

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 utilizing waste eggshell (WES) as an effective and excellent sustainable source of the heterogeneous catalyst under the application of environmental benign microwave heating technique. After preparing the CaO-based catalyst, diverse characterization techniques such as XRD, EDS, SEM, FITR, and BET were employed for measurements. Under different optimized conditions, a considerable high biodiesel yield of 91.7% was attained while employing the following reaction conditions; methanol to oil ratio (MTOR) 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 (E_a) of 9.58 kJ/mol demonstrated that the reaction is chemically controlled. The physico-chemical properties of the *Jatropha curcas* oil (JCO) biodiesel were studied in accordance with international standards such as ASTM D6751 and EN14214, among others. Therefore, the waste eggshell 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. Therefore, the proposed value increase method contributes to integrating the circular economy concept through an efficient biorefinery model.

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 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 an eco-friendly source of energy, owing to its advantage in producing less CO₂, SO_x, 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.

Meanwhile, shell waste, which is identified as inevitable food waste materials formed in food preparation or consumption thereof, has become a global environmental problem, specifically concerning the issue of waste management [7]. In this regard, eggshells waste is hugely generated from various food industries such as egg processing companies, and enormous amounts of waste shells are discarded to the landfills or incinerated, have virtually become a significant cause for organic pollution [5, 6].

Eggshells are a classic example of food waste emanating from the food processing industry that still contains recyclable parts [8, 9]. By 2030, global egg production will increase to around 90 million tonnes [10]. Because eggshells are considered worthless, most of this waste is usually disposed of in landfills without being converted into valuable materials [8]. 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 [9]. In addition, eggshells

are considered hazardous waste according to European Union regulations [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) [6]. 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 [4, 11]. 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 uses [11], through significant reusability as a substitute in the production of cement [6]. 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 [14]. CaO is also the cheapest and most durable heterogeneous catalyst [14]. Due to these properties, CaO was obtained from eggshells [15, 5], snail 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 [28].

Many researchers have been investigated the application of the microwave (MW) technique in 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 and the MW heating system, giving a quick way to *Jatropha curcas* oil (JCO) biodiesel synthesis. The MW heating system was equally more energy-efficient or energy-saving and more economical than the conventional heating system [5].

In the light of the above-mentioned, the present study attempts to promote the efficiency and synthesis of bio-waste derived CaO -based heterogeneous catalyst and transesterification of *Jatropha curcas* oil under the application of environmental 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

The waste eggshells (WES) were acquired from a fast-food establishment located in Kaohsiung City, Taiwan. The non-consumable oil (i.e., *Jatropha curcas* oil (JCO)) as a raw material was provided by the Chinese Petroleum Corporation (CPC) in Kaohsiung City, Taiwan. Methanol and sodium hydroxide were procured from Burdick & Jackson (UNI-ONWARD Crop, New Taipei City, Taiwan). The methanol and sodium hydroxide (NaOH) 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. The presence of high acidity means that the oil can go rancid.

2.2 Catalyst synthesis

2.2.1 Calcination of eggshells

The collected waste eggshells were washed thoroughly with clean tap water to eliminate dirt, impurities, and organic materials. Subsequently, the waste eggshells 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.

2.3 The transesterification procedures for the JCO

The JCO sample employed under the present work was provided by CPC in Kaohsiung City, Taiwan. In this research work, an MW synthesis reactor (PreeKem APEX, Shanghai, China) fitted out with an automated stirrer, and a capacitor was employed to enable MW reactions. The experimental system for this study is shown in Fig. 1. 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), MTOR (9:1), and a temperature reaction of (45, 55, 65, 75, and 85°C) were consistently investigated during the analysis. With the application of the 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. Rabelo et al. [31] quantified fatty acid methyl esters 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. [20] 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

The Chinese Petroleum Corporation (CPC) in Kaohsiung City, Taiwan, provided the JCO sample used in the experiments. The JCO sample used under this investigation was found to have an acid value of 2.8 mg KOH/g and a saponification value of 195.12 mg KOH/g. Additionally, the JCO sample had a free fatty acid (FFA) value of 2.5 wt.% with an average molecular weight of 861.01 g/mol and water content of 0.12 wt.% [4, 6]. The Hammett indicators were acquired from Sigma-Aldrich (Kaohsiung City, Taiwan). JCO sample was found to have a pH value of 5.8. The methanol, sodium hydroxide (NaOH) and all other chemicals employed in our experiments were of HPLC analytical grades.

2.5 Decontamination and separation procedures

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).

