Indian Journal of Chemical Technology
Vol. 33, September 2026, pp. 687-697
DOI: 10.56042/ijct.v33i5.30110

Biogenic synthesis of CaO nanoparticles from *Manilkara zapota* leaf extract and its application in biodiesel production from waste cooking oil

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Received 25 March 2026; accepted 4 August 2026

In this study, calcium oxide (CaO) nanoparticles were synthesised by a biogenic method using *Manilkara zapota* leaves. These nanoparticles were employed as a heterogeneous catalyst to yield biodiesel from waste cooking oil (WCO). The formation of nanoparticles was characterised using UV-visible spectroscopy, FTIR, scanning electron microscopy (SEM), and EDAX. The surface morphology of the nanoparticles revealed an irregular nanostructure. EDAX revealed the presence of calcium and oxygen as the primary elements. These results confirm the formation of calcium oxide nanoparticles. The catalytic activities of the synthesised nanoparticles were accessed via varying the reaction parameters such as concentration of the catalyst, the molar ratio of methanol to WCO, the reaction temperature, and the time. The maximum conversion efficiency of 96.1% was observed at catalyst concentration 1.5%, molar ratio of methanol to WCO 9:1 temperature 60°C and reaction time 120 min. GC-MS results revealed the formation of fatty acid methyl esters from the reaction between WCO and methanol. The results revealed that the calcium oxide nanoparticles synthesised by the green synthesis method using *Manilkara zapota* leaves act as an efficient heterogeneous catalyst to produce biodiesel from WCO.

Keywords: Biodiesel, Biogenic, CaO nanoparticles, Green synthesis, Heterogeneous catalyst, Waste cooking oil

Introduction

Environmental sustainability and energy security are two major issues in the 21st century. As fossil fuels are declining and climate change is becoming a serious issue due to greenhouse gas emissions, the use of alternative energy sources is becoming a priority. According to Rezaei *et al.*[1] fossil fuels, although holding a significant share in the global energy mix, emits a considerable amount of carbon, thus causing global warming and harming the environment. Therefore, to reduce climate change and build a sustainable energy system, a shift from conventional energy sources is required. A new alternative has emerged in the form of biodiesel, which is biodegradable and has low emission potential. Moreover, biodiesel is compatible with diesel engine technology since it is a viable alternative energy source[2].

The conversion process from triglyceride to fatty acid methyl esters (FAME) occurs by a process called transesterification, in which short-chain alcohols are used as catalysts. However, to achieve the best efficiency in the transesterification process, effective catalytic systems must be in place. Heterogeneous catalysts include NaOH and KOH, are recognized for having high reaction rates. In green chemistry, metal oxide nanoparticles are synthesized by using plant extracts, in which phytochemicals are used both as a reducing and capping agent. Calcium oxide (CaO), on the other hand, is a promising versatile and inexpensive catalyst with applications in various waste resources[3]. However, bulk calcium oxide exhibits restricted surface area and active site accessibility, which limits its catalytic activity. A recent research work reported biodiesel production using Jatropha oil with nano-CaO catalysts produced from waste snail shells with a maximum efficiency of 96.73% under optimum reaction conditions[4]. Basumatary *et al.*[5] demonstrated the use of CaO as a nano catalyst in biodiesel yield with higher surface area, basicity, and reactivity. Lakshmikanth *et al.*[6] reported a recent trend in the synthesis of nano-CaO using green technology involving plants due to their safety and economic benefits.

Waste cooking oil (WCO) resources have gained popularity in biodiesel yield owing to their economic and environmentally friendly characteristics in addition to reducing waste disposal costs and production costs[7]. Recent research was conducted to improve the yield of biodiesel from WCO using the base-catalysed transesterification method and zinc-doped calcium oxide as the catalyst. The optimal ratio of alcohol to oil was found to be 12:1 and the catalyst concentration was 5%. The biodiesel and its blends (B20–B100) showed similar properties to diesel fuel and produced less pollution. They also showed good performance in various engine operating conditions. Another study was conducted using green-synthesized iron oxide core-shell nanoparticles derived from *Camellia sinensis* leaves. The structure of the nanoparticles was analysed using XRD, FTIR, and SEM tests. The iron oxide core-shell nanoparticles were effective in the transesterification of algae oil to biodiesel using the optimal conditions and yielded 93.33% biodiesel. The nanoparticles showed good stability and could be reused up to five times[8]. Another research was conducted on the yield of biodiesel from WCO through a combination of methanol and ethanol as the alcohol source and a modified eggshell-derived calcium oxide nanocatalyst. The catalyst was treated using the hydration-dehydration method. This process was capable to attain a maximum yield of 94% under optimal conditions. A study also employed a CaO nanocatalyst synthesized by a thermal method for biodiesel synthesis from WCO. This study was able to attain a maximum biodiesel conversion of 96% under optimal conditions, such as 50°C, a 1:8 methanol-to-oil ratio, 1% catalyst, and 90 min reaction time. *Manilkara zapota* leaves contain antioxidants and polyphenolic compounds, making them a potential source for nanoparticles with improved catalytic activity. This is according to a study by Putson *et al*.[9] and Ayodhya *et al*.[10]. Previous studies have primarily investigated plant extracts such as *Polyalthia longifolia* and *Moringa oleifera* as well as waste-derived calcium sources, mainly eggshells. However, these studies focused on photocatalyst, antimicrobial and adsorption applications[11-13]. The *Manilkara zapota* leaves represent a relatively unexplored source for CaO nanoparticle synthesis. Since these leaves are rich in bioactive components, especially phenols, flavonoids, tannins, alkaloids, sterols and glycosides[14,15]. These phytochemicals can act as natural reducing, capping and stabilising agents during nanoparticle formation enabling the stable synthesis of CaO nanoparticles. Based on a literature survey, the use of *Manilkara zapota* leaf extract for the synthesis of CaO nanoparticles has not been previously reported. The present study introduces *Manilkara zapota* leaf extract as a novel phytochemical-rich biogenic synthesis for CaO nanoparticles. The synthesised nanoparticles will subsequently be utilised as an efficient heterogenous catalyst for biodiesel production from WCO.

