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Optimized Biodiesel Synthesis from Binary Mixture of Honne and Yellow Oleander Seed Oils for Sustained Availability of Alternative Fuel

*[1]Soji-Adekunle, A. R., [2]Betiku, E., [3]Adeboye, B. S., [2]Falowo, O. A., [4]Oyewusi, T. F. and [1]Oyerinde, A. Y.

[1]Department of Mechanical Engineering, Adeleke University, Ede, Nigeria
[2]Department of Chemical Engineering, Obafemi Awolowo University, Ile-Ife, Nigeria
[3]Department of Mechanical Engineering, Osun State University, Osogbo, Nigeria
[4]Department of Agricultural Engineering, Adeleke University, Ede, Nigeria

***Corresponding Author:** *soji-adekunle.ayowunmi@adelekeuniversity.edu.ng*

ABSTRACT

This research was carried out to optimize production of biodiesel achieved by fraternization of two non-edible oils, honne and yellow oleander, mixed in equal proportion to produce biodiesel. The binary oil (BO) was transformed to biodiesel by two steps. A biomass-based synthesized catalyst (BBSC) from blend of cocoa pod, plantain peel and kolanut pod ash was adapted to trans esterify the mixture. Taguchi experimental design tool was used to understudy the combined effects of irradiation power (W), methanol/oil ration (MeOH), time (S) and BBSC (wt %) on the percentage yield of the biodiesel from the BO. The production process was assisted by microwave irradiation. The results of Energy Dispersive X-ray Spectroscopy (EDX) of BBSC reveal potassium as the essential active catalytic element. The acid value of BO was reduced to < 2% via acid pre-treatment. A yield of 92.51% was achieved using microwave irradiation at MeOH of 9:1, microwave power of 450 W, BBSC of 3.5 wt % and reaction time of 1 minute. The biodiesel produced satisfied ASTM D 6751 and EN 14214 standards. Performance evaluation of the process input variables suggests weight of MeOH has the most significant process parameters for binary oil biodiesel (BOB) yield. Statistically, the predicted optimum yield differs from the actual by 0.01% thus proving the efficacy of the experimental design tool used. This study validates that biomass catalyst can adequately be applied to synthesis effectively biodiesel from blends of non-edible oils to supplement fossil diesel.

Keywords: Biodiesel, biomass, microwave irradiation, non-edible oil, optimization, transesterification.

1. INTRODUCTION

It is a known fact that there is major dependence on fuel from fossil for generation of energy globally. Energy is a major need to drive the economy of any country. The capacity and functionality of industrial, economical, technological and domestic activities depend directly on the amount of energy that a country can assess (Soji-Adekule *et al.*, 2019). The availability of energy in sufficient capacity assures sustenance of various sectors of the economy. It can then be deduced that for any country to continuously develop, the level of energy available is a germane factor. At present, the fuel converted to synthesize energy to drive transportation, agriculture, power generation, industry and domestic consumption is largely harnessed from fossil based fuel. This has raised a concern centered on how to reduce excessive reliance on its demand (Atabani *et al.,* 2012).

It is also worthy to note that the price of fossil based diesel has greatly increased recently so much that it is becoming uneconomical for commercial activities. In addition, the depletion of stratospheric layer, contamination of rain, disappearance of some animal and plant species surfaced due to some of the activities which entails the burning of fossil fuel for one purpose or the other (Bernstein *et al.*, 2008). Furthermore, activities in the transportation and power generation sectors have also contributed to undesired environmental conditions which include green-house effects, alterations of atmospheric concentration, unstable climate and global warming (Sanjel *et al.*, 2014). The aforementioned has necessitated a need for an alternative fuel that will be feasible and affordable and will also lessen the discussed undesirable effects (Silitonga *et al.,* 2013).

There have been several attempts to produce fuels to substitute fossil. Different biomasses have been transformed to possible substitute such as briquette, biogas, bioethanol and biodiesel (Jekayinfa *et al.*, 2018). Other sources include energy generated from micro hydro stations, wind farm and the use of photovoltaic materials to trap energy from sunlight. A major itch to these alternatives include the installation cost and instability in the weather and climatic conditions (Council, 2016). To mitigate this, the present age is envisaging that fuel from wastes viz- biological and agricultural could go a long way to supplement the existing ones in both unindustrialized and developed countries especially if the cost of production can be moderate and if such fuel is friendly to the environment (Soji-Adekunle *et al.,* 2019).

