



OPTIMIZATION OF BIODIESEL PRODUCTION FROM *KHAYA SENEGALENSIS* OIL USING HETEROGENEOUS CATALYST

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ABSTRACT

Global concerned for an ecofriendly environment and upholding minimal biodiesel production cost simultaneously has prompted the use of organic wastes as heterogeneous catalyst for its production. In this work, response surface methodology (RSM) was used to optimize the production of biodiesel through a two-step transesterification process. The extracted *Khaya senegalensis* oil (KSO) possessed a high free fatty acid (FFA) value of 3.10 % which was reduced to 0.329 % using H₂SO₄ processed at 60 min reaction time, 60 °C temperature and a methanol to oil ratio of 2.25:1 (mol/mol). A calcined plantain peels (CPP) heterogeneous catalyst produced at 700 °C for 3.5 h was characterized and was found to contain potassium, sodium, calcium, magnesium, iron, manganese and phosphorus which was used for the transesterification to obtain *Khaya senegalensis* oil methyl esters (KSOME). A 97.98 wt% optimal KSOME yield was attained from a central composite design (CCD) using a quadratic model at a methanol to oil ratio of 9:1 (vol/vol), 2.5 (wt%) catalyst loading, 40 min reaction time and 65 °C constant temperature condition which was validated at 95.875 (wt%). KSOME produced met the international ASTM D6751 and DIN EN 14214 biodiesel standard specification hence, it is projected a good prospect for biodiesel production.

Keywords: Optimization, Heterogeneous catalyst, Biodiesel, *Khaya senegalensis* oil

INTRODUCTION

Biodiesel production from biomass and wastes has become a subject of attention for most economies in the global quest for alternative energy sources to argument world energy reserves. Previous works focused on maximizing underutilized non-edible materials in biodiesel production with the view to reducing dependency on fossil fuels and price hike in many regions. Biodiesel apart from being renewable generally have good; lubricity, miscibility with conventional fuels, and low toxicity (Moser, 2009).

Researches were delved on the potentials of some edible oil seeds (Ogunsuyi, 2015; Rachimoallah et al., 2009; Shote et al., 2009) though it has aroused controversy on domestic feedstock versus energy-economic driven development preference (Gui et al., 2008). In this regard, many non-edible oil seeds have been explored (Betiku et al., 2017; Junaid et al., 2014; SandeepKumar et al., 2015; Vivek and Gupta, 2004; Ying-Zhi et al., 2016). The use of used cooking oil (Mohammed and Bhargavi, 2015) have also been reported, while wood ash (Meeta et al., 2012), sea sand (Muciño et al., 2014), plantain peel, (Betiku and Ajala, 2014), shells and bones (Farooq and Ramli, 2015; Roschat et al., 2016) have been documented for heterogeneous catalyst production. Catalyst reusability, non-edibility, cheap production cost, and waste conversion in environmental management are the significant pulls heterogeneous catalyst exacts in biodiesel production (Vicente and Mart, 2004). To strengthen the aforementioned advantages, investigations have been made in optimization study of oil extraction and transesterification yield with different

catalyst through various techniques and designs with the view of industrial profitability. Bhoopendra et al., (2017) reported using Taguchi design, Oyelade et al., (2017) used the D-optimal design, CCD was explored by Alok et al. (2007), and Betiku et al. (2017), while the comparison of Box-Behnken with Artificial neural network and genetic algorithm designs was investigated by Betiku and Ajala (2014) and Etim et al. (2018).

Khaya senegalensis also known as African Mahogany, Ogonwo among the Yoruba's (Orwa, 2009), Madachi for the Hausa's (Blench and Dendo, 2007) is available in good quantity within these zones of Nigeria and is also called Ono in Igbo. This study is focused on determining the best biodiesel production condition from KSO using the CPP catalyst as well as characterizing the KSOME and the catalyst.

MATERIALS AND METHODS

Materials

African mahogany seeds of 2.2kg net weight was obtained from the Forest Research Institute, Ibadan. The plantain peels were obtained from restaurants on Obafemi Awolowo University campus Ile-Ife. All reagents used were analytical grade BDH Chemicals Ltd., Poole England and GFS Chemicals, Inc., Columbus.

