JEE Journal of Ecological Engineering

Journal of Ecological Engineering, 2025, 26(1), 248–259 https://doi.org/10.12911/22998993/195650 ISSN 2299–8993, License CC-BY 4.0 Received: 2024.10.13 Accepted: 2024.11.15 Published: 2024.12.01

Microwave assisted production of biodiesel using CaO nano-catalyst produced from mango fallen leaves extract

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ABSTRACT

With technological development, the use of modern methods in producing biofuels, especially biodiesel, has become necessary to make the process more sustainable and time-efficient. In this study, the feasibility of biodiesel production from waste cooking oil using CaO nanocatalyst produced from mango fallen leaves extract with the aid of microwave was explored. The effect of key parameters such as microwave power, methanol to oil w/w ratio, reaction time, and catalyst loading was studied. The results demonstrated that the highest yield of 96% was obtained at 10% microwave power at 15 minutes, 40% methanol to oil w/w ratio, and 3% catalyst loading. In addition, the augmentation in the temperature may cause to reduced yield of biodiesel. The physical and fuel characteristics of the produced biodiesel were measured; it had a viscosity of 5 mm²/s, a density of 0.889 g/cm³, and a flash point of 130 °C. In addition, the produced biodiesel was characterized by FTIR and gas chromatography-mass spectrometer (GC-MS) analysis which ensured the presence of methyl ester.

Keywords: biodiesel, CaO nanocatalyst, microwave, waste cooking oil, mango fallen leaves, transesterification.

INTRODUCTION

In recent years renewable energy has more attention because of problems related to environmental and economic [Al-Yaqoobi et al., 2021; Saleem and Al-Jubouri, 2024] The significant depletion of fossil fuels and the increase in their prices has caused a major impact on the global economy [Alshahidy and Abbas, 2021]. It has been reported that fuel consumption will increase by 60% or more in the next 25 years, to reduce this consumption many countries have produced alternative energy such as wind, water, and biomass which convert into biofuel [Abd et al., 2024; Abd and AL-yaqoobi, 2024].

In the 1980s, a simple reaction known as transesterification was introduced, defined as the reaction of the oil with an alcohol to produce biodiesel and glycerol [Nakkash and Al-Karkhi, 2012]. Biodiesel has gained great attention because of its properties like no noxiousness high degradability, higher cetane number, and higher flash point than petroleum diesel [Abbas and Abbas, 2015; Abbas and Flayeh, 2019]. The biodiesel is a mixture of alkyl esters containing an amount of oxygen [Na-kkash and Al-Karkhi, 2014; Mohammed and Jabbar, 2015].

Biodiesel can be produced from different sources edible vegetable oil, non-edible vegetable oil, animal and chicken fats and waste cooking oil [Doyle et al., 2016; Jurmot and Abbas, 2022] The use of waste cooking oil (WCO) as row material to produce biodiesel comes from two important eliminates the increase in the price of vegetable oil and the food versus fuel argument [Huda et al., 2023]. However, WCO required treatment before being used in the transesterification reaction where it has a high content of free fatty acid (FFA), the high (FFA) react with the catalyst and causes the formation of soap which leads to a decrease in the yield of biodiesel [Banerjee and Chakraborty, 2009].

Catalysts are an important factor in biodiesel production, and there are three types of catalysts: homogenous, heterogenous, and enzymes. The conventional process uses homogenous catalysts such as KOH, NaOH, and H₂SO₄ these types of catalysts are linked with significant problems such as corrosion in equipment, are difficult to remove from the product and are nonreusable [Khemthong et al., 2012]. Moreover, the homogenous acids catalyst has shown a slow transesterification rate of reaction [Rived et al., 2001]. On the other hand, MgO, BaO, CaO, and Zeolite are examples of heterogeneous catalysts [Mat et al., 2012]. Several studies have introduced different types of catalysts such as carbonate [Ebiura et al., 2005], potassium salt impregnated with fluoride compound [Fatimah et al., 2018], and aluminaimpregnated potassium iodide [Evangelista et al., 2012]. The most important feature of the heterogenous catalyst is that it can be recycled and tolerate up to 3% free fatty acid (FFA) [Mmusi et al., 2021]. Calcium oxide CaO is categorized as heterogeneous nanocatalysts which show high activity and a large surface area [Winoto, 2019], and it's also affordable, nontoxic, biodegradable, less corrosive, and can be removed easily from the product [Cholapandian et al., 2022].

