1:12	-5.42	1.35	-4.02	0.028	1.50
1:18	0.77	1.35	0.57	0.606	1.50

The regression equation of the analysis is;

YIELD (%) = 94.57 + 0.0 REACTION TEMPERATURE (°C)_50 -
1.72 REACTION TEMPERATURE (°C)_55 - 0.70 REACTION TEMPERATURE (°C)_60
+ 0.73 REACTION TEMPERATURE (°C)_70 + 0.0 REACTION TIME (H)_1.0
+ 4.73 REACTION TIME (H)_1.3 + 3.56 REACTION TIME (H)_2.0 -
1.74 REACTION TIME (H)_2.3 + 0.0 CATALYST LOAD (wt. %)_0.5
+ 1.33 CATALYST LOAD (wt. %)_1.0 + 2.10 CATALYST LOAD (wt. %)_3.0 -
1.34 CATALYST LOAD (wt. %)_7.0 + 0.0 OIL TO METHANOL RATIO_1:06 -
3.46 OIL TO METHANOL RATIO_1:08 - 5.42 OIL TO METHANOL RATIO_1:12
+ 0.77 OIL TO METHANOL RATIO_1:18

The Response surface regression analysis was carried out to fit the response variable to predict the biodiesel yield. The regressors or terms incorporated in the model are those statistically tested to be significant. The model P- value is 0.135, which indicate the variables incorporated fit the model. The analysis as indicated in Table 5, showed that reaction time, catalyst load reaction time*reaction time and reaction temperature*catalyst load significantly affect the biodiesel yield. The reaction temperature, oil to methanol ratio, reaction temperature*reaction time, reaction temperature*methanol to oil ratio and catalyst load*methanol to oil ratio were found to be statistically insignificant. The model developed was successful in capturing the correlation

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between the transesterification conditions variables to the biodiesel yield. The result of regression analysis suggests that biodiesel yield was only significantly affected by reaction time, reaction temperature and catalyst load. Significant interaction terms were found to exist between the main factors reaction time and catalyst load.

Table 5: Response Surface Regression: Yield (%) Versus Reaction Temperature, Reaction Time, Methanol to Oil Ratio and catalyst Load.

Term	Effect	Coef	SE Coeff	T-value	P-value	Response
Constant		34.5	53.4	0.65	0.547	
Reaction temperature (°C)	-0.212	-0.106	0.940	-0.11	0.915	
Reaction time (H)	207.7	103.8	33.2	3.13	0.026	Significant
Catalyst load (g)	-17.92	-8.96	3.42	-2.62	0.047	Significant
Methanol to Oil ratio	-0.18	-0.09	3.65	-0.02	0.982	
Reaction time*Reaction time	-52.92	-26.47	5.65	-4.68	0.005	Significant
Reaction temp*Reaction time	-0.553	-0.277	0.420	-0.66	0.540	
Reaction temp*catalyst load	0.3159	0.1580	0.0942	2.91	0.033	Significant
Reaction temp*Methanol to oil R.O	0.0203	0.0101	0.0488	0.21	0.844	
Reaction time*Methanol to oil R.O	-0.437	-0.219	0.781	-0.28	0.791	
Catalyst load*Methanol to oil R.O	-0.1108	-0.0554	0.832	-0.67	0.535	

The optimization plot as indicated in Figure 1 (Appendix 1), predicts the maximum yield (%) that can be obtained as 99.6513 when target assigned at 100. The condition of the transesterification variables to obtain the maximum yield as predicts by the statistical model (optimization plot) were 18:1(0.05422) Methanol to oil ratio, 0.5 wt % catalyst load, 70 °C temperature and 1.3 h reaction time.

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With a predicted yield of 99.6514 % which is closely similar with experimental yield (99.68 %), it is clear that optimization of biodiesel production could be achieved through regression analysis using a known catalyst and other reaction variables.

