Comparative Study of Different Concentrations of NaOH and KOH Embedded Bentonite Clay as Heterogeneous Catalyst on Transesterification Variables of the Methyl Esters of Castor Seed Oils in Nigeria

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ABSTRACT

The oil seed production in Nigeria is still developing, however it is imperative to seek for alternative because conventional diesel may be out of vogue at any time due to innovation and development in science and technology. In this study, Castor oil was used to produce biodiesel while the objective was to determine the most effective ratio of NaOH and KOH embedded bentonite clay as heterogeneous catalyst that will give highest yield. Determination of optimal reaction conditions was also determined. Decoction and degumming processes were carried out on the seed oil in order to reduce the moisture content and gums respectively. Impregnation of both base accelerators were prepared in ratios 1:10, 1:5, 1:4, 1:3, 1:2 and 1:1. In order to optimize the yield of biodiesel during the transesterification process, a Box-Behnken experimental design and the response surface methodology were used. The studied factors were: reaction time, reaction temperature, catalyst loading, molar ratio and agitation speed. Results indicates that the optimal reaction conditions during transesterification of KOH accelerator, 60mins of reaction time, temperature of 59 ^{o}C, catalyst loading of 0.29g, 8:1 methanol to oil ratio and at agitation speed of 800 rpm.while 95 % yield of biodiesel conversion was obtained.

Keywords: Concentrations, NaOH, KOH, Bentonite, Clay, Heterogeneous Catalyst, Transesterification Variables, Methyl Esters, Castor Seed Oils & Nigeria

1. INTRODUCTION

Biodiesel is basically produced along with glycerin in transesterification where fat or oil reacts with an alcohol in the presence of a catalyst which is either sodium hydroxide or potassium hydroxide (Raja, Anthony, Robinson and Lindon, 2011). Much attention has been given to the development and utilization of biodiesel because of its renewable nature and compatibility with the existing engines without modification (Awolu and Layokun, 2013). It is also found to be environmentally friendly in that its use does not cause acid rain due to absence of sulphur compounds and its non-toxic nature informed the wide applications in highly sensitive environments like mining enclosures and marine ecosystems. (Helwani, Othman, Aziz, Fernando and Kim, 2009).

The transesterification reaction is characterized by the displacement of alcohol from an ester by another alcohol in a process similar to hydrolysis except that an alcohol is used instead of water (Murugesan, Umarani, Chinnusamy, Krishnan, Subramanian, and Neduchenzhain 2009). The process may either be one or two steps whereby esterification is adopted first in order to reduce the Free Fatty Acid (FFA) and then transesterification process follows in the presence of a catalyst that may be homogenous or heterogeneous in nature (Khalizani and Khalisanni, 2011; Mathiyazhagan and Ganapathi, 2011; Suranchai and Gumpon, 2011 and; Hassan, Chopade and Vinjamur, 2013).

The use of homogenous catalyst in producing biodiesel using either edible or non-edible feedstock is a popular practice. However, the resulting biodiesel causes severe engine deposits, injector coking and piston ring sticking in compression engines due to high viscosity, low volatility, high cetane number, poor atomization and auto-oxidization (Khalizani and Khalisanni, 2011). These drawbacks of the homogenous catalyst in biodiesel production brought about the development of heterogeneous catalysts. These exhibit less corrosive character due to ease of removal from bulk products and do not leave neutralization salt in the glycerol (Rubi *et al.,* 2011).Apart from the operational problems of homogenous catalyst based biodiesel and subsequent advantages that accompanied the emergence of heterogeneous process, many previous researches on biodiesel have been based on edible oil that resulted in the undesirable concept of competition with food (Suranchai and Gumpon, 2011).