Methods

ii *Khaya senegalensis* oil (KSO) extraction



The seeds were decorticated, dried and grinded for oil extraction using the chemical method in 250ml Soxhlet apparatus with n-Hexane. The seed granules were charged in a 24cm diameter thimble and into soxhlet which is fitted on 500ml flat-bottom flask. A condenser was fitted on soxhlet and everything was stand on a water-bath heating mantle whose temperature maintained 70 to 90 °C for a complete setup. The oil was separated from the refluxed n-Hexane after 9 h.

iii Preparation of calcined plantain peels (CPP)

Combination of ripe and over-ripe peels were washed for 5 times with distilled water, chopped into bits, sun dried to crispy and opened-air burnt before grinding into fine power. The powered peel was charged into a furnace (Carbolite – Parsons Lane, Hope Valley, England. Model S33 6RB) for calcination at 700 °C for 3.5 h and CPP obtained was stored in air-tight bottle.

iv Esterification step of KSOME production

This oil pretreatment step was necessary since the initial % FFA of the KSO of 3.10 is higher than the acceptable 1-2 % (Knothe et al., 2005) for good transesterification reaction. A 100g of oil was heated to 50 °C before charging with 225g methanol and 5g H₂SO₄ catalyst was allowed to react for 60 min at 60-65 °C temperature range. The was terminated after 60 min, esters were separated from the mixture after standing for 7 h in a separating funnel. This triplicate reaction yields a 0.329 % FFA and 95.70 % conversion for the transesterification step.

v A rotatable fractional CCD experiment

The transesterification was pursued through a rotatable fractional CCD of 3factor, 5level coded as shown in Table 1 using RSM to generate 15 experimental runs on Design-Expert version 10 (Stat-Ease Inc., Minneapolis, MN, USA) software. Methanol to oil ratio (X₁), catalyst loading (X₂) and reaction time (X₃) were the significant factors of interest as report in literature as well. The percentage yield was predicted from the second-order polynomial quadratic equation (1) to get the optimum model equation (2). Where

$$y = \beta_0 + \sum_{i=1}^k \beta_i X_i + \sum_{i=1}^k \beta_{ii} X_i^2 + \sum_{i < j=2}^k \sum_{j=2}^k \beta_{ij} X_i X_j + \epsilon \quad \text{i.}$$

y is the yield, β_i is the first-order model coefficients of main effects, β_{ii} stands for quadratic coefficients of quadratic effects, β_{ij} is the interactive effects coefficient for interacting effects, ϵ is a random error and i, j, and k are 1,2,3, ..., n.

$$y = 0.8143 + 10.3561X_1 + 29.5997X_2 + 0.9752X_3 - 0.5365X_1X_2 + 0.0366X_1X_3 - 0.2319X_2X_3 - 0.6257X_1^2 - 3.4911X_2^2 - 0.0102X_3^2 \quad \text{ii}$$

vi Optimized KSOME transesterification reaction step

Following the generated design from Table 1, a 12.26 ± 0.1 cm³ volume was held constant for all runs, measured

into a 500ml three-necked round-bottom flask with a corresponding volume of methanol for a run. This mixture was heated to 65 °C before adding the corresponding quantity of catalyst and the time was started with the temperature maintained.

Table 7: Independent factors for the optimized transesterification of KSOME on CCD

FACTOR	UNIT	CODED FACTOR LEVELS				
		-α	-1	0	+1	+α
Methanol : Oil (X ₁)	vol/vol	4.7574	6	9	12	13.2426
Catalyst loading (X ₂)	% wt	1.0858	1.5	2.5	3.5	3.9142
Reaction time (X ₃)	min	11.7157	20	40	60	68.2843

