Green composite Fe_3O_4/CaO (0.5 g) for biodiesel production with preliminary structural, functional and morphological characterization

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Abstract: The development of environmentally friendly heterogeneous catalysts is crucial to support sustainable biodiesel production. This study aims to develop an eco-friendly Fe_3O_4/CaO (0.5 g) composite synthesized from local iron sand of Tulungagung as the Fe_3O_4 source and waste eggshells as the CaO source for potential catalytic applications in biodiesel production. The synthesis was carried out through a simple method while maintaining sustainability aspects by utilizing natural resources and agricultural waste. The composite was characterized using X-ray Diffraction (XRD) to identify the crystal structure, Fourier Transform Infrared Spectroscopy (FTIR) to investigate functional groups, and Scanning Electron Microscopy–Energy Dispersive X-ray Mapping (SEM–EDX Mapping). The results showed that the obtained composite consisted of two dominant phases, namely Fe_3O_4 as the magnetic phase and CaO as the active basic phase, with average crystallite sizes of 10.08 nm and 31.07 nm, respectively, indicating a high degree of crystallinity. FTIR analysis confirmed the presence of characteristic Fe–O and Ca–O functional groups at the wavenumber range of 500–600 cm^{-1}, while SEM images revealed an agglomerated oval-spherical morphology with an average particle size of 57.36 ± 0.85 nm. EDX analysis further confirmed the presence of Fe, O, and Ca elements, with Ca distribution, though relatively small, remaining consistent as active basic sites. The combination of crystalline properties, nanometer-scale morphology, and elemental composition supports the role of Fe_3O_4/CaO as a heterogeneous catalyst that is not only easily separable by a magnetic field but also potentially enhances catalytic activity in transesterification reactions. Therefore, this material demonstrates strong prospects as an environmentally friendly heterogeneous catalyst derived from local resources to support sustainable biodiesel production.

Keywords: Fe_3O_4/CaO, iron sand, eggshells, biodiesel, green composite.

1. Introduction

In the past decade, research related to renewable energy has increasingly attracted the interest of many researchers across the globe. This growing interest is inseparable from the fact that global energy demand continues to rise in line with population growth and industrial development (Jie et al., 2023; Solarin, 2020; X. Wang et al., 2024). On the other hand, fossil fuel reserves, which have long served as the primary energy source, are increasingly depleting, while their utilization generates significant negative impacts on the environment, such as rising greenhouse gas emissions, air pollution, and climate change (Murphy, 2024) (Heras & Gupta, 2024). This circumstance has prompted concerted efforts to explore and advance alternative energy sources that can not only address long-term energy demands but also ensure environmental compatibility and sustainability. Accordingly, renewable energy technologies including biomass, solar, wind, hydro, and those based on cutting-edge functional materials have emerged as central themes in scientific investigations, ranging from fundamental laboratory research to industrial-scale implementation (Almrafee & Akaileh, 2024; Rahmawati et al., 2020; Shahid et al., 2025; Sher et al., 2025; Zhou et al., 2025).

One of the renewable energy sources that has received considerable attention over the past two decades is biodiesel. This fuel can be produced from vegetable oils, animal fats, or waste cooking oil, all of which are abundantly available and renewable. Thus, biodiesel not only reduces dependence on fossil fuels but also offers the potential to mitigate waste-related problems. (Cerón Ferrusca et al., 2023). From a chemical perspective, biodiesel primarily consists of fatty acid methyl esters (FAME), synthesized via the transesterification of triglycerides with short-chain alcohols—most commonly methanol—as the alcohol reagent (Rahim et al., 2024). In comparison with fossil-derived diesel, biodiesel exhibits several notable advantages: it is biodegradable, non-toxic, possesses a relatively high cetane number, and significantly contributes to the reduction of exhaust emissions—most prominently carbon monoxide, unburned hydrocarbons, and particulate matter (Rozina et al., 2023). An additional merit that underscores the growing relevance of biodiesel lies in its compatibility with fossil diesel, as it can be utilized in blends without necessitating significant modifications to conventional diesel engine systems (Wirawan et al., 2024). Accordingly, biodiesel represents not only a sustainable energy alternative but also a practical and immediately deployable solution to facilitate the global transition toward cleaner energy systems. However, its large-scale deployment remains constrained by several critical challenges, including high production costs, limited feedstock availability at the industrial scale, and suboptimal reaction efficiency, which is strongly influenced by the nature and performance of the catalyst utilized.

