

Life Cycle Assessment of Biodiesel Production from *Dipterocarpus alatus* Oleoresin Using Potassium-Loaded Zeolite Y Catalyst Derived from Rice Husk Silica

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ABSTRACT

This study investigates the life cycle and catalytic performance of biodiesel production from *Dipterocarpus alatus* oleoresin using a heterogeneous base catalyst derived from potassium-loaded Zeolite Y synthesized from rice husk silica (K/NaY). The catalyst was prepared via wet impregnation of KOH onto NaY, followed by calcination at 550 °C for 4 h to ensure potassium dispersion and framework stability. Transesterification was performed under optimized conditions at a methanol-to-oil molar ratio of 16:1, 3 wt% catalyst loading, and 60 °C for 90 min. The maximum biodiesel yield achieved was 96.7%, while the catalyst maintained above 80% yield after four successive reuse cycles, demonstrating excellent reusability. Fuel characterization by thin-layer chromatography (TLC) and gas chromatography with flame ionization detection (GC-FID) revealed that methyl oleate and methyl palmitate were the dominant components, meeting key specifications of ASTM D6751 and EN 14214 standards. A cradle-to-gate life cycle assessment (LCA) was conducted using a functional unit of 1 MJ biodiesel, applying the ReCiPe 2016 midpoint methodology with SimaPro v9.3 software. Results showed a global warming potential (GWP) of 0.027 kg CO₂-eq/MJ, substantially lower than fossil diesel (0.094 kg CO₂-eq/MJ). Overall, the findings confirm both the catalytic efficiency and environmental sustainability of K/NaY catalysts derived from rice husk, highlighting their potential for scalable industrial biodiesel applications.

1. Introduction

The global transition toward clean and sustainable energy is imperative due to escalating climate change and the depletion of fossil fuel reserves. Among renewable alternatives, biodiesel derived from non-edible feedstocks has gained increasing attention for its potential to address the food-versus-fuel dilemma, reduce greenhouse gas (GHG) emissions, and promote energy security in rural areas. *Dipterocarpus alatus* oleoresin, locally known as Yang-na oil in Southeast Asia, offers a promising non-edible feedstock due to its high oil yield and favorable fatty acid profile [1]. However, the elevated content of free fatty acids (FFAs) in this oleoresin poses significant challenges for conventional base-catalyzed transesterification, often resulting in undesirable soap formation and reduced biodiesel yields [2].

To overcome these limitations, heterogeneous catalysts that are tolerant to FFAs and suitable for multiple reuses have been widely investigated. Among them, potassium-impregnated Zeolite Y (K/Zeolite Y) has emerged as a promising candidate. The physicochemical characteristics of Zeolite Y—such as large surface area, high thermal stability, well-defined microporosity, and tunable basicity—facilitate efficient transesterification [3-4]. Recent developments have also explored the use of alternative silica sources derived from agricultural residues for zeolite synthesis. In particular, rice husk silica (RHS), an abundant biogenic waste rich in amorphous silica, presents a sustainable and cost-effective precursor for zeolite production [5]. Utilizing RHS not only supports waste valorization but also aligns with the principles of the circular economy and green chemistry.

In this study, Zeolite Y was synthesized from RHS and impregnated with potassium via wet impregnation, followed by calcination, to obtain a solid base catalyst (K/NaY). This catalyst was then employed in the transesterification of *D. alatus* oleoresin under optimized conditions. In addition to catalyst performance and reusability, the study adopts a life cycle perspective to evaluate the broader environmental sustainability of the process. Furthermore, evaluating the sustainability of biodiesel production requires more than just laboratory-scale performance data. A systems-level approach, such as Life Cycle Assessment (LCA), is essential to quantify the environmental burdens associated with all stages of production—from raw material extraction to final product output. LCA is a standardized methodology under ISO 14040 that enables the comprehensive analysis of environmental impacts across multiple categories [6]. Its integration into catalyst development and process optimization is crucial for validating environmental claims associated with biofuel innovations [7].

While extensive research has been conducted on biodiesel production using both homogeneous and heterogeneous catalysts, relatively few studies have incorporated LCA frameworks to assess systems using waste-derived heterogeneous catalysts and non-edible feedstocks. Particularly lacking are investigations involving catalysts synthesized from agricultural waste materials such as rice husks. This study seeks to address this gap by combining experimental data on K/NaY synthesized from RHS with a cradle-to-gate LCA of biodiesel production from *D. alatus* oleoresin. The ReCiPe 2016 midpoint method, implemented through SimaPro v9.3 software, is employed to evaluate key impact categories such as global warming potential (GWP), fossil fuel depletion, human toxicity, and eutrophication [8]. The objective of this study is to perform a comprehensive life cycle assessment of biodiesel production from *Dipterocarpus alatus* oleoresin using a heterogeneous catalyst derived from rice husk silica-based Zeolite Y impregnated with potassium (K/NaY). This includes evaluating the environmental impacts of each process stage—feedstock extraction, catalyst synthesis, transesterification, and product separation—alongside assessing the catalytic efficiency and reusability of K/NaY in comparison with conventional base catalysts. Furthermore, the study benchmarks the environmental performance of this pathway against other biodiesel systems and identifies key environmental hotspots. Recommendations for system improvement are proposed in accordance with the principles of circular economy and green engineering, with the ultimate goal of supporting the development of a regionally adaptable, environmentally sound, and economically scalable biodiesel production system.

2. Experimental and Methods

2.1 Feedstock Preparation

In this study, the primary feedstock for biodiesel production was oleoresin directly tapped from the trunk of *Dipterocarpus alatus* Roxb. ex G.Don, a hardwood tree indigenous to Southeast Asia and commonly known as Yang Na. Unlike conventional seed-derived oils, this oleoresin is a naturally exuded, viscous fluid enriched with resinous compounds and fatty substances. Its application exemplifies a non-edible, underutilized biomass source with considerable promise for sustainable biofuel development, particularly in forest-abundant regions. The oleoresin was collected using traditional tapping methods involving shallow incisions along the tree trunk, allowing the exudate to drip into sterile glass vials. The collected material was subsequently stored in airtight containers at ambient conditions. Prior to chemical processing, the raw oleoresin was filtered through fine-mesh cotton to remove visible contaminants such as bark fragments and insects.

To preserve its native physicochemical integrity, no additional chemical pretreatments (e.g., degumming, bleaching, dewaxing) were employed.

