

SYNTHESIS OF BIODIESEL FROM ANACARDIUM OCCIDENTALE (CASHEW) NUT SEED USING KI/AL₂O₃ CATALYZED IN-SITU TRANS ESTERIFICATION PROCESS

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ABSTRACT

In this research, biodiesel was produced from *Anacardium occidentale* seed with the aid of KI/Al₂O₃ catalyzes and in-situ trans esterification process. The biodiesel produced was analyzed using different instruments namely; FT-IR, X-ray diffraction (XRD), X-ray fluorescence (XRF) and Hammett basicity techniques. The Optimization of the reaction process was also carried out using respond Surface Method (RSM). The empirical model was obtained and the statistical significance of the reaction variables ($R^2=74.36\%$). The optimum yield of biodiesel (97.75%) was achieved under the optimum reaction conditions of 0.8 wt of KI/Al₂O₃, 1:6 methanol to seed ratio, 2hrs of reaction time and 80°C of temperature. The result of characterization and its physiochemical properties of biodiesel were in line with the ASTM specification for diesel.

Keywords: Anacardium occidentale, Biodiesel, trans esterification, optimization,

I.INTRODUCTION

The customarily use of fossil fuels have increasing emissions of combustion that generated hazardous gasses that have health impact on environment and the people [1]. These fossil fuels are usually exhaustible and reserves the concentrated gas in particular places of the earth. The cost and sometime unavailability of this fossil fuel in Nigeria's current petroleum reserves have call for various research interest in renewable energy search [1]. The issue has prompted the global search for alternative energy sources. More importantly, decreasing nature of conventional energy sources combined with the continual destruction of surrounding environment draw a lot of focus for the demand of renewable environment friendly fuel [2]

To boost the nation's economy, a dependable and sustainable energy supply is crucial, and this pressing requirement cannot be disregarded [5]. Merely 11% of the world's overall use of biofuel is derived from agricultural feedstock. Only around 4% of France's total agricultural production area is dedicated to biofuels; the other 96% will be utilized for other crop production industries [6]. As a viable alternative, biodiesel has several advantages over fossil diesel fuel, including lower exhaust emissions, enhanced biodegradability [7], decreased toxicity [8], superior lubrication [9], non-flammability due to its higher flash point [10], increased susceptibility to oxidative degradation [11], easy

availability, and a higher cetane number that can enhance performance and clean emissions [12].

In situ trans esterification (ISTE) is determined by various parameters, including such as the catalyst type and concentration, the reaction time, and the temperature [13]. In additionally, optimizing the reaction process and variables is crucial to achieving the highest biodiesel yield which in turn lowering production costs [14]. Numerous researchers have successfully carried out in situ trans esterification of plant and microalgae seeds using homogeneous alkaline [15] and acidic [16,17], or a combination of both, with high yields [18]. However, there is very little in the literature utilizing heterogeneous catalysts for the in situ trans esterification reaction of *Anacardium Occidentale* (Cashew) seeds. The objective of this research is to Utilizing KI/Al₂O₃ as a catalyst and optimizing the process variables temperature, catalyst, and time the research aims to create biodiesel from cashew oil.

II.MATERIALS AND METHODS

Sample collection and preparation

Cashew seeds were purchased from a farm located at Sokoto North- west of Nigeria. They were cleaned seeds and stored at room temperature for one week before dried in an oven at 105°C to remove residual moisture.

Oil extraction

Using a cellulose cartridge, the powdered sample was weighed (50.0 g) and extracted using Soxhlet process and n-hexane solvent for six hours. After the extraction was complete, the flask was heated to 35°C for 20 minutes in order to evaporate the solvent and retrieve the oil. To calculate the yield, the dried oil was moved to a bottle that had been previously weighed [19]. Equation 1 below was used to calculate the weight of oil of seed powder after repeated extraction:

$$\% \Delta\beta = \pi\alpha \Delta\beta (\pi) \times 100 \text{ -----(1)}$$

Catalyst preparation for KI/Al2O3

the catalyst was prepared using 0.5m of potassium iodide and incubated for three hours then transferred to a flask with a circular bottom.(250 cm3).After three hours of stirring at 700 rpm at 40 oC, the mixture is filtered. To activate and eliminate extra water, the gathered residue was calcined at 400, 600, and 700 °C for three hours at a rate of 10 °C per minute. It was then dried in an oven at 110 °C. In the catalyst, the mass ratio of KI to aluminum was 1:3 [20].

