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## Data Article

# Data on the derived mesoporous based catalyst for the synthesized of fatty acid methyl ester (FAME) from ternary oil blend: An optimization approach

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## ABSTRACT

This work presents datasets on fatty acid methyl ester (FAME) synthesized from the ternary blend of *Cucurbita pepo-chrysophyllum albidum* -papaya mix oils via methanolysis of mesoporous CaO heterogeneous catalyst derived from the mixture of *Citrullus lanatus* and *Musa acuminata* peels. The oils were extracted from the milled powdered using the solvent extraction method. Ternary oil mixed ratio of 33:33:34 with low acid value and density was achieved using simplex lattice design software. Characterization of the mixed calcined catalyst powder (MCCP) at 700 °C for 4 h was carried out using scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), X-ray diffraction analysis (XRD), and BET analysis. The thermal decomposition of mixed calcined catalyst powder (MCCP) produced 78.74% CaO with a strong basic site of 143 ( $\mu\text{mole.g}^{-1}$ ). Fatty acid methyl ester (FAME) was synthesized through the based catalyst transesterification of a derived catalyst by considering four variables data (reaction time, reaction temperature, catalyst amount and methanol/oil molar ratio) using response surface methodology (RSM). The maximum experimental FAME data of 94.29 (wt. %) was achieved at run 16, but the central

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composite design (CCD) software predicted value of 98.00 (wt. %) at a reaction time of 70 min, reaction temperature of 80 °C, catalyst amount of 5.0 (wt.) and methanol to oil molar ratio (MeOH/OMR) of 6.97, at the desirability of 97.90%. This was validated in triplicate, and the average FAME data obtained was 93.45 (wt. %). The produced FAME properties dataset meets the standard recommended value of ASTM and EN14214.

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## Specifications Table

|                                |   |
|--------------------------------|---|
| Subject                        | Material Science Engineering  |
| Specific subject area          | Renewable and Sustainable Energy  |
| Type of data                   | Table, Figure   |
| How data were acquired         | <p>A ternary mixture of oil was acquired through a simplex lattice mixture design. The significance of the variables was confirmed through analysis of variance (ANOVA) table. Physicochemical properties of the ternary blended oil and FAME produced were determined via AOAC (1997) standard method, Iodine value was determined through Wij's method [1]. The developed catalyst from the mixture of calcined <i>Citrullus lanatus</i> and <i>Musa acuminata</i> peels were characterized using SEM, EDS, XRD, and BET analysis.</p> <p>Experimental design and process optimization route of converting the blended oil to FAME was achieved through the simplex lattice and central composite experimental design.</p> <p>Catalyst activities, reactor wall accumulation, and catalyst purification were performed through catalyst reusability tests.</p> <p>The produced FAME fuel properties were confirmed by comparing with [2] and [3] biodiesel recommended standard.</p>  |
| Data format                    | Raw, Analyzed   |
| Parameters for data collection | <p><i>Cucurbita pepo</i>, <i>Chrysophyllum albidum</i>, and papaya seeds were milled into powders after drying. Mass extraction of oils from the powders was carried out through solvent extraction (soxhlet extractor).</p> <p>The blending of oil was carried out using simplex lattice experimental design with viscosity and acid value as the response variables.</p> <p>100 g each washed, mixed, dried and milled catalyst <i>Citrullus lanatus</i> and <i>Musa acuminata</i> peels were prepared for calcination in a furnace.</p> <p>Variable factors considered for experimental design during FAME production were reaction time (<math>K_1</math>), reaction temperature (<math>K_2</math>), catalyst amount (<math>K_3</math>) and methanol/oil molar ratio (<math>K_4</math>).</p>  |
| Description of data collection | <p>Oils were extracted from the powders through the soxhlet extractor using n-hexane as solvent. Excess n-hexane in the oil was recycled using a rotatory evaporator [4].</p> <p>The ternary oil blend was achieved by experimental design using a simplex lattice mixture (Raw data); the mixture of the three oils was varied in five level-three factors design, and the response variables were the viscosity and acid value [7, 8, 9, 10, 11].</p> <p><i>Citrullus lanatus</i> and <i>Musa acuminata</i> peels were oven-dried to constant weight at 110 °C for 3 h using an electrical oven (model DHG-9101-02). The dried samples were milled and then separated into a particle size of 0.30 mm powders [5]. The fine powders were mixed in ratio 1: 1, and then calcined at 700 °C for 4 h in a furnace with box-type resistance (model SX-5-12 with maximum control temperature of 1200 °C, 5 KW power rate). The calcined catalyst was then characterized by SEM, EDS, FTIR, and BET isothermal sorption (QUANTACHROME, 1 KE).</p> |

(continued on next page)

|                      |   |
|----------------------|---|
|                      | Since the acid value (FFA <1.50) of the blended oil was within the range of successful transesterification by a based catalyst, therefore, biodiesel was synthesized through the process route earlier adopted by [6] with few modifications. Catalyst reusability was stopped after 3rd usage due to a reduction in catalyst activity. |
| Data source location | The physicochemical properties of the blended oil and FAME produced were determined using the standard method of AOAC. The dataset obtained were compared with the biodiesel standard.  |
| Data accessibility   | Department of Chemical & Petrochemical Engineering, Akwa Ibom State University, Ikot Akpaden, Mkpato Enin L.G.A., Akwa Ibom State, Nigeria.<br>With the article   |

## Value of the Data

- Data on blend ratio can be used for the mixing of oils in the laboratory or industrial applications.
- Data on biodiesel synthesized can be modeled and optimized to examine the effect of variables on FAME yield
- Data will show authors in the field of engineering that calcined mixed *Citrullus lanatus* and *Musa acuminata* peels powder can produce an active CaO based catalyst for successful transesterification of oil to FAME.
- Dataset obtained shows that both calcined *Citrullus lanatus* and *Musa acuminata* peels powder can be used as a catalyst for FAME synthesized, but it mixed produced higher CaO conversion.
- Data on the physicochemical properties of the mixed oil and FAME produced shows that the produced FAME can serve as an alternative to conventional diesel.