Some of these previous researches include the conversion of palm kernel oil to biodiesel (Alamu, Waheed and Jekayinfa, 2007a), soybean oil (Hossain, Nasrulhaq, Boyce, Salleh and Chandran, 2010). Alternate feed stocks found in non-edible oils are rapeseed oil, tung oil, jatropha oil (Abebe, Yohannes and Rolando, 2011) and neem seed oil (Anya, Nwobia and Ofoegbu, 2012) which are also characterized by high FFA (Rubi, Sandra, Martínez and Reyna, 2011). Despite various feats in the biodiesel research, therefore, the evaluation of the KOH and NaOH embedded bentonite clay as heterogeneous catalyst and optimization of the process variables in order to improve the production of biodiesel through transesterification process using castor oil is the focus of this study.

2. MATERIAL AND METHODS

2.1 Collection and Pretreatment of Castor Seeds

Castor seeds were obtained from Masifa village, Ogbomoso, Nigeria (latitude 8.1333 ^{o}N and longitude 4.2500 ^{o}E) and prepared according to the method of Roetheli, Glaser and Brigham (1991). The seeds were sorted, cleaned, decorticated and boiled at 80°C under air tight conditions for twenty minutes to further reduce the moisture content and thereafter the seeds were grinded mechanically into powdered form.

2.2 Hot Batch Extraction Procedure

The hot continuous extraction process was carried out according to Akpan, Jimoh and. Mohammed, 2006 using soxhlet extractor at a temperature of 110°C for 210 minutes using methanol as the solvent because being polar in nature. Crushed castor seed powder of 20 g was placed in a porous bag made of strong filter paper, which was in turn placed in chamber of the Soxhlet apparatus with 100 ml of methanol. The percentage yield of the extracted oil was calculated according to equation 1.

$$Y(\%) = \frac{W_2}{W_1} x100 \qquad\qquad 1$$

2.2.1 Degumming Process

The extracted oil was degummed by the addition of boiling water (Kenneth and Robert, 1993). The mixture was stirred for 2 minutes and allowed to stand in the separating funnel. Thereafter, the aqueous layer was removed. The procedure was repeated to ensure removal of most gums.

2.3 Catalyst preparation

2.3.1 Impregnation of base accelerators

Impregnation of base accelerators was prepared according to Soetaredjo, Ayucitra, Ismadji and Maukar, (2011), whereby the raw bentonite clay was impregnated with NaOH, and KOH using wet impregnation method in a 500 ml beaker placed on a magnetic stirrer. Impregnation ratios of alkaline to bentonite clay prepared were 1:10, 1:5, 1:4, 1:3, 1:2 and 1:1 based on Equation 2

Impregnation ratio of alkaline to bentonite clay $= \dfrac{Mass\ of\ alkaline\ (g)}{mass\ of\ bentonite\ clay}$ 2

This process was conducted at a temperature of 60 ^{o}C for 24 hr under continuous stirring. After which the slurry was air dried and then oven dried at 110 ^{o}C for 24 hr to remove water content.

To produce the alkaline impregnated activated carbon with ratio 1:10, 1 g of alkaline was weighted and dissolved in 60 ml deionized water. Then, 10 grams of bentonite clay was added into the alkaline solution. After that, the mixture was placed on a magnetic stirrer and stirred for 60 minutes at ambient temperature. After 60 minutes, the solution was filtered and the impregnated bentonite clay was dried at 100 ^{o}C until completely dry. The prepared impregnated bentonite clay was labeled and kept for further use and analysis.

2.3.2 Preparation of heterogeneous catalyst

Potassium methoxide was prepared according to Manut and Satit (2007) method, whereby 100 g of methanol was weighed into a plastic container through funnel and 10 g of potassium hydroxide impregnated catalyst was carefully added using another funnel. The mixture was reflux at 80 ^{o}C for 1 hour for methanol to be completely dissolved. The final mixture was centrifuged at 800 rpm for 10 minutes and the supernatant – that is excess methanol was filtered and the catalyst was separated from the remaining residue, the catalyst was then activated in the furnace under 400 ^{o}C of temperature for duration of 5 hours which was used for transesterification process.