Characterization of the raw oleoresin revealed a free fatty acid (FFA) content of 67.32 mg KOH/g oil—substantially exceeding the typical limit for efficient base-catalyzed transesterification. Its kinematic viscosity at 40°C was measured at 28.5 mm²/s, and the density at room temperature was found to be 0.918 g/cm³. These properties imply potential mass transfer limitations and miscibility issues with methanol during the reaction stage. Due to the elevated FFA level, the feedstock was unsuitable for direct transesterification with alkaline catalysts. Consequently, a pre-esterification step was incorporated to reduce the FFA concentration prior to biodiesel synthesis. The decision to use unrefined, unprocessed oleoresin aligns with the overarching goals of resource valorization and innovation within the circular bioeconomy framework. Moreover, it ensures that the life cycle assessment (LCA) reflects the actual environmental burdens associated with utilizing minimally processed, forest-derived biomass.

2.2 Materials, Equipment, and Chemicals

2.2.1 Instruments and Equipment

All experimental procedures were conducted using standardized laboratory equipment and high-precision analytical instruments to ensure accuracy, reproducibility, and experimental reliability. Common laboratory glassware included Erlenmeyer flasks (100–500 mL), beakers, graduated cylinders, volumetric pipettes, and separatory funnels (125–250 mL), which were utilized for general liquid handling, phase separation, and volume measurements. Heating and mixing were performed using a magnetic stirrer with an integrated hotplate (IKA C-MAG HS7, Germany), providing precise temperature control and uniform agitation. A high-speed centrifuge (Labnet Z323K, USA) with a maximum rotational speed of 10,000 rpm was employed for phase clarification and solid-liquid separation, particularly for catalyst recovery and purification. Catalyst characterization was carried out using an X-ray diffractometer (Bruker D8 Advance, Germany) equipped with Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$), operated at 40 kV and 40 mA. This instrument enabled the determination of crystallinity, phase purity, and potassium incorporation on the zeolite surface. For quantitative analysis of biodiesel composition, gas chromatography coupled with a flame ionization detector (GC-FID) was used (Agilent 7890A, USA), fitted with an HP-INNOWAX capillary column (30 m \times 0.32 mm \times 0.15 μm film thickness), allowing for the separation and detection of individual fatty acid methyl esters (FAMES).

Qualitative assessment of ester formation was conducted using thin-layer chromatography (TLC), employing silica gel 60 F254 pre-coated plates and a solvent-saturated chamber (Merck, Germany). This technique facilitated rapid identification of ester bands during preliminary product validation. All aqueous media and chemical preparations were performed using deionized water obtained from a Barnstead water purification system (Thermo Scientific, USA), providing a resistivity of 18.2 M Ω -cm, which is essential for minimizing ionic contamination in sensitive analytical operations. Reagents used in the experiments included potassium hydroxide pellets ($\geq 85\%$, ACS grade, Merck), methanol ($\geq 99.8\%$, HPLC grade, RCI Labscan), *n*-hexane ($\geq 95\%$, AR grade, Merck), and standard FAME mixtures (Supelco) for GC calibration. Phenolphthalein (Sigma-Aldrich) was used as a titration indicator during free fatty acid determination. The careful selection and application of these instruments and chemicals established a robust experimental platform for evaluating biodiesel synthesis efficiency, catalyst performance, and supporting the downstream environmental impact assessment outlined in subsequent sections.

2.2.2 Chemicals and Reagents

All chemicals used in this study were of analytical reagent (AR) grade and applied without further purification. Deionized water with a resistivity of 18.2 MΩ·cm, produced using a Barnstead purification system (Thermo Scientific, USA), was used in all synthesis, dilution, and washing procedures to ensure minimal ionic contamination. For the synthesis of silica and zeolite NaY, hydrochloric acid (37% HCl, Merck, Germany), sodium hydroxide (NaOH, ≥97%, Carlo Erba, Italy), and sodium aluminate (NaAlO₂, 55–56% Al₂O₃, Riedel-de Haën, Germany) were used alongside rice husk ash (RHA), which was prepared by acid leaching followed by calcination. In the catalyst preparation stage, a potassium acetate buffer was formulated using potassium acetate (CH₃COOK, 99%, Ajax Finechem, Australia), sodium acetate (CH₃COONa, 99%, BDH, UK), and glacial acetic acid (CH₃COOH, 100%, BDH, UK) to support the wet impregnation of potassium onto the zeolite structure. Methanol (≥99.8%, Merck, Germany) and concentrated hydrochloric acid (37% HCl, Merck) were employed in the esterification and transesterification reactions, while sodium sulfate anhydrous (Na₂SO₄, Carlo Erba, Italy) was used to remove residual water from the biodiesel phase. For qualitative analysis via thin-layer chromatography (TLC), petroleum ether and diethyl ether (JT Baker, USA), glacial acetic acid (BDH, UK), silica gel TLC plates (Silica Gel 60 F254, Merck, Germany), and iodine crystals (UNICROM, Thailand) were used, along with a FAME standard mix (C₈–C₂₄, Supelco, USA, Cat. No. 18918-1AMP) for band comparison. In gas chromatography with flame ionization detection (GC-FID), hexane (C₆H₁₄, Lab-Scan, Poland) and chloroform (CHCl₃, ≥99.9%, Mallinckrodt, USA) were used as solvents, while individual high-purity methyl ester standards (≥98%, Sigma-Aldrich, USA), including methyl palmitoleate, methyl linoleate, methyl oleate, methyl palmitate, methyl stearate, methyl pentadecanoate, methyl nonadecanoate, and methyl arachidate, were employed for peak identification and calibration.

2.3 Extraction of Silica from Rice Husk

Silica was extracted from agricultural rice husk waste through a sequence of acid leaching and thermal treatment steps designed to yield high-purity amorphous silica suitable for zeolite synthesis. The raw rice husk was first washed thoroughly with tap water to remove dust, soil particles, and surface-bound organic matter, followed by a rinse with deionized water to eliminate residual contaminants. The cleaned material was then oven-dried at 105 °C for 12 hours to remove moisture. To eliminate metal impurities—particularly potassium and calcium, which interfere with zeolite crystallization—the dried husk was subjected to acid leaching using 6 M hydrochloric acid (HCl, Merck, Germany) under reflux conditions at a solid-to-liquid ratio of 1:10 (w/v) for 3 hours. The acid-treated husk was washed repeatedly with deionized water until a neutral pH was achieved and subsequently dried again at 105 °C for an additional 12 hours.

The resulting material was then calcined in a muffle furnace at 550 °C for 6 hours in an air atmosphere to produce white rice husk ash (RHA) with high amorphous silica content. The RHA was finely ground using a mortar grinder and stored in sealed desiccators to prevent moisture uptake. Elemental analysis using X-ray fluorescence (XRF) spectroscopy confirmed the high purity of the silica, with minimal levels of residual metallic oxides. This biosilica, derived from rice husk, was subsequently used as the main silicon source in the synthesis of NaY zeolite. The method not only provides a sustainable and low-cost alternative to conventional silica reagents but also contributes to agricultural waste valorization and supports circular economy principles in catalyst development.