IKA C-MAG HS 4 heating mantle and inbuilt magnetic stirrer at a maintained 1.5 stirring speed was used. At the end of the reaction time the mixture was separated into KSOME, glycerine and CPP phases with a centrifuge at 8000 rpm for 10 min before pouring into a separating funnel to stand for 15 min. KSOME obtained was purified with distilled water of 55 °C to eliminate residues till the water is colourless (Van Gerpen et al., 2004) as shown in Figure 1, dehydration was necessary with sodium sulphate. KSOME yield is therefore calculated with equation (3) and the process repeated for all runs. The run bearing the highest as reflected in Table 2 was used in validation through mass production of KSOME.

$$\% \text{ yield} = \frac{\text{weight of biodiesel}}{\text{weight of oil used}} \times 100\% \quad \text{ii.}$$

vi. Characterization of oil, KSOME and CPP

The vital physical and chemical properties of extracted oil and KSOME were experimentally determined in accordance with the Association of Official Analytical Chemists (AOAC, 1995) standard methods for percentage oil content yield, acid value, iodine value, peroxide value, saponification value, density, surface tension, viscosity. Cloud point was through ASTM D 5773 method, pour point by ASTM D 97-96a method, open cup method of ASTM D 93 for smoke and flash points, ASTM D 613 and ASTM D 1997 method for cetane number and higher heating value respectively. A gram weight of CPP was used in 50 ml³ of solute for elemental composition study using atomic absorption spectroscopy (AAS) method on Aanalyst 400, Perion Eleanor, Singapore 2010 while absorption method was also used in Fourier Transform Infrared (FT-IR) to determine the active functional group of the CPP on Cary 630 Agilent, U.K.

RESULTS AND DISCUSSION

The optimal production result presented in Table 2 indicates agreement with existing literature that the factors investigated in this work are significant production factors, presenting an optimum yield of 97.98 wt.% which was validated under created constraints methanol to oil ratio of 9:1 vol/vol, 2.5 wt.% catalyst loading and 40 min reaction time condition for the factors



yielding 95.875 wt.% for mass production. This met the objective of optimal KSOME yield in the production.



(a)



(b)

Figure 1: (a) Separated phased KSOME and glycerol. (b) Purified KSOME phased with water underneath.

Analysis of the model for KSOME production

ANOVA test result (Table 3) displayed a validation of the chosen model at 95 % confidence level with 0.0012 model p-value, accurately predicted that 0.12 % chances of noise existed at the model implementation. At the same confidence level was obtained 0.2327 p-value insignificant lack of fit, which was desired. In addition, regression equation R^2 of 0.9785 greater than the least expected 0.80 (Joglekar and May, 1987) for a good fit means the model explained 97.85 % response relationships between yields and varying reacting conditions of Table 2 and was in agreement with Chin et al. (2009) result. The residual demonstrates a good relationship between the predicted and actual yield values as strengthened by 1.79 % coefficient of variance (C.V.) which is within the require less than 10 % for a good regression coefficient demonstrated in Figure 2. P-values greater than 0.05 are insignificant at 95 % confidence level hence X_1 , X_2X_3 , X_1^2 , X_2^2 and X_3^2 factors are significant just as the model is. The F-values can be

used to present a contribution order of each factors to the reactions where X_1X_2 indicated as the lowest F-value is the least.

A three dimensional expression of the factors interactive effects on yield is demonstrated in Figure 3, pointing that an increase in X_3 beyond 40 min will

Table 8: Rotatable fractional CCD KSOME optimal production

Run order	X_1	X_2	X_3	Actual KSOME Yield	Predicted KSOME Yield	Residual
	vol/vol	% wt.	min	% wt.	% wt.	
1	0	0	$-\alpha$	91.16	90.54	0.6244
2	$+\alpha$	0	0	82.13	81.51	0.6244
3	0	$-\alpha$	0	92.51	91.89	0.6244
4	0	0	0	95.98	96.10	-0.1178
5	$-\alpha$	0	0	88.79	88.17	0.6244
6	0	0	0	95.44	96.10	-0.6578
7	0	0	0	95.94	96.10	-0.1578
8	0	$+\alpha$	0	86.97	86.35	0.6244
9	-1	-1	-1	84.37	84.99	-0.6244
10	0	0	0	97.98	96.10	1.88
11	-1	1	1	75.6	76.22	-0.6244
12	0	0	$+\alpha$	85.95	85.33	0.6244
13	0	0	0	93.9	96.10	-2.20
14	1	1	-1	80.63	81.25	-0.6244
15	1	-1	1	88.47	89.09	-0.6244

transmit a negative yield response with X_2 at constant X_1 on Figure 3(c). However, a decreasing of X_3 and increasing X_2 will favour KSOME yield.