## 1. Data

The dataset in this article describes the oil blend ratio which was carried out through simplex lattice design (expert 6.0.8 trial version) with three-factors (oils)-five levels design. Table 1a and 1b) shows the factors, the level and the results of the 16 experimental runs with response variables' (density and acid value), these values were used in the laboratory to obtain the experimental biodiesel yield. Table 2a and 2b shows the data on the ANOVA for a mixture of a cubic model as well as the point prediction, this was obtained through statistical analysis by a simplex lattice. Eqs. (1) and (2) showed the final equation in terms of real component generated by a design expert to show the density (d) and the acid value (AV) relationship with the variables data. Fig. 1(a-b) describes the ternary model blend ratio generated through the optimization technique of the design expert. Fig. 2(a) describes the results of the SEM used to determine the morphological characteristic of the derived catalyst, while Fig. 2b shows the FTIR used to confirm the presence of functional groups and verify the presence of characteristic absorption bands of CaO (Table 1c). Table 3 shows the data obtained for the BET surface, basicity, total pore

**Table 1a**

Five level three factors experimental design for oil blend.

| Variable | Units | Symbol | Levels |         |         |         |        |
|----------|-------|--------|--------|---------|---------|---------|--------|
|          |       |        | -2     | -1      | 0       | 1       | 2      |
| CPO      | (ml)  | $X_1$  | 0      | 0.16667 | 0.33333 | 0.66667 | 1.0000 |
| CAO      | (ml)  | $X_2$  | 0      | 0.16667 | 0.33333 | 0.66667 | 1.0000 |
| PO       | (ml)  | $X_3$  | 0      | 0.16667 | 0.33333 | 0.66667 | 1.0000 |

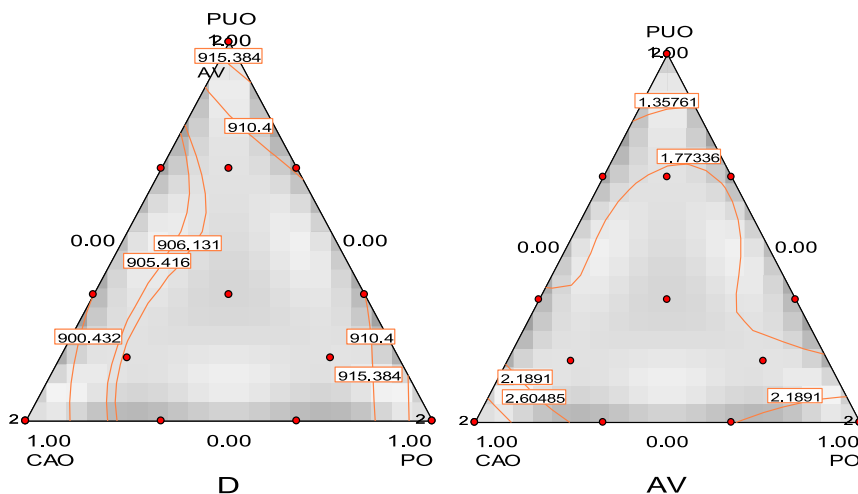
**Table 1b**  
Experimental runs with the response variables.

| R         | CPO           | CAO           | PO            | CPO            | CAO            | PO             | D (kg/m <sup>3</sup> ) | AV (mg KOH/g oil) |
|-----------|---------------|---------------|---------------|----------------|----------------|----------------|------------------------|-------------------|
| 2         | 1.0000        | 0.0000        | 0.00          | 100.0000       | 0.0000         | 0.0000         | 918.00                 | 0.53              |
| 9         | 0.6667        | 0.3333        | 0.00          | 66.6667        | 33.3333        | 0.0000         | 903.00                 | 1.63              |
| 13        | 0.6667        | 0.0000        | 0.3333        | 66.6667        | 0.0000         | 33.3333        | 911.00                 | 1.72              |
| 16        | 0.3333        | 0.6667        | 0.0000        | 33.3333        | 66.6667        | 0.0000         | 901.00                 | 1.80              |
| <b>15</b> | <b>0.3333</b> | <b>0.3333</b> | <b>0.3333</b> | <b>33.3333</b> | <b>33.3333</b> | <b>33.3333</b> | <b>907.00</b>          | <b>1.81</b>       |
| 12        | 0.3333        | 0.0000        | 0.6667        | 33.3333        | 0.0000         | 66.6667        | 910.00                 | 1.61              |
| 3         | 0.0000        | 1.0000        | 0.0000        | 0.0000         | 100.0000       | 0.0000         | 890.00                 | 3.02              |
| 10        | 0.0000        | 0.6667        | 0.3333        | 0.0000         | 66.6667        | 33.3333        | 908.00                 | 2.07              |
| 6         | 0.0000        | 0.3333        | 0.6667        | 0.0000         | 33.3333        | 66.6667        | 906.00                 | 2.18              |
| 14        | 0.0000        | 0.0000        | 1.0000        | 0.0000         | 0.0000         | 100.0000       | 920.00                 | 2.61              |
| 8         | 0.6667        | 0.1700        | 0.1667        | 66.6667        | 16.6667        | 16.6667        | 907.00                 | 1.82              |
| 5         | 0.1667        | 0.6667        | 0.1667        | 16.6667        | 66.6667        | 16.6667        | 907.00                 | 1.88              |
| 4         | 0.1667        | 0.1667        | 0.6667        | 16.6667        | 16.6667        | 66.6667        | 908.00                 | 1.86              |
| 7         | 1.0000        | 0.0000        | 0.0000        | 100.0000       | 0.0000         | 0.0000         | 918.00                 | 0.53              |
| 1         | 0.0000        | 1.0000        | 0.0000        | 0.0000         | 100.0000       | 0.0000         | 890.00                 | 3.02              |
| 11        | 0.0000        | 0.0000        | 1.0000        | 0.0000         | 0.0000         | 100.0000       | 920.00                 | 2.61              |

R= runs, V = viscosity, D = density and AV = acid value

**Table 1c**  
Peak assignment in the spectrum.

| Wavelength (cm <sup>-1</sup> ) | 1036.2 to 1442.5   | 1555.5 to 1636.3  | 2922.6 to 3338.7                           |
|--------------------------------|--|---|--|
| Transmittance <sub>0</sub>     | 44.962 to 76.401   | 63.439 to 66.145  | 74.179 to 53.157                           |
| Functional group               | Bending vibration of O-Ca-O group.<br>Presence of sp <sup>2</sup> in aldehyde/ketone and ester | C-H for sp <sup>3</sup> carbon,<br>C=O for sp <sup>2</sup> carbon<br>N-H bond | Presence of O-H of carboxylic acid and C-H |



