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SOLAR MICROWAVE-ASSISTED SYNTHESIS OF MODIFIED ALKALINE CATALYSTS FOR BIODIESEL PRODUCTION

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ABSTRACT

The global demand for eco-friendly energy sources has intensified the need for sustainable methods to synthesise modified alkaline catalysts for biodiesel production. Traditional methods are often costly and environmentally harmful. This study aims to develop high-efficiency, waste-based, modified alkaline catalysts using solar microwave-assisted synthesis. The focus is on optimising catalyst activity to enhance biodiesel yield from sunflower and waste cooking oil. Using the coprecipitation method, caO-rich hydrotalcite-like compounds (HLCs) were synthesised by impregnating Mg-Al hydrotalcite (HT Mg-Al) with 40 wt% eggshell-derived CaO. The study compared different drying methods, times, energy consumption, and costs involved in synthesising the catalysts. Characterisation techniques such as X-ray diffraction, BET, and SEM were used to analyse the catalysts' physicochemical properties. The catalytic activity was tested by transesterifying sunflower and waste cooking oil with methanol under microwave heating. Results showed that microwave drying significantly increased biodiesel yield compared to conventional methods. This study explores the originality of using a solar microwave-assisted method for catalyst synthesis and a conventional microwave for transesterification. The microwave-assisted technique achieved a 93.2% yield of fatty acid methyl esters (FAME) with a 30-minute reaction duration, 700 W microwave power, methanol-to-oil ratios of 6:1 and 9:1, and catalyst loading of 4 and 12 wt%. The catalysts were dried in 30 minutes, consuming only 0.9 kWh, demonstrating substantial energy savings when using a solar microwave power rating of 900W. In conclusion, this study successfully developed high-efficiency, waste-based, modified alkaline catalysts using solar microwave-assisted synthesis. This method enhances sustainability and economic viability in biodiesel production by promoting waste valorisation and reducing environmental pollution using waste-based materials.

Keywords: *Drying, eggshells, energy consumption, hydrotalcite-like compound, FAME, waste chicken sustainable*

INTRODUCTION

Biofuels encompass a diverse range of liquid, gaseous, and solid fuels primarily derived from biomass. They are crucial in enhancing energy security, reducing environmental impact, minimising reliance on fossil fuels, and promoting rural socioeconomic development [1]. Among biofuels, biodiesel has gained significant attention as a sustainable alternative to petroleum-based diesel due to its ability to reduce greenhouse gas emissions and dependence on finite crude oil reserves [2]. Biodiesel consists of long hydrocarbon chains with ester functional groups (-COOR) and is derived from plant oils, animal fats, or lipids, primarily triglycerides (TAGs). Its production relies on transesterification, a reaction catalysed by acids, bases, or enzymes that convert triglycerides into diglycerides, monoglycerides, and glycerol [3].

Catalyst selection is critical in biodiesel production, with heterogeneous catalysts offering advantages over homogeneous ones due to their ease of separation, reusability, and environmental benefits [4]. However, heterogeneous catalysis often requires higher reaction temperatures and alcohol-to-oil ratios to achieve high conversion efficiency [5]. Recent studies have explored novel catalyst preparation methods to enhance activity, selectivity, and stability, mainly focusing on microwave-assisted synthesis.

Microwave heating has been widely applied in biodiesel production because it provides rapid, uniform heating and improves reaction kinetics [6]. Conventional microwave-assisted transesterification has significantly reduced reaction times and energy consumption compared to traditional heating methods. For instance, studies have demonstrated that microwave-assisted transesterification can achieve over 90% biodiesel yield in less than 30 minutes, compared to several hours required for conventional heating [6].

Despite these advancements, limited research has been conducted on using solar microwave-assisted techniques for catalyst synthesis, which could further enhance the sustainability of biodiesel production [6]. Solar microwave technology integrates solar energy with microwave heating, reducing dependence on non-renewable electricity sources while maintaining the efficiency of microwave-assisted processes. This technique has shown promise in catalyst synthesis, improving surface area, porosity, and active site distribution, directly influencing catalytic performance [6]. However, comparative studies evaluating energy consumption, catalyst efficiency, and overall biodiesel yield using solar microwave technology versus conventional microwave or thermal methods remain scarce.

This study aims to address this gap by utilising a solar microwave-assisted approach for catalyst synthesis while employing a conventional microwave for transesterification to optimise biodiesel yield. The impact of microwave-assisted methods on catalyst structure, activity, and energy efficiency is critically evaluated, along with their implications for biodiesel yield. A detailed comparative analysis of catalyst performance, reaction conditions, and energy consumption is provided to highlight the benefits and potential limitations of solar microwave technology in biodiesel production.

METHODOLOGY

Catalyst Preparation

Waste eggshell-derived CaO was prepared by washing, drying in a 900W solar microwave, and crushing, followed by calcination in a furnace at 900°C for 3 hours [7]-[8]. The synthesis of HT Mg-Al via the coprecipitation method entailed dissolving magnesium and aluminium salts at the ratio of 2:1, adding sodium hydroxide and sodium carbonate, stirring, ageing, filtering, washing, drying in the 900 W solar microwave reactor for 30 minutes, sieving, and calcination in a furnace at 700°C for 5 hours [9].

Waste eggshell-derived hydrotalcite-supported CaO catalyst (CaO/MgAl) was prepared via the wet impregnation method according to a procedure available in the literature [10]. Accurately weigh the materials, dissolve them in distilled water, and heat them under magnetic stirring until drying, crushing, sieving, and calcination in a furnace at 800°C for 5 hours [11]. This work used two drying methods to synthesise CaO and CaO/MgAl catalysts.