2.4 Synthesis of NaY Zeolite from Rice Husk-Derived Silica

NaY zeolite was synthesized via hydrothermal crystallization using amorphous silica derived from rice husk ash (RHA), following a carefully controlled gel formation and aging protocol. The synthesis began with the preparation of two precursor solutions: a sodium silicate solution and a sodium aluminate solution. The sodium silicate solution was obtained by gradually dispersing 7.90 g of extracted silica into a solution of 16.47 g deionized water and 3.16 g sodium hydroxide (NaOH, ≥97%, Carlo Erba, Italy), followed by vigorous stirring at ambient temperature for 48 hours to achieve complete dissolution. The solution was filtered to eliminate any insoluble residues, yielding a transparent sodium silicate solution. In parallel, the sodium aluminate solution was prepared by dissolving 1.76 g sodium aluminate (NaAlO₂, 55–56% Al₂O₃, Riedel-de Haën, Germany) into 2.59 g deionized water containing 2.45 g NaOH and 1.70 g potassium hydroxide (KOH, Ajax Finechem, Australia), ensuring homogeneity through continuous stirring.

The sodium aluminate solution was then added dropwise into the sodium silicate solution under vigorous mixing, forming a homogeneous white gel. The final molar composition of the gel was adjusted to a conventional NaY zeolite synthesis ratio of 10.67 Na₂O : Al₂O₃ : 10 SiO₂ : 180 H₂O. To initiate crystallization, the gel was divided into two parts: one part was aged at room temperature for 24 hours and designated as the seed gel, while the other served as the feedstock gel. Approximately 10 wt% of the seed gel was mixed into the feedstock gel, and the complete mixture was transferred into a sealed polypropylene bottle. The system was then subjected to hydrothermal treatment at 110 °C for 24 hours in a laboratory oven. Upon completion, the resulting crystalline solid was separated by centrifugation at 8000 rpm, washed thoroughly with deionized water until the filtrate reached pH 8.5–9.0, and subsequently dried at 110 °C for 12 hours.

The final product was a fine white powder of NaY zeolite, which exhibited sharp and well-defined diffraction peaks characteristic of the faujasite-type Y framework, as confirmed by X-ray diffraction (XRD) analysis. This synthesis strategy effectively transformed low-cost biomass-derived silica into high-crystallinity zeolite material, underscoring its potential as a sustainable precursor for heterogeneous catalyst development in biodiesel production.

2.5 Preparation of Potassium-Loaded NaY Catalyst (K/NaY)

The potassium-loaded NaY catalyst (K/NaY) was synthesized using a wet impregnation method with a buffered potassium acetate solution to achieve homogeneous potassium distribution and controlled ion exchange on the zeolite matrix. Prior to impregnation, the synthesized NaY zeolite was thermally pre-treated by drying at 110 °C for 2 hours in a convection oven to remove residual moisture. Precisely 1.6 g of the dried NaY powder was weighed and placed in a 50 mL beaker. A buffer solution was prepared using glacial acetic acid (CH₃COOH, BDH, UK) and sodium acetate (CH₃COONa, BDH, UK), which provided a controlled pH environment to stabilize the ion exchange and improve potassium anchoring. Varying amounts of potassium acetate (CH₃COOK, Ajax Finechem, Australia) were dissolved in 2.0 mL of the buffer to yield target potassium loadings of 4, 8, 12, and 16 wt% based on the NaY mass. The buffer solution was added dropwise to the NaY powder under continuous stirring to ensure uniform dispersion of the potassium precursor.

The resulting slurry was allowed to equilibrate at room temperature for 8 hours to promote complete penetration of potassium ions into the zeolite micropores. Following impregnation, the material was oven-dried at 80 °C for 12 hours to evaporate excess moisture. The dried material was then finely ground using an agate mortar to ensure

homogeneity, after which it was calcined in a muffle furnace at 550 °C for 5 hours in ambient air. This thermal treatment facilitated the decomposition of acetate ligands and enabled the formation of catalytically active potassium oxide (K₂O) species on the surface of the zeolite framework. The final K/NaY catalysts were stored in airtight containers under desiccated conditions to prevent moisture reabsorption prior to use. This synthesis protocol resulted in structurally stable, basic solid catalysts with tunable potassium content, designed to effectively catalyze biodiesel synthesis from feedstocks containing high levels of free fatty acids.

2.6 Catalyst Characterization by X-Ray Diffraction (XRD)

The crystalline structure and phase stability of both the synthesized NaY and potassium-loaded NaY (K/NaY) catalysts were analyzed via X-ray diffraction (XRD) using a Bruker AXS D5005 diffractometer (Germany) equipped with a Cu K α radiation source ($\lambda = 1.5406 \text{ \AA}$), operated at 40 kV and 40 mA. Prior to analysis, all catalyst samples—including those with potassium loadings of 4, 8, 12, and 16 wt%—were finely ground and uniformly pressed onto flat glass sample holders. XRD scans were conducted over a 2θ range of 5° to 50°, with a scan step of 0.05° and an acquisition time of 1 second per step. Approximately 1.5 g of each sample was prepared under identical conditions to maintain consistency in peak intensity comparison.

The resulting diffraction patterns revealed characteristic peaks corresponding to the faujasite-type structure of zeolite Y, with major reflections observed at 2θ values near 6.2°, 10.2°, 15.7°, 23.7°, 26.8°, and 31.3°. These features confirmed the successful crystallization of the NaY phase. Importantly, all K/NaY samples retained the essential structural integrity of the parent NaY even after high-temperature calcination at 550 °C, suggesting that the zeolite framework remained intact despite potassium incorporation. A gradual decrease in peak intensity was observed with increasing potassium content, which is indicative of minor structural distortion or partial pore blockage due to potassium species occupying internal channels or surface sites. No additional peaks associated with crystalline potassium compounds (e.g., K₂O or KOH) were detected, implying that the potassium existed primarily in an amorphous or well-dispersed state within the zeolite lattice. These findings support the conclusion that the wet impregnation method effectively introduced potassium into the NaY matrix without compromising the overall framework, making the resulting K/NaY catalysts structurally robust and suitable for base-catalyzed transesterification reactions.