**Fig. 1.** Plots of ternary model blend of oils.

volume, and the percentage composition of CaO obtained by EDX-nitrogen adsorption-CO<sub>2</sub> TPD from the calcined catalysts (*Citrullus lanatus*, *Musa acuminata* peels, and the mixed). The experimental design factor, the coded level, the experimental, the predicted and the residual data are presented in [Table 4a](#). These datasets are used for experimental modeling and statistical analysis through CCD. [Table 4b](#) describes the results of the tests of a significant and fit statistic obtained through statistical optimization, while the final equation in terms of the coded value based on a

dataset that relates the response FAME with the variable data generated through the polynomial quadratic model are presented in Eq. (3). The results of the relationship between the predicted and experimental yield as well as the Box-cox plot for power transformation are presented in Fig. 3(a-b). These plots were used to know the difference between the real experimental value and the predicted value by the design expert. Fig. 4 (a-f) shows the three-dimensional interactive effect of data variables ( $P_1P_2$ ;  $P_1P_3$ ;  $P_1P_4$ ;  $P_2P_3$ ;  $P_2P_4$  or  $P_3P_4$ ) on the output (FAME), the plots explained the relationship that exists between the interaction of the variable factors on the response of FAME. Table 5 describes the qualities of the FAME and the blended oil obtained from a ternary mix of Cucurbita pepo oil (CPO), Chrysophyllum albidum oil (CAO) and Papaya oil (PO).

$$D = 918.43X_1 + 890.46X_2 + 920.37X_3 - 11.28X_1X_2 - 39.97X_1X_3 + 8.63X_3X_2 + 56.69X_1X_3X_2 - 44.48X_1X_2(X_1 - X_2) + 7.28X_1X_3(X_1 - X_3) + 78.76X_3X_2(X_2 - X_3) \quad (1)$$

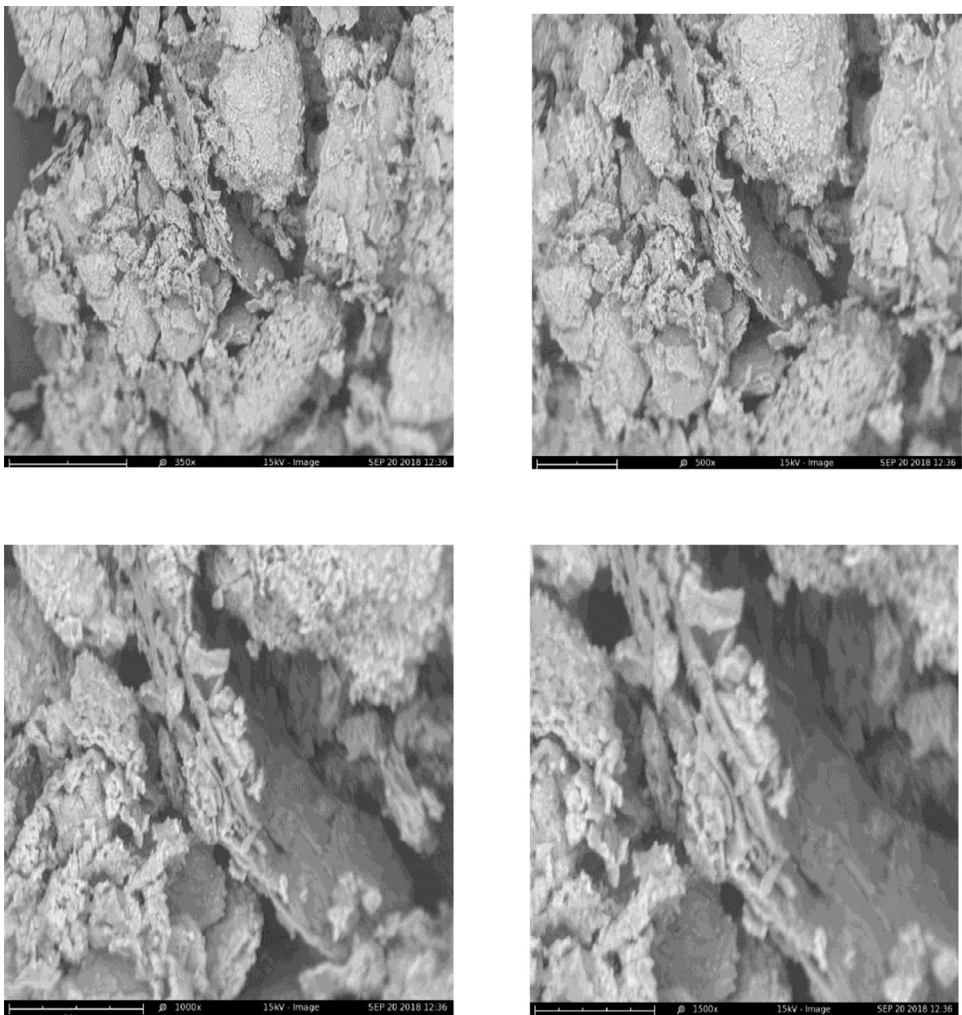


Fig. 2. (a) SEM image of a catalyst at different magnification. (b) FTIR analysis of the catalyst.