The materials used for catalyst synthesis in this study were selected based on their high purity and suitability for heterogeneous catalysis. The precursor materials included magnesium nitrate hexahydrate ($Mg(NO_3)_2{\cdot}6H_2O$, 99.9%), aluminium nitrate nonahydrate ($Al(NO_3)_3{\cdot}9H_2O$, ≥98%), sodium hydroxide (NaOH, 99%), and sodium carbonate (Na_2CO_3, 99%), all of which were sourced from Merck and HmbG Chemicals. Waste chicken eggshells were also utilised as a natural calcium source, and distilled water was used as the solvent.

Catalyst Characterisation

The synthesised catalysts were characterised using X-ray diffraction (XRD), Brunauer–Emmett–Teller (BET) surface area analysis, and Scanning Electron Microscopy (SEM) to evaluate their structural, textural, and morphological properties. These characterisation techniques were conducted following established methodologies in the literature [10]-[11].

XRD was performed using a Bruker D8 Advance diffractometer equipped with a Cu-Kα radiation source (λ = 1.5406 Å), operating at 40 kV and 30 mA. The diffraction patterns were recorded in the 2θ range of 10° to 80°, with a step size of 0.02° and a scan speed of 2°/min. The XRD data were analysed to determine phase composition, crystallite size, and crystallinity index using the Scherrer equation to estimate crystallite sizes.

The textural properties of the catalysts, including surface area, pore volume, and pore size distribution, were determined using N_2 adsorption-desorption isotherms via the BET method. A Micromeritics ASAP

2020 instrument was used, with catalysts pre-treated by degassing at 200°C under vacuum for 4 hours to remove adsorbed moisture and impurities. The Barrett–Joyner–Halenda (BJH) method was applied to analyse pore size distribution from the desorption branch of the isotherms.

The catalysts' morphological characteristics were examined using a Zeiss Supra 55 VP Field Emission Scanning Electron Microscope (FE-SEM). Before imaging, the samples were sputter-coated with a thin layer of gold to enhance conductivity. SEM micrographs were captured at varying magnifications (×500 to ×50,000) to observe surface texture, particle dispersion, and porosity. Comparative analysis assessed the impact of different drying techniques (oven drying vs. microwave drying) on the surface structure and particle agglomeration.

Catalytic Testing

The catalytic performance of the synthesised catalysts was evaluated through the transesterification of sunflower oil and waste cooking oil using methanol as the alcohol reactant. The selection of two different types of oils aimed at assessing the catalysts' efficiency in converting refined and waste-based triglycerides into biodiesel. The biodiesel yield was determined as follows:

$$Biodiesel\ Yeild\ (\%) = \frac{mass\ of\ FAME}{mass\ of\ oil} \times 100 \qquad (1)$$

The reaction mixture was prepared by mixing methanol with sunflower or waste cooking oil in the prescribed molar ratios, as detailed in Table 1. The catalyst was dissolved in methanol at a specific loading percentage before being mixed with the heated oil. The transesterification process was conducted at 65°C, the optimal temperature for methanol-based transesterification, and the reaction was carried out for 30 minutes, according to a procedure available in the literature reaction times for microwave-assisted biodiesel production [12]-[13].

The reaction was performed in a domestic microwave reactor (700 W power output) to ensure rapid and uniform heating. The mixture was continuously stirred at 600 rpm to enhance mass transfer and reaction efficiency. Following the reaction, the catalyst was removed via filtration, and the reaction mixture was centrifuged at 4000 rpm for 10 minutes to separate the biodiesel and glycerol layers. The biodiesel phase was washed with warm distilled water (50°C) until a neutral pH was reached, followed by drying at 105°C to remove residual moisture before further analysis.

The selection of methanol-to-oil ratios was based on their reported effectiveness in previous studies. For sunflower oil, a 6:1 methanol-to-oil ratio was employed, as it closely aligns with the stoichiometric requirement for effective transesterification and has been shown to facilitate high biodiesel yields with minimal excess methanol consumption [14]. In contrast, a 9:1 methanol-to-oil ratio was utilised for waste cooking oil due to its higher free fatty acid (FFA) content. The increased methanol proportion helps to shift the reaction equilibrium toward biodiesel formation while mitigating soap formation caused by saponification reactions [15].

Similarly, the catalyst loading percentages were selected based on literature recommendations and experimental feasibility. A 4 wt% catalyst loading was used for sunflower oil, as its lower FFA content allows for efficient conversion with minimal catalyst requirements [16]. In the case of waste cooking oil, 12 wt% catalyst loading was applied, as the presence of impurities and elevated FFA levels necessitated a higher catalyst concentration to enhance reaction efficiency and improve biodiesel yield [17].

The biodiesel composition was analysed using an Agilent 7890A system equipped with a DB-5MS capillary column (30 m × 0.25 mm × 0.25 µm). The system was operated under the following conditions (Table 1) to ensure accurate separation and identification of fatty acid methyl esters (FAMEs).

Table 1 The GC-MS operating conditions for analysis

Parameters	Condition
Injector temperature	250°C
Injection mode	Split injection (split ratio 50:1)
Carrier gas	Helium (99.99% purity)
Flow Rate	1.0 mL/min
Oven temperature program	50°C (2 min) → 300°C at 10°C/min → Hold for 10 min
Ion source temperature	230°C
Detector temperature	28°C
Mass scan range	50 – 500 m/z

The biodiesel samples were diluted with n-hexane before injection into the GC-MS system. FAMEs were identified and quantified by comparing the obtained retention times and mass spectra with those from the National Institute of Standards and Technology (NIST) library database. The final biodiesel composition and quality assessment were performed under ASTM D6751 and EN 14214 standards.