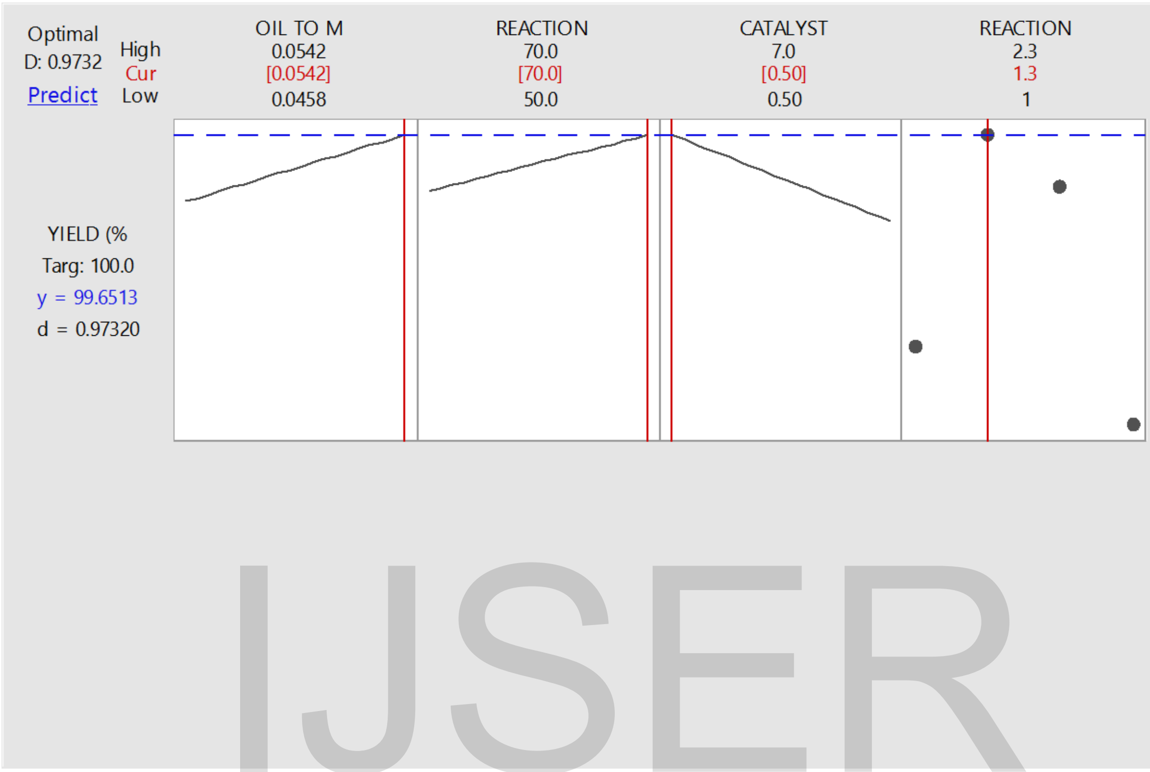

Fig. 1: Optimization plot

3.2 Effects of Transesterification Variables on Biodiesel Yield

The effects of methanol to oil ratio, catalyst load, reaction time reaction temperature and their interactions were studied to check the effect of each variable toward biodiesel production. The contour plots were used to analyze the interaction.

