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Performance Enhancement of Cuttlefish Bone – Derived CaO Catalyst via SrO Impregnation and NaOH Activation for Biodiesel Synthesis from Waste Cooking Oil

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Abstract

This study investigates the conversion of waste cooking oil (WCO) into biodiesel using a heterogeneous CaO-based catalyst derived from cuttlefish bone waste. To enhance catalytic performance, the CaO catalyst was modified through SrO impregnation at different compositions (98:2, 95:5, and 90:10 wt.%) and subsequent NaOH chemical activation (2, 4, and 6 g). The physicochemical properties of the synthesized CaO–SrO catalysts were characterized using SEM– EDS, XRD, and FTIR to evaluate surface morphology, elemental dispersion, and crystalline phase stability. The results demonstrate that SrO impregnation and NaOH activation significantly influence both catalyst structure and transesterification performance. Among the tested catalysts, the catalyst sample with a 90:10 CaO–SrO ratio and activated with 6 g NaOH (C90S10–N6) exhibited the highest biodiesel yield of 95.5% under a methanol-to-oil molar ratio of 9:1, catalyst loading of 1 wt.%, and reaction time of 2 h. Although catalysts with moderate SrO loading showed more homogeneous morphology, a higher SrO content combined with a stronger chemical activation resulted in superior catalytic performance, indicating that catalytic basicity plays a more dominant role than the morphological uniformity in achieving biodiesel yield. The produced biodiesel met key quality parameters of the Indonesian Standard for biodiesel, with a density of 0.87 g/mL, moisture content of 0.64%, a calorific value of 38.52 MJ/kg, and a reduction in free fatty acid content from 2.84% to 1.71%. These findings highlight the potential of synergistically modified CaO–SrO catalysts derived from cuttlefish bone waste as an effective and sustainable option for biodiesel production from low-cost feedstocks.

Keywords: Waste cooking oil (WCO); Cuttlefish bone-derived CaO; Catalyst; SrO; Biodiesel.

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INTRODUCTION

The increasing global dependence on fossil fuel – based energy has raised serious concerns related to resource depletion, price volatility, and environmental quality decline. Fossil fuels, including oil, natural gas, and coal, still account for more than 80% of the global energy consumption and are the primary contributors to greenhouse gas emissions (Osman *et al.*, 2024). In parallel with population growth and industrial expansion, global energy demand is projected to increase by nearly 50% by 2050, intensifying the urgency to develop sustainable and low–carbon energy alternatives (Kouider Elouahed et al., 2024). In this context, biodiesel has emerged as a promising renewable fuel due to its biodegradability, non-toxicity, and compatibility with the existing diesel engines (Asri *et al.*, 2016).

The feasibility and sustainability of biodiesel production are strongly influenced by the selection of feedstocks. In Indonesia, biodiesel is predominantly synthesized from refined vegetable oils, particularly palm oil, due to its low free fatty acid (FFA) content and simple processing. However, the high cost of refined oils significantly increases biodiesel production expenses, limiting its economic competitiveness with fossil diesel. To overcome this drawback, waste cooking oil (WCO) has attracted considerable attention as an alternative feedstock for its low cost, wide availability, and potential to mitigate environmental pollution (Asri *et al.*, 2017). The annual global generation of WCO reaches more than 16.5 million tons, and improper disposal of this waste possesses serious environmental and public health risks (Hosseinzadeh-Bandbafha et al., 2022). Therefore, converting WCO into biodiesel offers a dual benefit by addressing both energy security and waste management challenges.

Efficient conversion of WCO into biodiesel via transesterification requires the assistance of an effective catalyst. Among heterogeneous base catalysts, calcium oxide (CaO) is one of the most widely studied materials due to its strong basicity, low solubility in methanol, and ease of separation from reaction products (Xia *et al.*, 2024). CaO is generally produced by calcination of calcium carbonate ($CaCO_3$) – rich materials at temperatures above 800 °C (Ricinus *et al.*, 2024). In recent years, biogenic wastes, such as eggshells, fish bones, and cuttlefish bones have been explored as alternative CaO sources in order to reduce catalyst cost and improve sustainability (Herpandi *et al.*, 2022). Cuttlefish bone, a by-product of seafood processing, is particularly attractive due to its high $CaCO_3$ content and porous structure, making it a promising precursor for CaO-based catalysts.

