

# Optimization of biodiesel production from coconut oil using a bifunctional catalyst derived from crab shell and coconut shell

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## ABSTRACT

The utilization of renewable biomass resources for biofuel production has gained widespread popularity as an effective means of converting waste into a valuable resource. This study investigated the optimization of biodiesel production from coconut oil (CO) using a bifunctional catalyst derived from crab shell and coconut shell. The catalyst underwent characterization through Fourier transform infrared spectroscopy, scanning electron microscopy, X-ray diffraction, and Brunauer-Emmett-Teller. The acid precursor for the bio-based bifunctional catalyst was created from carbonized and sulfurized coconut shell, while the base precursor was obtained from calcined and KOH-treated crab shell. The experimental design was optimized using Box-Behnken design for the simultaneous esterification and transesterification of CO. Response surface methodology was employed to model and optimize the process. Under the determined ideal conditions, which included a methanol to oil ratio of 16.75:1, a 1.00% catalyst loading, a temperature of 58.19 °C, and a reaction time of 78.76 minutes, a CO methyl ester yield of 85.73% was achieved under experimental conditions. This yield closely aligned with the predicted range of 85.84% as per the model. The resulting methyl ester (biodiesel) obtained under these optimized conditions exhibited physicochemical properties well within the requirements specified by ASTM D6751 set by the International Organization of Standardization.

**Keywords:** biodiesel, biomass, biofuels, transesterification, bifunctional catalyst

## INTRODUCTION

Energy assumes a critical role in the everyday lives of individuals worldwide. The availability of energy is vital for domestic, industrial, and transportation activities (Ifeanyi-Nze & Akhiehiro, 2023). The energy demand within these

sectors heavily depends on petroleum-based fuels, which have been linked to numerous negative consequences, rendering their ongoing utilization unsustainable (Perin & Jones, 2019). Consequently, there is an increasing movement within the research and policymaking communities to investigate eco-friendly and sustainable alternatives. Among the various options being examined, biofuels have emerged as a feasible

choice to address worldwide energy needs while also alleviating the adverse impacts linked to the use of petroleum-based fuels (Ifeanyi-Nze & Akhiehiro, 2023).

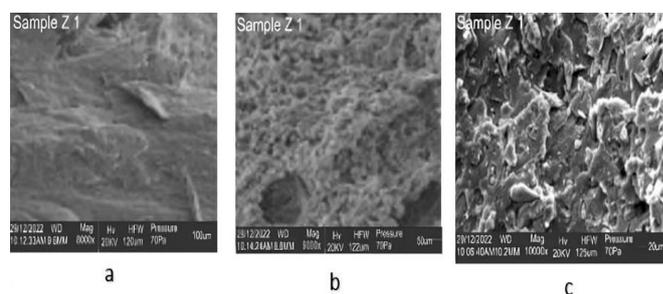
Biofuels, encompassing biogas, bioethanol, and biodiesel, constitute a class of fuels derived from biomass or organic matter. According to Wang et al. (2021), these fuels are widely acknowledged for their environmentally friendly characteristics for several reasons, including their capacity to produce minimal pollutant emissions, their biodegradability, and their renewable nature.

Biodiesel is traditionally manufactured from vegetable oils through a procedure known as transesterification. During this process, the triglycerides found in the oil react with a compatible alcohol, like methanol, in the presence of a catalyst. This chemical conversion results in the formation of fatty acid alkyl esters, commonly referred to as biodiesel (Sharma & Baredar, 2018). As a usual result of this reaction, glycerol is produced as a by-product, which is then separated from the resulting biodiesel using a separation method. The biodiesel goes through additional purification stages before it becomes suitable for use and storage (Atadashi et al., 2011).

The extensive acceptance of biodiesel as a feasible transportation fuel has encountered obstacles primarily influenced by economic factors. In particular, the expense of the raw materials employed in biodiesel production has been identified as a substantial component of the overall production expenses (Mahlia et al., 2020). The majority of seeds are grown mainly for their oil and for human use. The oil from most seeds is edible (for example, groundnut oil, fluted pumpkin seed oil, and soya bean seed oil), and approximately 80.00% of the world's vegetable oil production is for human consumption, while the remaining 20.00% is shared between animal agriculture and chemical processing industries (biofuel production) (Baud et al., 2018). Due to the high need for oil for both consumer and industrial purposes, hence the need for non-edible oil as bio lubricant feedstock. To position biodiesel as a competitive alternative fuel capable of satisfying worldwide energy requirements, it is crucial to tackle the elements that contribute to elevated production expenses. Therefore, the adoption of economically sustainable non-edible oils, like waste vegetable oil (Jaiswal et al., 2022; Topi, 2020), jatropha (Palitsakun et al., 2021), rubber seed oil (Sugebo et al., 2021), kapok (Pooja et al., 2021), castor seed (Keera et al., 2018), yellow oleander (Deka & Basumatary, 2011), assumes paramount importance.

Furthermore, the choice and affordability of catalysts used are pivotal in influencing the economics of the biodiesel production procedure. Conversely, the adoption of cost-efficient and exceptionally effective solid catalysts presents a promising path for progress (Alagumalai et al., 2021). Solid catalysts have attracted substantial research focus owing to their unique advantages, such as thermal stability, reusability, lack of corrosion, and avoidance of saponification (Bing & Wei, 2019).

Researchers have noted the use of crab shells as a traditional heterogeneous alkaline catalyst for biodiesel production (Shankar & Jambulingam, 2017; Vogt & Weckhuysen, 2022). Its efficiency has been thoroughly established and confirmed over time. Boey et al. (2009)



**Figure 1.** Calcined crab shell at (a) 8,000x; (b) 9,000x; & (c) 10,000x (Source: Authors' own elaboration)

examined biodiesel production through the transesterification of palm olein using waste mud crab (*scylla serrata*) shells as a heterogeneous catalyst. Characterization of the shells showed that their primary component was calcium carbonate, which converted to calcium oxide when activated at temperatures exceeding 700 °C for two hours. It was also discovered that the waste crab shell catalyst exhibited similar performance to laboratory-grade CaO with a yield of 91.00%, providing an alternative, cost-effective catalyst source for biodiesel production. This aligns with the findings of another study conducted by Karkal et al. (2023), where they utilized portunus sanguinolentus crab shells to create a heterogeneous base catalyst for the transesterification of marine fishmeal plant oil. In their study, they achieved a remarkable maximum biodiesel yield of 88.56% under specific conditions, including a 1:9 (mol/mol) oil to methanol molar ratio, a 3.00% concentration of calcined crab shell catalyst (**Figure 1**), a reaction temperature of 60 °C, and a reaction time of 60 minutes. Furthermore, the investigations conducted on the biodiesel produced from waste crab shells in their study confirmed its feasibility based on its physicochemical characteristics. Notably Karkal et al. (2023) also reported on the catalyst's reusability, demonstrating its favorable potential for reuse for up to four cycles, with only a minor 10.00% reduction in catalytic activity. This highlights the catalyst's significance in promoting sustainable biodiesel production.

