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Qualitative analysis of FAMEs of Biodiesel production from waste cooking oil using GC-MS technique

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Abstract

The global energy scenario faces a change in the sea due to an increasing alarm compared to climate change, the decline in fossil fuel resources and ecological costs of traditional energy sources [1]. One of the main advantages of using WCO as raw materials to produce biological diesel is relatively low cost. Waste oil is much cheaper than pure oil, and therefore, the production of bio -diesel oil from WCO is economically possible [2]. Increasing demand for biological diesel is also supported by government incentives and policies to apply renewable energy [3] . With concerns about energy security, environmental sustainability and climate change day by day, biological diesel has become an important player in the renewable energy space. The world trending towards sustainable energy solutions, biodiesel has become a prominent option as an alternative to fossil fuels. Biodiesel, dominated by Fatty Acid Methyl Esters (FAMEs), is a biodegradable, renewable fuel made from biological materials like vegetable oils, animal fat, and waste cooking oil [4]. Pretreatment acid catalysed esterification was carried out to reduce the free fatty acid number of oil. Physicochemical parameters of waste cooking oil (WCO), such as free fatty acid (FFA) value, moisture content, saponification value, were determined to evaluate the suitability of the oil for biodiesel production. Such parameters indicate the feasibility of transesterification and biodiesel conversion with good efficiency. Waste cooking oil was trans-esterified in a 250 ml three -neck round-bottom flask followed by controlled temperature 60-65^{O}C, waste cooking oil (20 grams), methanol (95% v/v) with a molar concentration 12:1, and 5wt% catalyst (5% Zn doped CaO) was mixed together to achieve maximum biodiesel. Chemical composition analysis by GC-MS shoed the presence of about 82.35% fatty acid methyl esters found. Fuel properties like kinematic viscosity ,flash ,pour,fire ,cloud point,acid value FFA conversion rate, density and viscosity of biodiesel found in accordance with standard American Society testing abd Materials biodiesel specification.

Keywords: Biodiesel, Waste Cooking Oil, Transesterification, GC-MS, Fatty Acid Methyl Esters (FAMEs), Renewable Energy

1. **Introduction**

The burning of fossil fuels such as petroleum diesel unlocks carbon stored under the ground for millions of years, contributing to atmospheric CO_2 levels and widening global warming. On the contrary, biodiesel is made from modern biological origin sources such as vegetable oils, animal fats, and waste cooking oils. They come from crops that uptake CO_2 when photosynthesizing. Diesel ingine contribute in economic value in developing countries such engines run with petroleum diesel this petroleum diesel produces consequently, the carbon dioxide emitted when biodiesel is burned is approximately equal to the quantity already taken in by the plants that were utilized to make the fuel creating a closed carbon cycle.. This makes biodiesel especially useful in urban areas, where high traffic densities lead to increased CO levels and serious health concerns for occupants. Smoke particulate matters oxides of nitrogen oxides of carbon unburned hydrocarbon etc and they have limited resources Biodiesel, as a result of having more oxygen content, so therefore we need to move towards alternative fuel like biodiesel [5]. Favors complete combustion, thus producing reduced carbon monoxide emissions