Despite these advantages, CaO catalysts derived from biogenic waste often suffer from limitations such as low surface area, poor dispersion of active sites, and susceptibility to deactivation caused by carbonation and moisture adsorption. To address these shortcomings, various modification strategies have been proposed, including impregnation in alkaline or alkaline - earth metal oxide solutions. Among these, strontium oxide (SrO) has been reported to enhance the basic strength, stability, and catalytic activity of CaO-based catalysts (Sulaiman *et al.*, 2021). Previous studies have demonstrated that SrO-modified CaO catalysts derived from eggshells or cuttlebone can improve biodiesel yields by increasing the density of strong basic sites (Tomano *et al.*, 2020; Zhang *et al.*, 2021).

In addition to metal oxide modification, chemical activation using alkali compounds, such as NaOH has been shown to further enhance the catalytic performance of CaO-based catalysts through the increase in accessibility and number of basic surface sites (Hawa *et al.*, 2020). Chinglenthoiba *et al.* (2020) reported that the use of NaOH-loaded CaO catalysts derived from fish bones achieved biodiesel yields as high as 98% when applied to WCO transesterification. However, most existing studies have focused on either metal oxide impregnation or chemical activation as individual modification strategies. The combined or synergistic effect of SrO impregnation and NaOH activation, particularly on CaO catalysts derived from cuttlefish bone waste, has yet to be systematically investigated.

Moreover, previous research has largely emphasized catalyst characterization or yield improvement independently, with limited discussion on the direct relationship between catalyst structure such as surface morphology, elemental dispersion, and crystalline phase stability and actual transesterification performance. As a result, the dominant factors governing catalytic activity, especially the relative importance of basicity versus morphological homogeneity, remain poorly understood.

Therefore, this study aims to investigate the effect of SrO impregnation combined with NaOH chemical activation on CaO catalysts derived from cuttlefish bone waste for biodiesel production from waste cooking oil. The novelty of this work lies in (i) the synergistic modification strategy integrating SrO impregnation and NaOH activation, and (ii) the systematic correlation between catalyst structural characteristics and biodiesel yield. By comparing catalysts with different SrO loadings and activation levels, this study provides new insight into the role of catalytic basicity and phase stability in controlling transesterification performance. The findings are expected to contribute to the development of cost-effective, sustainable, and high-performance heterogeneous catalysts for biodiesel production from low-quality feedstocks.

MATERIAL AND METHODS

Materials

The raw materials used in this study included cuttlefish bone residue obtained from Sumatra as a CaO source; $Sr(NO_3)_2$ (strontium nitrate, for analysis, Merck 107872); waste cooking oil (WCO) collected from Lhokseumawe; activated carbon; ethanol (96%, for analysis); methanol (for analysis, Merck); and sodium hydroxide (NaOH, for analysis, Merck).

Methods

Pretreatment of Waste Cooking Oil (WCO)

Prior to use as the raw material for biodiesel synthesis, the WCO was subjected to a preliminary treatment through an adsorption process using activated carbon. The WCO was first filtered to remove any type of unpreferable residue, followed by heating at 100 – 105°C for 30 min to eliminate moisture content. Then, the dehydrated WCO was transferred into a beaker glass, heated at 100°C and simultaneously stirred at a constant speed of 60 rpm for 1 h. Afterward, the mixture was allowed to cool to a warm temperature, and activated carbon was added under continuous stirring. The adsorption process was subsequently allowed to proceed for 24 h, after which the mixture was filtered to separate the adsorbent from the solution. Finally, the treated WCO was analyzed for its free fatty acid (FFA) content, moisture content, and density.