Experimental Section

All the chemicals and solvents used in the current study are of analytical reagent grade and used without further purification. Methanol (CH_3OH) was obtained from Rankem Laboratory, ethanol (C_2H_6O) from Changshu Hongsheng Fine Chemicals, calcium nitrate [$Ca(NO_3)_2$] from NICE Chemicals (P) Ltd., Kochi, and hydrochloric acid (HCl) from Molychem Chemical (P) Ltd., Mumbai. Hexane (C_6H_{14}) and potassium iodide (KI) were supplied by NICE Chemicals (P) Ltd., Kochi. Iodine, sodium hydroxide (NaOH), nitric acid (HNO_3), and starch were purchased from Reachem Laboratory Chemicals (P) Ltd., Chennai. Glacial acetic acid (CH_3COOH), phenolphthalein, and chloroform were from Molychem Chemical (P) Ltd., Mumbai. Potassium hydroxide (KOH) was obtained from HiMedia Laboratories (P) Ltd., Mumbai, and sodium thiosulfate ($Na_2S_2O_3$) from Qualigens Fine Chemicals, Mumbai. The experiments were conducted using standard equipment. Heating mantles, hot air ovens, and reflux condensers were supplied by Guna Enterprises, Chennai. A centrifuge and a magnetic stirrer were supplied by Remi Equipments (P) Ltd. Flash and fire point analysis was conducted using a standard flash and fire point apparatus. Mass was determined using a precise weighing balance from Madurai Metric Works.

Synthesis of CaO Nanocatalyst

A green and environmentally friendly route was employed in the synthesis of the calcium oxide (CaO) nanocatalyst. The green extract was derived from *Manilkara zapota* leaves. The leaves were employed as reducing and capping agents. Fresh leaves of MZ, about 30 g, were washed using deionised water to clean the leaves. The leaves were then boiled using 100 mL of deionized water at 60°C for 10 min. The light brown brew was cooled and filtered to obtain

clear liquid. The liquid was employed as the reducing and capping agent. To synthesize the calcium oxide nanoparticles, 20 mL of the *Manilkara zapota* leaf extract was mixed and heated by magnetic stirrer to a constant temperature of 50-60°C. Once the temperature was constant, 20 mL of 1M calcium nitrate solution was slowly added while stirring. To maintain the basic condition and promote the synthesis of the nanoparticles, a few drops of 2M NaOH were slowly added while mixed until the colour of the solution transformed from brown to light yellow. The solution was centrifuged at 10,000 rpm for 10 min. The precipitate was collected and dried. The precipitate was then heated at 400 °C using a muffle furnace for 3 h. The resulting CaO nanoparticles were white and derived from the *Manilkara zapota* leaves.[16]

Transesterification of WCO

The WCO was collected from local restaurants and filtered to remove food residues. The free fatty acid (FFA) was measured as 1.5% and the corresponding acid value is 2.99 mg KOH/g calculation using the correlation reported in Cho *et al*.[17]. A 500 mL round-bottom flask that had three ports was the reactor for the transesterification reaction. The flask was placed in a water bath on the hot plate to keep the temperature constant. A water-cooled condenser was connected to the middle port of the flask to collect the methanol that boiled off. A thermometer was also placed in one of the side ports of the flask. The waste cooking oil was weighed and was 100 mL. The oil was heated to 50 °C before the reaction. The catalyst was weighed and added to the preheated methanol[18]. This was then added to the preheated oil. The biodiesel yield was computed using the given formula.

$$Biodiesel\ yield\ (\%) = \frac{Volume\ of\ Biodiesel}{Volume\ of\ waste\ cooking\ oil} \times 100$$

Reaction parameter optimization

The aim was to get the maximum biodiesel yield from the oil. This was achieved by controlling some very important parameters, like how much catalyst was added, how much methanol was added in comparison to the oil, temperature, and how long the reaction was. These parameters were initially kept constant at 8:1 methanol/oil ratio, 60 °C, and 120 min, respectively, based on previous experiments. To see how the amount of catalyst added was affecting the process, it was varied from 0.5% to 2.5% in 0.5% increments, while keeping all other parameters constant. The amount of catalyst added was dependent on how much oil was taken. All these experiments were performed at constant speed, 600 rpm, while the mixture was being stirred. To produce the most biodiesel from waste oil, transesterification was carefully optimized. This was achieved by carefully monitoring a number of crucial factors, including temperature, reaction duration, and the ratio of methanol to oil. Throughout all these experiments, the amount of catalyst was kept constant at the optimal amount. First, experiments were conducted to see how the methanol/oil ratio was affecting the process. Ratios from 6:1, 7:1, 8:1, 9:1, 10:1, 11:1, and 12:1 were all tested. At all times, temperature was kept at 60 °C, time was kept at 120 min, and constant speed was kept at 600 rpm. This was achieved by dissolving catalyst in methanol, after which oil was added. The goal was to determine how much oil was needed in relation to methanol in order to maximize the yield of biodiesel while avoiding excessive methanol waste, which could lead to separation issues[19].

