Revisi_Publikasi_CeO2CaO-1737421928511

by By Turnitin

Submission date: 21-Jan-2025 08:12AM (UTC+0700)
Submission ID: 2567960335
File name: Revisi_Publikasi_CeO2CaO-1737421928511.pdf (759.61K)
Word count: 8655
Character count: 43831

Contents list available at CBIORE journal website

International Journal of Renewable Energy Development

Journal homepage: https://ijred.cbiore.id

Review/Research Article

Optimization of Biodiesel Production from Candlenut Oil via Simultaneous Reaction Using a Bifunctional CeO_2.CaO Catalyst

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Abstract. The biodiesel synthesis process with a high free fatty acid content can be accomplished in a single stage using solid catalysts that function simultaneously as both base and acid catalysts. In this study, CeO_2.CaO was used as a bifunctional catalyst for biodiesel synthesis from candlenut seed oil. Catalyst characterization includes FTIR, BET, SEM-EDX, and TPD analysis. Process optimization was carried out using the central composite design method on Design Expert software. To determine the effect of each process variable on the simultaneous reaction, the effect of methanol-to-oil molar ratio, catalyst loading, and reaction temperature on FAME yield was also analyzed. The optimum operating conditions to achieve high FAME yield were found at methanol-to-oil molar ratio of 10.3:1, 5.39% w/w catalyst loading, and a reaction temperature of 60°C.

Keywords: Bifunctional catalyst, biodiesel, process optimization, candlenut oil, catalyst characteristics

Received: xxx; Revised: xxxx; Accepted: xxx; Available online: xxx

1. Introduction

The current era of energy crisis is urging researchers and governments to limit the use of fossil fuels because their reserves are dwindling and are also causing environmental damage (Hussain et al., 2021; Martins et al., 2018; Munfarida et al., 2020). Researchers are beginning to see a glimmer of hope from biodiesel, where this fuel can be produced from vegetable oils that are abundant in nature (Vilas Bôas & Mendes, 2022). Seeing the huge potential of biodiesel, various countries, both developed and developing, have begun to encourage massive biodiesel production, which is estimated to reach 36.5 million tons in 2025 and 277 million tons in 2050 (Mishra & Goswami, 2018; Rouhany & Montgomery, 2019). This biodiesel is very spectacular because it has a high cetane number and flash point with a low sulfur content, making its emission gas safe to be released into the air (Alsultan et al., 2021; Hartono & Cahyono, 2020; Wu et al., 2020). Biodiesel has the potential to be an environmentally friendly alternative energy due to its renewability and lower emissions (Gebremariam & Marchetti, 2018; Syazwani et al., 2019).

One of the non-edible oils that can be used for biodiesel synthesis is candlenut seed oil. Candlenut (*Aleurites moluccana Wild*) is one of the promising raw materials for biodiesel production because the seeds have an oil content of about 55 - 66% by weight and can also grow in various places, including infertile soils such as sandy soils, saline soils, and gravelly soils (Shaah et al., 2022). Candlenut oil contains α-eleostearic acid which is a toxic compound that makes candlenut oil non-food (non-edible oil). Candlenut seed oil has a high FFA (Free Fatty Acid) content of about 5.4%, so to be used in the transesterification process must go through an esterification process to reduce the free fatty acid content to below 2% (Siregar et al., 2024).

The high oil content of candlenut seeds and classified as non-edible oil, makes many researchers conduct research related to the manufacture of biodiesel from candlenut seeds so as to encourage the development of renewable energy. Pham et al. (2018), conducted research on making biodiesel from candlenut seeds using a two-step co-solvent process with an acid catalyst in the form of H_2SO_4 and a base catalyst in the form of KOH. This process has the disadvantage of using co-solvent which requires additional separation and purification to remove residual solvent from the final product. In addition, the catalyst used is still a homogeneous catalyst so that the catalyst is difficult to separate from the product, cannot be reused, and can produce hazardous waste. The non-catalytic supercritical methanol transesterification method was used by Shaah et al. (2022), which can produce biodiesel without the use of a catalyst. This method requires very high temperatures and pressures to achieve supercritical methanol conditions, so it requires expensive and high-risk specialized equipment. In addition, at supercritical conditions, side reactions such as decomposition of triglycerides, methanol, and biodiesel products can occur. Juwono et al. (2023), conducted a photocatalytic process of candlenut seeds with ZnO nanocatalyst. This method requires a strong light source

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https://doi.org/10.61435/ijred.x.x.xxx-xxx
ISSN: 2252-4940/© 20xx.The Author(s). Published by CBIORE

(usually UV light) so it requires higher energy consumption and greater operational costs. In addition, photocatalytic often has lower conversion efficiency compared to conventional methods such as transesterification because it is greatly affected by the distribution and light intensity of the UV light. Siregar et al. (2024), synthesized biodiesel using an acid catalyst in the form of H_2SO_4 and a base catalyst in the form of NaOH/CaO/Ca. The esterification and transesterification processes carried out are still inefficient because they require a long series of processes.