Characterization of Waste Cooking Oil (WCO)

The pretreated WCO obtained was characterized using standard analytical methods. Free fatty acid (FFA) analysis was performed to evaluate the acidity of the oil as a feedstock, moisture content analysis was conducted to ensure allowable water content, and density measurement was also carried out as a reference.

Catalyst Preparation

The cuttlefish bone residue used in this study was obtained from Sumatra, Indonesia. It was first thoroughly washed with distilled water to remove the adhering impurities, then dried in an electric oven at 105°C for 24 h to eliminate moisture content. After drying, the cuttlefish bone was ground into a fine powder and sieved through an 80-mesh screen to obtain a uniform particle size. Subsequently, the cuttlefish bone powder ($CaCO_3$) was impregnated with strontium nitrate ($Sr(NO_3)_2$) at various ratios (98:2, 95:5, and 90:10 % w/v) for 24 h. Upon the completion of impregnation step, the mixture was then filtered and dried in an oven at 105°C. The dried sample was then calcined in a furnace at 900°C for 4 h to obtain the CaO–SrO catalyst.

The chemical activation process was carried out by mixing the catalyst with NaOH at loadings of 2, 4, and 6 g. The mixture was soaked for 24 h, then filtered and dried in an oven at 105°C to complete the activation process.

Catalyst Characterization

The synthesized CaO–SrO catalysts were characterized using several analytical techniques. Brunauer–Emmett–Teller (BET) analysis was employed to determine the specific surface area, pore size, and pore volume. The crystalline phases were identified using X-ray diffraction (XRD), while Fourier transform infrared (FTIR) spectroscopy was used to identify functional groups (Herpandi *et al.*, 2022) In addition, scanning electron microscopy (SEM) was utilized to analyze the catalyst morphology.

Table 1. Description of Sample Codes

Sample code	Description
C-N6	CaO activated with 6 g NaOH
C98S2-N2	98 g $CaCO_3$ with the addition of 2 g $Sr(NO_3)_2$ activated with 2 g NaOH
C98S2-N4	98 g $CaCO_3$ with the addition of 2 g $Sr(NO_3)_2$, activated with 4 g NaOH
C98S2-N6	98 g $CaCO_3$ with the addition of 2 g $Sr(NO_3)_2$, activated with 6 g NaOH
C95S5-N2	95 g $CaCO_3$ with the addition of 5 g $Sr(NO_3)_2$, activated with 2 g NaOH
C95S5-N4	95 g $CaCO_3$ with the addition of 5 g $Sr(NO_3)_2$, activated with 4 g NaOH
C95S5-N6	95 g $CaCO_3$ with the addition of 5 g $Sr(NO_3)_2$, activated with 6 g NaOH
C90S10-N2	90 g $CaCO_3$ with the addition of 10 g $Sr(NO_3)_2$, activated with 2 g NaOH
C90S10-N4	90 g $CaCO_3$ with the addition of 10 g $Sr(NO_3)_2$, activated with 4 g NaOH
C90S10-N6	90 g $CaCO_3$ with the addition of 10 g $Sr(NO_3)_2$, activated with 6 g NaOH

Transesterification of Waste Cooking Oil (WCO) into Biodiesel

The biodiesel production process via the transesterification stage was conducted using WCO with an FFA content of less than 5%. The process employed 100 mL of WCO with a methanol-to-oil molar ratio of 9:1. The catalyst concentration was set at 1 wt.%, and the transesterification reaction was carried out for 3 h.