Characterization of Catalysts

The Hammett base test was used to ascertain the catalyst's surface characteristics. The Hammett indicators that were employed were 4-nitroaniline (H=-18.4), 2,4-dinitroamine (H=-15.0), phenolphthalein (H=-9.8), and bromothymol blue (H=-7.2). Non-polar solvents like benzene [21] were used for the measurements. The capacity of a catalyst to change an adsorbed neutral acid (benzoic acid) into its conjugate base form is known as its basic surface resistance, and it is represented by the Hammett basicity function:

$$H - = \log [A-] [] + pKA \quad (2)$$

The Central Laboratory Complex (CASLEC), Usman Danfodiyo University Sokoto, used the KBr pellet technique to evaluate the sample's FTIR spectra. A Carry 630 FT-IR spectrometer was used to record the spectra, which had a resolution of 4 cm-1 and covered the 600 cm-1 to 4000 cm-1 X-ray fluorescence (XRF) of the catalyst that was synthesized (KI/). Thermo Scientific ARL OPTIM'X spectrometer was used to evaluate Al2O3 and raw Al2O3 utilizing wavelength dispersive X-ray fluorescence (WDXRF) techniques. The XRF method displays the percentage of oxides by element.

Response Surface for Experimental Design (Box-Behnken) for optimization, a statistical experimental design was employed. Based on initial investigations, four independent variables were chosen for the study: temperature, methanol/seed ratio, catalyst concentration

(KI/Al2O3), and reaction time [17]. For a total of 54 fully randomized trials, each test was replicated.

Description of the experimental trials

In each experiment, 10 g of cashew nuts were combined with 100 cm3 of n-hexane as a co-solvent for 10 minutes. The resulting slurry was then moved to a cellulose cartridge with a suitable volume (30, 45, or 60 cm3), and in a 250 cm3 round-bottom flask, it was combined with 0.46, 0.92, or 1.38 g of activated KI/Al2O3 catalyst at 60, 70, and 80 °C while being continuously stirred at 600 rpm for 1, 2, or 3 hours. Three phases (upper biodiesel layer, middle glycerol layer, and lower catalyst layer) were formed after the products were moved to a separatory funnel and 10 cm3 of distilled water and 10 cm3 of n-hexane were added for an hour [14].

Data Analysis

The statistical software MINITAB 17 was used to examine the biodiesel yields from an in situ trans esterification experiment in order to assess how the interaction of reaction variables (xi) affected the biodiesel yield (y).A full quadratic function was used to fit the biodiesel yield as a percentage. Regression analysis using a polynomial regression model (Eq.3). Where the intercept, linear, quadratic, and interaction coefficients are denoted by β0, βi, βii, and βi j, respectively. The coefficient of determination (R2) was used to evaluate model fit and analysis of variance (ANOVA) at the 95% confidence level.

Contour plots were drawn on the fitted quadratic polynomial equation obtained from the regression analysis, keeping two independent variables (reaction time, temperature, methanol and catalyst weight) at a constant value while varying the other variables.

$$\pi = \pi0 + 4\pi1\pi0 + 4\pi1\pi0^2 + 4 4\pi1\pi0 \pi1\pi0 - \pi4 \quad (3)$$

Biodiesel dosage $\pi=1\pi=1\pi=1\pi+1$

Biodiesel Assay

Analysis was done on biodiesel made from palm kernel seeds using in situ transesterification. use an Agilent 6890N GC/MS in conjunction with an Agilent 5973MSD.The process known as total ion current (TIC) was applied. It took five minutes to remove all of the produced fractions using solvents. The carrier gas, helium, flowed at a rate of 1.2 cm3/min. The gas chromatography machine had a 30 m long polysilolohexane capillary column with an internal diameter of 20 μm and a film thickness of 1.0 μm. One microliter (1 μl) of the samples was injected into the injection port of the machine at an injection temperature of 250 °C.

