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Evaluation of Chicken Bone-Derived CaO Catalyst for Biodiesel Production

Ilham Mufandi[a*], Muhammad Nur Kholis[a], Arief Rahmawan[a], Ratchaphon Suntivarakorn[b], Dhaifullah Nafis Nugraha[a], Raka Wyztyo Alana Prathista[a]

[a] Department of Agro-industrial Technology, Faculty of Science and Technology, Universitas Darussalam Gontor, Ponorogo, 63471, Indonesia

[b] Department of Mechanical Engineering, Faculty of Engineering, Khon Kaen University, 40002, Thailand

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ABSTRAK: Penelitian ini fokus pada pemanfaatan minyak jelantah (WCO) menjadi biodiesel dengan menambahkan katalis kalsium oksida (CaO) yang berasal dari limbah tulang ayam. Katalis CaO hasil sintesis dikarakterisasi menggunakan FTIR dan SEM-EDX untuk mengetahui sifat struktur dan kimianya. Percobaan produksi biodiesel dilakukan pada suhu 60°C dengan rasio minyak jelantah dan metanol sebesar 9:1 dan konsentrasi katalis CaO sebesar 5%. Biodiesel yang dihasilkan dikarakterisasi berdasarkan parameter kualitas utamanya, termasuk titik nyala, densitas, bilangan asam, viskositas, dan nilai kalor. Hasil penelitian menunjukkan bahwa katalis CaO mengandung berbagai senyawa organik, termasuk haloalkana, gugus fungsi 1,2-disubstitusi, alkohol primer, senyawa aromatik, dan alkohol. Selain itu, katalis CaO mengandung komposisi mineral seperti kalsium, karbon, oksigen, natrium, magnesium, dan fosfor. Yield biodiesel meningkat secara signifikan dengan bertambahnya loading katalis, mencapai maksimum 92,70% pada loading katalis 15%. Penelitian ini menunjukkan keefektifan katalis CaO yang berasal dari limbah tulang ayam untuk produksi biodiesel yang ramah lingkungan dengan menggunakan teknologi microwave.
Kata Kunci: Biodiesel; Katalis CaO; Limbah Tulang Ayam; Minyak Goreng Bekas

ABSTRACT: This research was focused on the utilization of waste cooking oil (WCO) into biodiesel by adding a calcium oxide (CaO) catalyst derived from chicken bone waste. The synthesized CaO catalyst was characterized using FTIR and SEM-EDX to determine its structural and chemical properties. The biodiesel production experiment was conducted at 60°C with a ratio of waste cooking oil and methanol of 9:1 and a CaO catalyst load of 5% concentration. The biodiesel produced is characterized by its main quality parameters, including flash point, density, acid number, viscosity, and heating value. The research results show that the CaO catalyst contains various organic compounds, including haloalkanes, 1,2-disubstituted functional groups, primary alcohols, aromatic compounds, and alcohols. In addition, the CaO catalyst contains mineral compositions such as calcium, carbon, oxygen, sodium, magnesium, and phosphorus. Biodiesel yield increases significantly with increasing catalyst loading, reaching a maximum of 92.70% at 15% catalyst loading. This research shows the effectiveness of the CaO catalyst derived from chicken bone waste for environmentally friendly biodiesel production using microwave technology.
Keywords: Biodiesel; CaO Catalyst; Chicken Bone Waste; Waste Cooking Oil

1. Introduction

Over the past three years, Indonesia has witnessed a significant decline in oil production, dropping from 708.32 million to 612.42 million barrels per day, leading to a corresponding increase in crude oil imports from 19.93 to 27.86 million kiloliters (Mineral, 2022). This trend highlights the urgent need to address escalating oil consumption. One sustainable alternative is renewable energy, specifically biodiesel, which has the potential to reduce emissions by up to 50% (Azzahro & Broto, 2021). Various studies have been carried out regarding the production of biodiesel from used cooking oil using heterogeneous catalysts. Research from (Maulana et al.,

