

## Catalytic Transesterification of Waste Cooking Oil using Fe-Modified Chicken Bone Catalyst: Characterization and Optimization

(Transesterifikasi Pemangkin Sisa Minyak Masak menggunakan Mangkin Tulang Ayam Diubah Suai Fe: Pencirian dan Pengoptimuman)

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### ABSTRACT

Food waste, including non-reusable materials like chicken bones, forms a significant portion of solid waste. In Malaysia, approximately 540,000 tons of waste cooking oil (WCO) is discarded annually without proper treatment. Chicken bones, rich in calcium, can be utilized as a heterogeneous catalyst in biodiesel production, addressing waste management issues. However, the use of chicken bone as a catalyst presents challenges such as the unmodified chicken bones often require a pre-treatment step to reduce high free fatty acid (FFA) content in WCO to prevent saponification, limiting their efficiency. Hence, this research endeavors to innovate by converting WCO into biodiesel via a transesterification reaction, leveraging waste chicken bones as a catalyst. The calcined waste chicken bone (CB) was modified to form 5 wt% Fe-CB, and 10 wt% Fe-CB. The catalysts were found to have similar physical characteristics in terms of the structure and surface morphology observed from XRD, N<sub>2</sub> adsorption-desorption, and SEM analysis. Among the catalysts, 10 wt% Fe-CB, produced the highest yield of fatty acid methyl esters (FAME), reaching 72.52%, under mild reaction conditions (10:1 methanol-to-WCO molar ratio, 1 wt% catalyst loading, 60 °C reaction temperature and 4 h reaction time). The capability of 10 wt% Fe-CB to produce a higher fatty acid methyl esters (FAME) yield than 5 wt% Fe-CB and calcined CB was due to the presence of CaO with binary transition metal oxides providing both acidic and basic sites, allowing for more efficient WCO conversion. Keywords: Fatty acid methyl esters; transesterification; waste chicken bone; waste cooking oil

### ABSTRAK

Sisa makanan, termasuk bahan yang tidak boleh diguna semula seperti tulang ayam, membentuk sebahagian besar sisa pepejal. Di Malaysia, kira-kira 540,000 tan sisa minyak masak (WCO) dibuang setiap tahun tanpa rawatan yang sewajarnya. Tulang ayam, yang kaya dengan kalsium, boleh digunakan sebagai mangkin heterogen dalam penghasilan biodiesel, sekali gus menangani masalah pengurusan sisa. Walau bagaimanapun, penggunaan tulang ayam sebagai mangkin menghadapi cabaran seperti tulang ayam yang tidak diubah suai sering memerlukan langkah pra-rawatan untuk mengurangkan kandungan asid lemak bebas (FFA) yang tinggi dalam WCO bagi mengelakkan saponifikasi yang membataskan kecekapan proses. Oleh itu, penyelidikan ini berusaha untuk berinovasi dengan menukar WCO kepada biodiesel melalui tindak balas transesterifikasi, memanfaatkan tulang ayam terbuang sebagai mangkin. Tulang ayam yang telah dikalsin (CB) diubah suai untuk membentuk 5 wt% Fe-CB dan 10 wt% Fe-CB. Mangkin ini didapati mempunyai ciri fizikal yang serupa dari segi struktur dan morfologi permukaan yang diperhatikan melalui analisis XRD, penyerapan-penyahjerapan N<sub>2</sub> dan SEM. Antara mangkin yang diuji, 10 wt% Fe-CB menghasilkan peratusan tertinggi ester metil asid lemak (FAME), mencapai 72.52% dalam keadaan tindak balas yang ringan (10:1 nisbah molar metanol-kepada-minyak, beban mangkin 1%, suhu tindak balas 60 °C dan masa tindak balas 4 jam). Keupayaan 10 wt% Fe-CB untuk menghasilkan asid lemak metil ester (FAME) yang lebih tinggi daripada 5 wt% Fe-CB dan CB terkalsin adalah disebabkan oleh kehadiran CaO dengan oksida logam peralihan binari yang menyediakan tapak berasid dan beralkali, membolehkan penukaran WCO yang lebih cekap. Kata kunci: Asid lemak metil ester; sisa minyak masak; sisa tulang ayam; transesterifikasi

## INTRODUCTION

Biodiesel is an alternative fuel derived from renewable sources. Since it is a biodegradable, renewable energy source and a non-toxic fuel with minimal exhaust emissions, biodiesel is a promising and potential energy source to replace fossil fuels (Alsharifi & Znad 2020; Ghanei et al. 2016). Biodiesel is typically derived from lipid sources such as vegetable oils and animal fats via a transesterification process with low-carbon alcohols (methanol or ethanol) aided by the presence of a homogenous or heterogeneous base catalyst (Hussain et al. 2021). The production of biodiesel from waste cooking oil (WCO) has gained significant attention as a sustainable alternative to using edible vegetable oils or animal fats, preserving valuable food resources and reducing environmental impact. Furthermore, it was reported that Malaysia disposes of around 540,000 tons of waste cooking oil (WCO) annually without adequate treatment (Suzihaque et al. 2022). However, the high free fatty acids (FFA) and water content of WCO pose a significant challenge in the transesterification reaction, which is the primary step in biodiesel production. Using high-FFA feedstocks leads to saponification, which reduces biodiesel yield. To overcome this challenge, the use of heterogeneous catalysts derived from biomass residues has emerged as a promising solution (Basumatary et al. 2023; Changmai et al. 2020) as they are more suitable for feedstocks with high FFA content greater than 1% and water content (Maheshwari et al. 2022).