3. EXPERIMENTAL DESIGN

The effects of reaction time, temperature, agitation speed, mole ratio of alcohol and different ratios of catalyst concentration were studied in the production of biodiesel in this research work according to Mathiyazhagan and Ganapathi, (2011). This was accomplished using the Box-Behnken factorial experimental design of Response Surface Methodology (RSM). The lower and upper levels of the factors used in the design are as shown on Table 1.

3.1 Batch process of biodiesel production

Transesterification method employed by Endalew, Yohannes and Rolando (2011) was adopted here for biodiesel production, using a three – necked round bottom flask glass reactor system as a batch reactor on a magnetic stirrer. The condenser was fixed at the center of the reactor; the agitator was placed inside the flask and the thermometer at the left hand side. For a particular run, 100 g of methanol and 10 g of calcined catalyst were first mixed and refluxed at 50 ^{o}C for 1 hr as the catalyst activation step. Then, 250 g of refined castor oil was added to transesterify for a stated experimental duration stipulated by design matrix as shown on Table 3.2. The reactor was heated to 65 ^{o}C temperatures, and then magnetic stirrer was switched on at 1000 rpm on after the 65 ^{o}C temperatures was attained and the reaction was allowed to take place for 3hr in order to obtain a clear phase separation.

The mixture was then filtered to remove catalyst particles; the clear solution obtained was then separated using separating funnel and the yield of biodiesel produced was calculated based on the principle of equation 3.1. All other experimental runs were performed in the same manner according to Table.2.

Table 1: Experimental range of independent variables and their limits

Independent variables	Lower Limit	Upper Limit
Reaction time (Hr)	1	3
Reacttion temperature (°C)	40	65
Catalyst concentration (wt %)	0.3	2
Methanol:Oil	3	9
Agitation rate (rpm)	800	1200

Table 2: Box Behnken Experimental Design for transeterification process

Std	Run	Reaction Time (Hr)	Reaction Temp. (oC)	Catalyst Conc. (g)	Methanol:Oil	Agitation rate (rpm)	Yield (Wt %)
9	1	2	40	1.13	6	800	
8	2	2	52.5	2	9	1000	
37	3	2	40	1.13	3	1000	
5	4	2	52.5	0.25	3	1000	
19	5	2	52.5	1.13	3	1200	
10	6	2	65	1.13	6	800	
11	7	2	40	1.13	6	1200	
6	8	2	52.5	2	3	1000	
17	9	2	52.5	1.13	3	800	
35	10	1	52.5	1.13	6	1200	
46	11	2	52.5	1.13	6	1000	
7	12	2	52.5	0.25	9	1000	
16	13	3	52.5	2	6	1000	
21	14	2	40	0.25	6	1000	
18	15	2	52.5	1.13	9	800	
20	16	2	52.5	1.13	9	1200	
26	17	3	52.5	1.13	3	1000	
43	18	2	52.5	1.13	6	1000	
33	19	1	52.5	1.13	6	800	
28	20	3	52.5	1.13	9	1000	
12	21	2	65	1.13	6	1200	
23	22	2	40	2	6	1000	
31	23	2	52.5	0.25	6	1200	
38	24	2	65	1.13	3	1000	
42	25	2	52.5	1.13	6	1000	
39	26	2	40	1.13	9	1000	
30	27	2	52.5	2	6	800	
2	28	3	40	1.13	6	1000	
22	29	2	65	0.25	6	1000	
34	30	3	52.5	1.13	6	800	
44	31	2	52.5	1.13	6	1000	
15	32	1	52.5	2	6	1000	
14	33	3	52.5	0.25	6	1000	
32	34	2	52.5	2	6	1200	
41	35	2	52.5	1.13	6	1000	
40	36	2	65	1.13	9	1000	
27	37	1	52.5	1.13	9	1000	
36	38	3	52.5	1.13	6	1200	
29	39	2	52.5	0.25	6	800	
24	40	2	65	2	6	1000	
25	41	1	52.5	1.13	3	1000	
3	42	1	65	1.13	6	1000	
1	43	1	40	1.13	6	1000	
4	44	3	65	1.13	6	1000	
13	45	1	52.5	0.25	6	1000	
45	46	2	52.5	1.13	6	1000	

4. **RESULTS AND DISCUSSION**

4.1 Effects of Various Impregnation Ratios and Types on Biodiesel Yield

The effects of two levels of bentonite impregnation (1:5 and 1:10) using NaOH and KOH combined with the variation of each of reaction time, reaction temperature, catalyst dose, methanol: oil ratio and agitation speed on the biodiesel yield in a batch reaction is as presented on Figures 1 – 5.

