

# The Effect of Catalyst Type and Concentration on the Yield of Biodiesel from Jathropa/Moringa Oil Mix

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**Abstract:** The exhaustible nature of petroleum diesel has made biodiesel an attractive alternative to diesel, but efforts at reducing its high production costs through the use of cheap poor-quality oil feedstock are not sufficient. A careful choice of catalyst concentration and type is also necessary. Binary blends of oil-feedstock and alcohol-feedstock were used to increase the efficiency of transesterification process. The effect of changing alkaline catalysts on the yield of transesterification reactions was also examined for similar and varied process conditions. This allowed the examination of the impacts of catalyst on yield with changing feedstock type and processing condition. Results of oil and alcohol blending showed that the partial use of ethanol permitted reactions to be completed in 45 minutes instead of 60- 120 minutes common to conventional ethanolysis. Yields were comparable to those of methanolysis. Ester yield increased with increase in the concentration of KOH and CaO catalysts within the 0.5-2.5 w/w% range. However, ester yield decreased with increasing concentration of NaOH beyond 0.2 w/w%. This indicated that reasonable contributions to cost saving were possible as smaller quantities of NaOH catalyst were required to increase ester yield when a Jathropa-Moringa oil-feedstock blend was transesterified with an alcohol blend of 4:1 methanol to ethanol mix ratio. Results for varying catalyst type showed that differences in ester yield as the alkaline catalysts were varied was significant at  $p=0.05$ . Catalyst performance with regard to ester yield for Jathropa/Moringa feedstock, was in the order  $\text{KOH} > \text{NaOH} > \text{CaO}$ .

**Keywords:** catalyst-type, concentration, transesterification, pretreatment, blending, ester.

## 1. Introduction

Energy is a basic requirement for most human activities. However, natural reserves of fossil fuel are running out and the fuels produced thereof are environmentally polluting. This fact has motivated attention in renewable alternatives such as biodiesel sourced from biomass. Biodiesel production though is not cost competitive with petroleum diesel because of high production costs. An important contributor to this high cost is the high cost of alkaline catalysts. Changing economic variables and other external factors affect the prices and availability of these catalysts. Hence, various homogenous alkaline catalysts may be used at different times in a small plant. However, the effect on ester yield of using an alkaline catalyst varies with oil feedstock. This work provides information required to make the best advantage of expensive catalysts by applying them only to feedstock with which they give the most yield during transesterification. This work also examined the yield trend of three alkaline catalyst as a function of their corresponding concentration to find gainful levels and ranges that supported lower use of catalyst. The purpose is to reduce production cost.

Biodiesel production from oil feedstock for example, has been remarkably noted for high production costs [1]. Contributions to these high costs include the use of expensive edible oil feedstock like soybean, palm oil and peanut oil [2, 3, 4, 5]. However, attempts to reduce production costs by using non-edible feedstock have proved insufficient because of land management practices required to grow those [6]. Furthermore, in some circumstances, up to 50% of net annual profit from biodiesel production are spent on catalysts alone [7]. The effect of varying these catalysts on ester yield depends on type of feedstock applied [8, 9, 10]. NaOH at 1 w/w% of oil feedstock resulted in yields of 98.4 and 87 % for waste sunflower oil and Canola oils respectively [11, 12]. Similarly, KOH at 1 w/w% of oil feedstock resulted in yields of 41 % and 79 % for Karanja oil and Jathropa oils respectively [13]. Thus reducing the cost of alkaline catalysts used for transesterification reactions is required to complement cost reduction efforts made by using nonedible feedstock. Since the exclusive use of non-edible feedstock does not completely solve cost problems, it may be appropriate to blend them with edible feedstock if it advances the goal of cost saving as in some previous works [14, 15, 16]. Information on the impact of alkaline catalysts

on ester yields from these new blends will also determine their preference.