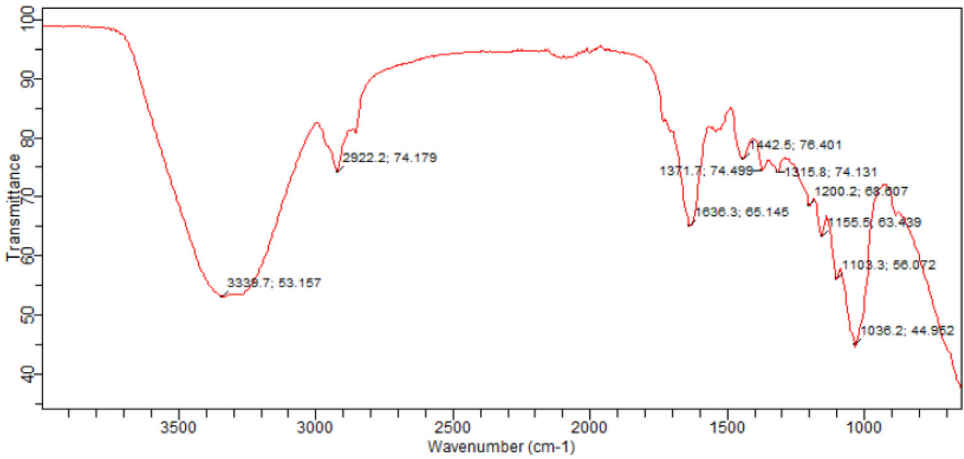


Fig. 2. Continued

**Table 2a**  
ANOVA for a mixture of cubic model.

| Source  | Sum of squares |                        | df | Mean square |                        | F value                | Prob > F |
|---|----------------|------------------------|----|-------------|------------------------|------------------------|----------|
|   | D              | AV                     |    | D           | AV                     |                        |          |
| Model   | 1233.92        | 7.60                   | 9  | 137.10      | 0.84                   | $6.366 \times 10^{-7}$ | < 0.0001 |
| LM  | 988.87         | 5.96                   | 2  | 494.44      | 2.98                   | $6.366 \times 10^{-7}$ | < 0.0001 |
| X <sub>1</sub> X <sub>2</sub>                                   | 8.57           | $4.085 \times 10^{-3}$ | 1  | 8.57        | $4.085 \times 10^{-3}$ | $6.366 \times 10^{-7}$ | < 0.0001 |
| X <sub>1</sub> X <sub>3</sub>                                   | 107.68         | 0.01                   | 1  | 107.68      | 0.01                   | $6.366 \times 10^{-7}$ | < 0.0001 |
| X <sub>2</sub> X <sub>3</sub>                                   | 5.02           | 0.65                   | 1  | 5.02        | 0.65                   | $6.366 \times 10^{-7}$ | < 0.0001 |
| X <sub>1</sub> X <sub>2</sub> X <sub>3</sub>                    | 3.90           | $6.045 \times 10^{-3}$ | 1  | 3.90        | $6.045 \times 10^{-3}$ | $6.366 \times 10^{-7}$ | < 0.0001 |
| X <sub>1</sub> X <sub>2</sub> (X <sub>1</sub> -X <sub>2</sub> ) | 28.80          | 0.29                   | 1  | 28.80       | 0.29                   | $6.366 \times 10^{-7}$ | < 0.0001 |
| X <sub>1</sub> X <sub>3</sub> (X <sub>1</sub> -X <sub>3</sub> ) | 0.77           | 0.43                   | 1  | 0.77        | 0.43                   | $6.366 \times 10^{-7}$ | < 0.0001 |
| X <sub>2</sub> X <sub>3</sub> (X <sub>2</sub> -X <sub>3</sub> ) | 90.31          | 0.04                   | 1  | 90.31       | 0.04                   | $6.366 \times 10^{-7}$ | < 0.0001 |

LM = linear mixture, D = density, AV = acid value

**Table 2b**  
Point prediction.

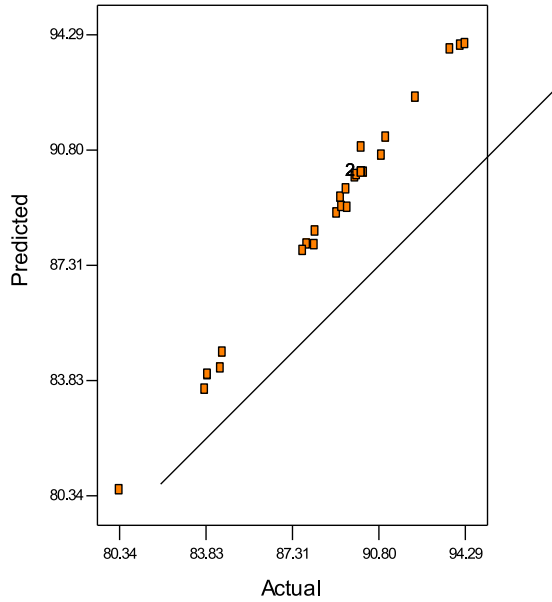
| Name           | Prediction | SE Mean | 95% CI low | 95% CI high | SE pred. | 95% PI low | 1.81   |
|----------------|------------|---------|------------|-------------|----------|------------|--------|
| Acid value     | 1.81       | 0.00    | 1.81       | 1.81        | 0.00     | 1.81       | 1.81   |
| Density        | 907.13     | 0.00    | 907.13     | 907.13      | 0.00     | 907.13     | 907.13 |
| Component      | Name       | Level   | Low level  | High level  | Std. Dev |            |        |
| X <sub>1</sub> | CPO        | 0.33    | 0.00       | 1.00        | 0.00     |            |        |
| X <sub>2</sub> | CAO        | 0.33    | 0.00       | 1.00        | 0.00     |            |        |
| X <sub>3</sub> | PO         | 0.34    | 0.00       | 1.00        | 0.00     |            |        |

Final equations in term of real component:

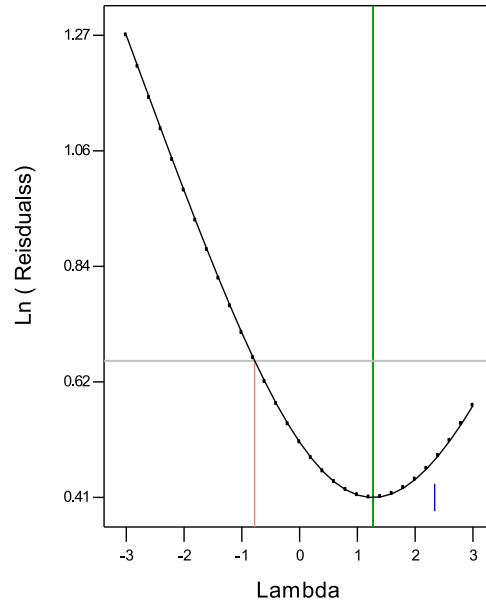
**Table 3a**  
Experimental design for FAME synthesized.

| Variables      | Units   | Symbol         | Levels |     |     |     |     |
|----------------|---------|----------------|--------|-----|-----|-----|-----|
|                |         |                | -2     | -1  | 0   | 1   | 2   |
| Reaction time  | (min)   | P <sub>2</sub> | 50     | 55  | 60  | 65  | 70  |
| MCCP amount    | (wt.%)  | P <sub>2</sub> | 3.0    | 3.5 | 4.0 | 4.5 | 5.0 |
| Reaction temp. | (°C)    | P <sub>3</sub> | 60     | 65  | 70  | 75  | 80  |
| MeOH/OMR       | (ml/ml) | P <sub>4</sub> | 3      | 4   | 5   | 6   | 7   |