Homogeneous catalysts, notably NaOH and KOH, have been extensively utilized in transesterification processes owing to their high catalytic activity and their effectiveness in achieving rapid biodiesel conversion within comparatively short reaction durations (Encinar et al., 2022). Nevertheless, the application of homogeneous catalysts is constrained by several critical limitations, such as the requirement for costly purification steps, excessive water and oil consumption, and the production of substantial amounts of wastewater, thereby raising considerable environmental concerns (Kibar et al., 2023; K. Wang et al., 2024). These limitations have prompted researchers to explore heterogeneous catalysts, which offer greater environmental compatibility, reusability, and ease of separation from reaction products. Calcium oxide (CaO) has emerged as one of the most extensively investigated heterogeneous catalysts, primarily owing to its strong basicity, low cost, wide natural abundance, and relatively benign environmental profile(Basumatary et al., 2023). Moreover, CaO can be synthesized from diverse biomass-derived or solid waste precursors, including eggshells, seashells, and animal bones, which is consistent with the principles of renewable resource utilization and waste valorization (Zhang et al., 2023) (Hart & Aliu, 2022). However, despite its promising potential, the practical application of pure CaO is hindered by considerable technical limitations. The catalyst is prone to structural instability and a gradual loss of catalytic activity, primarily as a result of Ca^{2+} ion leaching into the reaction medium, which in turn diminishes its efficiency over successive reaction cycles (Chavez-Esquivel et al., 2025; Mazaheri et al., 2021). This challenge underscores the rationale for the continuous development and modification of CaO, aimed at enhancing its structural stability, prolonging its operational lifetime, and optimizing its catalytic efficiency in biodiesel production.

Conversely, Fe_3O_4-based materials offer the distinctive advantage of inherent magnetic properties, allowing for efficient catalyst recovery under an external magnetic field. This feature significantly minimizes the reliance on conventional separation techniques such as filtration or centrifugation, which are often laborious, time-consuming, and costly (Helmi & Hemmati, 2021). Such characteristics render Fe_3O_4 an appealing supporting component in the design of heterogeneous catalysts. Nevertheless, the majority of prior studies have employed synthetic Fe_3O_4, the relatively high production cost of which has constrained its feasibility for large-scale industrial applications. (Kang et al., 2024). Attempts to integrate Fe_3O_4 with CaO derived from low-cost local resources, such as eggshells or other biomineral wastes, remain relatively scarce, even though this valorization strategy holds strong potential to yield catalysts that are not only efficient but also economically viable. Moreover, much of the existing research has predominantly emphasized the evaluation of catalytic activity in transesterification reactions, while comprehensive studies addressing the structure performance correlation—linking structural, morphological, and chemical properties with catalytic behavior are still limited. This knowledge gap presents a major obstacle to the rational design and optimization of heterogeneous catalysts, as an in-depth understanding of the intrinsic material properties is essential for improving stability, extending operational lifetime, and ensuring sustainability in biodiesel production. Consequently, advancing the development of Fe_3O_4/CaO composites from locally sourced precursors, combined with thorough physicochemical characterizations, constitutes a pivotal step toward the realization of efficient, environmentally benign, and industrially applicable heterogeneous catalysts for sustainable energy transition

In light of these challenges, the present study introduces a green synthesis strategy for Fe_3O_4/CaO composites, employing locally available iron sand from Tulungagung as the Fe_3O_4 source and waste chicken eggshells as the CaO precursor. This strategy aligns with the principles of green chemistry and the circular economy while simultaneously valorizing underutilized local resources. The novelty of this work resides in the fabrication of a magnetically recoverable green composite, coupled with an in-depth characterization of its structural and morphological attributes. Such an approach is expected to yield fundamental insights into material–property relationships and, at the same time, contribute to the development of stable, reusable, and locally sourced sustainable catalysts for biodiesel production.