Lastly, the effect of the reaction time was investigated by varying range from 60 to 180 min in increments of 20 min. The optimal time was determined based on yield and biodiesel separation. After the reaction was finished, the liquid was placed in a funnel and allowed overnight so that the biodiesel and the other components could clearly separate. This optimization approach was crucial in determining the most efficient and environmentally friendly method of turning used cooking oil and methanol into biodiesel[20].

Results and Discussion

UV-visible absorption spectroscopy analysis

UV-visible spectroscopy is a significant test that can be used to determine whether metal oxide nanoparticles were appropriately synthesized and to understand their response to light. The test was conducted within a range of 200-800 nm to observe the surface plasmon resonance and electron movement. The successful synthesis of CaO nanoparticles using *Manilkara zapota* plant leaf extract was demonstrated in this study by a peak at 330 nm. The peak results from the excitation of electrons within the nanostructure, which is influenced by the plant extract's active constituents. The findings were compared with CaO nanoparticles synthesized by utilizing *Moringa oleifera* plant leaf extract that demonstrated a peak at 323 nm illustrated in Fig. 1. The difference between the two findings

could be because of variations in size, crystal structure, or chemical composition of plant extracts[16]. The findings indicate that utilizing *Manilkara zapota* as a reducing agent is effective in synthesizing stable CaO nanoparticles that are sensitive to light.

FTIR analysis

FTIR analysis was used to determine the various chemical groups in the CaO nanoparticles obtained from leaf extract of *Manilkara zapota* and verify the formation of nanoparticles. Fig. 2 shows the FTIR spectrum. The wide band at 3395.63 cm^{-1} corresponds to the O-H stretching of hydroxyl groups. It may be due to the presence of water molecules adhering to the surface of the nanoparticles or remaining chemical compounds in the leaf extract. In green methods of synthesising nanoparticles, the presence of chemical compounds in plants like phenols, flavonoids, and alcohols aids in the development of hydroxyl groups on the surface of nanoparticles. The band at 2017.09 cm^{-1} may be due to secondary vibrations of the nanoparticles. It might be explained by the leaf extract's residual chemical constituents or reaction with atmospheric CO_2. The strong band at 1344.16 cm^{-1} corresponds to the CO_3^{2-} stretching of carbonate groups, as shown in Fig. 2. It may be due to the reaction of CO_2 in the atmosphere with the nanoparticles. It is a common property of CaO nanoparticles. Peaks at 871.45 cm^{-1} and 834.39 cm^{-1} correspond to the bending of C-O bonds in carbonate groups. The existence of calcium and oxygen bonding may potentially be responsible for these peaks. The stretching of calcium and oxygen bonds is shown by bands at 541.70 cm^{-1} and 469.70 cm^{-1}. These may be attributed to the calcium oxide nanoparticles.[16]

XRD analysis

The crystalline structure of CaO nanoparticles produced by biosynthesis were examined using X-ray diffraction (XRD). The diffraction pattern showed clear reflections at 2θ angles of 17.85°, 29.26°, 33.92°, 46.91°, 50.66°, 54.18° and 62.44°, proving that crystalline CaO nanoparticles had been formed in Fig. 3. The observed peaks are in good agreement with recent studies which report diffraction peaks for CaO nanoparticles confirming the successful formation of crystalline CaO nanoparticles. The small differences in the peak positions are due to lattice strain, the nanoscale effects of the crystallites, instrumental broadening, and the effect of the phytochemicals in the *Manilkara zapota* leaf extract during crystal growth. The crystallite size was determined by means of the Debye–Scherrer equation,

$$D = \frac{K\lambda}{\beta Cos\theta} \quad \ldots (1)$$

where K=0.94, λ=1.5406 Å, β (FWHM), and θ - Bragg angle. The calculated crystallite size varied from 21.76 to 27.55 nm, the average size being 24.45 nm, a value that is similar to the approximately 20 nm reported in Hemmami *et al.*[21]. The sharpness of the diffraction peaks together with the nanoscale dimensions of the crystallites show that the biosynthesised CaO nanoparticles are highly crystalline which use as catalysts in biodiesel production.

SEM analysis

The shape of CaO nanoparticles made from *Manilkara zapota* leaf extract was examined using scanning electron microscopy (SEM). From the SEM images at various magnifications, some important features of the catalyst are revealed. The SEM images indicate that CaO nanoparticles have an irregular shape and are found to be clumped together shown in Fig. 4. The clumping is a common phenomenon for CaO nanoparticles due to their high surface energy and interparticle interactions[19,20]. CaO nanoparticles

