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Synthesis of CaO Nanocatalyst and Its Application in Methyl ester (Biodiesel) Production from Edible and Non-Edible Oils via Transesterification

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Abstract

The sol-gel approach was used to synthesize the nanocatalyst, which was then characterized using XRD and SEM. An investigation was conducted into the optimization of transesterification parameters for the manufacture of methyl ester using edible linseed oil and non-edible neem oil. Within 180 minutes of initiating an in-situ transesterification process with linseed oil at temperature of 65 ^{0}C and a 1:10 oil-to-methanol ratio, a high yield of fatty acid methyl ester 90.0 wt%, whereas the yield of neem oil 94.0 wt% was achieved at optimized conditions. The feedstock neem oil had the greatest FAME conversion compared to linseed oil. It was found that the CaO nanocatalyst could be reused five times without a significant decrease in activity, according to the catalyst recyclability test. Nuclear magnetic resonance (NMR), Fourier transform infrared spectroscopy (FTIR), and gas chromatography–mass spectroscopy (GC–MS) were employed to characterize the feedstock and methyl ester.

Keywords: Methyl ester; Nanocatalyst; Transesterification; Linseed oil; Neem oil.

1. Introduction

The increasing global demand for energy, coupled with the depletion of fossil fuel reserves and escalating environmental concerns, has intensified the search for sustainable and renewable energy alternatives. Greenhouse gas emissions from traditional fuels derived from petroleum are a key cause of climate change and global warming [1]. Because of its renewable source, biodegradability, lack of toxicity, and lower emission profile than traditional diesel, biodiesel has gained attention as a potential alternative fuel in this regard [2]. By transesterifying triglycerides found in oils and fats with short-chain alcohols like methanol, the main component of biodiesel is fatty acid methyl esters (FAME) [3].

The sustainability and economic viability of biodiesel production are greatly affected by the feedstock choice. Linseed oil and other edible oils have a high conversion efficiency and a favourable fatty acid content, making them very useful [4]. Edible oils have many potential benefits, but they also increase production costs and bring up questions about the relative merits of food and fuel [5]. A lot of people are looking to non-edible oils, like neem oil, and inexpensive feedstocks, such recycled cooking oil, to solve these problems. Not only can these options save production costs, but they also solve problems with waste management and pollution [6,7]. Using a

variety of feedstocks, such as edible, non-edible, and waste oils, offers a holistic approach to producing biodiesel in a sustainable manner [8–12].

For biodiesel, the most common procedure is transesterification because it is easy to implement and produces good results. Glycerol and methyl esters are the byproducts of this process, which entails reacting triglycerides with alcohol while a catalyst is present [13–14]. Although sodium hydroxide (NaOH) and potassium hydroxide (KOH) and other homogeneous catalysts have a lot of catalytic activity, they have a lot of drawbacks, such as making soap and being bad for the environment [15]. Because of these drawbacks, heterogeneous catalysts, especially metal oxides like calcium oxide (CaO), have gained popularity. These catalysts have several benefits, including low environmental impact, facile separation, and reusability.

The effectiveness of heterogeneous catalysts has been significantly improved by recent advances in nanotechnology. In comparison to bulk catalysts, nanostructured CaO catalysts have a larger surface area, more active sites, and better catalytic performance [16]. Nanocatalysts are typically prepared using the sol-gel method, which is one of several synthesis strategies. This is because it yields particles that are homogeneous in size, very pure, and have improved surface properties [17]. There is promising evidence that CaO nanocatalysts produced using the sol-gel method can enhance Methyl ester production and reaction conditions.

It is essential to characterize the catalysts and Methyl ester in order to guarantee the quality and effectiveness of the process. The morphology and crystalline structure of the synthesized nanocatalyst are studied using scanning electron microscopy (SEM) and X-ray diffraction (XRD) [18]. To further verify the synthesis of fatty acid methyl esters and to identify functional groups in biodiesel, analytical procedures such Gas Chromatography-Mass Spectroscopy (GC-MS), Fourier Transform Infrared Spectroscopy (FTIR), and Nuclear Magnetic Resonance (NMR) are employed [19]. The chemical make-up and structural characteristics of the fuel that is produced can be fully understood with the use of these methods.

Factors crucial to the mass manufacturing of methyl ester include, but are not limited to, production, characterisation, the reusability of the catalyst, and the economic feasibility of the process. Reusing and regenerating CaO nanocatalysts can increase process sustainability and cut operational costs in half [21]. The current research intends to use the sol-gel method to create and analyze a CaO nanocatalyst, transesterify linseed and neem oils to make methyl ester, analyze FAME by GC-MS, FTIR, and NMR, and evaluate catalyst regeneration.

2. Materials and Methods

2.1 Feedstocks

In order to carry out the tests for the production of methyl ester (biodiesel), linseed and neem oil were bought from the local market. The compounds utilized were all of analytical grade. Researchers used ASTM criteria to analyze The physicochemical characteristics of feedstocks were analyzed by ASTM standards.

2.2. Synthesis of nanocatalyst using Sol-gel method and its characterization

Calcium oxide (CaO) nanocatalyst was synthesized via the sol–gel method by dissolving calcium nitrate tetrahydrate in ethylene glycol to form a sol, which was gelled, filtered, dried, and ground. The obtained powder

was calcined at 850 °C for one hour and stored in a desiccator for further use [22]. In this study, XRD and SEM analysis used for the identification and specification of Nano catalyst.