MeOH/OMR = Methanol/oil molar ratio



**a Predicted against Actual**



**b Box-cox plot for power transformation**

**Fig. 3.** (a) Predicted against Actual. (b) Box-cox plot for power transformation.

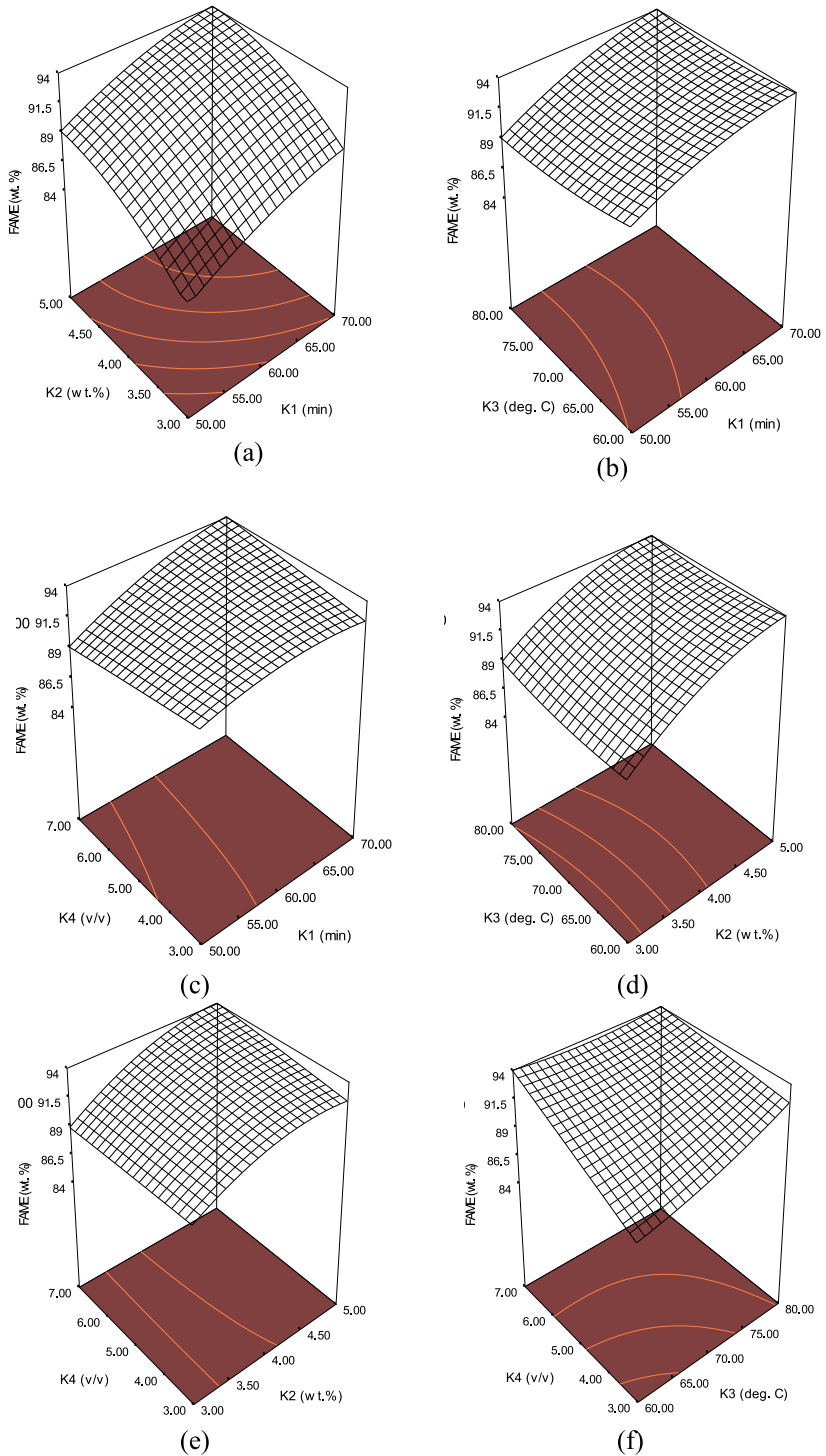


Fig. 4. (a-f): 3-D's plots.



**Table 3b**

Pro-catalytic activity of catalysts calcined at 700 °C for 4 h

| Catalysts | S <sub>BET</sub><br>(m <sup>2</sup> g <sup>-1</sup> ) | Total pore<br>volume<br>(cm <sup>3</sup> g <sup>-1</sup> ) | %CaO  | BS (μmole.g <sup>-1</sup> ) |                   |                   | TBS | BSD<br>(μmole.m <sup>-2</sup> ) |
|-----------|---|--|-------|-----------------------------|-------------------|-------------------|-----|---------------------------------|
|           |   |  |       | Weak <<br>450 °C            | Medium<br>>450 °C | Strong<br>>650 °C |     |                                 |
| CCL       | 0.80  | 5 × 10 <sup>-3</sup>                                       | 74.60 | -                           | 30                | 116               | 146 | 182.50                          |
| CMA       | 0.80  | 5 × 10 <sup>-3</sup>                                       | 62.80 | -                           | 22                | 102               | 124 | 155.00                          |
| MCCP      | 1.00  | 5 × 10 <sup>-3</sup>                                       | 78.74 | 8                           | 32                | 143               | 183 | 183.00                          |

BS = Basic site, TBS = Total basic site, BSD = Basic site density = TBS/N<sub>2</sub>-AA, CCL = Calcined Citrullus lanatus, CMA = Calcined Musa acuminata, MCCP = Mixed calcined catalyst powder