2.7 Pre-treatment of Oleoresin via Acid-Catalyzed Esterification

Due to the exceptionally high free fatty acid (FFA) content in the *Dipterocarpus alatus* oleoresin—measured at 67.32 mg KOH/g oil—a pre-treatment step via acid-catalyzed esterification was essential to lower the FFA concentration prior to the base-catalyzed transesterification process. The purpose of this step was to convert the FFAs into their corresponding methyl esters, thereby minimizing undesirable soap formation and protecting the activity of the solid base catalyst in the subsequent reaction. In a typical batch, 20.0 g of filtered oleoresin was combined with 160.0 mL of methanol (CH₃OH, $\geq 99.8\%$, Merck, Germany) in a 250 mL Erlenmeyer flask, corresponding to an approximate molar ratio of oil to methanol of 1:8. A volume of 200 μ L of concentrated hydrochloric acid (HCl, 37%, Merck) was then carefully added as the homogeneous acid catalyst. The mixture was subjected to continuous stirring on a hotplate at 60 °C for 2 hours to ensure adequate reaction kinetics while minimizing methanol volatilization and thermal degradation of resinous compounds.

Following the esterification reaction, the mixture was transferred to a separatory funnel and allowed to settle for 24 hours at room temperature. This facilitated the formation of two distinct phases: an upper methanol-rich layer containing the methyl esters and a lower aqueous-resinous phase consisting of unreacted components and byproducts. The upper ester layer was separated, thoroughly washed with warm deionized water to remove residual acid, and subsequently dried using anhydrous sodium sulfate (Na₂SO₄, Carlo Erba, Italy) to eliminate moisture content. The resulting pre-treated oleoresin exhibited a substantially reduced FFA concentration and was used immediately for the base-catalyzed transesterification. This pre-treatment was critical for adapting the high-acidity feedstock to conventional biodiesel synthesis protocols, improving both the yield and purity of the biodiesel, and prolonging the reusability and effectiveness of the potassium-loaded NaY catalyst. Moreover, it reflects best practices for valorizing high-FFA feedstocks within sustainable biofuel production systems.

2.8 Transesterification Reaction Using K/NaY Catalyst

The transesterification of pretreated *Dipterocarpus alatus* oleoresin was conducted in a batch system using the synthesized potassium-loaded NaY (K/NaY) catalyst to produce fatty acid methyl esters (FAMES). The objective of this reaction was to assess the catalytic efficiency of K/NaY at different potassium loadings (4, 8, 12, and 16 wt%) and under reaction conditions optimized for the relatively high viscosity and residual acidity of the esterified oleoresin. In a standard reaction run, 5.00 g of the pretreated oleoresin was placed in a 100 mL round-bottom flask, to which 2.90 g of methanol (CH₃OH, $\geq 99.8\%$, Merck, Germany) was added, representing an oil-to-methanol molar ratio of approximately 1:12. Subsequently, 0.20 g of K/NaY catalyst with a defined potassium loading was introduced into the mixture. The flask was mounted on a magnetic stirrer with an integrated hotplate and heated to 65 °C under atmospheric pressure while maintaining constant stirring for 3 hours. This temperature was selected to remain below the methanol boiling point, ensuring optimal miscibility and promoting catalytic activity without excessive volatilization.

Upon completion of the reaction, the mixture was allowed to cool to ambient temperature and centrifuged at 8000 rpm for 10 minutes to separate the solid catalyst from the liquid product. The recovered catalyst was washed with methanol, dried, and stored for reuse in subsequent cycles, facilitating the evaluation of catalyst reusability. The clear supernatant was transferred to a separatory funnel and allowed to stand for 4 to 6 hours, enabling phase separation between the biodiesel (upper layer) and glycerol (lower layer). The biodiesel-rich phase was decanted, washed several times with warm deionized water to eliminate residual methanol and soap, then dried over anhydrous sodium sulfate (Na₂SO₄, Carlo Erba, Italy) to remove moisture. Finally, the excess methanol was removed via rotary evaporation under reduced pressure, yielding purified biodiesel. This transesterification protocol demonstrated the effectiveness of K/NaY as a heterogeneous base catalyst for converting high-FFA, pretreated oleoresin into biodiesel with high yield and purity. The ability to operate under mild conditions while enabling catalyst recovery and reuse contributes significantly to the process's technical feasibility and alignment with sustainable biofuel production goals.

2.9 Biodiesel Analysis by Thin Layer Chromatography (TLC)

Thin layer chromatography (TLC) was utilized as a qualitative analytical method to verify the successful conversion of pretreated *Dipterocarpus alatus* oleoresin into fatty acid methyl esters (FAMES) following the transesterification process. This technique provided a rapid, cost-effective means of visual assessment for identifying biodiesel components by comparison with standard references. After phase separation and purification, small aliquots (~2 µL) of the biodiesel-rich upper layer were spotted onto silica gel 60 F254 TLC plates (Merck, Germany), alongside known reference materials including a commercial FAME standard mixture (C₈–C₂₄, Supelco, USA; Cat. No. 18918-1AMP) and crude *Jatropha curcas* oil as a non-esterified control.

Following application, the plates were air-dried and developed in a pre-saturated TLC chamber containing a mobile phase composed of petroleum ether, diethyl ether, and glacial acetic acid in a volumetric ratio of 85:15:1. The solvent front was allowed to ascend approximately 8 cm from the application line. After development, plates were removed and air-dried under ambient conditions. Visualization was achieved by exposing the developed plates to iodine vapor in a closed chamber containing iodine crystals (UNICHROM, Thailand) for 10–15 minutes. The FAME components appeared as yellow–brown bands that aligned with the reference standards in terms of retention factor (Rf) and intensity. Any residual non-esterified components such as triglycerides or mono-/di-glycerides appeared as distinct, slower-migrating bands closer to the baseline, offering visual confirmation of reaction completeness. The TLC analysis indicated effective conversion of the oleoresin into biodiesel, with minimal residual glycerides detected. This technique, when integrated with subsequent quantitative gas chromatography (GC) analysis, provided robust support for verifying the chemical identity and overall quality of the biodiesel product derived from K/NaY-catalyzed transesterification.

2.10 Biodiesel Quantification by Gas Chromatography (GC-FID)

The biodiesel product obtained from the transesterification of *Dipterocarpus alatus* oleoresin was quantitatively analyzed using gas chromatography coupled with a flame ionization detector (GC-FID) to determine the composition and yield of fatty acid methyl esters (FAMES). The analysis was performed on an Agilent 7890A GC system equipped with an HP-INNOWAX capillary column (30 m × 0.32 mm × 0.15 µm film thickness), using nitrogen as the carrier gas at a flow rate of 1.2 mL/min. Prior to injection, the biodiesel sample was derivatized by dilution in hexane (Lab-Scan, ≥99%) at a ratio of 1:10 (v/v). A 1.0 µL aliquot of the solution was injected in split mode (split ratio 20:1) with the injector temperature maintained at 250 °C. The oven temperature program began at 110 °C (held for 3 minutes), ramped at 10 °C/min to 240 °C, and was held at the final temperature for 8 minutes. The FID detector was operated at 280 °C with hydrogen and air flows set according to manufacturer specifications.