Conversely, scientists have determined that a catalyst derived from coconut shells displays exceptional efficiency in biodiesel production, particularly in the transesterification processes. From literature, Endut et al. (2017) conducted an optimization study for biodiesel production using a solid acid catalyst derived from coconut shells through response surface methodology (RSM). The study resulted in a significant biodiesel yield of 88.03%, demonstrating that coconut shell-derived solid acid catalysts contribute to environmentally sustainable biodiesel production methods. While researchers have separately explored the use of crab shell and coconut shell as heterogeneous catalysts, their combined application as a bifunctional catalyst for concurrent esterification and transesterification of oils has been relatively unexplored.

The findings of this investigation are consistent with earlier studies showing efficiency of bifunctional catalyst in simultaneously esterifying and trans esterifying seed oils to produce high yield of biodiesel. Akhabue et al. (2022) recorded a high biodiesel yield of 97.21% after utilizing a bifunctional catalyst gotten from ferric acid doped with poultry droppings in simultaneously esterifying and trans esterifying neem seed oil.

Also, in another study conducted by Akhabue et al. (2020) showed a high biodiesel yield of 92.89% after a novel bio-based bifunctional catalyst was prepared from sulfonated calcined corncobs and calcined poultry droppings by wet impregnation method.

In this research, we evaluated the catalytic performance of a bifunctional catalyst derived from crab shell and coconut shell. We combined the crab shell, which served as the base precursor with the coconut shell, which served as the acid precursor and conducted a thorough investigation. The utilization of these waste materials from the environment in the production of biodiesel catalysts presents an economical alternative, reducing the necessity to acquire costly catalysts for biodiesel synthesis. The only cost incurred is that of calcination (Gebremariam & Marchetti, 2018). Different factors that impact the outcome of biodiesel production, such as the methanol-to-oil ratio, catalyst quantity, reaction temperature, and duration, were fine-tuned using RSM in conjunction with Box-Behnken design (BBD). The optimized outcomes generated by BBD were then analyzed to evaluate the operating parameters in modeling and enhancing the production process.

The primary objective of this study was to evaluate the efficacy of RSM as a robust modeling and optimization tool for enhancing the transesterification process. Specifically, we aimed to assess the applicability of RSM in optimizing the simultaneous esterification and transesterification reactions in biodiesel production, using coconut oil (CO) as the raw material. A significant focus of this research was also directed toward investigating the advantages of employing a novel bifunctional catalyst derived from waste crab shell and coconut shell. This bifunctional catalyst was explored for its simultaneous esterification and transesterification capabilities, thereby offering a sustainable solution for biodiesel synthesis. The study comprehensively examined the modeling capabilities of RSM in predicting optimal reaction conditions. Furthermore, the resulting biodiesel's quality, synthesized using the bifunctional catalyst, was meticulously compared with established biodiesel standards to ensure its viability for commercial and industrial applications. This research contributes valuable insights to the fields of process optimization, waste utilization, and sustainable biodiesel production.

## METHODOLOGY

### Materials

The materials and equipment used in this study included distilled water, CO, methanol, sulfuric acid, acetic acid, chloroform, phenolphthalein indicator, starch indicator, lithium carbonate, crab shell, coconut shell, benzene, ethanol, a magnetic stirrer, conical flask, separating funnel, droppers, measuring cylinder, desiccator, beakers, oven, laboratory weighing balance, sieve, volumetric flask, and analytical instruments such as Brunauer-Emmett-Teller (BET), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and X-ray diffraction (XRD).

### Characterization of Coconut Oil

CO was characterized according to ASTM D6751 and EN 14214 protocols.

### Catalyst Preparation

#### *Preparation of precursor from crab shell*

The crab shells were cleaned by washing them repeatedly in warm distilled water to remove sand and other contaminants. They were then smashed into manageable chunks using a hammer. Subsequently, the crab shells underwent a final rinse in hot distilled water to ensure the removal of any remaining oil. After being exposed to sunlight for three days, the crab shells were baked at 110 °C for two hours to complete the drying process. The resulting powdered crab shell was sieved through a 0.3 mm screen Endecott's microplate sieves, and any unsaved particles were discarded.

Purified CaO was obtained by calcining the finely sieved crab shells in a muffle furnace at 900 °C for four hours (Correia et al., 2014). After calcination, the crab shell was promptly transferred to a desiccator to prevent further reactions with air. Once it had cooled, it was stored in an airtight container until it could be utilized or analyzed.

#### *Preparation of precursor from coconut shell*

The coconut shells were broken apart and cleaned in distilled water to remove any lingering debris or mud. To ensure the elimination of any remaining moisture, the coconut shells were sun-dried for a period of 14 days after undergoing thorough washing. After the drying process, the coconut shells were crushed and sieved through a 0.3 mm screen Endecott's microplate sieves to remove any remaining moisture. The finely sieved coconut shell was carbonized by heating it to 500 °C for three hours in a benchtop Thermo-scientific muffle furnace, resulting in ash formation. The carbonized coconut shell was carefully removed from the furnace and placed in a desiccator to prevent any further reactions with the air. It was stored in a sealed container for future use and analysis.