Alkaline transesterification of poor quality oil feedstock is hardly successful and consumes catalysts by soap formation when ester yield is significant. Nevertheless, an option of acid catalysis which can cause high ester yield without soap formation, has its challenges. It requires long reaction times and high temperatures, considerably adding to production costs [17, 18]. Considering the fact that alkaline catalysts do not share these limitations, this work intends to find gainful ranges and levels of concentrations for use of alkaline catalysts in the transesterification of two blended oil feedstock, Jathropa and Moringa oils. It also employed the partial use of ethanol to increase the efficiency of the transesterification process and consequently aid a better monitoring of the effects of concentration, temperature, and stirring speed on ester yield. While comparisons of catalysts are better done at identical processing conditions, some biodiesel plants may change the alkaline catalyst applied without maintaining a single process condition. Thus it will be beneficial to determine if the facts learned about the effect of catalyst type on yield at similar process conditions, can apply to varying process conditions for a single feedstock. This study examines this question using Jathropa/Moringa oil mix considering that a 90% extraction of the high oil content of Jathropa is attainable [19].

## 2. Materials and Method.

### 2.1 Materials

Jathropa and Moringa oil feedstock were purchased from local farmers in Kogi and Benue states respectively. A mechanical screw press was used for oil extraction. Methanol (CH<sub>3</sub>OH), and ethanol (C<sub>2</sub>H<sub>5</sub>OH) of 95 % and 99 % purity respectively, were procured in 2.5 L glass containers from chemical stores in Ibadan, Oyo state. NaOH, KOH, and CaO pelletised catalysts were also bought in 500 g packs from those stores. A laboratory scale biodiesel reactor designed to process small amounts of feedstock was used for transesterification. Other equipment used for this study were electronic weighing balance, and a set of apparatus for volumetric analysis.

### 2.2 Transesterification Reaction

The oil blends were stirred in the blender to ensure a proper mix. Alkoxide mixtures were also prepared in small glass bottles, one for each experimental run by dissolving the appropriate weights of catalyst pellets in a proportion of alcohol, the mass of which had been predetermined according to the alcohol to oil molar ratio for the run of experiment. The bottle of Alkoxide was closed and vigorously shaken for 10-15 minutes to ensure complete dissolution of the pelletised catalysts. 20 g of blended oil feedstock was measured and heated to 65 °C for 5 minutes. The heated oil was left to cool and emptied into the reactor which had been set to the temperature and stirring speed for the run of experiment. Each experiment lasted 45 minutes. The alkyl-esters yielded was washed and evaporated at 120°C for 24 hours and measured to determine yield. The

experiment was accomplished using a mixture/RSM method having 31 runs.

### 2.3 Fixed Reaction Condition

The three catalysts were studied under the same processing conditions. The transesterification condition was: Jathropa to Moringa blending ratio of 4:1, Methanol to ethanol blending ratio of 4:1, Concentration of 0.5 w/w% of oil blend, Mix speed of 1000 rpm, Temperature of 60°C and an Alcohol to oil molar ratio of 7.5. Every reaction was replicated 3 times for each catalyst such that there were 9 runs of experiment. A working weight of 20 g was used in these reactions.

### 2.4 Varied Reaction Condition

The process conditions are: Temperatures ranged from 40 - 60 °C; Stirring speeds of 500-1500 rpm; Alcohol to oil molar ratios of 45:1 to 9:1; methanol to ethanol blending ratio of 4:1; Moringa to Jathropa oil blending ratios of 1:4 - 4:1; and catalyst concentrations of 0.2 - 2.5 w/w%. These conditions were applied according to specifications in the experimental plan. A combined optimal custom experimental design (in 1- optimal mode) was used using Design expert 10 software.