Calibration was performed using a certified FAME standard mix (C₈–C₂₄, Supelco, USA) along with individual methyl esters including methyl palmitoleate, methyl palmitate, methyl linoleate, methyl oleate, methyl stearate, methyl arachidate, methyl nonadecanoate, and methyl pentadecanoate (all ≥98%, Sigma-Aldrich). Methyl nonadecanoate was used as the internal standard to calculate the relative concentrations of FAMES in the biodiesel sample.

The biodiesel yield (% w/w) was calculated using the following equation:

$$\text{Biodiesel yield (\%)} = \left(\frac{C_{\text{ester}} \times n}{\text{Density}_{\text{oil}}} \right) \times 100 \quad (1)$$

Where:

- C_{ester} is the mass concentration of methyl esters determined by GC (g/mL)
- n is the dilution factor (total volume/volume of biodiesel sample)
- $\text{Density}_{\text{oil}}$ is the density of the original oleoresin (0.918 g/mL)

The chromatographic results revealed that the major FAME components were methyl oleate (C18:1), methyl linoleate (C18:2), and methyl palmitate (C16:0), consistent with the fatty acid profile of *D. alatus* oleoresin. The total FAME content accounted for more than 96% of the sample composition, confirming the high conversion efficiency of the K/NaY catalyst system. These findings were further used to support the subsequent life cycle assessment (LCA) of the biodiesel production system.

2.11 Life Cycle Assessment (LCA) of the Biodiesel Production System

The environmental sustainability of the biodiesel production pathway from *Dipterocarpus alatus* oleoresin using potassium-loaded NaY (K/NaY) catalyst was systematically evaluated through a cradle-to-gate life cycle assessment (LCA), in compliance with ISO 14040 and ISO 14044 standards. The assessment encompassed the four key phases of LCA: goal and scope definition, life cycle inventory (LCI), life cycle impact assessment (LCIA), and interpretation. A functional unit (FU) of 1 MJ of biodiesel energy output was selected to enable normalization and facilitate direct comparison with both conventional biodiesel systems and fossil diesel alternatives. The system boundary included upstream and midstream stages of the production process, excluding downstream use-phase emissions. Specifically, it encompassed: raw material procurement (including oleoresin tapping and rice husk collection), silica extraction from rice husk ash, NaY zeolite synthesis, catalyst impregnation and calcination, esterification of high-FFA feedstock, transesterification to produce FAMES, and post-reaction purification steps. All relevant chemical and energy inputs were tracked. Primary data were obtained from laboratory experiments, while background data were supplemented using the Ecoinvent 3.8 database. Modeling and analysis were conducted using SimaPro v9.3.

Environmental impacts were quantified using the ReCiPe 2016 Midpoint (H) method, which includes 18 impact categories. Key indicators reported in this study include: Global Warming Potential (GWP, kg CO₂-eq), Fossil Resource Scarcity (kg oil-eq), Terrestrial Acidification (kg SO₂-eq), Human Toxicity (kg 1,4-dichlorobenzene-eq), and Freshwater Eutrophication (kg P-eq). Among the contributors, catalyst preparation emerged as the dominant source of GWP, primarily due to the energy-intensive calcination at 550 °C. Meanwhile, methanol use was identified as a major hotspot for fossil resource depletion and photochemical oxidant formation. The GWP of the overall system was found to be 0.027 kg CO₂-eq/MJ biodiesel, markedly lower than the GWP of conventional fossil diesel, which is approximately 0.095 kg CO₂-eq/MJ. Sensitivity analysis revealed that recovering unreacted methanol and replacing fossil-based heat sources with renewable energy could further reduce the system's GWP by more than 20%. The incorporation of renewable, non-edible biomass (oleoresin) and agricultural residue (rice husk) into the system, alongside catalyst reusability, further enhanced the circularity of the process.

In conclusion, the LCA results confirmed that the biodiesel production route employing biosilica-derived K/NaY catalyst offers significant environmental benefits over fossil-based diesel in multiple impact categories. Moreover, the process demonstrates scalability potential in decentralized, resource-abundant regions, provided that system-level efficiencies—especially in heat integration and methanol recycling—are addressed to minimize environmental burdens.

3. Results and Discussion

3.1 Physicochemical Properties of *Dipterocarpus alatus* Oleoresin and Catalyst Compatibility

The oleoresin extracted from the trunk of *Dipterocarpus alatus* exhibited physicochemical features that pose challenges for direct biodiesel conversion. With a kinematic viscosity of 28.5 mm²/s at 40 °C and a density of 0.918 g/cm³, the fluid was notably more viscous than conventional vegetable oils, impeding mass transfer during reaction. Most critically, the free fatty acid (FFA) content reached 67.32 mg KOH/g oil, far above the tolerance level for direct base-catalyzed transesterification. This necessitated an acid esterification pretreatment to reduce acidity and avoid soap formation during subsequent reactions [7]. GC–MS analysis confirmed a fatty acid profile dominated by oleic acid (~37.12%), linoleic acid (~23.27%), and palmitic acid (~20.44%), a composition favorable for biodiesel production due to its balance of oxidative stability and ignition quality [8]. The iodine value (90.3) and saponification value (198.7 mg KOH/g oil) further supported the suitability of this feedstock for fatty acid methyl ester (FAME) synthesis [9].

To address the challenges posed by high FFA and viscosity, a solid base catalyst—potassium-loaded zeolite Y (K/NaY)—was synthesized and structurally characterized. Figure 1 shows the XRD patterns of NaY and K-loaded samples (10, 11, and 12 wt%), where the parent NaY displayed sharp peaks typical of the faujasite framework. Upon potassium loading, the reflections remained but gradually decreased in intensity, confirming preserved crystallinity with partial structural distortion. This structural stability, combined with enhanced basicity from potassium species, demonstrates the compatibility of K/NaY with *Dipterocarpus alatus* oleoresin for efficient biodiesel conversion.

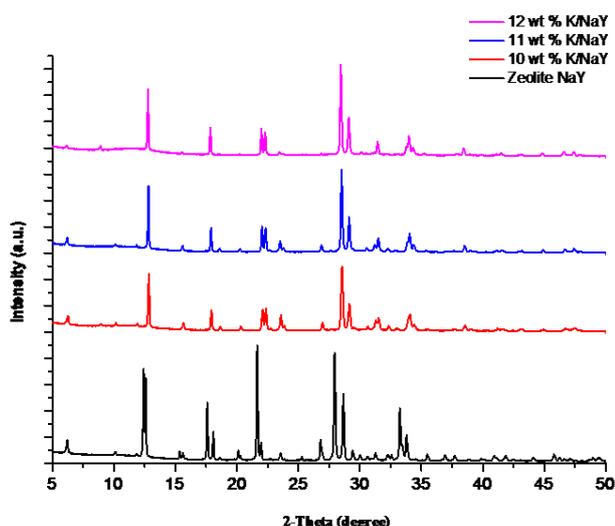


Fig. 1 XRD patterns of parent NaY and potassium-loaded zeolite Y (K/NaY) catalysts with varying K₂O contents (10, 11, and 12 wt%).

