

Effect of Calcination Temperature on the Structural and Catalytic Efficiency of APSR-Derived CaO for Biodiesel Production

Abstract

This study aimed to investigate the effect of varying the calcination temperature of calcium oxide (CaO) based biocatalysts for biodiesel production from low-cost feedstock (African Periwinkle Shell Residue, APSR). The synthesized APSR-derived biocatalyst was characterized through Energy Dispersive X-ray spectroscopy (EDX), Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD), and Fourier Transform Infrared spectra (FTIR). The catalytic performance was examined via catalyzed transesterification of soybean oil. The results indicate that the mass fraction of the main element (Calcium, Ca) present in the APSR, increased with temperature. Likewise, the morphological composition and percentage composition of crystalline phases (CaO, SiO₂ and calcite) of the derived biocatalyst increased with the increase in calcination temperature. Catalytic testing showed that the biodegradation of triglycerides in soybean oil was able to aid the conversion of oleic acid to biodiesel. The results of this study suggest that the composition and efficiency of CaO are dependent on the calcination temperature.

Keywords: Calcination temperature, African periwinkle shell, characterization, catalytic efficiency, biodiesel yield

1. Introduction

Growing concerns about environmental pollution, fuel security and sustainability have driven immense interest in biofuel as a renewable alternative fuel (Jekayinfa et al., 2020). Biodiesel is a non-toxic, biodegradable fuel that can be produced from plant oils and animal fats through processes such as supercritical methanol, micro-emulsion, and catalytic transesterification (Adila Fazliyana Aili Hamzah et al., 2021; Oloyede et al., 2022). However, transesterification is the most widely used method (Ogunkule et al., 2024; Oloyede et al., 2024). In the transesterification reaction, triglycerides in the oil react with an alcohol like methanol or ethanol in the presence of a catalyst to form fatty acid alkyl esters, commonly known as biodiesel, and glycerol as a byproduct (Pathak et al., 2018; Orisaleye et al., 2019). Calcium oxide (CaO) is a promising heterogeneous base catalyst that has been extensively studied for biodiesel production through transesterification from various biomass residues due to its advantages over homogeneous catalysts (Odude et al., 2017). The CaO displays high catalytic activity and reusability. However, its catalytic activity and efficiency can be influenced by factors related to the preparation and treatment of the CaO feedstock (Yin et al., 2016).

A calcination temperature is the exact temperature at which a material is heated, which causes thermal degradation, phase transition, or removal of volatile components and usually results in the formation of oxides and other stable forms (Aleman-Ramirez et al., 2021). In preparation of green heterogeneous catalyst from agricultural residues or waste, calcination of biomaterials over a range of temperature has been an effective approach (Jekayinfa, 2015;

Okoye et al., 2019). The calcination temperature at which CaO is heated during synthesis from agricultural residue source has been shown to significantly impact its surface properties and catalytic behaviour. Previous researchers such as Okoye et al. (2019) and Oloyede et al. (2023) had synthesized CaO from periwinkle shell over a range of temperatures from 300°C to 800°C. Results indicate catalytic activity is affected by calcination temperature up to an optimal point due to improved surface properties like basicity and porosity. However, the specific calcination temperature that maximizes the catalytic efficiency of CaO for biodiesel transesterification has not been definitively established and may depend on reaction parameters. Furthermore, characterizing how calcination temperature influences physicochemical properties like surface area, morphology and crystallinity could provide insights into structure-activity relationships.

This study aimed to systematically investigate the effect of varying the calcination temperature of CaO-based catalysts from 900°C and 1000°C for biodiesel production from low-cost feedstock (African Periwinkle Shell Residue, APSR). The findings seek to determine the optimal calcination temperature for maximizing biodiesel yields and provide knowledge to rationally design CaO biocatalysts with enhanced activity. Optimized heterogeneous catalysts could help advance the commercial viability and scale-up of biodiesel as a renewable transportation fuel. While CaO-based biocatalyst derived from periwinkle shell had shown promise as an effective heterogeneous base catalyst for biodiesel production via transesterification, the factors influencing its catalytic activity and efficiency have not been fully elucidated. In particular, the calcination temperature used in the synthesis of CaO-based biocatalysts may strongly impact the catalyst's physicochemical properties and performance, yet the optimal temperature for maximizing catalytic efficiency remains uncertain. **Determining the calcination temperature that yields a CaO biocatalyst with the highest activity is important for developing an economically viable biodiesel manufacturing process for the biofuel scientific community.** However, the relationship between calcination temperature, catalyst structure and activity has not been thoroughly studied.