### 2.5 Alcohol measurements

Molecular mass of Alcohol blend for all runs was determined using equation 1 [20]: 
$$N_a = \frac{4M+E}{5} \quad (1)$$

Ref. [21] reported the following equation as generic to all alkaline transesterification reactions in which the alcohol to oil ratio is 3:1 
$$TG + 3ME \rightleftharpoons 3B + G \quad (2)$$

If  $n_i$  and  $l_i$  are moles of oil blend and alcohol blend respectively in the equation above, it then follows that  $n_i = 1$  and  $l_i = 3$  so that and  $l_i = 3n_i$ . Therefore, for a run of experiment  $i$ , using alcohol to oil Mole ratio,  $l: 1$ ,

**Mass of alcohol blend required**, as expressed by [22].

$$= m_a = l_i N_a = 3n_i N_a \quad (3)$$

### 2.6 Oil Measurements

Molecular mass of oil blend for each run was determined using equation 4 [20].

$$N_i = \frac{xM+yJ}{x+y} \quad (4)$$

### 2.7 Yield of Biodiesel

Percentage yield was determined using equation 5 [23, 24].

$$\text{Percentage Yield, } Z = \frac{100A}{Y} \quad (5)$$

Where A=Actual yield in grams. A weight-wise rendering of the generic expression in equation 2 can be adapted as:



Application of proportionate mass assignment to the oil and ester species in Eq. (6) for 'm' grams **of oil gives:**

$$Y = \text{Theoretical Yield} = \frac{m(l_i H_i)}{N_i} \tag{7}$$

Where: M and E = RMM of Methanol and Ethanol;  $N_i$  and  $N_a$  = molecular mass of the oil blend and alcohol blend;  $M, J$  and  $H_i$  = molecular mass of Moringa, Jathropha, and ester:  $m$  and  $m_a$  = masses in grams of oil and alcohol blend:  $n_i$  and  $l_i$  = moles of oil blend and alcohol blend for the  $i$ th, run;  $x$  and  $y$  = proportions of Moringa and Jathropha.  $M = 927$  g/mol and  $J = 902$  g/mol [20, 25, 26];  $M = 32.04$  g/mol and  $E = 46.07$  g/mol [27]

### 3. Results and Discussion

#### 3.1 Effects of catalyst type on biodiesel yield for varied process conditions

Table 1 showed ester yield from different alkaline catalysts at various concentrations with average yields of 76.81, 72.00 and 28.84 % for KOH, NaOH and CaO. From the result, KOH performed better than sodium hydroxide, with calcium oxide as the least.

However, as shown in Table 2, the effect of the three alkaline catalyst type on the yield of biodiesel was significant at  $p=0.05$ . The significant differences in yield for the three catalyst indicated that NaOH cannot be used as a substitute for KOH if similarly high yields are to be achieved.

This result is similar to that obtained under a fixed condition for the three catalysts. Ref. [28] also rated potassium hydroxide catalyst as a better catalyst than sodium hydroxide. Furthermore, it agreed with [29] in describing

$$\text{Eq. 6 can be adapted as: } l_i H_i \rightleftharpoons (N_i + l_i N_a) - 92.10 \tag{8}$$

calcium oxide as a less efficient catalyst than sodium hydroxide. However, this result differs from the findings of [6] which rated sodium hydroxide catalyst as better performing than potassium hydroxide. This inconsistency had arisen from the fact that a different feedstock was used. Secondly, yield calculations by [6] were done only after washing and without neutralisation of the esters. This caused the effects and after effects of alkaline catalyst activity to be profound. NaOH soap (hard soap) as opposed to KOH soap (soft soap) settled faster, resulting in higher yield than KOH catalysed reactions for the same settling duration. Conversely, work done by [28] may have included neutralisation as in this work. This had tempered the soap formation and soap settling effects of NaOH and KOH catalyst in a way that affected yield measurements.

This result indicates that KOH is a more efficient alkaline catalyst than the others if a Jathropha/Moringa oil blend is transesterified in turns with the three catalyst under the same process condition. Hence it is recommended in preference to sodium hydroxide because it can be prepared from cheap agricultural materials such as cocoa pods. Calcium oxide with the least yield of biodiesel can still be used in the absence of a potassium hydroxide catalyst. This is because it is cheaper than the hydroxide catalysts and can be sourced from biomass.