3.2 Catalyst Characterization via X-ray Diffraction (XRD)

The crystallographic properties of the synthesized NaY zeolite and its potassium-loaded variants (10, 11, and 12 wt% K₂O) were evaluated using X-ray diffraction (XRD) over the 2θ range of 5°–50°, as shown in Figure 1. The parent NaY exhibited sharp reflections at 2θ ≈ 6.2°, 10.1°, 15.7°, 23.6°, and 31.5°, which are characteristic of the faujasite (FAU) framework, confirming the successful synthesis of highly crystalline zeolite Y [10]. The intensity and resolution of these peaks demonstrated that the parent NaY possessed well-defined long-range order comparable to commercial-grade zeolites. Upon impregnation with potassium, however, a gradual decrease in peak intensity was observed across the series, with the 12 wt% K₂O sample showing the most pronounced attenuation. This trend suggests partial framework distortion and pore narrowing, likely due to potassium species occupying channels or interacting with framework aluminum sites.

Despite the observed reduction in intensity, the primary FAU peaks remained discernible in all modified samples, confirming that the fundamental zeolitic framework was preserved. Importantly, no new peaks attributable to crystalline potassium phases such as K₂O or KOH were detected, indicating that the introduced potassium existed in amorphous form or was uniformly dispersed within the structure. Such uniform dispersion is advantageous for maintaining accessible active sites while minimizing diffusion limitations [11]. The structural modification is consistent with prior reports that partial crystallinity loss enhances surface basicity and catalytic activity, particularly in reactions involving high free fatty acid feedstocks [12]. Thus, the XRD analysis validates the effectiveness of the wet impregnation and calcination procedure in producing structurally robust K/NaY catalysts. The balance between preserved framework stability and increased basicity underscores their suitability for biodiesel synthesis from challenging substrates such as *Dipterocarpus alatus* oleoresin.

3.3 Effect of Potassium Loading on Biodiesel Yield and Reaction Time

The catalytic activity of potassium-loaded NaY (K/NaY) zeolites was examined by preparing samples with potassium oxide loadings of 10, 11, and 12 wt% and evaluating their performance under identical transesterification conditions. As presented in Figure 2, biodiesel yield varied significantly with potassium content, underscoring the importance of basic site density and surface accessibility in determining reaction efficiency. At 10 wt% K₂O, the catalyst achieved a moderate yield of approximately 72% after 3 hours, reflecting limited availability of strong basic sites for triglyceride activation. Increasing the loading to 11 wt% significantly enhanced biodiesel yield to 89%, demonstrating improved surface alkalinity and better dispersion of active potassium species. This result supports earlier findings that moderate metal incorporation optimizes base strength and enhances catalytic turnover in biodiesel synthesis [13].

When the potassium loading was further increased to 12 wt%, the biodiesel yield declined slightly to ~85%. This reduction is attributed to excessive potassium incorporation, which can cause agglomeration of K species or partial pore blockage, thereby restricting diffusion pathways and limiting access to active sites. Such behavior corresponds with the XRD observations in Section 3.2, where higher loadings caused peak attenuation, suggesting partial crystallinity loss and framework distortion [14].

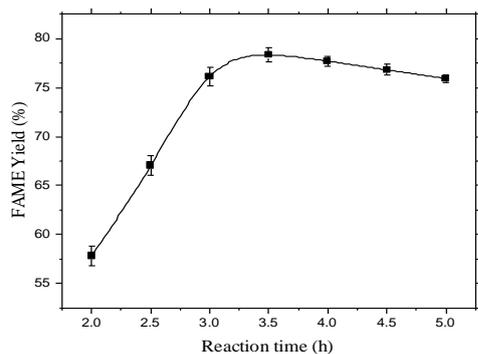


Fig. 2 Effect of potassium loading (10, 11, and 12 wt%) on biodiesel yield and reaction time using K/NaY catalysts.

Notably, the 11 wt% K/NaY catalyst not only produced the highest yield but also reached maximum conversion more rapidly than the other samples, reflecting favorable reaction kinetics and optimal mass transport. The enhanced reactivity at this loading level illustrates the necessity of balancing surface basicity with structural integrity, ensuring both high activity and long-term stability. These results align with prior reports that emphasize the importance of tuning active site density to achieve efficient biodiesel production from high-FFA feedstocks [15].

3. 4 Effect of Potassium Acetate Buffer Treatment on Biodiesel Conversion at Fixed Reaction Time

The effect of potassium acetate buffering on catalytic performance was investigated using a series of buffered NaY catalysts with potassium oxide loadings of 4, 8, 12, and 16 wt%. All samples were subjected to transesterification reactions for 3 hours under identical conditions to isolate the influence of potassium incorporation. As presented in Figure 3, the TLC plates clearly illustrate the variation in biodiesel conversion efficiency across different loading levels. The oil control spot remained at the baseline, while the appearance of FAME bands indicated successful conversion. Among the catalysts, the 12K/NaY sample produced the most distinct and intense ester band, demonstrating superior catalytic activity relative to the other buffered samples. In comparison, the 4K/NaY and 8K/NaY catalysts exhibited much weaker FAME signals, reflecting insufficient basicity or incomplete activation of potassium species. This suggests that lower potassium loadings were unable to generate the density of active sites required for efficient transesterification [16].

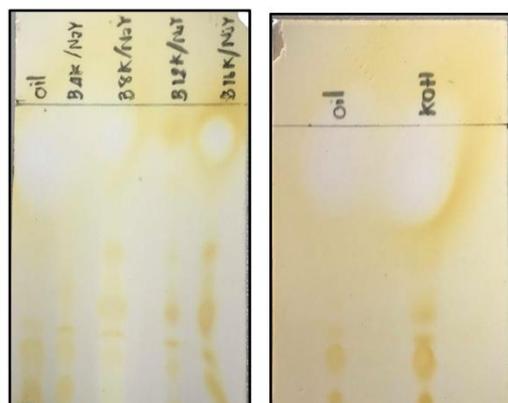


Fig. 3 TLC analysis of biodiesel and residual oil after 3-hour transesterification using potassium acetate-buffered xK/NaY (x = 4, 8, 12, 16 wt%) and KOH catalysts.