The biodiesel synthesis process in candlenut seeds, which have high FFA content, requires a two-stage reaction, i.e. esterification and transesterification. This two-stage reaction is certainly less effective because it requires a very long series of processes. A one-stage reaction is needed to overcome this problem. Solid-solid catalysts that work simultaneously are needed to be able to work as base catalysts and acid catalysts. The use of heterogeneous catalysts has several advantages such as being more environmentally friendly, easier to separate from liquid products, reusable, can be modified to increase activity, and longer service life (Faruque et al., 2020). Several researchers have synthesized acid-base bifunctional catalysts for biodiesel production in one reaction stage, including CeO_2.MgO (Manríquez-Ramírez et al., 2013), CeO_2.CaO (Wong et al., 2015), ZrO_2.CaO (H. Li et al., 2022), and V_2O_5.CaO (Mulyatun et al., 2023). In this study, CeO_2.CaO will be used as a bifunctional catalyst in the synthesis of biodiesel from candlenut seed oil. Process optimization will be conducted to find the best variables (methanol-to-oil molar ratio, catalyst loading, and reaction temperature) in the synthesis of biodiesel from candlenut seed with CeO_2.CaO catalyst.

2. Materials and Methods

2.1 Materials

The raw materials used for catalyst synthesis are limestone and cerium nitrate hexahydrate ($Ce(NO_3)_3.6H_2O$, 99%, Merck, Germany). The candlenut seeds used were from East Nusa Tenggara, Indonesia. Biodiesel synthesis was carried out using 96% methanol. N-hexane was used to regenerate the catalyst that had been used. KOH and phenolphthalein indicator were used to analyze the FFA content in the sample.

2.2 Raw Material Preparation

Limestone is crushed and then sieved using a sieve shaker with a size of 200 mesh. The limestone is then calcined using a furnace at 750°C for 4 hours to release the CO_2 and leave CaO (Zhang et al., 2018). CaO produced from calcination will be used for the synthesis of CeO_2.CaO catalyst.

Candlenut seeds are washed with warm water to remove dirt, then dried in the sun to dry the seeds. Extraction of candlenut oil using the pressing method will use a screw press machine which can produce large amounts of oil in a short time (Shaah et al., 2022). The oil that comes out of the press machine will be filtered, then cooled and its free fatty acid content will be checked.

2.3 Synthesis CeO_2.CaO Catalyst

CeO_2.CaO catalyst will be synthesized with a weight ratio of CeO_2 : CaO (1:4) through a physical mixing process. CeO_2 precursor in the form of $Ce(NO_3)_3.6H_2O$ powder and CaO will be mixed until homogeneous then ground together in a mortar until smooth. The bifunctional catalyst will be heated using a furnace at 600°C for 4 hours (Mulyatun et al., 2023). Before the catalyst is used, FTIR, BET, SEM-EDX, and TPD test will be carried out to see whether the catalyst is suitable for the reaction.

2.4 Catalyst Characterization

FTIR will be used to see the functional groups contained in the catalyst, as well as to see the bonds and interactions that occur on the catalyst surface (Mulyatun et al., 2023). FTIR testing will be carried out using Perkin-Elmer UATR Spectrum tool which is commonly used on a laboratory scale. BET testing will show the pore diameter and surface area of the bifunctional catalyst so that we can later determine the type of catalyst pores. This test will use the Quantachrome Autosorb-iQ tool. We also conducted SEM-EDX testing to see the appearance of the catalyst up close and to see what compounds were perfectly mixed in the catalyst. SEM-EDX test uses a JEOL JSM-6510LA SEM-EDX tool at 20 kV and 1.00000 nA probe. The acidic and basic properties of bifunctional catalysts play a crucial role in determining the success of simultaneous reactions, so TPD testing is required.

2.5 Biodiesel Production

Biodiesel will be synthesized in glassware in the form of a three-necked flask equipped with water as a coolant. Methanol and oil will mix in a three-necked flask with varying mole ratios (8:1, 10:1, 12:1). Bifunctional catalysts that play an important role will be varied at 2% w/w, 4% w/w, and 6% w/w. To see the effect of temperature on simultaneous reactions, the temperature of 50°C, 60°C, and 70°C were used. The reaction will last for 1 hour with stirring using a magnetic stirrer at 200 rpm. Separation by centrifuge at 5000 rpm for 5 minutes will be carried out to separate the catalyst from the reaction solution. The solution will then be separated further using a separating funnel for 24 hours (Al-Saadi et al., 2020).

2.6 Catalyst Reusability Test

To recover the catalyst, the catalyst will be washed using n-hexane solution. After washing, the catalyst will be heated for 6 hours at a temperature of 70°C. (Mulyatun et al., 2023). To test the reusability of the catalyst, repeated experiments were carried out at methanol-to-oil molar ratio of 10:1, loading catalyst of 4%w/w, and temperature of 60°C.