#### *Synthesis of heterogeneous composite catalyst*

The wet impregnation method was employed for the preparation of the catalyst by blending samples of crab shell and coconut shell to create a homogeneous slurry solution as described by Lani et al. (2016), and Akhiero et al. (2013). The blending process, lasting approximately 30 minutes, ensured thorough mixing of the two shell components. Subsequently, the slurry solution underwent heating on a magnetic stirrer, reaching a temperature of 80 °C, and was stirred until complete evaporation of water was achieved. Upon formation of the slurry, the resulting mixture was subjected to a drying process in a 110 °C oven for three hours, resulting in the formation of a solid material. This freshly prepared solid, comprising crab shell and coconut shell components, served as the precursor for the catalyst. To activate and enhance the catalytic properties of the precursor, a calcination step was employed. The precursor underwent calcination at 800 °C for a duration of three hours, facilitating the reactivation of the catalyst. This thermal treatment ensured the removal of any residual organic matter and promoted the development of a stable and effective catalytic material for subsequent applications.

### Catalyst characterization of coconut shell & crab shell

A number of analytical methods were used to characterize and analyze the prepared catalyst to ensure that it met all of the required specifications. These methods included FTIR for analyzing the functional group in the sample using the Shimadzu FTIR spectrometer, SEM for analyzing the surface morphology using the Thermo Fisher Scientific Scanning Electron Microscopes, XRD for analyzing the crystalline compounds in the catalyst and precursors using the Bruker X-Ray Diffractometers and BET for analyzing the surface area using BELSORP-mini-X sorption analyzer.

### Experimental Design & Response Surface Methodology

In this study, a BBD was utilized to systematically design a concurrent esterification and transesterification process for the conversion of carbon monoxide into biodiesel. The bi-functional catalyst, derived from a combination of crab shells and coconut shells, was employed. BBD was chosen for its suitability in modeling quadratic response surfaces, a common requirement in chemical engineering processes. Four key variables—methanol-to-oil molar ratio, catalyst concentration, reaction time, and temperature—were investigated for their significant impact on biodiesel production, with specified ranges outlined. The data from the 29 experiments conducted using BBD were analyzed using a quadratic regression model (Eq. [1]), where model terms were identified through multiple regression analysis, and their significance assessed through analysis of variance (ANOVA). The experimental design and statistical analysis were executed using design expert software version 13.0. The outcomes of the experiments facilitated the establishment of a mathematical model using response surface regression, incorporating a second-order polynomial equation, as depicted in Eq. (1).

$$Y = \sum b_i x_i + \sum b_{ij} x_i x_j + \sum b_{ii} x_i^2 + e_i \quad (1)$$

In Eq. (1),  $x_i$  and  $x_j$  represent the independent variables, while  $Y$  denotes the dependent variable, which is the biodiesel yield.  $b_i$  and  $b_{ij}$  refer to the coefficients for single and interaction effects, respectively, and  $e_i$  represents the experimental error term.  $b_0$  is the offset term.

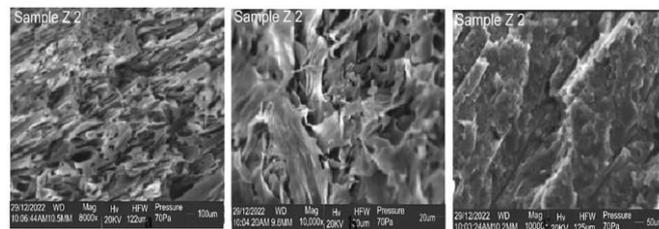
### Simultaneous Esterification & Transesterification of Coconut Oil

The process of simultaneous esterification and transesterification of CO was carried out in a 500 ml flask with two necks. This flask was equipped with a reflux condenser and a temperature probe and placed on a magnetic stirrer set at a constant temperature. We added a predetermined amount of catalyst to 50 g of CO that had been preheated in the flask. Then, we introduced the necessary quantity of methanol, which contained the catalyst, into CO. To prevent any loss of methanol, the reflux condenser captured and condensed any methanol that escaped when the temperature reached or exceeded 60 °C, returning it to the reaction mixture.

After each step, the resulting product mixture underwent separation from the catalyst using a centrifuge machine. Subsequently, we separated the biodiesel from the glycerol and excess methanol using a separating funnel. Following this separation process, the biodiesel was subjected to washing,

**Table 1.** Physiochemical properties of coconut oil

| Properties                   | Values  |
|------------------------------|---------|
| Acid value (mg KOH/g)        | 5.800   |
| FFA (%)                      | 2.900   |
| Saponification value         | 190.000 |
| Molecular weight (g)/mol     | 913.681 |
| Peroxide value               | 10.000  |
| Viscosity at 31 °C (MPa.S)   | 26.530  |
| Density (kg/m <sup>3</sup> ) | 925.00  |
| Moisture content (%)         | 0.060   |



**Figure 2.** Bifunctional catalyst containing mixture of crab shell & coconut shell at (a) 8,000x; (b) 9,000x; & (c) 10,000x (Source: Authors' own elaboration)

drying, and finally, it was stored in an airtight container, following the procedures outlined by (Akhabue & Falowo, 2019). To determine the biodiesel yield, we utilized Eq. (2).

$$\text{Biodiesel yield} = \frac{\text{Amount of biodiesel}}{\text{Amount of coconut oil}} \quad (2)$$

The properties of the biodiesel, including its kinematic viscosity, acid value, moisture content density, and iodine value were assessed using established standard methods.

## RESULTS

### Physiochemical Properties of Coconut Oil

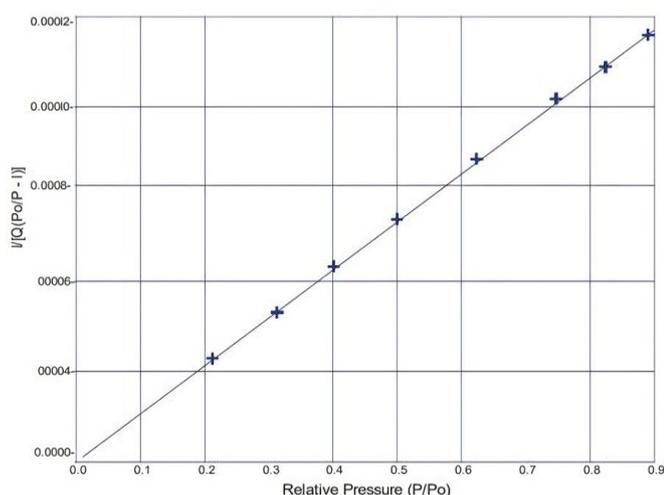
According to ASTM standards, the physiochemical characteristics of CO were examined as in the prior section. **Table 1** shows the physiochemical properties of CO.