The biodegradability and non-toxicity of biodiesel minimize the environmental impact of accidental spills, fuel leaks, and improper disposal, thus contributing to ecological conservation. Biodiesel has a high biodegradation rate based on its chemical makeup, which is mainly constituted by fatty acid methyl esters (FAMEs). Research has proven that in conditions conducive to environment (i.e., suitable temperature, microbial process, and amount of oxygen), biodiesel would be degraded three times or four times faster compared to petroleum diesel. The pollution of water sources due to WCO pollution is a serious community health risk, which can cause gastrointestinal diseases and other health problems in the community depending on water sources unreasonable treatment [6]. The entertainment of used cooking oil of drainage and discharge systems reduces the possibility of blocked drainage systems, the destruction of pests and odour pollution. The economic and environmental advantages of WCO recycling illustrate the ability to help sustainable development as well as cleaner and more effective energy industry [7]. The waist cooking oil or frying oil is considered to be much better replacement as its price is 65% less than pure oil [8]so rather than dumping this oil without treatment it can be utilised in biodiesel formation as a cheap and efficient alternative feedstock [9]. During pre treatment methods distinct parameters a waste cooking oil contain high proportion of free fatty acids as a result of repeated frying it hinders its conversion into esters. Therefore an initial pretreatment is carried out to lower the free fatty acid content of waste cooking oil [10].Most common pretreatment procedure include heat treatment glycerol treatment and acid catalysed esterification oil with the acid values less than 1% is selected for conversion into biodiesel as it gives higher biodiesel yield[11]. Microwave assisted catalyst,enzymatic green catalyst, pyrolysis ,microemulsion and transesterification are few methods that may be used to produce biodiesel .The transesterification method for producing biodiesel is a base catalysed transterification, due to its great efficiency and relative high conversion efficiency catalyst Zn doped CaO is utilised. The advantage of using heterogenous catalyst over homogenous catalyst is that Reusability and Regeneration of Zn-CaO solid catalyst, that can be reused multiple times with minimal loss in activity whereas Homogeneous catalysts cannot be recovered and reused, leading to higher operational costs.it also this proves the limitation for produces minimal soap formation even with oils containing free fatty acids (FFA).Zn modification enhances the acid resistance of CaO, making it more effective for feedstocks with moderate to high FFA, like waste cooking oil. Methanol Is preferred among other alcohols due to a slow cost and fast reaction.Glycerol is a main byproduct of transesterification reaction which requires complete separation from biodiesel to different purification process[12].

To accurately determine the composition of biodiesel, Gas Chromatography–Mass Spectrometry (GC-MS) was used to analyze the fatty acid methyl esters (FAMEs) present in the trans esterified product. Fuel characteristics are measured by ASTM protocols and compared with the standard range provided for biodiesel[13] .Projective of this study to generate biodiesel from waste cooking oil through transistorification procedure along with the parameter optimization for its efficient utilisation in biodiesel production this present research work focus on chemical characterization and fuel properties analysis of obtained biodiesel by GC MS and ASTM standard protocol.

2. **Materials and methods**

The waste cooking oil samples were collected from different sources restaurant kitchens street vendors and shopkeepers Of Jhansi Uttar Pradesh india in dried canes and bottles all the samples were analysed by physiochemical parameters among them household kitchen Sample was found to be highly efficient for conversion into biodiesel. samples was filtered out to remove food residues left in them after cooking or frying and heated at 100 degree Celsius to remove moisture content. Ethanol distilled water beaker measuring cylinder thermometer wire mesh philtre magnetic steerer heating chamber philtre paper sulfuric acid you know and weighing machines were all needed.

2.1. Physicochemical analysis

The properties like acid value density and saponification value were analysed and performed density of waste cooking oil was measured using mass over volume formula .Acid value of sample was obtained by taking 5 grams of oil into a 50 mL conical flask. On Adding 25 mL of neutral ethanol to the flask. To dissolve oil completely mixture was warmed and stirred. On Adding 2–3 drops of phenolphthalein, the solution should remain colourless at this point (if acidic). Titration with 0.1 N alcoholic KOH solution from a burette until a light pink colour persists for at least 15 seconds (endpoint). Against this a bank was run in parallel which contained similar components except oil which was replaced by water .This would determine the acid value of the WCO.

$$\text{Acid Value (AV)} = \frac{V \times N \times 56.1}{W}$$

Where:

V = Volume of KOH used (mL)

N = Normality of KOH (usually 0.1 N)

W = Weight of oil sample (g)

56.1 = Equivalent weight of KOH

Weight of WCO sample (W) = 5.0 g

Volume of 0.1 N KOH used (V) = 5.0 mL

Normality of KOH (N) = 0.1 N

$$AV = \frac{5 \times 0.1 \times 56.1}{5}$$

$$= 5.61 \text{mg KOH/g}$$

WCO was weighted about 5 grams into a 50 mL conical flask. On Adding 25 mL of 0.5 N ethanolic KOH to the flask that would be Attached to a reflux condenser to avoid evaporation. Mixture was heated gently on a boiling water bath for 60 minutes. It was stirred time to time to ensure complete saponification. After refluxing on adding 1–2 drops of phenolphthalein to the warm solution, the excess KOH with 0.5 N HCl was titrated until the pink colour just disappears. A blank was also run with the same volume of ethanolic KOH without oil.

$$SV = \frac{(B-S) \times N \times 56.1}{W}$$

Where:

B = Volume of HCl used in blank (50mL)

S = Volume of HCl used in sample (39.2mL)

N = Normality of HCl (0.5 N)