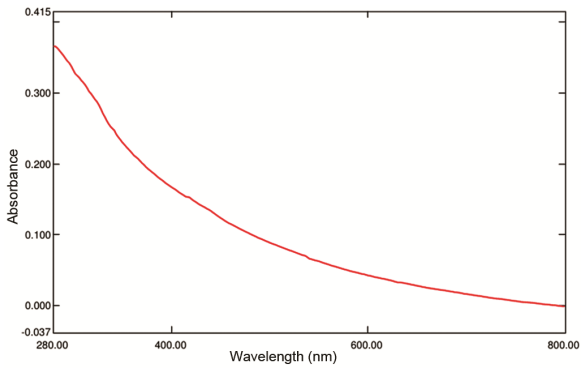

Fig. 1 — UV spectra of synthesised CaO nanoparticles

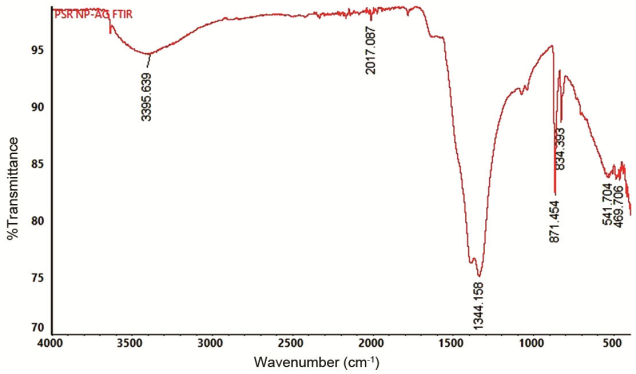

Fig. 2 — FTIR spectra of synthesised CaO nanoparticles

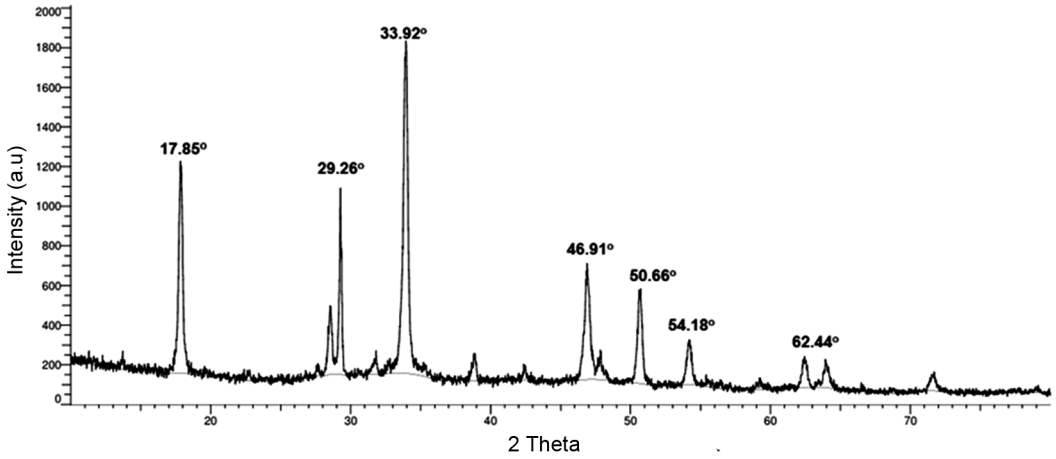

Fig. 3 — XRD plot of synthesised CaO nanoparticles

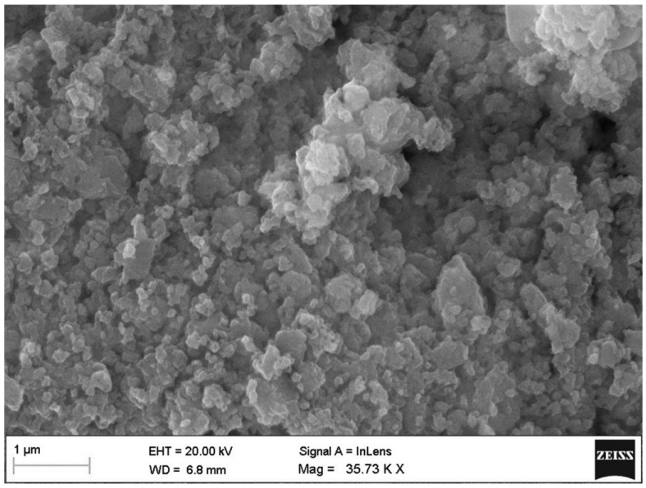

Fig. 4 — SEM image of synthesised CaO nanoparticles

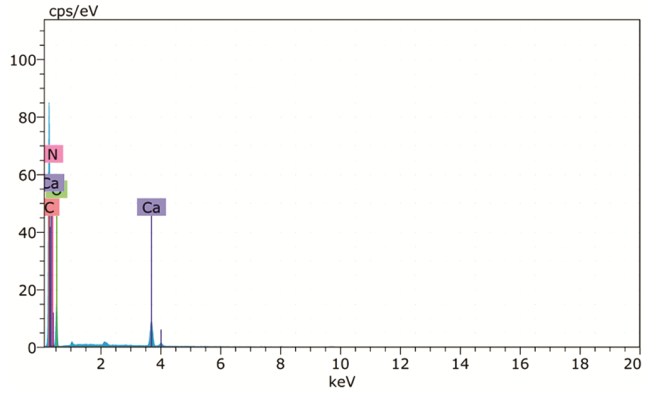

Fig. 5 — EDAX spectrum of CaO Nanoparticles

Table 1 — Elemental Composition of CaO Nanoparticles

Element	Weight (%)	Atomic (%)
Carbon	64.38	72.29
Oxygen	31.05	26.17
Calcium	4.57	1.54
Total	100.00	100.00

made from other plant extracts have previously been shown to have similar structures. The CaO nanoparticles had irregular shapes and helped as catalysts. The varying sizes of the particles may be due to various reasons like temperature/time of heating and the effect of plant chemicals during the growth of nanoparticles. The rough surface of CaO nanoparticles implies that the reaction can occur at many active sites. This is favourable for a catalyst as more reactant particles can stick to the catalyst's surface.