Microwave is an electromagnetic irradiation equipment that lies between infrared waves and radio waves with a frequency range of 0.3–300 GHz and wavelength between 0.01–1 m. To avoid intervention with telecommunication and cellular phone frequencies most microwave oven used in domestic applications has a frequency of 2.45 GHz. In industrial applications, typical bands of 915 and 2450 MHz are used. Most laboratory microwaves and chemistry experiments are conducted at 2450 MHz and 12.24 cm wavelength [Al-yaqoobi, 2023; Gude et al., 2013].

Microwaves produce heat through their interaction with the materials at the molecule level without changing the molecular structure [Refaat, 2010]. There are several benefits to using microwaves for heating rather than more traditional methods. These include the reduction of thermal gradients, material selection and volumetric heating, rapid startup and shutdown, non-contact heating (minimizing surface overheating), energy transfer rather than heat transfer (penetrative radiation), and reverse thermal effect (heating from inside the material body). Improved heating efficiency, rapid catalyst heating, smaller equipment, quicker responsiveness to process heating control, quicker startup, higher output, and the removal of process stages are all potential benefits to biodiesel production [Tidwell et al., 2007].

The heating mechanism of microwaves is intricate. Compared to traditional heating methods It would provide the groundwork for comparing various heating. Both traditional and supercritical heating techniques work by first heating the vessel's outside and then the materials inside the sample, with the former using heat transfer to the latter. This is also called "wall heating". The conduction of materials and convection currents cause a significant amount of the energy that is delivered by traditional sources to be lost to the environment. The standard approach exhibits a heterogeneous heating effect that is influenced by the thermal conductivity of materials, specific heat, and density. This leads to greater surface temperatures, which in turn facilitate heat transmission from the outer surface to the internal sample volume. Consequently, we see varying temperatures in the samples and greater differences in heat distribution [Gude et al., 2013; Groisman and Gedanken, 2008]. While in microwave when a molecule has a dipole moment, it attempts to align itself with the electric field when exposed to microwave radiation. Due to the oscillating nature of the electric field, the dipoles continuously attempt to realign themselves to track this motion. The constant realignment of the molecules leads to friction, which in turn generates heat. When a molecule carries an electric charge, the microwave radiation's electric field component causes the ions to oscillate within the sample and collide with one another. This motion once again produces thermal energy [Refaat, 2010; Taylor, 2005]. Furthermore, because of the rapid interaction between the energy and the molecules, the molecules are unable to relax, resulting in the generation of heat that can, momentarily, exceed the total measured temperature of the reaction mixture so the heating will be from inside the body to outside which make the heating in the microwave more efficient than in conventional heating [Chemat-Djenni et al., 2007; Groisman and Gedanken, 2008]. Microwave irradiation is employed in chemical reactions to accelerate reaction rates by directly transferring energy to the reactants, increasing energy transfer efficiency compared to conventional heating. This enables the response to be completed in a shorter duration [Banković-Ilić et al., 2012; Motasemi and Ani, 2012].

The benefits of microwave-assisted processes include enhanced product quality and yield, reduced energy usage, and shorter reaction and separation times. Environmentally friendly. Insufficient methanol-to-oil ratio, Reduced generation of by-products [Nodede, 2016]. Biodiesel can be produced in microwave by both batch and continuous transesterification [Ansori et al., 2019]. Studies have demonstrated that the reaction time for transesterification by batch microwave-assisted can be substantially decreased to a matter of minutes compared to traditional transesterification methods.

In this study, biodiesel produced by using waste cooking oil and CaO nanocatalyst synthesis from mango leaves extraction in microwave different parameters were studied such as methanol to oil weight ratio (w/w), catalyst content, reaction time and irradiation power. Also, the physical and fuel properties of the produced biodiesel were investigated.

EXPERIMENTAL WORK

Materials

Calcium nitrate tetrahydrate $(Ca(NO_3)_2.4H_2O)$ was supplied by Thomas Baker, India with 98% purity, sodium hydroxide (NaOH) was supplied from Thomas Baker, India, methanol purchased from chem-lab NV, Belgium with 99.8% purity, isopropyl alcohol purchased from Alpha chemika, India with the minimum assay of 99.5%, and deionized water grade-I (extra pure). Phenolphthalein indicator.

