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Optimization and kinetics studies of biodiesel synthesis from *Jatropha curcas* oil under the application of eco-friendly microwave heating technique: an environmentally benign and sustainable bio-waste management approach

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Abstract

This study attempts to synthesize biodiesel as a green liquid fuel from *Jatropha curcas* oil (JCO) utilizing waste eggshell (WES) as an effective and excellent sustainable source of the heterogeneous catalyst under the application of environmentally benign microwave heating technique. After preparing the CaO-based catalyst, diverse characterization techniques such as X-Ray Diffraction, Energy Dispersive Spectroscopy, Scanning Electron Microscopy, Fourier Transform Infrared, and Brunauer–Emmett–Teller were employed for characterization measurements. Under different optimized conditions, a considerable high biodiesel yield of 92% was attained while employing the following reaction conditions; methanol to oil molar ratio of 9:1, 5 wt% catalyst loading, 165 min reaction time with a microwave power of 800 W, and a 65 °C reaction temperature. The developed catalyst had significantly retained its reusability up to the 5th cycle of reuse. The catalysed transesterification process's activation energy of 38.5 kJ mol^{-1} demonstrated that the reaction is chemically controlled. Therefore, the WES has been utilized as a renewable base heterogeneous catalyst for novel biodiesel synthesis from JCO, which can be applied in diesel engines to lessen air pollution, specifically pollutant emissions from diesel vehicles. The results of this study are not for academic purposes only. They can also serve as models for industrial biodiesel production by exploiting bio-waste as catalysts and non-edible oils as feedstocks in microwave heating systems as environmentally friendly chemistry systems. In addition, our study uses non-consumable oil feedstock and bio-waste materials in an economical way to produce biofuel while contributing to environmental sustainability and sustainable bio-waste management. This approach extends to the existing state-of-the-art research.

Keywords: Waste eggshell valorization, *Jatropha curcas* oil, Biodiesel, Activation energy, Catalyst reusability, CaO-based catalyst

1 Introduction

The fast depletion of fossil-fuel-based resources and global warming as a result of climate change ramifications are the major driving forces towards the development

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of renewable and more environmentally benign energy resources. The forever fast-growing population has also prompted this, along with booming industrialization across the globe, which have similarly led to a serious shortage, uncertainty and rigorous ecological impact of fossil fuel-based energy utilization [1, 2]. In response to this, biodiesel has consequently been identified as one of the most promising green energy that can replace the unsustainable petrol diesel utilisation due to its vast environmental benefits [3, 4].

Biodiesel as a green fuel has been broadly considered as an eco-friendly source of energy, owing to its advantage in producing less CO_2, SOx, and hydrocarbon emissions, which contribute to environmental sustainability [5]. Conversely, over 95% of biodiesel is mainly synthesized from most notably edible oil crops (such as palm, rapeseed, soybean and sunflower), which compromise food security [6]. In view of this, various stakeholders such as government agencies, scientists, industries, and policymakers have aimed for the exploration of economical and non-edible oil-producing energy crops like *Jatropha curcas* for research.

Being an energy crop, *J. curcas* de-oiled seed cake can be used as a feedstock for the production of bio-oil via the pyrolysis process [7]. The *J. curcas* bio-oil has diverse potential for industrial applications. Among others, *J. curcas* bio-oil can also be used as bio dielectric fluids in the Electric Discharge Machining process. It is also reported that *J. curcas* bio-oil is an excellent substitute of the conventional hydrocarbon oils [7, 8].

Meanwhile, shell waste, which is identified as inevitable food waste materials formed in food preparation or consumption, has become a global environmental problem, specifically concerning the issue of waste management. In this regard, eggshells waste is generated from various food industries such as egg processing companies, and enormous amounts of waste shells that are discarded to the landfills or incinerated, have virtually become a significant cause for organic pollution.

Eggshells are a classic example of food waste emanating from the food processing industry that still contains recyclable parts. By 2030, global egg production will increase to around 90 Mt. Because eggshells are considered worthless, most of this waste is usually disposed of in landfills without being converted into valuable materials. However, managing these wastes requires appropriate strategies that take into account the increasing cost of disposal, environmental concerns associated with the risk of spreading pathogens, odours, and where they are disposed of [8–10]. In addition, eggshells as an agro-waste have the potential to cause environmental pollution according to European Union regulations. This is because the large amounts of the discarded eggshells contribute to food wastage which causes significant damage to the environment by increasing global carbon footprint when buried, which is one of the prime greenhouse gases contributing to the global warming [11]. Therefore, it is essential to find an alternative technique to transform eggshells into valued materials for further utilization.

Essentially, eggshells are considered high-value natural materials as opposed to waste, considering their most efficient calcified shells, comprised of over 96% calcium carbonate ($CaCO_3$). Eggshells are predominantly rich in $CaCO_3$ that mostly appears in the mineral calcite form, a better-decontaminated and thermodynamically persistent form of $CaCO_3$, which has a much lower level of contaminations. In view of this, eggshells could offer enormous opportunities for applications in the form of limestone ($CaCO_3$), if not lime (CaO), in a wide range of applications, through significant reusability as a substitute in the production of cement. In addition, eggshells signify an excellent substitute which is vastly plentiful, low-cost, and more sustainable source of filler for the rubber industry [4, 6, 11]. CaO, on the other hand, has a high bonding density with H_2O and CO_2, so it is easily degraded during the aqueous reaction [12, 13]. CaO also demonstrated improved stability and catalytic efficiency in biodiesel synthesis processes due to its high basic strength, low cost and inadequate dissolvability in methanol. CaO is also the cheapest and most durable heterogeneous catalyst [14]. Due to these properties, CaO was obtained from eggshells [5, 15], scallop shells [16], cockles shells *(Anadara granosa)* [17], and animal bones [18]. From this perspective, reusing eggshell waste for many purposes will benefit the environment and the economy. Furthermore, the reprocessing and recycling of waste eggshells (WES) integrates the fundamental principles of circular economy, which increases resource utilization efficiency through transforming waste or by-products into resources with both cost-effective and ecological beneficial impact [19].