**Table 4a**

FAME result of experimental run, predicted and the residual value

| Std       | Run      | Block    | P <sub>1</sub> | P <sub>2</sub> | P <sub>3</sub> | P <sub>4</sub> | FAME (wt. %) | Predicted FAME (wt. %) | Residual    |
|-----------|----------|----------|----------------|----------------|----------------|----------------|--------------|------------------------|-------------|
| 1         | 12       | 1        | -1.000         | -1.000         | -1.000         | -1.000         | 83.90        | 83.99                  | -0.094      |
| 2         | 3        | 1        | 1.000          | -1.000         | -1.000         | -1.000         | 83.90        | 84.01                  | -0.11       |
| 3         | 6        | 1        | -1.000         | 1.000          | -1.000         | -1.000         | 88.24        | 88.34                  | -0.10       |
| 4         | 30       | 1        | 1.000          | 1.000          | -1.000         | -1.000         | 87.90        | 87.95                  | -0.049      |
| 5         | 28       | 1        | -1.000         | -1.000         | 1.000          | -1.000         | 87.74        | 87.75                  | -0.013      |
| 6         | 2        | 1        | 1.000          | -1.000         | 1.000          | -1.000         | 89.27        | 89.37                  | -0.095      |
| 7         | 29       | 1        | -1.000         | 1.000          | 1.000          | -1.000         | 91.09        | 91.19                  | -0.095      |
| 8         | 4        | 1        | 1.000          | 1.000          | 1.000          | -1.000         | 92.29        | 92.39                  | -0.10       |
| 9         | 22       | 1        | -1.000         | -1.000         | -1.000         | 1.000          | 84.42        | 84.20                  | 0.22        |
| 10        | 19       | 1        | 1.000          | -1.000         | -1.000         | 1.000          | 88.21        | 87.93                  | 0.28        |
| 11        | 11       | 1        | -1.000         | 1.000          | -1.000         | 1.000          | 90.92        | 90.64                  | 0.28        |
| 12        | 20       | 1        | 1.000          | 1.000          | -1.000         | 1.000          | 94.10        | 93.97                  | 0.13        |
| 13        | 23       | 1        | -1.000         | -1.000         | 1.000          | 1.000          | 83.79        | 83.56                  | 0.23        |
| 14        | 1        | 1        | 1.000          | -1.000         | 1.000          | 1.000          | 89.11        | 88.89                  | 0.22        |
| 15        | 8        | 1        | -1.000         | 1.000          | 1.000          | 1.000          | 89.31        | 89.08                  | 0.23        |
| <b>16</b> | <b>5</b> | <b>1</b> | <b>1.000</b>   | <b>1.000</b>   | <b>1.000</b>   | <b>1.000</b>   | <b>94.29</b> | <b>94.01</b>           | <b>0.28</b> |
| 17        | 7        | 1        | -2.000         | 0.000          | 0.000          | 0.000          | 84.50        | 84.68                  | -0.18       |
| 18        | 10       | 1        | 2.000          | 0.000          | 0.000          | 0.000          | 89.49        | 89.62                  | -0.13       |
| 19        | 27       | 1        | 0.000          | -2.000         | 0.000          | 0.000          | 80.34        | 80.51                  | -0.17       |
| 20        | 14       | 1        | 0.000          | 2.000          | 0.000          | 0.000          | 89.85        | 89.98                  | -0.13       |
| 21        | 15       | 1        | 0.000          | 0.000          | -2.000         | 0.000          | 89.92        | 90.05                  | -0.13       |
| 22        | 26       | 1        | 0.000          | 0.000          | 2.000          | 0.000          | 93.68        | 93.85                  | -0.17       |
| 23        | 18       | 1        | 0.000          | 0.000          | 0.000          | -2.000         | 89.54        | 89.06                  | 0.48        |
| 24        | 13       | 1        | 0.000          | 0.000          | 0.000          | 2.000          | 90.10        | 90.88                  | -0.78       |
| 25        | 25       | 1        | 0.000          | 0.000          | 0.000          | 0.000          | 90.20        | 90.13                  | 0.075       |
| 26        | 21       | 1        | 0.000          | 0.000          | 0.000          | 0.000          | 90.10        | 90.13                  | -0.025      |
| 27        | 24       | 1        | 0.000          | 0.000          | 0.000          | 0.000          | 90.12        | 90.13                  | -0.005      |
| 28        | 17       | 1        | 0.000          | 0.000          | 0.000          | 0.000          | 90.11        | 90.13                  | -0.015      |
| 29        | 16       | 1        | 0.000          | 0.000          | 0.000          | 0.000          | 90.12        | 90.13                  | -0.094      |
| 30        | 9        | 1        | 0.000          | 0.000          | 0.000          | 0.000          | 90.10        | 90.13                  | -0.025      |

$$AV = 0.53X_1 + 3.02 + 2.61X_3 - 0.25X_1X_2 + 0.44X_1X_3 - 3.11X_3X_2 + 2.23X_1X_3X_2 + 4.44X_1X_2(X_1 - X_2) + 5.44X_1X_3(X_1 - X_3) - 1.64X_3X_2(X_2 - X_3) \quad (2)$$

**Final equation in term of coded**

$$FAME = +90.13 + 1.23X_1 + 2.37X_2 + 0.95X_3 + 0.46X_4 - 0.10X_1X_2 + 0.40X_1X_3 + 0.93X_1X_4 - 0.23X_2X_3 + 0.52X_2X_4 - 1.10X_2X_3 - 0.74X_1^2 - 1.22X_2^2 + 0.46X_3^2 - 0.038X_4^2 \quad (3)$$

**Table 4b**

Test of significance for every regression coefficient

| Source                        | Sum of squares | df | Mean Square | F-value | P-value  |
|-------------------------------|----------------|----|-------------|---------|----------|
| Model                         | 303.37         | 14 | 21.67       | 215.57  | < 0.0001 |
| P <sub>1</sub>                | 36.61          | 1  | 36.61       | 364.15  | < 0.0001 |
| P <sub>2</sub>                | 134.52         | 1  | 134.52      | 1338.23 | < 0.0001 |
| P <sub>3</sub>                | 21.70          | 1  | 21.70       | 215.85  | < 0.0001 |
| P <sub>4</sub>                | 4.99           | 1  | 4.99        | 49.61   | < 0.0001 |
| P <sub>1</sub> <sup>2</sup>   | 15.21          | 1  | 15.21       | 151.28  | < 0.0001 |
| P <sub>2</sub> <sup>2</sup>   | 40.80          | 1  | 40.80       | 405.85  | < 0.0001 |
| P <sub>3</sub> <sup>2</sup>   | 5.72           | 1  | 5.72        | 56.90   | < 0.0001 |
| P <sub>4</sub> <sup>2</sup>   | 0.040          | 1  | 0.040       | 0.40    | 0.5361   |
| P <sub>1</sub> P <sub>2</sub> | 0.16           | 1  | 0.16        | 1.63    | 0.2209   |
| P <sub>1</sub> P <sub>3</sub> | 2.56           | 1  | 2.56        | 25.47   | 0.0001   |
| P <sub>1</sub> P <sub>4</sub> | 13.84          | 1  | 13.84       | 137.67  | < 0.0001 |
| P <sub>2</sub> P <sub>3</sub> | 0.84           | 1  | 0.84        | 8.33    | 0.0113   |
| P <sub>2</sub> P <sub>4</sub> | 4.39           | 1  | 4.39        | 43.66   | < 0.0001 |
| P <sub>3</sub> P <sub>4</sub> | 19.36          | 1  | 19.36       | 192.60  | < 0.0001 |
| Residual                      | 1.51           | 15 | 0.10        | -       | -        |
| Lack of Fit                   | 1.50           | 10 | 0.15        | 104.94  | 0.3072   |
| Pure Error                    | 0.00715        | 5  | 0.0014      | -       | -        |
| Cor Total                     | 304.88         | 29 |             |         |          |