Table 1. Ester yield as a function of Catalyst type

CaO catalysed reactions			NaOH Catalysed reactions			KOH catalysed reactions		
S/N	Run	Yield (%)	S/N	Run	Yield (%)	S/N	Run	Yield (%)
1	13	14.78	1	12	84.66	1	8	78.48
2	14	18.13	2	19	87.89	2	10	68.13
3	20	20.47	3	23	73.63	3	16	87.95
4	30	5.92	4	18	62.02	4	15	75.12
5	9	56.97	5	4	79.06	5	26	85.83
6	1	27.13	6	6	68.43	6	3	71.83
7	21	21.26	7	25	64.37	7	27	80.16
8	22	37.09	8	31	76.6	8	17	80.41
9	2	34.04	9	5	55.59	9	24	71.67
10	7	52.58	10	11	67.73	10	28	73.99
						11	29	71.34
<b>Average</b>		<b>28.84</b>			<b>72.00</b>			<b>76.81</b>

Table 2 Mean separation for Effects of catalyst type on ester yield

Catalyst Type	Mean Yield (%)
CaO	20.88 <sup>a</sup> (3.06379)
NaOH	60.19 <sup>b</sup> (1.73127)

KOH

74.47<sup>c</sup> (4.22532)

Values with similar superscripts within the same column are not significantly different at  $p=0.05$ . Values enclosed in parenthesis are standard deviation

### 3.2 Effects of Catalyst Concentration

Figure 1 depicts a graph of yield patterns that varied with the concentration of three different catalysts. An increase in NaOH concentration reduces the yield of biodiesel. Therefore, 0.2 w/w concentration is adequate for increased biodiesel yield. Whereas, increase in biodiesel yield was observed with an increase in the concentration for the two other catalysts in the oil blend. The KOH catalyst yielded more biodiesel at the same concentration than CaO. However, in terms of the quantity required for increased yield, NaOH will be adequate. Transesterification at higher concentrations for this catalysts resulted in poorer yield. The negative trend line also revealed that it may be possible to achieve higher yields at lower concentrations. Ref. [28] achieved higher yields at 0.1w/w percent concentration of sodium hydroxide than at 0.2 w/w percent. The behaviour of sodium hydroxide in this study compares with studies by [30] in which the ethanolysis of castor oil using sodium hydroxide attained a maximum yield at exactly 0.2 w/w percent of oil feedstock, and resulted in decreasing yield at higher catalyst concentration. The result showed that it was not beneficial to increase the concentration of sodium hydroxide above its lowest point of 0.2 w/w percent.

However, the positive slopes of the potassium hydroxide and calcium oxide trend lines show that it was gainful to increase the concentration of calcium oxide beyond 0.5 w/w percent of oil feedstock as it improved alkyl-esters yield. A good comparison for the potassium hydroxide trend line in Fig. 1 was reported by [31] who found an upward trend of alkyl-ester yield until the concentration of potassium hydroxide was above 1 w/w percent of karanja oil. Ref. [32] similarly showed that yield increases with increasing concentration of calcium oxide in catalyst from 0.5 w/w percent of oil feedstock even up to 5 w/w percent where a decline in alkyl-ester yield begins. CaO as in this work has generally been observed in other studies, as poor performing [33].