Many researchers have been investigating the application of the microwave (MW) technique for the synthesis of biodiesel. It has been widely demonstrated that the MW heating system is one type of clean, fast, and suitable energy source that can expedite the procedure and enhance the selectivity of particular reactions [4, 5]. Lin et al. [5] reported the exploitation of ionic liquid as a green catalyst for the transesterification procedure in the MW heating system, giving a quick way to biodiesel synthesis from *J. curcas* oil (JCO). The MW heating system is equally more energy-efficient or energy-saving and more economical than the conventional heating system [5].

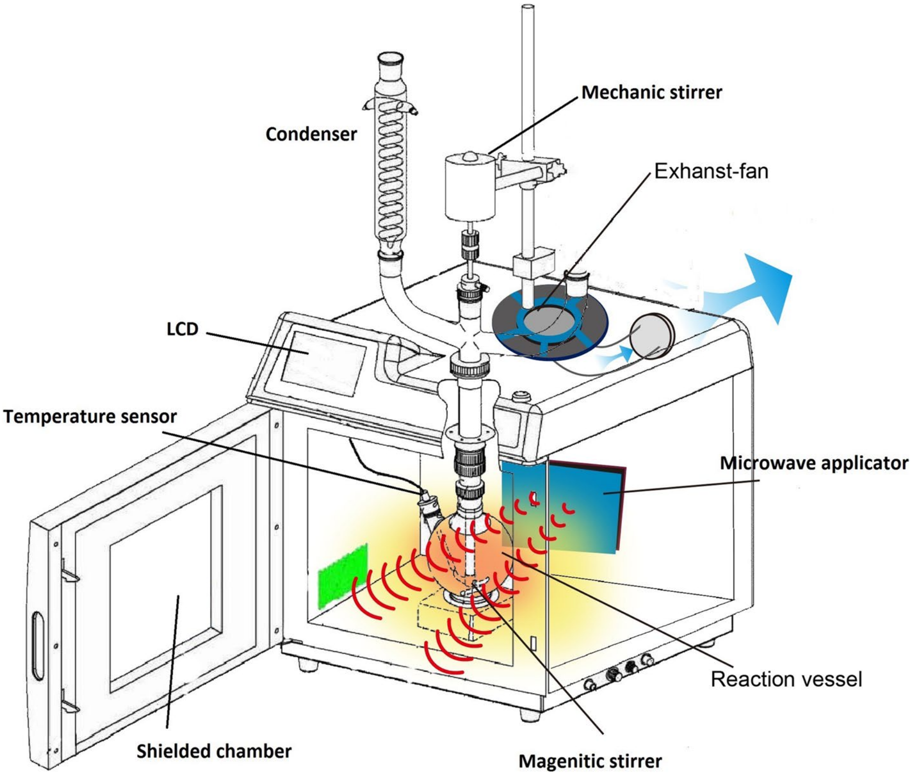

Fig. 1 Schematic diagram of the experimental setup (microwave system structure). (Reprinted with permission from Lin et al. [5]). Copyright (2020), Elsevier [License Number: 5270740247480]

In the light of the above-mentioned discussion, the present study attempts to promote the efficiency and synthesis of bio-waste derived CaO-based heterogeneous catalyst and transesterification of JCO under the application of environmentally benign MW heating technique as a sustainable bio-waste management approach. The developed technique can tremendously promote the integration of the circular economy concept through an efficient biorefinery model.

2 Experimental section

2.1 Materials and reagents

WES were acquired from a fast-food establishment in Taiwan. The non-consumable oil (i.e., JCO) as a raw material was provided by the Chinese Petroleum Corporation (CPC), Taiwan. Methanol and sodium hydroxide were procured from Burdick & Jackson (UNI-ONWARD, Taiwan). The methanol and sodium hydroxide used in our experimentations were of excellent analytical grade, and the purity of all solid chemicals were at least 99.5%. The feedstock was subsequently heated to remove the moisture content, whereas all other chemicals were used as received without further decontamination.

2.2 Catalyst synthesis

2.2.1 Calcination of eggshells

The collected WES were washed thoroughly with tap water to eliminate dirt, impurities, and organic materials. Subsequently, the WES were further rinsed using distilled water at least 2–3 times and, after that, stored in a hot air oven at the temperature of 110–140 °C, for approximately 24 h. At this stage, the desiccated eggshells were removed from the oven for further processing and reduced the materials to a fine powder due to continuous crushing in an agate mortar. Lastly, the calcination was conducted in a high-temperature furnace at 1000 °C for 4 h and consequently placed in a desiccator. Thus, the acquired product was CaO, which was kept in a cool, dark desiccator at a temperature in the range of 20–38 °C.

2.3 The transesterification procedures for the JCO

In this research work, an MW synthesis reactor (PreeKem APEX, Shanghai, China) fitted with an automated stirrer, and a capacitor was employed to enable MW reactions. Figure 1 shows the experimental system for this study. The stirrer was performed at 600 rpm with a hypnotizing (magnetic) midpoint. As a result, the eggshells were exploited to develop a heterogeneous catalyst, a product that has been calcined (CaO constituent), to achieve the waste products (discarded materials) reutilizing objective.

JCO and methanol were blended in the vessels, and subsequently, the catalyst was consequently supplemented to the vessel. The vessel was placed in an MW heating system. The samples were performed at several reaction intervals and temperatures during this time. Various catalyst loading (3–7 wt%), reaction time (120–210 min), methanol to oil molar ratio (MTOMR) (9:1), and a temperature reaction of (45, 55, 65, 75, and 85 °C) were investigated during the experiments. With the application of the Fourier Transform Infrared (FTIR) technique, the JCO conversion to biodiesel was thoroughly examined. After 32 scans, the FTIR spectra of the catalyst samples were determined to be in the range of 500–4000 cm^{-1}. These FTIR spectra were accordingly validated in addition to the documentation aimed at every sample. Krishnamurthy et al. [20] quantified fatty acid methyl esters (FAME) in microwave-mediated transesterification biodiesel using FTIR analysis and identified peaks that may be associated with the typical C=O stretching of the esters. These peaks were found in the 1800–1700 cm^{-1} spectral region and were familiar to both the FAME and refined oil spectra. Foroutan et al. [21] conducted FTIR analysis to determine the functional groups of the studied catalyst and produced biodiesel from edible waste oil using a calcium oxide@magnesium oxide nanocatalyst. Analysis was performed on 400–4000 cm^{-1} band range.