Catalyst preparation and characterization

The mango leaves were washed and cleaned and then dried at 80 °C after that the leaves crashed until they became powder. The powder of the leaves boiled with deionized water to 60 °C for 25 min. Thereafter it is left to cold and filtrated to get leave aqueous extraction. The aqueous extraction was heated to 55 °C and NaOH and calcium nitrate tetrahydrate was added the reaction stopped when we got yellow paste then its calcination in the muffle furnace to 700 °C where CaO nanocatalyst was produced the catalyst was investigated by using XRF and found the CaO percentage in the sample was 98.662% then the catalyst characterization by BET, FT-IR, SEM-EDX, XRD. BET analysis shows that the produced CaO has a specific surface area of 64.989 m² g⁻¹ with a pore volume of 0.057623 $\text{cm}^3 \text{ g}^{-1}$ and a 2–10 nm average diameter. The average crystal size of the CaO nanocatalyst was determined to be 24.650 nm using the Debye-Scherrer equation. The EDX analysis revealed that calcium (46.11%) and oxygen (51.95%) were found in greater quantities. The preparation method, catalyst properties and characterization are reported by Mahmood and Al-Yaqoobi (Mahmood and Al-Yaqoobi, 2024).

Transesterification process by microwave

The feedstock in this study was waste cooking oil (WCO). The WCO was treated to remove any impurities and solid particles. The free fatty acid and water content were checked and found to be 1.83% and 0.04% respectively which is acceptable to the heterogenous catalyst which tolerates up to 3% (FFA) Figure 1 shows the GC-MS analysis of the waste cooking oil and the average molecular weight of waste cooking oil was 812.0203 g/ mol. All batch experiments were conducted in an atmospheric pressure microwave chemical reactor (WB20230745 Gongyi Yuhua Instrument Co., Ltd). The microwave power can be controlled in the range of 80-800 W the setup used in the experiment is shown in Figure 2. A round three-neck flask connected to a condenser and temperature sensor. In each experiment, 30 g of waste cooking oil was used, while the weight % ratio of methanol to oil was 30%, 40%, 50%, 60%, catalyst loading was 1%, 2%, 3%, 4%, 5 wt.% from oil, power level used was 10%, 20%, 30% from the total power of the microwave, the time was 5, 10, 15, 20 minutes. The reaction product was left to settle overnight, thereafter three layers were obtained the lower layer is the catalyst, the middle layer is glycerol and the upper layer is biodiesel which was collected and then separated from the catalyst by centrifuge and left in a dryer for 24 hours at 65 °C to evaporate the remaining methanol then the conversion was calculated by the Equation 1.

Yield
$$\% = \frac{Weight of biodiesl}{Weight of oil} \times 100$$
 (1)

RESUL AND DISCUSSION

The effect of methanol to oil weight ratio

The effect of the methanol to oil weight ratio has an essential impact on the production of biodiesel. In this study, the effect was studied at a fixed parameter of catalyst concentration of 3.33% w/w, 20% microwave power, and 10 minutes irradiation time with different methanol to oil ratios of 30–60 w/w. Figure 3 (a) shows that



Figure 1. GC-MS of the waste cooking oil



Figure 2. Atmospheric pressure microwave chemical reactor

at 30% w/w methanol to oil the yield was 78.2% then it increased until it reached the maximum yield of 87.9 at 40% w/w. Increasing the methanol to oil ratio beyond this ratio will decrease the yield until it reaches 83.856 at 60% w/w. This result could be attributable to an excess amount of methanol, where the glycerol becomes more soluble in biodiesel and develops foam formation, which results in redaction of the yield of biodiesel [Sharma et al., 2019]. Furthermore, the overloading of methanol inactivates the catalyst and induces a backward reaction [Erchamo et al., 2021].