2.6 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 volume during this study was determined using the Gas Chromatography (GC-6890, Agilent, USA) method furnished with the flame ionization detector (FID). The content of methyl ester in this study was determined by employing the following equation:

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

where $\sum A$ is the total of the peak area of FAME from C_{14} to $C_{24:1}$; A_{EI} is the peak area of the interior average, 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 as follows:

$$\text{Methyl ester yield} = \text{methyl ester content} \times \text{biodiesel yield} = C \times \frac{W_B}{W_{oil}} \times 100\% \quad (2)$$

where C is the methyl ester volume (%); W_B is the quantity of biodiesel synthesis (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].

3 Results And Discussion

3.1 Catalyst characterization

3.1.1 Evaluation of eggshells through XRD technique

We studied the eggshells before and after calcination through the X-ray diffraction (XRD, HR-XRD D8 SSS, Bruker Co.) analysis technique. This was carried out and determined by XRD patterns of the uncalcinated and calcinated eggshells before (**Fig. S1 (a)**) and after calcination (**Fig. S1 (b)**) are illustrated in **Fig. S1** of the Supplementary Materials. According to the results, the occurrence and strong peaks were located at a bragg angle of (2θ) at 29.6° , 39.6° , 47.6° and 48.7° for the recycled WES upon calcination (**Fig. S1 (a)**). However, strong peaks were equally noticed after calcination (**Fig. S1 (b)**) and were detected an angle of (2θ) at 32.4° , 37.6° , 54.0° , 64.3° and 67.6° , in accordance with the bands of CaO [6] compared with the peaks of calcined and uncalcined snail shells through the application of the XRD technique.

In the earlier investigations, it has been discovered that the snail shells was calcined at 900°C and indicated the presence of CaO with strong peaks of bragg 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 involves of peaks 2θ located at several points coordinating from 26.242° , 33.172° and 45.889° , suggesting that snail shell component comprised of CaCO_3 [21]. 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 transformed into a fine white powder, firmly demonstrating that it was converted into CaO. By comparison, the band of XRD assessment and the points were discovered at an angle of (2θ) located at several degrees aligning from 32.46° , 37.64° , 54.12° and 62.60° , and this moreover, attests to the presence of CaO [6]. Takeno et al. [14] described cubic crystals in Calcined Silver Croaker Stone (CSCS), 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 reaction of soybean oil [22].

3.1.2 FTIR analysis

The FTIR analysis technique was applied to characterize eggshells before calcination (**Fig. S2(a)**) and after calcinated (**Fig. S2(b)**), though calcination was happening at 1000°C for 2 h. The assimilation bands, in this case, are presented in **Fig. S2** of the Supplementary Materials, recorded in the $400\text{--}4000\text{ cm}^{-1}$ bandwidth. According to the results, pre-calcination

absorption bands were situated at various peaks, stretching from 1417, 879 and 713 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 3639, 1413, 1099, 875, 532 and 432 cm^{-1} . The expanded matrix oscillations of the CaO bond are indicated by large, strong absorption peaks of approximately 713 and 524 cm^{-1} . Asymmetric expansion and contraction of C-O bonds in unknown carbonate species was associated with peaks of 1415 cm^{-1} and 875 cm^{-1} . There is a large peak of about 3643 cm^{-1} due to the creation of -OH bonds at $\text{Ca}(\text{OH})_2$ 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 $\text{Ca}(\text{OH})_2$ compounds forming soaps in acidic oils and leaching catalysts, which reduces yields in subsequent series or runs. These FTIR bands' 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 existing in the produced catalyst. In light of this, it was revealed (after calcination) that individual alkaline-earth metals such as C (6.25%), N (2.53%), O (20.52), Fe (2.28%), Mg (1.65%), and Ca (66.77%) as constituents of the developed CaO-based eggshells catalyst as displayed in **Table S1** of the Supplementary Materials.

The determined EDS results are consistent with the XRD. Rahman et al. [23] reported that the catalyst 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 [24]. Figure 2 shows the WES material structure, prior calcination (Fig. 3a) and after calcination (Fig. 3b) at high temperature. The images were obtained with the support of the Scanning Electron Microscope (SEM) technique, specific surface area (Micromeritics, ASAP 2020), and chemical configurations of the developed catalyst were determined. It is evidently illustrated from Fig. 3 that the pore structures in the eggshell showed extensive, substantial disparities before (Fig. 3a) and after calcination (Fig. 3b).