2.4 Determination of acid value and separation procedures

The JCO sample used under this investigation was found to have an acid value of 2.8 mg KOH g^{-1} and a saponification value of 195 mg KOH g^{-1}. Additionally, the JCO sample had a free fatty acid (FFA) value of 2.5 wt% with an average molecular weight of 861 g mol^{-1} and water content of 0.12 wt% [4, 6]. JCO sample was found to have a pH value of 5.8.

Under this investigation, the method on the transesterification reaction was anticipated to have had happened when the quantity of methyl ester was above 90%. In view of this, the mixture and assortment at the concluding phase of the transesterification process were separated with the help of a centrifuge running at 2000 rpm for 10 min, which resulted in the composition of an upper phase characterized by methyl ester and consequently a reduced phase composed of glycerin. This has also progressed into disproportionate methanol during the methyl ester stage, which was vaporized through heating at 80 °C. Afterwards, the catalyst was efficiently withdrawn with the support of silica gel supplemented to the reaction. Therefore, biodiesel was subsequently achieved, and its content was determined with the support of Gas Chromatography (GC-6890, Agilent, USA).

2.5 Product evaluation

The evaluation procedures employed in our experiments were validated in compliance with Taiwan CNS15051 for determining the amount of methyl ester. Additionally, the methyl ester content during this study was determined using the Gas Chromatography method furnished with the flame ionization detector (GC-FID). The conditions used for GC-FID to analyze the products and other instruments and methods used for other analysis such as X-ray Diffraction (XRD), Energy Dispersive Spectroscopy (EDS), Scanning Electron Microscopy (SEM), FTIR, and Brunauer–Emmett–Teller (BET), are reported in detail in our previous study [4, 5]. The content of methyl ester in this study was determined by employing the following Eq. (1) as reported in our preceding studies [4, 5]:

$$Methyl\ ester\ content = C = \frac{\left(\sum A\right) - A_{EI}}{A_{EI}} \times \frac{C_{EI} \times V_{EI}}{m} \times 100\% \tag{1}$$

where ΣA is the total of the peak area of FAME from C_{14} to $C_{24:1}$; A_{EI} is the peak area of the internal standard, methyl heptadecanoate ($C_{18}H_{36}O_2$); C_{EI} is the absorption of methyl heptadecanoate (mg mL^{-1}); V_{EI} is the quantity of methyl heptadecanoate (mL^{-1}); and m is the quantity of input biodiesel (g). The methyl ester yield is expressed using Eq. (2) as reported in our earlier studies [4, 5]:

$$Methyl\ ester\ yield = methyl\ ester\ content \times biodieselyield = C \times \frac{W_B}{W_{oil}} \tag{2}$$

where C is the methyl ester content (%); W_B is the quantity of synthesized biodiesel (g); and W_{oil} is the quantity of the initial volume of JCO (g). Consequently, biodiesel production was investigated and measured as part of the preliminary capacity of JCO by weight. A similar approach was used and demonstrated in our previous work [4, 6].

2.6 Physico-chemical properties of biodiesel from JCO

Table S1 of the Supplementary Materials shows the summarized physico-chemical properties of the JCO biodiesel. The biodiesel physico-chemical characteristics are unique, predominantly its density, which can substantially influence the engine's performance [6]. This emanates from the evidence that the fuel density can effectively split up the fuel spray from the injector. For that reason, the density at 15 °C for JCO biodiesel is mainly found to be within the scope and appropriate range of 0.859–0.891 g cm^{-3} for fuel standard (ASTM D6751) or EN 14,214 standard [22, 23].

It can also be observed from Table S1 that the JCO biodiesel could have a level of acidity of 0.18 mg KOH g^{-1} and saponification number of 191 mg KOH g^{-1} that are under the normal standard range 0.5–370 for biodiesel in consistent with the ASTM standards (ASTM D6751-02). The iodine value is used to establish the iodine absorption values in g, which could be engrossed by 100 g oil. It helps to determine the amount of unsaturation of biodiesel, and thus it is beneficial to examine the oil durability [6]. It is also worth noting that a significant proportion of desaturation can lead to fuel polymerization due to the development of epoxide in the buildup of oxygen in twofold links. As it can be observed from Table S1, the JCO biodiesel has an iodine value of 69 g I_2 100 g^{-1} oil that falls further down the recommended upper threshold of 120 (EN 14,214 standard). This iodine value is comparable with the results reported by Kumar et al. [22] for JCO biodiesel (75 g I2 100 g^{-1}). Additionally, other greater iodine concentrations of 119 and 122 g I_2 100 g^{-1} were recorded in the study conducted by Sarma et al. [24] and Nath et al. [25]. JCO biodiesel is reported to have a higher heating value (calorific value) of 40.5 MJ kg^{-1} [7].

3 Results and discussion

3.1 Catalyst characterization

3.1.1 XRD analysis

We studied the eggshells before and after calcination using the XRD (HR-XRD D8 SSS, Bruker) analysis technique. Figure S1 illustrates the XRD patterns of the uncalcinated and calcinated eggshells before (Fig. S1a) and after calcination (Fig. S1b). According to the results, the occurrence and strong peaks were located at a brag angle of (2θ) at 29.7, 39.3, 43.2 and 47.6° for the recycled WES before calcination (Fig. S1a). However, strong peaks were equally noticed after calcination (Fig. S1b) and were detected an angle of *(2θ)* at 29.8, 34.1, 39.4, 43.3, 47.6 and 48.5° in accordance with the bands of CaO [6].

In the earlier investigations, it has been discovered that the snail shells were calcined at 900 °C and indicated the presence of CaO with strong peaks of brag angle *(2θ)* located at several points, coordinating from 32.2, 37.3 as well as 53.8°. Conversely, the uncalcined snail shell band was similarly described to have peaks 2θ located at several points coordinating at 26.2, 33.2 and 45.9°, suggesting that snail shell component comprised of $CaCO_3$ [26]. Risso et al. [15] examined the calcination of waste shells, calcined for the duration of 4 h at 900 °C. The grinded clamshell powder had eventually been transformed into a fine white powder, firmly demonstrating that it was converted into CaO. By comparison, the bands of XRD assessment were discovered at an angle of *(2θ)* located at several degrees (32.5, 37.6, 54.1 and 62.6°), and this attests to the presence of CaO [6]. Takeno et al. [14] described cubic crystals in calcined Silver Croaker's stone, showing that the calcination temperature and time were adequate to decompose all the carbonates into oxides. CaO has been designated as one of the most suitable catalysts for the transesterification of soybean oil [27].