### Surface Morphology of Processed Catalyst

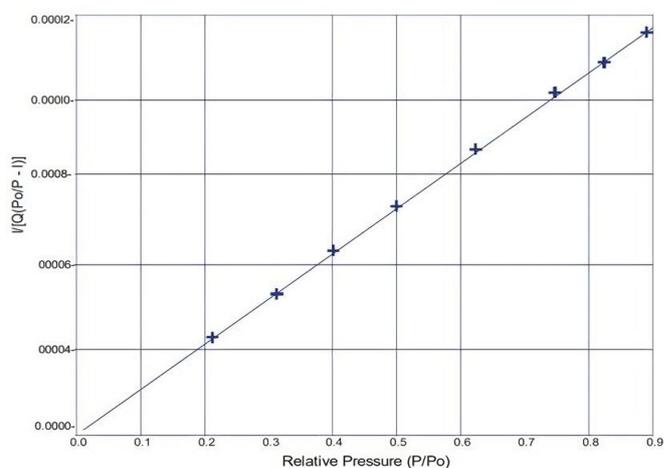
The surface morphology of the bifunctional catalyst derived from coconut and crab shells was comprehensively examined using SEM images at various magnifications. This SEM analysis provided valuable insights into the physical structure, surface area, and pore characteristics of the catalyst, all of which significantly impact biodiesel yield.

As illustrated in **Figure 2**, SEM images revealed a catalyst with well-defined pores and abundant active sites. The presence of these active sites, coupled with the porous structure, facilitated efficient esterification and transesterification reactions.

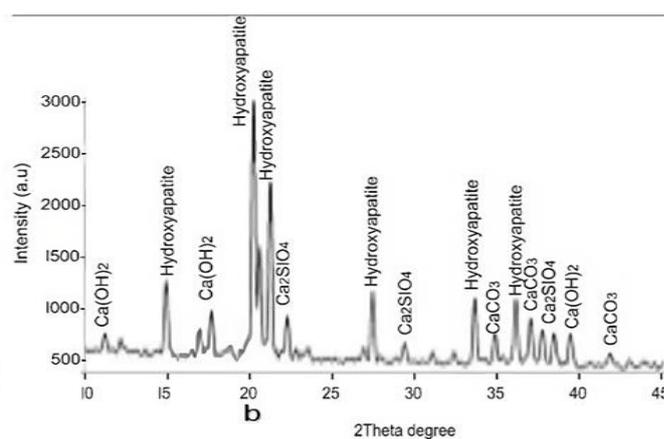
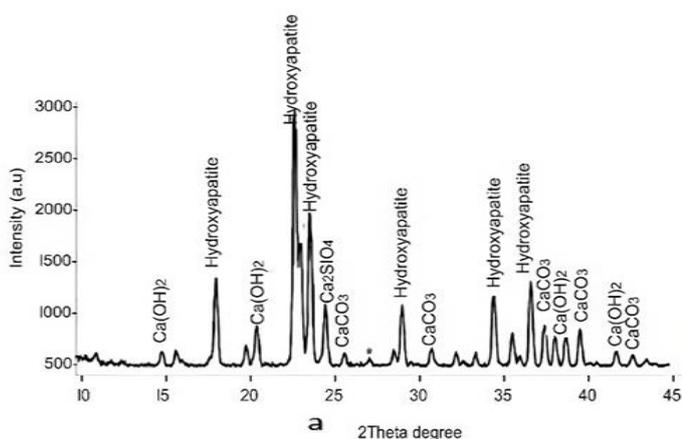
Despite constraints posed by unevenly distributed porous spaces, the catalyst exhibited a remarkable ability to promote reactant interactions, resulting in a notably high biodiesel yield. The findings from this analysis substantiate the catalyst's outstanding performance in achieving a significant biodiesel yield.



**Figure 3.** BET surface area plot of crab shell (Source: Authors' own elaboration)



**Figure 4.** BET surface area plot of synthesized bifunctional catalyst (Source: Authors' own elaboration)



**Figure 5.** XRD analysis plot for (a) crab shell; & (b) synthesized bifunctional catalyst (Source: Authors' own elaboration)

Consequently, the use of this bifunctional catalyst was confirmed to be superior to employing solely alkaline or acidic catalysts in biodiesel production.

#### Pore Volume, Surface Area, & Pore Diameter of Catalyst

The surface area of a catalyst plays a pivotal role in its catalytic activity, with the concept of active sites being central to understanding catalytic processes. Active sites represent specific surface atoms or groups of atoms with distinct structural and property characteristics, where catalytic transformations occur. In this study, the synthesized catalyst exhibited a BET surface area of  $268.330 \text{ m}^2/\text{g}$ , while the surface area of the calcined crab shell measured at  $265.720 \text{ m}^2/\text{g}$  as seen in **Figure 3** and **Figure 4**.

Notably, the synthesized catalyst displayed a larger surface area compared to the calcined crab shell, signifying its potential for heightened catalytic activity. The larger surface area of the bio-based catalyst suggests a greater number of active sites available for catalytic reactions, enhancing its suitability for biodiesel production.

#### X-Ray Diffraction Analysis

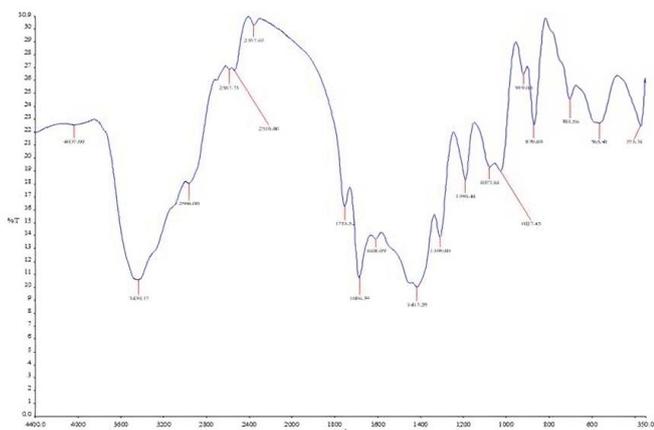
XRD analysis was conducted to investigate the crystalline structures of the produced bifunctional catalyst and calcined crab shell, the results of which are presented in **Figure 5**.