Consequently calcination, there should be significant modifications in the shells' physical structures, signifying that the transformation of CaCO_3 into CaO has subsequently 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 one revealed by Nisar et al. [18] for calcined waste animal bones. The calcined bones were irregular, and some stuck together. These results indicate that the calcination temperature affects crystallinity formation and that higher temperatures produce sharper cubic crystals than lower temperatures. For comparison, Krishnamurti 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 BET-BJH measurements

The dimensions for the surface area and the absorbency depiction of the developed catalyst were investigated using N_2 physisorption with a Brunauer-Emmer-Teller (BET) system (AUTOSORB-1C Quantachrome Corporation) technique. While the permeability and surface area of the developed CaO-based eggshell catalysts were investigated with the help of the BET system. As for the adsorption of nitrogen gas employing the micromeritics equipment, the general pore capacity and average pore dimension of the developed catalysts were calculated using the BJH technique. The outcomes are displayed in Table 1. 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 6.9592 to 6.2214 m^2/g with a typical BJH adsorption pore measurement of pore size between 19.35 and 31.39 nm as demonstrated in Fig. 3. Palitsakun et al. [25] reported a much lower significant surface area and pore volume measurements for CaO-T catalyst with a surface area of 1.95 m^2/g and an average pore volume of 0.0191 cm^3/g , while the CaO-H catalyst had a surface area of 23.30 m^2/g with an average pore volume of 0.2012 cm^3/g . The CaO-P catalyst was found to have a surface area of 1.76 m^2/g and an average pore

volume of 0.0155 cm³/g. Rahman et al. [23] have found that a solid catalyst's higher surface area can improve its catalytic performance.

Table 1 BET surface area measurements and porosity characterization of the CaO-based catalyst

Calcination temperature (°C)	Catalyst	Basic strength ^a (H _L)	Basicity ^a (mmol g ⁻¹)	Surface Area ^b (m ² /g)	Pore volume ^b (cm ³ /g)	Pore size (nm) ^b
800	WES 500	7.2 < H _L < 9.8	0.74 ± 0.34	6.9592 ± 0.0645	0.0178	19.35
9000	WES 600	15.0 < H _L < 17.2	0.97 ± 0.41	6.3672 ± 0.0769	0.1685	26.73
1000	WES 700	12.2 < H _L < 15.0	1.21 ± 0.36	6.2214 ± 0.0673	0.11.81	31.39
^a Determined by Hammett indicator.						
^b Analyzed by N ₂ -physisorption.						

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

3.2.1 Determination of transesterification process kinetics and activation energy

To investigate the transesterification reaction's kinetics, the effects of temperature and 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 Fig. 4, the graph reveals that the transesterification reaction rate is much slower at the initial phase of the reaction, and, subsequently, the reaction rate increases considerably. Because of plotting $-\ln(1-X)$ vs. t (Fig. 4), linear regression equation at diverse reaction temperatures or standards of kinetic model parameters was attained, and the results are presented in **Table S2**.

Regression equation appertaining to Fig. 4 was established, and it was observed to be linear with high regression coefficient ($R^2 = 0.9994$). Furthermore, the Arrhenius plot for determining activation energy of transesterification reaction was obtained by plotting $\ln k$ vs. $1/T$. Therefore, the reaction's activation energy (E_a) was determined as 9.58 kJ mol⁻¹. The acquired results in this regard are presented in Fig. 4.

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 E_a is attained by exploiting different feedstock (oils) in the transesterification reaction, as shown in Table 2. Mazubert et al. [28] 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 [29], Foroutan et al. [20] and Al-Sakkari et al. [11] have recorded high activation energy of 26.56, 45.53 and 48.56 kJ mol⁻¹. In comparison, Pavlović et al. [19] and Rathore et al. [30] reported very high activation energy (67.17; 58.03 and 54.5 ± 2 kJ mol⁻¹) as compared to the present study. Uzun et al. [31] used a conventional heating method and reported a very low activation energy of 11.741 kJ mol⁻¹. These different results could be guaranteed positive insight to the understanding of both practical and hypothetical in the generation of biodiesel.

Table 2
Activation energy (E_a) on transesterification reaction of biodiesel production from different studies

Feedstock	Heating Method	Catalyst	Catalyst Phase	Activation energy (kJ/mol)	Refs.
WCO	Microwave	Conventional	—	9.7	[28]
WCO	Microwave	Heterogeneous	Calcium diglyceroxide (CaDG)	26.56	[29]
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]
Karanja (Pongamia)	Conventional	Homogeneous base catalyst	KOH (9%)	54.5 ± 2	[30]
WFO	Conventional	Conventional	Basic catalysts	11.741	[31]
Soybean oil	Conventional	Heterogeneous	Cement kiln dust	48.56	[11]
WCO	Conventional	Heterogeneous	Biochar/CaO/K ₂ CO ₃ catalyst	45.53	[20]
<i>Jatropha curcas</i> oil	Microwave	Heterogeneous	CaO catalyst prepared from eggshells	9.58	This work

3.2.2 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 procedure. On the other hand, when the percentage of 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. 5 (a) revealed that catalyst loading significantly influenced biodiesel yield. As shown in Fig. 5 (a), the biodiesel yields increased when the catalyst loading was increased from 3–4%.