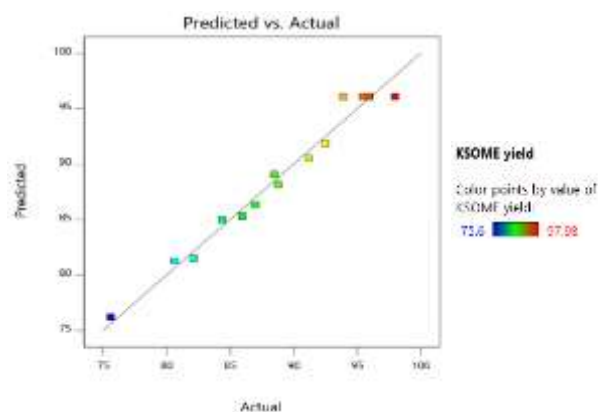


Figure 2: Plot of predicted versus actual KSOME yields

Fuel physicochemical properties

A compares of investigated properties with exiting literature on the same seed in Table 4, divulged some splashy differences in cetane number, flash point, cloud point and pour point which may be due to cultivation and geographical variation effects on seed (Moser, 2009).



Table 9: Quadratic model ANOVA summary for KSOME yield

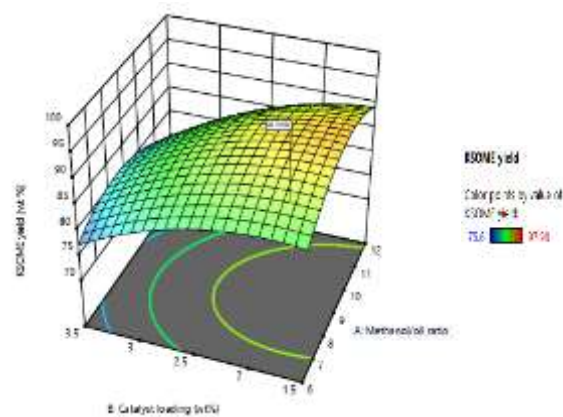
Source	Sum of Squares	df	Mean Square	F-value	p-value
X_1 (Methanol/Oil ratio)	22.18	1	22.18	8.70	0.0319
X_2 (CPP loading)	15.35	1	15.35	6.02	0.0577
X_3 (Reaction time)	13.57	1	13.57	5.32	0.0691
X_1X_2	5.18	1	5.18	2.03	0.2133
X_1X_3	9.63	1	9.63	3.78	0.1096
X_2X_3	43.01	1	43.01	16.87	0.0093
X_1^2	244.62	1	244.62	95.97	0.0002
X_2^2	94.02	1	94.02	36.89	0.0017
X_3^2	128.64	1	128.64	50.47	0.0009
ANOVA					
Model	579.20	9	64.36	25.25	0.0012
Lack of fit	4.21	1	4.21	1.97	0.2327
Residual	12.74	5	2.55		
Pure error	8.53	4	2.13		
R^2	0.9785				
Adequate precision	15.2459				
C.V %	1.79				

The 77.01 cetane number indicate that the biodiesel firing will demonstrate minimal ignition delay, while fine biodiesel atomization can be achieved with 5.862 mm²/s viscosity and good amount of energy could be liberated from the biodiesel with 37.443 MJ/kg of LHV in an internal combustion engine. Safe biodiesel portability is guaranteed by 124 °C flash point and a fairly low-temperature operability by 2 °C pour point. A minimal fuel corrosive effect is betokening by 0.794 KOH/g acid value.