2. Materials and methods

2.1 Sample Collection and Preparation

African Periwinkle shell residue (APSR) was sourced for from one of the regions (Ebute-pare, 6°21'N 4°47'E) in Ondo State, because of their abundance in these regions. The residue was washed with water to remove dissimilar materials and then sundried for three days. The sundried residue was oven dried at 100°C until a constant weight was obtained (Fattah et al., 2020; Oloyede et al., 2022). The sample was milled into powder via an attrition mill and sieved using a sieve with aperture size of 150 µm in laboratory mechanical shaker machine. The sieved sample was kept in air tight container for further analysis (Ogunkunle et al., 2017; Falowo et al., 2019).

2.2 Synthesis of CaO biocatalyst from APSR

The CaO was synthesized from APSR via calcination of 200 g each of a milled APSR sample at two different calcination temperatures of 900 and 1000 °C, respectively in a muffle furnace (Fig. 1a) at heating rate of 10 °C/min with retention time of 4 hours. This is to remove carbonaceous and volatile matter from the milled APSR and obtain a fine ash (CaO). The calcined ash sample (Fig. 1b) was kept in a desiccator and then in Ziploc bag (an air tight bag) for further studies (Oladipo & Betiku, 2020; Oloyede et al., 2023).



Figure 1: (a) Smokeless Muffle furnace (b) Synthesized CaO biocatalyst in ziploc bags.

2.3 Characterization of APSR Derived CaO Biocatalyst

The synthesized APSR derived CaO biocatalyst at the two varying calcination temperatures were characterized through Energy Dispersive X-ray spectroscopy (EDX), Scanning electron microscopy (SEM), X-ray diffraction analysis (XRD), Thermogravimetric analysis (TGA), and Fourier Transform Infrared spectroscopy (FTIR). These were done to test for the elemental composition, morphology, crystalline structures, thermal stability, and active surface functional group presents in the catalyst sample follow the procedure reported by Betiku et al. (2017) and Etim et al. (2018). The SEM-EDX was done using scanning electron microscope instrument (JEOL JSM-7600F, Japan) with a Gatan alto 2500 Cryo system. Rigaku Miniflex 600 (Miniflex, Japan) was used to determine the crystalline structure of each catalyst. The XRD was measured over Cu-K α_2 radiation at a wavelength of 1.5444 Å, 45 kV voltage and 40 mA current. The samples were scanned at a speed of 5°/min in the 2 θ range of 5 – 76°. The catalyst active surface functional were determined using FTIR spectrophotometer (Thermo-Nicolet iS10) measured in the wavelength range of 4000-400 cm^{-1} . The FTIR spectral of the samples were obtained in the wavelength range of 4000-400 cm^{-1} . The FTIR samples preparation was done via KBr pelleting method. The catalyst characterization was carried out at the National Research Institute for Chemical Technology (NARICT) laboratory, Zaria, Nigeria. The GC-MS analysis of the biodiesel produced using APSR derived CaO biocatalyst at optimal temperature to determine the composition of fatty acid methyl esters present in it was run using using a SRI 910 Gas Chromatography (Buck

Scientific Instruments, Norwalk, CT, USA) with 230 VAC \pm 10%, a 1725W power rating, and using Helium (He) as a carrier gas.

2.4 Catalytic performance of the APSR derived CaO Biocatalyst

The catalyzed transesterification of soybean oil was used to examine the performance of the APSR derived CaO biocatalysts. The transesterification of the oil was conducted in a three necked round bottom flask of 500 ml equipped with a reflux condenser, heating source controller, thermometer, and magnetic stirrer (Fig.2a) The transesterification procedure was carried out under the reaction condition with a methanol-to-oil ratio at 1:6, catalyst concentration at 7.0 wt.%, reaction temperature at 60 °C and reaction time of 60 minutes at an agitation of 750 rpm. At the end of the reaction, the catalyst was recovered through filtration using Wartman filter paper (100 microns)(Ropelewska et al., 2018; Oloyede et al., 2023). The mixture was then poured into a separating funnel and allowed to separate into two distinct layers, with the top layer being the biodiesel and the lower layer being glycerol (Fig.2b).



