

BIODIESEL SYNTHESIS FROM WASTE COOKING OIL USING CATALYST DERIVED FROM SEA CRAB SHELL AND NICKEL OXIDE

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ABSTRACT

As global energy demand increases and fossil fuel reserves decrease, alternative energy sources are becoming important. Biodiesel, derived from renewable sources, is a promising alternative to conventional diesel due to its environmental benefits. This paper investigates the synthesis and application of a NiO/CaO catalyst derived from sea-crab shells for biodiesel production. Waste cooking oil (WCO) was used as feedstock for the transesterification process. The catalyst was synthesized via a wet impregnation method and then characterized using SEM, XRD, FT-IR, and TG-DSC techniques. The transesterification reaction was optimized to achieve a maximum biodiesel yield of 90.29%. The optimal condition is 68 °C, a methanol/oil molar ratio of 24:1, a catalyst loading of 10 wt%, and the reaction time of 3 hours. The results demonstrate that NiO/CaO is an efficient catalyst in converting WCO to biodiesel, hence providing an environmentally friendly and cost-effective solution.

Keywords: WCO, biodiesel, sea-crab shell, catalysis, waste cooking oil.

1. INTRODUCTION

The depletion of fossil fuels and environmental concerns such as greenhouse gas emissions have led to the search for alternative, renewable energy sources. Biodiesel, derived from vegetable oils, animal fats, and waste oils, is a promising candidate [1]. Compared to traditional diesel, biodiesel offers several advantages, such as biodegradability, non-toxicity, and reduced emissions of sulfur oxides and particulates [2].

Biodiesel is most produced via transesterification of oils and fats, in which triglycerides react with an alcohol, typically methanol, in the presence of a catalyst to generate fatty acid methyl esters (FAME) and glycerol [3]. A key factor in determining the cost and efficiency of biodiesel synthesis is the choice of catalyst. Sodium hydroxide (NaOH) and other homogeneous catalysts have been widely used in the past, but they have disadvantages such as difficulty in separation and the need for neutralization, which increase production costs and cause environmental problems [4].

Heterogeneous catalysts, including calcium oxide (CaO), provide several benefits over homogeneous catalysts, such as simpler separation, greater reusability, and a smaller environmental effect [5, 6]. A variety of biological materials, such as eggshells, bones, and sea-shells, can be used to make calcium oxide. The CaO/ZnO catalyst employed eggshell as a CaO source [7]. A surplus and inexpensive source of CaO is from sea-crab shells that can be obtained through thermal method [8]. In addition, the use of nickel oxide (NiO) as a promoter enhances catalytic activity, especially during the conversion of waste cooking oils that contain high levels of free fatty acids [9]. The combination of CaO and NiO results in a catalyst that has high basicity and stability, which makes it perfect for the transesterification.

Waste biomaterials have been investigated in a numerous of research as sources of CaO for catalyst production. Previous research has shown that calcium oxide (CaO) obtained from eggshells, oyster shells, and bones has good catalytic performance in transesterification reaction [10, 11]. By increasing dispersion on the catalytic surface and offering more base sites, NiO has been demonstrated

to increase the activity of CaO-based catalysts [12]. Consequently, the current work investigates the use of sea-crab shells, a waste from the seafood industry, as a catalyst for transesterification [13].

Waste cooking oil (WCO) is an attractive feedstock for biodiesel production because of its abundance, low cost, and role in waste reduction. Cooking oil is discarded every year after use in restaurants and households. Improper disposal of WCO poses serious environmental risks, including water pollution. WCO serves not only as a low-cost feedstock for biodiesel production but also as a sustainable approach to mitigating waste management problems through transesterification [4]. However, it typically contains a high concentration of free fatty acids (FFA), which can be a challenge to transesterification process, hence it requires the use of efficient catalysts that can tolerate FFA without producing soaps through saponification.

The combination of WCO and biowaste-derived catalysts such as NiO/CaO from sea-crab shell represents an economy and sustainable approach to biodiesel production. WCO serves as a cost-effective feedstock, while sea-crab shell provides an environmentally friendly catalyst source. The aim of this study is to provide an environmental and cost-effective solution for biodiesel production.