2.3 Production of Methyl ester from purified feedstock via transesterification and its optimization

Pretreatment of the feedstock included drying it out and neutralising free fatty acids (FFA) to stop soap from forming. After being heated to 110 ± 5 °C, the linseed and neem oils were filtered. Titration with 0.1 N KOH and phenolphthalein was used to determine the FFA content, which was then neutralized as needed. A reflux condenser, thermometer, and magnetic stirrer were used in conjunction with a 500 ml two-necked flask to conduct the transesterification. Reactions with pretreated oil were carried out using a calcium oxide nanocatalyst/methanol mixture under carefully regulated conditions, including temperature (50-60 °C), duration, and molar ratio. After letting the reaction mixture settle, layers of glycerol, catalyst, and methyl ester formed. After separating the methyl ester, it washed with acidified water using the air bubble method and then dried at 110 °C. Optimization of parameters such as temperature, reaction time, methanol ratio, and catalyst loading was performed to enhance Methyl ester production efficiency. The equation shown below was used to compute the yield of Methyl Ester.

$$Methyl\ Ester\ Yield(\%) = \frac{Volume\ of\ Methyl\ Ester}{Volume\ of\ Feedstock}\ \text{X}\ 100 \qquad \text{---(1)}$$

2.4 Characterization of Methyl ester and Regeneration of catalyst

The crystal structure of the resulting powder samples was analyzed by X-ray diffraction (XRD, Model BRUKER AXS D8, Cu Ka radiation, λ = 0.154056 nm). The vibrational frequencies of the samples were examined using a Perkin Elmer (model series 2000) FTIR spectrophotometer with a resolution of 1 cm^{-1} in the region of 400–4000 cm^{-1}. The transformation of neem oil into methyl ester was verified using gas chromatography in conjunction with mass spectrometry (GC-MS). Gas chromatography-mass spectrometry was carried out in Agilent 7890a gas chromatograph equipped with a 30 m HP-5 MS column of internal diameter 0.25 mm and 0.15 µm film thickness. Sample was introduced in split injection with a split ratio of 50:1. The oven temperature was started at 25 °C and then ramped at 50 °C per min to 230 °C withhold time of 3 and 5 min, respectively. Helium gas was introduced at the rate of 1 µl/min in a constant mode. The NIST library 11 was used to identify and validate the existence of fatty acid methyl ester peaks in the biodiesel GC-MS spectra. ^{H}NMR spectroscopy was utilized to determine the conversion of linseed and neem oil to FAMEs. The product was evaluated using a Bruker NMR 400 MHz spectrometer with deuterated chloroform ($CDCl_3$) as a solvent, in which case 10 mg of biodiesel product was dissolved in 1 mL of deuterated chloroform with 0.05% tetra-methyl silane. Heterogeneous CaO catalyst was recovered by centrifugation, washed with ethanol, vacuum-dried, and reused in transesterification, demonstrating effective regeneration and comparable performance to previous studies.

3. Results and discussion

3.1 Characterization of Synthesized CaO Nano catalyst

3.1.1 XRD Analysis:

The synthesized catalyst principal components were determined by X-ray diffraction (XRD), which also served to measure the crystallite sizes. There was clear evidence of the synthesized catalyst's value in the 16-71° range, as well as the CaO nanoparticles XRD diffraction intensity (pattern) and the powder's considerable crystalline. Near the peak positions of 16.024,28.639, 29.361, 34.069, 47.088, 50.743, 54.592, 62.719, 64.229 there distinct peaks as shown in the **Figure-1**. These results were qualitatively confirmed as similar trends were observed in other studies conducted by Davoodbasha et al., 2021 [22].

$$D=k\lambda/(\beta*\cos\theta)$$

Where D = Diameter of particles(nm)

k = Scherrer constant

λ = XRD radiation of wavelength in Nano meters

β = Full width at half maximum in radians

2θ = Peak position

Debye-Scherer equation, was used to compute the CaO nanoparticle crystallite size diameter (D), which is measured in nanometers (nm).The synthesized CaO has a mean crystal size of 39.17 nm and a particle size range of 13 to 77 nm as shown in the **Table 1.**

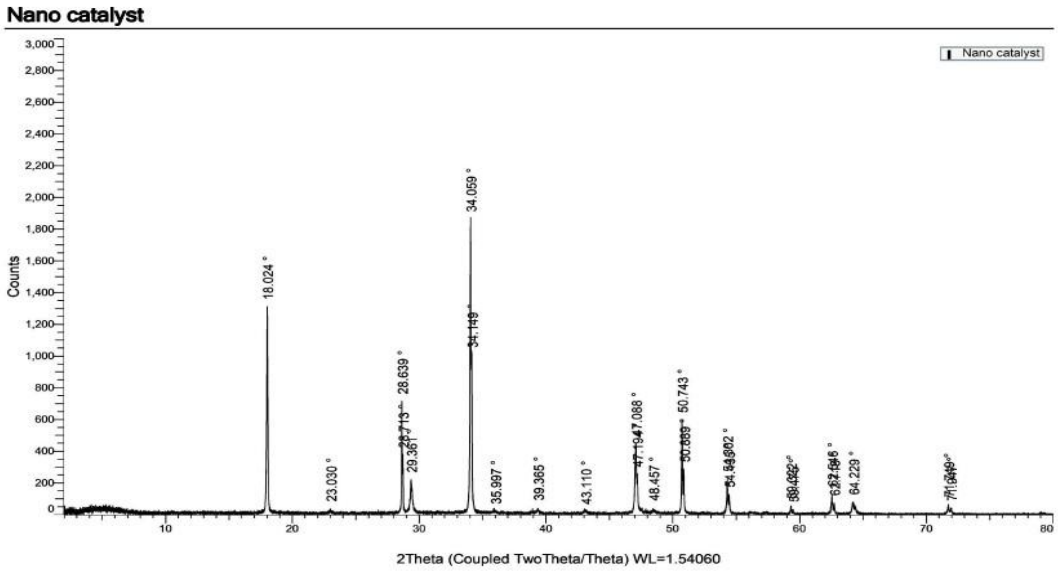

Figure 1. XRD pattern of synthesized CaO nanoparticle.