2.7 Biodiesel Characterization

Various tests will be carried out to meet the applicable SNI 7182:2015 standards. Biodiesel density testing can be done using a pycnometer. The formula for calculating biodiesel density is as equation (1) (Buchori et al., 2020):

$$\boldsymbol{\rho} = \frac{(\textbf{mass of pycnometer+sample}-(\textbf{mass of pycnometer})}{\textbf{volume of pycnometer}} \quad (1)$$

Biodiesel viscosity testing can be done using an Ostwald viscometer by calculating the time required for the biodiesel to travel from one point to another. Viscosity is then calculated with the equation (2) (Buchori et al., 2020):

$$\boldsymbol{\eta_x} = \frac{\boldsymbol{\rho_x}.\mathbf{t_x}}{\boldsymbol{\rho_a}.\mathbf{t_a}} \times \boldsymbol{\eta_a} \quad (2)$$

Where $\boldsymbol{\eta_x}$ shows the viscosity of the sample (mm^2/s), $\boldsymbol{\eta_a}$ shows the viscosity of the aquadest (mm^2/s), $\boldsymbol{\rho_x}$ shows the density of the sample (kg/m^3), $\boldsymbol{\rho_a}$ shows the viscosity of the aquadest (kg/m^3), $\mathbf{t_x}$ shows the travel time of the sample (s), and t_a shows the travel time of the aquadest (s).

FFA testing is carried out using KOH as a titrant. 3 ml of biodiesel will be mixed with 5 ml of methanol and 2 drops of phenolphthalein indicator then titrated using 0.1 N KOH. FFA

content can be calculated with equation (3) (Susilowati et al., 2019).

$$\%\mathbf{FFA} = \frac{\mathbf{MW}\times\mathbf{V}\times\mathbf{N}}{\mathbf{m}\times\mathbf{1000}} \times \mathbf{100}\% \quad (3)$$

Where MW is the molecular weight of fatty acids (gr/mol), V is the volume of KOH (ml), N indicates the normality of KOH (N), and m is the mass of the sample (gr).

Yield overall is the weight ratio between the reaction product and the raw material, i.e. candlenut oil. Yield overall can be calculated by equation (4) (Prameswari et al., 2022).

$$\mathbf{Yield\ overall}\ (\%) = \frac{\mathbf{mass\ of\ biodiesel}}{\mathbf{mass\ of\ candlenut\ oil}} \times \mathbf{100}\% \quad (4)$$

To identify the composition of the biodiesel product, a SHIMADZU QP 2010S Gas Chromatography-Mass Spectrometry (GCMS) equipped with a DB-1 column was used to analyze the FAME composition. FAME yield can be calculated by equation (5) (Mulyatun et al., 2024).

$$\mathbf{Yield\ FAME}(\%) = \%\mathbf{GC\ area\ FAME} \times \frac{\mathbf{m\ biodiesel}}{\mathbf{m\ candlenut\ oil}} \quad (5)$$

3. Results and Discussion

3.1 Characteristic of candlenut oil

One of the raw materials for biodiesel which is non-edible oil and has a high oil content is candlenut seed. Based on testing the Free Fatty Acid (FFA) content, the FFA value of candlenut oil is 2.287%. In the simultaneous reaction, FFA will be converted into biodiesel through esterification, while triglycerides will produce biodiesel and glycerol through transesterification. GC-MS analysis was also conducted to determine the fatty acid components of candlenut oil. Table 1. shows the results of GC-MS (Gas Chromatography Mass Spectrometry) analysis of candlenut oil.

Oils generally contain saturated fatty acids and unsaturated fatty acids. Saturated fatty acids do not have double bonds, while unsaturated fatty acids have double bonds (Li et al., 2019). Saturated fatty acids usually have a higher melting point than unsaturated fatty acids (Devi & Khatkar, 2016). GC-MS results show that candlenut oil contains saturated fatty acids in the form of hexadecanoic acid (palmitic acid) and octadecanoic acid (stearic acid). Candlenut oil also contains unsaturated fatty acids in the form of 9,12-octadecanoic acid (linoleic acid).

3.2 Characteristics of CeO_2.CaO catalyst

FTIR analysis was carried out to see functional groups in the CeO_2.CaO bifunctional catalyst. Fig. 1. shows the results of the FTIR analysis. The peak appears at a wavelength of 876 cm^{-1} refers to the C-O group of CO_3^{2-} which shows the presence of $CaCO_3$ on the catalyst (Maneerung et al., 2016). $CaCO_3$ can be formed due to the high affinity of Ca-O (Kesserwan et al., 2020). At a wavelength of 1408 cm^{-1} there is an O-Ca-O group, which indicates the presence of CaO formed (Mekonnen & Yesuf, 2024). Hydroxyl (O-H) groups, i.e. $Ca(OH)_2$ are seen at a wavelength of 3644 cm^{-1}. O-H groups are formed when there is water adsorbed to the surface of the catalyst. When the decomposition of nitrate is followed by a reaction with water, it will produce a $Ca(OH)_2$ compound (Maneerung et al., 2016). Peak formed at 515 cm^{-1} indicates the presence of O-Ce-O group which indicates the presence of CeO_2 on the catalyst (Prabaharan et al., 2016).