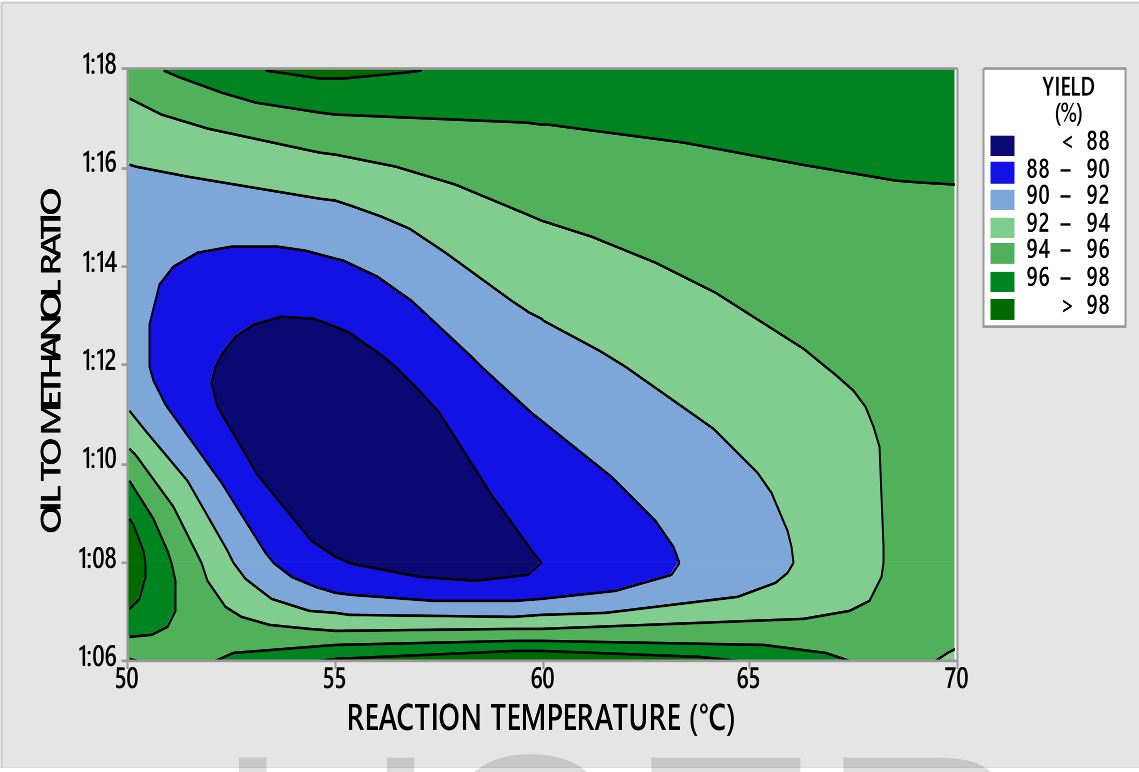

Fig. 2: effects of methanol to oil ratio and reaction temperature on biodiesel yield