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 91.7–87.4%. Other researchers highlighted that excessive catalyst loading might impede the mixing of methanol, oil and catalyst, which may further lead to the separation stage [4, 6]. Therefore, 5 wt.% catalyst loading was the best catalyst amount during this study.

3.2.3 Effect of reaction time

The effect of reaction time on biodiesel yield is reported in Fig. 5 (b). Different experimental conditions were as follows: MTOR 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. 5 (b), the biodiesel yield had significantly increased as the reaction time continued to increase. Subsequently, this trend has resulted in a high biodiesel yield of 91.7% in 180 min. However, and afterwards, the biodiesel yield began to decline insignificantly as the time was further increased from 195 min to 210 min. The possible elucidation of this result could be that as the reaction time was at 180 minutes, the reaction had probably already reached its equilibrium phase. In addition to this, 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 [32].

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 eggshell waste 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 [32]. Palitsakun et al. [25] argued that methanol normally activates CaO. A small amount of CaO is converted to the parental $\text{Ca}(\text{OCH}_3)_2$, which is more catalytically active than inactivated CaO. On the other hand, the FAME yield rose substantially from 54.78–88.09% in 80 min and remained steady as the reaction time intensified to 120 min, indicating that the system reached equilibrium.

3.2.4 Effect of MTOR

The influence of MTOR on biodiesel yield was investigated under the following conditions: MTOR of 9:1, 5 wt% catalyst loading, 180 min reaction time, temperature 65 °C and MW power of 800 W. MTOR has a similar effect analogous to the loading of catalysts, specifically on the reaction frequency and biodiesel yield in particular. As a result, the study on the transesterification reaction was conducted by adjusting the MTOR while employing the enhanced catalyst loading (5 wt%) at 65 °C (Fig. 5 (c)). The results of this effect are presented in Fig. 5 (c). It can be determined from the results in Fig. 5 (c) that as MTOR increased from 7:1 to 9:1, a considerable increase in biodiesel harvest from 86.9 to 91.7% was notably observed. Despite that, as the MTOR was increased to 10:1, there was an insignificant decrease in biodiesel yield (90.1%). When the MTOR was further increased to 10:1, the biodiesel yields gradually decreased to 87.1% 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 initial results was likewise affirmed in the study conducted by Nath et al. [27] and Basumatary et al. [26]. Their studies recorded that large amount to MTOR 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 MTOR 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 [26]. In the present work, the most promising level for JCO transesterification to biodiesel was determined to be 9:1 of MTOR with 5 wt.% of CaO-supported catalyst generated from eggshell waste materials. For that reason, 9:1 was the optimal MTOR in this study.

3.2.5 Effect of reaction temperature

The effect of temperature on the biodiesel yield was studied under the following diverse conditions: MTOR of 9:1, 5 wt% catalyst loading, 180 min reaction time, temperature 65 °C and MW power of 800 W. The results are presented in Fig. 5 (d). According to the results in Fig. 5 (d), it was found that when the temperature was much lower than 65 °C, the biodiesel yield increased with increasing temperature and achieved the highest considerable yield at 65 °C. At this stage, the biodiesel yield was observed to be decreasing insignificantly as the temperature rose. Various studies envisaged reaction of this nature to be thermic [26], whereas other researchers anticipated it to be marginally exothermic [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.

Although high temperatures enhanced catalytic performance significantly, advanced reaction temperatures may subsequently lead to fast methanol evaporation, causing detrimental effects on improving biodiesel yields [4, 5]. Higher temperatures could speed up the saponification of triglycerides, which can cause adverse consequences on product yields [34].

3.3 Physico-chemical properties of biodiesel from JCO

Table 3 shows the summarized physico-chemical properties of the JCO biodiesel. The biodiesel physico-chemical characteristics are indispensable, 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 in the scope of 0.859–0.891 g/cm³, which is generally within the scope and appropriate range of fuel standard (ASTM D6751) or EN 14214 standard [35, 26]. In conforming to the low viscosity that is indispensable for satisfactory features of combustion in addition to non-deposition in the engine, the JCO biodiesel viscosity at 40 °C for JCO biodiesel is reported to be 3.624 mm²/s (as shown in Table 3) which complies with the normal limits [26]. Several studies have recounted on JCO biodiesel high values of viscosity at 40 °C, such as 4.75 mm²/s reported by Kumar et al. [35]. The JCO biodiesel is also equally reported to have a cetane number of 55.50, as it can be seen from Table 3, and this surpasses the lowest limit value of international standards, particularly for the ASTM D6751 and EN 14214, as well as near to the values of the cetane number of 56.67 as re-counted by Nath et al. [27] for soybean biodiesel. Conversely, JCO biodiesel is reported to have a pour point of -6 and a cold filter plugging point of -3.