DISCUSSION

Catalyst Characterisation

The catalytic characterisation results present a thorough understanding of the structural and morphological features of the solar microwave-assisted and oven-assisted synthesised CaO/MgAl catalyst. The XRD study revealed different diffraction patterns for each catalyst, showing its composition and structural features. In the case of Ca-containing catalysts, Figure 1 illustrates diffractogram peaks corresponding to $Ca(OH)_2$ (18.269°, 20.422°, 29.482°, and 32.318°), CaO (37.465°, 53.957°, and 62.413°), and MgO (39.478°, 43.006°, and 62.360°) phases. Notably, Al_2O_3 phases are absent, suggesting the dispersion of Al^{+3} ions within MgO without spinel structure formation [18]. The XRD patterns affirm that increasing Ca content enhances the crystallinity of Ca-containing phases, yielding well-crystallised Ca oxides post-calcination at 800°C. All catalysts exhibit mixed metal oxide phases, indicating the successful formation of frameworks comparable to oven-assisted synthesised catalysts, as reported in the literature [11].

SEM research revealed similar surface morphologies for solar microwave-assisted and oven-assisted synthesised CaO/MgAl, demonstrating surface homogeneity and exhibiting mesoporous structures, as shown in Figure 2. Following Ca impregnation, the change from platelet-like MgAl structures to CaO morphologies was evident and consistent, as reported in the literature [10].

BET surface area analysis provided critical insights into the structural properties of the solar microwave-assisted and oven-assisted synthesised CaO/MgAl. Both catalysts have similar nitrogen adsorption-desorption isotherms, as shown in Figure 3, due to hysteresis patterns consistent with plate-like structures and slit-shaped pores. However, solar microwave-assisted catalysts have a diminutive difference in surface area, pore volume, and pore size compared to conventional synthesised CaO/MgAl as the presence of bulky calcium impregnation was noted to obstruct MgAl pores, limiting internal surface accessibility for adsorption [10].

These results demonstrated the suitability of the synthesised catalysts for the transesterification of sunflower oil and waste cooking oil with methanol, highlighting their catalytic efficacy. These findings support the dependability and effectiveness of the solar microwave-assisted synthesis approach, which yielded catalysts with surface properties comparable to those produced using conventional techniques.

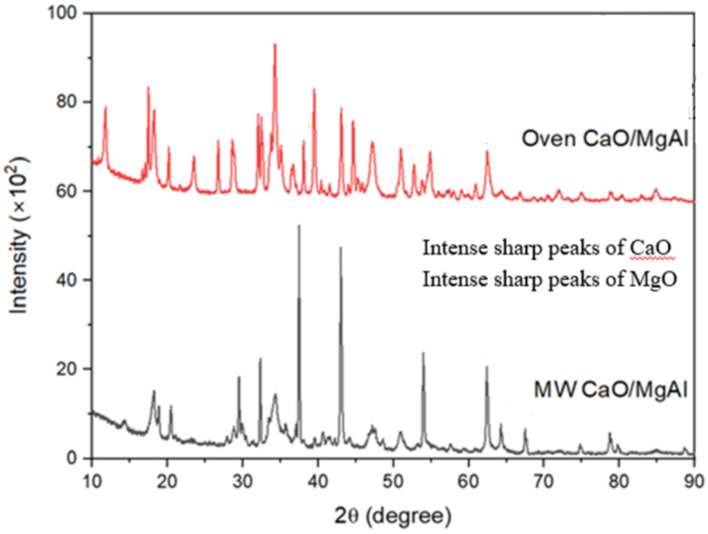
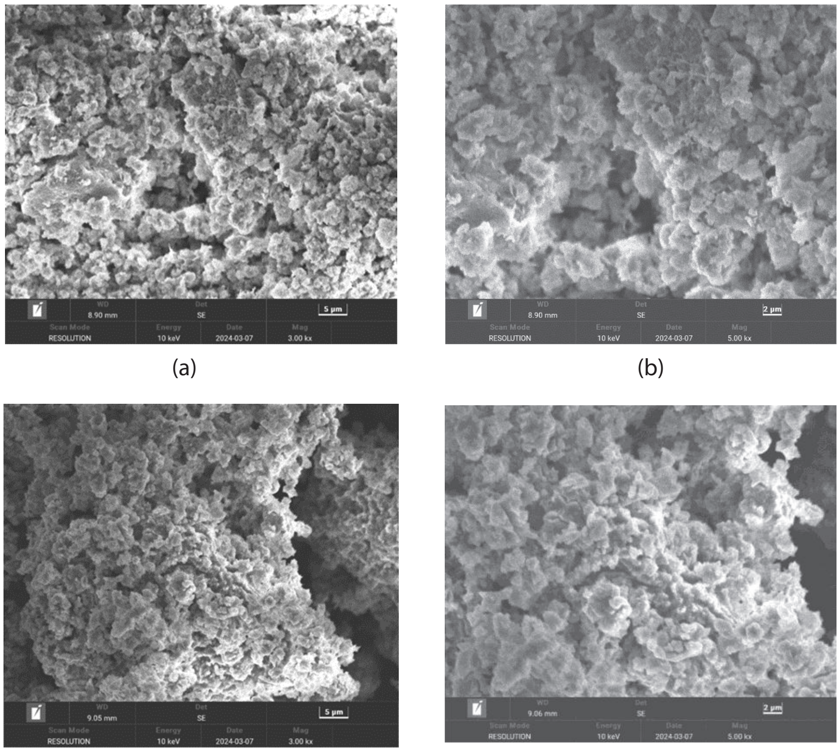