The effect of catalyst loading

The catalyst is used to accelerate the reaction rate so the determination of the exact concentration of the catalyst is considered as an important parameter in the transesterification process. The effect of different concentrations of the

catalyst on the production of biodiesel is shown in Figure 3b. In this set of experiments, fixed parameters of 20% microwave power, 10 minutes irradiation time, and 40% w/w methanol to oil ratio were adopted. It can be noticed that the yield increases from 78.397% at a catalyst concentration of 1% to 86.3% at 2% until it reaches the highest yield of 91.2% at a concentration of 3%. Thereafter, the yield started to decrease with the increase of the catalyst concentration until it reached 71.3% at a catalyst concentration of 7%. The increase of catalyst concentration above the optimum value promotes saponification reaction, resulting in redaction of biodiesel yield [Chen et al., 2012]. In addition, the use of an excess base catalyst leads to the formation of the organic layer which causes the FFA to react with the catalyst to form a gel as shown in Figure 3 (c), consequently, the yield of the biodiesel is demoted [Ansori et al., 2019].

The effect of irradiation time

The irradiation was studied at different times 2, 5, 10, 15, 20, 30 and 40 min with fixed parameters of 20% microwave power, 40% w/w methanol to oil ratio, and 3% w/w catalyst loading. The effect of irradiation time on the transesterification reaction can be seen in Figure 3 (d). The yield of biodiesel increases from 79.34% at an irradiation time of 2 min to 87.4823% at 5 min until it reached its maximum yield of 91.1736% at 10 min. After that, the yield started to decrease to 84.248% by increasing irradiation time to 15 min until it reached 70.5016% at 40 min. From this, we found that after the optimum point, any increase in heat will decrease the yield by making the methanol evaporation and it will become insufficient amount in the reaction [Hsiao et al., 2020], also transesterification is a reversible reaction and saponification reaction will occur [Ansori et al., 2019].

The effect of irradiation time at different power

Figure 4a shows that the maximum yield time shifts with increasing microwave power, at 10% microwave power the maximum yield of 96% was obtained at an irradiation time of 15 min and at a microwave power of 20% the maximum yield of 91% was obtained at 10 min while increasing the power to 30%, the maximum yield of 88.87% was obtained at 5 min with the increasing the microwave power the maximum yield decreases and the time needed for reaction also decreases. This could result from a rise in the temperature of the mixture with the augmentation of the microwave power, which reaches above 250 °C at a microwave power of 30%. This speeds up the evaporation rate of methanol. Furthermore, microwave power has a strong impact on catalyst activity, where the increasing of reaction temperature above the optimum decreases the biodiesel yield and increases the saponification reaction [Mathiyazhagan and Ganapathi, 2011].

The effect of methanol to oil weight ratio at different power

The effect of methanol to oil weight ratio at different microwave power was studied at a fixed parameter of 10 min irradiation time and 3.33% catalyst loading with methanol to oil ratio of 30, 40, 50, and 60% w/w and different power 10, 20, and 30% of the total microwave power.

From Figure 4b it can be noticed that the higher yield of 90.4% was obtained at 40% w/w methanol to oil ratio at 10% microwave power, with a microwave power of 20% the highest yield of 87.94% obtained at also 40% w/w methanol to



Figure 3. (a) Study the effect of methanol to oil ratio w/w at 20% microwave power, 10 minutes irradiation time, and 3.33% w/w catalyst loading; (b) study the effect of catalyst loading at 20% microwave power, 10 minutes irradiation time, and 40% w/w methanol to oil ratio; (c) the organic layer gel formation around the catalyst; (d) study the effect of irradiation time at 20% microwave power, 3% catalyst loading, and 40% w/w methanol to oil ratio

oil, which is less than the yield achieved at 10% microwave power. This may be due to an increase in the temperature which increases the evaporation rate of methanol, decreasing the yield. At microwave power of 30% the maximum yield of 84.3% was obtained at 50% w/w methanol to oil ratio. At this power, the reaction needs more amount of methanol than the other power where the temperature is augmented in a short time which makes the methanol evaporate in large amounts and make it insufficient in the reaction system.

The effect of catalyst loading at different power

Figure 4c shows that the highest yield of 93.944% was obtained at 3% catalyst concentration at 10% microwave power, at 20% microwave power the maximum yield of 91.173% was also obtained at 3% catalyst concentration but it is less than the yield obtained at the same concentration of the catalyst at 10% microwave power this is may be due to the high increase in the temperature which caused to evaporation of the methanol and make the catalyst in excess amount in the mixture so the gel will formation around the catalyst and decrease the yield of biodiesel, with the 30% microwave power the maximum yield of 90.2243 was obtained at 2% catalyst concentration in this power the optimum catalyst concentration was decreased this is due to the highly rising in temperature which caused to speed up the methanol evaporation and make the catalyst in excess amount in the mixture in turned the gel layer will formation.