Figure 2. (a) Transesterification set-up, (b) Biodiesel mixture

2.5 Determination of Biodiesel Yield

The biodiesel yield obtained from the catalytic testing of the APSR derived CaO biocatalysts was calculated using equation 1(Akhabue & Okwundu, 2019).

$$B_y (\%) = \frac{W_b}{W_o} \times 100 \quad 1$$

Where: B_y = Biodiesel yield

W_b = Weight of biodiesel produced

W_o = weight of oil used

2.6 Data Analysis

The particles in the SEM micrographs were analyzed using Imagej software and the data obtained were statistically analyzed using frequency distribution in Origin software (Version 2021) to show the occurrence of particle sizes in relation with calcination temperatures. The graphs were also drawn using Origin software.

3. Results and Discussions

3.1 Effect of calcination temperatures on elemental composition of APSR derived CaO biocatalyst

The effect of calcination temperatures (900 and 1000 °C) on the elemental composition of the CaO-based biocatalyst is presented in Fig. 3. It was observed from the Figure that as calcination temperature increased, the percentage mass of calcium (Ca), increases. The mass percentage of Ca increased from 94.31 to 95.50 wt. % at 900 and 1000 °C, respectively. The Figure also detailed that the raw sample of APSR contained 92.23 wt. % of Ca. The increase in percentage mass of Ca, in the calcined ash sample (CaO derived catalyst) of APSR from 94.31 to 95.50 wt. % could be attributed to the removal of organic matter and volatile impurities during the calcination process, leading to a higher concentration of calcium in the remaining material. Okoye et al. (2020) stated that at higher calcination temperatures, there might be enhanced decomposition of organic components, further concentrating the Ca content in the sample. The other metallic element such as Sodium (Na), Magnesium (Mg), and Aluminum (Al) were present as trace elements. Thus, shown that Ca is the main metal present in the calcined APSR. Oloyede et al. (2022) reported similar percentage mass of 60.60 wt. % for calcined African periwinkle shell derived ash catalyst at 800 °C. Thus, for a complete thermal decomposition of calcite (CaCO_3) present in APSR to obtain a pure green CaO, scientists should focus on temperature range of 1000 °C and above.

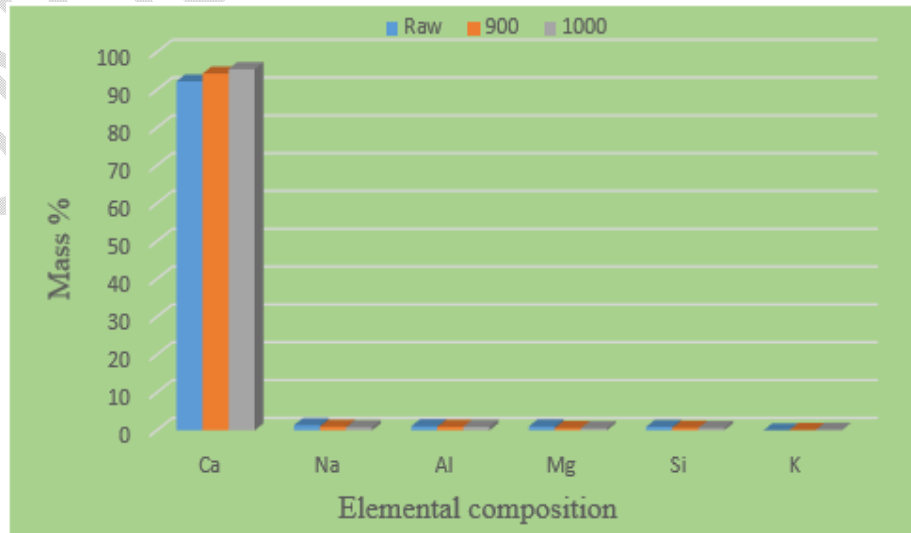
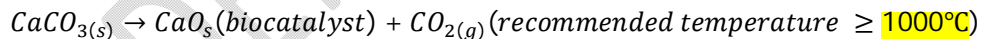


Figure 3: Effect of calcination temperature on elemental composition of raw and calcined ash samples of APSR.