Utilizing waste biomass-derived materials as heterogeneous solid catalysts is an excellent choice for more sustainable biodiesel production. Several wastes such as eggshells (Ayoola et al. 2019; Sulaiman et al. 2021), crab shells (Yuliana et al. 2021), dead coral (Zul & Hussin 2019), fish bones (Sulaiman, Jamaludin & Kabbashi 2019; Widiarti et al. 2017), chicken bones (Andas & Jusoh 2022), ostrich bones (Khan et al. 2020), and cow bones (Aghel et al. 2023) have been benefited as heterogeneous catalysts in the formation of biodiesel. The feasibility of chicken bones as a calcium source of biodiesel catalyst has been recently reported (Andas & Jusoh 2022; Basumatary et al. 2023). Bernama (2023) reported that food waste constitutes the largest category of disposed items, accounting for approximately 36%, with chicken bones comprising around 24% of this food waste. Chicken bones can be utilized as a biomass source for producing catalysts as they are rich in calcium in the form of oxides, alkoxides, carbonates and hydroxides calcium phosphate, which after calcination at high temperatures produces calcium oxide (CaO) (Bharti, Singh & Dey 2019). Andas and Jusoh (2022) reported the success of using chicken bones that have been treated to calcination-hydration-dehydration for biodiesel production from WCO.

However, the use of chicken bone as a heterogeneous catalyst comes with limitations. An esterification process catalyzed by concentrated acid such as  $H_2SO_4$  as a pre-treatment step was required to reduce the FFA content prior to the transesterification process for FAME formation using

the treated chicken bones (Andas & Jusoh 2022). This is due to the fact that chicken bone-derived catalysts are primarily composed of calcium oxide and thus highly basic. When WCO contains high levels of FFAs, the basic nature of chicken bones leads to saponification, a side reaction that forms soap instead of biodiesel, reducing the overall yield and efficiency of the process. This drawback of requiring a two-step process can be addressed by enhancing the acidic properties of the chicken bone catalyst.

The acidic property of biomass-derived catalysts with base properties can be enhanced through activation with transition metals, such as  $Fe^{2+}$ ,  $Zn^{2+}$ ,  $Co^{2+}$  or  $Ag^+$  (Borah et al. 2019; El-sherif et al. 2023; Satraidi et al. 2019; Xia et al. 2022), which can increase the acid site strength and improve the catalyst's effectiveness. This improved acidic property is required when a high FFA and water content of feedstock such as WCO is used in biodiesel formation. The activation with transition metals can create Lewis acid sites which can contribute to the enhanced catalytic activity of the biomass-derived catalysts (Pandian et al. 2020; Yang & Yu 2023). Introducing this functionality not only improves the acid site strength but enhances the catalyst's surface properties, such as porosity and hydrophobicity, which can further optimize the biodiesel production process (Amal & Usman 2024). Moreover, using a base/acid bifunctional catalyst eliminates the need for a separate acid treatment step for FFA, simplifying the process and reducing both reaction time and costs. This makes the process more economically viable and environmentally friendly (Enguilo Gonzaga et al. 2021).

In this study, continuing our focus on developing cost-effective heterogeneous catalysts (Elgubbi, Othman & Harun 2021; Yahya, Wahab & Harun 2020; Zahari et al. 2023), the waste biomass i.e., waste chicken bone is utilized as a heterogeneous catalyst to convert waste cooking oil to biodiesel. This research highlights the potential of using WCO in biodiesel production thus mitigating both environmental and economic issues.

## MATERIALS AND METHODS

### PREPARATION OF CATALYST FROM WASTE CHICKEN BONES

Catalyst from waste chicken bone was prepared following the method adapted from Farooq, Ramli and Naem (2015) and was modified accordingly. The chicken bones were washed to remove excess meat and fat from the bones and boiled in water for about 20-30 min to remove impurities or oil from the bones. The cleaned chicken bones were then left to dry on the pan to remove excess water before being dried under the sunlight for two consecutive days. By using a mortar and pestle, the dried bones were crushed and ground to a fine powder. The fine powder of chicken bone was calcined in a muffle furnace for 4 h at 900 °C. The calcined chicken bone later known as CB was stored in a desiccator prior to use.

#### PREPARATION OF MODIFIED CB CATALYST

A wet impregnation method was used to impregnate the CB powder with  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  precursor. During this process, a series of catalysts were prepared including the unmodified CB, 5 wt% Fe-CB, and 10 wt% Fe-CB. Firstly, 0.5 g or 1 g of iron precursor was added to 100 mL of distilled water in a 250 mL round bottom flask to produce 5 wt% Fe-CB, and 10 wt% Fe-CB, respectively. Then, 10 g of bone powder was added to each solution and impregnated at 80 °C for 6 h at 350 rpm to ensure complete homogeneity. Following vacuum filtration of the slurry, the solid was dried for 1.5 h at 100 °C and then calcined for 3 h at 600 °C (Satraidi et al. 2019).