2. MATERIALS AND RESEARCH METHODOLOGY

2.1. Materials

Waste cooking oil, collected from several restaurants in Ho Chi Minh City, was used as the feedstock for biodiesel production. Sea crab shells, obtained from seafood restaurants, were cleaned, dried, and processed to extract CaO. Other chemicals, including methanol (HPLC grade), and nickel nitrate hexahydrate $[\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}]$, were sourced from Fisher Scientific and Merck.

2.2. Catalyst preparation

The sea crab shells were initially washed with distilled water to remove organic matter and impurities. The cleaned shells were then dried at 105 °C for 6 hours and crushed into fine powder. The powdered shells were calcined at 900 °C for 4 hours to convert CaCO_3 into CaO. The CaO was further screened with a 0.125 mm diameter sieve to obtain uniform CaO.

The NiO/CaO catalyst was synthesized using the wet impregnation method. 9.88 g of nickel nitrate hexahydrate $[\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}]$ was mixed with 35 mL of deionized water. This solution was then gradually added to a beaker containing 28g calcined CaO. The mixture was stirred during the adding process. After that, the mixture was covered by aluminum foil and left at room temperature for 15 hours to completely impregnate the salt solution into CaO. It was further dried at 105 °C for 14 hours and then calcined at 500 °C for 4 hours to form the NiO/CaO catalyst. The catalyst was screened with a 0.125 mm sieve.

2.3. Catalyst characterization

The synthesized NiO/CaO catalyst was characterized using several techniques such as SEM-EDX, XRD, FT-IR. Scanning electron microscopy – energy dispersive X-ray (SEM-EDX) was used to analyze the surface morphology and the elemental composition. X-ray diffraction (XRD) was used to identify the crystalline phases and compared with database of characteristic peaks of CaO and NiO. Fourier transform infrared spectroscopy (FT-IR) provided detailed information on the functional groups present on the catalyst surface. Finally, Thermogravimetric-Differential Scanning Calorimetry (TG-DSC) was used to evaluate the thermal stability and decomposition behavior.

2.4. Transesterification experiment

The transesterification experiments were carried out in a 100 mL round-bottom flask fitted with a magnetic stirrer and reflux condenser. The amounts of catalyst and methanol, as well as the reaction temperature and time, were varied according to the specific experimental conditions. In each run, 10 g of waste cooking oil was combined with methanol at various molar ratios, ranging from 6:1 to 30:1. The NiO/CaO catalyst was subsequently added to the flask at different concentrations ranging from 5% to 25% weight percent of the oil. The reaction mixture was heated to the desired temperature (ranging

from 35 °C to 68 °C) and stirred for 1 to 5 hours depending on the experiment. After the reaction, the mixture was cooled and separated into two phases: biodiesel (upper phase) and glycerol (lower phase). The resulting biodiesel was subsequently washed with warm water to eliminate any remaining catalyst and methanol.

Gas Chromatography with Mass Spectrum Detector (GC – MS) was used to determine the FAME yield ($H\%$) of the transesterification.

$$H\% = \frac{\sum A_{FAME}}{A_{ISTD}} \times \frac{m_{ISTD}}{m_p} \times 100\% \quad (1)$$

where

- A_{FAME} is the peak area of FAME in the sample,
- A_{ISTD} is the peak area of internal standard (ISTD),
- m_{ISTD} is the weight of ISTD in the sample,
- m_p is the weight of the biodiesel sample (transesterification product).

3. RESULTS AND DISCUSSIONS

3.1. Catalyst characterization results

To gain a deeper understanding of the structure, composition, and surface properties of the NiO/CaO catalyst synthesized from sea crab shells, SEM-EDX, XRD, FT-IR, and TG-DSC were employed.