EDAX

The elemental composition of synthesized nanoparticles was studied using energy-dispersive X-ray spectroscopy (EDS) illustrated in Fig. 5 and Table 1. The spectrum exhibited characteristic emission peaks of Ca Lα (0.34 keV) and Ca Kα (3.69 keV) which confirmed the presence of calcium in the synthesized material. The oxygen signal also supports the formation of CaO. The quantitative EDS analysis showed the presence of C (64.38 wt.%), O (31.05 wt.%) and Ca (4.57 wt.%). The relatively high carbon content is attributed to residual phytochemicals adsorbed on the nanoparticle surface during green synthesis and conductive carbon tape used for the SEM specimen mounting[22]. EDS is a localized semi-quantitative microanalytical technique. The elemental composition measured is influenced by the interaction volume, specimen geometry, particle distribution and substrate contribution. The EDS results were mainly used to confirm the elemental presence of calcium and oxygen, while the crystalline phase of CaO was confirmed by X-ray diffraction analysis[16].

Table 2 — Comparative physiochemical analysis of WCO and Biodiesel

S. No	Physiochemical properties	Waste cooking oil (WCO)	Standard WCO[13,23]	Biodiesel (9:1,60°C,120mins, 1.5wt% catalyst conc)	Standard of biodiesel[15,25]
1.	Free fatty acid (%)	1.5	1.54	4.90	5.28
3.	Iodine value (gI2/100g)	90	84	80	90.6
4.	Saponification value	210	204.77	178	180
5.	Flash point (^{o}C)	300	309	120	150

Physiochemical characteristics

From the analysis of the properties of the biodiesel using the CaO nanocatalyst derived from *Manilkara zapota*, it was established that the properties of the biodiesel derived from the CaO nanocatalyst were like the properties of normal biodiesel. Compared to the biodiesel derived from waste cooking oil, the biodiesel derived from the CaO nanocatalyst has low iodine and saponification values. The flash point and fire point of the biodiesel derived from the CaO nanocatalyst were close to the values of normal biodiesel was mentioned in Table 2. Therefore, the CaO nanocatalyst is effective in the yield of biodiesel from waste cooking oil.[19,20]

Impact of nano catalyst concentration on biodiesel yield

In this study, the impact of catalyst concentration on biodiesel yield was studied at different concentrations of CaO nanocatalyst, ranging from 0.5% to 2.5% by weight, while keeping all other conditions constant, i.e., temperature at 60 °C, methanol/oil ratio at 8:1, and reaction time at 120 min. The obtained results were observed that an increase in catalyst concentration from 0.5% to 1.5% increased biodiesel yield from 71.99% to 93.97%. However, further increase in concentration decreased biodiesel yield, i.e., 85.11% at 2%, and 72.70% at 2.5% was shown in Fig. 6. The biodiesel yield was significantly affected by catalyst concentration. These results are in accordance with those in previous studies, where it was observed that moderate amounts of catalyst are required in transesterification, which enhances the efficiency of the reaction. Excessive amounts of catalyst result in soap formation due to saponification, thereby completely halting biodiesel production. It was also reported in Degfie *et al*.[19] 1% catalyst concentration produced the highest yield, i.e., 96%, under mild conditions, i.e., 50°C, and 90 minutes. In our study, it was observed that 1.5% concentration produced the highest yield. This shows that conditions play an important role in catalyst usage. Hence, 1.5% concentration was observed to be the optimal concentration in our study. Thus, it can be inferred that catalyst concentration affects biodiesel yield and that, because of its affordability and environmental friendliness, nano-CaO may be successfully used in biodiesel production.

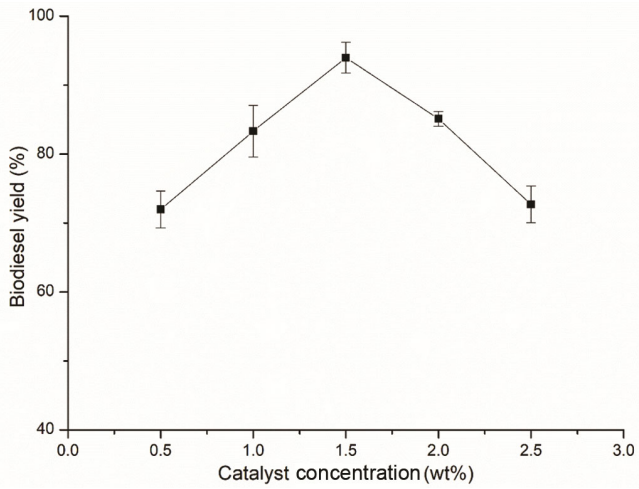

Fig. 6 — Effect of catalyst concentration on biodiesel yield

Impact of methanol to oil ratio on biodiesel yield

By changing the methanol-to-oil molar (MO) ratio from 6:1 to 12:1, the impact of this ratio was investigated. The catalyst concentration was maintained at 1.5%, the reaction time was set at 120 min, and the temperature of the biodiesel manufacturing process was maintained at 60 °C. The biodiesel yields increased from 65.96% with a MO molar ratio of 6:1 up to a maximum of 96.10% with a MO molar ratio of 9:1. However, biodiesel yields decreased as the MO molar ratio increased from 9:1 up to a MO molar ratio of 12:1, with a biodiesel yield of 74.47% was illustrated in Fig. 7. This further confirms that an increase in methanol content will facilitate the forward reaction by forming methoxy groups on the catalyst's surface. However, once the optimal ratio is exceeded, the increased solubility of the glycerol will make it harder to be separated from biodiesel, thus shifting the reaction towards the reverse direction and causing a decline in biodiesel yields. These findings are comparable with those of Degfie *et al*.[19], who discovered that a molar ratio of 8:1 resulted in the highest biodiesel yields of 96% under mild operating conditions of 50 °C, 90 min, and 1% catalyst concentration. Erchamo *et al*.[14] found that a molar ratio of 12:1 produced a biodiesel yield of