#### CHARACTERIZATION OF CATALYSTS

X-ray diffractogram (XRD) (Rigaku Miniflex) was used to determine the crystallinity of the prepared catalyst. The composition of the catalysts produced was identified using x-ray fluorescence (XRF) (Bruker S8 TIGER), and field emission scanning electron microscopy (FESEM) (JSM-IT800) was used to analyze the surface morphological analysis of the catalyst. The surface area, porosity, and pore volume of the catalyst were determined by  $\text{N}_2$  adsorption/desorption analysis (Micromeritic).

#### CATALYTIC ACTIVITY

The activity of the catalysts was carried out under conditions: 60 °C reaction temperature, 10:1 methanol-to-oil molar ratio, 4 h reaction time, and 1 wt% of catalyst loading at 430 rpm agitation speed. Initially, WCO evaluated previously by Yahya, Razali and Harun (2019) was filtered to remove insoluble materials by using fine cloth and heated for 1 h at 100 °C. This WCO source was

reported to have ca. 3% of FFA content. Meanwhile, the catalyst was first activated in a two-necked 250 mL round bottom flask with a thermometer, condenser, and magnetic stirrer in the presence of methanol for 40 min at 40 °C. In the same flask, the preheated WCO was added to the solution and the reaction was carried out at 60 °C for 4 h.

After the reaction completion, the mixture was allowed to cool before being filtered using a vacuum filtration to separate the catalyst (Ghanei et al. 2016). The FAME product was collected and dried in an oven at 80 °C for 12 h to remove water by-products and excess methanol (Yahya, Wahab & Harun 2020). The sample mixture was transferred to a 250 mL separatory funnel and allowed to settle to separate into two distinct phases, glycerol at the bottom and biodiesel on top (Figure 1). The upper layer was kept in a sealed bottle for further analysis using gas chromatography (GC) equipped with a flame ionized detector (FID) and HP-INNOWAX column. The methyl esters in the product were quantified by comparing the peak areas of their corresponding standards and calculated using Equation (1) (Liu et al. 2015). The activity of 5 wt% Fe-CB, and 10 wt% Fe-CB were compared to unmodified CB catalyst.

$$\% \text{ yield} = \frac{m_{\text{FAME}}}{m_{\text{WCO}}} \times 100 \% \quad (1)$$

#### OPTIMIZATION OF BIODIESEL

The most active catalyst that produces the highest biodiesel yield was chosen to optimize the biodiesel yield. The parameters were varied for methanol-to-oil molar ratio (5:1, 10:1 and 20:1), catalyst loading in relation to the mass of oil (1, 3 and 5 wt%), and reaction time (2, 4 and 6 h) at a constant 60 °C reaction temperature.



FIGURE 1. Two separate layers of FAME (upper layer) and glycerol (bottom layer)

## RESULTS AND DISCUSSION

## CATALYST CHARACTERIZATION

The crystallinity structures of CB, 5 wt% Fe-CB, and 10 wt% Fe-CB catalysts were examined using an XRD pattern, as shown in Figure 2. The XRD patterns showed that the prepared catalysts had a high crystallinity, as evidenced by sharp, narrow, and distinct peaks in the diffraction patterns. All peaks that appeared in the XRD patterns were referred to the Joint Committee on Powder Diffraction Standards (JCPDS). It was discovered that the peaks observed indicate the presence of a mixture of hydroxyapatite ( $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ ), tricalcium phosphate beta- ( $\text{Ca}_3(\text{PO}_4)_2$ ), calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ), and calcium oxide ( $\text{CaO}$ ). The formation of these compounds could serve as a potential catalyst for the transesterification of WCO (Bitire, Jen & Belaid 2021; Farooq, Ramli & Naeem 2015). No diffraction peak that could contribute to the presence of calcium carbonate ( $\text{CaCO}_3$ ) from chicken bone was observed; thus, it indicates  $\text{CaCO}_3$  phase underwent a complete transformation into a crystalline phase (Amal et al. 2024). The Fe-modified CB samples show no new peak on the XRD patterns. The absence of diffraction peaks corresponding to the metal nitrate  $\text{Fe}(\text{NO}_3)_2 \cdot 9\text{H}_2\text{O}$  in the XRD patterns of all the prepared catalysts suggests that the metal ions are uniformly dispersed across the surface of the chicken bone with very small crystallite size which cannot be observed by XRD (Nur 2006; AlSharifi & Znad 2020). This observation was supported by SEM photographs shown in Figure 3.

FESEM was used to examine the surface morphologies of the prepared catalysts which are shown in Figure 3. The surface morphology of the CB and modified CB catalysts were arranged in sequence and considered as crystallinity parallel due to the high-temperature calcination (Bitire, Jen & Belaid 2021; Tan et al. 2019). The surface of the

catalysts shows a heterogeneous distribution of spherical-shaped and rod-shaped particles of irregular size that appear in such a non-uniform manner that they cover almost the entire surface of the catalyst. This is consistent with a study by Sulaiman et al. (2021), which found that ZnO/CaO catalysts have a mixture of spherical and rod-like structures.