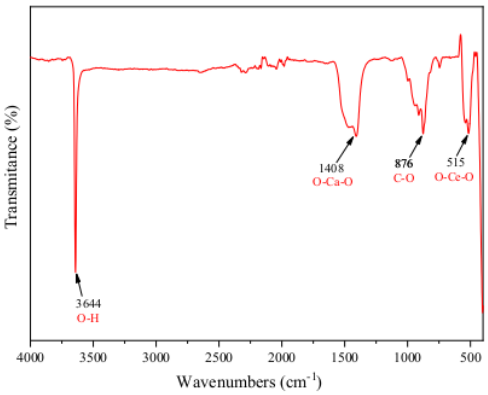

Fig. 1. FTIR spectrum of CeO_2.CaO

BET analysis results show that the average surface area of CeO_2.CaO catalyst is 9.536 m^2/g. Large surface area is triggered by high porosity in the catalyst. High porosity can form due to the presence of $CaCO_3$ which releases CO_2 during high temperature calcination (Mulyatun et al., 2023). The average pore diameter of the bifunctional catalyst is 5.876 nm. The pore diameter of CeO_2.CaO catalyst is in the range of 2-50 nm, which means including mesoporous catalyst. Mesoporous catalysts are excellent for transesterification reactions that require a lot of reactants. (Zhang et al., 2018). Based on BET analysis, the average pore volume of the catalyst is 0.028 cm^3/g. The $CaCO_3$ calcination process will release CO_2 gas so that pores appear in the catalyst (Mulyatun et al., 2023). Increased catalyst pores will increase the pore volume of the catalyst.

The adsorption-desorption curve of CeO_2.CaO bifunctional catalyst is shown in Fig. 2. The curve displays type IV characteristics which indicates that a mesoporous catalyst is formed. The curve also forms a hysteresis loop of type H3, where the loop closes at relative pressure more than 0.4 (P/P_0 > 0.4) which also indicates the presence of mesopores in the bifunctional CeO_2.CaO catalyst (Kingkam et al., 2024). The pore size in mesoporous materials ranges from 2-50 nm. Closed loops at P/P_0 between 0.4 - 0.5 indicate that the pore diameter is more than 2 nm (Mulyatun et al., 2024). The BET analysis also shows that the bifunctional catalyst CeO_2.CaO has a mesopore size of 5.876 nm which is more than 2 nm. Mesoporous catalysts provide advantages, where the reactants can contact the active site of the catalyst. Mesoporous catalysts can help in the transesterification reaction because triglyceride molecules can diffuse into the interior and contact with the active sites of the catalyst (Nayebzadeh & Hojjat, 2020).

SEM results show that CeO_2 and CaO are perfectly distributed with consistent size and shape. It can be seen that the CeO_2.CaO catalyst has macro and meso pore sizes which greatly support transesterification reactions. Triglyceride molecules will be able to penetrate the macropores so they can reach the active side of the catalyst (Zheng et al., 2017). The presence of mesopores can increase the surface area of the catalyst and also the pores will increase so that the active side of the catalyst will also be greater (Qian et al., 2022).

Table 1. Composition of candlenut oil

Compound	Trivial Name	Molecular formula	Area%
Hexadecanoic acid	Palmitic Acid	$C_{17}H_{34}O_2$	30.194
9,12-Octadecanoic acid	Linoleic Acid	$C_{19}H_{34}O_2$	67.821
Octadecanoic acid	Stearic Acid	$C_{19}H_{34}O$	1.985

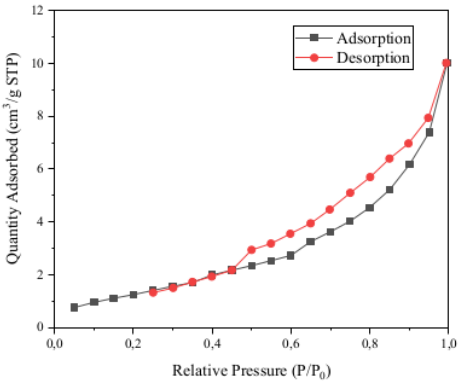

Fig. 2. Adsorption-desorption curve of CeO_2.CaO catalyst

The acidic and basic properties of bifunctional catalysts need to be tested to see their success in simultaneous reactions later. Peaks formed at temperatures below 300°C indicate weak acidic or basic properties. Peaks formed at temperatures between 400 - 500°C indicate medium acidic or basic properties, and peaks observed at temperatures above 500°C indicate high acidic or basic properties (Gil, 2023).

Fig. 4. is a picture showing the results of the TPD-NH_3 and TPD-CO_2 test. TPD-CO_2 provides data on the total base site density, which is 1.1971 mmol/g. Based on Fig. 4., it can be seen that two CO_2 desorption peaks were formed at temperatures between 200°C - 300°C and 400°C - 600°C. This shows that the CeO_2.CaO catalyst has a low and high basicity level. Strong basic properties can occur due to the presence of oxygen in the Ca-O compound. The synergistic bond between CaO and CeO_2 also increases the basicity level of the catalyst (Zhang et al., 2018). Fig. 4. also shows two peaks formed in the TPD NH_3 result. The peak formed at 150°C - 300°C indicates low acidity, while the peak formed at 400°C - 700°C indicates high acidity. The total acid site density obtained is 1.7202 mmol/g. The incorporation of CaO oxide with transition metal oxides such as ZnO, CeO, ZrO_2, dan W-Mo can cause changes in the acidity level of the catalyst (Basumatary et al., 2023). The presence of metal oxides in CaO, for example CeO_2 can encourage the transesterification reaction of non-edible oil in one reaction stage.