The column oven temperature was programmed between 50 and 300 °C at 50 C/min and held at the final temperature for 10 min. The compounds were identified using their respective mass spectra compared with standard mass spectra from the NISTII reference library.

Determination of biodiesel properties

The ASTM 6751D technique was used to determine the characteristics of the biodiesel. Specific gravity, kinematic viscosity, and acid number were calculated using the methods used by Mohibbe et al [22]. The properties like cetane number (CN) and higher heating value (HHV), iodine value (IV) and saponification index were also determined using earlier reported methods[22].

IV.RESULTS AND DISCUSSION

Catalyst characterization

Table 1: Surface Property of catalyst

Indicator	Original colour	Basic colour	Pka	Vol. of Benzoic acid (cm ³)	Basicity (mmol)
Bromothymol Blue	Colourless	Blue	7.2	48.0	0.00758
Phenolphthalein	Colourless	Red	9.3	48.5	0.00968
2,4-dinitroamine	Yellow	Violent	15	48.2	0.01530
4-nitroaniline	Yellow	Orange	18.4	48.3	0.01850

FT-IR-Analysis

The result of the FT-IR analysis of the catalyst (Al₂O₃/KI) is shown in Figure 1. The OH groups that are directly bonded to the aluminum are responsible for the wide band of strong vibrations in the 3400–3480 cm⁻¹ region of the catalyst's spectrum. Similarly, the stretching vibration of the OH groups for water in the solid catalyst may be represented by the broad band at 3480-3500 cm⁻¹ [23]. The large peak at

667 cm⁻¹ is also ascribed to the vibration frequencies of octahedral AlO₆ [24,25] Demonstrates that the primary locations of the aluminum vibration band were 714 cm⁻¹, 633 cm⁻¹, and 567 cm⁻¹.These groups are most likely regarded as the KI/Al₂O₃ catalyst's basic active species or sites. These findings are consistent with those published by [21].

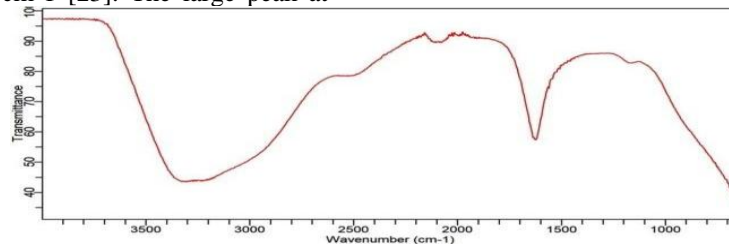


Figure 1 FT-IR Spectra of the catalyst (Al₂O₃/KI)

Table 2 The results of XRF Analysis

Elemental Composition of Crude KI/Al ₂ O ₃		Elemental Composition of Crude Al ₂ O ₃	
OXIDE	CONTENT (%)	OXIDE	CONTENT (%)
Al ₂ O ₃	73.21	Al ₂ O ₃	82.44
CaO	4.09	CaO	3.97
Fe ₂ O ₃	0.19	Fe ₂ O ₃	0.08
K ₂ O	0.86	K ₂ O	0.15
MgO	0.11	MgO	0.09
P ₂ O ₅	ND	P ₂ O ₅	ND
SiO ₂	6.09	SiO ₂	10.86
SO ₃	ND	SO ₃	ND
LOI	2.43	LOI	0.38
SUM	84.55	SUM	97.59

Table 2. Above reveals that the major elemental constituent of the Al_2O_3 and KI/Al_2O_3 are majorly $SiO_2 + Al_2O_3 + Fe_2O_3$ which are $\geq 100\%$. The difference in the LOI between the Al_2O_3 and KI/Al_2O_3 was 2.051

which signify that the physisorption of KI on the surface of the alumina were achieved. This leads to an increase in the % of K_2O from 0.15 to 0.86%.