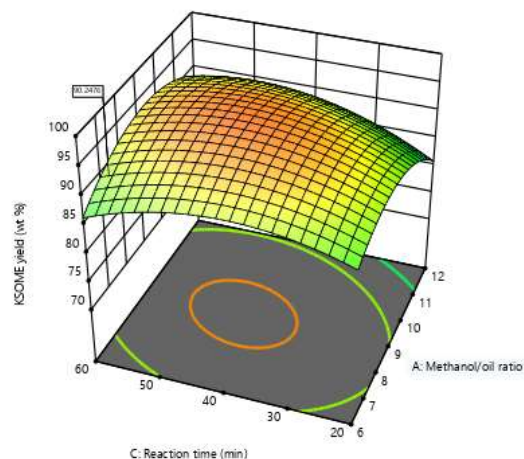
Catalyst properties: elemental and FT-IR analysis

An evidence projected in Table 5, pointed that K is the dominant catalytic element of CPP with 78.9 % mass fraction similar to (Betiku and Ajala, 2014) and (Etim et al., 2018) with the same plantain peel at 700 °C of calcination. The higher mass fractions this study presented over others could be argued to be stemming from a combination of ripped and overripe peels in this study as against the others. Calcination was necessary as it aids the prevention of leaching elements and will promote CPP reusability as presented by Meeta et al. (2012). A wavenumber ranges of 4000 – 650 (cm⁻¹) and Transmittance range of 95 – 62 at a resolution of 4 presented in Figure 4 for CPP FT-IR, has been

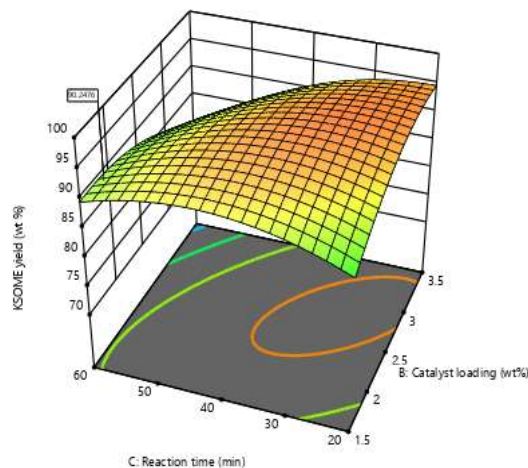
summarized in Table 6 for speculated stretching vibration band and their corresponding active functional groups using the detected peak on the graph.



(a)



(b)



(c)

Figure 3: Interaction effects of reacting factors on KSOME yield

Carboxylic acid and phosphine are noted as the dominating groups. The identified vibration suggested that element like N, S, Cl and F are present in CPP organic mixture due to the presence of K₂MgSiO₄, KNaSO₄ and KCl compounds as revealed in X-ray diffraction chromatogram result of (Etim et al., 2018).



Table 10: Comparing fuel physicochemical properties

Properties	KSO		KSOME			ASTM D6751	DIN EN 14214
	Present study	(Danbature et al., 2015)	Present study	(Danbature et al., 2015)	(Soja et al., 2017)		
Density at 15 °C (Kg/m ³)	923	907.8 (at 20 °C)	874	871.3 (at 20 °C)	905	840	860 - 900
Kinematic viscosity at 40 °C (mm ² /s)	50.61	19.16	5.862	3.509	5.09	1.9 – 6.0	3.5 -5.0
Surface tension at 15 °C (N/m)	0.03053	-	0.0333	-	-	-	-
API	21.804	-	30.399	-	-	36.95	-
Percentage Yield	50.15	58.6	75.6 - 97.98	97.2	90	-	-
Percentage FFA	3.10	0.0704	0.399	0.0905			
Acid value (mg KOH/g)	6.171	0.14	0.794	0.18	0.72	0.80 max	0.50 max
Saponification value (mg KOH/g)	79.66	91.6	121.74	-	-	-	-
Iodine value (g iodine/100 g)	32.18	92.4	62.78	-	-	-	120 max
Peroxide value (meq/kg)	2.67	-	-	-	0.225	-	-
Cetane number	107.58	-	77.01	-	54	47 min	51 min
Smoke point (°C)	-	-	89.3	-	-	-	-
Flash point (°C)	-	-	124	155	134	93 min	120 min
Pour point (°C)	-	-	2	- 1.5	- 15	-	-
Cloud point (°C)	-	-	8.3	4.5	- 5	-	-
Calorific value (LHV) (MJ/kg)	-	36.55	37.443	37.897	37	-	-