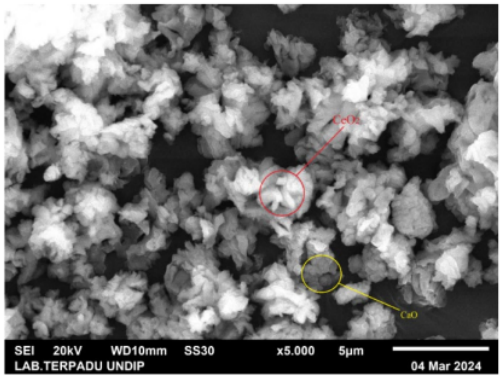

Fig. 3. SEM results of CeO_2.CaO catalyst with 5,000x magnification

3.3 Process Optimization

The experimental data obtained were then processed using Design Expert software with the Central Composite Design (CCD) process optimization method to see the optimum parameters. The number of runs in this study was 16 times with 2 center points. The model obtained is a quadratic equation as equation 4.

$$\text{FAME Yield} = 79,27 + 1,76X_1 + 10,62X_2 - 0,3271X_3 + 1,39X_1X_2 - 2,37X_1X_3 + 0,485X_2X_3 - 9,28X_1^2 - 7,76X_2^2 - 4,35X_3^2 \quad (6)$$

The dependent variable in the equation is FAME yield, while X_1, X_2, and X_3 are independent variables, i.e. the methanol-to-oil molar ratio, catalyst loading, and reaction temperature. The interaction between the three variables is described by $X_1.X_2$, $X_1.X_3$, and $X_2.X_3$. The square effect of each independent variable is described by X_1^2, X_2^2, and X_3^2.

Based on Table 2., it can be seen that the F value obtained is 165.31, indicating that the model is significant. The P value in the model is less than 0.05, indicating that the model is significant (Njoku & Otisi, 2023). A significant P value is also shown in variables B, AB, AC, BC, A^2, B^2, and C^2. The F value on Lack of Fit is 47.56 which indicates Lack of Fit is not significant. Lack of Fit which is not significant indicates that the model used is appropriate. The R^2 value obtained is 0.9960 where the value is close to 1. This shows that the model is good at predicting the response.

Biodiesel production from candlenut oil with CeO_2.CaO catalyst was carried out by varying the methanol-to-oil molar ratio, catalyst loading, and reaction temperature. The methanol-to-oil molar ratio was varied from 8:1, 10:1, and 12:1. The catalyst loading was varied from 2% w/w, 4% w/w, and 6% w/w. The reaction temperature was varied from 50°C, 60°C, and 70°C.

Fig. 5(a). shows the three-dimensional plot of the interaction between the methanol-to-oil molar ratio (A) and catalyst loading (B) on FAME yield. The three-dimension surface response shows that the Fatty Acid Methyl Ester yield increases as the methanol-to-oil molar ratio increases up to 10:1. The FAME yield decreased when it reached a mole ratio of 12:1. The increase in the amount of catalyst added to the reaction caused the FAME yield to increase as well. Al-Muhtaseb et al. (2021) producing biodiesel from loquat oil using a bifunctional CeO_2.CaO catalyst. The study showed that the optimal condition to achieve the highest yield of 90.14% was at a methanol-to-oil molar ratio of 9:1 and a catalyst loading of 4%w/w. These results are in line with the research conducted,

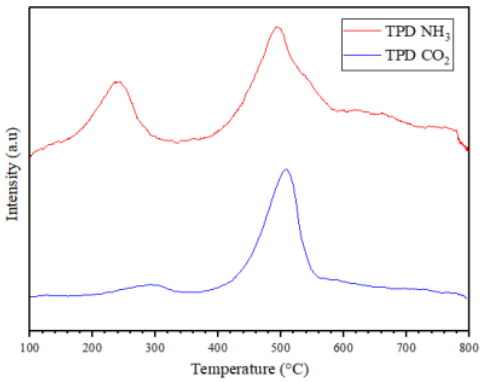

Fig. 4. TPD NH_3 and TPD CO_2 result

https://doi.org/10.61435/ijred.x.x.xxx-xxx
ISSN: 2252-4940/© 20xx.The Author(s). Published by CBIORE

where the optimal methanol-to-oil molar ratio for simultaneous reactions is ± 10:1 with a catalyst loading of ± 6% w/w. Esterification and transesterification reactions are reversible reactions so that excess methanol can drive the reaction towards the product. However, too much methanol can saturate the active sites of the catalyst thus inhibiting the protonation of triglycerides into carbonyl groups (Mulyatun et al., 2024). The more catalyst used in the reaction, the greater the number of active sites that contribute to biodiesel formation (Al-Muhtaseb et al., 2021).