XRF was performed to determine the composition of elements present in the catalysts produced. XRF analysis in Table 1 shows that Ca and P were the most abundant elements in the CB, 5 wt% Fe-CB and 10 wt% Fe-CB catalysts. Based on the XRD results, these Ca and P exist as  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ , beta- ( $\text{Ca}_3(\text{PO}_4)_2$ ),  $\text{Ca}(\text{OH})_2$ , and  $\text{CaO}$ . Other elements were detected in trace amounts which are less than 2%. The presence of trace elements in the samples could be beneficial in increasing the catalytic activity of the catalysts produced (Hart 2020). The significant increase of Fe element for 10 wt% Fe-CB sample from ca. 0.08% to 1.96% indicates that the impregnation of  $\text{Fe}(\text{NO}_3)_2 \cdot 9\text{H}_2\text{O}$  was successfully conducted. However, the amounts of Fe detected by XRF are much lower than the amount of Fe used in the impregnation step and this could be due to the leaching of the impregnated Fe that occurs during the post-impregnation step. Selpiana et al. (2023) claimed that the filtration step conducted after impregnation in the wet impregnation process for Zn/ $\text{Al}_2\text{O}_3$  catalyst formation resulted in a significant loss of Zn ions with distilled water.

The physical characteristics of the prepared catalyst as shown in Table 2 were analyzed by  $\text{N}_2$  adsorption/desorption analysis. The catalysts produced in this work have a high BET surface area that indicates the compositional transformation of the calcined  $\text{CaO}$ , in which  $\text{CO}_2$  molecules are released during the high-temperature calcination process (Laskar et al. 2018). The BET surface area was slightly altered for the modified CB compared to the unmodified CB and this was probably