* Corresponding author
Email address: ilhammufandi@unida.gontor.ac.id

2019) explains the method of producing biodiesel from coconut waste using the in-situ transesterification method, which shows great potential in utilizing organic waste for biodiesel production. In addition, research from (Jaya et al., 2021) discusses the production of biodiesel from used cooking oil using heterogeneou catalysts, which can increase the efficiency and quality of the biodiesel produced. Furthermore, research from (Murni et al., 2018) explores the production of biodiesel from used cooking oil with the help of ultrasonic waves, which is proven to speed up the transesterification process and increase biodiesel yields. The pre-treatment process also plays an important role in biodiesel production, as explained in the Research from (Dewati et al., 2014). This article explains various pre-processing techniques that can optimize the quality of used cooking oil before it is further processed into biodiesel. Finally, research from (Haryono et al., 2016) discusses the production of biodiesel from used cooking oil using the heterogeneous catalyst CaO, which shows that the use of this catalyst can produce high quality biodiesel with a more efficient process.

Cooking oil, commonly used and reusable three to four times, undergoes complex processes during heating, making it hazardous to health due to increased saturation. Waste cooking oil (WCO) is identified when total polar compounds exceed 25% (Adhikesavan et al., 2022). Additionally, the byproduct of increased chicken consumption, chicken bone waste, poses health risks(Nur Ajizah et al., 2022). Despite being traditionally undervalued, chicken bones, containing calcium carbonate and calcium phosphate, have potential as catalysts for biodiesel production from various animal sources(Ferriansyah & Hadiantoro, 2021; Iqbal et al., 2023)

Catalysts, compounds that accelerate reactions, play a crucial role in compound synthesis and can be categorized as homogeneous or heterogeneous (Jamilatun et al., 2022). While homogeneous catalysts face challenges in recycling and may interfere with biodiesel production, leading to glycerol separation constraints(Oko & Feri, 2019), heterogeneous catalysts, especially solid acid-base catalysts, offer advantages such as efficient biodiesel production, ease of separation, and product purification. Heterogeneous catalysts are non-corrosive, non-toxic, and can be regenerated after use(Simpen et al., 2021). In the realm of transesterification reactions, CaO emerges as a potential heterogeneous catalyst, providing easy separation through filtration, reusability, and significant non-corrosive properties (Widiarti & Kusumastuti, 2015).

Analyzing functional groups and molecular interactions is crucial in biodiesel production, and Fourier Transform InfraRed (FTIR) is a method commonly used for this purpose (Chen et al., 2023). On the other hand, Scanning Electron Microscopy (SEM), developed in the 1930s, has become a valuable tool in various domains, offering insights into the topography, morphology, and composition of diverse materials (Sharma & Bhardwaj, 2019).

Innovative process intensification methods, such as membrane reactors, reactive distillation, ultrasonic irradiation, and microwaves, have been employed for biodiesel production. Notably, microwave and ultrasonic methods significantly impact biodiesel yield by reducing reaction time and affecting molecular-level heating, improving separation steps with fast and even internal heating (Sharma et al., 2019)

Therefore, this research focuses on utilizing chicken bone waste as a source for synthesizing a CaO catalyst for biodiesel production from waste cooking oil. The investigation was carried out to assess the impact of varying catalyst loading levels on its properties. The synthesized CaO catalyst undergoes characterization using FTIR and SEM-EDX analyses. Finally, the produced biodiesel is evaluated for key quality parameters, including yield, flash point, viscosity, density, and heating value.

2. Materials And Methods

2.1. Materials

The main raw materials used in this study were waste cooking oil (WCO), chicken bone, aquadest, 10% of sodium hydroxide (NaOH), 90% of methanol (CH_2OH), and KOH. Waste cooking oil (WCO) was collected from the canteen center of Universitas Darussalam Gontor, Indonesia. The waste cooking oil is first filtered to remove impurities and solid particles. Then, the waste cooking oil is heated at 120°C for 2 hours in a closed container. The temperature of 120°C is chosen because it is high enough to evaporate water without causing decomposition of the waste cooking oil. During the heating process, the waste cooking oil is stirred periodically to ensure even heating.