Figure 1 XRD of conventional and solar microwave-assisted synthesised CaO/MgAl



(a) (b)

(c) (d)

Figure 2 SEM of (a, b) solar microwave-assisted synthesised CaO/MgAl and (c, d) conventional synthesised CaO/MgAl

Table 2 BET properties of solar microwave-assisted and conventionally synthesised CaO/MgAl catalysts

Catalysts	Brunauer – Emmett – Teller Properties		
	Surface area (m^2/g)	Pore volume (cm^3/g)	Pore size (nm)
[a]CaO/MgAl	7.2133	0.038060	24.9974
[b]CaO/MgAl	5.9666	0.027479	22.2293

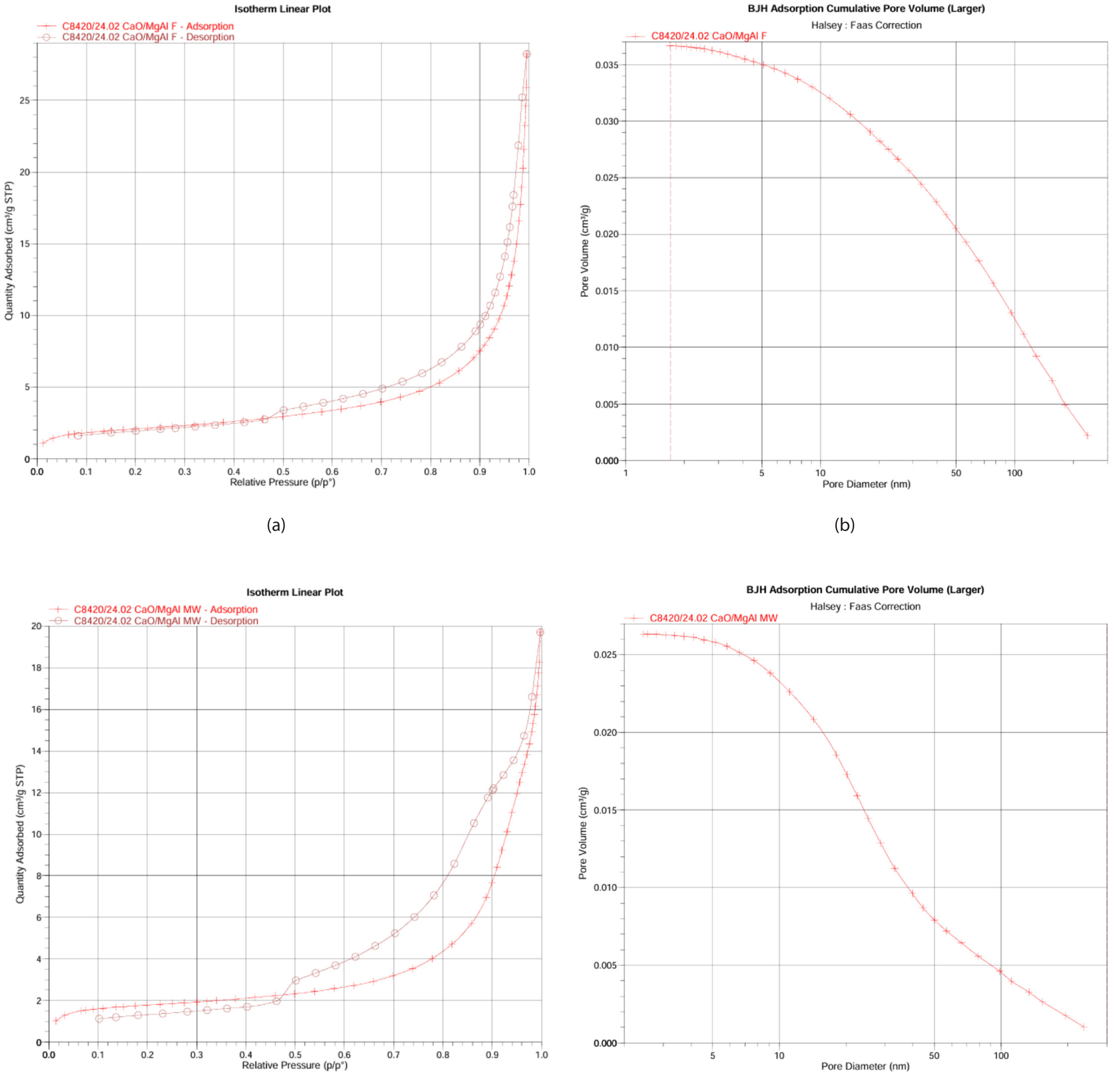
[a] Drying process is carried out in an oven at 120°C for 24 hours.
[b] Drying process carried out in a solar microwave reactor at 120°C for 30 minutes.

Catalyst Testing

The catalytic testing determined the effectiveness of solar microwave-assisted synthesised CaO/MgAl catalysts in transesterifying sunflower oil and waste cooking oil with methanol. The experimental setup included using a catalyst loading of 4 and 12 wt%, maintaining specific methanol-to-oil molar ratios of 6:1 for sunflower oil and 9:1 for waste cooking oil, and conducting the reaction at 65°C for 30 minutes [19].

These conditions were selected based on literature and optimisation studies to ensure efficient biodiesel production. The 4 wt% catalyst loading was used for sunflower oil due to its lower FFA content, which allows for effective transesterification with minimal catalyst consumption. Conversely, 12 wt% catalyst loading was applied to waste cooking oil as its higher FFA content and presence of impurities necessitate a greater catalyst concentration to enhance reaction efficiency and minimise side reactions such as saponification [20]-[21].