3.2 Effect of calcination temperature on morphological analysis of APSR derived CaO biocatalyst

The effect of calcination temperature on morphological character of the synthesized CaO-based biocatalyst was explained using the **particle sizes measure via Imagej software and their nature**. The relationship between the calcination temperature and the size of the CaO-based biocatalyst is statistically given in Figures 4. It was observed from the Figures that the particle size in term of length ranged between 0-100 μm is higher at calcination temperature of 1000 $^{\circ}\text{C}$ than at 900 $^{\circ}\text{C}$. At both temperatures, the occurrence of the particle sizes ranged between 100-200 μm are similar. While no particle size observed for between 200-500 μm , however, particles having size of 500 μm at frequency between 0-1.0 occurred at 900 $^{\circ}\text{C}$. This implies that the particle size in micrometer changes with increased in temperature. Also, the particles area as observed in Fig. 5 increased with increased in temperature. The occurrence of the particle area decreased from 58 to 3 with corresponding particle area of 0 to 1000 μm^2 as calcination temperature increased from 900 to 1000 $^{\circ}\text{C}$. At frequency of 2.0, the constant particle area between 2000 to 4000 μm^2 was observed in the CaO-based biocatalyst. It was similarly observed that the morphology at both temperatures exhibits a microporous nature (Fig 6). The micrographs (Fig 6) of the APSR derived CaO biocatalyst shown an increase in both the quantity and size of pores on the calcined APSR. Their physical structure presented greater clumping together; the presence of additional openings on the APSR particles was noticed in CaO catalyst which indicates that it possesses a larger surface area (Nwosu-Obieogu et al., 2024).

Moreover, increasing the calcination temperature to 1000 $^{\circ}\text{C}$ further enhances the particle area of the biocatalyst. Thus, elevating the calcination temperature within the examined range results in a more developed porous network structure and enhanced microporosity or crystallinity in the resulting CaO-based biocatalyst. Nath et al. (2019) reported similar morphological observation for *Brassica nigra* derived biocatalyst for transesterification of soybean oil which showed particle agglomeration and the presence of layered spongy stringy microstructures in the derived catalyst.

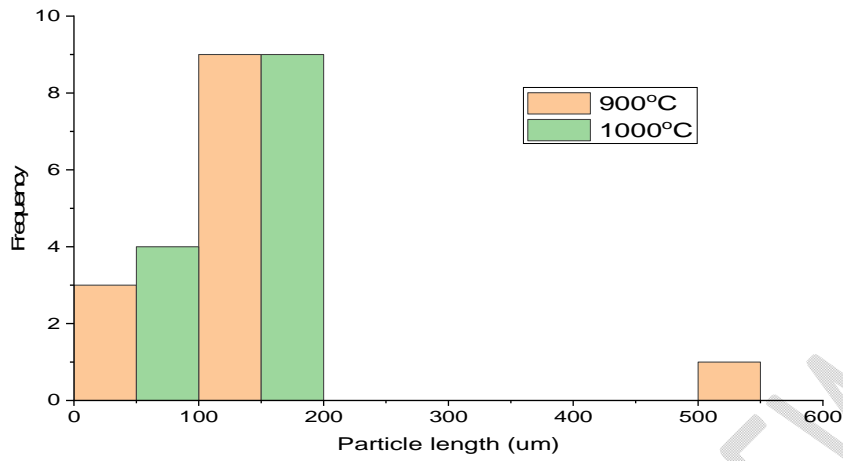


Figure 4: Effect of calcination temperature on particle length.

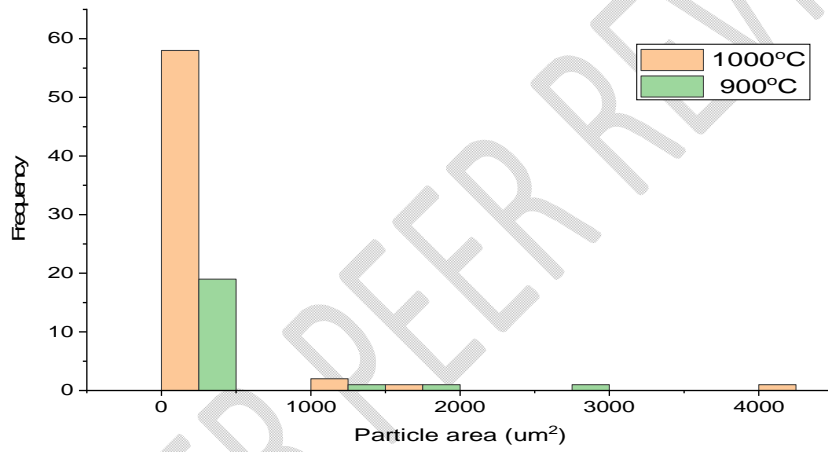


Figure 5: Effect of calcination temperature on particle area.