Table 3
Physico-chemical properties of the synthesized *Jatropha curcas* biodiesel (ASTM D6751, EN 14214)

Properties	ASTM D6751	EN 14214	Jatropha biodiesel
Density (15°C, g/cm ³)	NS	0.8–0.90	0.878
Kinematic viscosity (40°C, mm ² /s)	1.9-6.0	3.5-5.0	3.624
Acid number (mg KOH/g)	0.5	NS	0.18
Cetane number	47 (min)	51 (min)	55.50
Cetane index	NS	NS	59.23
Pour point (°C)	NS	NS	-6
CFPP (°C)	NS	NS	-3
Saponification number (mg KOH/ g)	NS	NS	191.64
Iodine value (g I ₂ / 100 g)	NS	120 (max)	69.11
API	36.95	NS	34.28
Diesel index	50.4	NS	68.37
Aniline point	331	NS	199.47
HHV (MJ/kg)	NS	NS	40.53
Max-maximum; min-minimum; NS-Not specified; SN-Saponification number; CFPP-Cold filter plugging point; API-American petroleum index; HHV-Higher heating value (Calorific value). Sources: [26, 35].			

In contrast, it can be observed from Table 3 that the JCO biodiesel could have a saponification number of 191.64 mg KOH/g and a level of acidity of 0.18 mg KOH/g that are under the normal standard range 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 worthy to note that a significant proportion of desaturation can lead to fuel polymerization due to the development of epoxide due to the buildup of oxygen in twofold links. As it can be observed from Table 3, the JCO biodiesel has an iodine value of 69.11 g I₂ /100 g oil that falls further down the recommended upper threshold of 120 (EN 14214 standard). This iodine value is comparable with the results reported by Kumar et al. [35] for JCO biodiesel (74.5 g I₂/100 g). Additionally, other greater iodine concentrations of 119 and 121.91 g I₂/100 g were recorded in the study conducted by Sarma et al. [59] and Nath et al. [27]. JCO biodiesel is reported to have a higher heating value (calorific value) of 40.53 MJ/kg [26].

3.5 Comparative study of the synthesized catalyst

The results showed a significantly high yield of 91.7% for biodiesel within 180 min reaction time within ideal requirements of 9:1 MTOR and 5 wt% catalyst loading with 65 °C reaction temperature. The results in this study have been subsequently compared to various preceding studies in other related literature, specifically on waste biomass derivatives for catalysts of the heterogeneous nature that were utilized for the generation of biodiesel, as indicated in Table 4. Biodiesel production from JCO while exploiting various solid base heterogeneous catalysts derived from different sources such as by Teo et al. [36] and Vyas et al. [37] 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 91.7% biodiesel yields after the 9th and 5th cycles, respectively.

Table 4
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 (mins)	Reaction temperature (°C)	Yield (%)	Reusability	Refs.
1	Jatropha oil	CaSO ₄ /Fe ₂ O ₃ -SiO ₂ core-shell	9:1	12	4 h	120	94	9 cycles	[36]
2	Jatropha oil	Chicken eggshell (CaO)	12:1	5	1	65	98.2	4 cycles	[39]
3	Jatropha oil	Eggshell (CaO)	1:8	2	2.5 h	65	90	6 cycles	[38]
4	Jatropha oil	Heterogeneous catalyst from <i>Heteropanax fragrans</i> (Kesseru)	12:1	7	65	65	97.75	3 cycles	[26]
5	Jatropha oil	Heterogeneous KNO ₃ /Al ₂ O ₃	12:1	6	70	360	87	5 cycles	[37]
6	Jatropha oil	Eggshell	9:1	5	165	65	91.7	5 cycles	This study.

In other studies, the synthesis of biodiesel while exploiting different waste biomass derived heterogeneous catalysts, Chavan et al. [38] and Teo et al. [36] have recounted a diminished catalytic performance with lengthy reaction time (2.5 h and 4 h). This can perhaps be ascribed to significant smaller surface areas of the various catalysts used, particularly the WES catalyst under this research work. However, Joshi et al. [39] and Basumatary et al. [26] reported CaO-based heterogeneous catalysts with relatively outstanding catalytic performance (with 98.2% and 97.75% biodiesel yields) even though they may have low catalysts reusability or recyclability of 4 and 3 cycles as compared to that of the catalyst utilized under this present study which was recycled 5 times. Teo et al. [36] 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 discovered the existence of a high quantity of calcium (Ca) 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 [22, 23].

3.6 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]. The results of the reusability study of the catalyst are reported in **Fig. 6**.