The methanol-to-oil ratios were chosen based on their impact on reaction equilibrium. A 6:1 ratio for sunflower oil follows the stoichiometric requirement for transesterification, promoting high conversion efficiency without excess methanol waste [22]. A 9:1 methanol-to-oil ratio was adopted for waste cooking oil to counteract FFA's inhibitory effects, ensuring sufficient methanol availability to drive the reaction forward while reducing unwanted soap formation [26].



(a) (b)

(c) (d)

Figure 3 Nitrogen (a, c) adsorption-desorption isotherms and (b, d) adsorption cumulative pore volume of the conventional synthesised CaO/MgAl catalyst and solar microwave-assisted synthesised CaO/MgAl catalyst, respectively

The reaction time of 30 minutes at 65°C was selected based on prior studies demonstrating that microwave-assisted transesterification significantly accelerates reaction kinetics compared to conventional heating methods. The temperature of 65°C was maintained to stay within the boiling point of methanol (64.7°C), preventing excessive evaporation and ensuring optimal reaction efficiency. The shorter reaction duration also aligns with the benefits of microwave-assisted processes, which facilitate rapid heating and improved molecular interactions, thereby enhancing biodiesel yield within a reduced time frame [12],[23].

The catalytic experiments yielded biodiesel, as shown in Table 3, indicating that the solar microwave-assisted synthesised catalyst successfully facilitated the transesterification process. The solar microwave-assisted catalysts demonstrated higher biodiesel yields than oven-assisted synthesised catalysts, indicating enhanced catalytic activity due to improved physicochemical properties. For sunflower oil, the highest yield (93.20%) was achieved using solar microwave-assisted CaO/MgAl, while the yield for waste cooking oil was slightly lower (89.44%), likely due to the presence of FFA that may interfere with transesterification efficiency.

In comparison, oven-assisted synthesised CaO and CaO/MgAl catalysts exhibited lower biodiesel yields, particularly for waste cooking oil, with yields of 69.68% and 74.36%, respectively. This suggests that higher FFAs and impurities in waste cooking oil affect catalyst performance, necessitating optimised conditions for maximum conversion. The significant improvement in biodiesel yield with solar microwave-assisted catalysts (CaO/MgAl) highlights the advantages of solar microwave drying, which enhances catalyst surface area, porosity, and active site availability. These findings confirm that the synthesis method plays a crucial role in catalyst efficiency and demonstrates the potential of solar microwave-assisted synthesis as a superior technique for biodiesel production.

The GC-MS analysis was conducted to determine the composition and purity of the produced biodiesel. The total ion chromatograms (TICs) for the transesterified sunflower oil and waste cooking oil using different catalysts are presented in Figure 4. The retention times and peak intensities indicate the presence of key FAMEs, confirming the successful conversion of triglycerides into biodiesel. The significant peaks observed around 9.0, 14.3–14.9, and 15.3 minutes correspond to methyl palmitate, methyl stearate, and methyl oleate, respectively, which are the primary constituents of biodiesel. The variations in peak intensity and additional minor peaks at higher retention times suggest the presence of trace impurities or unconverted intermediates, particularly in waste cooking oil samples due to their higher FFA content.

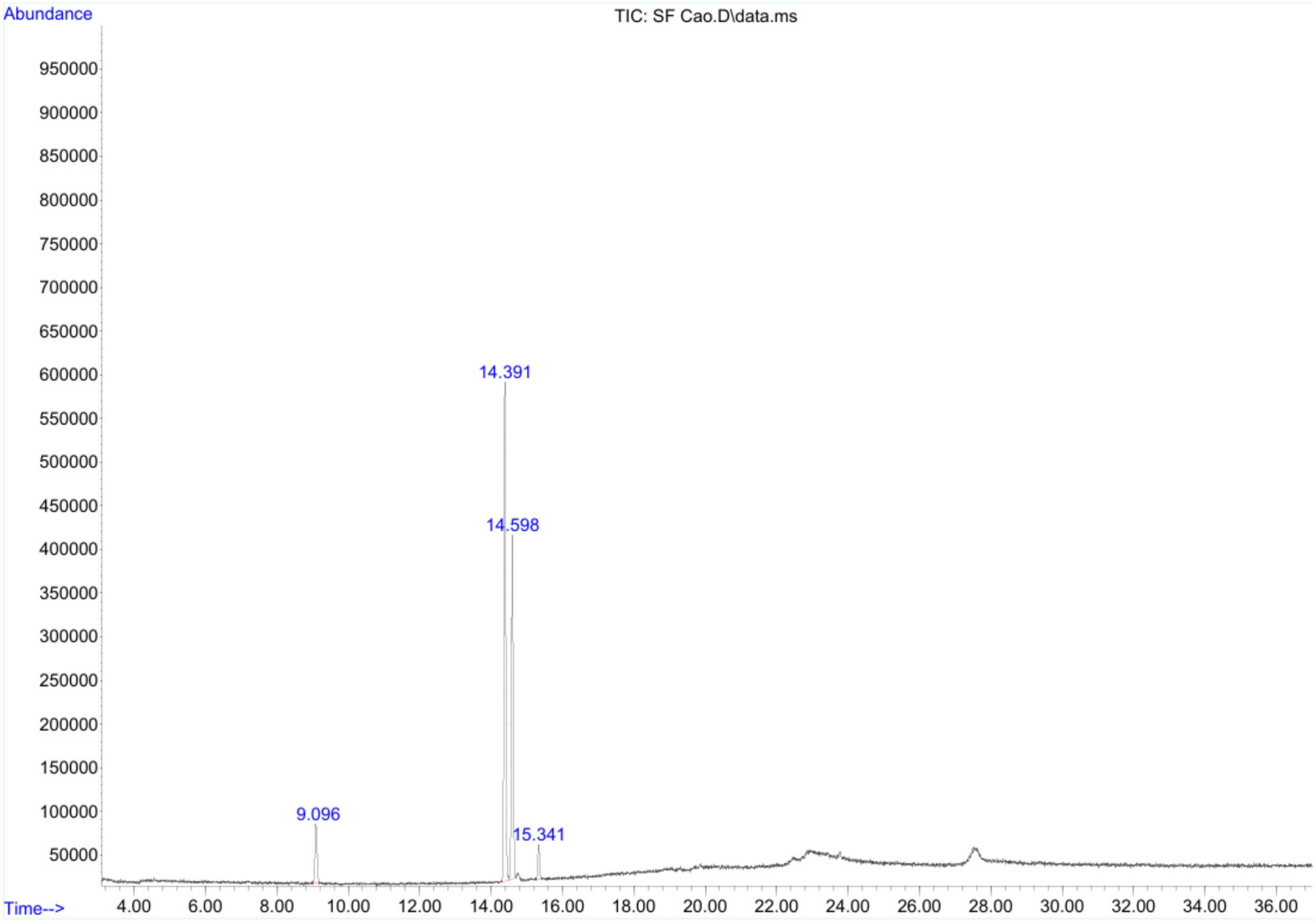
A comparative evaluation of the chromatograms reveals that biodiesel from sunflower oil exhibited cleaner spectra with fewer secondary peaks, indicating a higher degree of conversion. In contrast, the TICs for waste cooking oil biodiesel exhibited additional peaks in the 18–28 min range, suggesting the presence of longer-chain esters or minor impurities. The catalysts synthesised via solar microwave-assisted methods resulted in higher peak intensities for major FAMEs, aligning with the increased biodiesel yields observed in the literature. The differences in peak intensities and retention shifts highlight the impact of catalyst composition and synthesis methods on biodiesel quality. The improved performance of CaO/MgAl catalysts synthesised via solar microwave heating can be attributed to their enhanced surface area and catalytic efficiency, leading to more complete transesterification reactions.