Biodiesel Characterization

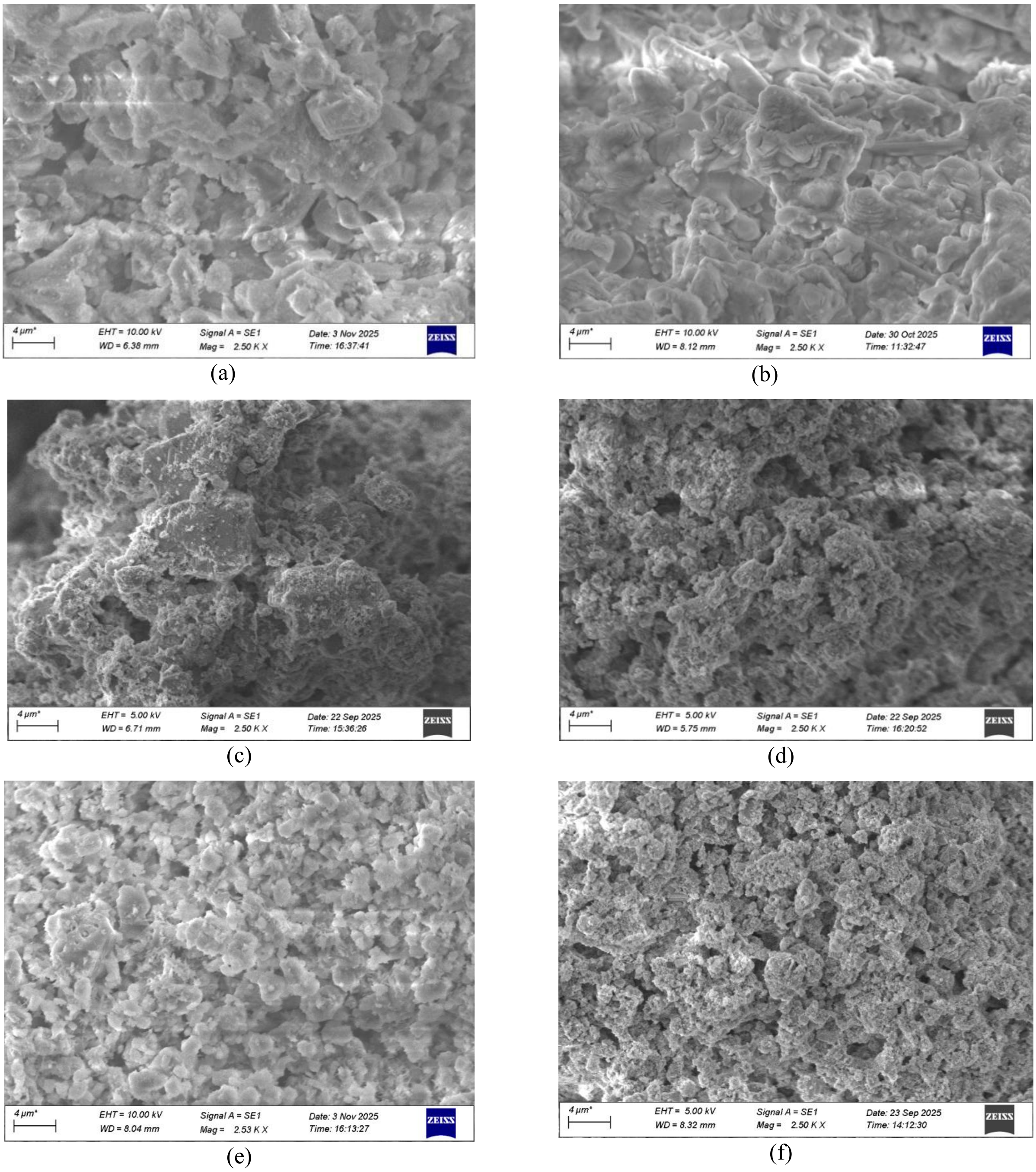
Product analysis involved several important parameters, including yield, which indicates the efficiency of the production process; calorific value, which measures the energy released during combustion; and flash point, defined as the temperature at which the product vapor becomes ignitable. Density represents the mass per unit volume of a substance, while viscosity indicates resistance to flow, which affects both processing and end-use performance.