| Fits statistics     |        |
|---------------------|--------|
| R squared           | 99.51% |
| Adjusted R squared  | 99.04% |
| Predicted R squared | 97.16% |
| Adequate precision  | 60.219 |

**Table 5**

Properties of TMO and FAME.

| Parameter                                   | TMO    | FAME   | ASTM D6751 | EN 14214 [1] |
|---|--------|--------|------------|--------------|
| Density (kg/m <sup>3</sup> ) @ 25 °C        | 907    | 886    | -          | 860-900      |
| Viscosity @ 40 °C/ (mm <sup>2</sup> /s)     | 4.40   | 2.10   | 1.9-6.0    | 3.5-5.0      |
| Moisture content (%)                        | 0.002  | 0.001  | <0.03      | 0.02         |
| %FFA (as oleic acid)                        | 0.90   | 0.40   | 0.40 max   | 0.25 max     |
| Acid value (mg KOH/g oil)                   | 1.80   | 0.20   | 0.80 max   | 0.5 max      |
| Iodine value (g I <sub>2</sub> /100g oil)   | 98.20  | 80.52  | -          | 120 max      |
| Saponification value (mg KOH/g oil)         | 172.00 | 140.20 | -          | -            |
| Peroxide value (meq O <sub>2</sub> /kg oil) | 10.20  | 11.21  | -          | 12.85        |
| HHV (MJ/kg)                                 | 40.91  | 42.47  | -          | -            |
| Cetane number                               | 55.93  | 67.10  | 57 min     | 51 min       |
| API   | 24.51  | 28.21  | 39.95 max  | -            |
| Diesel index                                | 63.76  | 79.31  | 50.4 min   | -            |

TMO = Ternary mixed oil

## 2. Experimental Design, Materials, and Methods

Response surface methodology (Simplex lattice design) and central composite design (expert 6.0.8 trial version) were employed to determine the blend ratio and the effects of variation of reaction time, catalyst amount, reaction temperature and methanol to oil molar ratio on the FAME synthesized. Materials used include CH<sub>3</sub>OH, Ethanol, Sulphuric acid, Wij's solution, etc. (Chem-Sciences Nig. Ltd.), Cucurbita pepo-Chrysophyllum albidum-papaya seeds, Citrullus lanatus and Musa acuminata peels. Equipment used are scanning electron microscopy (SEM) to examine the surface morphology of the calcined catalysts (CaO) derived from the mixture of Citrullus lanatus and Musa acuminata peels calcined powder, energy dispersive spectroscope for determination

of elemental analysis of the samples and the quantitative composition of the catalysts, X-ray diffraction analysis equipped with  $K\alpha$  and Cu radiation source, accelerated at 20 mA and 40 kV used to determine the angular scanning electron performed in the range of  $20^\circ < 2\theta < 80^\circ$  at speed of  $2^\circ \text{C min}^{-1}$ , Fourier transform infrared spectroscopy used for determination of the presence of functional group and verify the presence of characteristic absorption bands of CaO, and QUANTACHROME, 1 KE, BET isothermal sorption was used to determination of the surface area of the catalysts through  $\text{N}_2$ -adsorption  $\text{CO}_2$  TPD thermal.

Cucurbita pepo-Chrysophyllum albidum-papaya seeds were washed with deionized water to remove dirt's, sun dried for 15 days until a constant weight was achieved before milled to powders.

The solvent extraction method by the Soxhlet apparatus was used for oil extraction from the powders. 100 g each powder was measured, tightly placed in a muslin bag, and the solvent, n-hexane was measured into the round bottom flask of Soxhlet extractor. A 4-place combo heating mantle unit was loaded with four 500 ml capacity Soxhlet extractors. The reaction time was 60 min and the heating temperature was adjusted to the temperature range of  $68\text{--}70^\circ \text{C}$ . At the end of the reaction, excess n-hexane in the extracted oil was recycled using an evaporator. The percentage of oil-free of n-hexane was determined using the ratio of the Eq. (4)

$$\text{Oil yield \% (v/v)} = \frac{W_{\text{OIL}}}{W_{\text{POWDER}}} \times 100 \quad (4)$$

Ternary oil blend was carried out by using three variables (Cucurbita pepo oil, Chrysophyllum albidum oil, and papaya oil) as input factors and two response variables (density and acid value). The simplex lattice design predicted a ratio of 33:33:34 ternary blend, this was used for oil mixed and the oil was kept in the jar.

Citrullus lanatus and Musa acuminata peels were washed to remove dirt, then oven-dried to constant weight in an electrical oven. The dried peels were milled into powders, separated into smaller particle sizes using a mesh strainer (mesh size:  $125 \text{ mm--}20 \mu\text{m}$ ) to aid calcination. Each of the powder and the blend (100 g Citrullus lanatus peel powder + 100 g Musa acuminata peel powder) were calcined at  $700^\circ \text{C}$  for 4 h in an electrical furnace. After cooling, the calcined powders were characterized using scanning electron microscopy, energy dispersive spectroscopy, X-ray diffraction analysis equipped with  $K\alpha$  and Cu radiation source, accelerated at 20 mA and 30 kV, with angular scanning electron performed in the range of  $20^\circ < 2\theta < 80^\circ$  at speed of  $2^\circ \text{C min}^{-1}$ , Fourier transform infrared spectroscopy, and BET isothermal adsorption and Hammett indicator method [12].