3.1.2 FTIR analysis

The FTIR analysis technique was applied to characterize eggshells before calcination (Fig. S2a) and after calcination at at 1000 °C for 2 h (Fig. S2b). According to the results, pre-calcination absorption bands were situated at various peaks at 1397.0, 862.9 and 710.3 cm^{-1}. These types of bands are essentially caused by carbonate's existence on the surface of the catalyst [4].

Lastly, the absorption bands after calcination were located at 3650.9, 1397.1, 873.9, and 710.1 cm^{-1}. The expanded matrix oscillations of the CaO bond are indicated by large, strong absorption peaks of approximately 873.9 and 710.1 cm^{-1}. Asymmetric expansion and contraction of C-O bonds in unknown carbonate species was associated with peaks of 1397.1 and 873.9 cm^{-1}. There is a large peak at 3650.9 cm^{-1} due to the creation of -OH bonds at $Ca(OH)_2$ after H_2O adsorption on the metal surface by CaO. This means that water is not the cause of catalyst deactivation [16]. It may also be due to $Ca(OH)_2$ forming soaps in acidic oils, which reduces yields in subsequent runs. These FTIR results acquired in the current study are comparable with related results described in our previous work [4, 6].

3.1.3 EDS analysis

In this study, the EDS analysis was conducted with a single goal to determine particular elements that might be present in the produced catalyst. In light of this, it was revealed (after calcination) that individual chemical elements such as C (6.5%), O (19.1), and Ca (74.4%) as constituents of the developed CaO-based eggshells catalyst as displayed in Table 1.

The determined EDS results are consistent with the XRD. Rahman et al. [28] reported that the catalyst

Table 1 EDS analysis results for elemental composition of eggshell

Elements	Before calcination		After calcination	
	wt%	At%	wt%	At%
Carbon (C)	13.6	23.5	2.3	6.5
Nitrogen (N)	5.1	7.6	N.D	N.D
Oxygen (O)	33.8	43.8	9.05	19.05
Sodium (Na)	1.2	1.1	N.D	N.D
Calcium (Ca)	46.4	24.0	88.6	74.4

N.D Not detected

performance could be potentially strengthened by the complementary effect of different metal oxide-based catalysts. The catalyst constituents are relatively compatible with the banana peel ash recounted by Balajii and Niju [29]. SEM diagrams in Fig. 2 show the WES material structure, prior calcination (Fig. 2a) and after calcination (Fig. 2b) at high temperature. The specific surface area (Micromeritics, ASAP 2020) and chemical configurations of the developed catalyst were determined. It is evidently illustrated from Fig. 2 that the pore structures in the eggshell showed extensive, substantial disparities before (Fig. 2a) and after calcination (Fig. 2b).

After calcination, there should be significant modifications in the shells' physical structures, signifying that the transformation of $CaCO_3$ into CaO has happened. SEM images of the developed CaO-based catalyst showed packs of appropriately structured cubic crystals with visible edges. These images are quite similar to the ones revealed by Nisar et al. [18] for calcined waste animal bones. The calcined bones were irregular, and some stuck together. For comparison, Sirisomboonchai et al. [16] developed nano-sized CaO-based catalysts from snail shells using SEM micrographs showing significant accumulation of catalyst particles due to a high specific area and a spherical structure without scattering morphology.

3.1.4 Brunauer–Emmett–Teller (BET)-Barrett-Joyner-Halenda (BJH) measurements

The dimensions for the surface area and the absorbency depiction of the developed catalyst were investigated using N_2 physisorption with a BET technique (AUTOSORB-1C Quantachrome). As for the adsorption of nitrogen gas employing the micrometrics equipment, the general pore capacity and average pore dimension of the developed catalysts were calculated using the BJH technique. The Hammett indicators were (acquired from Sigma-Aldrich, Kaohsiungy, Taiwan) used to measure the basic strength and the basicity of the catalyst samples. The outcomes are displayed in Table 2. The analysis was conducted at a temperature range of 800–1000 °C. In this respect, the assessment via BET suggests that the developed catalyst's surface area had considerably declined from 7.0 to 6.2 $m^2\ g^{-1}$ with a typical BJH adsorption pore measurement of pore size between 19 and 31 nm. Palitsakun et al. [30] reported a much lower surface area and pore volume measurements for CaO-T catalyst with a surface area of 1.95 $m^2\ g^{-1}$ and an average pore volume of 0.019 $cm^3\ g^{-1}$, while the CaO-H catalyst had a surface area of 23.3 $m^2\ g^{-1}$ with an average pore volume of