Comparison analysis

Table 1 illustrates a comparison of the optimal conditions employed for biodiesel synthesis from various feedstocks using CaO as a catalyst. The current study achieved a biodiesel yield of 96% at a microwave power of 80 W, utilizing a 40% w/w methanol/oil ratio, 3 wt.% catalyst, and a reaction duration of 15 minutes, indicating a superior yield under optimal conditions compared to prior research. However, Sharam et al. [Sharma et al., 2019] produced biodiesel by using CaO and waste cotton-seed cooking oil with a maximum yield of 89.94% but it needs a higher power of 180 W. Khemthong et al. [Khemthong et al., 2012] produced 96.7% biodiesel by using CaO nanocatalyst produced from waste eggshell and palm oil in 4 min but it needed a higher methanol to oil molar



Figure 4. (a) Study the effect of irradiation time with different power at 3% catalyst loading, and 40% w/w methanol to oil ratio; (b) study the effect of methanol to oil ratio w/w with different power at 10 minutes irradiation time, and 3.33% w/w catalyst loading; (c) study the effect of catalyst loading with different power at 40% w/w methanol to oil ratio, and 10 min. irradiation time

Feedstock (Oil)	Catalyst source	Methanol/oil (Molar ratio)	Temperature and power	Catalyst (wt%)	Reaction time (min.)	Yield% (wt%)	Ref.
wco	Mango leaves (NPS)	10.137	- 80 W	3	15	96	Current study
Waste cotton-seed cooking oil	Commercial	9.6:1	50 °C and a plus power (half min cycle: 8 s on and 22 s off) 180 W		9.7	89.94	[Sharma et al., 2019]
Palm	Waste eggshell	18:1	122 °C 900 W	15	4	96.7	[Khemthong et al., 2012]
Calophyllum inophyllum L.	Waste eggshell	9:1	- 325.24 W	3.88	12.47	98.9	[Ansori et al., 2019]
Jatropha Oyster and pyramidella 15:1 shell		- 800 W	4	5	93	[Buasri et al., 2015]	
Microalgae Nannochlorop-sis	Commercial	6:1	60 °C	3	3	96.46	[Hindarso et al., 2015]

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ratio of 18:1 and higher microwave power of 900 W and higher catalyst loading of 15 wt.%. In addition, biodiesel was produced by using jatropha oil and CaO nanocatalyst driven from oyster and pyramidella shell [Buasri et al., 2015], Despite a maximum yield of 93% in a short time of 5 min was obtained, a higher power of 325.24 W, higher methanol to oil ratio of 15:1, and higher catalyst concentration of 4% was needed.

Physical and fuel properties

The produced biodiesel was characterized for its physical and fuel properties and the results are shown in Table 2 along with the (ASTM-D6751) for biodiesel and (ASTM-D975) for petroleum diesel. The determined density was found to be 0.889 g cm^{-3} at 40 °C which is within the (ASTM D6751) range of biodiesel and equivalent to the value measured for petroleum diesel which is shown in Table 2. The flash point is the minimum temperature at which the liquid begins to evaporate and form an ignitable mixture within the air near the surface of the liquid, and this property is important in the storage and safety of fuel. The flash point of the produced biodiesel was found to be (130 °C) which is within the range of the ASTM for the biodiesel, and higher than the flash point of the petroleum diesel. This indicates that biodiesel is safer than petroleum diesel.

Viscosity is an important property of the fuel that may affect its operation. Viscosity is one of the properties that is affected by temperature change. At low temperatures the viscosity increases, and this may affect the fluidity of the produced fuel and impact the performance of fuel in injection equipment. The kinematic viscosity was found to be 5 mm² s⁻¹ at 40 °C which is within the range of ASTM value for biodiesel.