Design of Experiment

Table 3 Variables in *In-situ* Transesterification and their Levels used in Box-Behnken Design

Independent Variables	Coding	Lower level	Upper level
Temperature, ($^{\circ}C$)	θ	60.0	80.0
Time, (hr)	T	1.00	3.00
Methanol, (cm^3)	M	30.0	60.0
Catalyst concentration, (g)	C	0.46	1.38

Analysis of variance table

Table 4: Result of Analysis of Variance for Cashew Nut Biodiesel Yield (%)

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	15	12932.9	862.20	7.35	0.000
Blocks	1	4.4	4.45	0.04	0.847
Linear	4	4381.9	1095.46	9.34	0.000
Temp. (θ)	1	3649.9	3649.93	31.11	0.000
Time.(hr) (t)	1	16.1	16.10	0.14	0.713
Con. of Methanol.(m)	1	17.8	17.82	0.15	0.699
Mass of Catalyst (C)	1	698.0	698.01	5.95	0.020
Square	4	5459.0	1364.75	11.63	0.000
θ^2	1	1332.3	1332.31	11.36	0.002
t^2	1	240.3	240.33	2.05	0.161
m^2	1	5118.4	5118.37	43.62	0.000
C^2	1	556.2	556.20	4.74	0.036
2-Way Interaction	6	3087.6	514.61	4.39	0.002
θ .* t	1	2779.2	2779.22	23.69	0.000
θ .* m	1	176.7	176.72	1.51	0.227
θ .* C	1	11.0	11.04	0.09	0.761
t * m	1	84.8	84.76	0.72	0.401
t * C	1	35.7	35.74	0.30	0.584
m * C	1	0.1	0.15	0.00	0.972
Error	38	4458.5	117.33		
Lack-of-Fit	34	4388.4	129.07	7.36	0.032
Pure Error	4	70.2	17.55		
Total	53	17391.5			

$R^2=74.36\%$ $R^2(\text{adj}) = 64.24\%$ $R^2(\text{pred})=46.37\%$,Key
 DF= degree of freedom. Seq SS = Sequential Sum of Squares. Adj SS= Adjusted Sum of Squares.
 Adj MS= adjusted mean Squares. $F=f$ -Statistic . $P=p$ -value.

The model's linear, quadratic, and interaction terms are statistically significant ($p < 0.05$, with $\alpha = 0.05$), according to analysis of variance. The model effectively captures the empirical relationship between biodiesel yields and process variables, as evidenced by the cashew-based biodiesel's highest coefficient of determination

(R^2) of 74.36%. All other linear and quadratic terms of process variables are statistically significant ($p < 0.05$). The revised regression model with six significant terms (adjusted $R^2 = 64.24\%$ and $R^2 = 74.36\%$) was therefore produced by removing the non-significant terms from the model. Overall, the empirical model has strong prediction power and is a better depiction of the in situ transesterification process.

$$\% \text{ Yield of Biodiesel} = -426 + 11.98 \theta - + 60.6 C - 0.1118\theta^2 - 0.0974 m^2 - 34.1C^2 + 1.864 \theta$$

Figure 2 and 3 contour plots describing the relationship between one of the reaction variables while holding the other two variables at constant as they affect the biodiesel yield of cashew nut. The combined effect of reaction time and temperature (Figure 2) proves that biodiesel yield increases with reaction time and temperature. At the lowest reaction time and temperature (< 1 h, < 600°C), the biodiesel yield was 55%. The highest yields, above 75%, are obtained with a reaction time of 2 h and a

reaction temperature of 70°C. the methanol volume and catalyst weight also interact positively to affect biodiesel yields. When the methanol volume was 30 cm³ with a maximum catalyst loading of 1.3 g, the biodiesel yield obtained was 50%. The maximum biodiesel yield (>80%) was obtained when the in situ transesterification was carried out under a methanol volume of 50 cm³ to 55 cm³ and a catalyst loading of 0.8 g.