W = Weight of oil sample (5 g)

56.1 = Equivalent weight of KOH

$$SV = \frac{(50 - 23.1) \times 5 \times 56.1}{5}$$

$$= 151.17 \text{mg KOH/g}$$

The moisture content of waste cooking oil (WCO) was determined using the oven drying method. 5 grams of the oil sample were accurately weighed into a clean, dry crucible, and the initial weight was recorded. The crucible containing the sample was placed in a hot air oven maintained at 105 °C for 1.5 to 2 hours to allow complete evaporation of moisture. After drying, the crucible was cooled in a desiccator to prevent moisture absorption from the atmosphere, and the final weight was recorded. The percentage of moisture was calculated based on the weight loss before and after drying using the formula:

$$\text{Moisture (\%)} = \underline{\text{Initial weight} - \text{Final weight}} \quad \text{X } 100$$

Sample weight

Free fatty acid % was also analysed simultaneously .It was found to be 2.82%

2.2. Pre-treatment

To achieve the lowest free fatty acid content acid treatment was applied on the waste cooking oil by pretreatment procedure .Acid treatment, was applied on all the waste oil samples to achieve the lowest free fatty acid (FFA)content. Esterification with the use of homogenous acids is a commonly employed procedure in which 5gm of oil was added with methanol, acid catalyst (conc. H2SO4, HCl and H3PO4) and reaction mixture was transferred to round bottom three necked flask attached with water cooled condenser placed on hot plate.

Typical reaction efficiencies of pretreatment of waste cooking oil with acid catalyst (H_2SO_4) under the specified conditions (5 wt% catalyst loading, 12:1 methanol to oil molar ratio, 60–65 °C, 5-hour reaction time. Reducing FFA below 1% makes the oil suitable for base-catalyzed transesterification.

2.3. Biodiesel conversion (Transesterification)

Pretreated waste cooking oil was transesterified in a 250 ml three -neck round-bottom flask equipped with an oil bath, magnetic stirrer, and a water-cooled reflux condenser. At a controlled temperature 60-65^{O}C, waste cooking oil (20 grams), methanol (95% v/v) with a molar concentration 12:1, and 5wt% catalyst (5% Zn doped CaO) was mixed together and continuously stirred. The catalyst was first doped with CaO and then used in transterification reaction to produce nanocrystallinity.

Wet impregnation was used to create the nanocrystalline Zn/CaO. Prepared Zinc Sulphate Solution weighing accurately $ZnSO_4$. $7H_2O$ to match the desired doping level (0.075g for 1.5% to 0.350g for 7%). Dissolved it in 10 mL of deionized water to form a clear zinc sulphate solution. Taking 5 g of CaO and suspend it in 40 mL of deionized water in a 250 mL beaker. Stirred continuously using a glass rod or magnetic stirrer to form a homogenous slurry. Slowly added the zinc sulphate solution into the CaO suspension while stirring. While continued stirring for 2–3 hours at room temperature for uniform dispersion of Zn^{2+} ions onto CaO particles. After stirring, mixture settled, and dried in an oven at 110°C for 12 hours to remove moisture. The dry mixture was then calcined for four hours at 700°C in a muffle furnace. When the transesterification reaction was completed the reaction share was transferred to separating funnel for overnight to separate biodiesel from glycerol layer[14].

2.4. Purification of biodiesel

The biodiesel produced through the transesterification process was subjected to purification to eliminate residual catalysts, unreacted methanol, and soap content. Both wet and dry washing techniques were employed to achieve a clean final product. In the wet washing method, water was heated to 60 °C and used to repeatedly wash the biodiesel until a clear, distinct product layer was formed. The purified biodiesel layer was then dried by heating at 100 °C to remove any remaining moisture [15].For the dry wash method, silica was used as an adsorbent to eliminate impurities. Approximately 5% silica by weight was added to the biodiesel and the mixture was gently stirred at 60 °C for two hours. Following this, the mixture was left undisturbed to allow the silica to settle at the bottom. The purified biodiesel layer was then carefully separated from the settled adsorbent [16].

2.5. GC-MS analysis

GC-MS is a widely accepted technique for the qualitative and quantitative assessment of individual fatty acids, offering high sensitivity and precise identification of molecular structures.