Taguchi experimental design method is a technique for optimizing inconsistency of a single factor while keeping another factor constant. The two basic concept is to restrict reduction in quality by estimating the divergence from the expected objective and to obtain the topmost quality level in a profitable way (Mamourian *et al.,* 2016). The method is based on orthogonal array which is capable of reducing the number of experiments that will be performed and still optimize the process within few numbers specified (Dhawane *et al.,* 2016a). It also give sufficient information about the relationship between reaction variables based on the orthogonal array which assist to optimize the process with less effort and reduced time (Abu-Jrai *et al.,* 2017). Microwave irradiation entails the use of an alternative energy stimulant in the form of microwave irradiation for transesterification process (Patil *et al.,* 2010). Microwave assisted method is a fast and easy means to accomplish transesterification because it requires shorter duration for the completion of the reaction, lower ratio of triglyceride to alcohol, the set up can be easily operated, marked reduction of by-product and consumption of lesser energy (Kumar *et al.,* 2011).

Biodiesel is a bio-based fuel type formulated by reacting oil from plant origin with catalyst in transesterification reaction. This fuel type has been found to be usable directly in compression ignition engine with no need for modification and with output performance characteristics of the engine being similar to that operated on automotive gas oil. This bio-based fuel can also be mixed in different proportional ratio to obtain different fuel blends which can enhance the performance of the compression ignition engine (Ang *et al.,* 2015)**.** Biodiesel has been found to be almost totally free of combustion contaminant such as sulphur and burns better because of its chemical structure. The fuel is also more environmentally friendly because of its ability to cut down on SOx and COx during combustion (Chavan *et al.,* 2013).

In the past, different eatable vegetable oils had been used for almost greater percentage of the biodiesel produced (Wakil *et al.,* 2015), the chance of continuous use of eatable oil as biodiesel feedstock is slim as this may result in food fuel crisis in the future thus non-eatable oils are recommended (Morshed *et al.*, 2011). The adoption of non-dibble oil and its mixtures as feedstock oils will solve the problem of food crisis, provide further useful purpose for the non-edible oils and also increase the varieties of oil that can be used as feedstock oil. The use of biomass wastes for catalyst production will reduce environmental pollution. Thus, this study optimized biodiesel synthesis from binary mixture of honne and yellow oleander seed oils for sustained availability of alternative fuel.

2. METHODOLOGY

2.1 Materials

Honne and yellow oleander seeds were collected from Obafemi Awolowo University, Ile-Ife, Nigeria compound, processed and taken to the Pharmacy Laboratory of the institution for oil extraction via Soxhlet apparatus using n-hexane to obtain honne oil (HO) and yellow oleander oil (YO). Biomass materials were obtained from a village within Obafemi Awolowo University campus, Ile-Ife. Reagents and chemicals which include di-ethyl ether, ethanol, methanol, potassium iodide, starch solution, cyclohexane, sulphuric acid, sodium sulphate, phenolphthalein, potassium hydroxide and Wij's solution of analytical grade were also used for the study.

2.2 Preparation of Biomass-Based Synthesized Catalyst (BBSC)

The obtained biomass materials were expurgated, washed 3 times with distilled water, sundried for 2 weeks and thereafter dried in the oven at 80^oC till constant weight was achieved. These wastes were separately seared into ashes in the open air after which each ash was manually minced to fine powder using porcelain mortar and pestle. The resulting powdered samples were weighed in equal proportion, mixed and calcined in a muffle furnace at different temperatures for 4 h. The calcined BBSC ash produced was then stored in corked plastic vessels.

2.3 Determination of Characteristic Features of BBSC

The calcined BBSC ash was characterized using Energy Dispersive X-ray Spectroscopy (EDX) tool to determine the elemental composition and the active element in the catalyst. This was accomplished using Cambridge S200SEM equipped with an energy dispersive X-ray analyzer.

2.4 Preparation of Binary Oil (BO)

Equal volume of honne and yellow oleander seed oils were mixed together by first heating them separately in a 500 ml conical flask at 60^oC for 15 minutes and then decanted one after the other into a 1000 ml flat bottom flask placed on a hot plate equipped with stirrer having magnetic property for agitation and proper fraternization at 60^oC for another 30 minutes to get BO.

2.5 Experimental Design for Tranesterification

Taguchi experimental design which generated orthogonal array of nine experiment tool was used. Four factors at three levels were input into the design tool to generate nine experimental runs to investigate the effects of MeOH, BBSC, irradiation power and reaction time on the yield of biodiesel from BO. The reaction was conducted within the confinement of the microwave set up.