RESULTS AND DISCUSSION

Morphological Analysis

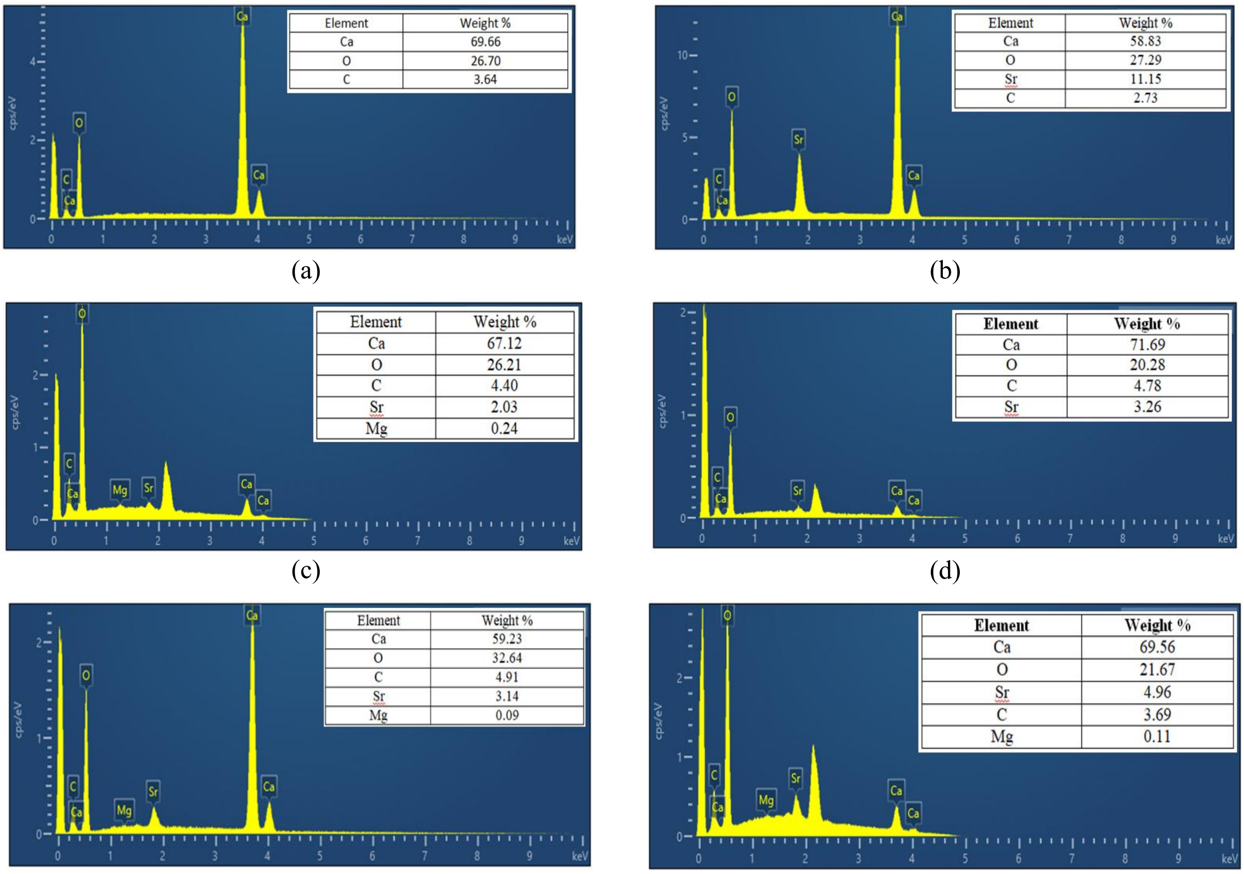
Figure 1. shows the morphology of CaO catalysts derived from cuttlefish bone modified by SrO impregnation and NaOH activation, namely C–N6, C90S10, C98S2–N6, C95S5–N6, C90S10–N2, and C90S10–N6. Variations in Sr and $CaCO_3$ composition were observed to significantly influence the catalyst surface structure.