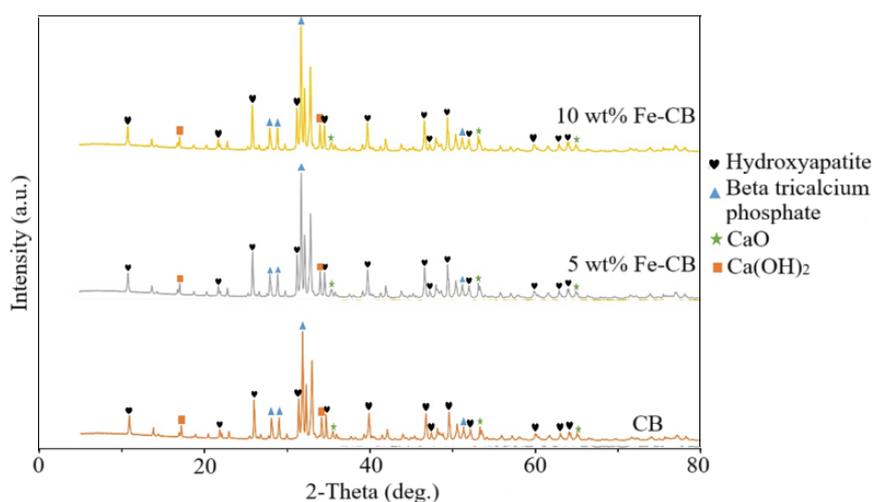


FIGURE 2. XRD patterns of catalysts

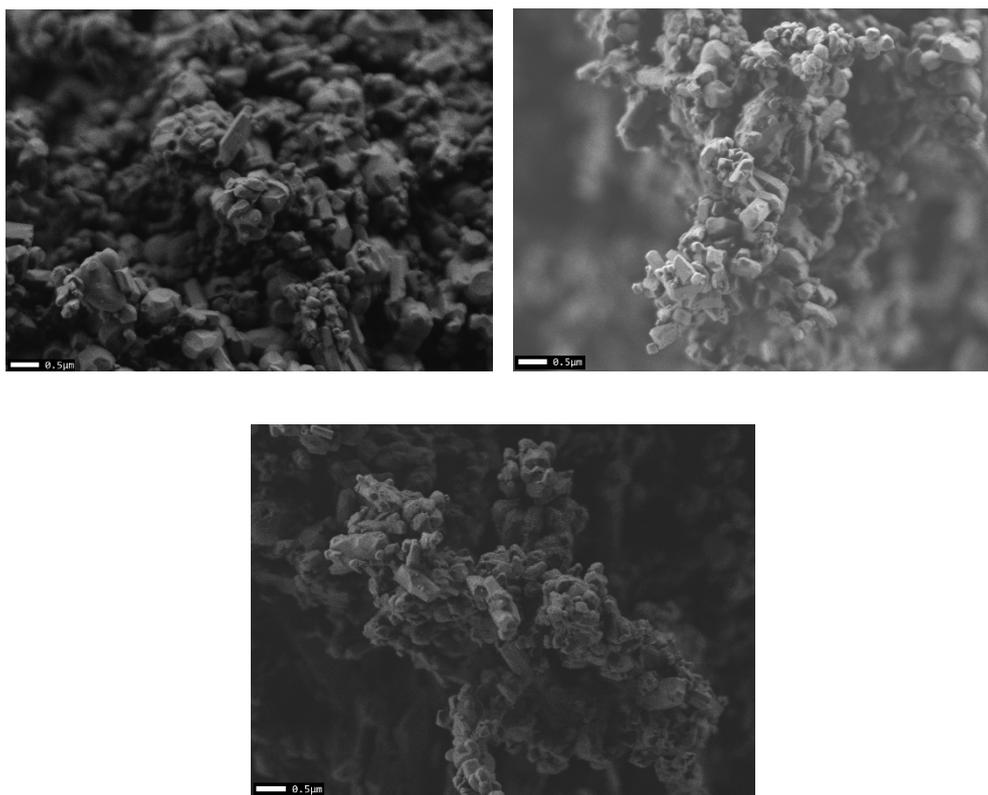


FIGURE 3. FESEM micrographs of the catalysts: (a) CB, (b) 5 wt% Fe-CB and (c) 10 wt% Fe-CB with magnification 15000x

TABLE 1. The compositions of catalysts determined by XRF technique

Composition (%)	CB	5 wt% Fe-CB	10 wt% Fe-CB
Ca	44.9	42.6	40.7
P	32.5	30.2	29.7
Fe	0.08	1.06	1.96
Mg	1.31	0.67	0.61
Na	1.06	0.65	0.62
Si	0.06	0.06	0.05
Cu	0.008	0.008	0.008

TABLE 2. Structural characteristics of the prepared catalysts

Catalyst	Surface area (m <sup>2</sup> /g)	Pore size (Å)	Pore volume (cm <sup>3</sup> /g)
CB	197	44	0.21
5 wt% Fe-CB	180	46	0.21
10 wt% Fe-CB	182	47	0.21

due to the covering of the CB pores with the metal ions (AlSharifi & Znad 2020). At the same time, the pore size and pore volumes were unaffected by the presence of  $\text{Fe}^{2+}$  in the pore structure of CB. The insignificant difference in surface properties could be due to the small ionic size of  $\text{Fe}^{2+}$  (ca. 0.078 nm) to be effectively inserted in the pore structure of CB with the average pore size between 44 and 47 Å (ca. between 4.4 and 4.7 nm) and Fe species should adhere to both the internal and external surfaces (Cao et al. 2019). This finding is consistent with the XRD results as there is no change in the diffraction planes between CB and Fe-CB samples indicating no accumulation of iron complex across the surface of chicken bone. Besides, the catalysts' average pore sizes are observed to be less than 50 Å (<5 nm), which is classified as mesoporous material (Bashah et al. 2019).

#### CATALYTIC ACTIVITY

All samples were screened for the transesterification reaction of WCO at constant reaction conditions: a reaction temperature of 60 °C, methanol-to-oil molar ratio of 10:1, reaction time of 4 h and catalyst loading of 1 wt%. It was found that the modified CB produced more biodiesel yield than the unmodified CB, where the highest biodiesel yield was obtained from 10 wt% Fe-CB (72.52%), followed by 5 wt% Fe-CB (45.67%) and CB (40.53%), as shown in Table 3.

The higher production of biodiesel when using the modified catalysts than the unmodified CB can be explained by the theory that the binary conjugation of transition metal ion ( $\text{Fe}^{2+}$ ) and base metal oxide (CaO) from CB offered excellent acidic and basic active sites for the conversion of high FFA feedstocks, such as WCO, in transesterification reaction (Mansir et al. 2017). It was anticipated that  $\text{Fe}^{2+}$  would create an acidic active phase that could esterify FFA in WCO, and that CaO from CB would supply the basic site needed to convert triglycerides to FAME. Strong Lewis acid sites such as those found in  $\text{Fe}^{2+}$  were also mentioned by Yassin et al. (2015) as favoring the more effective esterification process. The presence of notable Fe element in the Fe-CB catalyst's from XRF analysis served as evidence. Additionally, the presence of additional trace acidic oxides such as  $\text{SO}_3$ ,  $\text{P}_2\text{O}_5$ , and  $\text{SiO}_2$  enhanced the catalysts' acidic properties and increased the yield of biodiesel (Mustapha et al. 2022).

Besides, 10 wt% Fe-CB yields substantially more biodiesel than 5 wt% Fe-CB. This resulted from the iron content in 10 wt% Fe-CB being higher than in 5 wt% Fe-CB. Ahmed et al. (2022) claimed that the number of active sites on the catalyst is directly proportional to the percentage of iron content. Based on XRF analysis, it was found that 10 wt% Fe-CB, which has a higher iron content, offers more active sites for the reaction. Consequently, this plays a major part in enabling a greater conversion of triglycerides into biodiesel (Fatimah et al. 2023). The catalytic cycle for the transesterification reaction of WCO into FAME on Fe-CB catalyst is shown in Figure 4. The cycle depicts several steps, such as adsorption of the oil on  $\text{Fe}^{3+}$ , methanol activation, attack by methanol to form intermediates, and the formation of FAME. Additionally, 10 wt% Fe-CB has a slightly larger surface area than 5 wt% Fe-CB, according to the  $\text{N}_2$  adsorption/desorption study. It has been demonstrated that 10 wt% Fe-CB catalyst provides more accessible active sites, which increase the catalyst-reactant contact point during the reaction and may lead to higher conversion rates (Ahmed et al. 2022). Therefore, 10 wt% Fe-CB was selected to be further optimized in terms of reaction parameters to produce biodiesel.