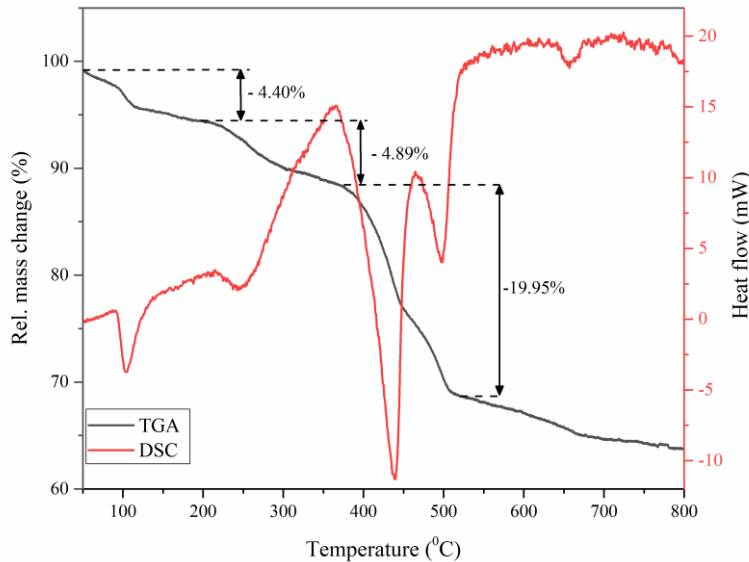


Figure 1. TG and DSC curve of uncalcined NiO/CaO

TG-DSC were performed to evaluate the thermal properties and stability of the uncalcined NiO/CaO. As illustrated in Figure 1, the TG curve shows a weight loss of approximately 4.4% below 250 °C, attributed mainly to the evaporation of physically adsorbed water [14]. Within the temperature range of 250 – 500 °C, a notable mass loss of about 25% was observed, accompanied by two endothermic peaks on the DSC curve. This suggests that the removal of NO_3^- groups from $\text{Ni}(\text{NO}_3)_2$ took place, leading to the formation of NiO on the CaO support, along with the decomposition of $\text{Ca}(\text{OH})_2$. At temperatures above 500 °C, the sample exhibited only a slight weight loss, indicating that $\text{Ni}(\text{NO}_3)_2$ completely decomposed to NiO at that temperature. Thermogravimetric analysis revealed that

the NiO/CaO catalyst exhibited good thermal stability up to 800 °C. Therefore, a calcination temperature of 500 °C is suitable for synthesizing the catalyst.

Based on the TGA results of CaO (data not shown), we selected an initial calcination temperature of 900 °C for the sea crab shells to obtain CaO. Additionally, a calcination temperature of 500 °C was chosen to thermally treat the CaO impregnated with nickel nitrate for the synthesis of the NiO/CaO catalyst.

SEM image of the synthesized NiO/CaO catalyst is illustrated in Figure 2. SEM image showed the structure of catalyst surface, while the EDX analysis confirmed the presence of key elements, including Ni, Ca, Mg, and O. It also demonstrated that NiO particles successfully dispersed on the CaO surface as in Table 1 and Figure 2d.

Table 1. Elemental characterization from EDX

Element	Weight (%)	Atom (%)
C	9.12	17.21
Ca	42.99	29.37
O	37.42	64.05
Mg	2.57	2.90
Ni	7.90	3.68
Total	100	100

This uniform distribution of NiO (Figure 2d) is crucial for increasing the catalytic activity by providing active sites for the biodiesel conversion reaction. The structure enhances the catalyst's performance by promoting efficient diffusion of reactants into the pores, facilitating the transesterification process [15, 16].