Table 1: Diameter of particles using XRD report

Peak position (2θ)	Scherrer constant (κ)	Full width at half maximum(β) in radians	XRD radiation of wavelength(λ) in Nano meters	Diameter of particles(nm)
16.024	0.98	0.2796	1.54	56.77
28.639	0.98	0.4998	1.54	32.31
29.361	0.98	0.5124	1.54	63.27
34.069	0.98	0.5946	1.54	28.74
47.088	0.98	0.8218	1.54	18.35
50.743	0.98	0.8856	1.54	19.18
54.592	0.98	0.9528	1.54	42.08
62.719	0.98	1.0946	1.54	13.87
64.229	0.98	1.1210	1.54	77.92

3.1.2 SEM analysis:

A scanning electron microscopy (SEM) investigation was conducted with sample image scales of 5µm, 10µm, 20µm, and 100µm, and magnifications of 500X, 2.5KX, 5KX, and 8KX, respectively. The SEM pictures shown in the **Figure-2(a-d)** that the produced CaO nanocatalyst usually has irregularly shaped particles with a porous structure and active sites. The catalyst's larger surface area for reaction is another possible explanation, given the wide range of particle sizes and shapes. Using the scanning electron microscope examined the resulting CaO Nano catalyst for structural defects. Therefore mentioned results were qualitatively validated, since comparable patterns were noted in the study by Abd Malek et al., 2021[23].

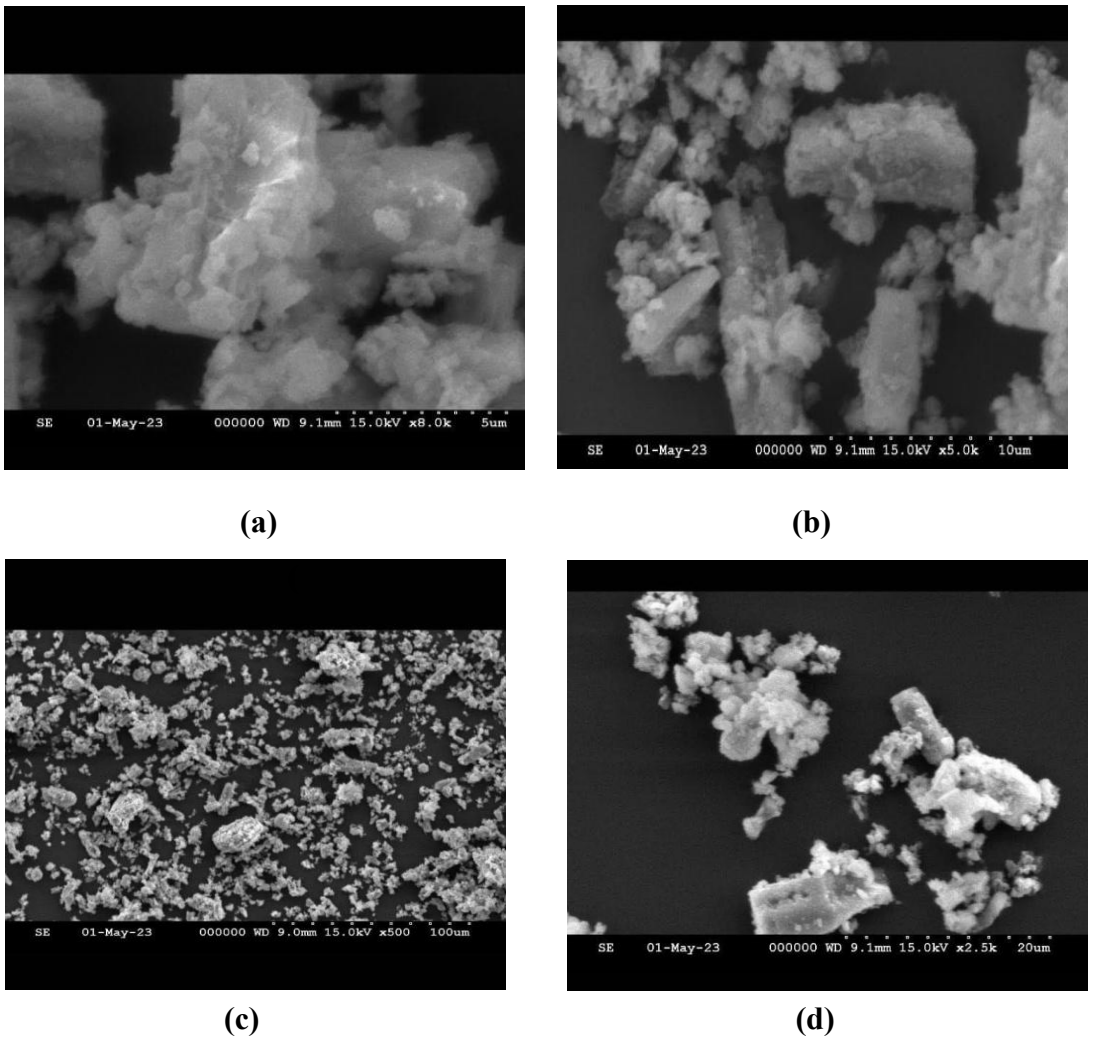

Figure-2 (a-d) shows the SEM images of synthesized nanocatalyst at 5µm, 10µm, 20µm, and 100µm magnification, respectively.

