

# Performance Profiles of Heterogeneous Catalyst Classes in Biodiesel Transesterification: A Decision-Making Framework via Statistical Modelling

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**Abstract:** This study develops a study-aware, data-driven framework for comparing biodiesel catalyst families and identifying favourable yield–time performance profiles, with particular emphasis on biomass-derived catalysts. A literature-derived database comprising 302 catalyst observations from 166 studies was assembled, of which 198 observations met the biodiesel-yield eligibility criteria (149 FAME and 49 FAEE). Catalyst systems were classified into 12 families and evaluated using descriptive statistics, non-parametric tests, correlation analysis, clustered regression, yield–time productivity (YTP), Pareto analysis and study-level sensitivity analyses. Biomass-derived catalysts showed the highest overall mean biodiesel yield (95.89%; median 97.75%), with strong performance for both FAME (95.75%; median 97.70%) and FAEE (97.53%; median 97.80%). Catalyst-family differences were significant in both observation-level (Kruskal–Wallis  $H = 25.44$ ,  $p = 0.00785$ ) and study-level ( $H = 20.08$ ,  $p = 0.0442$ ) analyses. However, the pooled FAME–FAEE difference was not significant ( $p = 0.4385$ ), and the catalyst-family  $\times$  pathway interaction was not significant ( $p = 0.9962$ ). Yield–time analysis showed that catalyst families with high final yields did not necessarily achieve the highest YTP. Biomass-derived catalysts therefore emerged as the preferred catalyst family within the evaluated dataset based on their combination of high yield and consistent pathway performance, while the YTP and Pareto analyses demonstrate the importance of considering reaction time alongside final biodiesel yield in catalyst selection.

**Keywords:** Biodiesel; Heterogeneous Catalysts; Biomass-Derived Catalysts; FAME; FAE.

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## I. INTRODUCTION

The increasing demand for renewable and lower-carbon transportation fuels has sustained interest in biodiesel as an alternative to conventional petroleum-derived diesel. Biodiesel consists primarily of fatty acid alkyl esters produced through the transesterification or esterification of triglyceride- or fatty-acid-containing feedstocks with short-chain alcohols. Its production can accommodate a broad range of lipid resources, including non-edible vegetable oils and other alternative feedstocks, thereby creating opportunities to reduce dependence on edible oil resources while promoting the utilization of underused biomass resources [1,2]. The characteristics of the resulting biodiesel are influenced not only by feedstock composition but also by the alcohol, catalyst, reaction conditions and production pathway employed [1,3].

Heterogeneous catalysis has attracted considerable attention in biodiesel production because solid catalysts can provide advantages over conventional homogeneous systems, particularly with respect to catalyst separation, recovery and potential reuse. A broad range of heterogeneous catalytic materials has been investigated, including alkaline earth oxides, mixed metal oxides, hydrotalcite-derived materials, supported catalysts and solid acid catalysts [4–7]. The catalytic behaviour of these materials depends on properties such as surface basicity or acidity, active-site density, porosity, catalyst preparation and activation, as well as the conditions under which transesterification is performed. Consequently, reported biodiesel yields cannot always be interpreted independently of the experimental conditions used to obtain them [4,5].

An important development within heterogeneous biodiesel catalysis is the conversion of biomass- and waste-

derived materials into functional catalytic materials. Agricultural residues, plant-derived wastes, shells, animal residues, ashes and carbonaceous biomass can contain mineral or structural constituents that can be transformed into catalytically active materials. Their conversion into catalysts provides an additional valorization pathway in which low-value or discarded biomass is transformed into a functional material for another production process [8–10]. Reviews of biomass-derived heterogeneous catalysts have documented the growing diversity of basic, acidic, bifunctional and carbon-based catalytic systems prepared from renewable or waste resources and applied to biodiesel synthesis [8–12].