The GC-MS analysis was performed using an Agilent 7890B gas chromatograph coupled with a 5977A mass spectrometer. The FAMEs were separated on a DB-WAX capillary column (30 m × 0.25 mm ID × 0.25 µm film thickness)was used. Helium act as carrier gas used at a constant flow rate of 1.0 mL/min,Injection Volume of sample 100 µL in 900 µL (split ratio 10:1)of chloroform solvent in an Eppendorf taken .Column Temperature Program was Initially maintained at 60°C (held for 2 min) and then increased to 350°C at 10°C/minutes to Final ramp at 400°C at 5°C/min (held for 10 min).The column Injector Temperature was set at 250°C

MS Conditions were Electron Ionization (EI) mode at 70 eV, scan range 50–550 m/z,IS standard -Palmitic acid Molecular weight= 650, mass=1mg in sample (biodiesel) 20mg of weight was used to measured.

2.6. Fuel properties analysis

The fuel properties of biodiesel such as kinematic viscosity carbon residue test cloud port flash point fire point were determined according to the astm protocols carbon residue of sample was measured by using Conradson carbon residue apparatus, flash point by Pensky Martens closed cup tester,calorific value by bomb calorimeter,kinematic viscosity by viscometer and ash content in a muffle furnance.

3. **Results and discussion**

3.1 Physical properties of waste cooking oil

Waste cooking oil exhibits altered properties compared to pure oil, as it is repeatedly used for frying at high temperatures. These changes in characteristics vary among different waste oils, depending on the frequency of heating, the peak temperatures reached, and the type of food cooked in the oil. Waste cooking oil was Collected from household kitchens was found to be have different density with acid value as well as saponification value due to access hydrolysis and polymerization reaction. Density and saponification value of samples were 919 kg/m^3, 151.17 mg KOH/g and moisture content was 0.29%.

Table.1 Physicochemical Properties of Waste Cooking Oil (WCO)

Parameter	**Measured Value**
Acid Value	5.61
Moisture Content (wt.%)	0.29
Saponification Value (mg KOH/g sample)	151.17

3.2 pretreatment

Waste cooking oil sample was treated with pretreatment procedure and the acid value of oil after the methods are given in the table 2 it was found to be lower Acid value with acid treatment and free fatty acid content was found to be less than before and it was found to be free fatty acid conversion rate about 84% that would be better to use for further transfer reactions.

Table 2. parameters for waste cooking oil samples after pre-treatment procedures.

Parameter	Before Pretreatment	After Pretreatment
Acid Value (mg KOH/g)	5.61	**0.9**
FFA (%)	2.82%	**0.45%**
FFA conversion rate	——	**84%**

3.3 Characterisation of biodiesel

Transesterification procedure was catalysed by using zinc doped calcium oxide catalyst and physical and chemical parameters were observed on final biodiesel yield. The physical properties of biodiesel derived from waste cooking oil were evaluated, including kinematic viscosity, density, flash point, calorific value, ash content, and carbon residue. These properties were then compared with those of conventional diesel fuel based on ASTM standard protocols. The refined biodiesel exhibited a higher density (approximately 872 kg/m^3) compared to the typical diesel range of 830–860 kg/m^3. Its kinematic viscosity was also higher at 5.57 mm^2/s, while diesel generally falls within 1.9–4.1 mm^2/s. Due to this higher viscosity, biodiesel from recycled cooking oil is often blended with diesel to enhance performance. The flash point—defined as the temperature at which a fuel emits vapours that can ignite in air—was also notably higher for biodiesel, measured around 152 °C, compared to diesel's minimum standard flash point of 52 °C. A higher flash point indicates safer handling, storage, and transportation characteristics. The measurements of these and other relevant fuel properties are summarized in Table 3.

Table 3. fuel properties for Zn doped CaO catalysed biodiesel compared to pure diesel on ATSM protocols.