3.2 Production of methyl ester (biodiesel) from Linseed oil at optimized parameters

3.2.1 Effect of transesterification reaction variables:

Parameters' effects on methyl ester synthesis were investigated, as demonstrated in Figure - 3 (a–d). Factors such as catalyst loading, reaction time, oil-to-methanol molar ratio, and temperature have a substantial impact on transesterification [24]. Increasing temperature from 45°C to 65°C improved methyl ester yield from 75.5% to 86.0% due to enhanced reaction rates; however, further increase reduced yield because of methanol evaporation and possible thermal degradation. Similarly, reaction time played a crucial role, with yield (88.0%) increasing up to 180 minutes, beyond which no improvement was observed due to reverse reactions and soap formation. Catalyst loading also affected conversion, as increasing CaO nanocatalyst from 1.0 g to 1.5 g enhanced yield (88.5 %) by providing more active sites, while excess catalyst (2.0 g) led to soap formation and reduced yield. The oil-to-methanol molar ratio showed optimal performance at 1:10, where maximum yield (90.0%) was achieved. Further increase to 1:12 decreased yield due to excess methanol promoting glycerol formation and shifting equilibrium backward. Thus, optimal conditions were identified to maximize methyl ester production efficiency. Similar reports were also seen in the study conducted by Gashaw and Lakachew, 2014 [25].

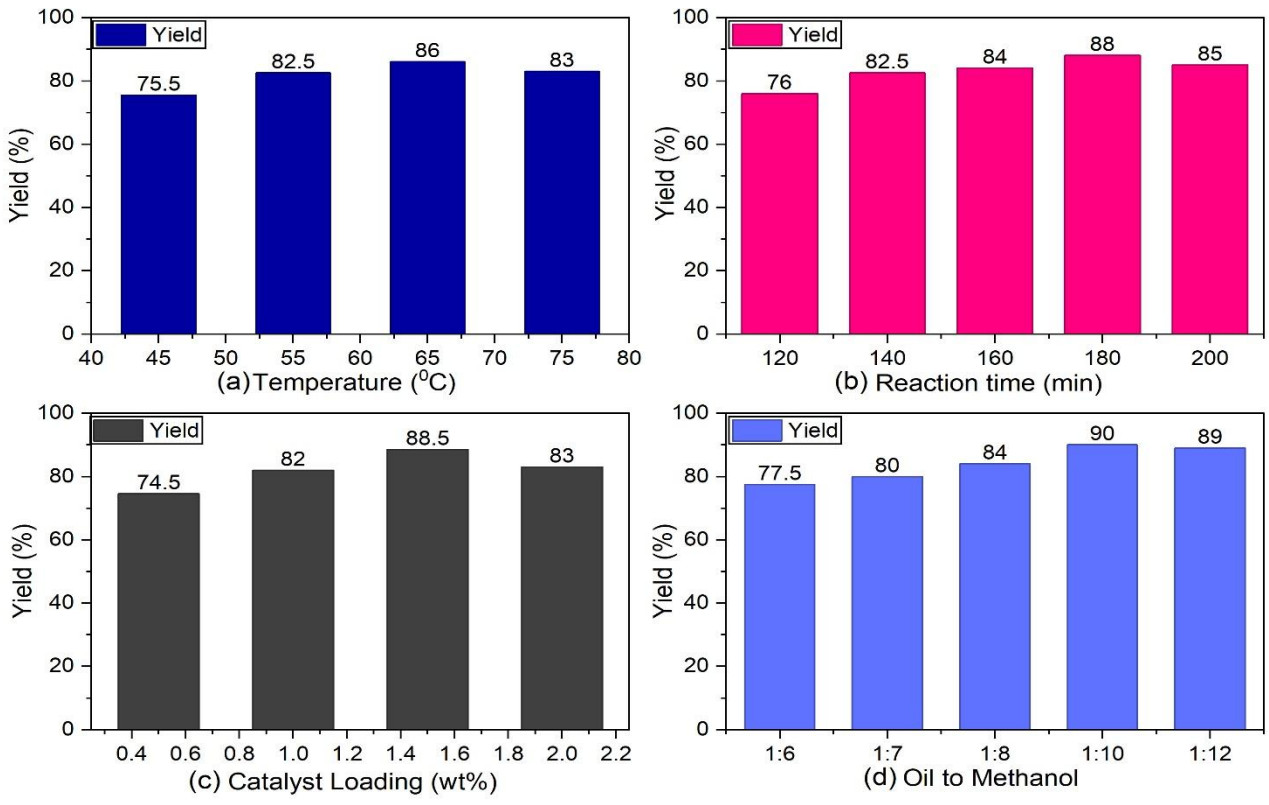

Figure-3 (a-d): Effect of Temperature, reaction time, catalyst loading & Oil to methanol ratio

3.3. Characterization of LOME

3.3.1. FTIR studies of Linseed methyl ester (LOME):

FTIR analysis of LOME confirmed the presence of key functional groups indicative of methyl ester formation. A peak at 3010.6 cm^{-1} corresponds to O–H and C–H stretching, associated with carboxylic acids, alkenes, and alcohols. Peaks at 2924.9 cm^{-1} and 2853.5 cm^{-1} indicate O–H, N–H, and C–H bonds, confirming the presence of alkanes, alcohols, and amine-related groups. A strong absorption at 1741.9 cm^{-1} represents C=O stretching, characteristic of ester functional groups, confirming successful transesterification. The peak at 1437.9 cm^{-1} corresponds to C–H and O–H bending, suggesting alkanes and carboxylic acids. Further peaks at 1168.7 cm^{-1} indicate C–O, C–N, and related bonds, supporting the presence of esters and alcohols (**Figure-4**). The absorption at 717.9 cm^{-1} is attributed to C–Cl and C=C bonds, indicating alkenes and minor halo compounds. Overall, the FTIR spectrum confirms the formation of methyl esters with characteristic functional groups, validating the successful conversion of linseed oil into methyl ester.