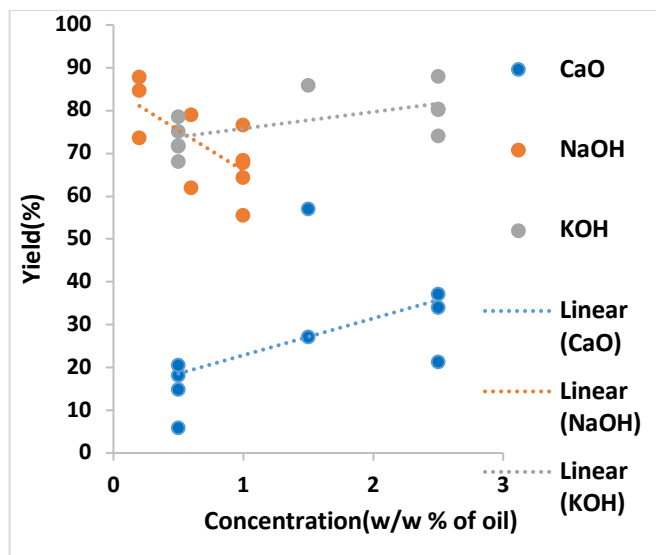


Figure 1. Effect of catalyst type and concentration on yield of biodiesel

### 3.3 Comparison of Partial Ethanolysis to Methanolysis

As shown in table 3, the calculated F-value of 0.507613 was less than the tabulated F-value of 4.413873, indicating that differences in alkyl-ester yield by methanolysis and ethanolysis are not significant.

Therefore these results indicated that the yield patterns from the use of ethanol and methanol are sufficiently similar. The result is similar to what was obtained by [34] that yields through ethanolysis of castor oil with hydroxide catalysts compared closely with those of methanolysis if sufficient reaction time was assigned. This showed that significant losses in yield may not occur in attempting to produce biodiesel through the use of methanol-ethanol blend instead of methanol.

### 3.4 Performance of the Alcohol blend at various Alcohol to Oil Molar Ratios

As shown in Fig. 2, an increase in alcohol to oil molar ratio decrease the yield at first, at a ratio of 4.5:1 to 6:1. An increase in the yield was observed from the ratio from 6:1 to 7.5:1, and finally decreased beyond 7.5:1. This was observed for all the catalysts used.

The initial reduction in yield was due to incomplete reaction that resulted from the high concentration of alkaline catalyst in the alcohol phase at low alcohol-oil molar ratio. This caused a high diffusion of alkaline catalysts from the alcohol

phase to the oil reacting with free fatty acids to form soap, thereby reducing the amount of catalyst available to drive the reaction [28].

Table 3. ANOVA table comparing partial Ethanolysis to Methanolysis

Groups	Count	Sum	Average	Variance
E	10	592.24	59.224	312.6585
M	10	651.46	65.1464	378.3168

ANOVA				
				F=0.507613
				F <sub>c</sub> =4.413873
				p=0.485308
SV	SS	df	MS	
BG	175.37	1	175.3741	
WG	6218.77	18	345.4877	
Total	6394.15	19		

Note: E = Ethanolysis; M = Methanolysis; SV = Source of variation; SS = Sum of squares; df = degrees of freedom; MS = Mean squares; F = F-calculated; F<sub>c</sub> = F-critical; P = P-value; BG=Between groups; WG= within groups

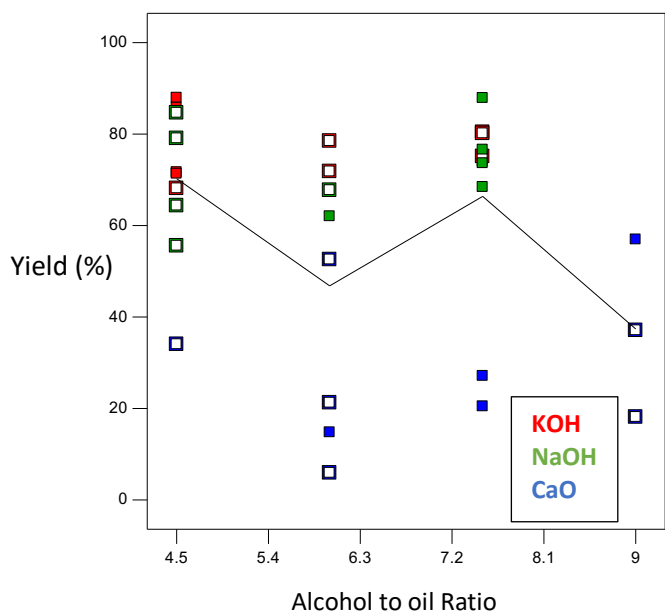


Figure 2. General effect of alcohol to oil molar ratio on yield

However, beyond the alcohol to oil molar ratio of 6:1, yield rose because the high volume of alcohol had reduced the concentration of alkaline catalyst in the alcohol phase, causing minimal diffusion into the oil phase. Hence, less catalyst was lost to soap formation and more was available for the transesterification reaction. Moreover, beyond the

alcohol to oil ratio of 7.5:1, excess alcohol increased the solubility of the alcohol blend, preventing glycerol from separating. This glycerol then encouraged backward reaction and decreased yield [35]. The low yield was worsened by the fact that a very poor catalyst, calcium oxide was used at this high alcohol-oil molar ratio.