Property	**Typical Range of diesel ASTM std.**	**biodiesel**
Density @31°C	830–860	872kg/m^3

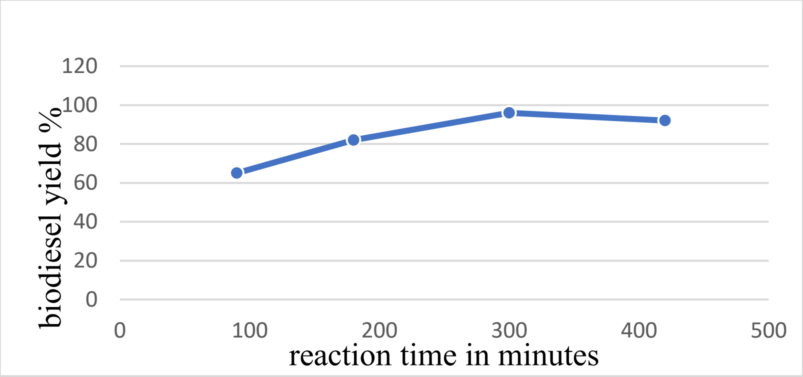
Kinematic Viscosity @40°C	1.9–4.1 mm²/s	5.5 cSt
Flash Point	≥ 52 °C	152 °C
Cloud Point	-15 to +5 °C (varies)	5
Pour Point	-35 to 0 °C (varies)	2
Carbon Residue	≤ 0.35% (by mass)	0.04%
Ash Content	≤ 0.01% (by mass)	0.02%
Fire Point	~65–96 °C	164
Calorific value	~45,000–46,000 kJ/kg	38 MJ/kg

3.4 Impact of reaction time on Biodiesel

The transesterification reaction was tracked for different time periods—90 minutes, 180 minutes, 300 minutes, and 420 minutes—to find out the time needed for the reaction to achieve equilibrium. The yield of biodiesel was quantified at every time interval, and the progress of the reaction was monitored by withdrawing samples of the reaction mixture at intervals. The reaction conditions like the methanol-to-oil molar ratio (12:1), catalyst loading (5 wt%), and temperature (60–65°C) were maintained constant during the experiment. By monitoring the conversion of waste cooking oil (WCO) to biodiesel at different time intervals, the research intended to determine the optimum reaction time required to obtain maximum biodiesel yield and determine the time taken to attain equilibrium in the process of transesterification.

The conversion was 65% at 90 minutes, 82% at 180 minutes, 96% at 300 minutes, and 92% at 420 minutes, showing that the reaction was close to completion. Beyond 90 minutes, prolonging the reaction time to 180 minutes only slightly increased the yield (82%), which indicated that the reaction had almost reached equilibrium at 180–300 minutes. Thus, a reaction time of about 300 minutes was found to be ideal for maximum biodiesel yield since prolonging the time further offered insignificant advantages and would only consume unnecessary energy. Yield of biodiesel rose constantly with reaction time, achieving a level of about 65% conversion at 90 minutes and 82% at 180 minutes. Exceeding 300 minutes, a further prolongation of the reaction time up to 400 minutes resulted decrease in a (96%), reaction proving that by 300 minutes, the reaction was practically at the stage of equilibrium. Thus, an optimal duration of reaction around 300 minutes is adequate to produce the highest biodiesel content without wastage of energy.

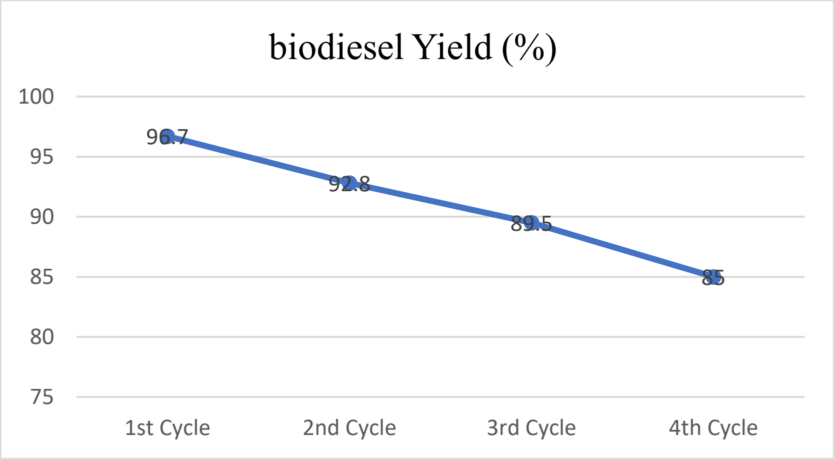
Figure 1. Effect of reaction time on biodiesel yield



3.5 . Impact of reusability of catalyst

The reusability of the 5-Zn/CaO-700 catalyst was investigated through successive transesterification reaction using the catalyst for repeated cycles.The 5-Zn/CaO-700 catalyst had outstanding catalytic activity for the initial three cycles, producing biodiesel yields of 96.7%, 92.8%, and 89.5%, respectively. This indicates that the catalyst was still efficient for multiple uses with minimal drop in yield between the first and second cycles. However, a significant reduction in yield to 85.0% was noted in the fourth cycle after the third cycle, reflecting some level of catalyst deactivation. This reduction in yield is thought to result from loss of active sites or changes in structure of the catalyst following repeated use, which restricted its performance. In spite of this, the catalyst was highly recyclable for three cycles and showed promise for sustainable biodiesel production with very little loss of efficiency.