89% with a temperature of 60°C for 180 minutes, and a concentration of 2.5%. Therefore, in our study, it was found that a molar ratio of 9:1 produced the highest biodiesel yields. However, it is significant to vary the methanol content to attain the highest biodiesel yields

Impact of temperature on biodiesel yield

The effect of temperature on the yield of biodiesel was investigated by varying the temperature from 40 to 80 °C while maintaining the concentration of the catalyst at 1.5%, the molar ratio of methanol to oil at 9:1, and the reaction time at 120 min. As indicated in Fig. 8, the biodiesel yield rose to a maximum of 96.10% at 60 °C from 79–80% at 40 °C. Therefore, the temperature of 60 °C is the most appropriate. The yield of biodiesel increases as the temperature increases because the reaction occurs more quickly and the oil becomes less thick. Moreover, methanol and oil mix well. This improves the reaction because it reduces the resistance of the three-phase system. Similar findings were obtained in other research on the production of biodiesel. Raising the temperature promotes the oil's transesterification because it supplies the energy required to enhance the reactants' diffusion. However, the yield of biodiesel decreased beyond 60 °C. The yield was 90% at 70°C and 86-87% at 80°C. This could be because methanol has a boiling point of around 64.7°C. Therefore, the temperature of 60 °C is the most appropriate. Additionally, the use of higher temperatures could cause the production of soaps because of the basic nature of the CaO nanocatalysts[19]. Therefore, maintaining the temperature at 60°C is the most appropriate because it gives the best yield of biodiesel and the best performance of the CaO nanocatalysts produced from the *Manilkara zapota* leaf extract.

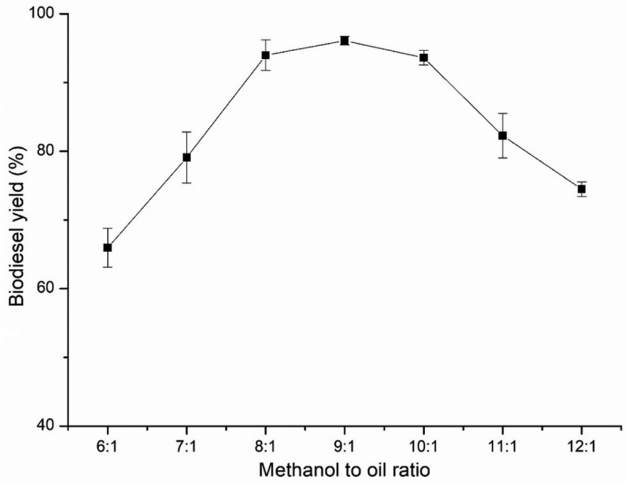

Fig. 7 — Effect of Methanol to Oil ratio on biodiesel yield

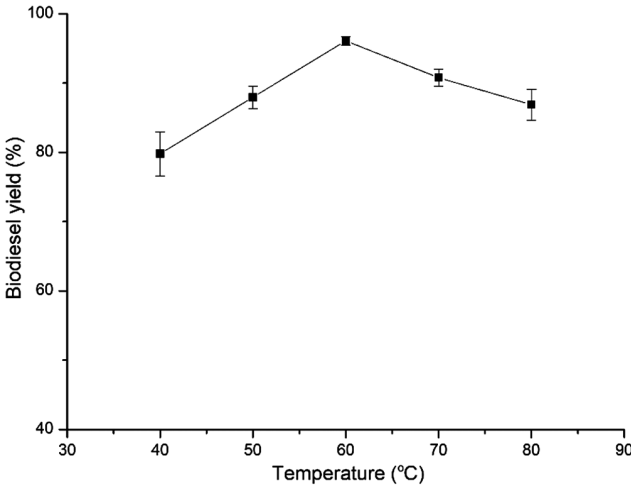

Fig. 8 — Effect of temperature on biodiesel yield

Impact of reaction time on biodiesel yield

The impact of reaction time was investigated by altering the reaction time from 60 to 180 min. The temperature, methanol-to-biodiesel molar ratio, and catalyst concentration were maintained at their ideal values of 60 °C, 1.5 weight percent, and 9:1, respectively. As shown in Fig. 9, the biodiesel yield raised with the increase of the reaction time from 60 min up to a maximum of 96.10% after 120 min, indicating that the optimal time for the biodiesel production is 120 min was elucidated in Fig. 9. The initial increase in biodiesel yield is because of the transesterification reaction, where triglycerides are converted into fatty acid methyl esters with the help of a CaO nanocatalyst, which was synthesized from *Manilkara zapota* leaf extract. The increase in the