This result agrees with the findings of [35] who transesterified cynara oil at various alcohol to oil molar ratios. They found that at alcohol to oil molar ratios less than 6, ester yield decreased with increasing alcohol to oil molar ratio until the ratio got to 6:1 beyond which yield started to increase with increasing alcohol to oil molar ratio. However, the yield again traced a downward path with further increase of alcohol to oil molar beyond 12:1. The ideal alcohol to oil molar should be within the range of 6:1 and 7.5:1 without which losses in yield will occur.

### 3.5 Effects of Temperature

As temperature increased, alkyl-ester yield increased from 40 °C to a peak of 50 °C and then decreased with continued increase in temperature (Fig. 3). This trend agreed with studies conducted by [36] in which methanolysis of neat and used frying oils were carried out between 30 °C and 70 °C. The initial rise and subsequent fall in yield of esters with temperature increase has been attributed to the impact of temperature rise on the viscosity of the feedstock blend and saponification effects [36].

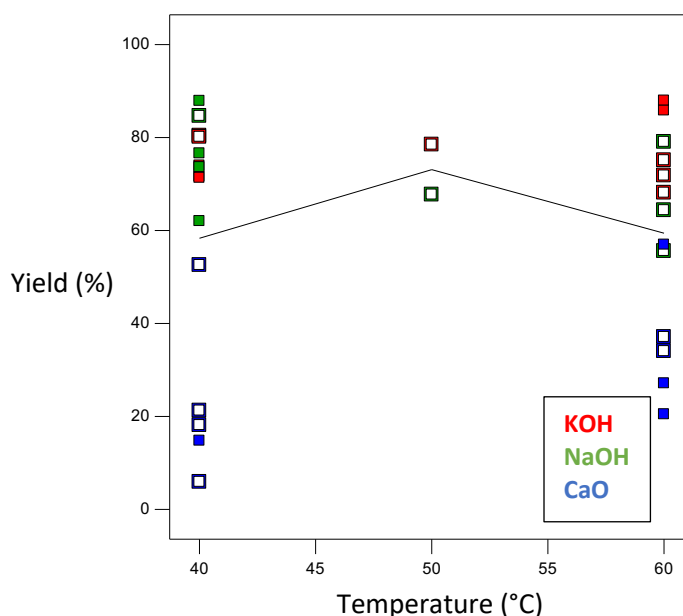


Figure 3. Effect of temperature on biodiesel yield for different catalyst types.

The initial increase in temperature had reduced the viscosity of the oil, allowing the oil phase and alcohol phase to mix easily, hence speeding up the reaction and causing more yield. However, further increase in temperature had made

saponification of triglycerides to become more pronounced, thereby, causing a decrease in the yield.

Therefore, high ester yields were possible at the temperature of 40°C when sodium hydroxide and potassium hydroxide catalysts were used. However, high yields were not possible even at high temperatures when calcium oxide -a less reactive catalyst- was used.

### 3.6 Effects of Stirring Speed

Ester yield initially increased with stirring speed as shown in Fig. 4, but slightly decreased as increments in stirring speed were sustained. This also meant that surface area was increased with increased stirring speed. However, in a similar study involving the methanolysis of beef tallow using sodium hydroxide catalyst, Ref. [37] claimed that methyl-ester yield was not affected by stirring speed, but reaction time. This disagreement can be explained by considering studies published by [21] which explained that increase in surface area by thorough mixing was only required at the beginning of a reaction to bring the oil, and alkoxide phases into contact. This indicates that for cases, as in the current study in which a constant reaction time of 45 minutes was maintained for all experiments, continued and increased stirring beyond complete phase combination amounted to disturbances that hindered the progress of the reaction and reduced the rate of increase in yield. However, high stirring speed did shorten the blending time and brought the two reacting phases together in less time leaving more reaction time for transesterification, and so increasing yield.