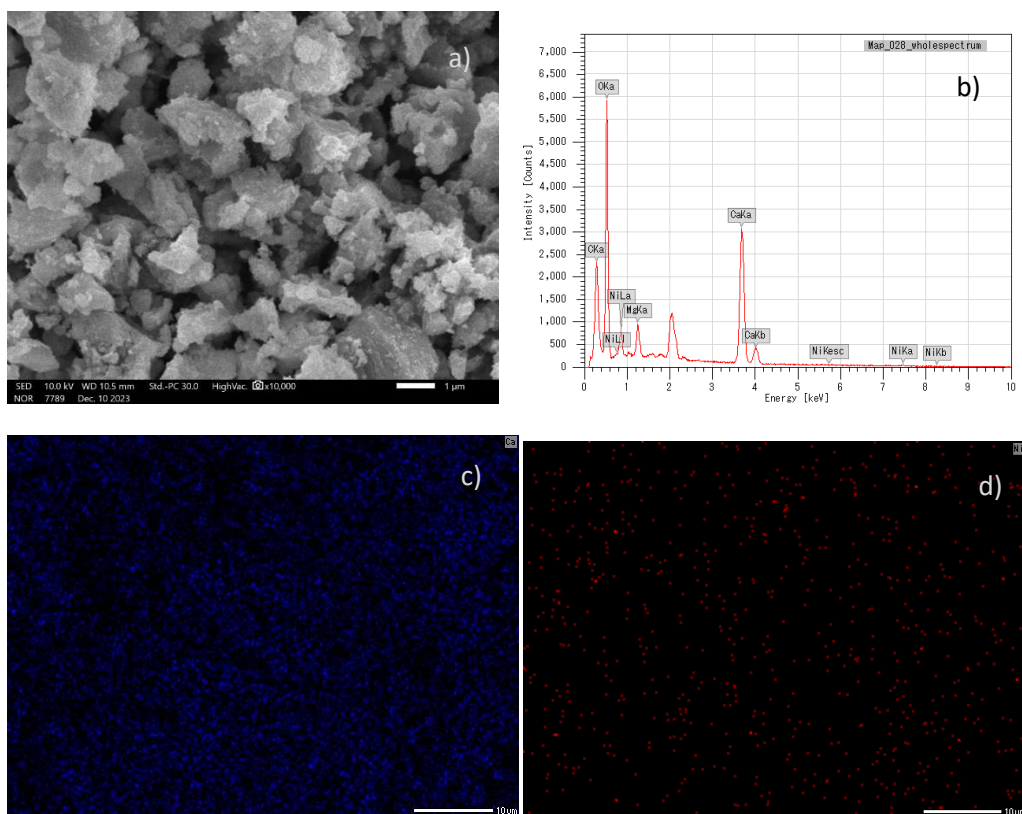


Figure 2. SEM image (x10,000) (a), EDX spectrum (b), Ca distribution (c), and Ni distribution (d) in NiO/CaO catalyst sample.

In Figure 3, the XRD pattern of the calcined NiO/CaO catalyst showed sharp peaks corresponding to both CaO and NiO. The strong diffraction peaks for CaO at $2\theta = 32.2^\circ$, 37.4° , and 53.9° indicate the presence of highly crystalline CaO. Additionally, the peaks observed at $2\theta = 37.4^\circ$, 43.3° and 62.5° are characteristic of NiO, confirming that NiO presents on the CaO surface. The peaks of NiO overlapped with the peaks of MgO because CaO source was obtained from sea crab shell (data not shown). In addition, the peaks of HAp which is formulated as $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ also appeared in the XRD pattern at $2\theta = 31.9^\circ$, 33.0° . The existence of HAp is also found in the research of Marwa Abdul Muhsien Hassan *et al.* [17]. The peaks of CaCO_3 also appear in the XRD pattern at $2\theta = 29.4^\circ$; 39.4° ; 47.4° which can be from the reformation between CaO and CO_2 during the impregnation and calcination. The NiO size was found to be relatively small, around 18.98 nm, while the CaO crystallites were larger, with an average size of approximately 37.49 nm. Smaller NiO crystallites offer a higher specific surface area and a greater number of active sites for the catalytic reaction, which is crucial for enhancing the overall performance of the catalyst [17].