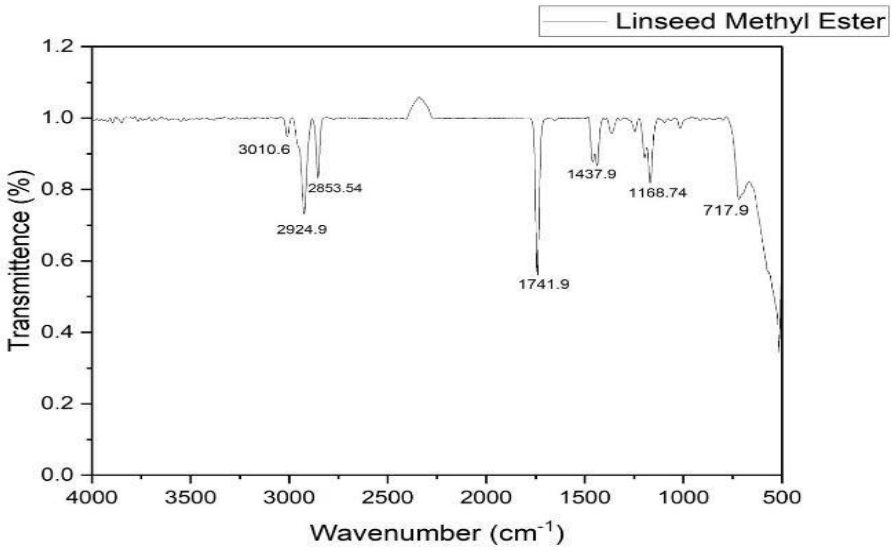

Figure-4 FTIR analysis of LOME

3.3.2. Nuclear Magnetic Resonance (H-NMR) studies of LOME:

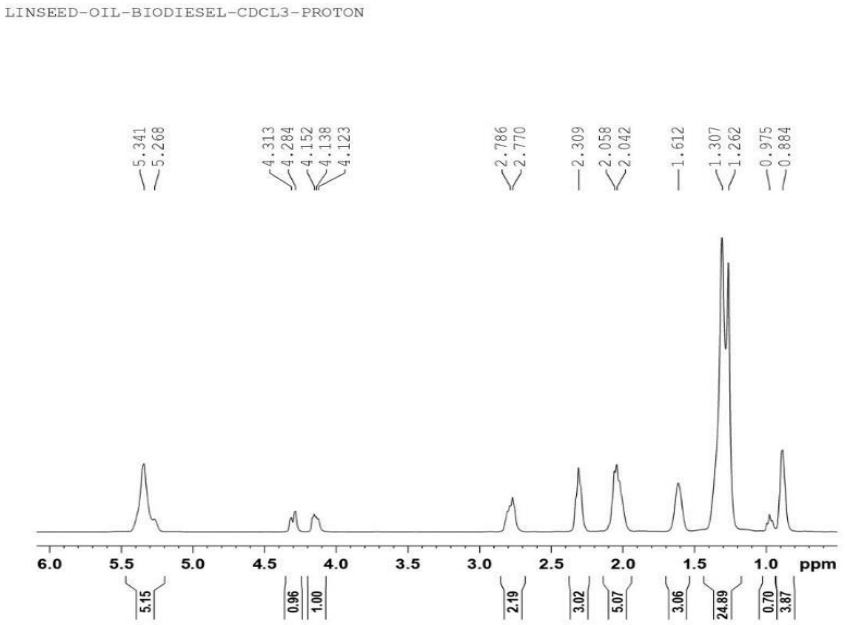

Figure -5 H-NMR studies of LOME

The results as shown in the **Figure-5.** ^{1}H NMR analysis of LOME in $CDCl_3$ confirmed the formation of fatty acid methyl esters (FAME), the main component of methyl ester. Peaks at 5.268–5.341 ppm correspond to alkenyl protons of unsaturated fatty acids, indicating high unsaturation. Signals at 4.113–4.313 ppm represent methylene protons adjacent to the ester group, confirming conversion of triglycerides into methyl esters. Peaks at 2.770–2.786 ppm are due to methylene protons near the carbonyl group, while 2.042–2.309 ppm indicate allylic protons. The methylene protons along the aliphatic chain appear at 1.612 ppm and 1.262–1.397 ppm. Terminal methyl groups are observed at 0.884–0.975 ppm. Overall, the spectrum confirms successful biodiesel production with characteristic ester functional groups and unsaturated fatty acid chains.

3.4 Production of Neem oil methyl ester (NOME) at optimized parameters

As shown in **Figure-6 (a-d),** the influence of parameters like effect of temperature, reaction time, catalyst loading, and oil–methanol ratio on methyl ester yield was investigated. Increasing temperature from 40°C to 50°C improved yield from 74% to 85.5%, with 50°C identified as optimum; further increase reduced yield due to methanol vaporization. Reaction time optimization showed maximum conversion (87.0%) at 120 minutes, beyond which yield decreased due to reverse reactions with glycerol. Catalyst loading enhanced conversion by providing more active sites, with optimum yield (89.5 %) observed at 1.0 wt% for neem oil. The oil-to-methanol molar ratio significantly influenced yield and increased to 94.0%, with an optimum ratio of 1:20. Higher methanol content initially increased conversion by driving the reaction forward; however, excessive methanol reduced yield by diluting reactants and limiting effective contact between triglycerides and catalyst. These results confirm that optimal conditions are essential to maximize biodiesel production efficiency [26].

