

## The Effects of Process Variables on Biodiesel Production from Various Catalyst Types

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### ABSTRACT

This research work investigated the effects process variables involved in biodiesel production has on the rate of conversion taking different catalyst types into consideration. Castor seed oil was used as feed stock. The characterization of the castor seed oil was done using American Society for Testing and Materials (ASTM). The castor seed oil was then mixed with methanol and four catalysts (NaOH, KOH, H<sub>2</sub>SO<sub>4</sub>, and H<sub>2</sub>PO<sub>4</sub>) to undergo a transesterification reaction separately. The transesterification reactions were repeated with varying catalyst weight, oil to methanol ratio, reaction time and reaction temperature. High-Performance Liquid Chromatography (HPLC) was used to analyze the various biodiesel samples produced to identify the level of conversion to methyl ester and also identify the compositions of the fatty acid methyl esters (FAMES). Optimal value of each of the reaction with different catalyst show that the optimal reaction conditions was obtained at a temperature of 333k, Molar Ratio of 8, Catalyst weight of 3kg and a maximum reaction time of 97mins, with KOH catalyst having the highest conversion 95.42%, followed by NaOH catalyst with a conversion of 93.77%. H<sub>2</sub>PO<sub>4</sub> catalyst gave a conversion of 62.46%, while H<sub>2</sub>SO<sub>4</sub> catalyst gave the lowest conversion of 57.77%. This research work has shown that reaction parameters combination determines the rate of conversion in biodiesel production not minding the catalyst type used. It also defined the optimum parameter conditions for different catalyst types.

**KEYWORDS:** High-Performance Liquid Chromatography (HPLC), Transesterification, Fatty acid methyl esters (FAME)

### I. INTRODUCTION

Among the biofuels, biodiesel seems to be at the forefront because of its environmental credentials such as renewability, biodegradability and clean combustion behavior [4]. The vegetable-oil derivative 'biodiesel' offers several advantages

as an alternative fuel for diesel engines. These include improved fuel performance and lubricity, a higher octane rating than petro-diesel [4], [9], [3]. It is also a local renewable source of energy and highly biodegradable [6]. The use of castor seed oil as a feed stock for biodiesel production has gained a lot of attention due to several advantages attached to its usage and characteristics. Apart from the fact that castor seed plant is a native to Africa, it's a vegetable oil plant with generally high oil yield of above 35%, it's also one of the major non-edible oil that grows fast with lots of varieties of species and a high unsaturated fatty acid composition [8].

Lots of studies and researches have been done on biodiesel production from various catalysts and the outcome has been a subject of concern since there tend to be inconsistencies in the out puts. It can be seen from the outputs that the effects of the different catalyst types on transesterification process has actually not been considered simultaneously likewise the effect of the reaction parameters on different catalyst types reaction under same reaction conditions have also not been properly looked into.

### II. MATERIALS AND METHODS

Castor seed used in this research work was obtained from PlateauStateNigeria, West Africa. It was then processed and the castor seed oil extracted using Kemtech America synthware Soxhlet Extractor 40MM ID before pre-treatment with NaOH.

#### Fatty Acid Profile and Physiochemical analysis of Castor Oil Feed Stock

The Fatty acid profile was determined using Gas chromatography; Shimadzu Gas Chromatograph-Mass Spectrometer (GCMS-QP 2010 Plus) with a flameionizationdetector(FID) while the physiochemical properties of the pre-treated castor seed oil was determined following standard methods.

#### Transesterification of the Pretreated Castor Seed Oil Sample

Four different catalyst was used namely: Sodium hydroxide (NaOH), Potassium hydroxide (KOH),

Sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) and Phosphoric acid (H<sub>2</sub>PO<sub>4</sub>). 200ml of castor seed oil and 40ml of methanol (i.e. 20% by volume of oil) were utilized in the batch production. 200ml of castor seed oil was pre-heated to a steady temperature of 60°C using a magnetic heater/stirrer. With the aid of the measuring cylinder 40ml of methanol was measured and poured into the beaker. 0.5g of NaOH pellet was measured using the weighing balance and added to the methanol. The content of the beaker was stirred vigorously using the second magnetic stirrer until the NaOH was completely dissolved in the methanol. The mixture formed is called sodium Methoxide. The Methoxide was poured into the conical flask containing the preheated oil. The content of the conical flask was stirred with the magnetic stirrer at a steady speed and temperature of 55°C. Then heating and stirring was stopped after 2.5 hours and the product was poured into a separating funnel mounted on a clamp stand. The mixture was allowed to settle down for about 20 hours. The separating funnel was opened at the bottom allowing the glycerin at the bottom to be run off after

which the biodiesel was collected in a beaker and poured into a container for storage. The experimental procedure was repeated for the three other catalyst types at different varying temperatures, reaction time, catalyst weight and molar ratio of methanol and the FAME produced purified with warm distilled water and dried. The biodiesel produced was also analyzed using HPLC equipment (Agilent 120 series MWD) to determine the composition by percentage of FAME in each sample and subsequently characterized using standard methods

### III. RESULTS AND DISCUSSION

The results obtained from the characterizations of the raw and pre-treated castor seed oil sample are presented in Tables 1 & 2. The physiochemical characteristic (Table 1) identified the crude castor seed oil as pale straw colour oil, which is in line with an earlier study [5]. The fatty acid profile (Table 2) revealed that Ricinoleic acid was higher in percentage composition, with 89.5% composition in the oil sample. This agrees with an earlier investigation that puts Ricinoleic acid as the major fatty acid content in castor seed oil [7].