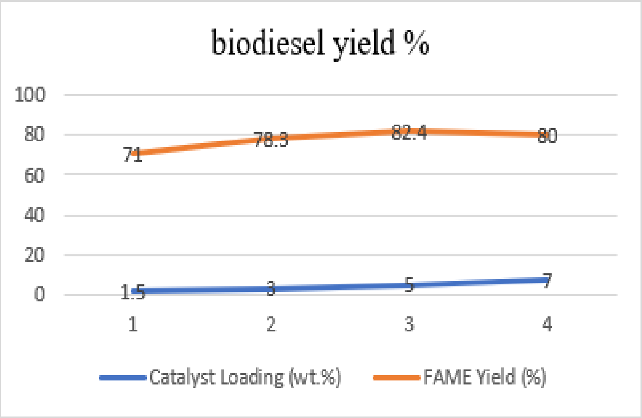
figure 2.: Effect on biodiesel yield on reusability of 5-Zn/CaO-700 catalyst in successive transesterification cycles



3.6. Impact of catalyst loading

Loading of catalyst greatly impacted the yield of biodiesel. The yield was increased by raising the loading from 1.5 wt% to 5 wt%, with 5 wt% yielding the highest biodiesel production (82.4%). If the loading was further increased to 7 wt%, however, yield decreased (80%) because of mass transfer restrictions and rising mixture viscosity, which makes 5 wt% the best catalyst loading for effective transesterification.

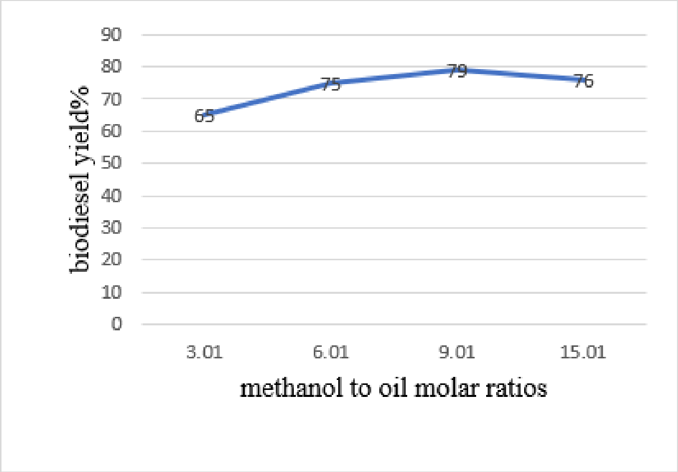
Figure 3. Effect of catalyst loading on biodiesel yield%.



3.7. Impact of methanol to oil molar ratio on biodiesel yield

The methanol-to-oil molar ratio is a critical factor in the transesterification process, as it significantly influences the formation of fatty acid methyl esters (FAMEs). Determining the optimal molar ratio is essential for maximizing biodiesel yield while minimizing production costs. In the case of heterogeneous catalysis, various molar ratios—3:1, 6:1, 9:1, and 12:1—were tested given in fig(4). Among these, the most effective ratios were found to be 9:1 and 12:1.Under catalytic conditions, methanol reacts readily with triglycerides at a temperature range of 60–65 °C, completing the reaction within 300 minutes. A 12:1 molar ratio produced the highest biodiesel yield of 82.3%. However, increasing the methanol ratio beyond 12:1 led to incomplete reactions due to difficulties in separating glycerol, some of which remained in the biodiesel phase. This residual glycerol lowered the apparent yield and affected the purity of the final product

Figure 4. Effect of methanol to oil molar ratio on biodiesel yield



3.8. Gas chromatography

Gas chromatography (GC) is a highly efficient analytical technique widely used for both qualitative and quantitative assessments due to its rapid analysis time and high resolution. In this study, the waste cooking oil underwent multiple rounds of washing and purification prior to conversion, and the final yield was analyzed using GC-MS. As illustrated in the figure, GC-MS effectively identified the major components present in the waste cooking oil, offering valuable insight into the success of the purification process before biodiesel production.A total of seven distinct components were identified through GC-MS based on their chemical composition. This technique enabled precise separation and detection of individual compounds, including various fatty acids, glycerides, and lipid-related substances. The identification of these components is critical in assessing the oil's quality, level of purity, and the presence of any residual contaminants or impurities.The integration results, presented in Table 5, include details such as retention time, area percentage, and peak height for each compound.