Part a in **Figure 5** illustrates XRD plot of crab shells, revealing four distinctive peaks corresponding to various minerals. Notably, portlandite peaks were observed at  $2\theta$  angles of  $14.08^\circ$ ,  $21.00^\circ$ ,  $38.00^\circ$ , and  $42.00^\circ$ , while calcite exhibited peaks at  $2\theta$  angles of  $26.00^\circ$ ,  $31.00^\circ$ ,  $35.00^\circ$ ,  $39.75^\circ$ , and  $43.00^\circ$ . Hydroxyapatite minerals were identified with peaks at  $2\theta$  angles of  $16.78^\circ$ ,  $23.24^\circ$ ,  $23.87^\circ$ ,  $28.45^\circ$ ,  $34.00^\circ$ , and  $37.16^\circ$ .

Part b in **Figure 5** presents XRD plot for the synthesized catalyst crystals, where four distinct peaks were observed, corresponding to portlandite, calcite, hydroxyapatite, and  $\text{CaSiO}$ . The portlandite peaks were identified at  $2\theta$  angles of  $12.00^\circ$ ,  $21.00^\circ$ , and  $39.00^\circ$ , while calcite exhibited a peak at  $2\theta=35.00^\circ$ . Hydroxyapatite minerals were observed at  $2\theta$  angles of  $15.00^\circ$ ,  $21.23^\circ$ ,  $22.00^\circ$ ,  $23.00^\circ$ ,  $27.00^\circ$ ,  $34.00^\circ$ , and  $37.16^\circ$ . The presence of these minerals in both the crab shell and the catalyst highlights the catalyst's mineralogical composition and its potential for catalytic activity in biodiesel production.

#### Fourier Transform Infrared Spectroscopy

In **Figure 6**, FTIR spectra are presented, providing essential insights into the functional groups present on the surface of the catalyst. The analysis revealed distinctive peaks in the range of  $373.31\text{-}1919.00 \text{ cm}^{-1}$ , attributed to  $\text{CaO}$



**Figure 6.** FTIR spectrum (Source: Authors' own elaboration)

vibrations, indicating the presence of active calcium oxide species on the unused catalyst. Additional peaks between 2,966.00 and 4,037.80  $\text{cm}^{-1}$  were consistent with O-H stretching vibrations, indicating hydroxyl groups on the catalyst's surface. A notable signal at 2,357.63  $\text{cm}^{-1}$  was observed, likely corresponding to carbonate C-O bonds, highlighting the catalyst's composition. Furthermore, the presence of the peak at 1686.39  $\text{cm}^{-1}$  indicated the presence of the carbonyl functional group (C=O). These findings offer valuable insights into chemical composition and functional groups present on the catalyst's surface, underlining its potential for catalytic activity in biodiesel production.

#### Response surface methodology analysis

The outcomes of the 29 biodiesel production experiments are presented in **Table 2**. Following the estimation of the coefficients for the model terms (expressed in terms of coded factors), Eq. (3) represents the resulting equation. **Table 2** displays the predicted biodiesel yield using Eq. (3) for comparison with the experimental results. The model's effectiveness is evident from the close alignment between the predicted and experimental yields, which ranged from 60.75% to 85.86%.

$$\begin{aligned} \text{COME yield} = & 78.524 + 1.94167A + \\ & 10.4907B + 1.55833C - 0.00841667D - \\ & 0.5825AB + 0.8575AC - 1.25AD - 0.885BC + \\ & 1.62275BD - 1.2325CD - 0.266625A^2 - \\ & 4.44525B^2 - 0.396625C^2 - 0.34525D^2, \end{aligned} \quad (3)$$

where Y is COME yield, A is MeOH: oil (mol/mol), B is temperature ( $^{\circ}\text{C}$ ), C is catalyst loading (weight percent), and D is time (min).

Examining Eq. (3), it becomes evident that factors A, B, C, AC, and BD contribute positively to increasing COME yield, as indicated by their positive coefficients. Conversely, factors D, AD, BC, CD,  $A^2$ ,  $B^2$ ,  $C^2$ , and  $D^2$  exhibit negative coefficients.

To anticipate the reaction at specific factor levels, one can utilize an equation expressed in terms of real factors. It's essential to express the levels for each factor in their original units. However, it's crucial to note that this equation should not be employed to gauge the relative significance of each factor. This is because the coefficients are scaled to