Table 3 The biodiesel yield for sunflower and waste cooking oil transesterification using solar microwave-assisted and conventional synthesis catalysts

Catalysts	Type of Oil	Biodiesel Yield (%)
[a]CaO	Sunflower oil	80.16
[a]CaO	Waste cooking oil	69.68
[a]CaO/MgAl	Sunflower oil	84.52
[a]CaO/MgAl	Waste cooking oil	74.36
Solar microwave-assisted [b]CaO	Sunflower oil	85.68
Solar microwave-assisted [b]CaO	Waste cooking oil	88.04
Solar microwave-assisted [b]CaO/MgAl	Sunflower oil	93.20
Solar microwave-assisted [b]CaO/MgAl	Waste cooking oil	89.44

[a] Drying process carried out in an oven at 120°C for 24 hours.

[b] Drying process carried out in a solar microwave reactor at 120°C for 30 minutes.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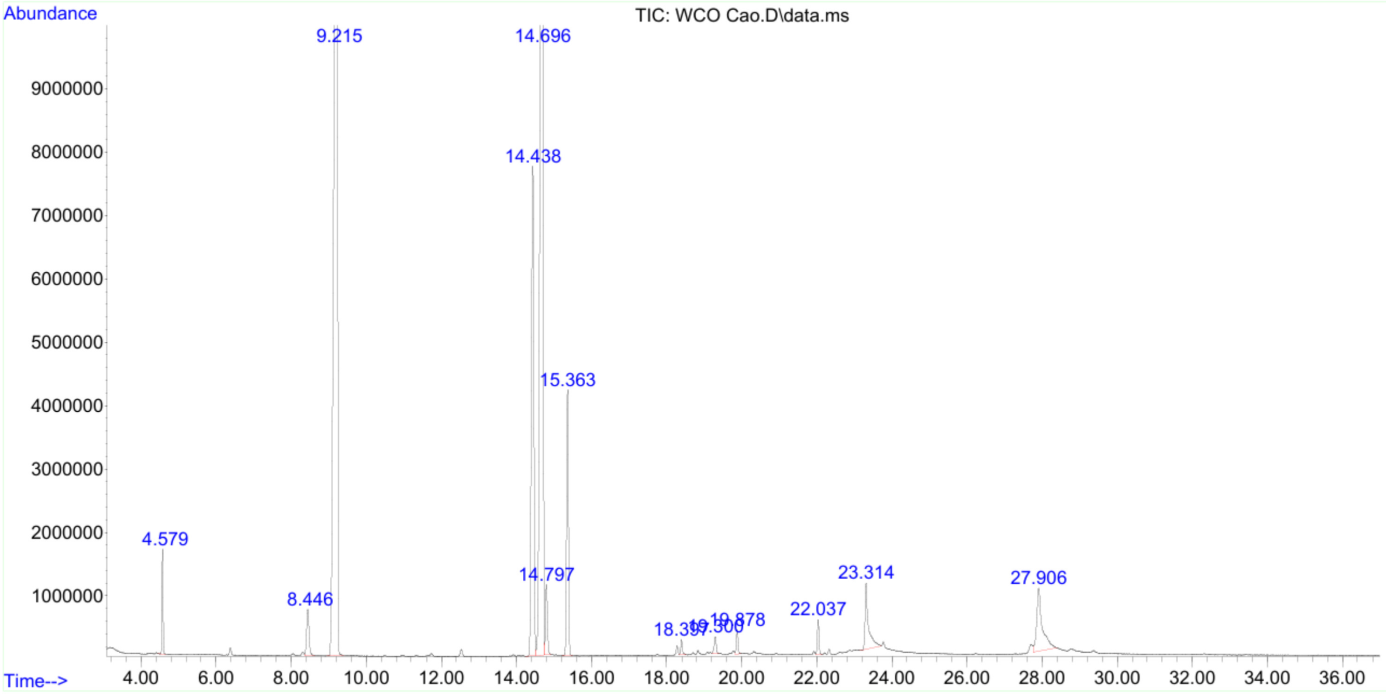
(a)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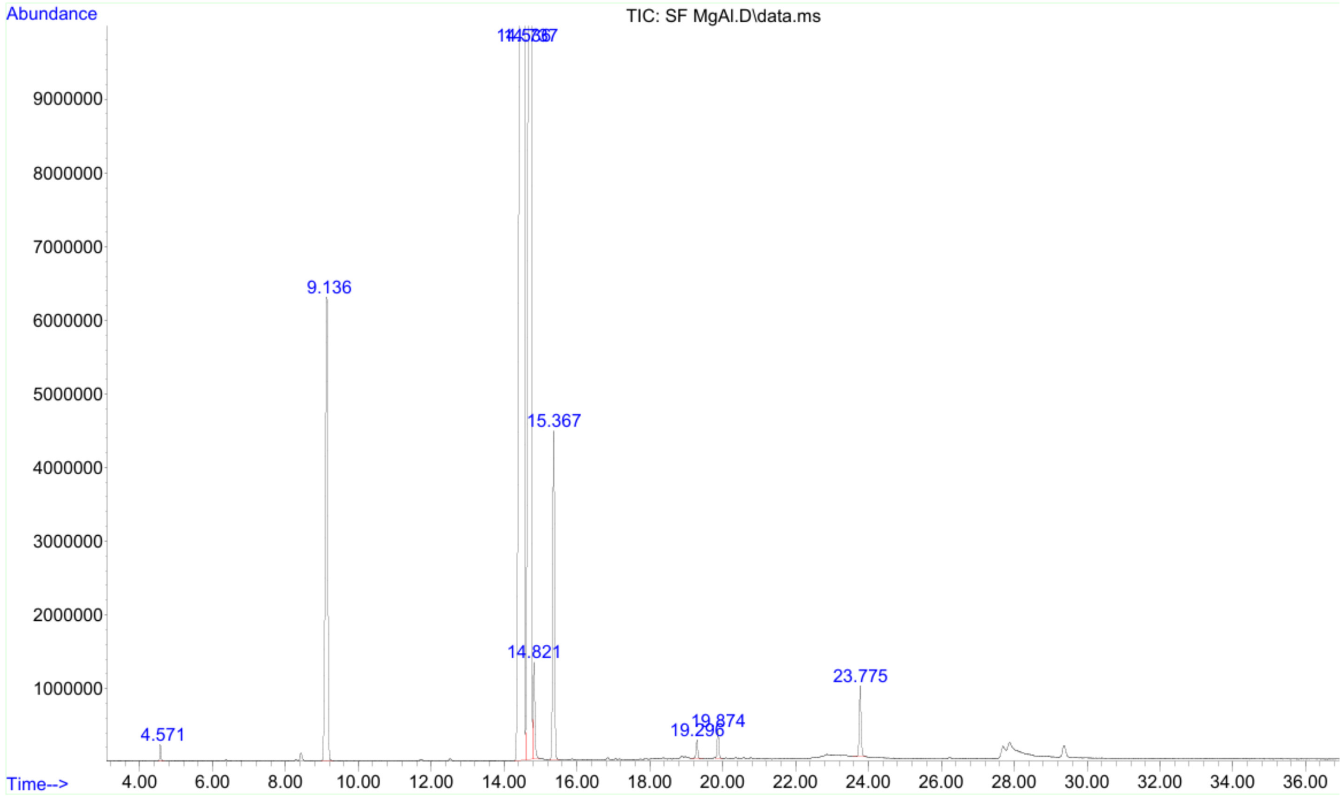

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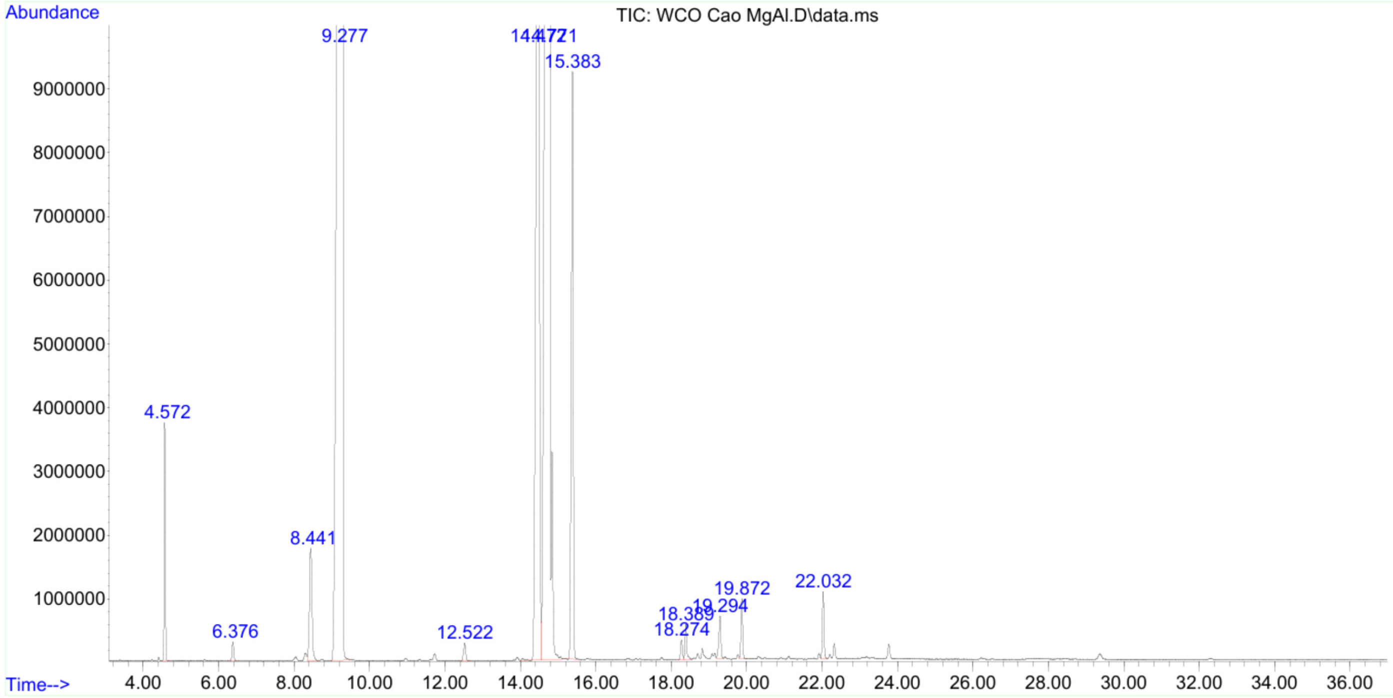