Chicken bones weighing 10 kg were obtained from a chicken satay vendor in the satay village area of Ponorogo, Indonesia. The chicken bones were purchased fresh and immediately washed with running water to remove dirt and leftover meat. Then, the chicken bones were soaked in saltwater for 30 minutes to kill bacteria. Next, the chicken bones were cut into small pieces and boiled in a large pot at 100°C. The purpose of boiling was to extract calcium and collagen from the chicken bones.

2.2. Preparation of CaO Catalyst from Chicken Bones

The process of making CaO catalyst from chicken bones references previous research conducted by Kedir et al. (2023). This research applies modifications to the calcination temperature. The chicken bone pre-treatment process has been explained in a previous section. At this stage, Dried chicken bones are ground using a ball mill to obtain a fine powder. Uniform particle size ensures a larger surface area of the catalyst, thereby increasing its catalytic activity. Chicken bone powder is filtered with a 100 mesh sieve to separate coarse particles that can clog the reactor during the calcination process. Chicken bone powder is calcined in a kiln at 950°C for 3 hours in atmospheric air. Calcination converts $CaCO_3$ in chicken bones into CaO, which is an active catalyst for transesterification of waste cooking oil. The resulting CaO catalyst is stored in a vacuum desiccator to prevent water and air contamination which can reduce catalytic activity.

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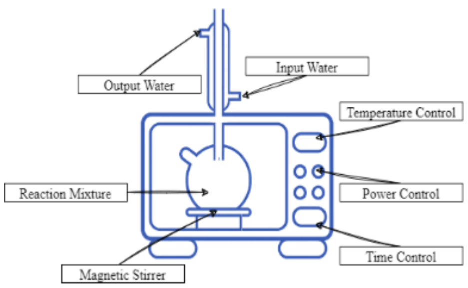
2.3. CaO Catalyst Characterization

This section describes the methods employed to characterize the physicochemical properties of the CaO catalyst prepared from chicken bones. Two primary techniques were utilized: Fourier Transform Infrared (FTIR) spectroscopy and Scanning Electron Microscopy (SEM) with Energy-Dispersive X-ray spectroscopy (EDX).

FTIR spectroscopy, is a valuable analytical tool that analyzes the interaction of infrared radiation with molecules(C. Chen et al., 2018; Mohamed et al., 2017). In the context of characterizing the CaO catalyst, FTIR spectroscopy will be used to identify the functional groups present in the catalyst. This information can confirm the presence of calcium oxide (CaO) and potentially reveal the presence of any residual impurities remaining from the chicken bone precursor.

SEM is a high-resolution imaging technique that utilizes a focused beam of electrons to scan the surface of a sample(González-Santamaría et al., 2021). In the context of characterizing the CaO catalyst, SEM was used to examine the morphology of the catalyst particles, including their size, shape, and aggregation behavior. EDX analysis will be employed to determine the elemental composition of the catalyst, confirming the presence of calcium and oxygen as the primary elements in CaO.



a)

b)

Figure 1. Biodiesel production via microwave (a) microwave design, (b) biodiesel purification

2.1. Biodiesel Production Via Microwave

In the biodiesel production process, waste cooking oil is filtered to remove impurities and contaminants that can interfere with the transesterification reaction and is heated at 120℃ for 2 hours to remove water content because water can hinder the reaction and produce soap. The biodiesel production process is carried out based on the transesterification process conducted by (Khan et al., 2020). The transesterification reaction is carried out in a 1000 mL three-necked distillation flask with a microwave.

Before adding the catalyst to the waste cooking oil, the waste cooking oil is first heated to 70℃. Once the oil temperature is appropriate, the catalyst and modified methanol are added to the waste cooking oil. The molar ratio of methanol to waste cooking oil is 9:1 with a catalyst percentage of 5,10,15% (catalyst/oil ratio weight), the reaction mixture is stirred at a speed at a temperature of 65ºC for 1 hour in the microwave using a voltage of 300 watt. The microwave helps to speed up the reaction rate. After the reaction is complete, the catalyst is separated using centrifugation. The product mixture is allowed to stand in a separating funnel for 24 hours. Glycerol, as a by-product, will separate in the lower layer because it has a higher density. Residual methanol is evaporated using vacuum evaporation and can be recycled. The upper layer containing biodiesel is washed with water several times to remove contaminants and residual methanol.Biodiesel production via microwave can be seen in Figure 1.