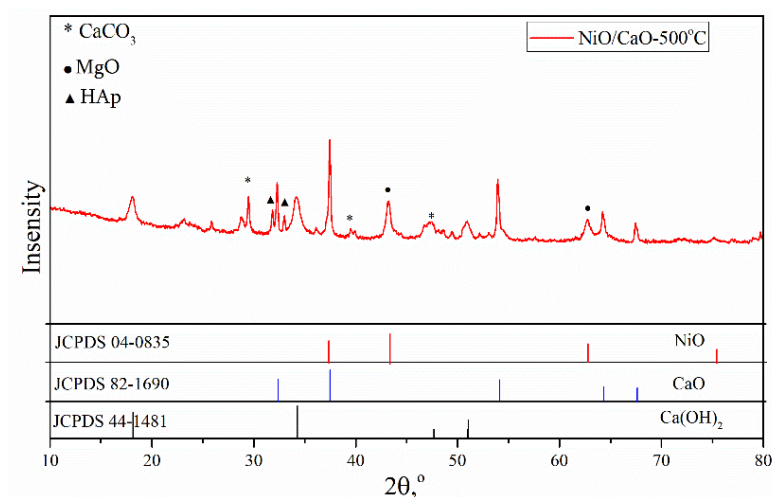


Figure 3. XRD spectrum of CaO before impregnation and the NiO/CaO catalyst

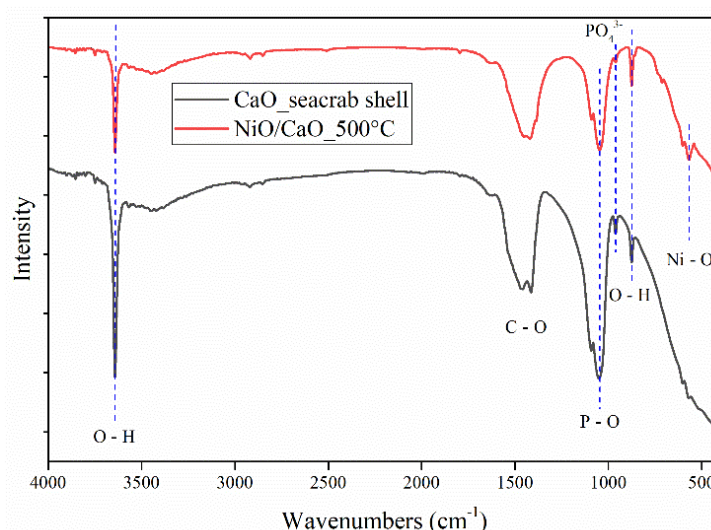


Figure 4. FTIR spectrum of calcined CaO and Ni/CaO

Figure 4 illustrated FT-IR spectra of calcined CaO (obtained from sea-crab shell) and NiO/CaO catalyst. The spectrum of the NiO/CaO catalyst displayed a strong absorption band at 3640 cm^{-1} , indicating the presence of hydroxyl groups (-OH). These hydroxyl groups can act as weak basic sites, facilitating the

conversion of triglycerides into methyl esters (biodiesel). Additionally, absorption bands at 1426 cm^{-1} were attributed to the unsymmetric stretching vibration of carbonate (CO_3^{2-}) groups, which may form due to carbonation when CaO is exposed to CO_2 from calcination of catalyst [18]. The FT-IR spectrum also showed a peak at 569 cm^{-1} , which corresponds to the Ni-O bond presented in catalyst [19].

3.2. Transesterification of waste cooking oil (WCO)

The effectiveness of the NiO/CaO catalyst was tested through transesterification of WCO. The effect of various reaction parameters, including reaction temperature, MeOH:oil molar ratio, catalyst loading, and reaction time, was performed to maximize biodiesel yield.

3.2.1. Effect of reaction temperature

The transesterification reaction was conducted at different temperatures, from 35 to 68 °C (near the boiling point of MeOH) and depicted in Figure 5. The results showed that the biodiesel yield increased significantly from 1.76% to 90.29% as the temperature increased from 35 to 68 °C. This temperature corresponds to the boiling point of methanol, at which mass transfer between methanol, oil, and catalyst is most effective [20]. Higher temperatures could favor the reaction rate, but new reactors should be applied such as autoclave or high-pressure fix-bed reactors.

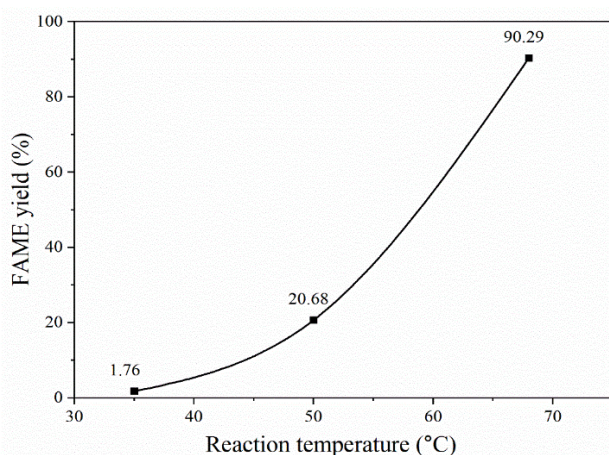


Figure 5. Effect of reaction temperature to FAME yield (Reaction condition: cat. = 10 wt.%, MeOH:oil = 24:1, τ = 3 hours)

According to Liu et al. (2008), the ideal temperature for producing biodiesel from soybean oil with a CaO catalyst was around 65 °C. The evaporation of methanol was cited by the authors as the reason why the biodiesel output started to decrease at temperatures over 65 °C [15].