#### OPTIMIZATION OF BIODIESEL

##### *Effect of Catalyst Loading*

The effect of catalyst loading on the transesterification of WCO was examined using 10 wt% Fe-CB at various catalyst loadings i.e., 1, 3, and 5 wt%. The reaction was carried out at the conditions of 60 °C reaction temperature, methanol-to-oil molar ratio of 10:1 and reaction time of 4 h. According to Figure 5, the analysis showed that the FAME yield of 1 wt% of 10 wt% Fe-CB catalyst had the highest yield (72.52%) and the yield for 5 wt% was the lowest (38.20%). A notable decrease in FAME yield was observed upon adding catalyst from 3 wt% to 5 wt%. This is likely due to the catalyst clumping together within the system, causing the reaction mixture to become more viscous (Lokman et al. 2014). As a result, the catalyst's surface area was decreased due to the agglomeration of particles, making it more difficult for the reactants to mix and diffuse toward the active sites of the catalyst. This is consistent with the finding reported by Ezzah-Mahmudah et al. (2016). Another possible explanation for the decreasing FAME levels in this system might be the saponification

TABLE 3. Percentage of biodiesel yield from catalyst produced

Catalyst	FAME yield (%)
CB	40.53
5 wt% Fe-CB	45.67
10 wt% Fe-CB	72.52

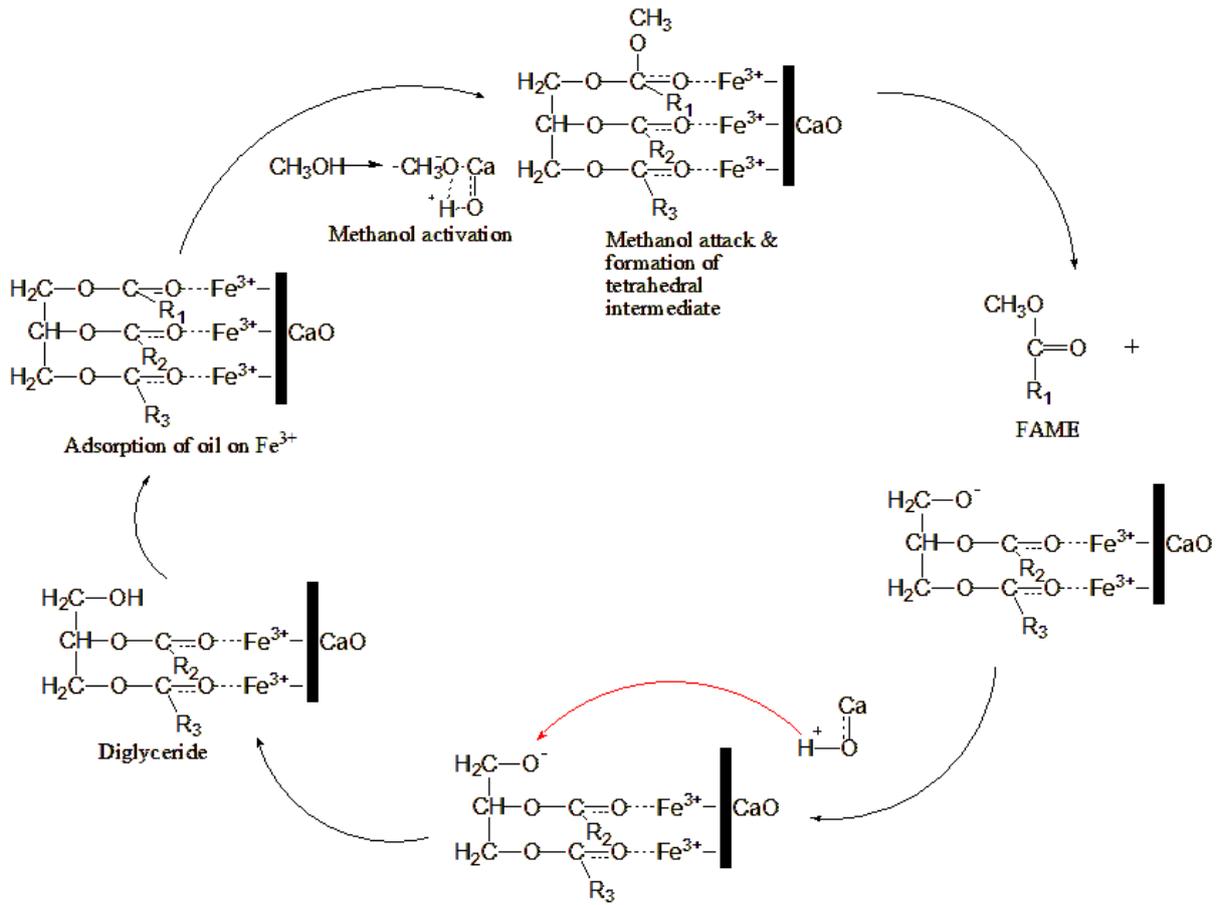


FIGURE 4. Catalytic cycle of Fe-CB catalyzed transesterification reaction

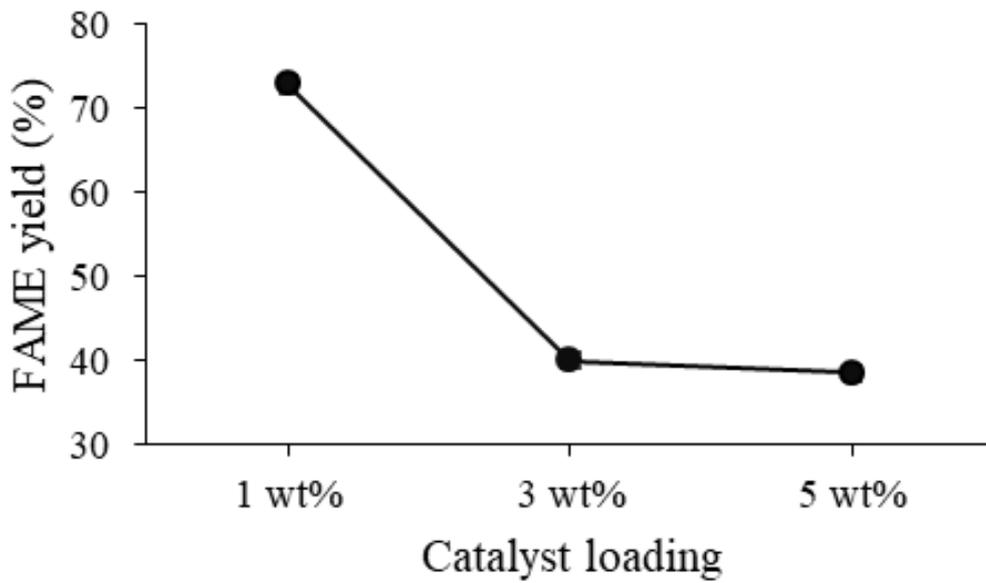


FIGURE 5. Percentage of FAME yield from different catalyst loading

reaction between FFA in the WCO feedstock and the basic sites of the CaO in the CB catalyst. This reaction occurs at a high catalyst loading and restricts mass transfer (Changmai et al. 2020). Considering this outcome, 1 wt% catalyst loading of 10 wt% Fe-CB was chosen for further study to examine the effect of other reaction parameters.

#### EFFECT OF METHANOL-TO-OIL MOLAR RATIO

The effect of methanol-to-oil molar ratio was investigated using the ratio of 5:1, 10:1, and 20:1. The yield of biodiesel is shown in Figure 6. The constant reaction conditions were 60 °C, catalyst loading 1 wt% and reaction time of 4 h. Theoretically, the transesterification reaction process needs one mole of triglyceride to react with three moles of methanol to produce one mole of FAME and three moles of glycerol as a by-product (Ezzah-Mahmudah et al. 2016). It was observed that the 10:1 methanol-to-oil molar ratio recorded the highest biodiesel yield. A sharp decrease in yield was observed with an increase in the molar ratio to 20:1. The optimum methanol quantity was 10:1 suggests that the reaction was driven towards the completion of FAME formation. However, an excess of methanol of more than 10:1 creates excess reactant which favors the reverse direction according to Le Chatelier's principle, resulting in the reduction of FAME yield (Ezebor et al. 2014). Additionally, an excess of methanol can inhibit the phase separation of glycerol and biodiesel, while moisture content in the methanol solution may induce undesirable hydrolysis of the FAME, which further reduces the

biodiesel yield (Ghoreishi & Moein 2013). Furthermore, the insolubility of methanol in the oil could dilute or deactivate the catalyst in the reaction, decreasing the catalyst efficiency (Essamlali et al. 2019). By referring to the trend of the results, it was obvious that the methanol-to-oil molar ratio played an important role in increasing the FAME yield. Therefore, a methanol-to-oil ratio of 10:1 was used to optimize the reaction time.

#### EFFECT OF REACTION TIME

The effect of reaction time was investigated using reaction time at 2 h, 4 h, and 6 h, as shown in Figure 7. The constant reaction conditions were 60 °C, catalyst loading 1 wt% and methanol-to-oil molar ratio of 10:1. This analysis showed that the FAME yield increased significantly between 2 h and 4 h. However, the yield decreased as the duration increased from 4 h to 6 h. This may have resulted from increased FAME production during the initial stages of the reaction, which reached equilibrium at 4 h. Nevertheless, the biodiesel yield was only 46% when the reaction time was further increased. Yaşar (2019) suggests that a backward reaction that produced more glycerol than FAME was likely the cause of the reduction in catalytic performance. Furthermore, as the reaction time increases, side product formation also increases and it may become more difficult to remove the separated glycerol layer if it dissolves in the ester layer (Haq et al. 2021). Therefore, the ideal reaction time for the transesterification reaction using this synthesized catalyst from chicken bone waste is 4 h.