2.2. Characterization of Physicochemical Properties of Biodiesel

The characterization of the physicochemical properties of CaO catalyst derived from chicken bones was conducted using two complementary techniques: Fourier-Transform Infrared (FTIR) spectroscopy and Scanning Electron Microscopy (SEM) equipped with Energy-Dispersive X-ray spectroscopy (EDX).

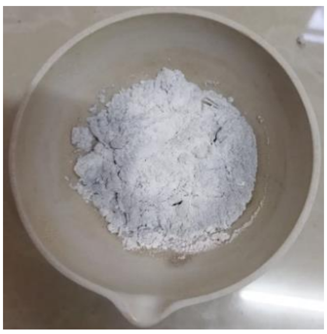


Figure 2. CaO catalyst

FTIR spectroscopy is a radiation-based analytical technique that elucidates the chemical functionalities present in the CaO catalyst(Chen et al., 2018). SEM-EDX provides visual information about the catalyst's morphology in the micrometer range. Additionally, EDX allows for a semiquantitative determination of the elemental composition within the analyzed area. In terms of physical properties, biodiesel has specific quality standards that must be met before it can be used. These standards ensure that biodiesel has the appropriate properties for diesel engines and does not harm the environment. Some of the biodiesel test parameters used in this study include: biodiesel yield, flash point, density, viscosity: acid number, and calorific value.

Table 1: FTIR waveform absorption

Wavenumber	Group	Compound Class
551.64	C-Br stretching	Halo compound
628.79	C-Br stretching	Halo compound
879.54	C-O bending	1,2-disubstituted
1083.99	C-O stretching	Primary alcohol
1456.26	C-H bending	Aromatic compound
3251.98	O-H stretching	Alcohols
3570.24	O-H stretching	Alcohol

3. Result And Discussion

3.1. CaO Catalys From Chiciken Bone

In this research, the CaO catalyst fabrication process involves several stages, including separating chicken bones, cleaning, drying, grinding, and calcination. The CaO catalyst obtained is in the form of a white fine powder as seen in Figure 2. Evaluation of CaO products was carried out using FTIR and SEM-EDX tests described in the next section.

3.2. CaO Catalys Characterization Using Fourier Transform InfraRed (FTIR)

In this study, FTIR analysis was carried out to characterize the chemical structure obtained from chicken bones that cah be seen in Figure. FTIR works based on the interaction between infrared radiation and chemical molecules to identify the elements contained in chicken bones. The rotational energy of the molecules is formed from the radiation absorbed by the sample. After that, the signal is captured by the detector and translated into spectral form (Aini et al., 2023).

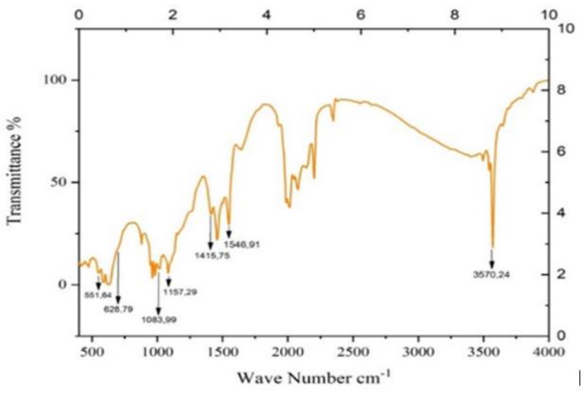


Figure 3. FTIR Result Test

Based on Figure 3, FTIR analysis of the CaO catalyst sample derived from chicken bones indicates the presence of stretching vibrations between Carbon (C) and Bromine (Br) atoms, as shown at wave numbers 551.64 cm^{-1} and 628.79 cm-1. Additionally, both wave numbers indicate the presence of haloalkane groups resulting from the bond between alkane compounds and halogen atoms. These findings are consistent with the discoveries of(Arul et al., 2018; Tan et al., 2015), who reported similar wavenumbers for C-Br vibrations in haloalkane compounds. Research conducted by(Aini et al., 2023) also noted similar wavenumbers for C-Br vibrations in haloalkanes.