**Table 2.** Results from experiments & predictions for COME yield

| RN | MeOH   | T       | CL       | TI      | ACOME | PCOME |
|----|--------|---------|----------|---------|-------|-------|
| 1  | 6 (-1) | 50 (0)  | 1.0 (0)  | 60 (-1) | 75.73 | 74.73 |
| 2  | 18 (1) | 50 (0)  | 0.5 (-1) | 75 (0)  | 76.63 | 77.39 |
| 3  | 12 (0) | 50 (0)  | 1.0 (0)  | 75 (0)  | 79.36 | 78.52 |
| 4  | 6 (-1) | 50 (0)  | 1.0 (0)  | 90 (1)  | 78.36 | 77.21 |
| 5  | 18 (1) | 40 (-1) | 1.0 (0)  | 60 (-1) | 80.37 | 81.11 |
| 6  | 6 (-1) | 50 (0)  | 1.0 (0)  | 75 (0)  | 60.83 | 60.80 |
| 7  | 12 (0) | 60 (1)  | 0.5 (-1) | 60 (-1) | 73.47 | 75.00 |
| 8  | 12 (0) | 50 (0)  | 1.0 (0)  | 60 (-1) | 84.20 | 82.61 |
| 9  | 18 (1) | 40 (-1) | 1.5 (1)  | 75 (0)  | 82.46 | 82.22 |
| 10 | 12 (0) | 60 (1)  | 1.0 (0)  | 90 (1)  | 59.65 | 61.61 |
| 11 | 6 (-1) | 50 (0)  | 1.0 (0)  | 75 (0)  | 81.00 | 82.94 |
| 12 | 12 (0) | 50 (0)  | 1.0 (0)  | 75 (0)  | 80.46 | 78.52 |
| 13 | 12 (0) | 50 (0)  | 1.0 (0)  | 75 (0)  | 77.60 | 78.52 |
| 14 | 12 (0) | 50 (0)  | 1.5 (1)  | 60 (-1) | 79.40 | 80.58 |
| 15 | 6 (-1) | 40 (-1) | 0.5 (-1) | 75 (0)  | 74.60 | 75.22 |
| 16 | 12 (0) | 60 (1)  | 0.5 (-1) | 75 (0)  | 61.00 | 60.75 |
| 17 | 12 (0) | 40 (-1) | 1.5 (1)  | 75 (0)  | 85.00 | 84.85 |
| 18 | 12 (0) | 40 (-1) | 1.5 (1)  | 75 (0)  | 64.54 | 65.63 |
| 19 | 18 (1) | 50 (0)  | 1.0 (0)  | 75 (0)  | 67.76 | 65.85 |
| 20 | 12 (0) | 50 (0)  | 1.0 (0)  | 75 (0)  | 77.60 | 78.52 |
| 21 | 12 (0) | 50 (0)  | 1.5 (1)  | 90 (1)  | 79.60 | 78.10 |
| 22 | 12 (0) | 50 (0)  | 0.5 (-1) | 90 (1)  | 78.60 | 77.45 |
| 23 | 6 (-1) | 50 (0)  | 1.5 (1)  | 75 (0)  | 77.00 | 76.62 |
| 24 | 12 (0) | 60 (1)  | 1.0 (0)  | 75 (0)  | 77.60 | 78.52 |
| 25 | 12 (0) | 60 (1)  | 1.0 (0)  | 90 (1)  | 84.60 | 85.84 |
| 26 | 18 (1) | 50 (0)  | 1.0 (0)  | 75 (0)  | 85.60 | 85.66 |
| 27 | 18 (1) | 40 (-1) | 1.0 (0)  | 90 (1)  | 78.00 | 78.60 |
| 28 | 12 (0) | 60 (1)  | 1.0 (0)  | 60 (-1) | 65.74 | 64.87 |
| 29 | 12 (0) | 50 (0)  | 0.5 (-1) | 75 (0)  | 85.00 | 83.50 |

Note. RN: Run no; MeOH: MeOH: oil (mol/mol); T: Temperature ( $^{\circ}\text{C}$ ); CL: Catalyst loading (weight percent); TI: Time (min); ACOME: Actual COME yield (weight percent); & PCOME: Predicted COME yield (weight percent) RSM

accommodate the units of each element, and the intercept is not centered within the design space.

The results of ANOVA for COME yield are presented in **Table 3**. The sum of squares for COME yield in ANOVA model of this study is determined to be 1,557.49, with an F-value of 40.60 and a p-value of 0.0001 ( $<0.0001$ ). ANOVA performed on COME yield outcome reveals that temperature, methanol-to-oil ratio, and the second-order effect of temperature have the most significant impact on COME yield. Temperature, in particular, exerts the greatest influence, with a sum of squares value of 1,320.67, an F-value of 481.95, and a p-value of 0.0001.

As for lack of fit F-value, which stands at 1.79, it indicates that the lack of fit is not statistically significant when compared to pure error. A significant lack of fit F-value suggests a 30.21% chance of being attributed to random noise. Therefore, given our objective of achieving a well-fitting model, a lack of fit that is not statistically significant is considered desirable.

Following the simultaneous esterification and trans esterification of CO, the proposed quadratic model boasts the highest  $R^2$  value, which amounts to 0.9760. A high  $R^2$  value, nearing unity, signifies that the fitted model predicts COME yield with a commendable level of accuracy. The discrepancy

**Table 3.** Results for ANOVA for COME yield

| Source          | SS         | df | MS         | F-value  | p-value  | Result |
|-----------------|------------|----|------------|----------|----------|--------|
| <b>Model</b>    | 1557.49    | 14 | 111.2500   | 40.6000  | <0.0001  | S      |
| A-MO            | 45.2400    | 1  | 45.2400    | 16.5100  | 0.0012   |        |
| B-T             | 1,320.6700 | 1  | 1,320.6700 | 481.9500 | < 0.0001 |        |
| C-CL            | 29.1400    | 1  | 29.1400    | 10.6300  | 0.0057   |        |
| D-Time          | 0.0009     | 1  | 0.0009     | 0.0003   | 0.9862   |        |
| AB              | 1.3600     | 1  | 1.3600     | 0.4953   | 0.4931   |        |
| AC              | 2.9400     | 1  | 2.9400     | 1.0700   | 0.3178   |        |
| AD              | 6.2500     | 1  | 6.2500     | 2.2800   | 0.1532   |        |
| BC              | 3.1300     | 1  | 3.1300     | 1.1400   | 0.3030   |        |
| BD              | 10.5300    | 1  | 10.5300    | 3.8400   | 0.0701   |        |
| CD              | 6.0800     | 1  | 6.0800     | 2.2200   | 0.1586   |        |
| A <sup>2</sup>  | 0.4611     | 1  | 0.4611     | 0.1683   | 0.6879   |        |
| B <sup>2</sup>  | 128.1700   | 1  | 128.1700   | 46.7700  | <0.0001  |        |
| C <sup>2</sup>  | 1.0200     | 1  | 1.0200     | 0.3724   | 0.5515   |        |
| D <sup>2</sup>  | 0.7732     | 1  | 0.7732     | 0.2822   | 0.6036   |        |
| <b>Residual</b> | 38.3600    | 14 | 2.7400     |          |          |        |
| Lack of fit     | 31.3600    | 10 | 3.1400     | 1.7900   | 0.3021   | NS     |
| Pure error      | 7.0100     | 4  | 1.7500     |          |          |        |
| <b>Total</b>    | 1,595.85   | 28 |            |          |          |        |

Note. SS: Sum of squares; MS: Mean square; A-MO: A-Methanol: oil ratio; B-T: B-Temperature; C-CL: C-Catalyst loading; S: Significant; & NS: Not significant

**Table 4.** Evaluation of RSM performance

| Parameter               | RSM        |
|-------------------------|------------|
| R                       | 0.98240606 |
| R <sup>2</sup>          | 0.96030000 |
| Adjusted R <sup>2</sup> | 0.95368333 |
| MSE                     | 1.32153383 |
| RMSE                    | 1.14957985 |
| SEP (%)                 | 1.50730419 |
| MAE                     | 0.99934483 |
| AAD (%)                 | 1.32300107 |

between the predicted R<sup>2</sup> (0.8800) and the adjusted R<sup>2</sup> (0.9519), which is in reasonable agreement, is less than 0.2.