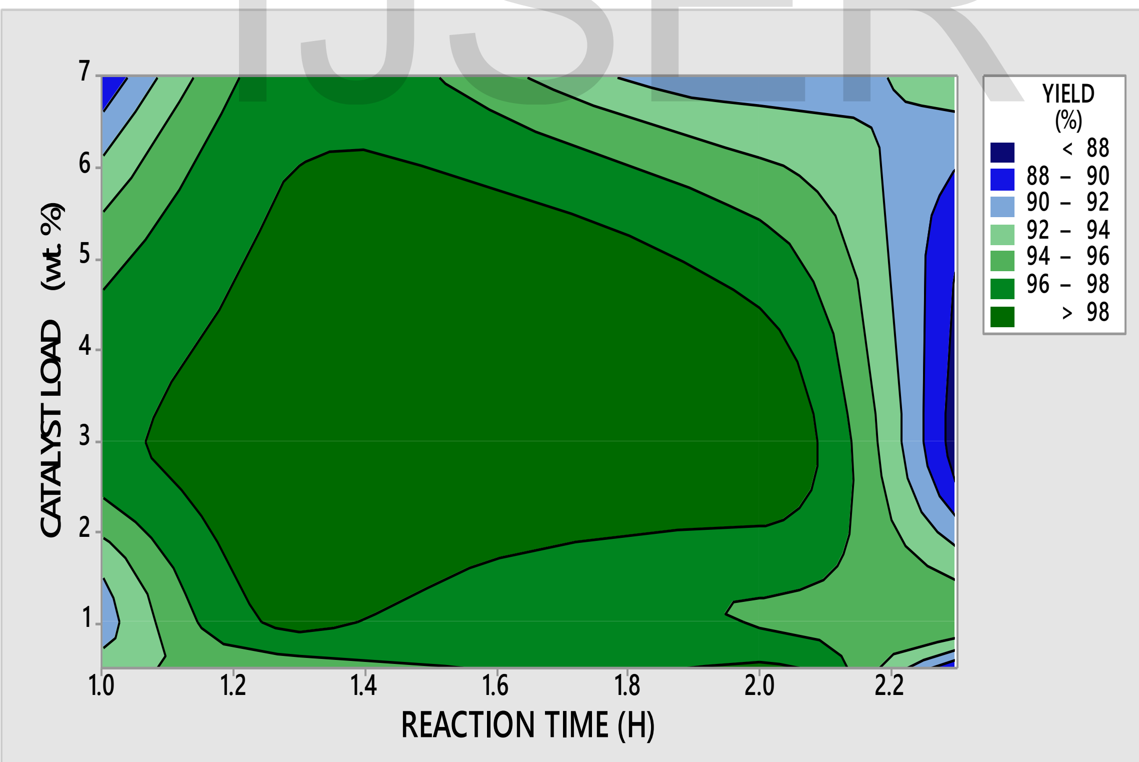

Fig. 3: Effect of catalyst load reaction time on biodiesel yield

I. Effect of Oil to Methanol Ratio on Biodiesel Yield

The contour plot as illustrated in Fig. 2 indicates that the molar ratio of oil to methanol has a significant impact on biodiesel yield. The optimum yield (99.68 %) was obtained at 1:06 oil to methanol ratio. The excess of methanol is necessary because it can increase the rate of methanolysis. The high amount of methanol promoted the formation of methoxy species on the CaO surface, leading to a shift in the equilibrium towards the forward direction, thus increasing the rate of biodiesel conversion (Ismail *et al.,* 2016). However, the yield was slightly reduced when the oil to methanol ratio was high than 1:06 at 60 ° C temperatures as indicated in the Fig. 2. Continues increase in oil to methanol ratio after optimal ratio 1:06 would lead to a reduction in biodiesel yield. This is due to excessive methanol beyond the optimal point which does not promote the reaction. The glycerol which is a byproduct of the reaction would largely dissolve in methanol and subsequently inhibit the reaction of methanol to reactants and catalyst, thus interfering with the separation of glycerine, which in turn lowers the conversion by shifting the equilibrium in the inverse direction (Ismail *et al.,* 2016).

II. Effects of Reaction Temperature on Biodiesel Yield

The Fig. 2 shows the biodiesel yield from transesterification of refined castor oil at the different reaction temperature from 50 °C to 70 °C. The biodiesel yield increases with reaction temperature until at an optimal point of 60 °C with biodiesel yield of 99.68%, it agreed with optimal temperature obtained by Ismail *et al.,* (2016). Beyond this temperature, yield decreased to averagely 94.77% at 70 °C. Initially, some thermal energy was needed for transesterification as the reaction was endothermic (Samart *et al.,* 2009). Since reaction mixture constitutes a three-phase system (Oil- methanol- catalyst), the thermal energy was sufficiently needed to overcome the diffusion resistance between different phases (Ismail *et al.,* 2016). However, the high temperatures are not preferred, as the temperature increase and reached the boiling point of methanol (64.7 °C), the methanol immediately vaporize and form a large number of bubbles, which inhibits the reaction on the two-phase interface and thus decreases the biodiesel yield (Long *et al.,* 2010).

III. Effect of Reaction Time on Biodiesel Yield

Fig. 3 shows the biodiesel yield for transesterification of castor oil in different reaction time from 1 to 2.5 hours. In the initial stages of the transesterification, production of biodiesel was rapid until the reaction has reached equilibrium. Beyond the optimum reaction time (2 hours), the reaction starts to progress in a backward direction towards reactants. This phenomenon occurred due to the reversibility of transesterification reaction (Samart *et al.,* 2009). CaO catalyst has a tendency to absorb the product (Ismail *et al.,* (2016). Hence it is important to identify the optimum reaction time for transesterification reaction. In this experiment, the optimum reaction time was 2 hours at 3 g catalyst load (Fig. 3). The similar result of optimum reaction time was obtained by Ismail *et al.,* (2016).

IV. Effect of Catalyst Load on Biodiesel on Biodiesel Yield

The catalyst loads play a vital role in optimizing the yield of the transesterification reaction. From the Fig. 3, it can be observed that the biodiesel yield increase with an increase in catalyst load from 0.5 to 3 wt% with respect to time. And also the yield was slightly decreased with further increase of catalyst load beyond optimal value 3 g. Optimal catalyst load was determined to be 3 g with biodiesel yield of 99.68%. The excess catalyst has slightly reduced the biodiesel yield due to soap formation in the presence of high amount of catalyst concentration. Furthermore, this excess amount of catalyst increases the viscosity of reactant which also results in lowering the biodiesel yield as reported by Yang *et al.,* (2009). The transesterification is not solely catalyzed by the basic sites generated on the surface of CaO catalyst but also the soluble substance leached away from CaO catalyst (Granados *et al.,* (2007).