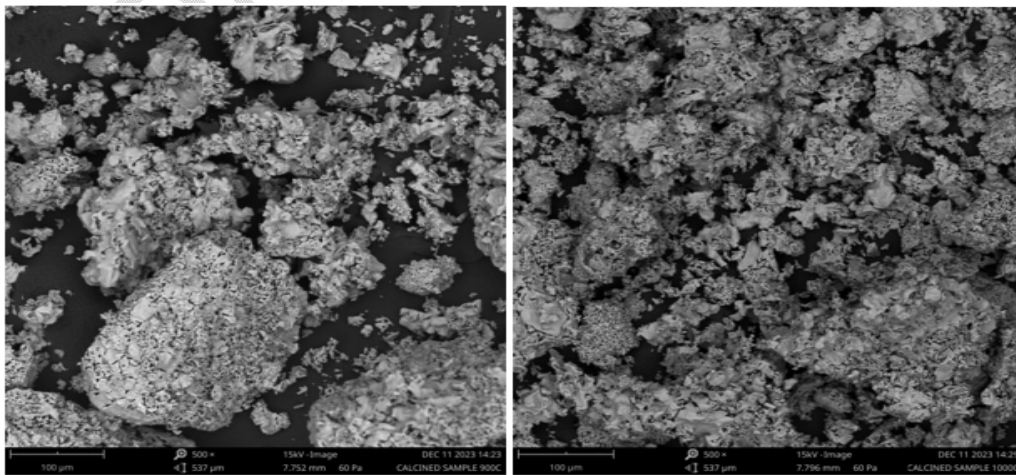


Figure 6: SEM micrographs of APSR derived CaO at 900 and 1000°C at 500 ×

3.3 Effect of calcination temperature on crystalline phase of APSR derived CaO-based biocatalyst

The Figure 7 presented the effect of calcination temperature on crystalline structures of the APSR derived CaO biocatalyst. As shown from the figure, the crystalline phases of CaO (lime) present in the derived catalyst (CaO) increased with the increased in calcination temperatures. The percentage occurrence (88.3%) of the CaO phase was higher at 1000 °C than at 900 °C which has percentage occurrence of 85% for CaO. This implies that increase in calcination temperature led to higher crystalline phase of CaO in the calcined APSR. However, the histogram shown that there is no much significant difference between the two temperatures. The crystalline phases of aragonite ($CaCO_3$ and SiO_2) present in the derived catalyst were also affected by calcination temperature. The $CaCO_3$ and SiO_2 decreased with an increased in temperature with percentage occurrence of 2 and 13.2% at 900 °C and 1.2 and 10.5% at 1000 °C. This shown that crystalline structure or phases in the APSR derived CaO is temperature dependent which should be given a prior consideration by the scientist in while developing CaO from APSR. Oloyede et al. (2022) obtained similar composition of phases for APS calcined as 800 °C with percentage occurrence of 61.1%. Suryaputra et al. (2013) reported similar phases of CaO and calcite in Capiz shell calcined at 900 °C. This shows that the crystalline structure of the derived CaO biocatalyst is temperature dependent.

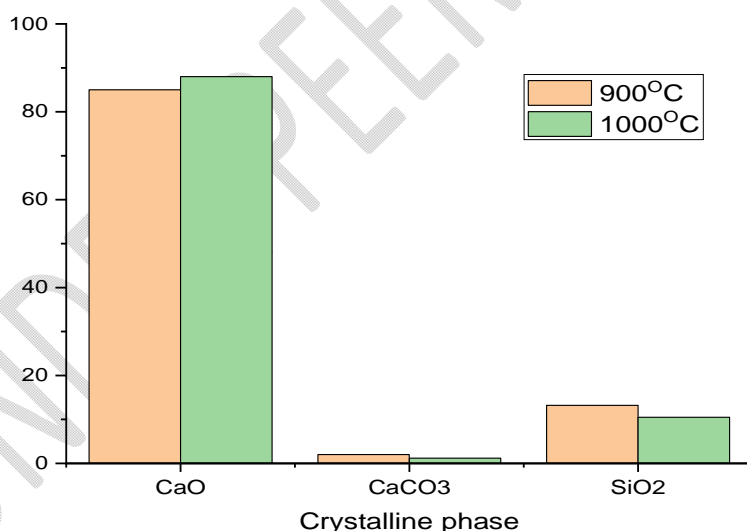


Figure 7: Effect of calcination temperature on crystalline phase of CaO-based biocatalyst.