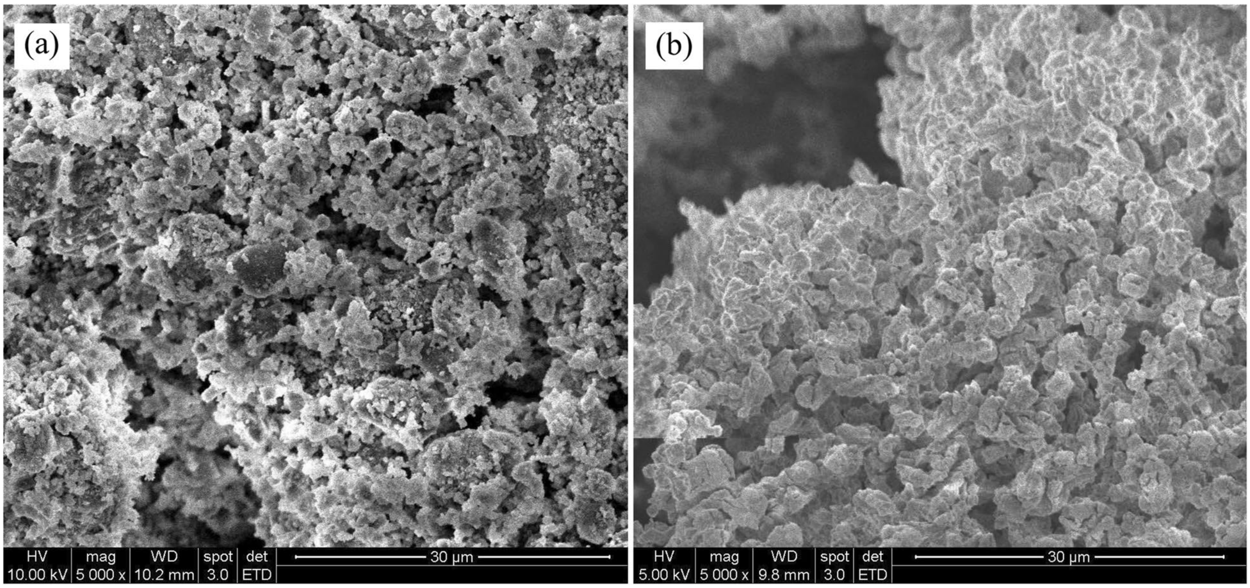

Fig. 2 SEM image of eggshell before (**a**) and after calcination (**b**) at high temperature

Table 2 BET surface area measurements and porosity characterization of the CaO-based catalyst

Calcination temperature (°C)	Basic strength [a] (H_)	Basicity[a] (mmol g^{-1})	Surface area [b] (m^2 g^{-1})	Pore volume[b] (cm^3 g^{-1})	Pore size (nm)[b]
800	7.2 < H_ < 9.8	0.74 ± 0.34	7.0	0.02	19
900	15.0 < H_ < 17.2	0.97 ± 0.41	6.4	0.17	27
1000	12.2 < H_ < 15.0	1.21 ± 0.36	6.2	0.12	31

[a] Determined by Hammett indicator

[b] Analyzed by N_2-physisorption

0.201 cm^3 g^{-1}. The CaO-P catalyst was found to have a surface area of 1.76 m^2 g^{-1} and an average pore volume of 0.0155 cm^3 g^{-1}. Rahman et al. [28] have found that a solid catalyst's higher surface area can improve its catalytic performance.

3.2 Optimization of transesterification over the developed catalyst and exploring the optimum reaction conditions

3.2.1 Effect of catalyst loading on biodiesel yield

The catalyst effect substantially influences biodiesel yield, and thus, it is an indispensable parameter in optimising the transesterification reaction. On the other hand, when the percentage of free fatty acids (FFAs) in the feedstock is much high, the base catalyst can react with FFAs, resulting in saponification. This could make the product partition and purification process more complex and reduce the biodiesel yield [5, 6]. Accordingly, various catalyst loadings on biodiesel yield were studied to examine their effect on biodiesel production yield. In this regard, the results in Fig. 3a revealed that catalyst loading significantly influenced biodiesel yield. As shown in Fig. 3a, the biodiesel yields increased when the catalyst loading was increased from 3 to 4%.

Notwithstanding, as the catalyst loading was increased to 5%, the biodiesel yield had consequently increased to more than 90%. Furthermore, when the catalyst loading was increased to 6%, biodiesel yield was subsequently observed not to be increasing any longer and started to decline from 91 to 87%. Other researchers highlighted that excessive catalyst loading might impede the mixing of methanol, oil and catalyst, which may further lead to the separation phase [4, 6]. Therefore, 5 wt% catalyst loading was the best catalyst amount during this study.

3.2.2 Effect of reaction time

Figure 3b shows the effect of reaction time on biodiesel yield. Different experimental conditions were as follows: MTOMR of 9:1, 5 wt% catalyst loading, 180 min reaction time, temperature 65 °C and MW power of 800 W. As it can be observed from Fig. 3b, the biodiesel yield had significantly increased as the reaction time continued to increase. Subsequently, this trend has resulted in a high biodiesel yield of 91% in 180 min. However, the biodiesel yield began to decline slightly as the time was further increased from 195 to 210 min. The possible elucidation of this result could be that as the reaction time was at 180 min, the reaction had probably already reached its equilibrium phase. In addition, the backward reaction might occur after reaching the equilibrium period because this reaction, in effect, holds a reversibility nature of the reaction and consequently declines the yield [28, 31].

Alternatively, the highest conversion of JCO to biodiesel can be attributed to various factors such as high basic strength, high absorbency, and large surface area of the developed CaO-supported catalysts from WES materials. It is also worthy of highlighting that a very long reaction time could reduce biodiesel yield in such a way that the transesterification reaction will shift to the left and lead to the formation of soap [15]. Such soap formation can result from esters that could have probably been affected by hydrolysis by increasing reaction time further [31]. Palitsakun et al. [30] argued that methanol normally activates CaO. A small amount of CaO is converted to the parental $Ca(OCH_3)_2$, which is more catalytically active than inactivated CaO. On the other hand, the FAME yield rose substantially from 54 to 88% in 80 min and remained steady as the reaction time intensified to 120 min, indicating that the system reached equilibrium.

3.2.3 Effect of MTOMR

The influence of MTOMR on biodiesel yield was investigated under the following conditions: MTOMR of 9:1, 5 wt% catalyst loading, 180 min reaction time, temperature 65 °C and MW power of 800 W. MTOMR has a similar effect analogous to the loading of catalysts, specifically on the reaction rate and biodiesel yield in particular. As a result, the study on the transesterification reaction was conducted by adjusting the MTOMR while employing the enhanced catalyst loading (5 wt%) at 65 °C (Fig. 3c). It can be determined from the results in