3.3 Effect of Transesterification Variables on the Quality of Biodiesel

The quality of biodiesel is measured mainly in terms of viscosity, since this one of the important specification in ASTM standards and it directly governs the flow of fuel in the engine. Hence, the quality of biodiesel is discussed in terms of change in viscosity with the variables like reaction time, reaction temperature, oil to methanol ratio and catalyst concentration.

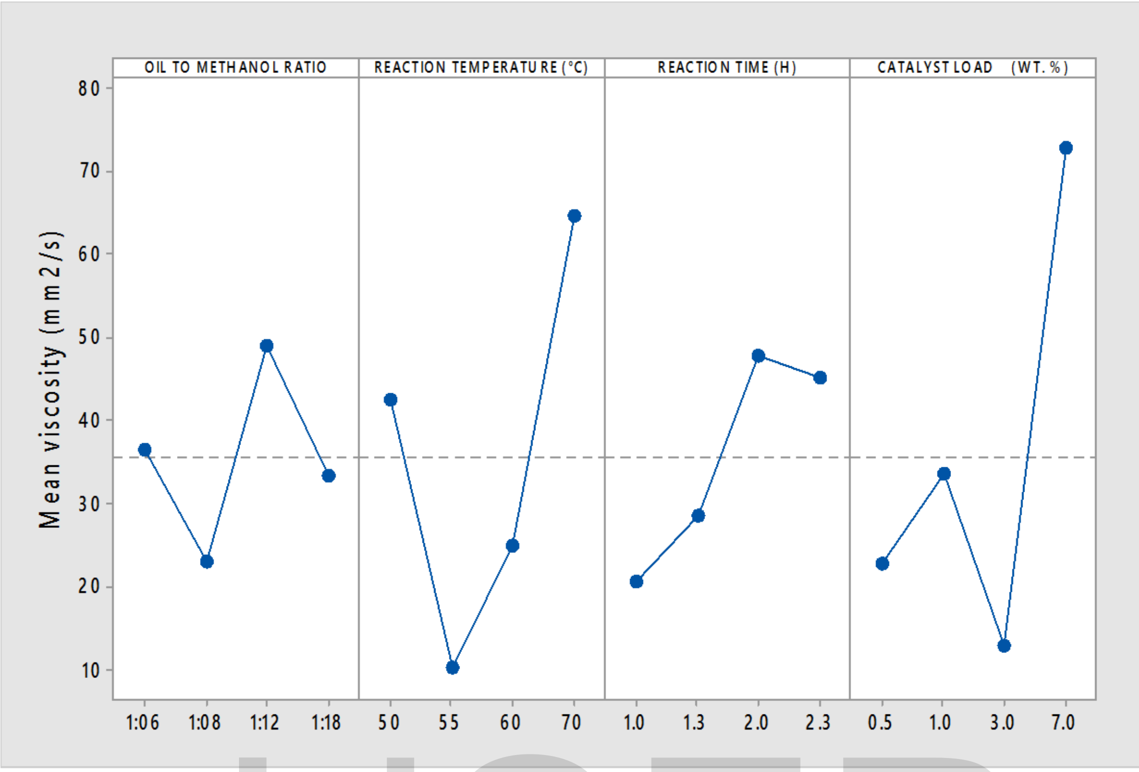

Fig. 4: Effects of transesterification variables on the viscosity of biodiesel

I. Effect of Oil to Methanol Ratio on Viscosity of Biodiesel

The experimental results illustrated in the Fig. 4 shows that the oil to methanol ratio has an effect on the viscosity of the biodiesel produced. It can be observed that the average viscosity was slight decreases as molar ratio increase from 1:6 to 1:8 and also significantly increase to 50 mm^2/s as the molar ratio increases to 1:12 accordingly, this may be due to the excess methanol that has reached saturation point with glycerol, catalyst and soap, which virtually make the separation of biodiesel difficult, thereby increasing the impurities in the biodiesel, hence, increases the viscosity (Deshpande *et al.*, 2012). The viscosity of the oil significantly reduced to averagely 35 mm^2/s as molar ratio increases further to 1:18. Similar results were obtained by Despande *et al.*, (2012) whereby using sodium hydroxide catalyst, as the oil to methanol ratio increase from 1:6 to 1:9, the viscosity of biodiesel decreased from 15.68 to 13.10 mm^2/s. This is because the glycerol, soap, catalyst and other impurities are readily soluble in the excess methanol (lower than saturation point), thereby making them easier to be removed, hence, lower the viscosity.