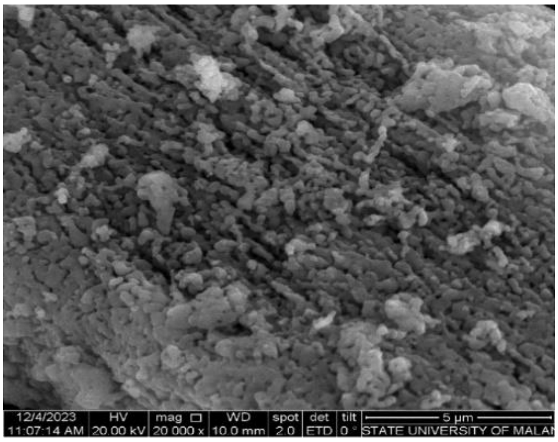


Figure 4. SEM characterization of catalysts

The wavenumber of 879.54 cm^{-1} indicates bending vibration in the C-O bond. This wavenumber value is specific for bending vibration in 1,2-disubstituted compounds, meaning that there are two substituents other than H on the C atom. Organic compounds that are 1,2-disubstituted can detect similar wavenumbers for C-O bending vibration (Haris et al., 2022). The wavenumber of 1083.99 cm^{-1} indicates stretching vibration between Carbon (C) and Oxygen (O) atoms. This wavenumber value indicates the presence of primary alcohol functional groups. The emergence of primary alcohol functional groups in C-O vibration is consistent with the research conducted by(Hsiao et al., 2020). The wavenumber of 1456.26 cm^{-1} indicates bending vibration in C-H atoms in aromatic compounds. With the detection of this wave, the chemical bonds show the presence of an aromatic ring in one of the compounds. The wavenumbers of 3251.98 cm^{-1} and 3570.24 cm^{-1} are in the typical range for stretching vibration in hydroxyl groups (O-H). This indicates the presence of alcohol functional groups in two different compounds. Although their wavenumbers are slightly different, this could be due to differences in the chemical environment of these hydroxyl groups.

3.3. CaO Catalys Characterization Using Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) analysis was employed to investigate the surface morphology of the CaO catalyst derived from chicken bones. SEM operates by

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focusing a beam of high-energy electrons onto the sample surface. The principle of SEM involves examining the surface morphology of a sample using radiation from an energy source that is sufficiently capable of penetrating and inducing specific transitions, which results in emissions at the surface of the energy powder that can be analyzed. In this study, the morphology of the CaO catalyst from chicken bones can be seen in Figure 4.

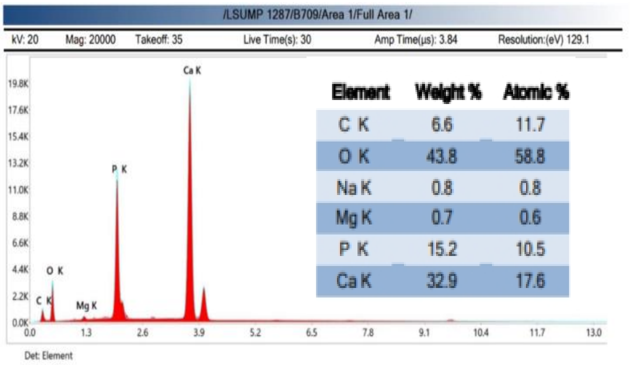

Figure 5. EDX characterization of catalysts

Based on Figure 5, the morphology of CaO catalyst from chicken bone with magnification of 20,000 times and diameter of 5 µm was obtained. The SEM characterization results show that the CaO catalyst from chicken bone with 5% concentration has a morphological structure in the form of granules that tend to be clustered, non-porous, and dense. In addition, the morphological form of CaO catalyst from chicken bone is also supported by the presence of mineral components detected in EDX analysis which can be seen in Figure 5. The mineral components consist of main elements such as calcium (Ca), carbon (C), oxygen (O), sodium (Na), magnesium (Mg), and phosphorus (P). The analysis shows that the CaO catalyst from chicken bone contains calcium (Ca) with an atomic percentage of 17.6%, carbon (C) of 11.7%, oxygen (O) of 58.8%, Na of 0.8%, Mg of 0.6% and phosphorus (P) of 10.5%.