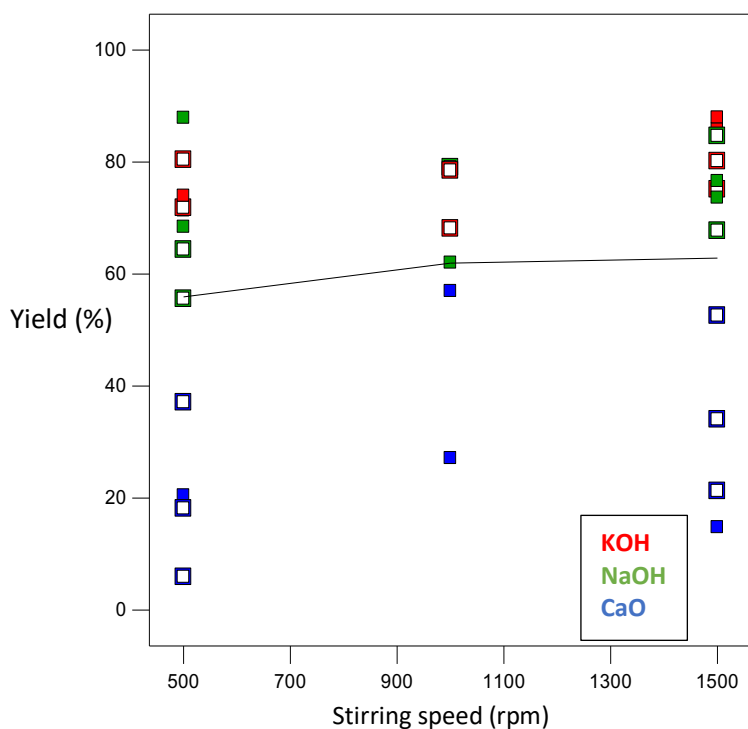


Figure 4. Effect of stirring speed on yield

Conversely, a low stirring speed will require more time to harmonise the two reacting phases, reducing available

reaction time and consequently decreasing ester yield. Therefore, it may be beneficial to increase surface area by using high stirring speeds for a very short time at beginning of a transesterification reaction, after which stirring speed may either be reduced or totally stopped.

## 4. Conclusions

The fixed methanol to ethanol blending ratio of 4:1 supported high yields for all catalysts.

The range of catalyst concentrations used for this study can support high ester yield. Although NaOH and KOH catalysts are more expensive than CaO, they can be used to successfully achieve high ester yields and this is desirable if other parameters are combined to defray high catalyst costs.

Considering the inverse relationship for NaOH catalyst, it is possible to achieve higher yields with lesser amount of NaOH if catalyst concentrations below 0.2 w/w% are applied. This has important business implications for biodiesel plants and firms that run them. The blending ratio for alcohol feedstock which was fixed in this study can also be varied to determine the highest permissible amount of ethanol in the blend, beyond which gainful ester production is no longer possible. KOH and CaO are recommended given their biomass origin.

Alcohol to oil ratios between 6:1 and 7.5:1, temperatures of 40-50 °C and high but short lasting stirring speeds can boost the performance of these catalysts if the appropriate level or range of catalyst concentration are applied.

Comparisons between the results of this study and those of previous studies indicated that a change in feedstock does affect the performance of alkaline catalysts used for transesterification reaction. However, changes in process conditions did not affect the performance ranking of the three catalysts studied for *Jathropa Moringa* feedstock despite variations in its blending ratio. It is also not appropriate to use these alkaline catalysts interchangeably if similarly high ester yields are required for a given Biodiesel plant. This is true for similar and varied process conditions if the same feedstock is applied.

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