Therefore, 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 redevelopment approach. Furthermore, the recycled catalyst was ascertained to produce a more than 90% biodiesel yield in the 1st cycle and gradually decreased to 89.7%. 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 less than 76% after the fifth cycle. However, in the 5th cycle, the yield declined to a more significant value. This may be due to the contamination of the active site with CO₂ and H₂O and 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 leaching can affect the lifetime of the developed CaO catalyst, and biodiesel can be contaminated with leached Ca particles [19].

This sluggish decline in the catalytic performance can be ascribed to various contributing factors such as the saturation and blockage of active surfaces sites as a result of absorption of organic molecules [23], the incidence of physical alteration of the catalyst during regeneration period, and the incomplete cleansing of the active species from the catalyst [23].

6 Economic And Environmental Cost-benefit Analysis

The circular economy notion is characterized by the prospect of reprocessing, recycling and producing new materials from existing products. From this perspective, waste 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 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 waste eggshell 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].

7 Conclusions

This research developed 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 91.7% under the measured, optimized reaction conditions. While the kinetic study parameters and activation energy (E_a) of the reaction were determined to be 9.58 kJ mol⁻¹, culminating that it is not an energy-intensive procedure. This study hypothesized that the regression coefficient (R^2) of correlations has a value of 0.9994, signifying 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 microwave heating systems as environmentally friendly methods for sustainable bio-waste management approaches. In addition, this study expands on existing state-of-the-art research we know about biofuel production using non-consumable oil feedstocks and bio-waste economically.

Declarations

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Authors' contributions

Kassian T.T. Amesho: Conceptualization, Visualization, Formal analysis/Software, Validation, Writing-original draft. **Yuan-Chung Lin:** Conceptualization, Funding acquisition, Methodology, Supervision. **Chin-En Chen:** Data curation, Investigation. **Pei-Cheng Cheng:** Data curation, Investigation.

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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.

Availability of data and materials

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

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Figures

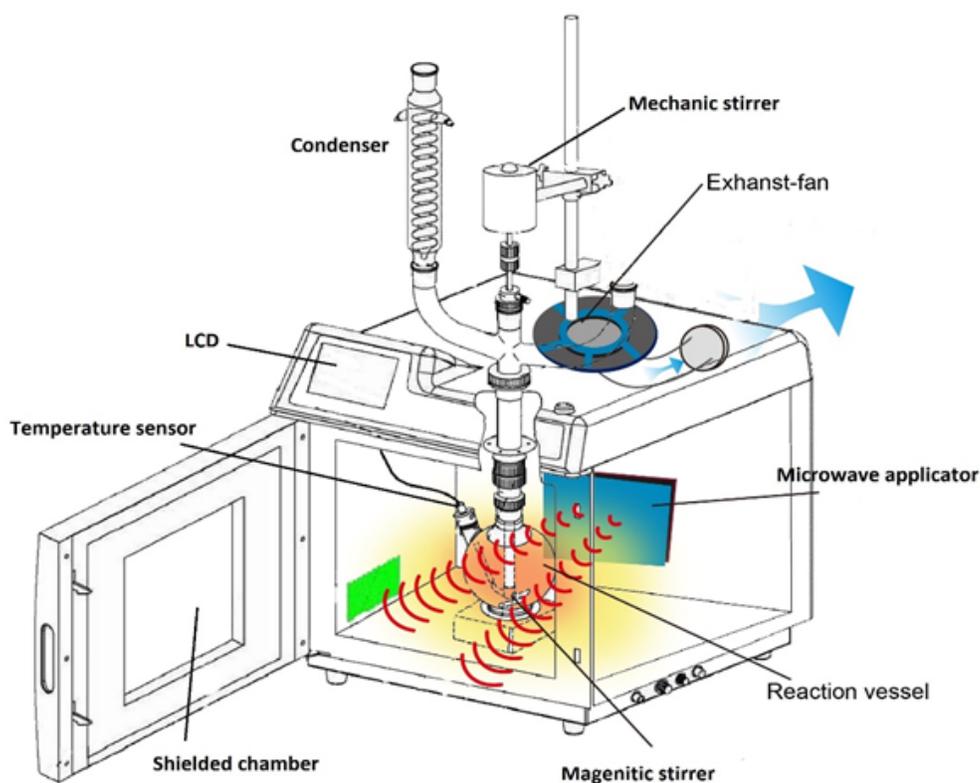
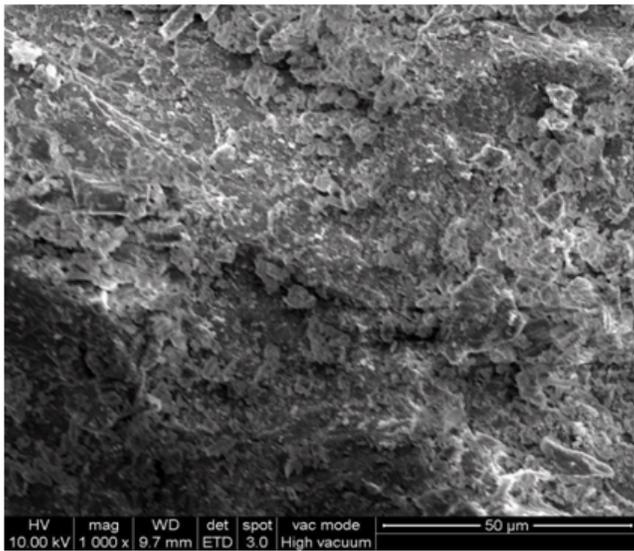
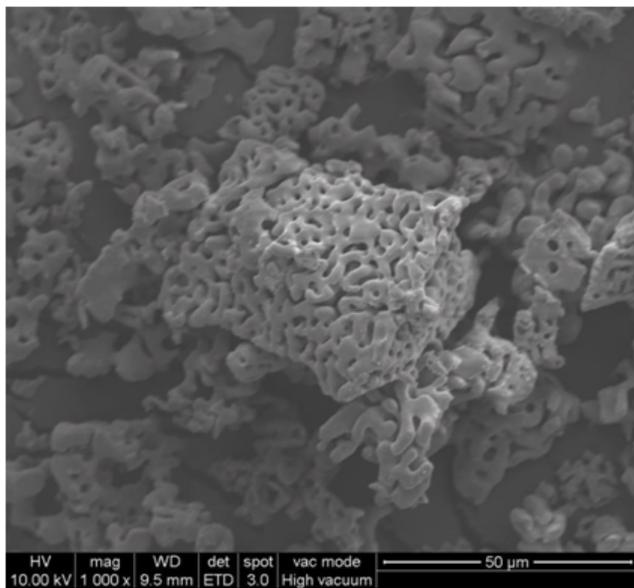