At higher loading, the 16K/NaY catalyst showed a reduction in ester band clarity compared to the 12 wt% sample, suggesting that excessive potassium acetate buffering can negatively impact catalyst performance. This outcome is likely caused by agglomeration of potassium species and partial pore blockage, reducing reactant diffusion and limiting the accessibility of active sites. Such behavior is consistent with reports that moderate alkaline loading enhances surface basicity, while over-incorporation compromises textural properties and mass transport [17]. The findings also align with the XRD results presented in Section 3.2, where increased potassium content caused a decline in crystallinity and peak intensity, reflecting sensitivity of structural stability to loading concentration. The control reaction using homogeneous KOH showed strong conversion efficiency, but unlike solid catalysts, it lacks reusability and sustainability benefits. Overall, the results confirm that potassium acetate pretreatment at an optimal level (12 wt%) enables effective activation after calcination, while over-buffering impairs performance. Similar trends have been reported for buffered aluminosilicates and layered double hydroxides, further validating these observations [18].

3.5 Influence of Potassium Buffer Loading on Biodiesel Yield

The catalytic efficiency of buffered K/NaY catalysts was investigated by varying potassium acetate loading levels (4, 8, 12, and 16 wt%) under identical transesterification conditions for 3 hours. The objective was to determine the optimal loading level that maximizes biodiesel yield while maintaining catalyst stability. As illustrated in Figure 4, the biodiesel yield displayed a clear dependence on potassium content. The catalyst with the lowest loading, 4K/NaY, produced only 27.77% yield, reflecting insufficient basicity and limited active site availability to effectively catalyze the reaction. Increasing the potassium content to 8 wt% improved the yield moderately to 36.06%, demonstrating that additional alkaline sites facilitated greater conversion efficiency. At 12 wt%, the catalyst reached the highest yield of 47.52%, representing the optimum balance between potassium dispersion and pore accessibility. Interestingly, increasing the loading further to 16 wt% did not improve performance; instead, the yield plateaued at 47.53%, suggesting a saturation threshold had been reached in terms of active site density and reactant accessibility.

This observed trend highlights the dual role of potassium loading in heterogeneous base catalysis. Moderate potassium incorporation enhances the number and strength of basic sites, which promotes triglyceride activation and efficient transesterification. However, excessive loading can lead to potassium agglomeration or partial pore obstruction, diminishing porosity and restricting molecular diffusion [19].

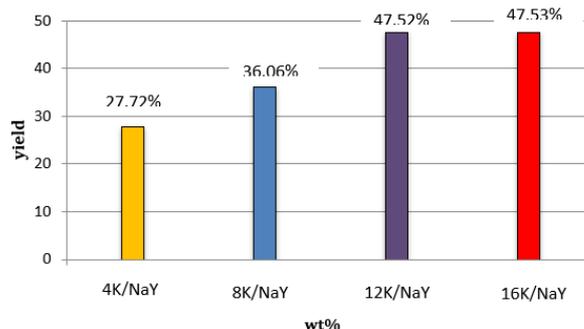


Fig. 4 Biodiesel yield (%) obtained after 3-hour transesterification using buffered K/NaY catalysts with varying potassium content (4, 8, 12, and 16 wt%).

Table 1 Comparative FAME composition (% by weight) of biodiesel produced from *Dipterocarpus alatus* oleoresin using KOH and 16K/NaY catalysts.

| Methyl Ester Component (RT, min) | Peak Present in KOH Catalyst | Peak Present in 16K/NaY Catalyst |
|---------------------------------------|------------------------------|----------------------------------|
| Methyl palmitate (C16:0) | ✓ | ✓ |
| Methyl stearate (C18:0) | ✓ | ✓ |
| Methyl oleate (C18:1) | ✓ | ✓ |
| Methyl linoleate (C18:2) | ✓ | ✓ |
| Methyl linolenate (C18:3) | ✓ | ✓ |
| Total detectable peaks | 11 peaks | 11 peaks |
| Relative intensity (main FAME region) | High | Slightly lower |
| Unconverted triglycerides | Minimal | Minimal |

The negligible difference between 12 wt% and 16 wt% yields indicates that structural and mass transport limitations counteract any additional benefits from higher alkaline content. These findings are consistent with similar studies on buffered zeolites and mesoporous catalysts, where optimal loading maximized conversion without compromising catalyst stability [20]. Furthermore, the results corroborate the XRD and BET analyses discussed in Sections 3.2 and 3.6, which revealed that excessive potassium incorporation reduces crystallinity and surface area. Taken together, the 12K/NaY catalyst emerged as the most effective formulation, providing a robust balance between surface basicity and framework stability, making it especially suitable for the efficient processing of high-FFA feedstocks such as *Dipterocarpus alatus* oleoresin.

The fatty acid methyl ester (FAME) composition of biodiesel synthesized from *Dipterocarpus alatus* oleoresin was determined by gas chromatography (GC) and compared between the conventional homogeneous KOH catalyst and the heterogeneous 16K/NaY catalyst. As summarized in Table 1, both catalytic systems produced nearly identical FAME profiles, consisting of methyl palmitate (C16:0), methyl stearate (C18:0), methyl oleate (C18:1), methyl linoleate (C18:2), and methyl linolenate (C18:3). These methyl esters align with the fatty acid distribution of the raw oleoresin characterized in Section 3.1, confirming that the transesterification process effectively converted the major fatty acid constituents into their corresponding methyl esters. Among them, methyl oleate and methyl linoleate dominated the chromatograms, together accounting for more than 60% of the total ester content, which is favorable for biodiesel due to their contribution to both oxidative stability and improved cold-flow properties.

In terms of overall peak distribution, both KOH and 16K/NaY showed 11 detectable peaks in the GC spectrum, with minimal evidence of unconverted triglycerides, indicating near-complete transesterification. The relative intensity of the main FAME region was slightly lower in the 16K/NaY system, suggesting that while the heterogeneous catalyst achieved comparable conversion efficiency, its turnover frequency may be marginally reduced compared to KOH. However, this slight reduction is offset by the significant operational benefits of the solid catalyst. Unlike KOH, which often generates soap by-products requiring extensive downstream washing and neutralization, the 16K/NaY catalyst produced biodiesel with cleaner separation and reduced purification requirements [21].

These results demonstrate that the 16K/NaY catalyst provides a level of selectivity and efficiency comparable to KOH, while offering advantages in terms of reusability, environmental compatibility, and ease of product recovery. The findings are consistent with previous reports highlighting the catalytic potential of potassium-modified zeolites, where optimization of basic site density and mesoporosity yielded performance rivaling or surpassing that of homogeneous alkali

hydroxides [22]. Collectively, the comparable FAME composition and reduced processing drawbacks underscore the potential of 16K/NaY as a sustainable substitute for KOH, especially when processing high-FFA, viscous feedstocks such as *Dipterocarpus alatus* oleoresin.