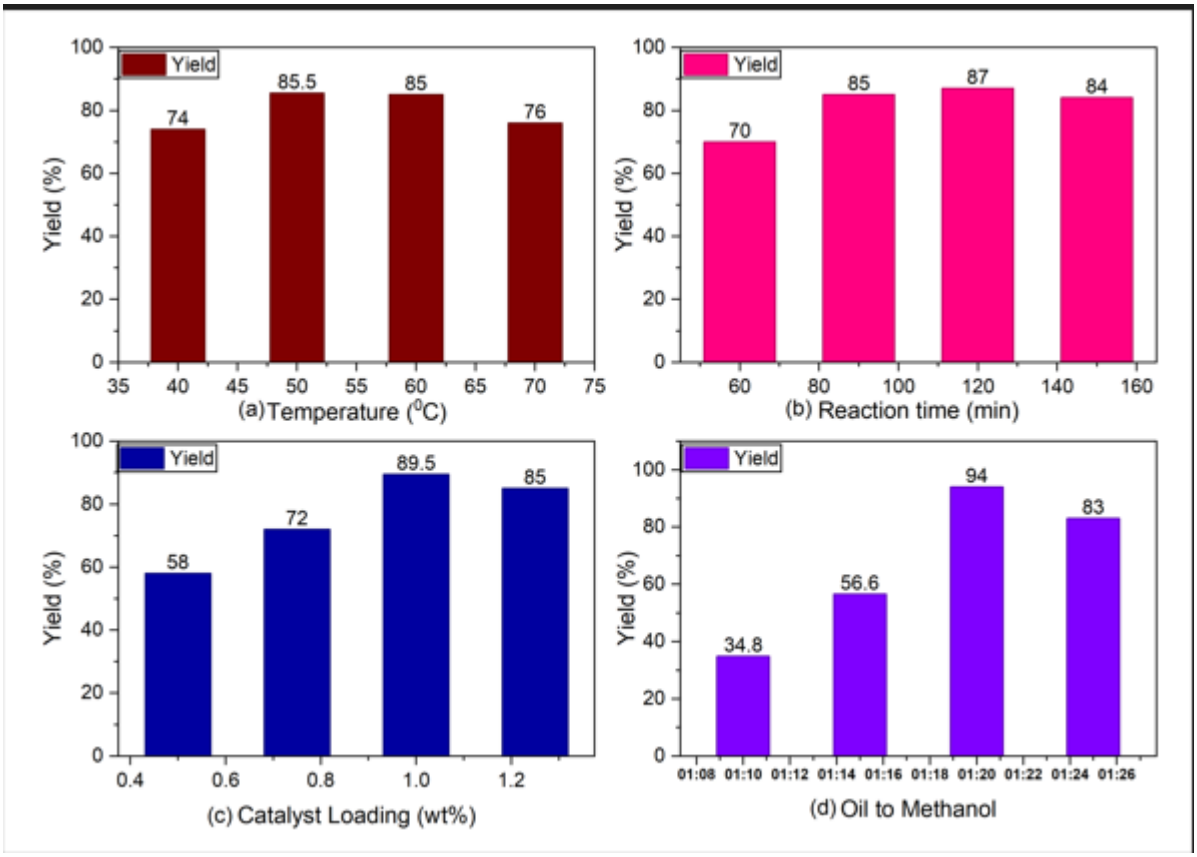

Figure 6 (a-d): Effect of Temperature, reaction time, catalyst loading & Oil to methanol ratio

3.5 Characteristics of NOME

3.5.1 NOME characterization by FTIR*:*

The FT-IR spectra of neem oil was shown in **Figure-7**. The bands between 2924 cm^{-1} and 2831 cm^{-1} are the strong peaks of the carboxylic acid's OH stretching. Seen at 1741 cm^{-1} was the distinctive peak that corresponds to the ester C=O stretching. The C-H bending in the alkane methylene group is corresponding to the area between 1460 cm^{-1} and 1452 cm^{-1}. All FTIR peaks are consistent with values in the previous reports [27].

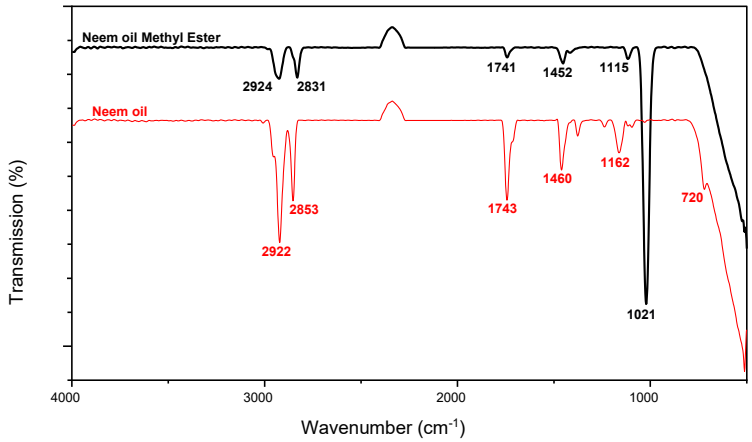

Figure -7 FTIR analyses of neem oil and NOME.

3.5.2 NOME characterization by Nuclear Magnetic Resonance (H-NMR):

The conversion of neem oil to neem oil methyl ester (NOME) was analyzed by using H-NMR as shown in the Figures-8 & 9. The solvent used internal references for H-NMR with deuterated chloroform ($CDCl_3$) [28]**.** The conversion of raw neem oil and neem oil to methyl esters was determined by the ratio of signals at 3.209 ppm (NOME) (methoxy groups of the methyl esters) and 1.123 ppm (NOME), (α-carbon CH_2groups of all fatty acid derivatives). The peaks at 5.153 ppm and 5.086 ppm were due to olefinic hydrogen. The NOME yield from neem oil could be quantified from H-NMR integration area.