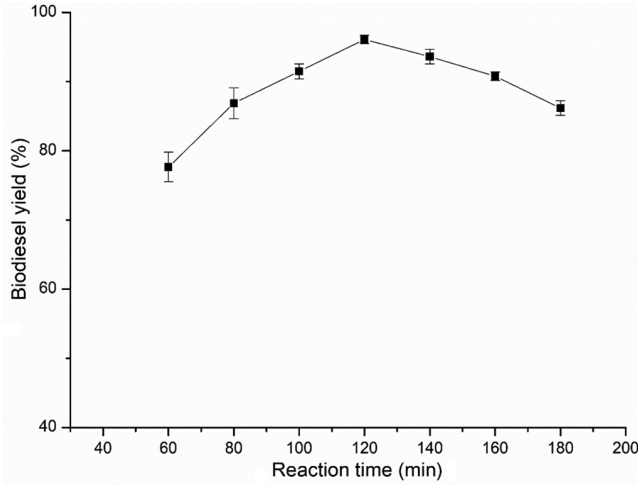

Fig. 9 — Effect of reaction time on biodiesel yield

biodiesel yield with the increase of the reaction time up to 120 minutes may be due to the longer duration, which gives enough time for methanol and biodiesel to mix properly, for better movement of the reactants and products, and for better interaction of methanol with biodiesel using a CaO nanocatalyst. This agrees with the results of earlier researchers, where biodiesel yield was found to increase with the increase of the reaction time up to a certain point where equilibrium is reached, i.e., the optimal time for the reaction. After that, the biodiesel yield decreases, which may be due to the reverse reaction, where the produced biodiesel is converted into smaller parts, and soap is formed due to the basic nature of the catalyst[19,20]. Thus, the optimal time to produce biodiesel is 120 min, where the highest biodiesel yield of 96.10% is obtained, which agrees with the results of earlier researchers for transesterification and proves that CaO nanoparticles, which were synthesized from *Manilkara zapota* leaf extract, are better catalysts for biodiesel production.

Gas Chromatography – Mass Spectroscopy

The Gas Chromatography – Mass Spectroscopy (GC-MS) analysis was used to determine the chemical constituents in WCO and biodiesel made with calcium oxide (CaO) nanocatalyst derived from *Manilkara zapota* leaves. The GC chromatogram of the WCO indicates the presence of fatty acid derivatives and organic compounds. Major compounds were identified as glycerin, hexadecanoic acid methyl ester, 9,12-octadecadienoic acid methyl ester, 11-octadecenoic acid methyl ester, and oleic acid derivatives was shown in Fig. 10 and Table 3. These

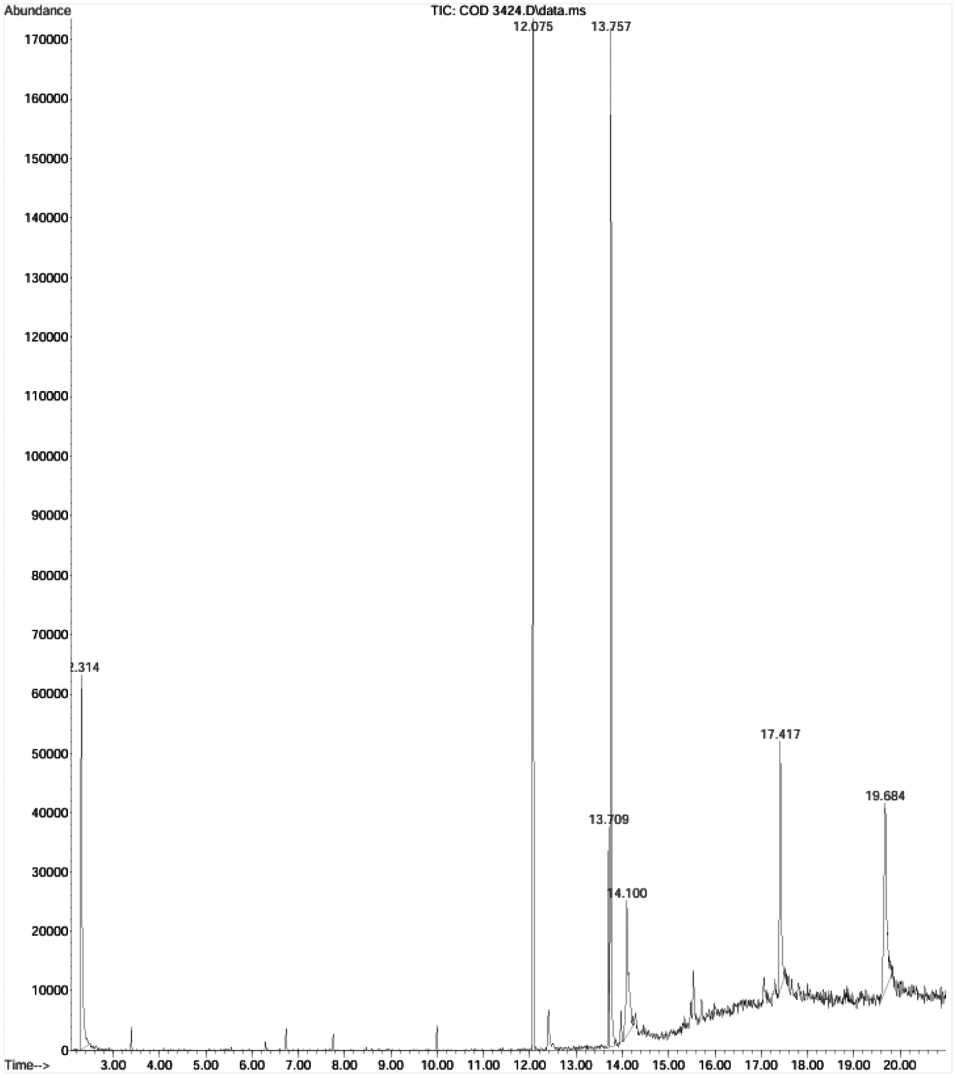

Fig. 10 — GC-MS of caste cooking oil

Table 3 — GC-MS analysis of waste cooking oil

	Waste Cooking Oil		Biodiesel	
Retention time	Name of compound	Area%	Name of compound	Area%
2.314	Glycerine	13.5	Methanamine	7.38
12.072	Hexodecanoic acid, Methyl ester	24.24	Hexodecanoic acid, Methyl ester	29.86
13.708	9,12- Ocadecodienoic acid Methyl ester	5.21	9,12- Ocadecodienoic acid Methyl ester	6.56
13.755	11-Octadecenoic acid Methyl ester	25.56	11-Octadecenoic acid Methyl ester	34.53
14.105	Oleic acid	8.36	10-Octadecenoic acid, methyl ester	9.02