The attraction of biomass-derived catalysts is therefore not limited to catalytic activity. Such materials may combine locally available precursor resources with relatively simple preparation routes, while some biomass-derived systems provide alkaline or alkaline-earth active species suitable for transesterification. Calcium- and potassium-containing residues, for example, have been investigated as sources of catalytic functionality, whereas carbonaceous biomass can provide porous supports that can subsequently be functionalized to introduce acidic, basic or other active sites [8–10]. Recent research has also emphasized catalyst preparation, calcination, activation, functionalization, reusability and catalyst stability as important factors controlling the performance of biomass-derived catalytic systems [11,12]. These characteristics place biomass-derived catalysts at the intersection of heterogeneous catalysis and biomass valorization.

Despite the expanding literature, comparison of heterogeneous biodiesel catalysts remains difficult because reported performance is generated under substantially different experimental contexts. Feedstock composition, alcohol type, alcohol-to-oil ratio, catalyst loading, reaction temperature and reaction time can vary considerably between studies [3,5,10]. These variables may interact with catalyst characteristics and can therefore influence the reported biodiesel yield independently of catalyst family. A comparison based solely on the highest reported yield may consequently favour catalyst systems investigated under particularly favourable operating conditions without adequately reflecting the processing requirements associated with achieving that yield.

Reaction time is particularly relevant to this comparison. Two catalyst systems may produce similarly high biodiesel yields while requiring markedly different processing times. Conversely, a catalyst may produce a slightly lower yield but achieve that performance substantially faster. From a process-development perspective, these situations represent different performance profiles. Catalyst assessment therefore benefits from considering yield together with the time required to achieve the reported yield. This distinction is particularly relevant when comparing catalyst families because a family with the highest final-yield profile may not necessarily provide the highest yield relative to reaction time.

A systematic comparative framework is consequently required to distinguish apparent catalyst-family differences from variation arising from experimental conditions and study-specific characteristics. Such a framework should combine descriptive characterization of the literature with statistical assessment of catalyst-family differences, evaluation of relevant operating variables and an explicit treatment of yield–time performance. Because multiple observations may originate from the same publication, the dependence among observations also requires consideration when estimating catalyst-family effects. Study-level sensitivity analyses can provide an additional assessment of whether the principal family-level patterns persist when the study, rather than the individual experimental observation, is given greater influence.

The present study develops a data-driven framework for evaluating heterogeneous catalysts used in biodiesel production across the published literature. The analysis integrates catalyst-family classification, biodiesel yield, reaction conditions and reaction time with descriptive statistics, non-parametric statistical analysis, correlation analysis, clustered regression and yield–time productivity assessment. Yield–time productivity (YTP), defined as biodiesel yield divided by reaction time, is used as a secondary indicator of processing efficiency, while Pareto analysis is employed to identify catalyst–condition combinations offering favourable trade-offs between reported yield and reaction time. Study-level analyses and sensitivity assessments are used to examine the robustness of catalyst-family patterns to the dependence and heterogeneity inherent in the literature-derived dataset. Particular attention is given to biomass-derived catalyst systems because of their potential to provide a functional outlet for underutilized biomass and waste resources.

Accordingly, this study aims to develop a study-aware, data-driven framework for comparing heterogeneous biodiesel catalysts and identifying favourable yield–time performance profiles, with particular emphasis on the valorization potential of biomass-derived catalytic materials. Specifically, the study seeks to: (i) characterize a literature-derived dataset of biodiesel catalyst systems according to catalyst family, feedstock, reaction pathway and operating conditions; (ii) evaluate differences in reported biodiesel yield among catalyst families and examine their association with key process variables; (iii) assess catalyst–condition performance using yield–time productivity and Pareto analysis; and (iv) examine the robustness of catalyst-family comparisons using study-level and sensitivity analyses that account for the heterogeneous and study-dependent nature of the literature-derived evidence.