2. Research Methods

The synthesis of the Fe_3O_4/CaO composite in this study was carried out via a coprecipitation method using abundant local raw materials, namely iron sand from Tulungagung as the Fe source and chicken eggshells as the CaO precursor. The initial step involved the preparation of eggshells, in which the samples were thoroughly washed with running water to remove impurities and residual organic membranes, then sun-dried to significantly reduce moisture content. Subsequently, the eggshells were calcined in a furnace at 800 °C for 4 h to convert calcium carbonate ($CaCO_3$) into high-purity calcium oxide (CaO). A total of 0.5 g of the resulting CaO powder was then dispersed in 5 mL of ethanol under magnetic stirring to form a homogeneous suspension. This suspension was gradually titrated into the Fe precursor solution to prevent excessive agglomeration and ensure uniform CaO distribution. The Fe precursor solution was prepared by dissolving 20 g of iron sand in 58 mL of concentrated HCl under controlled conditions. The dissolution process yielded a concentrated brown FeCl solution, which was subsequently filtered using Whatman filter paper to obtain a clear filtrate of 18 mL as the source of Fe^{3+} and Fe^{2+} ions. The mixture of FeCl solution and CaO/ethanol suspension was then continuously stirred at room temperature to maintain system homogeneity. Coprecipitation was initiated by the slow addition of 25 mL NH_4OH solution until the system reached a basic condition (pH > 10), leading to the formation of a black Fe_3O_4/CaO precipitate. The precipitate was filtered and repeatedly washed with deionized water until neutral pH was achieved, ensuring the removal of residual ions and unreacted species. The resulting precipitate was dried at 100 °C in an oven for several hours to obtain structurally stable Fe_3O_4/CaO composite powder. The final product was comprehensively characterized to evaluate its physicochemical properties. X-ray diffraction (XRD) was employed to identify crystalline phases and crystallite sizes; scanning electron microscopy (SEM) combined with energy-dispersive X-ray spectroscopy (EDX) was used to examine surface morphology and elemental distribution; while Fourier-transform infrared spectroscopy (FTIR) was utilized to observe the functional groups formed. The magnetic properties of the material were analyzed using a vibrating sample magnetometer (VSM) to confirm its capability for catalyst recovery under an external magnetic field. The results of these characterizations were integrated to assess the potential of the Fe_3O_4/CaO composite as an environmentally friendly heterogeneous catalyst for biodiesel production.

3. Results and Discussion

The X-ray diffraction (XRD) pattern revealed that the sample consisted of two dominant phases, Fe_3O_4 (magnetite) and CaO (calcium oxide). The diffraction peaks at $2\theta \approx 30.1°$, 35.5°, 43.2°, 53.5°, 57.1°, and 62.6° correspond to the (220), (311), (400), (422), (511), and (440) crystal planes of Fe_3O_4, while the peaks at $2\theta \approx 32.2°$, 37.4°, and

53.9° indicate the presence of CaO (Istadi et al., 2015). This phase identification is further supported by the lattice parameters obtained, namely a = 8.366 Å for Fe_3O_4 and a = 4.808 Å for CaO, both of which are consistent with their respective reference values (8.37 Å and 4.81 Å) (Irianti et al., 2021; Sahadat Hossain et al., 2023), This confirms the successful formation of Fe_3O_4/CaO composite as a pure phase without additional phases. The lattice parameters were calculated using Bragg's law:

$$n\lambda = 2n \sin \Theta \qquad (1)$$

Furthermore, the crystallite size can be calculated using the Scherrer equation.:

$$D = \frac{K\lambda}{\beta cos\theta} \qquad (2)$$

The calculation results indicate that the average crystallite size is approximately 10.08 nm for the Fe_3O_4 phase and 31.07 nm for the CaO phase. These values are consistent with previous studies, which reported crystallite sizes in the range of 9–53 nm for Fe_3O_4 and 27–47.9 nm for CaO (Upadhyay et al., 2016), (Eddy et al., 2024; Singh & Sunil, 2025). The crystallite size within the nanometer range confirms that the synthesized sample is not amorphous but rather exhibits a high degree of crystallinity. This finding is consistent with the presence of sharp and well-defined diffraction peaks in the XRD pattern, further supporting the existence of crystalline structures in the Fe_3O_4/CaO material. The X-ray diffraction pattern of the synthesized sample is presented in Figure 1.

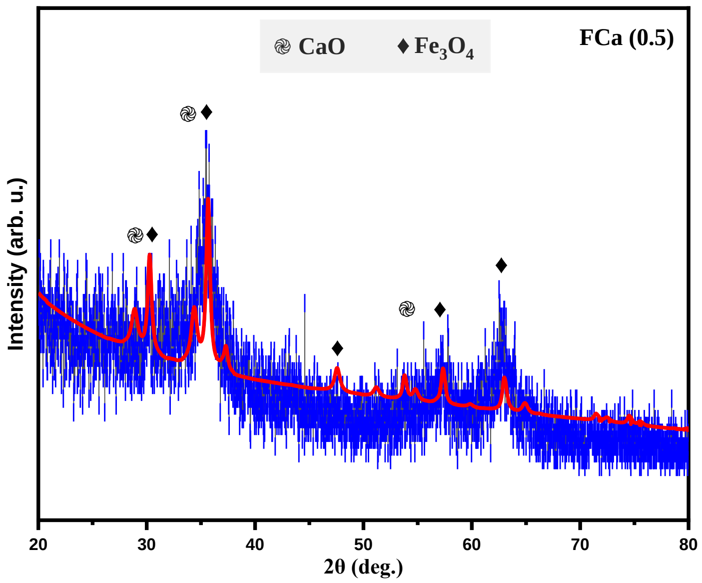

Figure 1. X-ray diffraction (XRD) pattern of the Fe_3O_4/CaO sample.

The results indicate that Fe_3O_4 serves as a magnetic phase that allows easy catalyst separation using an external magnetic field, while CaO acts as an active basic site essential for the transesterification reaction in biodiesel production. The crystallite size in the nanometer range provides a larger surface area, thereby enhancing the catalytic activity. This finding is further supported by FTIR analysis, which was employed to identify the functional groups present in the material, as shown in Figure 2.