GC-MS

Gas chromatography-mass spectrometer (GC-MS) Agelint (7820A, USA) is used to detect

 Table 2. Comparison between the properties of waste cooking oil biodiesel and commercial diesel fuel [Tahira et al., 2012]

Fuel property	ASTM methods	Petro-diesel (D975)	Biodiesel standard (D6751)
Flash point, °C	D 93	60–80	100–170
Ash contents, wt%	D 482	0.01	0.01
Kinematic viscosity (40 °C, mm ² /sec)	D 445	1.9–4.1	1.9–6.0
Sulphur contents, wt%	D 4294	0.05	0.05
Cloud point,°C	D 2500	-15 to 5	-3 to 12
Pour point, °C	D 97	-35 to -15	-15 to 16
Centene number	D 613	> 46	47 min.
Density (40 °C, g/cm ³)	D 5002	0.834	0.86–0.90
Acid value (mg KOH/g)	D 664	0.50	0.80 max

chemical components of the produced biodiesel. Figure 5 shows the chromatogram for the synthesis of biodiesel. The results are demonstrated in Table 3 which shows the components identified in the biodiesel. Nineteen peaks were detected from the chromatogram. In the produced biodiesel methyl ester was the dominant ester.

FT-IR analysis

The waste cooking oil and produced biodiesel were analysed by infrared spectrometry to detect

the functional groups and bands in the range from 4000 cm^{-1} to 500 cm⁻¹.

Many studies inspected the composition of biodiesel by FTIR spectrum, the ester can be found in the peak from 1725 cm⁻¹ to 1700 cm⁻¹ [Kamaronzaman et al., 2020], the starching vibration band of carbonyl groups C = O for ester are found at the range of 1800–1700 cm⁻¹ according to Rabelo et al. [Rabelo et al., 2015]. Figure 6 shows FTIR analysis for waste cooking oil (WCO) and produced biodiesel according to the FTIR analysis the ester was observed at the absorption peak

Table 3. GC-MS Components identified in the produced biodiesel

Peak	Retention time	Name of component	Molecular formula	MW(g/gmol)	Peak area%
1	8.773	Undecanoic acid, methyl ester	C ₁₂ H ₂₄ O ₂	200	0.18
2	13.442	9-Hydroxy-decanoic acid, methyl ester	C ₁₁ H ₂₂ O ₃	202	0.12
3	17.179	Tridecanoic acid, 12-methyl-, methyl ester	C ₁₅ H ₃₀ O ₂	242	1.22
4	18.312	Pentadecanoic acid, methyl ester	C ₁₆ H ₃₂ O ₂	256	0.05
5	19.215	9-Hexadecenoic acid, methyl ester	C ₁₇ H ₃₂ O ₂	268	1.40
6	19.444	Pentadecanoic acid, 14-methyl-, methyl ester	C ₁₇ H ₃₄ O ₂	270	21.42
7	21.196	9,12-Octadecadienoic acid (Z,Z)-, methyl ester	C ₁₉ H ₃₄ O ₂	294	8.76
8	21.724	Methyl stearate	C ₁₉ H ₃₈ O ₂	298	10.01
9	21.833	10-Octadecynoic acid, methyl ester	C ₁₉ H ₃₄ O ₂	294	10.50
10	21.919	9- Octadecenoic acid (Z)-, methyl ester	C ₁₉ H ₃₆ O ₂	296	0.34
11	22.113	Methyl 9-cis,11-trans-octadecadienoate,	C ₁₉ H ₃₄ O ₂	294	0.95
12	22.793	Methyl 5,9-heptadecadienoate	C ₁₈ H ₃₂ O ₂	280	0.35
13	23.014	Oxiraneoctanoic acid, 3-octyl-, methyl ester	C ₁₉ H ₃₆ O ₃	312	0.87
14	23.163	9-Octadecenoic acid, 12-hydroxy-, methyl ester	C ₁₉ H ₃₆ O ₃	312	0.51
15	23.264	Methyl 5-eicosenoate	C ₂₁ H ₄₀ O ₂	324	4.07
16	23.506	Eicosanoic acid, methyl ester	C ₂₁ H ₄₂ O ₂	326	5.03
17	24.117	2(1H)-Naphthalenone, octahydro-4a-methyl-7-(1- methylethyl)-, (4a.alpha.,7.beta.,8a.beta.)-	C ₁₄ H ₂₄ O	208	0.59
18	26.029	Hexadecanoic acid, 2-hydroxy-1-(hydroxymethyl) ethyl ester	C ₁₉ H ₃₈ O ₄	330	17.58
19	26.228	Methyl 20-methyl-heneicosanoate	C ₂₃ H ₄₆ O ₂	354	16.05