In the C98S2–N6 sample, the surface appeared denser with smaller pores and a relatively homogeneous distribution. This behavior is attributed to the low Sr content (2 g), which limits the number of basic sites despite the high NaOH activation (6 g). The C95S5–N6 sample exhibited a more developed morphology, characterized by more distinct pores and a uniform distribution of SrO particles. This indicates an increase in active surface area and a more optimal number of basic sites, which may enhance catalytic activity. In contrast, the C90S10–N6 sample showed



(a) (b) (c) (d) (e) (f)

Figure 1. Morphological characteristics of (a) C–N6, (b) C90S10, (c) C98S2–N6, (d) C95S5–N6, (e) C90S10–N2, and (f) C90S10–N6



(a)

Element	Weight %
Ca	69.66
O	26.70
C	3.64

(b)

Element	Weight %
Ca	58.83
O	27.29
Sr	11.15
C	2.73

(c)

Element	Weight %
Ca	67.12
O	26.21
C	4.40
Sr	2.03
Mg	0.24

(d)

Element	Weight %
Ca	71.69
O	20.28
C	4.78
Sr	3.26

(e)

Element	Weight %
Ca	59.23
O	32.64
C	4.91
Sr	3.14
Mg	0.09

(f)

Element	Weight %
Ca	69.56
O	21.67
Sr	4.96
C	3.69
Mg	0.11

Figure 2. EDS spectra of (a) C-N6 (b) C90S10 (c) C98S2-N6 (d) C95S5-N6 (e) C90S10-N2 (f) C90S10-N6

a rough surface with noticeable SrO particle agglomeration due to the excessively high Sr content (10 g). Such agglomeration can block some pores and reduce the effective surface area, thereby decreasing catalyst efficiency despite an increased number of basic sites. Overall, the SEM results demonstrate that the amount of Sr strongly affects catalyst morphology. A moderate Sr loading (C95S5–N6) produces the most balanced surface structure, combining pore development, particle distribution, and homogeneity that are favorable for transesterification performance.

Numerous previous studies have demonstrated that Sr impregnation into CaO can influence catalyst morphology through changes in porosity, active particle distribution, and agglomeration tendency (Alhanif et al., 2025). (Tomano et al., 2020) reported that Sr loadings of 1-10% on Sr/CaO catalysts derived from cuttlebone resulted in a nearly homogeneous Sr distribution, although morphological changes were not pronounced at low loading.

The EDS results are presented in Figure 2 for (a) C98S2–N6, (b) C95S5–N6, and (c) C90S10–N6. The EDS analysis supports the previous SEM morphological observations. An increase in the amount of impregnated Sr led to a change in the elemental composition of the catalyst from Ca-dominated to a more balanced distribution between Ca and Sr. The C95S5–N6 sample exhibited the most favorable condition, as the Sr content was sufficiently significant without excessively reducing Ca dominance, resulting in a more homogeneous elemental distribution. In contrast, although C90S10–N6 showed a high Sr content, the agglomeration tendency observed in the SEM images may reduce the effective surface area. Therefore, a moderate combination of Ca and Sr (C95S5–N6) is considered the most optimal for producing a catalyst with high activity in biodiesel transesterification reactions.

The distribution of elements such as strontium within the CaO matrix after impregnation is commonly confirmed through EDS elemental mapping. (Tavizón-Pozos et al., 2025a) reported a uniform distribution of K^+ and Sr^{2+} in K–Sr/CaO catalysts derived from eggshells, indicating that Sr addition can be homogeneously dispersed within the catalyst structure.