(d)

Figure 4 Total ion chromatogram of (a) ^{b}CaO transesterified sunflower oil, (b) ^{b}CaO transesterified waste cooking oil, (c) ^{b}CaO/MgAl transesterified sunflower oil, and (d) ^{b}CaO/MgAl waste cooking oil, biodiesel showing the composition of fatty acid methyl ester

Drying Cost and Energy Consumption Analysis

This study demonstrated that solar microwave technology significantly reduces the drying time of eggshells and MgAl hydrotalcite samples, achieving complete drying within 30 minutes using a 900 W solar microwave reactor. In contrast, conventional oven-assisted drying at 1300 W required 24 hours for the same process [7],[9]. While these two methods differ in power rating and drying mechanism, the comparison is based on their ability to achieve a similar final drying state, ensuring that the moisture content of the materials was sufficiently reduced for catalyst synthesis.

The basis for this comparison lies in the fundamental differences between microwave and conventional drying mechanisms. Conventional drying relies on conductive and convective heat transfer, gradually removing moisture through surface evaporation, leading to longer processing times and higher energy consumption. Conversely, solar microwave drying employs dielectric heating, where microwaves penetrate the material and generate heat internally, allowing for rapid moisture removal and significantly shorter drying durations. The solar microwave-assisted drying method further enhances efficiency by utilising solar-generated electricity, eliminating direct energy costs.

The impact of these drying methods was interpreted based on energy consumption, and cost analyses were performed. Table 4 compares directly, considering the total energy consumed (kWh). Despite the conventional drying system having a higher power rating (1300 W), its prolonged duration (24 hours) resulted in an energy consumption of 62.40 kWh, leading to an estimated drying cost of MYR 22.78 based on an electricity tariff of 0.365 MYR/kWh [24]. In contrast, solar microwave drying consumed only 0.90 kWh, eliminating electricity costs. This resulted in a 100% reduction in drying costs, demonstrating the economic and environmental advantages of solar microwave technology. Thus, while the two methods differ in operational parameters, the comparison remains valid in the context of achieving complete drying and evaluating energy efficiency. The findings emphasise the sustainability and cost-effectiveness of solar microwave drying, highlighting its potential for scaling up industrial catalyst synthesis while reducing energy dependency and carbon footprint.

Table 4 Comparison between solar microwave and conventional drying for CaO/MgAl synthesis

	Solar Microwave	Conventional
Reactor power (W)	900	1300
Eggshell drying time (min)	30	1440
HT Mg-Al drying time (min)	30	1440
Energy consumption (kWh)	0.90	62.40
Electricity cost (MYR/kWh)	0.000	0.365
Total drying cost (MYR)	0.00	22.78
Drying cost reduction (%)	100.00	0.00

CONCLUSION

This study successfully developed highly efficient modified alkaline catalysts (CaO/MgAl) from waste materials using an innovative solar microwave-assisted synthesis method. The proposed method significantly reduced synthesis time from 24 hours to 30 minutes while consuming only 0.9 kWh of energy, compared to 62.4 kWh in conventional oven-assisted drying. This results in a 100% reduction in drying costs when utilising solar energy. Furthermore, the solar microwave-assisted catalysts exhibited superior catalytic activity, achieving 93.2% fatty acid methyl ester (FAME) yield for sunflower oil and 89.4% for waste cooking oil. In contrast, catalysts synthesised via conventional methods yielded 85.7% and 88.0%, respectively, highlighting the enhanced efficiency of solar microwave-assisted synthesis. Compared to other heterogeneous catalysts reported in the literature, such as CaO derived from eggshells (yield ~80%) and Mg-Al hydrotalcite (yield ~85%), the synthesised catalysts demonstrated improved performance. The solar microwave reactor's rapid heating and uniform energy distribution likely contributed to higher catalyst reactivity and biodiesel yield. The findings of this study demonstrate that solar microwave-assisted synthesis is an eco-friendly, cost-effective, and highly efficient approach to catalyst preparation. The significant reduction in energy consumption, reaction time, and production cost highlights its potential for industrial-scale biodiesel production, aligning with global sustainability goals.

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