3.4. Biodiesel Characterization

The biodiesel obtained was subsequently subjected to quality testing in accordance with the Biodiesel Quality Standard outlined by the Ministry of Energy and Mineral Resources of Indonesia. Specifically, the specification adhered to is SNI 04-7182-2015, which integrates internationally recognized biodiesel standards such as ASTM D6751 in the United States and EN 14214:2002 for European Union nations. The results of the biodiesel, augmented with a CaO catalyst derived from chicken bones, are detailed in the following Table 2.

Based on Table 2, the use of CaO catalyst derived from chicken bones improves most of the biodiesel quality parameters compared to the control (without catalyst). Biodiesel yield increased 0.09% from 89.89% in the control to 92.47% with the addition of the catalyst. This indicates the effectiveness of CaO catalyst in enhancing the conversion of vegetable oil into biodiesel. The catalyst improved the efficiency of the transesterification reaction and converted more triglycerides from waste cooking oil into biodiesel. The flash point of biodiesel increased from 150°C in the control to 152°C with the addition of the catalyst. This suggests that biodiesel produced with CaO catalyst has a higher flash point, indicating better thermal stability.

Table 2. Biodiesel characteristics from waste cooking oil

Parameter	Control (no catalyst)	Addition of catalyst (5 %)	ASTM	Standard
Yield	89.89%	92,47%	-	-
Flash Point (°C)	150	152	ASTM D93-10	130-170
Density (gr/cm^3)	0.83	0.85	ASTM D4052-09	0.87810
Acid Number (KOH ml/g)	00.03	0,05	ASTM D664	00.05
Viscosity (cSt)	03.46	4,6	ASTM D445	1.9–6.0
Calorific Value (MJ/Kg)	21.11	22,72	ASTM D240	32.94

The density of biodiesel increased from 0.83 gr/cm3 in the control to 0.85 gr/cm3 with the addition of the catalyst. This indicates that biodiesel produced with CaO catalyst has slightly higher density. The acid number of biodiesel increased from 0.03 KOH ml/g in the control to 0.05 KOH ml/g with the addition of the catalyst. Although slightly higher, this acid number is still within the ASTM D664 standard limits. The viscosity of biodiesel increased from 3.46 cSt in the control to 4.6 cSt with the addition of the catalyst because low viscosity can cause leakage on the fuel injection pump. The calorific value of biodiesel increased 1.61% from 21.11 MJ/kg in the control to 22.72 MJ/kg with the addition of the catalyst. This indicates that biodiesel produced with CaO catalyst has higher energy potential.

4. Conclusion

This research successfully synthesized a fine white powder CaO catalyst from chicken bones through a process of separation, cleaning, drying, grinding, and calcination. The CaO catalyst was evaluated using FTIR and SEM-EDX tests. FTIR analysis showed that the CaO catalyst from chicken bones contains various organic compounds, including haloalkanes, 1,2-disubstituted functional groups, primary alcohols, aromatic compounds, and alcohols. The CaO catalyst derived from chicken bones has a dense grain structure and a mineral composition that includes important elements such as calcium, carbon, oxygen, sodium, magnesium, and phosphorus. The use of CaO catalyst from chicken bones improves most of the biodiesel quality parameters. This shows the potential use of CaO catalyst from chicken bone waste in efficient and environmentally friendly biodiesel production. Unwanted organic molecules, such as toluene (C_7H_8) and oxalic acid ($C_2H_2O_4$), are detected in the biodiesel GC chromatogram. Water-soluble oxalic acid could corrode machinery and equipment, whereas water-insoluble toluene may be hazardous to such items.

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