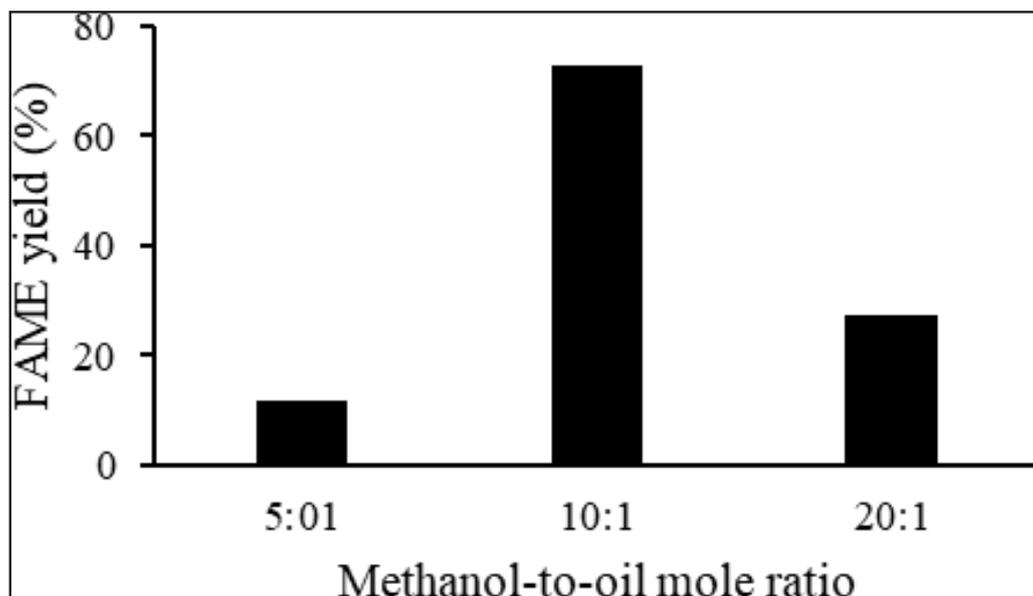


FIGURE 6. Percentage of FAME yield from different methanol-to-oil ratio

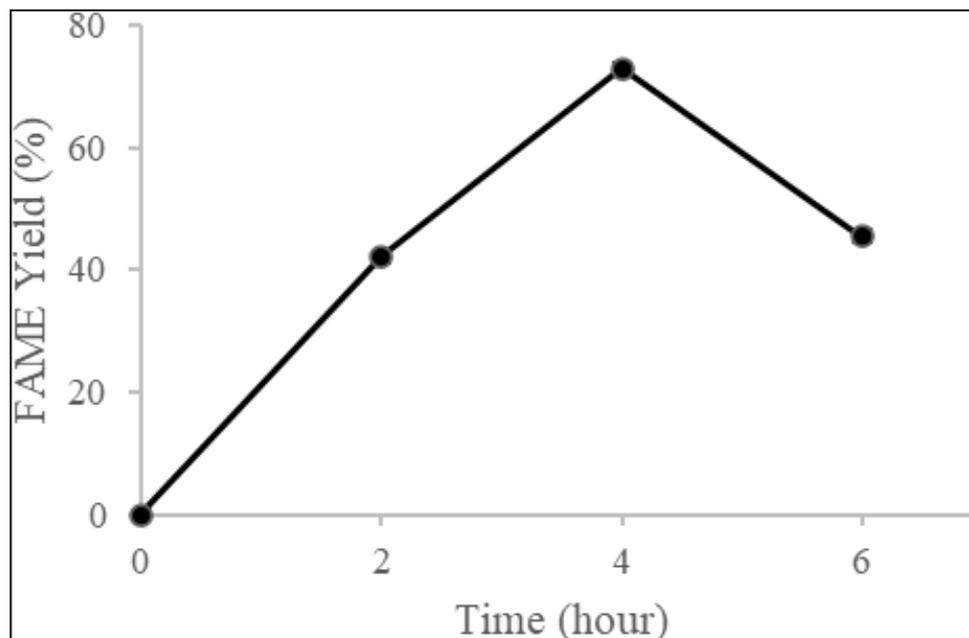


FIGURE 7. Percentage of FAME yield from different reaction times

#### CONCLUSION

In conclusion,  $\text{Fe}^{2+}$  modified on CB catalyst with two distinct weight percentages i.e., 5 wt% Fe-CB and 10 wt% Fe-CB were successfully synthesized from waste chicken bone and characterized. The modified catalyst surpassed pure CaO in terms of biodiesel yield from the WCO, with the 10 wt% Fe-CB catalyst producing the highest FAME yield. The research indicates that the optimal conditions for transesterification reaction are 10:1 methanol-to-oil molar ratio, 1 wt% catalyst loading, and a 4 h reaction time. It has been demonstrated that evenly distributed iron on the surface of the CB catalyst acts as an active site, enhancing the catalyst's capacity to catalyze transesterification for the synthesis of biodiesel. The results showed that it is possible to produce FAME from a high FFA content of WCO by using Fe-CB as a catalyst with further study focusing on optimizing the impregnation method of  $\text{Fe}^{2+}$  on the calcined chicken bone.

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