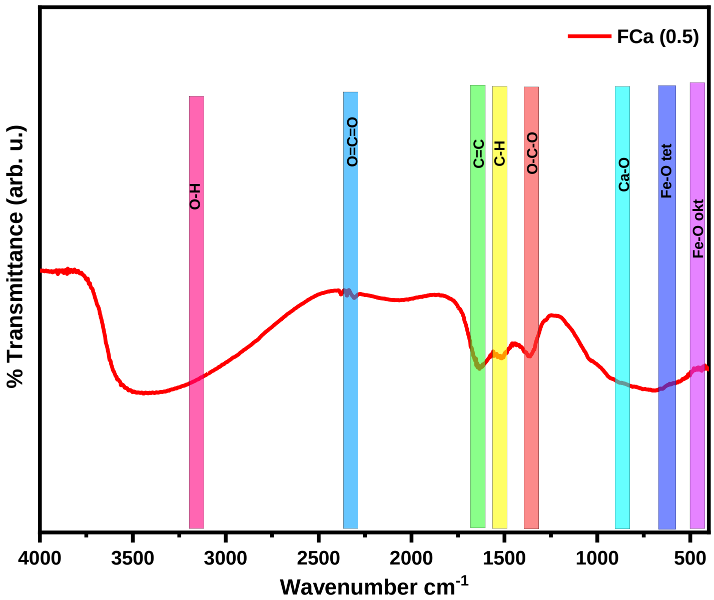

Figure 2. FTIR spectrum of Fe_3O_4/CaO material

The FTIR spectrum of the FCa(0.5) composite exhibits several characteristic absorption bands that confirm the presence of key functional groups on the material surface. The broad band in the range of 3400–3200 cm^{-1} is associated with O–H stretching vibrations, typically arising from adsorbed water molecules or hydroxyl groups on the surface of CaO and Fe_3O_4. This observation is consistent with previous studies reporting O–H bands at approximately 3425 cm^{-1} in CaO–Fe_3O_4 composite materials (Reyes-Vallejo et al., 2025). The band at 2920–2850 cm^{-1} indicates C–H stretching vibrations, which are likely derived from organic residues or minor contamination. In the region of 1650–1500 cm^{-1}, H–O–H bending bands are observed, indicating the presence of bound water molecules, while the band at 1380–1350 cm^{-1} is related to C–O symmetric vibrations of carbonate or residual precipitate anions. Furthermore, the band at 1100–1000 cm^{-1} can be attributed to C–O vibrations or Fe–O–C interactions, indicating the interphase interaction between Fe_3O_4 and CaO in the composite. In the low wavenumber region, around 500–600 cm^{-1}, strong absorption bands appear, corresponding to Fe–O vibrations of magnetite and Ca–O of calcium oxide, confirming the presence of both major phases in the composite. (Reyes-Vallejo et al., 2025). Thus, FTIR analysis confirmed that the FCa (0.5) composite consisted of Fe_3O_4 and CaO phases with characteristic functional groups of each component, without showing the presence of foreign functional groups. The presence of hydroxyl and carbonate groups, as well as interphase interactions are thought to play an important role in providing active sites and increasing surface stability, which ultimately supports the catalytic activity of the material in the biodiesel transesterification reaction. In addition to the characterization of functional groups through FTIR, SEM observations are presented in Figure 3 to evaluate the surface morphology and particle distribution of the FCa (0.5) composite, providing additional information regarding its microstructure and catalytic potential.

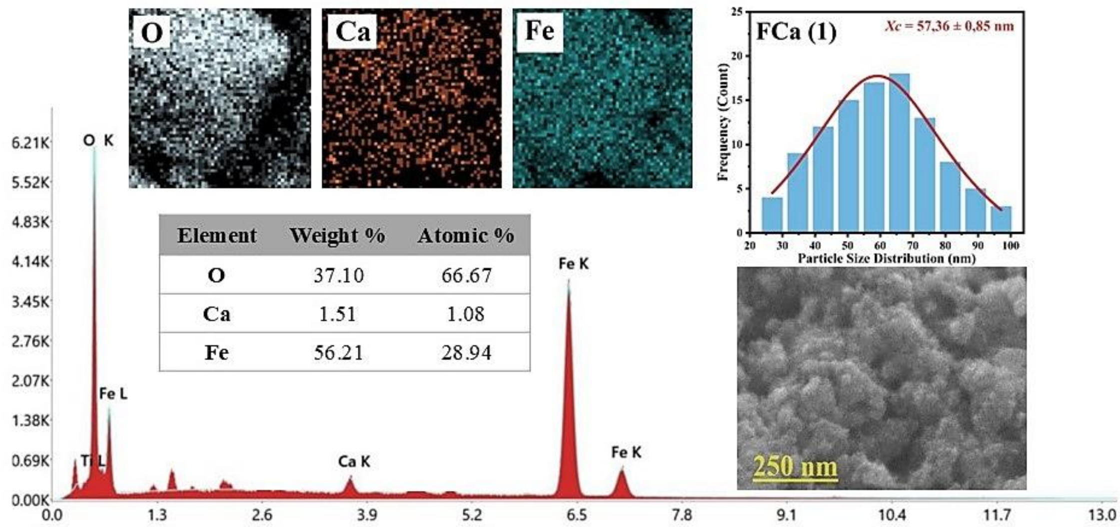

Figure 3. SEM images and EDX spectrum of the FCa (0.5) composite showing characteristic functional groups and surface morphology.