4.2 Effect of reaction time on biodiesel yield

The effect of reaction time on the biodiesel yield while other parameters were kept constant is as presented on Figure 1. The yield of biodiesel was found to increase with the reaction time for the four types of solid catalyst used. The effect of the KOH impregnated catalyst (1:5) was the highest and then followed in this order: NaOH (1:5), KOH (1:10) and NaOH (1:10). As pointed out by Xie and Li (2006), the product yield would increase steadily with the time until the full conversion is reached and then remained nearly constant thereafter. It is thus deduced from this study the KOH impregnated catalyst at ration 1:5 was the best in terms of biodiesel yield.

4.3 Effect of reaction temperature on the yield of biodiesel

The yield of biodiesel was found to be strongly influenced by the reaction temperature as shown on Figure 2. Generally, the reaction was conducted at atmospheric pressure. Here, biodiesel yield increased as the temperature increased. Transesterification can occur at different temperatures, depending on the oil used. Higher reaction temperature increases reaction rate and shortened the reaction time due to reduction in viscosity of oil (Mathiyazhagan and Ganapathi, 2011). Transesterification occurred at temperature below the boiling point of methanol (65^oC) which informed the choice of temperature range between 40 and 65^oC for this study. This is in line with the findings of Leung and Guo, (2006), Ma *et. al.* (1999) and Freedman *et. al.* (1984) that specified the range of optimal transesterification temperature between $50\ ^oC$ to $60\ ^oC$ depending upon the types of oils and fats used. The KOH impregnated solid catalyst gave the highest yield of biodiesel as temperature increased in similar manner to the effect of time.

4.4 Effect of catalyst concentration on biodiesel yield

Biodiesel production is affected by the amount of catalyst used in the reactions. For homogenous catalyst, biodiesel production is carried out using approximately 1 % catalyst (w/w). However, for heterogeneous catalyst the amount of catalyst used depends on the type of solid catalyst and the type of oil. Here, at constant reaction time of two (2) hours, reaction temperature of 40^oC, methanol: oil ratio of 6:1 and agitation rate of 1000 rpm, the results obtained from the experiment show that as catalyst concentration increased so also the biodiesel yield as shown on Figure 3. The experiment was carried out using 0.25-2.0 wt. % catalyst concentration of bentonite clay, maximum conversion was obtained at 0.29 wt. % of catalyst and $59\ ^oC$ temperature for KOH (1:5) impregnated catalyst . This result shows a similar trend with the work of Leung and Guo (2005), Garcia, Teixeira, Marciniuk and Schuchardt (2008).

4.5 Effect of methanol to oil ratio on biodiesel yield

Ratio of alcohol to oil is another important variable affecting the yield of biodiesel. Stoichiometrically, ratio for the transesterification requires three moles of alcohol and one mole of triglyceride to yield three moles of fatty acid ester and one mole of glycerol. However, in transesterification reaction a large excess alcohol is required to drive the reaction in favour of the product, as higher molar ratio of alcohol to vegetable oil causes an increase in the solubility of glycerol in methyl ester layer. In this study, constant reaction time of three hours, reaction temperature of $59\ ^oC$, and catalyst concentration of 0.29wt. % and agitation rate of 800 rpm, it could be seen from Figure 4 that biodiesel yield increased with increased methanol oil ratio, that is, as the molar ratio increase so also the yield of biodiesel and here maximum conversion was achieved at ratio 8:1.