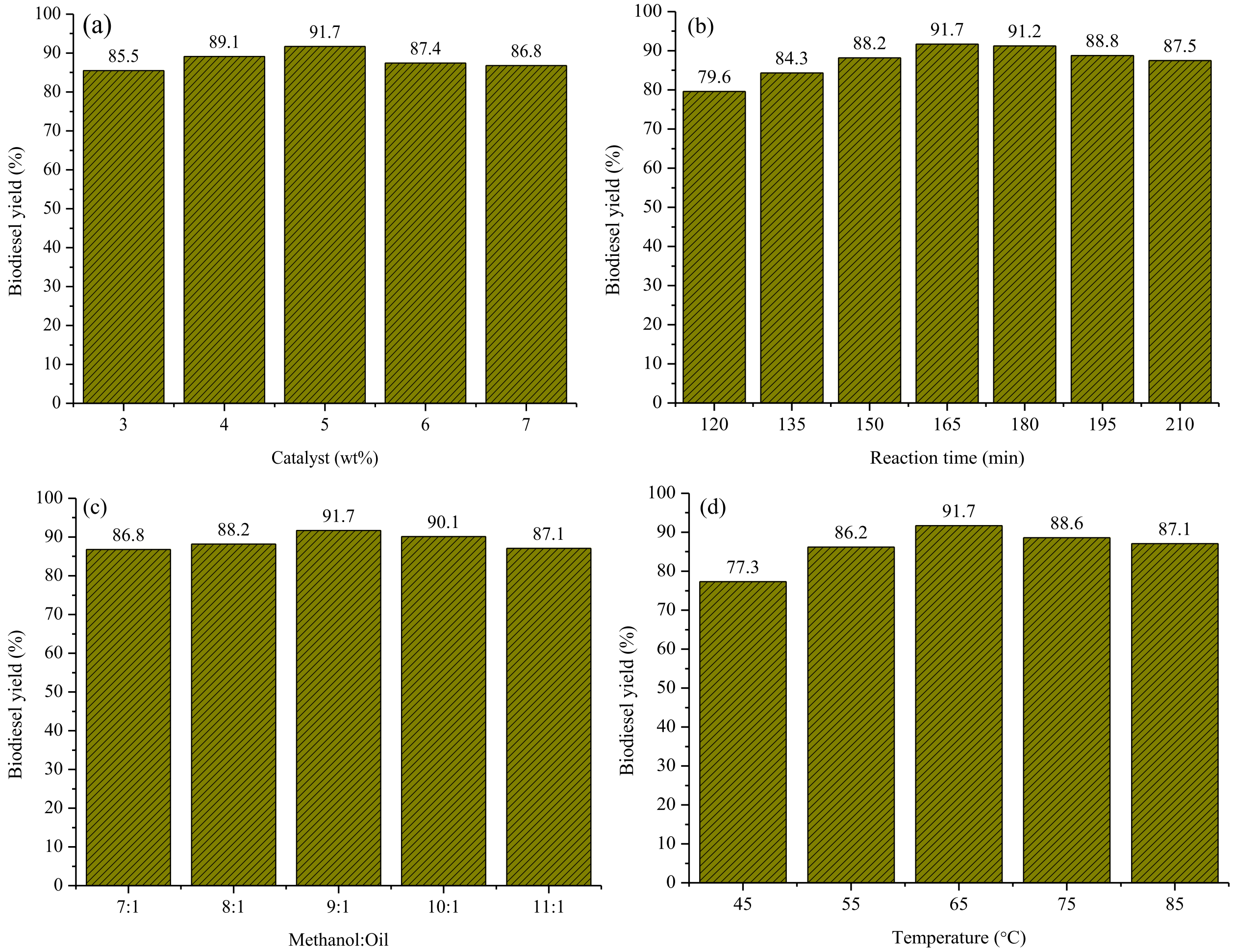

Fig. 3 Effects of **a** catalyst loading, **b** reaction time, **c** methanol to oil molar ratio (MTOMR), and **d** reaction temperature on biodiesel yield. Conditions: MTOMR 9:1; catalyst loading 5 wt%; reaction time 165 min; 65 °C; microwave power of 800 W

Fig. 3c that as MTOMR increased from 7:1 to 9:1, a considerable increase in biodiesel harvest from 86 to 91% was notably observed. Despite that, as the MTOMR was increased to 10:1, there was an insignificant decrease in biodiesel yield (90%). When the MTOMR was further increased to 10:1, the biodiesel yields gradually decreased to 87% at 11:1.

Conversely, as the methanol concentration continued to increase, the catalyst's elements of the catalyst and reaction agents were diluted by methanol, which could trigger a reverse reaction and result in declined biodiesel yield [6]. A comparable pattern of the results was affirmed in the study conducted by Nath et al. [25] and Basumatary et al. [7]. Their studies recorded that large amount to MTOMR above best reaction prerequisite can weaken the reaction assortments, causing submerging of catalyst's active sites, thus declining their essential interactions for effective reaction and subsequently, the reaction rate and decreasing biodiesel yield product. Additionally, above the ideal MTOMR level, there is always a possibility of increasing biodiesel's hydrolysis to produce soap. Such an effect would trigger a substantial reduction in biodiesel yield [7, 32]. In the present work, the most promising level for JCO transesterification to biodiesel was determined to be 9:1 of MTOMR with 5 wt% of CaO-supported catalyst generated from eggshell waste materials.

3.2.4 Effect of reaction temperature

The effect of temperature on the biodiesel yield was studied under the following diverse conditions: MTOMR of 9:1, 5 wt% catalyst loading, 180 min reaction time, temperature 65 °C and MW power of 800 W. According to the results in Fig. 3d, it was found that when the temperature was lower than 65 °C, the biodiesel yield increased with increasing temperature and achieved the highest considerable yield at 65 °C. The biodiesel yield was observed to be decreasing slightly as the temperature above 65 °C. Various studies envisaged reaction of this nature to be thermic [7], whereas other researchers anticipated it to be marginally exothermic [32, 33]. It is also imperative to highlight that the molecular activity increased with the increasing temperature, increasing the reaction rate. Hitherto, as the temperature continues to increase to 65 °C, which is assumed to be the boiling temperature of methanol, the methanol will be evaporated and disappear from the reaction mix. Accordingly, the biodiesel yield was found declining. In addition, higher temperatures could speed up the saponification of triglycerides, which can cause adverse consequences on product yields [34].

3.3 Determination of transesterification process kinetics and activation energy

To investigate the transesterification reaction's kinetics, the effects of temperature and constant reaction time on the reaction were studied. The kinetics of transesterification reaction was investigated at various reaction temperatures of 45, 55, 65, 75 and 85 °C under the ideal operational conditions. As indicated in Table 3, the linear regression equation at diverse reaction temperatures was attained, and the results are presented thereof. Regression equation was established, and it was observed to be linear with high regression coefficient ($R^2 = 0.9994$). The reaction's activation energy (*Ea*) was determined as 38.5 kJ mol^{-1}.