Observations showed that the FCa (0.5) sample consisted of spherical, oval agglomerates with an average particle size of 57.36 ± 0.85 nm, as determined by size distribution analysis. This morphology demonstrates the tendency of particles to agglomerate, a common phenomenon in iron oxide nanoparticles due to magnetic dipole interactions. However, the agglomeration formed was within reasonable limits and provided a large surface area, making it advantageous for catalytic applications and interface-based processes. EDX analysis revealed the presence of Fe, O, and Ca as the main components of the material. The quantification of the obtained elements is presented in Table 1.

Table 1. Results of EDX (Energy Dispersive X-ray Spectroscopy) analysis

Element	Weight %	Atom %
O	37,10	66,67
Ca	1,51	1,08
Fe	56,21	28,94

The EDX analysis results on the FCa(0.5) composite showed strong intensity peaks associated with the presence of Fe (Fe-L, Fe-Kα, Fe-Kβ) and O-K elements, while the small peak in Ca-Kα indicated the presence of calcium in relatively low amounts. This phenomenon is in line with the report of Helwani et al. (2020) which showed that the Fe_3O_4/CaO catalyst from eggshells had dominant Fe and O peaks, with Ca being detected consistently even in small amounts, indicating good dispersion of Ca on the material surface. (Helwani et al., 2020). The lower Fe/O atomic ratio compared to the magnetite stoichiometry in this analysis is thought to be due to the EDX detection bias towards light elements, as well as the contribution of surface species such as hydroxides or carbonates that are commonly formed in oxide-based catalysts. Similar findings were also reported by Hanif et al. (2023), who found that the nano-magnetic catalyst CaO/Fe_2O_3/feldspar exhibited a distribution of Fe, O, and Ca on the surface, confirming the successful integration of Ca without forming a significant amount of separate phase (Hanif et al., 2023). Thus, these EDX results strengthen the assumption that FCa(0.5) has a suitable composition and morphology for use as a magnetic heterogeneous catalyst, where the Ca dispersion on the surface is expected to play a role in providing active base sites while maintaining the magnetic properties of the material required for separation and practical application in biodiesel transesterification reactions.

4. Conclusion

The results of this study indicate that the FCa(0.5) composite was successfully synthesized with a crystalline structure consisting of two dominant phases, namely Fe_3O_4 as the magnetic phase and CaO as the active basic phase. XRD analysis revealed diffraction peaks consistent with reference data, with crystallite sizes of 10.08 nm for Fe_3O_4 and 31.07 nm for CaO, indicating a high degree of crystallinity. The presence of these two phases was further confirmed by FTIR, which exhibited characteristic absorption bands of Fe–O and Ca–O at wavenumbers 500–600 cm^{-1} along with several other supporting functional groups. Meanwhile, SEM observation showed an agglomerated morphology with oval-spherical shapes and an average particle size of 57.36 ± 0.85 nm, which still provides a large surface area. EDX analysis confirmed the main composition of Fe, O, and Ca, with Ca consistently dispersed on the surface, albeit in a relatively small amount. The combination of these characterization results indicates that FCa(0.5) possesses structural and morphological properties that support its performance as a heterogeneous magnetic catalyst. The presence of active basic sites from CaO enables the transesterification reaction, while Fe_3O_4 imparts magnetic properties that facilitate catalyst separation and reusability. The nanometer-scale crystallite size also contributes to a larger surface area and enhanced catalytic activity. Therefore, FCa(0.5) demonstrates strong potential for application in efficient, stable, and sustainable biodiesel production.

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