2.5.1 Acid catalyzed esterification of BO

A predetermined quantity of BO was put into a 500 ml two necked glass reactor. The esterification process was carried out using MeOH 25:1, H_2SO_4 1 wt. %, heat up power 300 w and reaction time of 15 minutes. The reaction took place in a glass reactor which was placed in the microwave oven. At the completion of the reaction, the mixture was allowed to separate by gravity and the residual methanol was removed by heating off on the hot plate before determining the acid value of the esterified oil.

2.5.2 Transesterification of pretreated BO with biomass-catalyzed

A predetermined amount of esterified BO was put in a two necked glass reactor. Known volume of methanol was added as well as known amount of BBSC. This was placed in a pre-timed microwave and the reaction was stopped based on the time indicated by the experimental design. The factors and levels Taguchi experimental design is presented in Table 1. MeOH, reaction time, irradiation power and weight of BBSC were combined on three levels to generate orthogonal array of nine experimental conditions by Taguchi design tool to model the production of BO methyl esters. At the completion of the reaction, the resulting product was allowed to separate by gravity, glycerol was tapped off and the BO biodiesel (BOB) was washed with distilled water at 50ºC to get rid of residual catalyst, glycerol, methanol and soap. The washed BOB was dried over heated anhydrous sodium sulphate powder and the BOB yield was determined gravimetrically according to equation 1.

$$\text{BOB yield (wt.\%)} = \frac{\text{weight of oil sample}}{\text{weight of biodiesel sample}} \times 100 \quad (1)$$

Table 1: Factors and levels Taguchi experimental design

Factors	Unit	Level for factors		
		-1	0	1
MeOH	v/v	6	9	12
Catalyst amount	wt %	2.0	3.5	5.0
Microwavw power	Watt	150	300	450
Reaction time	Mins	1	3	5

2.6 Determination of Properties of BOB

The physicochemical properties of the BOB produced were carried out according to Betiku *et al.* (2015). The properties determined include kinematic viscosity, density, moisture content, acid value, saponification value (sv), iodine value, higher heating value, cetane number diesel index and mean molecular mass.

2.7 Analysis of Data

The data obtained from the experimental runs were analyzed using Analysis of variance (ANOVA) and significance test were both applied on the experimental yield to determine the quality of the quadratic polynomial response model. The regression equation for BOB yield was mathematically described and the level of significance for the process was established by the ANOVA result.

3. RESULTS AND DISCUSSION

3.1 Properties of BO

The biodiesel production related properties for BO are presented in Table 2. The acid value of BO shows that there is need to reduce the acid value to prevent saponification. The acid value of BO was determined to be 38.03 mg of KOH/g of oil. The acid pretreatment of BO was carried out using methanol/oil molar ratio of 25:1, catalyst loading (H_2SO_4) of 1 wt.%, microwave power of 300 W and time of 15 minutes. This reduced the acid value to <1 mg KOH/g oil. Thus the tendency of soap formation was reduced.

3.2 Composition of BBSC

Table 3 shows the composition of BBSC. It could be observed that at different temperatures, the elemental composition of the BBSC were O, Mg, Ca, Cl, S, P, Si, Fe and Al. Based on the analysis, it was observed that the calcination temperature had a significant effect on the elemental composition of the catalyst. It can be seen that K has the highest percent mass fraction at 500ºC, and is the major active constituent responsible for the catalytic activity in biodiesel production (Etim *et al.,* 2018). From previous studies on processing of various biomass for production of catalysts, potassium (K) has been reported to have the highest concentration among other metals in calcined cocoa pod hush ash (Betiku *et al.,* 2017b), ripe banana fruit peel (Betiku *et al.,* 2016) and unripe plantain fruit peel (Betiku and Ajala, 2014) when calcined at 500ºC for 4 h, 700ºC for 4 h and 500ºC for 3.5 h, respectively. It can be seen that increase in temperature between 300 and 1100ºC caused the elemental contents to increase. The characterization of BBSC by calcination also reveals that heavy metals such as cadmium, arsenic, titanium, and lead were absent, implying that the synthesized catalyst is non-toxic and eco-friendly (Onoji *et al.,* 2017).