Additionally, a comparison of the activation energy for biodiesel production of the synthesized catalysts with several solid heterogeneous base catalysts from the previous studies has been made, signifying how the diverse *Ea* is attained by exploiting different feedstock (oils) in the transesterification reaction, as shown in Table 4. Mazubert et al. [35] had reported activation energy of 9.7 kJ mol^{-1} while utilizing waste cooking oil (WCO) under an MW heating system. While using the same WCO as a feedstock and heterogeneous catalysts, Gupta and Rathod [36], Foroutan et al. [21] and Al-Sakkari et al. [11] have recorded high activation energy of 27, 46 and 49 kJ mol^{-1}. In comparison, Pavlovic et al. [19] reported very high activation energy (67; 58 kJ mol^{-1}) as compared to the present study. Uzun et al. [37] used a conventional heating method and reported a very low activation energy of 12 kJ mol^{-1}.

3.4 Comparative study of the synthesized catalyst

The results showed a significantly high yield of 92% for biodiesel within 180 min reaction time within ideal requirements of 9:1 MTOMR and 5 wt% catalyst loading with 65 °C reaction temperature. The results in this study

Table 3 Linear regression equation at different reaction temperatures

Temperature (°C)	Reaction time (min)	Temperature (K)	Biodiesel yield (%)	1/T (K^{-1})	1/t (s^{-1})	ln (1/t)
45	165	318	79	3.14E-03	6.06E-03	-5.11
55	165	328	84	3.05E-03	6.06E-03	-5.11
65	165	338	88	2.96E-03	6.06E-03	-5.11
75	165	348	91	2.87E-03	6.06E-03	-5.11
85	165	358	91	2.79E-03	6.06E-03	-5.11

Reaction temperatures = 45, 55, 65, 75 and 85 °C; Reaction time = 165 min; Activation energy (E_a) = 38.5 kJ mol^{-1})

Table 4 Activation energy (E_a) on transesterification reaction of biodiesel production from different studies

Feedstock	Heating method	Catalyst	Catalyst phase	Activation energy (kJ mol^{-1})	Refs
WCO	Microwave	Conventional	--	9.7	[35]
WCO	Microwave	Heterogeneous	Calcium diglyceroxide (CaDG)	26.56	[36]
Sunflower oil	Rotating miniature autoclave reactor system	Heterogeneous	CaO/zeolite-based catalyst prepared from waste chicken eggshell coal fly ash	67.17; 58.03	[19]
Hydnocarpus wightiana oil	Conventional	Heterogeneous	CaO nano catalyst prepared from waste snail shells	67.21; 73.15	[20]
WCO	Conventional	Heterogeneous	Biochar/CaO/K_2CO_3 catalyst	45.53	[21]
Jatropha curcas oil	Microwave	Heterogeneous	CaO catalyst prepared from eggshells	38.5	This work

WCO Waste cooking oil

were compared to various preceding studies, specifically on waste biomass derivatives for catalysts of the heterogeneous nature that were utilized for the generation of biodiesel, as indicated in Table 5. Biodiesel production from JCO while exploiting various solid base heterogeneous catalysts derived from different sources such as by Teo et al. [8] and Vyas et al. [38] have all recounted high reaction temperatures (360 and 120 °C, respectively) for the transesterification procedure with pretty much similar yields to the catalyst used under this investigation. They have reported 94 and 92% biodiesel yields after the 9th and 5th cycles, respectively.

In other studies, the synthesis of biodiesel while exploiting different waste biomass derived heterogeneous catalysts, Chavan et al. [39] and Teo et al. [8] have reported a diminished catalytic performance with lengthy reaction time (2.5 and 4 h). This can perhaps be ascribed to significant smaller surface areas of the various catalysts used. However, Pratika et al. [40] and Basumatary et al. [7] reported CaO-based heterogeneous catalysts with relatively outstanding catalytic performance (with 98 and 98% biodiesel yields) even though they may have low catalysts reusability or recyclability of 4th and 3rd cycles as compared to that of the catalyst utilized under this present study which was recycled 5 times. Teo et al. [8] have also described similar catalytic performance for JCO biodiesel production utilizing solid based heterogeneous catalysts and produced biodiesel yields in the range of 90–97%. Consistently, the current WES derived catalyst has displayed exceptional catalytic activity for the generation of biodiesel. In the current research work, EDS analysis has reported the existence of a high quantity of calcium in the WES catalysts as compared to other elements (Table S1), and the Ca is an outstanding source of calcium oxide (CaO), which has been broadly utilized in several commercial enterprises as a heterogeneous catalyst. CaO has a fundamental role in the catalysis for the application of biodiesel production [21, 23].

3.5 Reusability and leaching study of the catalyst

It is worth mentioning that the economic sustainability or viability of the transesterification procedure regarding biodiesel production is immensely influenced by the recyclability of a catalyst [23]. Reusability is one of the most essential and promising characteristics of the solid base heterogeneous catalysts that should be considered in the continuous reaction process for the low-cost biodiesel generation [26, 27]. Figure 4 shows the results of the reusability study of the catalyst.

The synthesized catalyst was regained from the reaction mixture by separation and consequently rinsed with hexane and subsequently calcinated at 1000 °C for 4 h. Thus, the restored catalyst was acquired and immediately reused in successive 5 catalytic cycles using the same experimental and reusability approach. Furthermore, the recycled catalyst was ascertained to produce a more than 90% biodiesel yield in the 1st cycle and stayed the same at 90%. Yet even in the 3rd cycle of the five progressive catalytic runs, the recycled catalyst performed well with an excellent biodiesel yield of more than 80%. Conversely, the results were observed to have been drastically reduced to 72% after the 5th cycle. This may be due to the contamination of the active site with CO_2 and H_2O and, hence, it shortened lifespan of the developed CaO-based catalyst. As a result, the CaO-based catalyst developed in our study exhibits outstanding reusability in biodiesel fuel production with a significant reduction in yield up to 5 cycles. The degree of Ca leaching can affect the lifetime of the developed CaO catalyst, and biodiesel can be contaminated with leached Ca particles [19].