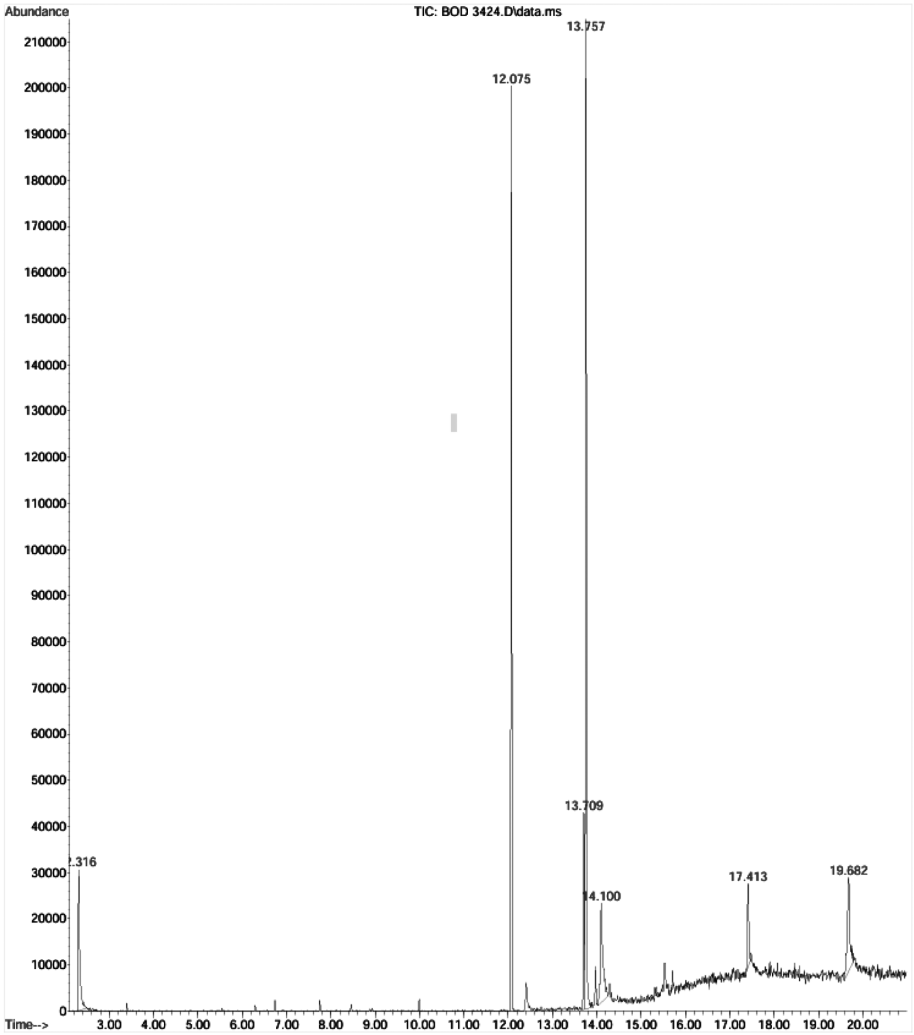

Fig. 11 — GC-MS of biodiesel

compounds show that fatty acids are present and their derivatives, which are long-chain fatty acids and lipid derivatives of the molecules of the feed oil. The compounds, such as glycerin, present in the GC chromatogram indicate the presence of glycerol and its derivatives, which are usually present in the molecules of the waste cooking oil. These compounds are usually formed during the repeated heating of the oil during cooking. The produced biodiesel was examined by GC-MS following transesterification with methanol in the presence of the CaO nanocatalyst. Significant peaks on the chromatogram were mostly associated with substances that have methyl ester functional groups. The major compounds identified in the biodiesel sample are hexadecanoic acid methyl ester, 9,12-octadecadienoic acid methyl ester, and 11-octadecenoic acid methyl ester was illustrated in Fig. 11 and Table 3. These compounds are composed of fatty acid methyl esters, which is formed during the methanolysis of triglycerides. The

presence and increased amount of methyl ester compounds in the biodiesel GC-MS chromatogram confirm the effective conversion of waste cooking oil into biodiesel. By comparing the GC-MS chromatogram of the waste cooking oil and biodiesel sample, it is evident that there is an increase in methyl ester compounds and a decrease in glycerol compounds[26].. This confirms the higher catalytic activity of the CaO nanocatalyst in this conversion process. The GC-MS data confirm that the CaO nanocatalyst synthesized from *Manilkara zapota* leaves is active in transforming waste cooking oil into biodiesel containing methyl ester compounds.

Conclusion

Green synthesis of *Manilkara zapota* leaf extract successfully produced CaO nanoparticles. The structural and morphological characteristics of synthesised CaO nanoparticles were confirmed by FTIR, XRD, and SEM-EDAX. The synthesised CaO nanoparticles showed potential catalytic performance for the transesterification of waste cooking oil. The process optimisation identified 1.5 wt% catalyst loading, 9:1 methanol-to-oil ratio, 60°C temperature and a time of 120 min. The maximum biodiesel yield of 96.10% was achieved. Furthermore, GC-MS analysis confirmed the potential conversion of triglycerides into fatty acid methyl esters, while the physicochemical properties of the produced biodiesel support the effectiveness of the transesterification process. It is suggested that the superior catalytic activity of green-synthesis CaO makes it a feasible material in the sustainable process of converting waste cooking oil into biodiesel.

Conflict of Interest

The authors declare that there are no conflicts of interest associated with this work.

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