Table 11: Elemental composition of CPP by atomic absorption spectrometer (AAS) method

Element	Calcium	Magnesium	Potassium	Sodium	Manganese	Iron	Phosphorus
	Ca	Mg	K	Na	Mn	Fe	P
Concentration (mg/l)	386.752	372.511	4,455.10	286.54	1.411	2.326	141.95
% Mass Fraction (Present Study)	6.85	6.60	78.90	5.08	0.03	0.04	2.51
% Mass Fraction (Betiku and Ajala, 2014)	1.13	ND	54.73	ND	0.05	0.04	1.99
% Mass Fraction (Etim et al., 2018)	ND	1.15	51.02	ND	ND	ND	1.84

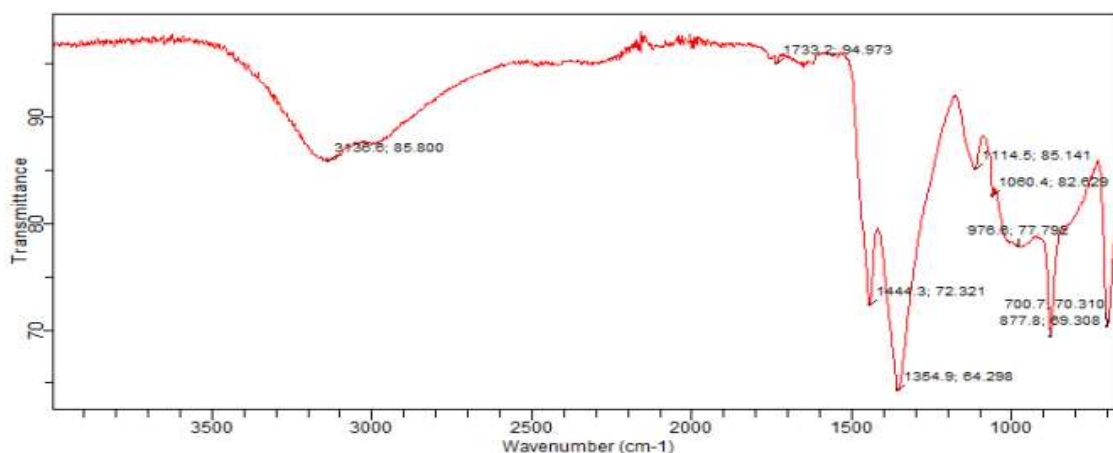


Figure 4: FT-IR result of CPP catalyst

Table 12: Summary of FT-IR CPP catalyst interpreted result

Wavenumber (cm ⁻¹)	Vibration Band	Speculated Function Groups
3138.8	O—H Stretch	Carboxylic acids
1733.2	C=O Stretch	Carboxylic acids and Esters
1444.3	N=N Stretch and O—H Stretch	Azo and Carboxylic acids
1354.9	C—H in-plane bend	Alkenes
1114.5	C—O Stretch, Aromatic C—N Stretch, C—O—C Stretch, —NO ₂ , C—F Stretch and Stretch (aromatic)	Alcohols, Amines, Ethers, Haloalkane and Nitro
1060.4	P—H bend and S=O Stretch	Phosphine and Sulfonate
976.6	P—H bend and S—O Stretch	Phosphine and Sulfonate
877.8	P—H bend	Phosphine
700.7	C—Cl Stretch	Haloalkane

CONCLUSION

It is therefore substantiating by this study that using heterogeneous catalyst for biodiesel production is cost reduction implicative and will as well contribute to maintaining an ecofriendly environment due to its element non-leaching effect if practiced. Furthermore, 50.15 % oil and 97.98 % biodiesel yields is certified satisfactorily high as optimum production is feasible at minimal cost using RSM. Hence, KSOME will be lucrative on mass production, satisfactorily meeting international standards as a projected viable green energy source.

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