4 Economic and environmental cost–benefit analysis

Waste such as eggshells can be transformed into high value-added products, contributing to sustainable economic development and, more so, to sustainable bio-waste management. Undoubtedly, it costs about

Table 5 Comparative study of biodiesel production from Jatropha oil with other reported basic heterogeneous catalysts

Sl. No	Feedstock	Catalyst	Methanol: Oil ratio	Catalyst amount (wt%)	Reaction time (min)	Reaction temperature (°C)	Yield (%)	Reusability	Refs
1	Jatropha oil	$CaSO_4/Fe_2O_3$-SiO_2 core–shell	9:1	12	4 h	120	94	9 cycles	[8]
2	Jatropha oil	SO_4/TiO_2 and TiO_2/CaO	1:15	3	90	65	79	N/A	[40]
3	Jatropha oil	Eggshell (CaO)	1:8	2	2.5 h	65	90	6 cycles	[39]
4	Jatropha oil	Heterogeneous catalyst from *Heteropanax fragrans* (Kesseru)	12:1	7	65	65	98	3 cycles	[7]
5	Jatropha oil	Heterogeneous KNO_3/Al_2O_3	12:1	6	70	360	87	5 cycles	[38]
6	Jatropha oil	Eggshell	9:1	5	165	65	91	5 cycles	This study

N/A Not available

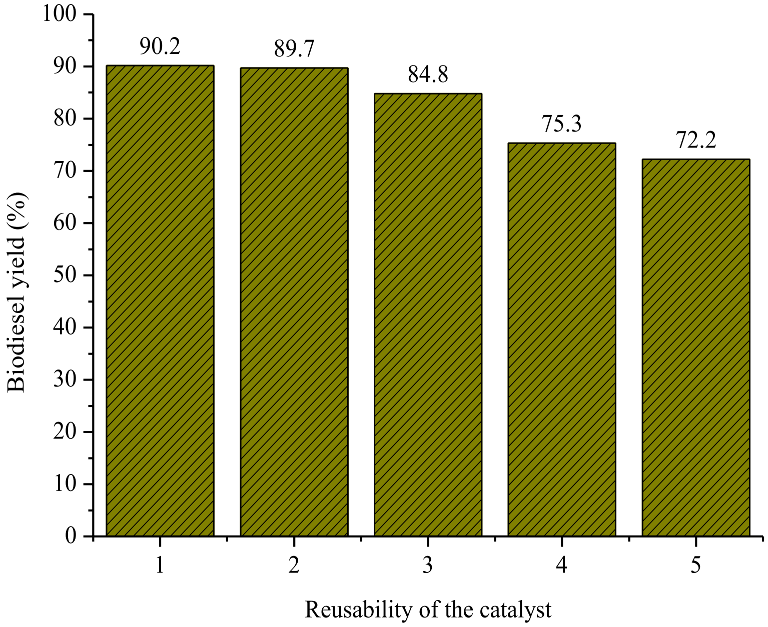

Fig. 4 Reusability of the WES derived catalyst (CaO-based catalyst). Conditions: MTOMR 9:1; catalyst loading 5 wt%; reaction time 165 min; 65 °C; microwave power of 800 W

US$100,000 per annum to discard the eggshell waste produced by the average egg processing plant in the United States [10]. In contrast, the industrial scale of recycling WES to CaO-based catalysts can provide economic benefits that are at least five times higher than the cost of conservative disposal methods [8]. At the same time, by minimizing the exposure and likelihood of pathogens transmission, abbreviating discarding costs and synthesizing potential CaO-based heterogeneous catalysts for economical biodiesel production, great environmental benefits can be achieved that greatly support the sustainability of the anticipated technique [8]. One of the environmental benefits of reutilization of WES is that the eggshell is made up of calcium carbonate, which is a rich source of calcium. Eggshells are collected as waste from residences, hotels, and egg processing facilities, among other places. Eggshells are still difficult to dispose of in an environmentally acceptable manner because of their limited decomposition capacity, which generates pollution. To reduce microbiological species such as molds and bacteria, such shells can be dried at high temperatures (about 80 °C). Eggshell powder can then be made by powdering dried shells in a grinder [29]. Eggshell powder has been demonstrated in studies to be effective as a calcium supplement for plants suffering from blossom-end rot disease, such as tomatoes and berries [6].

5 Conclusions

This research has successfully synthesized an economical, eco-friendly, and highly efficient bio-waste-derived CaO solid base heterogeneous catalyst for low-cost biodiesel synthesis. The developed catalyst has demonstrated outstanding catalytic performance with a biodiesel yield of 92% under the measured, optimized reaction conditions. While the kinetic study parameters and *Ea* of the reaction were determined to be 38.5 kJ mol^{-1}, culminating that it is not an energy-intensive procedure. We hypothesized that the high coefficient of determinations (R^2) value of 0.9994 signified the prospect and excellent suitability of this model can be applied in future. The eggshell catalyst, produced from renewable and cheap raw materials under MW-assisted technology, is an excellent and environmentally friendly technique. These capabilities are required to develop high-performance heterogeneous catalysts that can be upgraded to a commercial scale in the future or to industrial applications for large-scale biodiesel production. The results of this study are not for academic purposes only. They can also serve as prototypes for the industrial production of biodiesel using non-consumable oils as feedstock and biowaste as catalysts for MW heating systems as environmentally friendly methods for sustainable bio-waste management approaches. In addition, this study expands on existing state-of-the-art research as we know about biofuel production using non-consumable oil feedstocks and bio-waste economically.

Supplementary Information

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Additional file 1: Table S1. Physico-chemical properties of the *Jatropha curcas* biodiesel (ASTM D6751, EN 14214) from [1, 2]. **Fig. S1.** XRD analysis of eggshells (a) before and (b) after calcination. **Fig. S2.** FTIR analysis of eggshells before (a) and after calcination (b).

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Authors' contributions

Kassian T.T. Amesho: Writing-original draft. Yuan-Chung Lin: Conceptualization, Funding acquisition, Methodology, Supervision. Chin-En Chen: Data curation, Investigation, Formal analysis/Software. Pei-Cheng Cheng: Data curation, Investigation. Vinoth Kumar Ponnusamy: Writing- review & editing. All authors read and approved the final manuscript.

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Availability of data and materials

The datasets supporting the conclusions of this article are included within the article and supplementary materials.

Declarations

Competing interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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