## II. MATERIALS AND METHODS

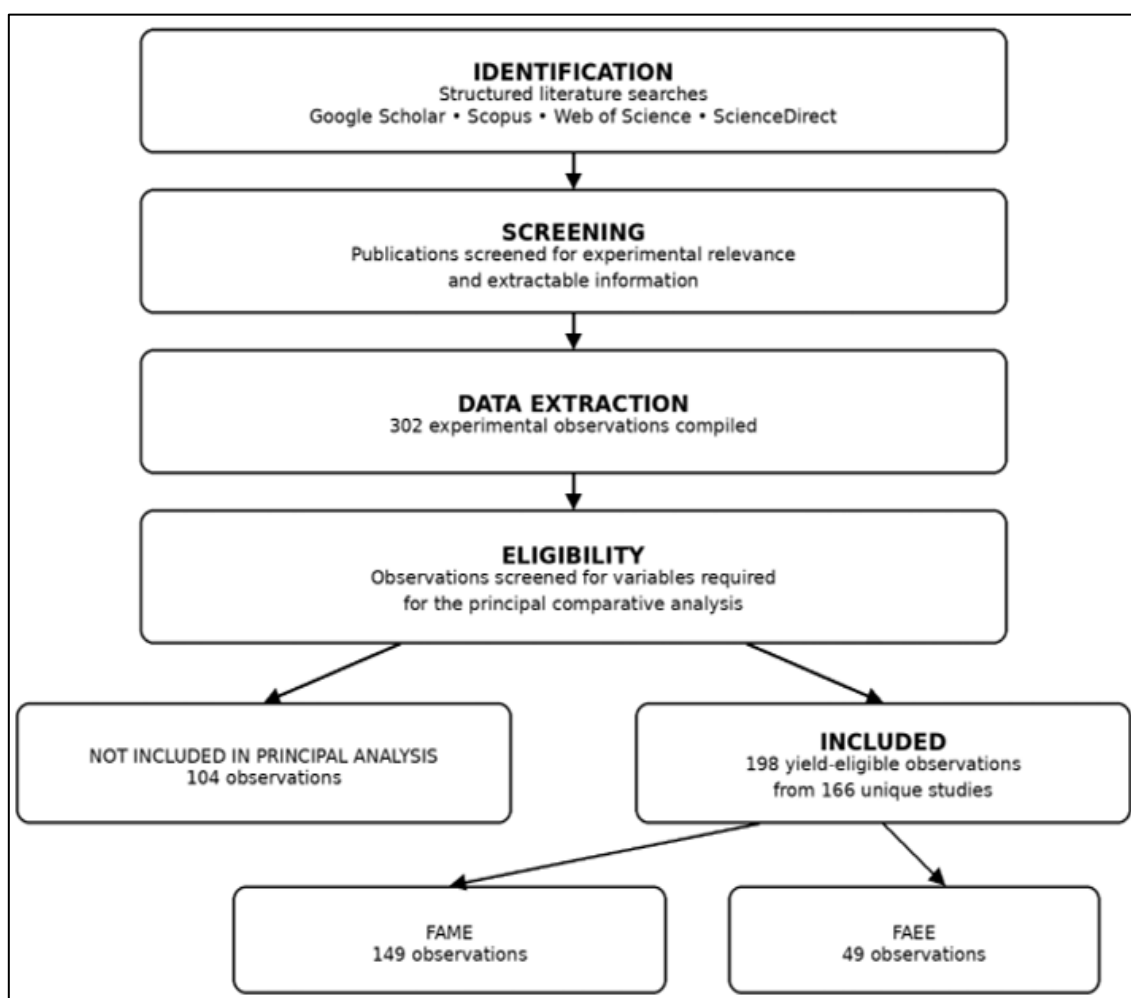


Fig 1 Literature Search, Screening, and Eligibility Assessment of Observations on Biodiesel Catalysts.

### ➤ Literature Search and Study Selection

A structured literature search was conducted to identify experimental studies reporting biodiesel production using catalytic systems relevant to comparative assessment of biodiesel catalyst performance. Searches were performed across Google Scholar, Scopus, Web of Science and ScienceDirect using combinations of terms related to biodiesel production, transesterification, heterogeneous catalysis, solid catalysts, biomass-derived catalysts, catalyst performance, biodiesel yield, reaction temperature, catalyst loading, alcohol-to-oil ratio and reaction time. Search terms were combined in different configurations to capture studies reporting conventional inorganic catalysts, supported and composite materials, biomass- and waste-derived catalysts, and other catalyst systems for FAME and FAEE production. The literature search and data extraction were conducted on 28 August 2026.