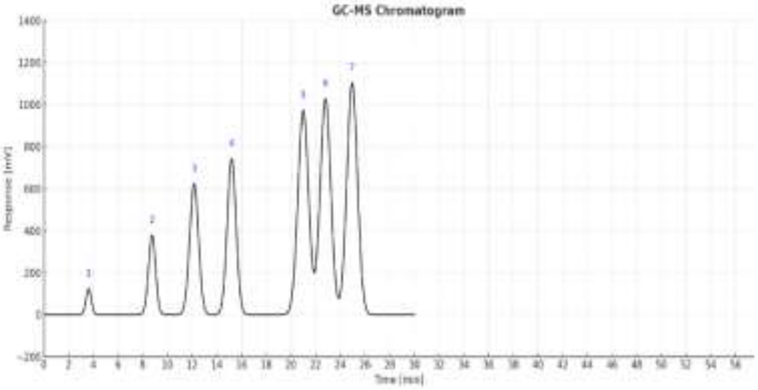


Table 5. The details of integration results.

Compound Name	**Height (mV)**	**Retention time**	**Area %**	**wt. %**
1.Methyl heptanoate (C7:0)	123.17	3.625	2.48%	1.28%
2.Methyl tetradecanoate (C14:0)	378.13	8.755	7.60%	6.59%
3.Methyl pentadecanoate (C15:0)	623.72	12.162	12.54%	11.50%
4.Methyl hexadecanoate (C16:0)	742.31	15.202	14.93%	14.44%
5.Methyl linoleate (C18:2)	1028.38	22.448	20.69%	21.77%

6.Methyl oleate (C18:1)	1103.25	24.963	22.19%	23.52%
7.Methyl stearate (C18:0)	973.21	21.529	19.57%	20.90%
Total	**5230.23**		**100.00**	**100.00**

Higher ester content, particularly fatty acid methyl esters (FAMEs), indicates a successful transesterification reaction and good-quality biodiesel with minimal side-product formation. The measured FAME content was used to evaluate the biodiesel yield and overall conversion efficiency. The methyl ester content of the biodiesel sample was calculated based on the area percentage obtained from GC-MS analysis, using the following formula:

$$\text{Methyl Ester Content (\%)} = \frac{\text{Methyl Ester Area (\%)} \times \text{Weight of the Product (g)}}{\text{Weight of the feedstock used(g)}}$$

Methyl Ester Area (%) = 97.52
Weight of the Product = 16.9 g
Weight of the Feedstock = 20 g

$$\text{Methyl ester \%} = \frac{97.52 \times 16.9}{20} = 1647.088$$

$$= 1647.088 / 20 = 82.3544$$

The methyl ester content of the biodiesel sample was found to be 82.35%. Overall these findings suggest that biodiesel produced from this cooking oil can be a promising alternative fuse source to reuse the environmental impact of traditional diesel fuel.

Conclusion

This research demonstrated that waste cooking oil serves as an efficient and cost-effective feedstock for biodiesel production. Key reaction parameters—including reaction time, catalyst concentration, and methanol-to-oil ratio—significantly influenced both the yield and the properties of the final biodiesel product. GC-MS analysis confirmed a high concentration of fatty acid methyl esters (FAMEs), indicating successful transesterification. Furthermore, the produced biodiesel exhibited favourable fuel properties that align with ASTM standards. When compared with conventional diesel, biodiesel showed notable improvements in performance characteristics. The findings suggest that biodiesel production from waste cooking oil is not only technically viable but also economically and environmentally sustainable. Since waste cooking oil can be collected from household and commercial kitchens at little to no cost, this approach reduces overall production expenses. Additionally, it helps lower greenhouse gas emissions and promotes the reuse of a valuable resource that would otherwise be discarded, supporting cleaner energy initiatives.

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Disclosure statement

No potential conflict of interest was reported by the author(s)

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