**Table 1: Result of Physiochemical Analysis of castor seed oil feed stock**

CHARACTERISTICS	Crude castor oil	Pretreated castor oil	Units
Appearance @ 25°C	-	Pass	-
Odour	-	Pass	-
Free Fatty Acid %	5.42	0.985	%
Specific Gravity @ 25°C	0.963	0.961	-
Saponification value	-	180.08	mgKOHg
Iodine value	-	86	gI <sub>2</sub> /100g
Calorific value	-	40.76	MJ/kg
Acid value	10.84	1.97	mgKOHg
viscosity @ 40°C (St/dpas)	-	9.5	mm <sup>2</sup> /s

**Table 2: Fatty Acid Profile of castor seed oil sample feed stock**

FATY ACID NAME	ACTUAL %
Ricinoleic Acid	89.5
Oleic Acid	5
Linolenic acid	3.5
&- linolenic Acid	0.5
Stearic Acid	0.4
Palmitic Acid	0.5
Dihydroxystearic Acid	0.3
Others (unknown)	0.3

**Results of Characterization of Biodiesel Samples**

The properties of the biodiesels presented in table 3 are in line with a prior investigation and studies by known researchers [1]

The physiochemical analysis in table 3 revealed that biodiesel from H<sub>2</sub>PO<sub>4</sub> catalyst has the highest calorific value while biodiesel from H<sub>2</sub>SO<sub>4</sub>

has more water content. The analysis result also showed that the water content in biodiesel is very high when compared to fossil diesel fuel not minding the high flash point and cetane number of the biodiesel. These are similar to previous research findings [2].

**Table 3: Result of Physiochemical Analysis of different biodiesels samples compared to petro-diesel**

CHARACTERISTICS	UNITS	SAMPLE CATALYST				REFERENCE (ASTM/ISO)	FOSSIL DIESEL FUEL
		KOH	NaOH	H <sub>2</sub> S O <sub>4</sub>	H <sub>2</sub> PO <sub>4</sub>		
FAME	%	95	93	57.7	62.4		
Density	Kg/m <sup>3</sup>	887	892	865	867	860-900	848
Viscosity	mm <sup>2</sup> /s	3.8	3.8	3.9	4.1	3.5-5.0	2.37
Flash Point	°C	128	132	145	140	120-130	70
Pour Point	°C	- 12	-12	-11	- 8	-15 to 10	-25 to -15
Calorific Value	MJ/kg	41.64	41.9	41.6	41.89	42	
Cetane Number		49	49.4	49	48	51min	41
Water content	mg/kg	350	360	400	370	500max	26.2
Acid value	mgKOH/g	0.2	0.25	0.41	0.42	0.50max	0.002
Iodine Value	gI <sub>2</sub> /100g	90	88	99	91	120max	

**Effects of process variables on conversion for different catalyst types**

The tables 4 – 7 below show results experimental results of the conversion of FAME for the different catalyst types as a result of changes in reaction parameters.

**Table 4. Effects of temperature on conversion off all the catalyst types**

Temp(K)	CONVERSION (%)			
	Cat A	Cat B	Cat C	Cat D
316	63.26	60.63	46.19	41.85
323	95.39	92.56	55.87	53.02
333	95.42	93.77	62.46	57.77
343	76.47	70.47	48.06	45.41
350	94.46	91.57	48.2	50.23

Cat A=KOH, Cat B=NaOH, Cat C=H<sub>2</sub>PO<sub>4</sub>, Cat D= H<sub>2</sub>SO<sub>4</sub>

**Table 5. Effects of Molar Ratio on conversion off all the catalyst types**

Molar Ratio	CONVERSION (%)			
	Cat A	Cat B	Cat C	Cat D
1	86.72	79.28	49.13	45.58
4	95.39	92.56	55.87	53.02
8	95.42	93.77	62.46	57.77
12	94.63	92.87	54.2	52.47
15	95.19	93.24	49.13	52.09

Cat A=KOH, Cat B=NaOH, Cat C=H<sub>2</sub>PO<sub>4</sub>, Cat D= H<sub>2</sub>SO<sub>4</sub>

**Table 6. Effects of Catalyst weight on conversion off all the catalyst types**

Catalyst weight (%)	CONVERSION (%)			
	Cat A	Cat B	Cat C	Cat D
0.35	62.5	57	44.58	40.73
1	89.22	83.22	49.99	47.02
3	95.42	93.77	62.46	57.77
5	95.39	92.56	55.87	53.02
6.36	71.89	74.86	48.46	44.45