As pointed out by Encinar *et al.* (2005) higher methanol/oil molar ratio requires longer time for conversion and this explains the reason for the 3 hours reaction time. Comparatively, the KOH (1:5) impregnated catalyst gave the best biodiesel yield out of the four ratios considered.

4.6 Effect of agitation rate on biodiesel yield

Rate of mixing of the biodiesel reactants is very important in transesterification reaction, since the reactants initially were in three-phase system. The reaction is diffusion-controlled and poor diffusion between the phases results in a slow rate of reaction. The effect of the agitation rate is as presented in Figure.5. The biodiesel yield increased with increased agitation and the maximum yield was obtained using KOH (1:5) impregnated catalyst. This result is in agreement with Encinar, Juan, Gonzalez, Rodriguez-Reinares (2005). The mixing effect is significant during the slow reaction rate region and the increase in mixing rate improves the mass transfer in the surface of the solid catalyst. As the single phase is established, mixing becomes insignificant.

Table 3: Summary of some important analytical factors of both base accelerators

Catalyst	Ratio	Lack of Fit	F- value	C.V.	R^2	Adj. R^2
NaOH						
	1:5	6.53	21.38	10.33	0.9145	0.8717
	1:10	2.04	16.64	8.69	0.8927	0.8391
KOH						
	1:5	43.81	52.19	4.83	0.9766	0.9579
	1:10	17.60	28.91	6.42	0.9586	0.9254

4.7 Analysis of Variance (ANOVA) for Response Surface Model

The Model F-value of 21.38 and 16.64 of ratios 1:5 and 1:10 NaOH, then 52.19 and 28.91 of ratios 1:5 and 1:10 KOH respectively (table 3) imply the models are significant. There are only 0.01% chance that a "Model F-Value" this large could occur due to noise.

Lack of fit of 6.53 and 2.04 of ratios 1:5 and 1:10 NaOH and then 43.81 and 17.60 of KOH respectively, is the weighted sum of the squared deviation between the mean response at each parameter level, and the corresponding fitted value. Here, these show there were no significance relative to a pure error in both ratios (i.e. non- significant lack to fit is good). At the other end, the low value of the coefficient of variation 10.33 and 8.69 for both ratios 1:5 and 1:10 of NaOH and 4.83 and 6.42 of ratios 1:5 and 1:10 KOH respectively indicated that the results of the fitted model are reliable.