X-ray Diffraction (XRD) Analysis of CaO/SrO Phases

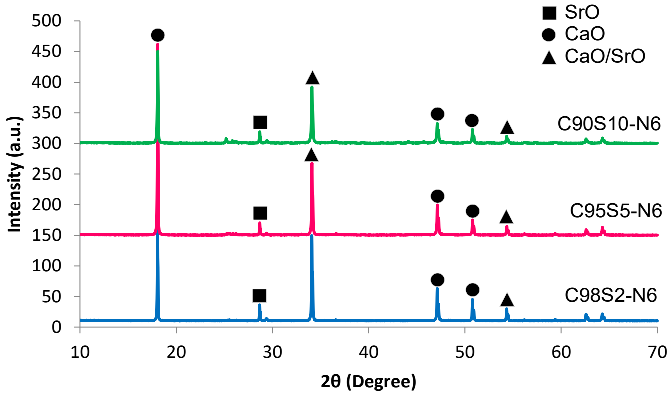


Figure 3. XRD patterns of CaO–SrO catalysts for samples C–N6, C98S2–N6, C95S5–N6, and C90S10–N6

The X-ray diffraction patterns of CaO, SrO, and CaO–SrO catalysts with various compositions are presented in Figure 3. The diffraction patterns of CaO and CaO–SrO exhibited similar features; however, the CaO–SrO pattern represents a combination of the characteristic peaks of both CaO and SrO. The characteristic CaO peaks observed at $2\theta = 18.066°$, 48.054°, and 52.306° showed slight shifts toward lower angles, while the SrO peak appeared at $2\theta = 29.657°$. In the CaO–SrO catalysts, new diffraction peaks were observed at $2\theta = 34.769°$ and 54.809°, accompanied by peak shifting and reduced intensity, indicating the influence of SrO modification on the CaO catalyst. (Zhang *et al*., 2021) reported similar changes in XRD peak positions and intensities due to SrO impregnation, including the attenuation of several CaO peaks and the emergence of SrO-related peaks around 29–31° (2θ). These phenomena were attributed to the formation of mixed phases as well as changes in crystallite size and lattice strain in SrO–CaO systems.

The XRD results indicate that the modification of CaO with SrO successfully formed a bifunctional catalyst with stronger basic sites. Variations in Sr content influenced the intensity and distribution of diffraction peaks, with a moderate Sr loading (C95S5–N6) producing the most balanced XRD pattern between CaO and SrO phases. This observation is consistent with the previous SEM and EDS results, which showed that moderate Sr addition leads to a homogeneous morphology, uniform elemental distribution, and good phase stability. Therefore, the C95S5–N6 sample is predicted to exhibit the highest catalytic activity in transesterification reactions due to the optimal combination of crystalline phase stability and availability of basic sites.

Fourier Transform Infrared (FTIR) Analysis of CaO–SrO Catalyst

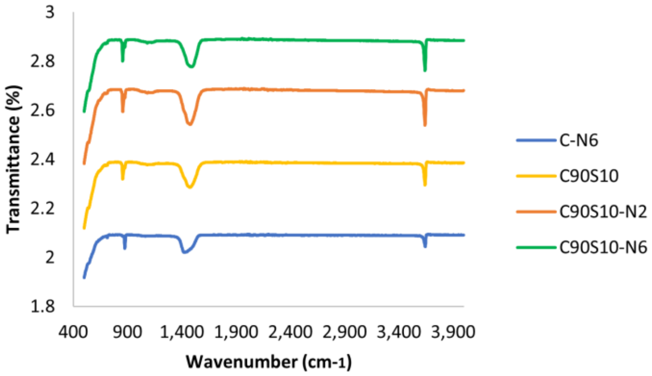


Figure 4. FTIR spectra of CaO–SrO catalysts for samples C–N6, C90S10, C90S10–N2, and C90S10–N6

FTIR analysis was conducted on selected CaO–SrO catalyst samples, namely C–N6, C90S10, C90S10–N2, and C90S10–N6, which represent different catalyst modification conditions, including pristine CaO, SrO-impregnated catalysts, and NaOH-activated catalysts. In contrast to XRD analysis, which focuses on the identification of crystalline phases, FTIR analysis was employed to confirm changes in functional groups, carbonate formation, and Ca–O and Sr–O bond vibrations on the catalyst surface. Therefore, the use of representative samples was considered sufficient to capture the main chemical trends without generating repetitive spectra.