Cat A=KOH, Cat B=NaOH, Cat C=H<sub>2</sub>PO<sub>4</sub>, Cat D= H<sub>2</sub>SO<sub>4</sub>

**Table 7. Effects of Reaction time on conversion off all the catalyst types**

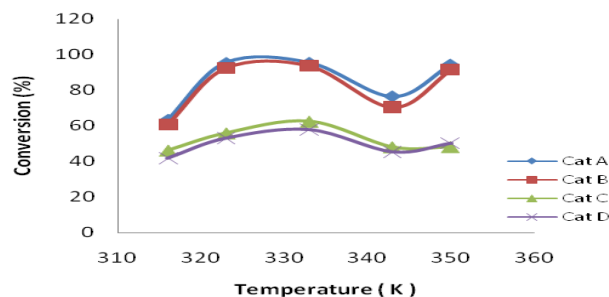
Reaction Time(min)	CONVERSION %			
	Cat A	Cat B	Cat C	Cat D
13	87.15	81.74	49.57	45.78
30	95.39	92.56	55.87	53.02
55	95.19	93.24	49.13	52.09
80	94.63	92.87	54.2	52.47
97	95.42	93.77	62.46	57.77

Cat A=KOH, Cat B=NaOH, Cat C=H<sub>2</sub>PO<sub>4</sub>, Cat D= H<sub>2</sub>SO<sub>4</sub>

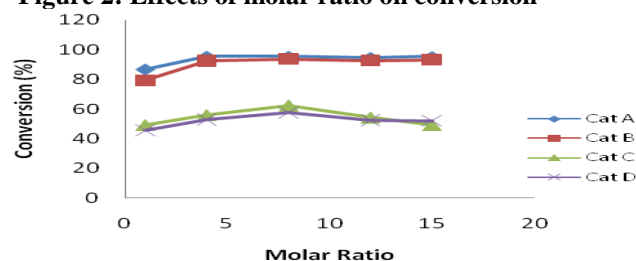
Figure 1 – 4 shows how each of the process variables affect the conversion with respect to each catalyst types while Cat. A, Cat. B, Cat. C and Cat. D represents KOH, NaOH, H<sub>2</sub>PO<sub>4</sub> and H<sub>2</sub>SO<sub>4</sub> catalyzed reactions respectively. Figure 1 shows high decline in conversion after the optimum temperature of 333.15k for all the catalyst types although with little stability in the acid catalyzed reaction, this could be as a result of the fact that high temperature tends to favour acid catalyzed reactions. Figure 2 shows the response of the catalyst types to change in molar ratio which shows no significant changes for the alkaline catalyzed reactions after the initial slight increase in conversion percentage.

Figure 4 illustrates the effect of catalyst weight on conversion for all the catalyst types, this show a gradual decline in conversion for all the catalyst types as the percentage catalyst weight increases after the optimal level. Finally figure 5 shows the effect of reaction time on conversion for all the catalyst types. Although the conversion percentage is higher for alkaline catalyzed reaction at all the time, the conversion for alkaline catalyzed reactions tend to maintain a steady rate as time increases after the initial sharp increase within the first 30mins, whereas a higher reaction time tends to favour the acid catalyzed reactions as it tends to increase at higher reaction time above 55mins.

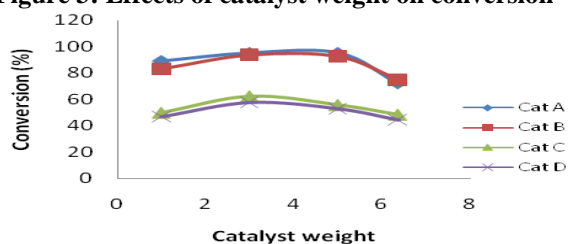
**Figure 1: Effects of temperature on conversion**



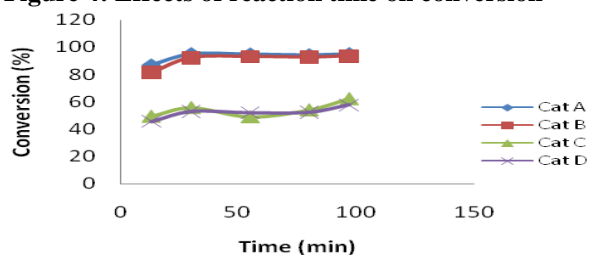
**Figure 2: Effects of molar ratio on conversion**



**Figure 3: Effects of catalyst weight on conversion**



**Figure 4: Effects of reaction time on conversion**



#### IV. CONCLUSION

The production of biodiesel from castor seed oil was achieved successfully with different level of conversion which is dependent on the level of interaction of the reaction parameters involved in the reaction. These shows that the rate of interaction of reaction parameters is a major determinant of the FAME produced not minding the type of catalyst used

It can also be said that certain catalyst types are favoured by certain process variables and an increase conversion can be achieved by manipulating such various to its advantage.

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