Table 2: Properties of BO

Parameters	Values Honne	Oleander Seed	BO
Physical properties			
Physical State at 25 ºC	Liquid/dark green	Liquid/dark brown	Liquid dark brown
Moisture Content (%)	0.640 ± 0.001	0.050 ± 0.001	0.026
Specific Gravity	0.901 ± 0.005	0.961 ± 0.001	0.925
Kinematic Viscosity (mm^2/s) at 40 ºC	37.01 ± 0.76	124.43 ± 0.04	80.53
Refractive Index at 25 ºC	1.475 ± 0.00	1.4764 ± 0.001	1.47
Density (kg/m^3) at 25 ºC	901 ± 0.014	964 ± 0.001	925
Chemical properties			
%FFA (as oleic acid)	20.24 ± 0.35	7.32 ± 0.91	13.64
Acid Value (mg KOH/g oil)	40.28± 0.00	14.57± 0.00	38.03
Iodine value (g I_2/100g oil)	80.44 ± 0.65	84.53 ± 0.74	81.45
Saponification value (mg KOH/g oil)	226.5 ± 0.94	178.86 ± 0.81	201.68
Higher heating value (MJ/kg)	51.69± 0.00	49.39± 0.00	50.54
Other Properties			
Cetane number	52.29± 0.00	58.00± 0.00	54.12

Table 3: Composition of BBSC

Temperature ºC	Element (mass%) O	Mg	Si	P	S	Cl	K	Ca	Fe	Zn	Na	Al
300	40.43	4.06	0.90	1.74	1.41	1.97	43.99	5.50	0.00	0.00	0.00	0.00
500	**37.21**	**4.21**	**0.56**	**1.65**	**1.26**	**1.88**	**47.67**	**5.56**	**0.00**	**0.00**	**0.00**	**0.00**
700	41.20	3.05	0.79	1.61	0.92	1.88	47.93	3.93	0.00	0.00	0.00	0.00
900	41.59	1.85	1.41	1.77	1.29	1.43	45.55	4.68	0.64	0.00	0.00	0.00
1100	45.30	0.81	2.82	5.20	1.17	1.03	43.90	0.00	0.00	0.00	0.00	0.75

3.3 Modeling and Parameter Optimization for Transesterification of BO

The result of experimental yield of BOB via BBSC catalyzed methanolysis of BO to BOB using orthogonal array of nine experiments is presented in Table 4. The model equation for the reaction which can be used to predict the yield when actual factors are applied is represented by equation 2.

$$BOB\ yield\ (wt\ \%) = +89.71 - 2.56A_1 + 3.21A_2 + 0.048B_1 - 2.07B_2 - 3.07D_2 \qquad (2)$$

Where A, B and D were MeOH, BBSC and time respectively. BOB yield varied between 82.06 wt. % and 94.85 wt. %. The maximum yield was obtained at third experimental run. This corresponds to 2 wt. %, 300 W, 9:1 and 5 minutes. This experimental run number also has the best combination of process parameter. The fuel properties of BOB are presented in Table 5. BOB has improved cetane number which implies better fuel strength; the iodine value is very low implying good shelf life. The viscosity is a tad higher than specified therefore BOB will require blending with AGO to present clogging of filters of unmodified IC engine and to ensure long time usage.

Table 4: Orthogonal Array of Experimental Runs for BO Transesterification

Run	MeOH	BSC (wt.%)	Power (W)	Time (min)	Actual Yield (wt.%)
1	12	3.5	150	5	88.36
2	6	5	450	5	91.43
3	9	2	300	5	94.85
4	6	3.5	300	3	82.06
5	6	2	150	1	87.97
6	9	3.5	450	1	92.51
7	9	5	150	3	91.4
8	12	2	450	3	86.46
9	12	5	300	1	92.37

Table 5: Fuel Properties of BOB

Properties	BOB	ASTM D6751	EN 14214
Physical state and colour	Liquid/golden yellow	NS	NS
Kinematic viscosity(mm^2/s) at 40 ºC	6.83	1.9 - 6.0	3.5 - 5.0
Density at 25 ºC (kg/m^3)	868.	NS	860 – 900
Moisture content (%)	0.025	<0.03	0.02 max
Acid value (mg KOH/ g oil)	0.20	0.50 max	0.50 max
SV (mg KOH/g oil)	180.82	-	-
Iodine value (g I_2/100g oil)	33. 18	NS	120 max
Higher heating value (MJ/kg)	40.31	NS	NS
Flash point	ND	130 minimum	120 minimum
Pour point	ND	NS	NS
API gravity (deg.)	ND	39.95	NS
Diesel index	72.6	331.00	NS
Aniline point	ND	NS	NS
Mean molecular mass	24.05	-	-
Cloud point	ND	NS	NS
Cetane number	62.28	47 mi	51 min