Figure 1

Schematic diagram of the experimental setup (microwave system structure)



(a) Eggshells - before calcination.



(b) Eggshells - after calcination

Figure 2

SEM image of eggshell before (a) and after calcination (b) at high temperature

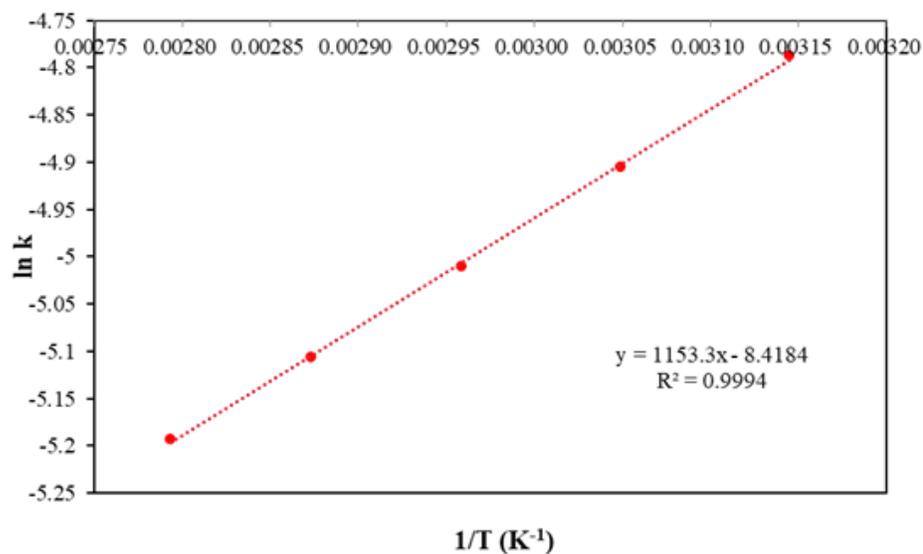


Figure 3

Arrhenius plot for determining activation energy. The relation curve in $\ln k$ and $1/T$. The plot of $\ln k$ versus $1/T$ in **Fig. 3** is found to be linear with high regression coefficient ($R^2 = 0.9994$). Reaction temperatures = 45, 55, 65, 75 and 85 °C; Activation energy (E_a) = 9.58 kJ mol^{-1} .