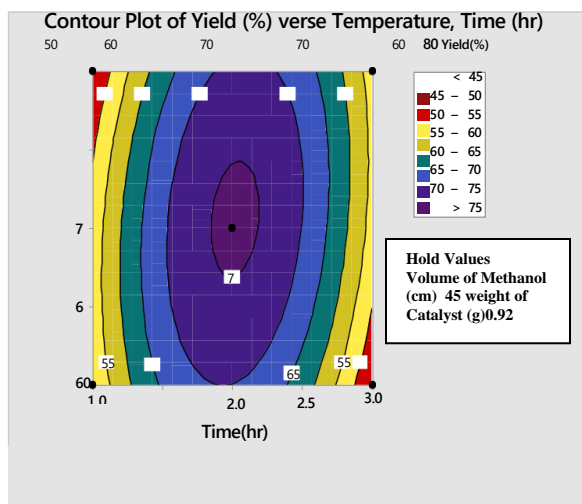


Fig.3

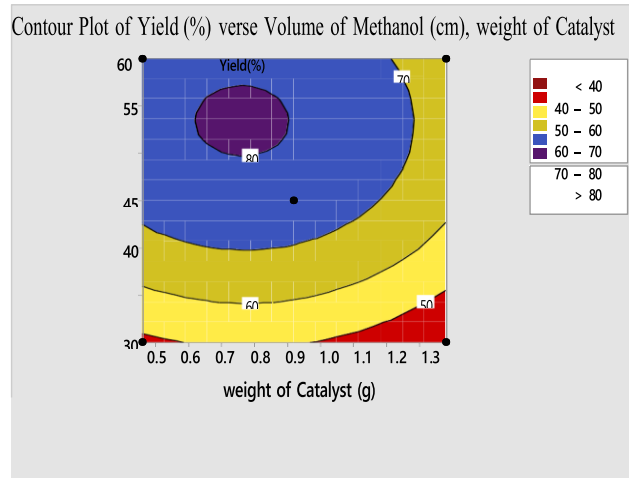


Fig.4

Fig.3 and Fig.3 :contour plots describing the relationship between one of the reaction variables

Table 5: Result of Optimization of Cashew Nut Biodiesel Yield

Solution	MeOH (cm ³)	Temp(°C)	Time(hr)	Catalyst(g)	Yields (%)	Desirability
Solution 1	60	80.000	2.19192	0.81	97.7559	1.00000
Solution 2	60	71.096	2.11006	0.46	77.2013	0.80511
Solution 3	50	80.000	2.94698	0.46	76.8722	0.79949
Global Solution	60	80.000	2.19192	0.81	97.7559	1.00000

From the above result, the most significant difference between the solutions is solution 2 with catalyst loading (0.46 g) which gives a yield of 77. 20%, while increasing the catalyst leads to an increase to 0.813 g of yield from 77.20% to 97. 75%. The decrease in the amount of methanol and the higher reaction time of 2.9 h in (solution 3) lead to a decrease in the percentage yield of biodiesel. The general solution (solution 1) which requires a temperature, a methanol volume, a catalyst weight of 60 cm³, 80 0C, 2 hours and 0.81 g,

respectively, may therefore be interesting for the in situ transesterification of cashews in FEME via. in situ transesterification.

Biodiesel Characterization

FT-IR spectrophotometers and GC-MS were used to identify the biodiesel formation. The FT-IR spectra of all biodiesel samples showed a similar pattern. The FT-IR spectrum of biodiesel was different with triglycerides at a peak area of 1200 cm⁻¹, which is assigned to methoxy functional groups (-OCH₃) as shown in Figure 4.