3.4 Effect of calcination temperature on functional group present in APSR derived CaO-based biocatalyst

The FTIR pattern of the derived CaO-based biocatalyst at calcination temperature of 900 and 1000 °C were presented in Fig. 8a-b, respectively. The strong peak at 3041.0cm^{-1} for the two temperatures revealed band that represent the C=O group in CO_3^{2-} and the peak

might be due to OH in $Ca(OH)_2$ which formed during adsorption of H_2O by CaO (Kirubakaran & Arul Mozhi Selvan, 2018; Goli & Sahu, 2018). Boonyuen *et al.* (2018) also identified the presence of C=O group in the raw sample of *Turbo jordani* shells. The other characteristics absorption peaks positioned at 2974.4, 2117.1, 1408.9, 1066.0, and 875.9 cm^{-1} represent the stretching vibrations of C-O group also in CO_3^{2-} and Ca-O which suggests the presence of CaO and aragonite. This implies that calcination temperature did not significantly affect the position of functional group present in the synthesized biocatalyst. Oloyede *et al.* (2022) also reported similar stretching vibrations for calcined APSR at 800°C for peaks at 721.05, 897.35 and 1600.65 cm^{-1} that are due to the stretching vibrations of Ca-O and Na-O bonds suggesting the presence of CaO and Na_2O in the calcined ash sample derived catalyst. Similar observation was detailed by Nath *et al.* (2019) for the calcined *Brassica nigra* ash catalyst which shown the stretching vibration of Ca-O and K-O bonds at infrared peak of 624 cm^{-1} .

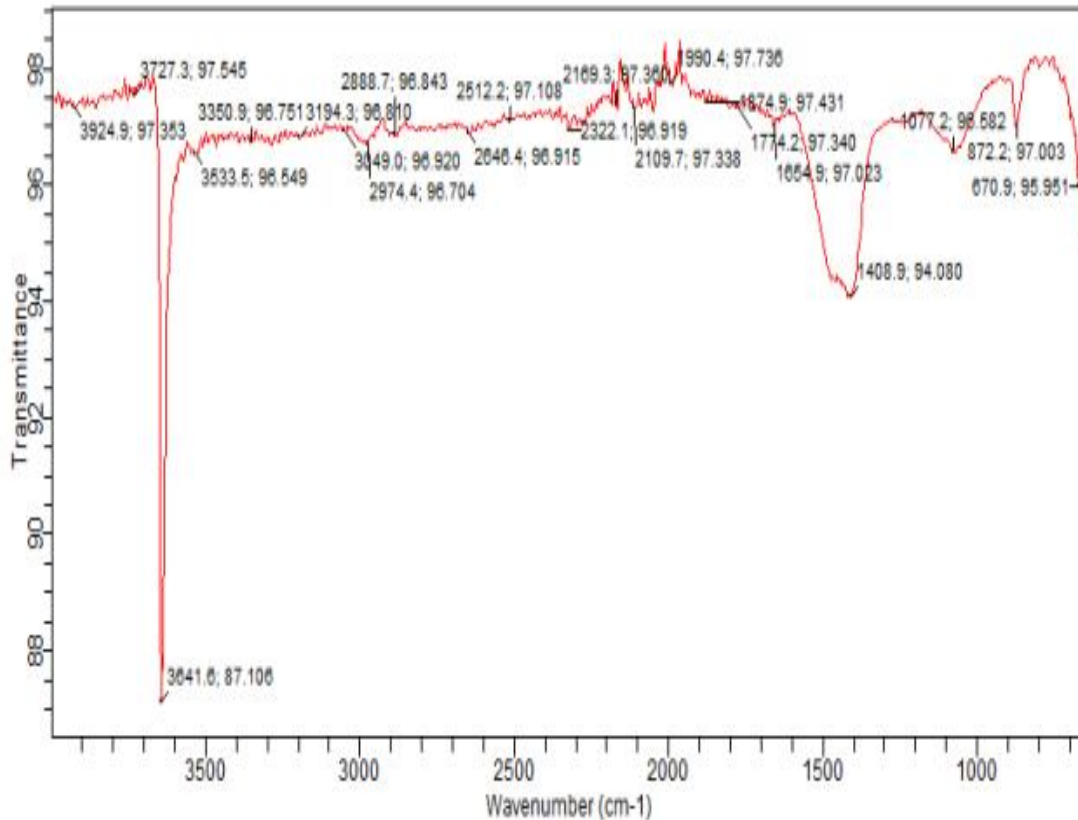


Figure 8a: The FTIR spectra of derived APSR CaO biocatalyst at 900°C .