NS = not specified

The regression analysis for this study tested by means of ANOVA shows that $p < 0.05$ (Table 6) for all model terms meaning that statistically the result is significant at 95% level of sureness and that the most influential parameters affecting the yield of BOB is BBSC. The F value of 33.35 and p value of 0.0294 of the model assert that all the model terms are significant (Table 6). The model terms was developed by eliminating the term that is not significant on the response. Only the power is insignificant on the yield of BOB and that was why it was not contained in the model terms. MeOH has the highest influence on BOB yield when placed side by side with other significant factors; this is clearly spelt out in the p-values of the factors. From the value of the R^2 (0.9931) the model can explicate 99% of the changeability of the process (Betiku *et al.*, 2017a). For the signal of the design process to be strong enough, the adequate precision which measures the signal to noise ratio must be > 4 thus 18.735 denotes strong signal of the design of this study. Similarly, the adeptness of the model is reflected in its percentage coefficient of variation (CV) which is required to be less than 10. The CV for this study is 0.86%. This model is fit to adequately describe the process.

Table 6: Significance Result for Modeling of Methanolysis of BOB

Source of variance	Sum of squares	Degree of freedom	Mean square	F-value	p-value
Model	119.89	6	19.98	33.35	0.0294
A- MeOH:NOYO	51.78	2	25.89	43.20	0.0226
B - BBSC	25.10	2	12.55	20.95	0.0456
D – Time	43.01	2	21.50	35.89	0.0271
Fitting statistics					
Standard Deviation		0.77			
Mean of response		89.71			
Coefficient of Variation (CV) (%)		0.86			
R^2		0.9901			
Adequate precision		18.735			
Adequate Precision		18.735			

3.4 Effects of Process Parameter on BOB Yield

The influence of the three process parameters that have profound effect on the yield of BOB diesel are represented as diagnostic plots in Figure 1. Considering the effect of MeOH on BOB yield, molar proportion of methanol to oil at three levels (6:1, 9:1, and 12:1) were adopted to investigate their influence on BOB yield. The effect of these ratios is represented in Figure 1a. BOB yield increased from 6:1 to 9:1. Any ratio higher lowers the yield. The ratio that favoured BOB yield most was 9:1 with maximum yield of 94.85 wt%.

Catalyst loading at three levels; 2, 3.5 and 5 wt. % were investigated for the study of influence of amount of catalyst on BOB yield while keeping other parameters constant. The diagnostic plot Figure 1b reveals that the maximum yield was achieved with BBSC of 2 wt. %. This is a plus for the BBSC catalyst used because low quantity is required for transesterification, thus, lowering the production cost of BOB. Dhawane *et al.* (2016b) also reported similar observation with the amount of catalyst required for transesterification in their study.

Time taken for completion of a reaction is one of the key factors that assist biodiesel production. Reaction time of 1, 3 and 5 minutes were investigated to study their effect on BOB yield. This is represented in Figure 1c. The maximum BOB yield of 94.85 wt%.was achieved at 5 minutes yield. A closer percentage yield was achieved for the ninth experimental run but there was an associated increase in the amount of methanol and BBSC. The reduction in the yield at the other power investigated may be due to the possibility of drying-off of the alcohol in the reaction mixture because of the intensity of heat generated in the microwave.