II. Effect of the Reaction Temperature on the Viscosity of the Biodiesel

From Fig. 4, as the temperature increases from 50 to 55 °C, then mean viscosity of the biodiesel drastically decreases from 40 mm^2/s to about 9 mm^2/s, this may be due to that, at lower temperature the methanol smoothly reacts with the oil to form good quality biodiesel (Deshpande *et al.* 2012). As the temperature rises from 55 to 70 °C, the mean viscosity was significant rises from about 9 mm^2/s to above 60 mm^2/s, this is due to fact that as temperature increase and reached the boiling point of methanol (64.7 °C), the methanol immediately vaporize and form a large number of bubbles, which inhibits the reaction on the two-phase interface and thus decreases the biodiesel yield (Long *et al.*, 2010) and also affects the quality of the biodiesel.

III. Effect of Reaction Time on the Viscosity of the Oil

From the Fig. 4 it can be seen that, as the time of the transesterification increases from 1 hour to 2 hours, the viscosity drastically increased from 20 mm^2/s to about 50 mm^2/s. The mean viscosity decreases as the time exceed optimum 2 hours reaction time. The reduction may be due to enough time that the reaction needed to completely transesterified the oil completely.

IV. Effect of Catalyst Load on the Viscosity of the Biodiesel

In Fig. 4, when catalyst concentration increase from 0.5 to 1.0 wt%, the mean viscosity increase from about 25 to 35 mm^2/s and hence, drastically reduces to less than 10 mm^2/s at optimal catalyst load (3.0 g). After then, as the catalyst load increase from optimal point to 7.0 g, the mean viscosity significantly increases from about 9 to above 70 mm^2/s. The excess catalyst has slightly reduced the biodiesel yield due to soap formation in the presence of high amount of catalyst concentration. Furthermore, this excess amount of catalyst increases the viscosity of reactant which also results in lowering the biodiesel yield and its quality as reported by Yang *et al.*, (2009).

3.4 Conclusion

The optimization of biodiesel production from castor seed oil using refluxed calcined snail shell as a catalyst based on Taguchi orthogonal array design was successful and able to produce the optimum yield of 99.68 % at 1:6 oil to methanol ratio, 60 °C, 2 hours and 3 w% catalyst load. The biodiesel yields range 88.02 – 99.68 %. The response surface regression show that reaction time, catalyst load reaction time*reaction time and reaction temperature*catalyst load significantly affect the biodiesel yield. The reaction temperature, oil to methanol ratio, reaction temperature*reaction time, reaction temperature*methanol to oil ratio and catalyst load*methanol to oil ratio were found to be statistically insignificant. The model developed was successful in capturing the correlation between the transesterification conditions variables to the biodiesel yield.

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Appendices

Appendix 1: Response Optimization: YIELD (%)

Parameters

Response	Goal	Lower	Target	Upper	Weight	Importance
YIELD (%)	Target	86.99	100	110	1	1

Solution

Solution	OIL TO METHANOL RATIO	REACTION TEMPERATURE (°C)	CATALYST LOAD (g)	REACTION TIME (H)	YIELD (%) Fit	Composite Desirability
1	1:18	70	0.5	1.3	99.6513	0.973196

Multiple Response Prediction

Variable	Setting
OIL TO METHANOL RATIO	1:18
REACTION TEMPERATURE (°C)	70
CATALYST LOAD (g)	0.5
REACTION TIME (H)	1.3

Response	Fit	SE Fit	95% CI	95% PI
YIELD (%)	99.65	3.00	(92.87, 106.43)	(88.55, 110.75)