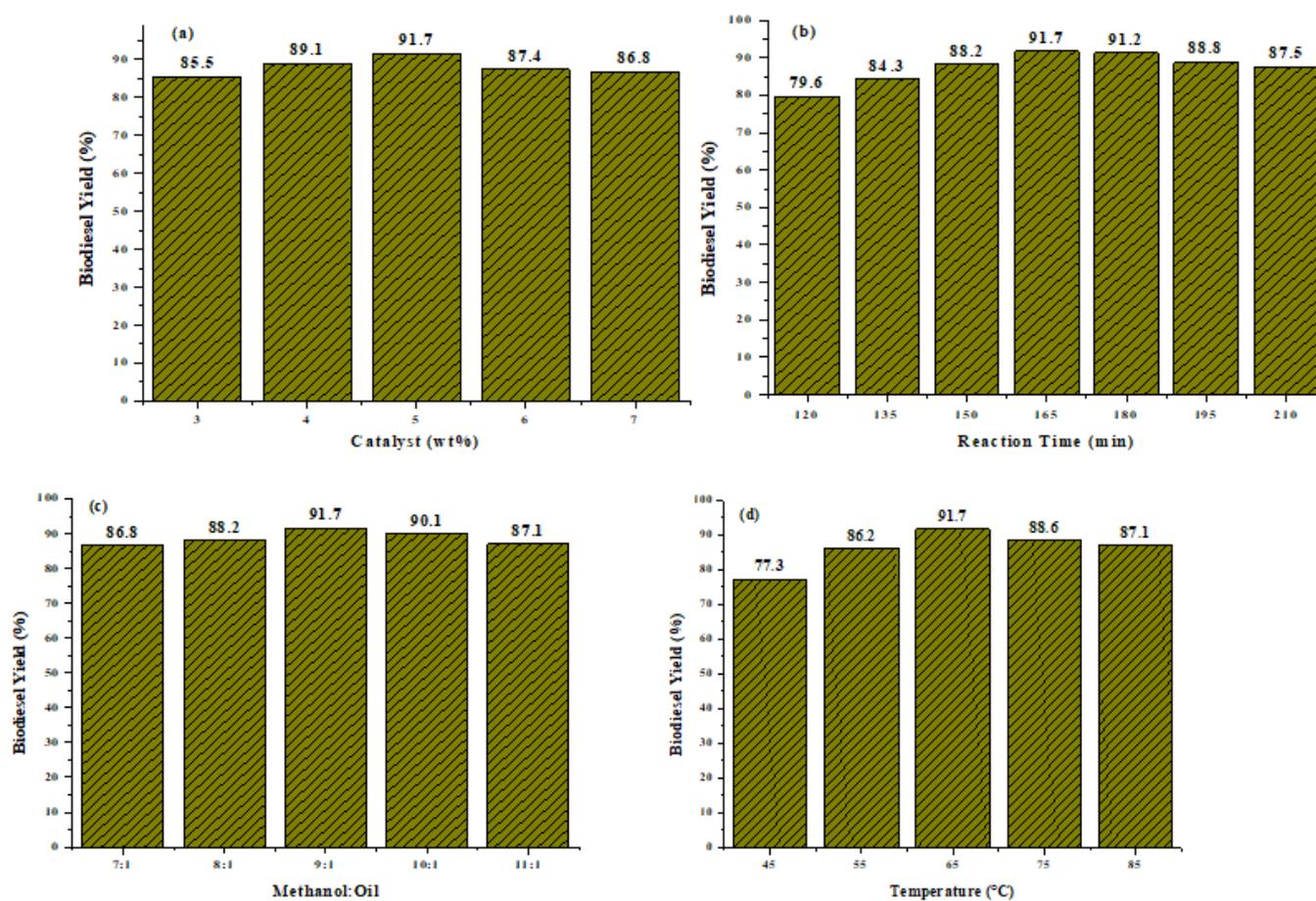


Figure 4

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

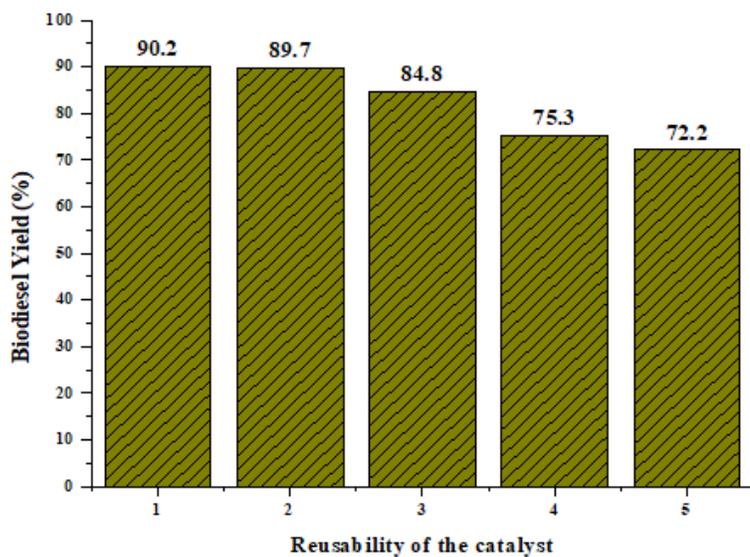


Figure 5

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

Supplementary Files

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