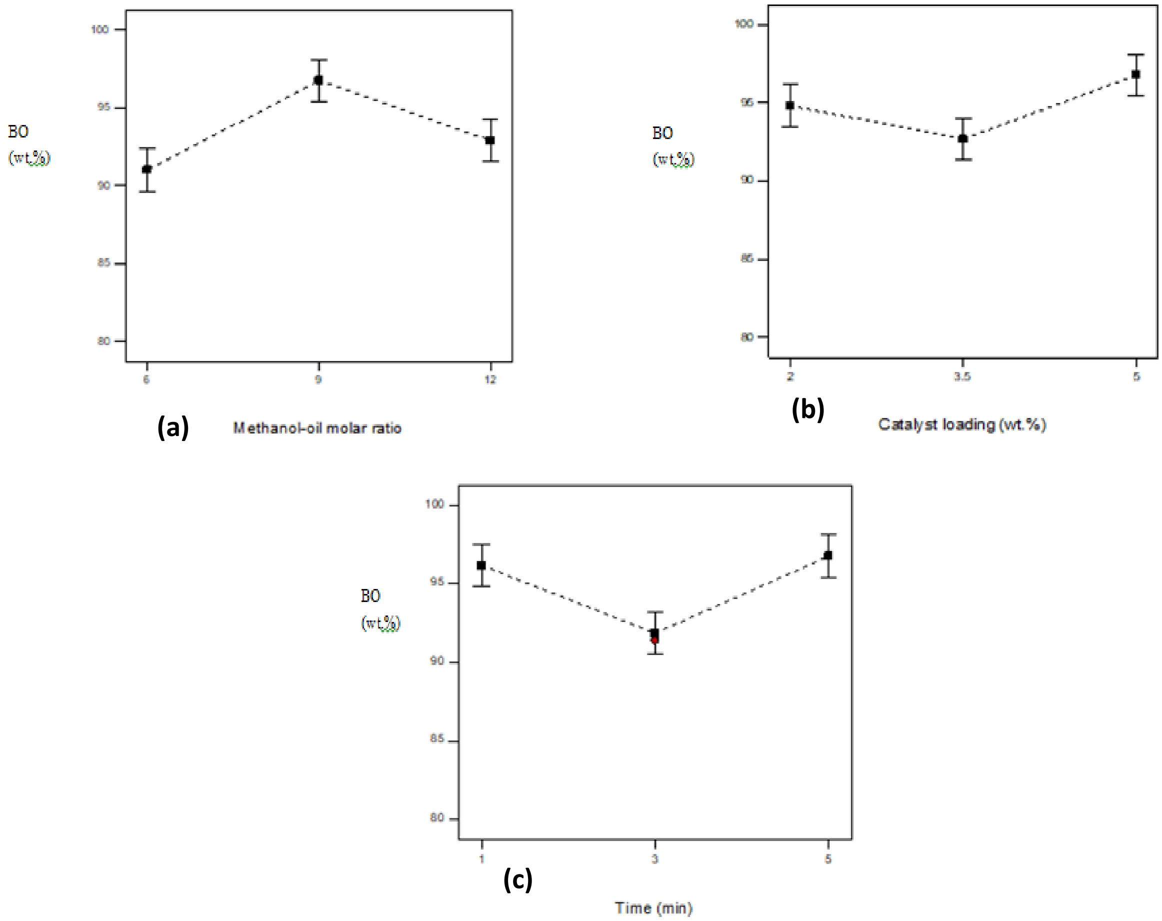

Figure 1: Diagnostic Plots for BOB Production: (a) Effect of MeOH (b) Effect of BBSC (c) Effect of Time

3.5 Model Validation

The optimization of each parameter considered was performed to determine the best combination that will give optimum BOB yield based on the mathematical model developed. The optimum values of the process parameters and the biodiesel yield obtained from this study for conversion of BO into BOB using BBSC were methanol to oil molar ratio of 9:1, catalyst weight of 2 wt.%, heat up power of 300 W and reaction time 5 minutes. The optimum BOB yield projected at this condition was 100 wt. % with desirability of 1. The optimum condition forecast by the regression model equation was validated by performing the experiment in triplicates. It was observed that the results of the experimental and that predicted are close which implies that the chosen model is appropriate for predicting the optimum condition for BOB production. The average BOB yield observed was 98.57 ± 0.58 wt.%.

4. CONCLUSION

The physicochemical properties of binary oil showed that it is a feasible feedstock for biodiesel production but requires acid pre-treatment prior its trans-esterification. The prime conditions established from this study for conversion of binary oil into binary oil biodiesel using bio-based synthesized catalyst was methanol oil molar ration of 9:1, catalyst loading of 2 wt.%, reaction power of 300 W and reaction time of 5 minutes with binary oil biodiesel yield of 98.57 ± 0.58 wt.%. The contribution of the process parameters were; methanol oil molar ratio (51.32%), bio-based synthesized catalyst (33.30%) and time (15.00%) showing methanol oil molar ratio as the most influential process parameter on binary oil biodiesel yield. Reaction power had least contribution factor (0.68%) and so was eliminated by the model. Binary oil biodiesel produced is of high quality since most of its properties agreed satisfactorily with biodiesel standard specifications (ASTM D6751 and EN 14214) but requires blending to be used in unmodified internal combustion engine because of its viscosity which is a little higher than specified standard. This will prevent the tendency of clogging of the engine and as well ensure elongation of the life span of the engine for long term usage.

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