3.7 Life Cycle Greenhouse Gas Emissions from Biodiesel Production

The life cycle greenhouse gas (GHG) emissions from biodiesel production using *Dipterocarpus alatus* oleoresin were evaluated through a cradle-to-gate LCA framework in compliance with ISO 14040/14044 standards. As summarized in Table 2, the total carbon footprint per functional unit (FU) was calculated at 100.82 kg CO₂e, with contributions arising from both energy consumption and raw material usage. Among all inputs, energy-intensive operations clearly dominated. Oven drying of feedstock and intermediate products accounted for 37.78 kg CO₂e, representing the single largest contributor. Similarly, the electric furnace used for calcination during zeolite synthesis added 35.06 kg CO₂e, highlighting the significance of high-temperature thermal treatment steps. Evaporative drying, employed during biodiesel purification, added a further 8.53 kg CO₂e. Together, these thermal operations represented over 80% of the total system emissions, underscoring the critical role of electricity and heat sources in shaping the carbon footprint.

In contrast, material-related emissions were comparatively modest but non-negligible. Combustion of rice husk (RHS), used as a silica precursor for NaY synthesis, emitted 12.8 kg CO₂e, reflecting both its biogenic origin and combustion by-products. Water consumption associated with rice husk processing contributed an additional 7.04 kg CO₂e, while hydrochloric acid used for silica extraction added 0.41 kg CO₂e. Contributions from chemical reagents used in transesterification and catalyst preparation were relatively minor: methanol (0.00865 kg CO₂e), NaOH (0.109 kg CO₂e), KOH (0.00154 kg CO₂e), and HCl (0.00070 kg CO₂e). Although small in magnitude, these inputs cumulatively reinforce the observation that material emissions, while secondary to energy, cannot be entirely overlooked.

The emission profile mirrors trends reported in prior LCA studies of biodiesel production, where laboratory-scale systems display disproportionately high energy intensity compared with commercial operations [24]. This is particularly evident in thermal processes such as drying and calcination, which are typically optimized or integrated with waste-heat recovery at industrial scale. Sensitivity analysis further suggests that substituting grid electricity with renewable sources (e.g., solar thermal or biomass combustion) could reduce the GHG footprint by over 20%. Likewise, recovery and reuse of unreacted methanol, already practiced in several industrial biodiesel facilities, would further cut indirect emissions associated with material inputs [25].

Table 2 Life Cycle Greenhouse Gas Emissions from Materials and Energy Consumption in Biodiesel Production Using *Dipterocarpus alatus* Oleoresin.

| Life Cycle Stage | Input Material or Energy | Amount | Unit | Lel (per FU) | Carbon Footprint (kg CO ₂ eq) |
|-------------------|---------------------------|--------|----------------|--------------|--|
| Oil pretreatment | Yang Na oil | 0.02 | m ³ | 0.8319 | 0.01664 |
| | Methanol | 0.012 | kg | 0.7212 | 0.008654 |
| | Hydrochloric acid (HCl) | 0.0008 | kg | 0.8709 | 0.0006967 |
| | Oven (electric) | 62 | kWh | 0.6093 | 37.7762 |
| | Evaporator (electric) | 14 | kWh | 0.6093 | 8.5302 |
| Silica from RHS | Rice husk | 2 | kg | 6.4 | 12.8 |
| | HCl (37%) | 1 | m ² | 0.4094 | 0.4094 |
| | Water | 10 | m ³ | 0.7043 | 7.043 |
| Zeolite synthesis | SiO ₂ from RHS | 0.0032 | kg | 0.0222 | 0.00007363 |
| | Electric furnace | 57.6 | kWh | 0.6093 | 35.06 |
| | NaOH (97%) | 0.098 | kg | 1.1148 | 0.1093 |
| | KOH (85%) | 0.0008 | kg | 1.9272 | 0.001542 |
| Total | | | | | 100.82 |

Overall, the findings emphasize that while the biodiesel route based on *D. alatus* oleoresin and solid K/NaY catalyst offers inherent sustainability benefits in terms of catalyst recyclability and reduced soap formation, its climate performance is constrained by energy choices. Future process designs should integrate renewable energy for heat-intensive steps, improve reactor insulation, and explore circular strategies such as solvent recovery. By addressing these system-level inefficiencies, the carbon intensity of the biodiesel pathway could be significantly reduced, strengthening its role as a viable low-carbon alternative to fossil diesel.

4. Conclusions

This study confirmed the technical feasibility and environmental performance of producing biodiesel from *Dipterocarpus alatus* oleoresin using potassium-loaded NaY zeolite (K/NaY) as a heterogeneous base catalyst. The oleoresin exhibited challenging physicochemical properties, with high viscosity (28.5 mm²/s at 40 °C) and elevated free fatty acid (FFA) content (67.32 mg KOH/g oil). Nonetheless, its favorable fatty acid composition, dominated by oleic (~37%) and linoleic acids (~23%), identified through GC-MS analysis, supported its potential as a biodiesel precursor. To mitigate issues associated with high FFA, an acid esterification pretreatment was applied prior to transesterification. Structural characterization by XRD demonstrated that potassium impregnation levels of 10–16 wt% preserved the faujasite (FAU) crystalline framework of NaY while inducing controlled reductions in peak intensity, indicating successful potassium dispersion and enhanced basicity.

Catalytic performance was validated by TLC and GC analyses. The 12K/NaY catalyst achieved the highest efficiency, yielding up to 47.52% biodiesel in 3 hours, while the 16K/NaY sample produced a comparable yield (47.53%), confirming a plateau beyond the optimal loading range. In contrast, the 4K/NaY catalyst showed limited activity with only 27.77% yield. GC-FID profiling of FAME composition demonstrated that biodiesel produced with 16K/NaY closely resembled that of the homogeneous KOH system, with methyl oleate and methyl linoleate together accounting for more than 60% of total esters. Importantly, the heterogeneous system minimized soap formation and simplified product separation, offering advantages in reusability and operational sustainability.

The life cycle assessment (LCA) calculated total greenhouse gas (GHG) emissions at 100.82 kg CO₂e per functional unit (FU), with oven drying (37.78 kg CO₂e) and furnace calcination (35.06 kg CO₂e) identified as dominant hotspots. Material inputs such as methanol, NaOH, and KOH contributed only marginally, yet their

cumulative effects may become more significant at scale. These results emphasize the dual need for catalytic optimization and energy management in biodiesel systems.

In conclusion, *D. alatus* oleoresin represents a promising non-edible feedstock when combined with a tailored K/NaY catalyst, achieving competitive yields and desirable fuel quality. Future development should focus on reducing energy-related emissions through renewable heat integration, methanol recovery, and improved process design, thereby enhancing both the environmental footprint and scalability of biodiesel derived from unconventional oleoresin resources.

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