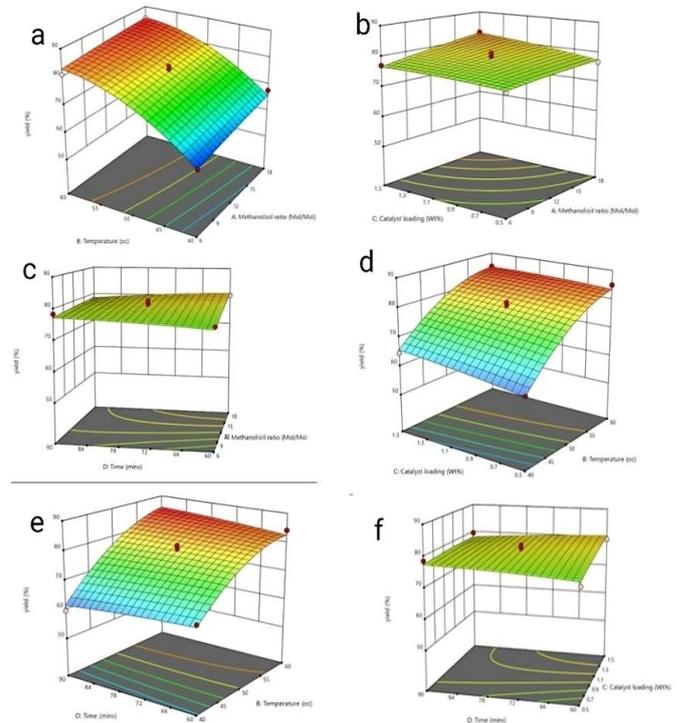
A relatively low coefficient of variation of 2.17% indicates good reproducibility for the model. The sum of PRESS, totaling 191.560, is a measure of how well the model fits each part of the design. A signal-to-noise ratio of 21.075 demonstrates sufficient precision. Ideally, this ratio should exceed four. Consequently, this approach can be effectively employed to explore the design space.

**Table 4** shows the evaluation of RSM performance.

#### **Effect of reaction parameters on COME yield (3-D response surface plots for optimization process)**

The effect of the temperature and methanol ratio relationship on COME yield is depicted in part a in **Figure 7**. Methanol ratio was adjusted between 6:1 and 18:1 while maintaining catalyst loading constant at 75 minutes and 1.00 weight percent, respectively, at 40 °C. When the temperature was at 60 °C and the methanol ratio was changed from 6:1 to 18:1, COME yield achieved was 60.83 weight percent and 67.76 weight percent, respectively. COME yields achieved were 81.00% and 85.60%, respectively.

Part b in **Figure 7** shows the impact of the methanol ratio and catalyst loading relationship on COME yield. While keeping the temperature and duration at 50 °C and 75 minutes,



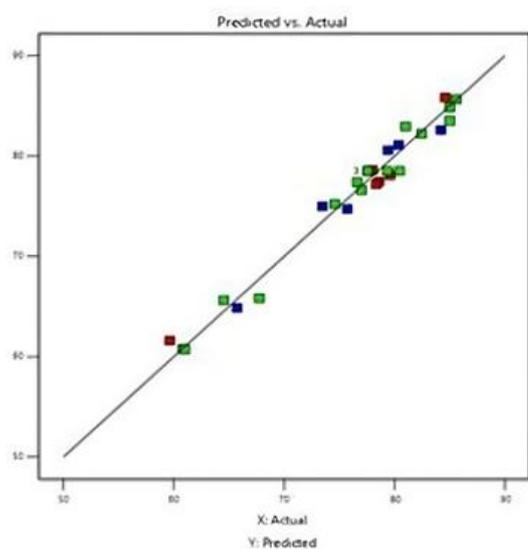
**Figure 7.** Response surface plots for COME yield as a function of (a) methanol ratio & temperature; (b) catalyst loading & methanol ratio; (c) time & methanol ratio; (d) catalyst loading & temperature; (e) time & temperature; & (f) time & catalyst loading (Source: Authors' own elaboration)

respectively, the methanol ratio was changed between 6:1 and 18:1 when the catalyst loading was at 0.50 weight percent. COME yield was 74.60% and 76.63 weight percent, respectively, with 1.50 weight percent catalyst loading. A 6:1 to 18:1 methanol ratio adjustment was made. Both 77.00 and 82.46 weight percent of COME yields were achieved.

Part c in **Figure 7** shows how time and methanol ratio interaction affect COME yield. Over the course of the 60 minutes, the methanol ratio was varied between 6:1 and 18:1, while keeping the catalyst loading at 1.00% and the temperature at 50 °C, respectively. COME yield was obtained at 75.73 and 80.37 weight percent when the time was 90 minutes and the methanol ratio was changed between 6:1 and 18:1. COME yields of 78.36% and 78.00% were attained, respectively.

Part d in **Figure 7** shows how the catalyst loading, and temperature interact to affect COME yield. While keeping time and methanol ratio constant at 75 minutes and 12:1, COME yield was obtained at 61.00 weight percent and 85.00 weight percent, respectively, when catalyst loading was at 0.50 weight percent and temperature was varied between 40 °C and 60 °C, and when catalyst loading was at 1.50 weight percent and temperature was varied between 40 °C and 60 °C, respectively.

Part e in **Figure 7** illustrates the influence of time and temperature interactions on COME yield. COME yield was produced at 65.74 weight percent and 84.20 weight percent, respectively, when the time was 60 minutes, and the temperature was altered between 40 °C and 60 °C. The identical process was carried out again when the time had passed 90 minutes and the temperature had been changed



**Figure 8.** Predicted vs. actual COME yield RSM model (Source: Authors' own elaboration)

from 40 °C to 60 °C. COME yields of 58.65 weight percent and 84.60 weight percent, respectively, were obtained.