FT-IR Analysis

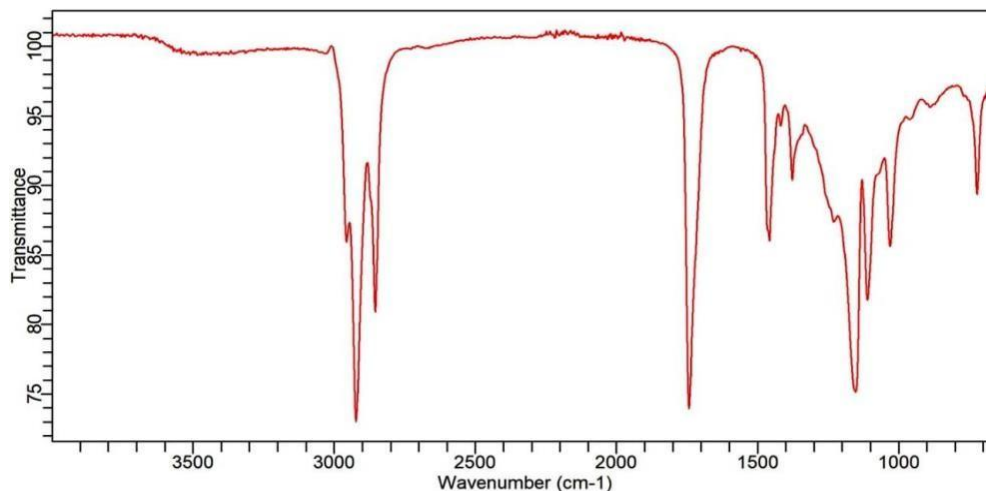


Figure 4: FT-IR Spectra of Biodiesel Sample from *Anacardium Occidentale* (Cashew nut)

GC-MS Analysis

The GC-MS profile of cashew fatty acid methyl ester is dominated by; Methyl hexadecanoate, methyl (Z)-19-octadecanoate, methyl 13-16-octadecanoate, methyl 16-heptadecanoate and oleic acid methyl ester are

produced. From Table 5 above, approximately 99.99% biodiesel conversion was achieved with the catalyst and (Z)-methyl-9-octadecanoate obtained was 69.44%, implying that approximately 69.44% of cashew oil is (Z)-methyl-9-octadecanoic (Z) acid (oleic acid).

Table 6: GC-MS Fatty Acid Methyl Ester Compositions of Cashew nut Biodiesel

Name of fatty esters	Retn Time(Min)	Molecular Formular	Peak Area (%)	(%) of FEME A
Methyl hexadecanoate	19.252	C17H34O2	348528.18	8.3
(Z)-Methyl 9-octadecanoate	24.133	C19H36O2	2915902.02	69.44
13-16-Methyl octadecadiynoate	24.877	C19H30O2	347589.52	8.28
16-methyl heptadecanoate	24.877	C19H38O2	347589.52	8.28
Oleic acid methyl ester	26.839	C19H36O2	239289.03	5.69

Table 7 Physicochemical Properties of Biodiesel

PROPERTIES	CNK FAME	ASTM D6751
Acid value (mg KOH/g)	0.86 ± 0.18	0.8 max
Saponification value (mg KOH/g)	160.44 ± 2.30	—
Specific gravity @ 15°C (g/cm ³)	0.963 ± 0.00	0.827
Kinematic viscosity @ 40°C (mm ² /s)	4.87 ± 0.00	1.90 – 6.00
Cetane Number (min)	61.33 ± 0.00	51min
Higher Heating Value (MJ/Kg)	41.58 ± 0.00	—
Values are means ± SD triplicates analysis of the sample		

The results of the physicochemical properties of biodiesel show that the saponification index obtained is consistent with previous publications; (173.91 mg KOH/g oil) by Ogungbenle and Afolayannew [26] even lower than (205.29 mg KOH/g oil) obtained by Mohibbe, et al [22]. A saponification index below 190 mg/KOH is an indicator of the presence of high molecular weight fatty acids [27].

The kinematic viscosity at 400°C (4.87/4.78 mm²/s) is consistent with previous publications by Onyema et al [27]. The maximum acceptable viscosity limit according to ASTM D6751 range is (1.9-6.0 mm²/s)

V.CONCLUSION

This study revealed that biodiesel could be successfully produced from cashew nuts using the in-situ transesterification process catalyzed by KI/AL₂O₃. The effect of several parameters such as acid value, saponification value, specific gravity, kinematic viscosity, cetane number and high catalyst dosage of heater valve in biodiesel was analyzed. The optimum yield of 97.75% was achieved at a temperature of 80°C, a reaction time of 2 h and a catalyst dosage of 0.8 wt%. Catalyst reuse studies should be carried out to determine the extent of its catalytic activity. and other domestic waste substrates should be considered for the production of calcium oxide catalyst for the esterification reaction of methyl esters in biodiesel.

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