In Figure 4, the FTIR spectra exhibit three main absorption bands that reflect the surface chemical characteristics of CaO–SrO basic oxides. The broad band in the wavenumber range of 3500–3700 cm^{-1} is attributed to the stretching vibrations of –OH groups from Ca–OH and Sr–OH species formed due to moisture adsorption. The presence of these –OH groups is commonly observed in CaO- and SrO-based materials owing to their highly hygroscopic nature.

The strong absorption band observed in the range of 1400–1500 cm^{-1} is attributed to the vibration of carbonate ions (CO_3^{2-}), indicating surface carbonation resulting from the reaction of CaO/SrO with atmospheric CO_2 (Tavizón-Pozos et al., 2025b), This result is consistent with the XRD analysis, which also confirms the presence of carbonate phases, and is further supported by previous reports stating that CaO readily transforms into $CaCO_3$ upon exposure to air. In addition, the the absorption band in the range of 800–900 cm^{-1} is associated with Ca–O and Sr–O vibrations, confirming the presence of active basic oxide phases. Variations in the intensity of this band indicate structural and chemical changes in the oxides induced by SrO impregnation and activation processes, which may influence the strength and distribution of basic sites on the catalyst *(Kingkam et al*., 2024).

Yield Biodiesel

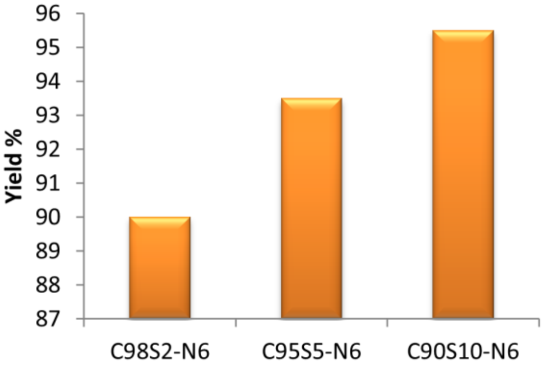


Figure 5. Biodiesel yield for samples C98S2–N6, C95S5–N6, and C90S10–N6

Figure 5 presents the biodiesel yield obtained from the three sample variations. The highest yield was achieved by the C90S10–N6 sample at 95.5%. Meanwhile, the C95S5–N6 sample produced 93.5%, and the C98S2–N6 sample showed the lowest yield of 90.0%.

These data confirm a significant increase in biodiesel yield with the formulation change from C98S2–N6 to C90S10–N6. The 95.5% yield observed for C90S10–N6 indicates optimal conditions and a very high conversion efficiency in the transesterification process. The achieved yield underscores the importance of selecting an appropriate catalyst composition to ensure efficient triglyceride conversion in the transesterification process (No *et al.*, 2024).

CONCLUSION

In conclusion, cuttlefish bone waste was demonstrated to be an effective and sustainable source of CaO for heterogeneous catalyst development in biodiesel production from waste cooking oil. Modification with $Sr(NO_3)_2$ and subsequent NaOH activation significantly enhanced catalyst surface morphology, elemental dispersion, and phase stability. Among the catalysts evaluated under the studied conditions, the catalyst formulation comprising a 90:10 CaO–SrO ratio with 6 g NaOH activation (C90S10–N6) exhibited the best performance, achieving a high biodiesel yield of 95.5%. The resulting biodiesel exhibited a density of 0.87 g/mL, a moisture content of 0.64%, a calorific value of 38.52 MJ/kg, and a substantial reduction in free fatty acid (FFA) content from 2.84% to 1.71%. These results indicate that cuttlefish bone–derived CaO–SrO catalysts offer an environmentally friendly, cost-effective, and promising alternative for supporting sustainable biodiesel production.

CONFLICT OF INTEREST

The authors declare that there are no conflicts of interest in the conduct of the research or the preparation of this manuscript.

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