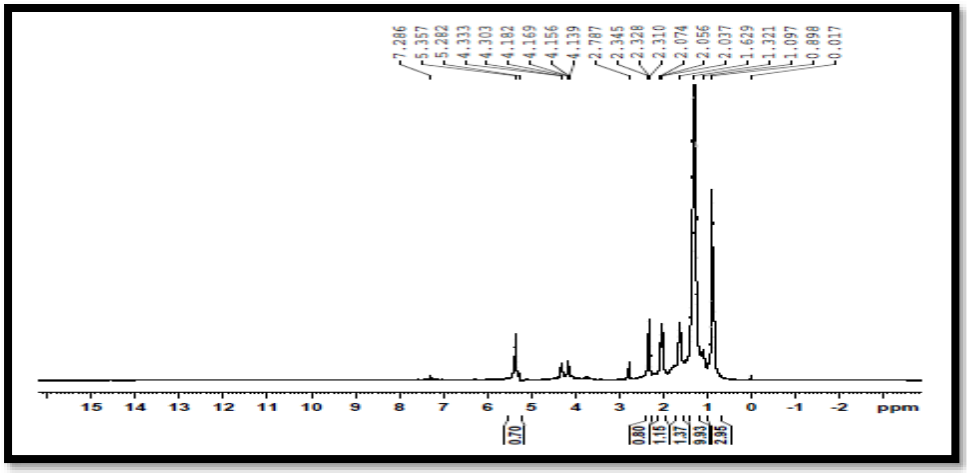

Figure 8: H-NMR studies of Raw Neem oil.

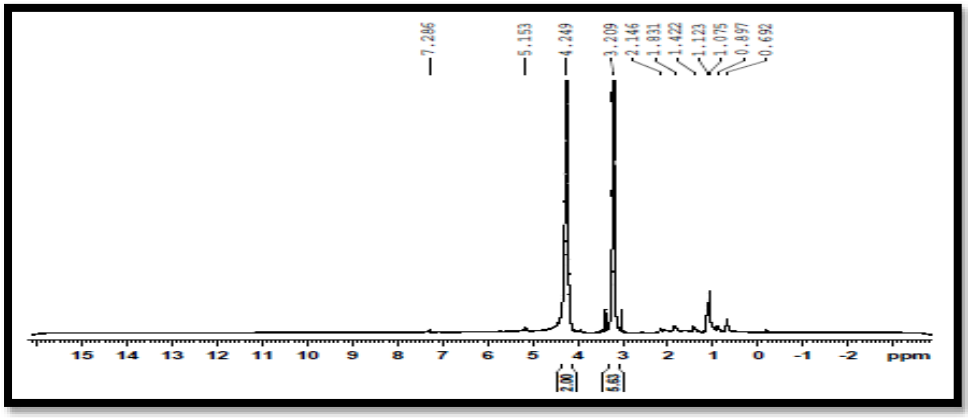

Figure 9: H-NMR studies of NOME

3.6 Gas Chromotography - Mass Spectroscopy(GCMS) analysis of NOME

Gas chromatography (GC) was used to determine the composition of esters in biodiesel and to characterize pure biodiesel. MS chromatogram with the mass spectra available in the library (NIST). After analyzing the NOME, 12 components were identified through the GCMS chromatogram as shown in the **Figure-10** shows the biodiesel components, which constitute 93% of the (NOME). The results shown in **Table 2** indicate that the main components of methyl ester, according the percentages, are as follows 9-Octadecenoic acid(Z)-, methyl ester(13.95%) Hexadecanoic acid, methyl ester (15.21%), Methyl stearate (3.92%).Unidentified esters of lard biodiesel go uncounted and compounds present in minor amounts(less than 0.1% peak area) were not considered further [29].

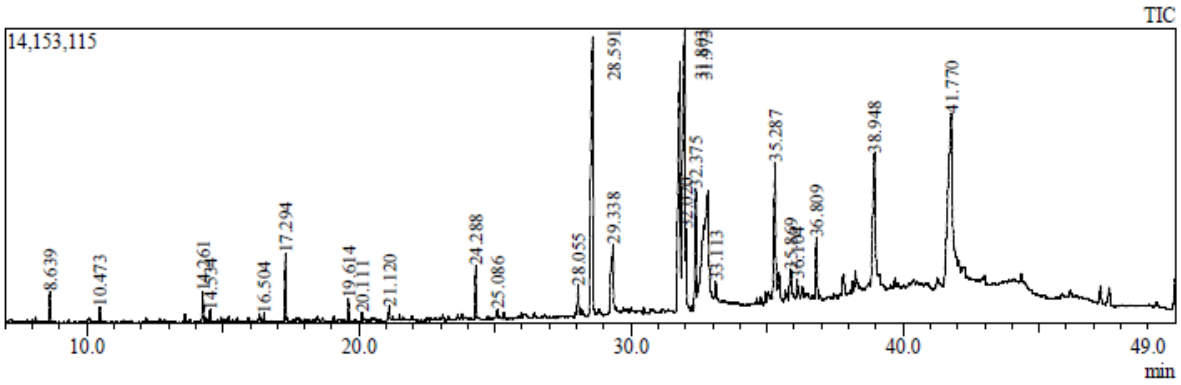

Figure 10: GCMS report of NOME

Table-2 Composition of fatty acid methyl ester in NOME

S.No	Compounds	Molecular formula	Molecular wt	Retention time	Area %
1	**9,12-Octadecadienoic acid (Z,Z)-, methyl ester**	$C_{15}H_{34}O_2$	294	31.803	14.48
2	**9-Octadecenoic acid, methyl ester, (E)-**	$C_{16}H_{36}O_2$	296	31.973	13.95
3	**Hexadecanoic acid, methyl ester**	$C_{17}H_{34}O_2$	270	28.591	15.21
4	**Methyl tetradecanoate**	$C_{15}H_{30}O_2$	242	24.288	1.27
5	**Methyl stearate**	$C_{19}H_{38}O_2$	298	32.375	3.92
6	**9-Octadecenoic acid, 12-hydroxy-, methyl ester, (Z)-**	$C_{19}H_{36}O_3$	312	35.287	6.24
7	**n-Hexadecanoic acid**	$C_{16}H_{32}O_2$	256	29.338	3.87
8	**Heneicosanoic acid, methyl ester**	$C_{22}H_{44}O_2$	340	35.869	1.48
9	**Hexadecanoic acid, 2-hydroxy-1-(hydroxymethyl)ethyl ester**	$C_{19}H_{38}O_4$	370	38.948	8.07
10	**6-Octadecenoic acid, methyl ester, (Z)-**	$C_{19}H_{36}O_2$	296	32.020	1.51
11	**Tricyclo[20.8.0.0(7,16)]triac**	$C_{30}H_{52}O_2$	444	36.809	2.14

	ontane, 1(22),7(16)-diepoxy-				
12	**9-Octadecenoic acid (Z)-, 2,3-dihydroxypropyl ester**	$C_{21}H_{40}O_4$	356	41.770	20.66