Retrieved publications were screened for experimental relevance and data availability. A study was considered eligible when it reported an experimentally determined biodiesel yield and provided sufficient information to identify the catalyst system and the principal reaction conditions required for comparative analysis. Studies were excluded

from the yield-eligible dataset when they did not provide a quantifiable experimental yield, did not provide sufficient catalyst information for classification, or lacked the process information required for analysis of the reported performance. Reviews, conceptual articles, and other publications without extractable experimental observations were not treated as individual experimental records.

The search and extraction process initially generated a master database containing 302 experimental observations. The database was subsequently screened for the variables required for the principal comparative analysis, resulting in 198 yield-eligible observations from 166 unique studies. The eligible observations comprised 149 FAME and 49 FAEE records and represented multiple catalyst families, feedstocks and reaction conditions. The study was therefore structured around experimental observations rather than publications alone, while retaining publication DOI as the study identifier. Accordingly, the resulting database represents literature identified and extracted up to 28 August 2026.

Multiple experimentally distinct catalyst formulations or reaction configurations reported within the same publication were retained as separate observations when

sufficient corresponding data were available. This approach preserved experimentally reported variation within individual studies while allowing observations from the same publication to be identified and accounted for in subsequent study-level analyses. The resulting structure also permitted comparison between observation-level and study-level results to assess the influence of within-publication replication and study-specific reporting.

#### ➤ *Data Extraction and Dataset Construction*

Data were extracted directly from the experimental reports and entered into a structured database using a predefined set of variables. The principal fields comprised publication DOI, catalyst identity, catalyst family, precursor or support material where reported, feedstock, biodiesel pathway, biodiesel yield, reaction time, reaction temperature, catalyst loading and alcohol-to-oil ratio. The DOI was retained as the primary study identifier so that multiple observations originating from the same publication could be distinguished from observations originating from independent studies.

The extraction procedure retained the reported experimental configuration rather than selecting only the highest-performing result from each publication. Thus, where a study reported several catalyst formulations, operating conditions or experimentally distinct runs, the corresponding observations were entered separately when the required variables were available. This preserved the performance variation reported within individual studies and avoided reducing the evidence base to a single arbitrarily selected observation. A separate highest-yield-per-study sensitivity analysis was subsequently used to examine whether the principal catalyst-family findings depended on retaining multiple observations from individual publications.

Numerical values were harmonized before analysis. Biodiesel yield was expressed as percentage (%), reaction time as hours (h), reaction temperature as °C, and catalyst loading as wt%. Alcohol-to-oil ratio was retained according to the molar-ratio convention reported in the source study and harmonized to permit numerical comparison. Reaction times reported in units other than hours were converted to hours before calculation of yield–time productivity. No values were experimentally generated or substituted for measurements that were not reported.

Where the literature used different terminology for closely related performance measures, the reported measurement was retained in the database and its original context was preserved. In particular, reported biodiesel yield and conversion were not assumed to be analytically identical. The principal comparative analysis was restricted to observations with an identifiable reported yield, while conversion values were retained as contextual information where available. This distinction was maintained because differences in analytical definition can contribute to heterogeneity when results are compiled across independent studies.

The database also retained feedstock identity and catalyst-specific information even when these variables were not included as primary predictors in every statistical analysis. Their retention provided contextual information for interpreting between-study heterogeneity and permitted identification of the specific catalyst formulations underlying family-level patterns. No attempt was made to homogenize chemically distinct feedstocks or catalyst formulations beyond the defined classification framework.