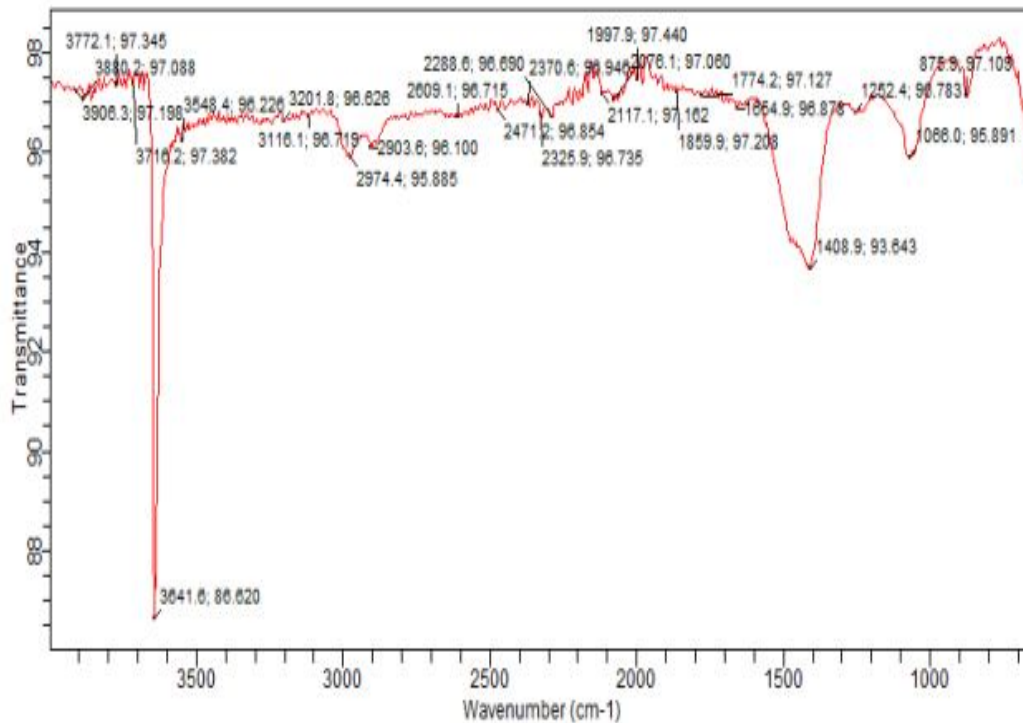


Figure 8b: The FTIR spectra of APSR derived CaO biocatalyst at 1000□.

3.5 Effect of calcination temperature on catalytic efficiency of synthesized CaO-based biocatalyst

The catalytic performance of the APSR derived CaO biocatalyst at the calcination temperature (900 and 1000□), was examined through transesterification of soybean oil. It was observed that the APSR derived biocatalyst was able to aid the conversion of triglycerides in soybean oil to biodiesel (Fig2). The biodiesel yield was 92.4 and 95.2 wt.% at 900 and 1000□, respectively. This shows that the biodiesel yield increased with an increase in calcination temperature. The increase in biodiesel yield may be as a result of the percentage occurrence of CaO phase in the APSR derived CaO biocatalyst as contained in XRD at temperature of 1000□ (Sandesh & Ujwal, 2021). The yield of biodiesel at the two temperatures are greater than the reported yield by Okoye et al. (2019) when calcined periwinkle shell at 500□ was used to produce biodiesel. Comparing the biodiesel yield obtained using APSR derived CaO biocatalyst with the one produced using KOH (homogeneous catalyst) which has a yield of 96.3 wt.%. It was observed that there is no much significant difference between the yields of biodiesel produced using synthesized CaO biocatalyst and that of KOH.

3.6 GC-MS analysis of the biodiesel produced using APSR CaO derived biocatalyst

The conversion of triglyceride of Soya bean oil into methyl ester using the derived APSR CaO is presented in Figure 9. The highest peaks (74, 87) in the figure indicate the presence of methyl esters. The lower peaks (101-228) to the right indicate the presence of mono-, di- and tri-glyceride which suggest that the triglycerides were converted successfully

into biodiesel. Thus, causing a reduction in their level to form biodiesel (Nwosu-Obieogu et al., 2024; Falowo et al., 2019)