3.7 Regeneration of nanocatalyst

Transesterification of triglycerides into biodiesel and glycerol is made possible by nanocatalysts, which have recently become essential in the biodiesel production process. However, nanocatalysts can deactivate or foul with time, reducing their effectiveness and lifespan. One environmentally friendly way to increase process efficiency and extend the lifetime of catalysts is by regeneration of nanocatalyst. According to this study, iterative cycles result in a decrease in yield. The removal of active sites during deactivation could account for the observed drop in catalytic activity. Another factor to think about is the likelihood of undesirable byproducts being formed and then deposited on the surface of the catalyst [30]. The CaO nanocatalyst can be reused for up to five cycles, according to several experiments **(Figure-11**).

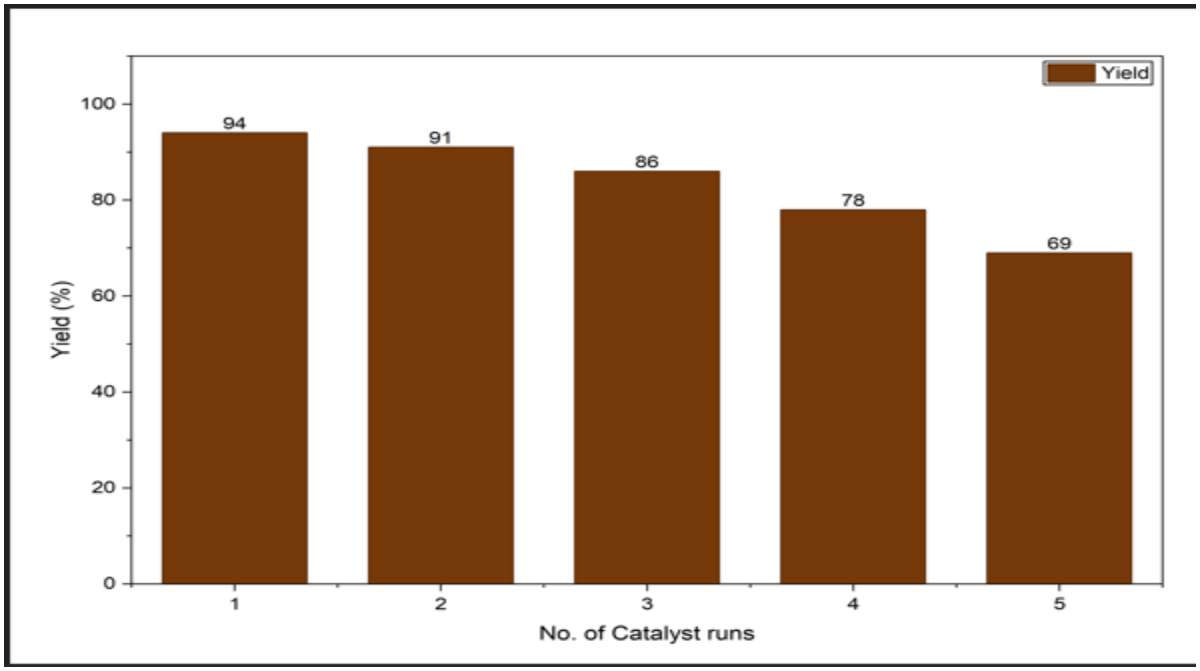

Figure-11: No of catalyst cycles for methyl ester production.

The CaO-catalyzed transesterification, which used calcined hydrated lime, demonstrated remarkable water tolerance in the oil. The FAME yield of neem oil remained constant at 94 percent as long as the oil's water content was below 5 wt%. An increase in the catalytic activity of CaO during transesterification can be achieved by adding a tiny quantity of water to the reaction stream. This is

because water generates an OH group, which speeds up the creation of methoxide anion. But, the yield of FAME dropped to 69% when the water concentration in the reaction exceeded 5 wt%, as this level of water could induce soap to forms. According to the study of Sarah Shakir Mahmood and Atheer Mohammed Al-Yaqoobi, 2024 the obtained results were similar [31].

Conclusion

Using CaO as a nanocatalyst may increase the output of methyl ester from linseed and neem oil. Nano CaO provides a realistic and cost-effective way to speed up the transesterification process, with the added benefit of being easy to recover and reuse. Because nano CaO is easily extracted from the methyl ester and reused up to five cycles, the approach is more sustainable. Using a 1:20 oil-to-methanol ratio for 2 hours at 50 °C with a 1.0 wt % catalyst, this synthetic nanocatalyst achieved a NOME production yield of 94%; conversely, at its optimal levels, the LOME yield was 90%. By weight, NOME primarily consists of the following ingredients: 9-Octadecenoic acid(Z)-methyl ester (13.95%), hexadecanoic acid (15.21%), and methyl stearate (3.92%). It is possible to lessen dependence on fossil fuels and crude oil imports by using methyl ester produced from linseed and neem oil, which has characteristics that are quite similar to petroleum diesels.

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