3.2.2. Effect of reaction time

The effect of reaction time on biodiesel yield was evaluated under fixed conditions: a reaction temperature of 68 °C, a MeOH:oil molar ratio of 24:1, and a catalyst loading of 10 wt%. The results are presented in Figure 6, showing that the biodiesel yield increased with time. The FAME yield reached a maximum yield of 76.69 and 90.29% after 3 hours of reaction for CaO and NiO/CaO catalysts, respectively. As we extended the reaction time beyond 3 hours, the FAME yield slightly decreased, may be because of the reverse transesterification reaction or the formation of side products such as soap through saponification.

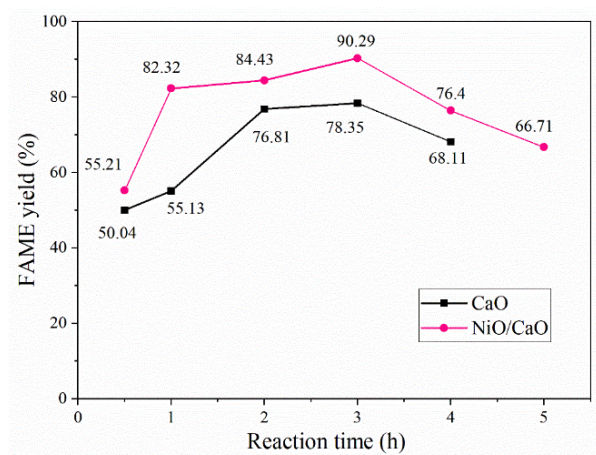


Figure 6. Effect of reaction time to FAME yield (Reaction condition: $T=68\text{ }^{\circ}\text{C}$, cat. = 10 wt.%, MeOH:oil = 24:1)

This behavior is consistent with findings reported in the literature. Liu et al. (2008) observed a similar trend, where the biodiesel yield increased by time, and reached maximum after 3 hours for the transesterification of soybean oil using CaO catalyst [15]. Similarly, Madhuvilakku and Piraman (2013) also found that biodiesel yield increased with time when using a $\text{TiO}_2\text{-ZnO}$ mixed oxide nanocatalyst, with an optimal reaction time of 5 hours, after which the yield began to decline, likely due to methanol evaporation and the onset of side reactions [21].

Figure 6. also showed that the addition of NiO to CaO facilitated the reaction rate significantly as the FAME yield was higher in the case of NiO/CaO.

3.2.3. Effect of MeOH:oil ratio

The MeOH:oil ratio is a critical factor influencing the transesterification reaction as it affects the operation cost of biodiesel production [22]. The ideal MeOH:oil ratio was determined by testing a range of ratios, from 6:1 to 30:1. The biodiesel output rose with the MeOH:oil ratio, according to Figure 7, reaching a maximum yield at a ratio of 24:1 [23]. The increase in the MeOH:oil ratio can increase the solubility of FAME in methanol, therefore, made it more difficult to separate the biodiesel from glycerol, and further resulted in a drop in the yield of biodiesel [24].

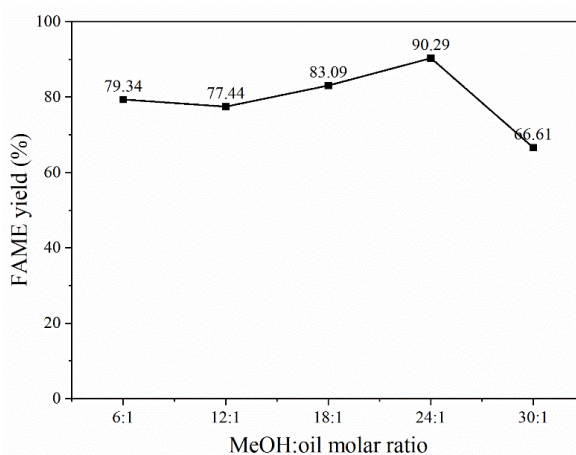


Figure 7. Effect of MeOH:oil Ratio to FAME yield (Reaction condition: $T = 68\text{ }^{\circ}\text{C}$, cat. = 10 wt.%, $\tau = 3$ hours)

Liu et al. (2008) reported that a MeOH:oil molar ratio of 12:1 was optimal for the transesterification of soybean oil when using CaO as a solid base catalyst [15]. Although raising the

MeOH:oil ratio at first increased the output of biodiesel, they observed that ratios greater than 12:1 had decreased in FAME yield [15].