The 3D response surface between the methanol-to-oil molar ratio (A) and reaction temperature (C) is shown in Fig. 5(b). Based on the figure, it can be seen that the yield of FAME increases at an increase in the methanol-to-oil molar ratio up to 10:1 and an increase in temperature up to 60°C. There is a decrease in FAME yield when the methanol-to-oil molar ratio reached 12:1 and the temperature of 70°C. Similar results have been found in biodiesel production using neem oil and heterogeneous copper doped zinc oxide nanocatalysts by Gurunathan and Ravi (2015). They found that the optimal operating conditions were at a methanol-to-oil molar ratio of 10:1 and a temperature of 55°C with a FAME yield of 97.18%. This is in line with the results of the study where the optimal methanol-to-oil molar ratio for biodiesel production from non-edible oil is 10:1 and at a reaction temperature of ± 60°C. The transesterification reaction is a reversible reaction so the more methanol is used, the more the equilibrium will shift to the right. Adding too much methanol can reduce FAME yield due to the catalyst dilution effect and makes methanol insoluble (Mulyatun et al., 2024). Reaction temperature plays a very important role in a reaction because the higher the temperature, the faster the molecules will move. Too high a temperature can cause methanol to evaporate quickly and reduce the concentration of reactants (Lani et al., 2022).

Fig. 5(c). shows the interaction plot between catalyst loading and reaction temperature on FAME yield. An increase in catalyst loading causes an increase in FAME yield. The FAME yield also increased at an increase in temperature up to 60°C. The increase in temperature up to 70°C causes a decrease in FAME yield obtained. Wong et al. (2015) produced biodiesel from palm oil using CeO_2.CaO catalyst and obtained the highest yield (95%) at 5%w/w catalyst loading and 65°C temperature. This is in line with the research conducted, where the optimal catalyst loading is ± 6% w/w and the temperature is ± 60°C. The higher the concentration of catalyst used, the active side of the catalyst used as a reaction site will also increase (Mulyatun et al., 2022).

The relationship between reaction temperature and kinetic energy of reactant particles is directly proportional, where the higher the temperature will cause collisions between particles and the reaction rate becomes faster. However, reaction temperatures higher than the boiling point of methanol will cause methanol to vaporize and reduce the concentration of reactants (Al-Muhtaseb et al., 2021).

Based on optimization using CCD in Design Expert software, it was found that the optimum parameters for biodiesel production from candlenut oil were at a methanol-to-oil molar ratio of 10.3:1, catalyst loading of 5.39%w/w, and temperature of 60°C. These optimum conditions are estimated to produce a FAME yield of 83.1125% with a desirability value of 0.959. The desirability value shows that from the FAME yield target (85%) there is a 95.9% possibility that the target can be achieved.

3.4 Characteristics of biodiesel

The constituent components of biodiesel can be determined by GC-MS (Gas Chromatography Mass Spectrometry) testing. The data presented are the GC-MS results from variable 4 which has the highest %GC Area FAME. GC-MS analysis showed the presence of FAME compounds in the resulting product with the main components contained in biodiesel are methyl palmitate (44.07%) and methyl stearate (15.53%). This proves that the bifunctional CeO_2.CaO catalyst is successfully used for simultaneous esterification-transesterification reactions and produces methyl esters.

Tests on the calorific value, density, viscosity, and %FFA of biodiesel were also carried out to determine the characteristics of the products produced. The value of the analysis results was then compared with the value of the Indonesian National Standard (SNI) 7182: 2015 to determine the success of the CeO_2.CaO catalyst in producing biodiesel products. The results of biodiesel characterization can be seen in can be seen in Table 4.

FAME yield is the main standard in biodiesel production because consumers have minimum requirements that need to be met by biodiesel producers. Based on SNI 7182:2015, biodiesel should contain at least 96.5% FAME. The results we obtained are still below SNI standards. High FAME content indicates biodiesel contain less contaminants such as glycerol, methanol, or free fatty acids remain from the process.

Table 2. ANOVA process optimization

Source	SS	DF	MS	F	P
Model	2616.71	9	290.75	165.31	< 0.0001
A-Mole ratio of methanol : oil	42.11	1	42.11	23.94	0.0027
B-Catalyst loading	1540.40	1	1540.40	875.81	< 0.0001
C-Reaction temperature	1.46	1	1.46	0.8310	0.3971
AB (Interaction)	15.41	1	15.41	8.76	0.0253
AC (Interaction)	44.84	1	44.84	25.50	0.0023
BC (Interaction)	1.88	1	1.88	1.07	0.3408
A^2 (Squared)	797.14	1	797.14	453.22	< 0.0001
B^2 (Squared)	558.01	1	558.01	317.26	< 0.0001
C^2 (Squared)	175.07	1	175.07	99.54	< 0.0001
Residual	10.55	6	1.76		
Lack of fit	10.51	5	2.10	47.56	0.1096
Pure error	0.0442	1	0.0442		
Cor Total	2627.27	15			