The quality of model developed could be evaluated using correlation coefficient. The coefficient of determination (R^2) of ratio 1:5 and 1:10 NaOH gave 91.45% and 89.27% then ratio 1:5 and 1:10 KOH yielded 97.66% and 95.86% respectively, these show that the experiment data confirmed compatibility with the predicted data of the model (R^2) which is always between 0 and 1 and its magnitude indicates the aptness of the model, (from a good statistical model, the R^2 value should be close to 1.0). Adjusted coefficient of determination value reconstructs the expression with all the significant terms included. Here, the values of regression coefficients R^2 = 0.8927 and 0.9145 and adj. R^2 of 0.8391 and 0.8717 for ratios 1:5 and 1:10 NaOH then R^2 = 0.9766 and 0.9586 with adj. R^2 of 0.9579 and 0.9254 for ratios 1:5 and 1:10 KOH show high correlation between the experimentally observed and predicted values and give details of any variability in the response.
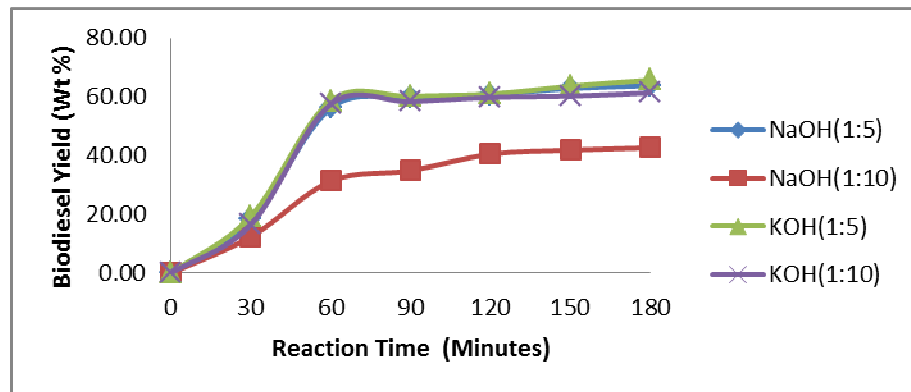
Figure 1: Effects of Reaction Time and Type of Solid Catalyst on Biodiesel Yield (at 59°C, 0.29g catalyst dose, methanol: oil ratio (8:1) and 800rpm)
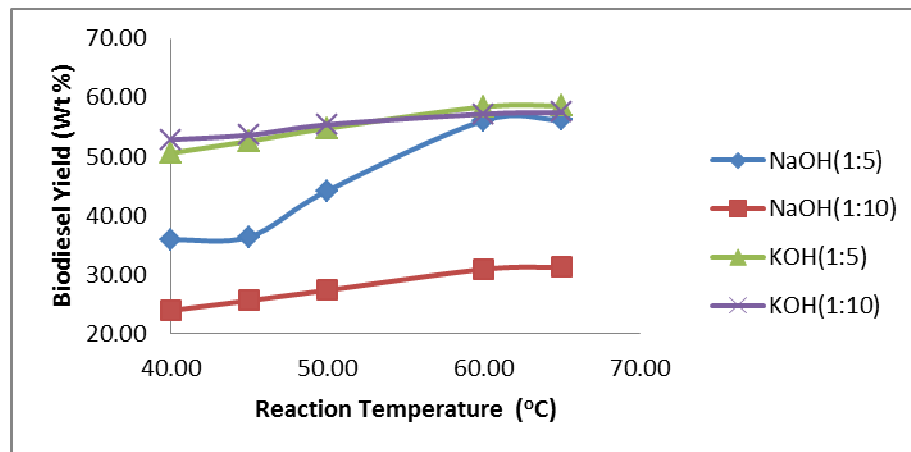

Figure .2: Effects of Reaction Temperature and Type of Solid Catalyst on Biodiesel Yield (at 60 minutes, 0.29g catalyst dose, methanol: oil ratio (8:1) and 800 rpm).
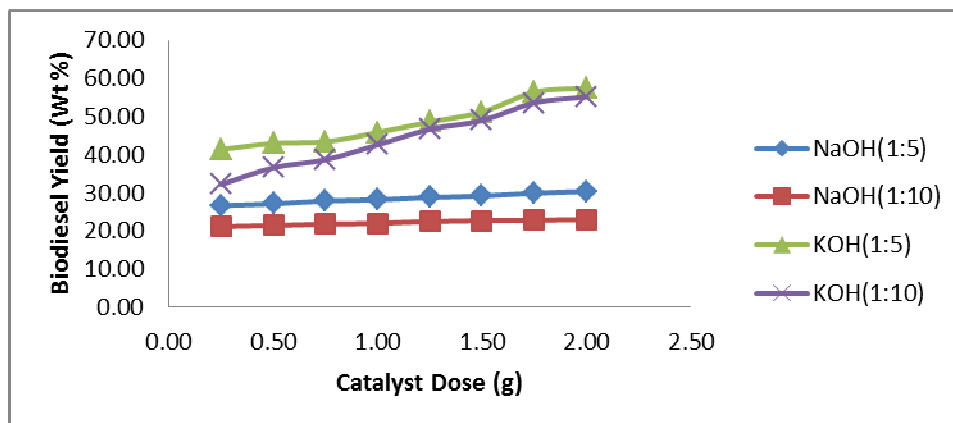