On COME yield, part f in **Figure 7** depicts the effects of time and catalyst loading. When the timer reached 60 minutes, the catalyst loading was changed from 0.50 weight percent to 1.50 weight percent while the temperature and methanol ratio were maintained at 75 minutes and 1.00 weight percent, respectively. COME yield obtained varied between 73.47 and 79.40 weight percent when the catalyst loading was altered between 0.50 weight percent and 1.50 weight percent. 78.60 weight percent and 79.60 weight percent were the respective COME yields.

**Figure 8** shows the predicted vs. actual COME yield RSM model.

### Process Parameter Optimization & Model Validation

In order to maximize production efficiency while minimizing costs, it is essential to optimize the process parameters involved in simultaneous esterification and transesterification.

Based on the actual COME yield data gotten from the experimental procedure, the highest yield was attained when using a 1.00% catalyst loading, an 18:1 methanol-to-oil molar ratio, a reaction temperature of 60 °C, and a reaction time of 75 minutes, resulting in an 85.60 weight percent COME yield.

Interestingly, the parameters suggested by BBD for optimal results closely aligned with these findings, proposing a 16.75:1 methanol-to-oil molar ratio, a 1.28% catalyst loading, a reaction temperature of 58.19 °C, and a reaction duration of 78.76 minutes, yielding an 85.73 weight percent COME. This remarkable similarity between the two sets of results underscores the accuracy of our initial assumptions.

### Characterization of Come

COME generated under the optimum reaction conditions was characterized to determine its fuel characteristics. Kinematic viscosity and density of COME were found to be 3.76 mm<sup>2</sup>/s and 854.76 kg/m<sup>3</sup>, respectively, which were within the ASTM and EN biodiesel standard limits. COME's AV, flash

**Table 5.** Physiochemical attributes of biodiesel obtained

| Attribute                              | Value   |
|--|---------|
| Acid value (mg KOH/g)                  | 0.280   |
| FFA (%)                                | 0.140   |
| Saponification value                   | 99.860  |
| Viscosity at 31 <sup>o</sup> C (MPa.S) | 3.760   |
| Density (Kg/m <sup>3</sup> )           | 854.760 |
| Moisture content (%)                   | 0.006   |
| Iodine value                           | 65.730  |
| Pour point (°C)                        | -10.100 |
| Cloud point (°C)                       | -2.000  |
| Flash point (°C)                       | 128.000 |
| Cetane number                          | 68.520  |

point, and calorific value were determined to be 0.28 mg KOH/g, 128 °C, and 39.23 MJ/kg. The values that were obtained for each characterization of COME are shown in **Table 5**.

## CONCLUSIONS

The usage of biodiesel in the power sector has been claimed to assure both energy security and ecological balance. Commercial biodiesel production would need to use inexpensive and plentiful biomass as a raw source in order to be profitable.

This study used CO, which was assessed and found to have an acid value of 5.8 mg KOH/g, to optimize the synthesis of biodiesel, catalyzed by a bio-based bi-functional catalyst, by esterifying and trans-esterifying the CO at the same time. The crab shells were calcined and treated with KOH to form a base precursor, while the carbonized and sulfurized coconut shell was utilized to create an acid precursor. Both acid and base precursors were then impregnated to create the bio-based bi-functional catalyst. SEM, XRD, FTIR, and BET/BJH techniques were used to analyze the characteristics of the synthesized bio-based catalyst. At the optimum conditions of 18:1 methanol to oil molar ratio, 75 minutes reaction time, 60 °C reaction temperature, and a 1.00 weight percent catalyst loading, the catalysis produced a COME yield of 85.60 weight percent and an AV of 0.28 mg KOH/g, whereas the predicted COME yield was discovered to be 85.73 weight percent, at 58.19 °C, 16.75:1 methanol ratio, 1.28 weight percent, and 78.76 minutes.

ANOVA results revealed that the catalyst loading, reaction temperature, and methanol ratio were the key factors of COME yield.

The parameters of COME created in ideal conditions fell within the acceptable range of ASTM D6751 and EN 14214 criteria, and the synthesized catalyst was found to be an economical and environmentally friendly means of producing biodiesel.

### Recommendations

In order to advance the knowledge in the field of simultaneous esterification and transesterification of CO for biodiesel production using a bio-based bi-functional catalyst derived from coconut shells and crab shells, the following critical areas merit further investigation:

1. **Effect of temperature on calcination & carbonization:** Detailed examination of the impact of

temperature during the calcination and carbonization processes of crab and coconut shells is essential. Investigating a range of temperatures will provide insights into the optimal conditions for catalyst development, ensuring its efficiency in the esterification and transesterification reactions.

2. **Effect of impregnation ratios of waste biomasses:** Systematic analysis of various impregnation ratios concerning the waste biomasses (crab and coconut shells) is imperative. This study will shed light on the ideal proportions required for effective catalyst preparation, leading to enhanced catalytic activity and stability during biodiesel production.
3. **Exploration of reaction kinetics:** In-depth exploration of the reaction kinetics involved in the esterification and transesterification processes is crucial. This includes determining rate constants, understanding reaction mechanisms, and evaluating the influence of factors such as temperature, catalyst concentration, and reactant ratios. A comprehensive kinetic analysis will provide valuable data for process optimization.
4. **Impact of catalyst particle size:** Thorough investigation into the influence of the synthetic catalyst's particle size on the simultaneous reaction is necessary. Evaluating different particle sizes will offer valuable insights into the catalyst's surface area and reactivity. This understanding is vital for achieving optimal mixing and efficient conversion of CO into biodiesel.
2. **Comprehensive reusability study:** A comprehensive reusability study of the catalyst is essential for determining its long-term viability and economic feasibility. Conducting multiple reaction cycles and assessing changes in catalytic activity and selectivity will provide critical information on the catalyst's stability and potential for industrial-scale applications.

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**Data sharing statement:** Data supporting the findings and conclusions are available upon request from corresponding author.

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