R^2	Adjusted R^2	Predicted R^2	Adeq Precision
0.9960	0.9900	0.9660	37.9726

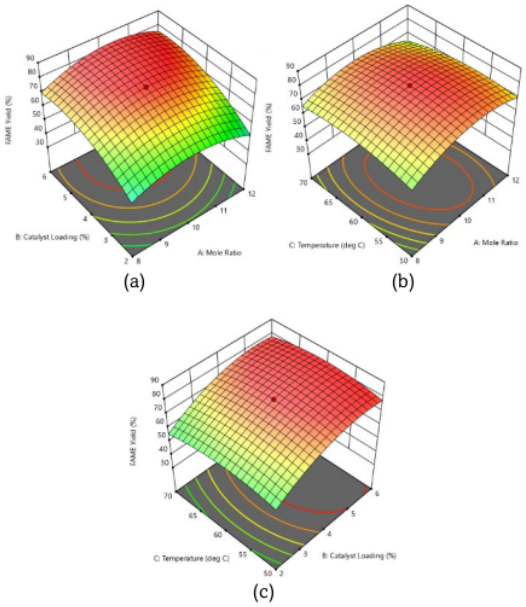

Fig. 5. Optimization graph of: (a) methanol : oil molar ratio vs catalyst loading (b) methanol : oil molar ratio vs temperature (c) catalyst loading vs temperature

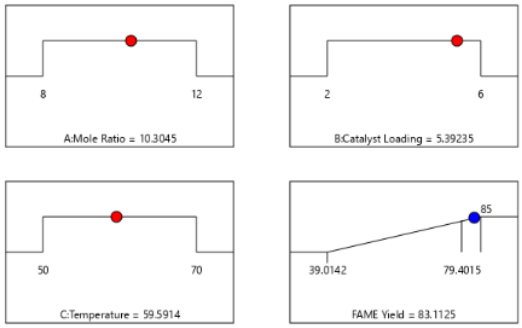

Fig. 6. Optimization of simultaneous reaction operating conditions

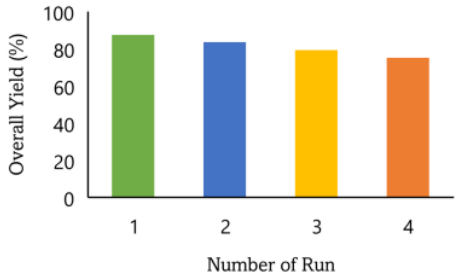

Fig. 7. Reusability of CeO_2.CaO catalyst

Table 3. FAME components in variable 4

Component	Molecular Formula	Area%
Hexadecanoic acid, methyl ester	$C_{17}H_{34}O_2$	44,07
11,14-Eicosadienoic acid, methyl ester	$C_{21}H_{38}O_2$	6,25
9,12-Octadecadienoic acid, methyl ester	$C_{19}H_{34}O_2$	10,71
13-Octadecenoic acid, methyl ester,	$C_{19}H_{36}O_2$	9,29
9,12,15-Octadecatrienoic acid, methyl ester	$C_{19}H_{32}O_2$	6,33
Octadecanoic acid, methyl ester	$C_{19}H_{38}O_2$	15,53
9-Octadecenoic acid, methyl ester	$C_{19}H_{36}O_2$	5,48

Contaminants contained in biodiesel can reduce engine performance and even damage the engine. High FAME content can minimize the presence of unsaturated fatty acids, which are more susceptible to oxidation (Tomić et al., 2019). High FAME content can also reduce the risk of fuel degradation and increase its shelf life.

The calorific value indicates how much heat can be released during combustion or indicates the energy capacity in the fuel. The calorific value of biodiesel from candlenut oil shows results of 9000 Cal/g. Cahyono et al. (2018) tested the calorific value of biodiesel from candlenut oil and obtained a value of 9387 Cal/g. This is in line with the research conducted, where the calorific value of biodiesel from candlenut oil is approximately 9000 Cal/g. A large calorific value indicates that the fuel is more efficient (Karmakar et al., 2018). A low calorific value of biodiesel will require more biodiesel mass to burn.

Another parameter to determine the quality of biodiesel can be seen from its density value. Based on SNI 7182:2015, the density requirement that must be met for biodiesel is between 0.85 - 0.89 g/cm^3 at 40°C. The results showed that some variables met the requirements, but there were nine variables that did not meet the requirements, i.e. variables 1, 2, 3, 5, 8, 9, 10, 11, and 13. The lowest density value produced was 0.87 g/cm^3, while the highest value obtained was 0.91 g/cm^3. The density value is related to the calorific value of biodiesel, where the higher the density, the higher the calorific value (Ozcanli et al., 2013). The high density value of biodiesel can be caused by the presence of glycerol in the biodiesel (Elgharbawy et al., 2021). Glycerol has a relatively high density of 1.26 g/cm^3. If glycerol is not completely separated from biodiesel, the density of biodiesel will increase. Biodiesel density that is higher than the quality requirement will cause an increase in particulate matter and NOx emissions (Yildiz et al., 2022).