Figure 5. GC-MS spectra of the produced biodiesel

Component	Waste cooking oil	Biodiesel
Methyl: O-CH ₃	1159 cm ⁻¹	1168.86 cm⁻¹
Methylene: O-CH ₂	1377 cm ⁻¹	1365.60 cm ⁻¹
Methyl: -CH ₃	1459 cm ⁻¹	1454.33 cm ⁻¹

Table 4. Comparison of absorption peaks between waste cooking oil and produced biodiesel by current study

Wavenumber (cm⁻¹)

Figure 6. FTIR spectrum for waste cooking oil and produced biodiesel

at 1741.72 cm⁻¹. Also Farooq et al. [Farooq et al., 2013] reported that the stretching vibration peaks of C – O ester groups appear in the peaks from 1125–1095 cm⁻¹ and this study shows peaks lie in this range which is 1107.14 and this ensures the existence of ester component in the biodiesel produced from this study. The stretching vibration of CH₃, CH₂, and CH groups appears at 2980-2950, 2950-2850, and 3050–3000 cm⁻¹ and the bending vibration CH₂ of these groups appears at 1475–1350, 1350–1150, and 723 respectively [Tariq et al., 2011].

From Figure 6 we notice that the FTIR spectrum of biodiesel and waste cooking oil are similar and there is a small difference that can be appeared by shifting the absorption peaks from 3010, 1743, 1459, 1377, 1245, 1159, 1097, 721, 596 cm⁻¹ in waste cooking oil to 3009, 1741.72, 1454.33, 1365.60, 1242.16, 1168.86, 1107.14, 723.31, 594.08 cm⁻¹ respectively in biodiesel. A new band appeared at 2677.20, 916.19, and 1653 cm⁻¹ as the waste cooking oil was transferred to biodiesel.

The value of absorption peaks appears in the produced biodiesel according to Figure 6 which are 3009, 2924.09, 2856.58, 1741.72, 1454.33, 1365.60, 1242.16, 1168.86, 1107.14, and 723.31 are agree and similar to absorption peaks found by Qiu et al. [Qiu et al., 2011] and Kamaronzaman

et al. [Kamaronzaman et al., 2020]. According to Goli and Shau [Goli and Sahu, 2018] biodiesel can be confirmed to be found by the presence of three peaks 1170.16, 1366.39, and 1458.19 cm⁻¹. In this study, we obtained similar peaks of 1168.86, 1365.60, and 1454.33 cm⁻¹ Table 4 shows a comparison of absorption peaks between unreacted oil and biodiesel obtained by the current study.

CONCLUSIONS

This study focuses on the production of biodiesel by using heterogenous catalyst CaO nanocatalyst produced from fallen mango leaves in microwaves by using different power and various reaction time under high temperatures above 100 °C and found that the production of biodiesel by using microwave application technology and heterogeneous nanocatalyst CaO appeared to be quite efficient concerning biodiesel yield, catalytic activity, reaction time, and energy efficient.

This means that the production of biodiesel by microwave reduced the reaction time needed for the transesterification process with improved catalytic activity compared to conventional heating. Also, it observed that the separation process needed the least time (a few hours) compared to the conventional process which need 24 hours to separate the biodiesel from glycerol.

Different powers were used to produce biodiesel 10%, 20%, and 30% of microwave power with different reaction times, methanol to oil w/w ratio, and catalyst loading this study shows that there is a shifting in maximum yield time with an increase in the power of the microwave, the yield was 96% at 10% microwave power with reaction time of 15 min. then the maximum yield reaction time shifting to 10 min. at 20% microwave power with 91% yield after that the maximum yield time of 30% microwave power was shifting to 5 min. with 88.87% yield.

Increasing the power caused to increase in the temperature above 250 °C caused to decrease in the activity of the catalyst by forming a gel layer around it. The produced biodiesel was analysed by FT-IR and GC Mass analysis which confirmed the production of methyl ester, and the physical properties of the produced biodiesel were calculated, and it was found to be compatible with ASTM.

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