3.2.4. Effect of catalyst loading

The catalyst loading was adjusted from 5 to 25 wt% to study its effect on FAME yield. As illustrated in Figure 8, increasing the catalyst loading to 10 wt% resulted in a considerable increase in FAME yield, and highest yield reached 90.29%. However, further increase in catalyst loading beyond 10 wt% resulted in a decrease in yield due to catalyst aggregation, which reduced the accessible surface area. Furthermore, excess catalyst can lead to the formation of soap, which competes with transesterification reaction for available methanol, and hence reduces overall yield [6].

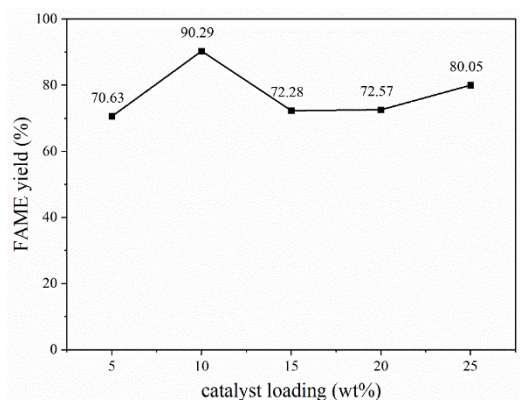


Figure 8. Effect of catalyst loading to FAME yield (Reaction condition: $T = 68\text{ }^{\circ}\text{C}$, MeOH:oil = 24:1, $\tau = 3$ hours)

4. CONCLUSION

This study successfully synthesized a NiO/CaO catalyst from sea-crab shells and proved its usefulness in the transesterification of WCO into biodiesel. The catalyst exhibited excellent activity, achieving a maximum biodiesel yield of 90.29% under the optimized reaction conditions. The addition of NiO to CaO support increased catalytic performance considerably by increasing the catalyst's basicity. The utilization of sea-crab shells as a raw material for CaO production is a sustainable and cost-effective approach for biodiesel synthesis that adheres circular economy principles. Besides, transesterification of WCO to biodiesel can help reduce environmental risk.

The findings indicate that the NiO/CaO catalyst is a good choice for biodiesel synthesis from WCO, particularly in developing countries where biowaste materials are abundant and readily available.

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TÓM TẮT

NGHIÊN CỨU TỔNG HỢP BIODIESEL TỪ DẦU ĂN PHẾ THẢI BẰNG XÚC TÁC TỔNG HỢP TỪ VỎ GHE VÀ NICKEL OXIDE

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Ngày nay, nhu cầu năng lượng toàn cầu ngày càng tăng và trữ lượng nhiên liệu hóa thạch giảm dần, các nguồn năng lượng thay thế ngày càng trở nên quan trọng. Nhờ được tạo ra từ các nguồn tái tạo và thân thiện hơn với môi trường, biodiesel được xem như một phương án thay thế triển vọng cho dầu diesel thông thường. Bài báo này nghiên cứu quá trình tổng hợp và ứng dụng xúc tác NiO/CaO được chiết xuất từ vỏ ghe trong sản xuất biodiesel. Dầu ăn phế thải (WCO) được sử dụng làm nguyên liệu trong quá trình phản ứng transester hóa. Xúc tác được tổng hợp thông qua phương pháp tâm ướt và được đánh giá đặc tính thông qua các kỹ thuật SEM, XRD, FT-IR và TG-DSC. Phản ứng transester hóa tối ưu trong nghiên cứu này đạt được hiệu suất FAME tối đa là 90,29% dưới các điều kiện: 68 °C, tỷ lệ mol methanol-dầu là 24:1, tải trọng xúc tác 10% khối lượng và thời gian phản ứng là 3 giờ. Kết quả này chứng tỏ xúc tác NiO/CaO là lựa chọn hiệu quả để chuyển hóa WCO thành biodiesel, cung cấp giải pháp năng lượng bền vững, tiết kiệm chi phí và thân thiện với môi trường.

Từ khóa: WCO, biodiesel, vỏ ghe, quá trình xúc tác, dầu ăn phế thải.