#### ➤ *Catalyst Classification and Classification Rationale*

Catalyst classification was performed at the material-family level to provide a consistent basis for statistical comparison while preserving the identity of individual catalyst formulations in the underlying database. Classification was based primarily on the reported chemical composition, material origin, structural form and catalytic mode of the catalyst. Where a catalyst contained multiple components, classification was based on the principal catalytic material or dominant material characteristic reported in the source study.

The final classification comprised 12 catalyst families: biomass-derived, metal/mixed-metal oxide, homogeneous, supported catalyst, MOF-based, enzymatic, LDH/hydrotalcite, solid-acid, ionic-liquid, magnetic catalyst, metal alkoxide and zeolite-based catalysts. This classification was designed to distinguish chemically and functionally recognizable groups without creating an excessive number of small categories that would limit statistical comparison.

Biomass-derived catalysts were defined as catalytic materials prepared wholly or principally from biomass or biomass-derived residues. These included catalysts produced from agricultural residues, plant-derived materials, shells, ashes and other biomass-based precursors through processes such as calcination, thermal conversion, activation, carbonization, impregnation or related modification. The defining criterion was the principal origin of the catalytic material rather than simply the presence of a biomass-derived component.

Metal/mixed-metal oxide catalysts comprised inorganic oxide systems based on one or more metals, including promoted or mixed-metal formulations that were not more appropriately assigned to a distinct structural family. LDH/hydrotalcite catalysts were retained as a separate family because of their characteristic layered structure and their frequent use as precursors to mixed-metal oxide catalytic phases. Supported catalysts were defined by the presence of a distinct support material carrying the active catalytic phase, while MOF-based systems were classified according to their metal–organic framework structure.

Solid-acid catalysts were classified according to their predominant acidic catalytic functionality, whereas ionic-liquid systems were grouped according to the ionic-liquid catalytic phase. Magnetic catalysts were distinguished by the incorporation of magnetic components intended to facilitate catalyst recovery or separation. Metal alkoxide and zeolite-based catalysts were retained as separate families because

their characteristic chemical or structural features provided a meaningful basis for comparison.

Homogeneous and enzymatic systems appearing within the comparative evidence base were retained as separate comparator families rather than being forced into heterogeneous material categories. Their separate classification prevented fundamentally different catalytic modes from being obscured within broader material groups and allowed their performance to be evaluated transparently alongside the heterogeneous catalyst families.

The classification was therefore intended as an analytical grouping framework rather than a claim of identical catalytic mechanisms within each family. Individual catalysts within a family can differ substantially in composition, preparation, surface properties and catalytic activity. Family-level analysis was used to identify broad performance patterns across the literature, while catalyst identity and precursor information were retained to preserve the underlying experimental specificity.

#### ➤ *Experimental and Process Variables*

Biodiesel yield was designated as the principal response variable because it was the most consistently extractable performance measure across the eligible literature. Reaction time, reaction temperature, catalyst loading and alcohol-to-oil ratio were selected as the principal continuous process variables because they were sufficiently represented across the database and directly relate to the operating conditions under which the reported yields were obtained.

Catalyst family and biodiesel pathway were treated as categorical variables. Biodiesel pathway was classified as FAME for methanol-derived biodiesel and FAEE for ethanol-derived biodiesel. Catalyst identity, precursor material and feedstock identity were retained as contextual variables to preserve information concerning the specific experimental systems represented in the database.

The analysis used the experimental values reported by the individual studies. No unreported experimental measurements were generated or substituted. Where a process variable was unavailable for a particular observation, that observation was retained for analyses for which the required variables were available but was not assigned an artificial value for the missing measurement. Consequently, the number of observations contributing to individual analyses could differ according to variable availability.

The process variables were evaluated primarily as associational descriptors of reported biodiesel performance. Their inclusion in correlation and regression analyses was intended to determine whether differences in reported yield were associated with catalyst family and operating conditions after accounting for the structure of the literature-derived dataset. These analyses were not interpreted as controlled experimental estimates of causal effects because catalyst composition, feedstock characteristics and operating conditions varied simultaneously among published studies.