Figure 3: Effects of Catalyst Dose and Type of Solid Catalyst on Biodiesel Yield (at 60 minutes, 59 °C methanol: oil ratio (8:1) and 800 rpm).
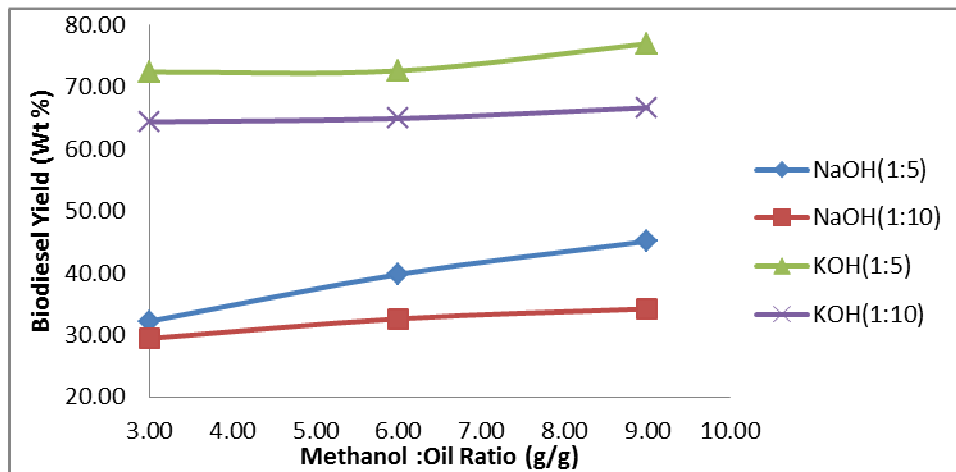
Figure 4: Effects of Methanol: Oil Ratio and Type of Solid Catalyst on Biodiesel Yield (at 60 minutes, 59 ^{o}C, 0.29g Catalyst Dose and 800 rpm).
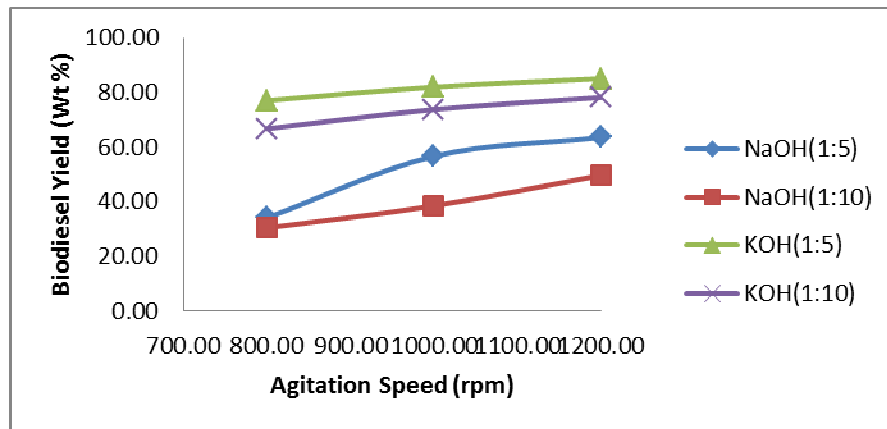

Figure 3.5: Effects of Agitation Speed and Type of Solid Catalyst on Biodiesel Yield (at 60 minutes, 59 ^{o}C, 0.29 g Catalyst Dose and Methanol :Oil ratio (8:1)).

5. CONCLUSION

In this work, different ratios of NaOH and KOH embedded bentonite clay as heterogeneous catalyst and optimization of the process variables in order to improve the production of biodiesel through transesterification process using castor oil were investigated. The results show that the response surface methodology and Box-Behnken design are useful in order to determine the effect of the reaction time, temperature catalyst loading, mole ratio and agitation speed on the transesterification reaction of Castor seed oil. This allowed finding the optimal values at which the process variables maximizing the yield of biodiesel. The yield of biodiesel was found to increase with the reaction time, temperature, catalyst loading, methanol to oil ratio and agitation speed for the four types of solid impregnated catalyst used. Transesterification occurred at temperature below the boiling point of methanol (65°C) which informed the choice of temperature range between 40 and 65°C for this study. Eventually, 1:5 KOH embedded bentonite clay catalyst produced biodiesel of higher yield than the NaOH with 95 % conversions.

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