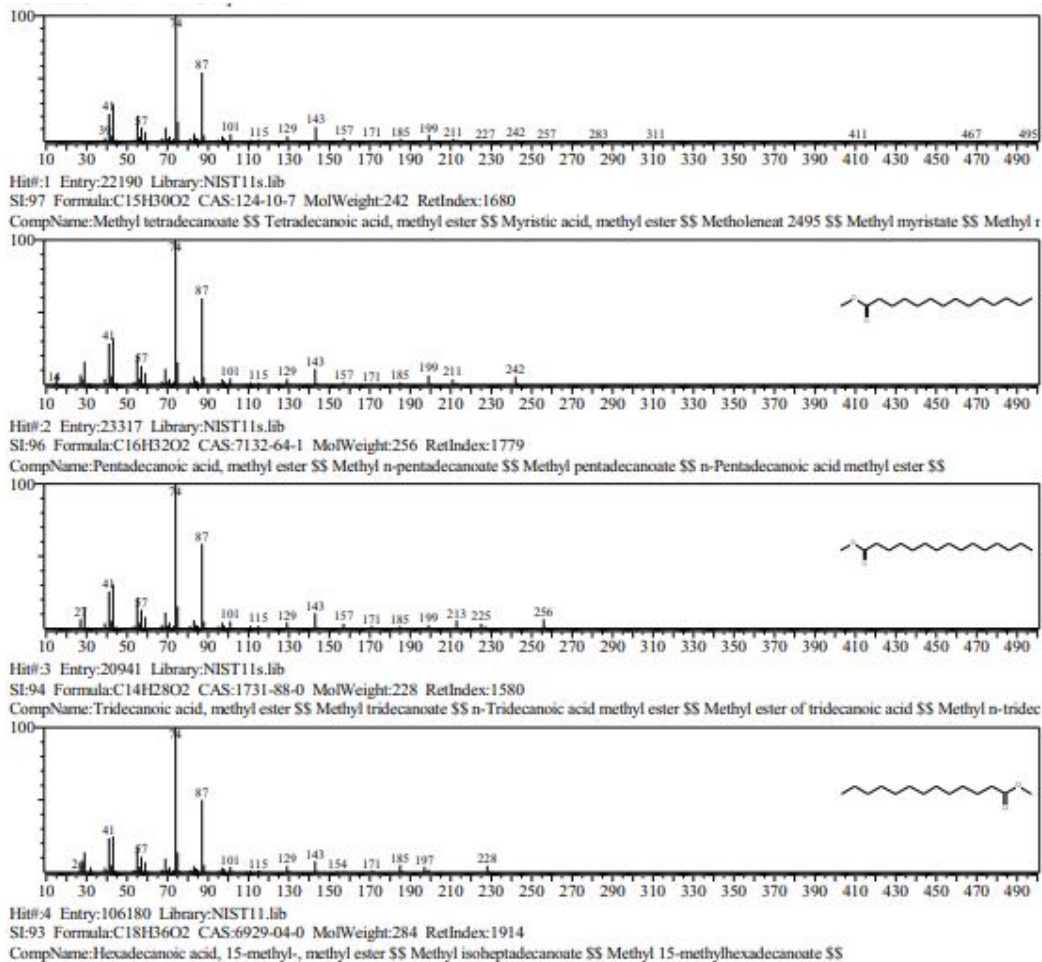


Figure 9: The GC-MS spectral of soya bean oil biodiesel using APSR derived CaO biocatalyst

4. Conclusion

This study examined the effect of calcination temperatures on characterization of synthesized CaO-based biocatalyst from African periwinkle shell residue for biofuel production. The following conclusions were drawn, based on the experimental results:

- i. The increase in calcination temperature affects the mass fraction of the elemental composition present in the APSR derived CaO-based biocatalyst and Ca being the major element present in the sample. Likewise, the biocatalyst morphological composition, and crystalline phases, were affected with increase in calcination temperature. However, the calcination temperature has no effect on the functional group present in the synthesized biocatalyst.
- ii. Catalytic testing showed the synthesized APSR CaO biocatalyst effectively convert soybean oil to biodiesel via catalysed transesterification. The biodiesel yield

increased with increased in calcination temperature. The GC-MS spectral confirmed the conversion of triglycerides in soya bean oil to biodiesel.

- iii. Hence, the study concludes that the composition and efficiency CaO derived biocatalyst is dependent on the synthesized calcination temperature. Likewise, to develop a green CaO for APSR, an optimum calcination temperature of 1000 °C is recommended. Moreover, further studies on the stability of APSR CaO derived biocatalyst is also recommended.

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Details of the AI usage are given below:

1. for correcting the grammatical structure of the manuscript, and
2. for abstract suggestion.

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