For FAME synthesized, the predicted acid value of 1.81 mg KOH/g oil was validated as 1.80 mg KOH/g oil (FFA = 0.90) through the design, the ternary mixture of the oil (TMO) containing 33:33:34 of PO: CAO: PO blend meets the require conditions for biodiesel production via transesterification with catalyzed methanolysis of derived based catalyst CaO (d-CaO). FAME was synthesized through the procedure employed by [12] with little modifications on data factor varied and catalyst reusability steps as follows: A three-necked-reactor was used to carry out the FAME production, a total of thirty experimental runs was generated and carried out via four variable factors were considered namely; reaction time of 50-70 min, MCCC amount 3.0-5.0 (wt.), reaction temperature of  $60\text{--}80^\circ \text{C}$ , and MeOH/OMR of 3-7, respectively. Initially, 80 ml of the oil was preheated at  $60^\circ \text{C}$  for 1 h, a measured catalyst amount was added to a measured volume of methanol in 250 ml flask, heated at  $65^\circ \text{C}$  for 20 min, and then transferred into the preheated oil in the reactor, and the reaction was monitored for a period of time until it reaches completion. At the end of the reaction, the catalyst was separated by decantation and the biodiesel phase was separated from the methanol phase by separating funnel. The leach catalyst in the biodiesel was removed by washing with a mixture of 2.0 g  $\text{NaCO}_3$  and 40 ml ethanol thermally heated for 2 h under agitation. The mixture was filtered, washed with distilled water trice before the separation of biodiesel through gravity settling was carried out. Washed biodiesel was then dried over anhydrous  $\text{Na}_2\text{SO}_4$ , and then separated by filtration to obtain pure biodiesel (FAME).

For catalyst reusability, the derived CaO was recycled for reuse at the end of the reaction with reduction in the 4th, 5th and 6th cycle. Hence, the catalyst reusability was stopped after

3rd usage. For experimental design for FAME synthesized, a central composite design was used to generate a total of 30 (thirty) experimental runs, which includes the plus and minus axial points, plus and minus factorial points and the central-point with factors low and high entered in terms of alpha. For every combination of categorical factor levels, central composite design was duplicated.

The density, viscosity, the moisture content, acid value, the iodine value, and the peroxide value of the mixed oil were higher than the FAME values confirming that the synthesized product is consistent with biodiesel and that the conversion of mixed oil to FAME was complete with negligible resistance to flow and reduce internal drag in engine.

The ternary mixed ratio of oil is shown in Table 1b and Fig. 1. The SEM image and FTIR of the calcined catalysts and the mixed catalyst are shown in Fig. 2(a-b). Compositions of the calcined catalyst by XRD and BET sorption are listed in Table 3. Variable factors, the experimental yield, and the predicted value data for FAME are illustrated in Table 4a, while the test of significant and fits statistics by CCD optimization are shown in Table 4b. Fig 4(a-b), displayed the predicted against the actual FAME yield, as well as the three-dimensional plots that exist between the four factors and the FAME response. Table 5 provides the properties of the ternary mixed oil (TMO) and the FAME produced.

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## Conflict of interest

The authors declare that they have no competing financial interests or personal relationships which have, or could be perceived to have, influenced the work reported in this article.

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## Supplementary material

Supplementary material associated with this article can be found, in the online version, at doi:[10.1016/j.dib.2020.105514](https://doi.org/10.1016/j.dib.2020.105514).

## References

- [1] E. Betiku, T.F. Adepaju, Sorrel (Hibiscus sabdariffa) Seed Oil Extraction Optimization and Quality Characterization, *American Chemical Science Journal* 3 (4) (2013) 449–458.
- [2] ASTM D6751- Standard Test Method for Gross Calorific Value of Oil, Water, Coal and Coke by the adiabatic Bomb Calorimeter from SAI Global.
- [3] EN14214- European committee for standardization describing the requirements and test methods for FAME
- [4] E. Betiku, T.F. Adepaju, K.O. Akinbiyi, E.A. Seyi, Statistical Approach to the Optimization of Oil from Beniseed (*Sesamum indicum*) seed oil, *JFSE* 2 (2) (2012) 351–357.
- [5] F. Sharifah, Sy. Mohamad, S. Mohamad, Z. Jemaat, Study of calcination condition on decomposition of calcium carbonate in waste cackle shell to calcium oxide using thermal gravimetric analysis, *ARPN JEAS* 11 (16) (2016) 9917–9921.
- [6] Kostic M.D., V.V. Ana, M.J. Natasa, S.S. Olivera, B.V. Vlada, Optimization and kinetic modeling of esterification of the oil obtained from waste plum stones as a pretreatment step in biodiesel production, *Waste Manag* 46 (2016) 619–629.
- [7] M.D. Joana, A.F. Conceicao, F.A. Manuel, Using mixtures of waste frying oil and pork lard to produce biodiesel, *World Academ of Scienc, Engineer and Tech* 44 (2008) 258262.

- [8] I. Khalil, A.R.A. Aziz, S. Yusup, M. Heikal, M. El-Adawy, Response surface methodology for the optimization of the production of rubber seed/palm oil biodiesel, IDI diesel engine performance, and emissions, *Biomass Conversion and Biorefinery* 7 (2017) 37–49.
- [9] F. Qiu, Y. Li, D. Yang, X. Li, P. Sun, Biodiesel production from mixed soybean oil and rapeseed oil, *Applied Energy* 88 (2011) 2050–2055.
- [10] K.V. Yathish, R. Suresh, E. Amruth, Optimization of Biodiesel production from mixed oil (Karanja & Dairy waste Scum oil) using Homogeneous Catalyst, *IOSR Journal of Applied Chemistry (IOSR-JAC)* 3 (6) (2016) 9–15.
- [11] J. Milano, H.C. Ong, H. Masjuki, A. Silitonga, W-H. Chen, F. Kusumo, S. Dharma, A. Sebayang Optimization of biodiesel production by microwave irradiation-assisted transesterification for waste cooking oil-*Calophyllum inophyllum* oil via response surface methodology, *Energy Conversion and Management* 158 (2018) 400–415.
- [12] G. Minakshi, L. Khairujjaman, K.P. Atanu, D. Niran, M. Mrutyunjay, K.G. Imon, H. Anil, B. Utpal, D. Dhanapati, Carica papaya stem: A source of versatile heterogeneous catalyst for biodiesel production and CeC bond formation, *Renewable Energy* 147 (2020) 541–555.