Viscosity is a parameter that also needs to be considered in determining the quality of biodiesel. The requirements determined by SNI 7182: 2015 for biodiesel viscosity are 2.3 - 6 cSt. The results showed that most of the variables met the specified requirements, but there were several variables that did not meet the requirements, i.e. variables 4, 12, 13, 14, 15, and 16. The lowest viscosity value obtained was 1.1 cSt, while the highest viscosity value was 5.0 cSt. Viscosity that is too low will complicate the distribution of fuel so that the fuel will be difficult to burn and has the potential to cause leaks in the pipe (Gülüm & Bilgin, 2017). Viscosity of biodiesel that is less than the quality standard can also reduce combustion efficiency because the fuel will flow faster and the contact time with engine components will be reduced (Ferreira et al., 2021).

https://doi.org/10.61435/ijred.x.x.xxx-xxx
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Free fatty acid (FFA) levels can be a parameter for the success of esterification reactions in biodiesel production. If the FFA level in biodiesel is low or less than 2%, it indicates that the conversion reaction of FFA into biodiesel is successful. The research conducted showed that the FFA content in all variables had decreased from the initial candlenut oil FFA content of 2.287%. The decrease in FFA levels showed that the bifunctional catalyst CeO_2.CaO successfully carried out simultaneous reactions which included esterification and transesterification. Too high FFA content in biodiesel indicates a high amount of free fatty acids that can cause corrosion in diesel engines (Oni et al., 2022).

3.5 Catalyst reusability

In this study, the catalyst was reused 4 times in simultaneous reactions to see the reusability of the CeO_2.CaO catalyst. The operating condition used was the 15th run condition (center point), which was at a methanol-to-oil molar ratio of 10:1, 4%w/w catalyst loading, and a temperature of 60°C. After the reaction was complete, the catalyst was separated from the product, then washed with n-hexane several times and dried in an oven for 6 hours before being used in the next cycle. Fig. 7. illustrates the reusability of the CeO_2.CaO catalyst.

Based on Fig. 7., it can be seen that after reusing the catalyst 4 times, the overall yield can be maintained above 75%. Run 15 produced an overall yield of 89.3328%, then after reuse, the overall yield continued to decrease to 87.5135% after being used again for the first time, 83.6182% for the second times, 79.3382% for the third time, and 75.1719% for the fourth times. Deactivation of CaO-based catalysts can occur due to the leaching of CaO into the reaction medium. In addition, a decrease in yield can occur due to the adsorption of fatty acids, glycerol, or glycerides (triglycerides, diglycerides and monoglycerides) on the active sites (Zhang et al., 2018). Based on the catalyst reusability testing conducted, it can be seen that the bifunctional CeO_2.CaO catalyst has good stability. Reusability is an important factor of heterogeneous catalysts which can later be developed for industrial needs.

4. Conclusion

The acid-base bifunctional catalyst CeO_2.CaO was successfully synthesized by physical mixing method. From the catalyst characterization, it can be seen that CeO_2 and CaO are evenly distributed. The catalyst formed has acid-base properties and also has a mesoporous structure that is useful for transesterification reactions. The reuse of CeO_2.CaO catalyst for 4 times successfully maintained the overall yield value above 75%. In this study, it was found that the optimum operating conditions for biodiesel production from candlenut oil with CeO_2.CaO bifunctional catalyst were at a methanol-to-oil molar ratio of 10.3:1, catalyst loading of 5.39%w/w, and reaction temperature of 60°C.

Table 4. Characteristics of biodiesel

	FAME Yield (%)	Calorific Value (Cal/g)	Density (g/cm^3)	Viscosity (cSt)	FFA (%)
SNI 7182:2015	96.5	-	0.85-0.89	2.3-6	-
Variable 1 (8:1, 2%w/w, 50°C)	46.5495	9240.63	0.90	3.6	0.182
Variable 2 (12:1, 2%w/w, 50°C)	49.5539	9132.81	0.90	4.0	0.201
Variable 3 (8:1, 6%w/w, 50°C)	61.8463	9101.33	0.90	2.6	0.238
Variable 4 (12:1, 6%w/w, 50°C)	73.8413	9099.03	0.89	1.8	0.148
Variable 5 (8:1, 2%w/w, 70°C)	48.3060	9170.57	0.90	4.3	0.128
Variable 6 (12:1, 2%w/w, 70°C)	45.2787	9107.57	0.89	5.0	0.147
Variable 7 (8:1, 6%w/w, 70°C)	68.9815	9263.26	0.89	4.1	0.240
Variable 8 (12:1, 6%w/w, 70°C)	68.0677	9228.92	0.91	2.7	0.217
Variable 9 (6.6364:1, 4%w/w, 60°C)	49.3174	9024.41	0.90	2.5	0.292
Variable 10 (13.3636:1, 4%w/w, 60°C)	57.0013	9094.02	0.90	3.8	0.200
Variable 11 (10:1, 0.6364%w/w, 60°C)	39.0142	9215.68	0.90	3.9	0.237
Variable 12 (10:1, 7.3636%w/w, 60°C)	75.8753	9188.39	0.87	1.1	0.132
Variable 13 (10:1, 4%w/w, 43.2°C)	68.0849	9145.42	0.90	1.9	0.073
Variable 14 (10:1, 4%w/w, 76.8°C)	66.1164	9044.00	0.89	2.0	0.166
Variable 15 (10:1, 4%w/w, 60°C)	79.1042	8984.85	0.88	2.1	0.149
Variable 16 (10:1, 4%w/w, 60°C)	79.4015	8105.85	0.88	1.9	0.130

Acknowledgments

This study is funded by Faculty of Engineering, Diponegoro University, Indonesia

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