#### ➤ *Statistical Analysis*

Descriptive statistics were calculated to characterize biodiesel yield within catalyst families and biodiesel pathways. Because the compiled observations represented heterogeneous experimental systems and did not satisfy the assumptions required for conventional parametric group comparisons, catalyst-family differences in biodiesel yield were evaluated using the Kruskal–Wallis test [13].

Where the omnibus test indicated a statistically significant difference, pairwise comparisons were conducted using Dunn's test [14]. The resulting  $p$ -values were adjusted using the Holm sequential procedure to account for multiple comparisons [15]. Statistical significance was assessed at  $p < 0.05$ . Effect sizes were considered alongside statistical significance to characterize the magnitude of between-group differences. Catalyst families were compared using their observed yield distributions rather than maximum reported yield alone, allowing central tendency and dispersion to be considered.

#### ➤ *Correlation Analysis*

Rank-based correlation analysis was used to examine associations between biodiesel yield and continuous process variables. Spearman's rank correlation coefficient was used to assess monotonic relationships between yield and reaction time, reaction temperature, catalyst loading and alcohol-to-oil ratio [16]. The correlation analysis was performed using available paired observations for each variable. Correlation coefficients were interpreted as measures of association within the literature-derived dataset and were not interpreted as evidence of causal relationships.

#### ➤ *Regression Analysis*

Multivariable regression analysis was used to evaluate the combined relationship between biodiesel yield and the principal catalyst and process descriptors. Predictor variables included catalyst family, biodiesel pathway, reaction temperature, reaction time, catalyst loading and alcohol-to-oil ratio, subject to data availability. Because multiple observations could originate from the same publication, publication DOI was retained as the study-level identifier. Regression inference incorporated cluster-robust standard errors at the study level to account for potential dependence among observations originating from the same study [17].

The regression model was used to characterize conditional associations between catalyst and process variables and biodiesel yield. Model coefficients were interpreted within the range of experimental conditions represented in the literature and were not treated as evidence of causal effects.

#### ➤ *Yield–Time Productivity Analysis*

Yield and reaction time were jointly evaluated to provide a process-oriented assessment of catalyst performance. Yield–time productivity (YTP) was calculated as:

$$YTP = \frac{Y}{t}$$

Where  $Y$  represents biodiesel yield (%), and  $t$  represents reaction time (h). YTP is therefore expressed as percentage-yield units per hour (yield-units  $\text{h}^{-1}$ ).

The measure was used to distinguish catalyst–condition combinations that achieved high biodiesel yield rapidly from systems requiring substantially longer reaction periods. YTP was treated as a comparative performance descriptor rather than as a comprehensive economic, environmental or energy-efficiency indicator.

#### ➤ *Pareto Frontier Analysis*

Pareto analysis was applied to evaluate the simultaneous objectives of maximizing biodiesel yield and minimizing reaction time. An experimental configuration was considered Pareto-efficient when no other configuration provided both a higher biodiesel yield and a shorter reaction time. The resulting Pareto frontier identifies experimentally reported catalyst–condition combinations representing favourable yield–time trade-offs.

Pareto-efficient configurations were interpreted as candidates for further consideration based on the combined objectives of high reported biodiesel yield and shorter processing duration. The analysis was complementary to YTP because Pareto efficiency evaluates dominance between configurations rather than reducing the two objectives to a single ratio.

#### ➤ *Study-Level Sensitivity Analysis*

Study-level sensitivity analysis was performed to determine whether the principal comparative patterns were consistent when the publication rather than the individual experimental observation was considered the primary analytical unit. Where multiple observations were reported within the same publication, study-level summaries were generated for comparison with observation-level results. The analysis provided an additional assessment of the influence of study-specific experimental reporting on the observed catalyst-family differences.

Additional sensitivity analyses examined the stability of the family-level findings under alternative observation-selection approaches, including exclusion of homogeneous and enzymatic systems and selection of the highest-yield observation from each study. These analyses were used to determine whether the principal catalyst-family patterns were dependent on particular subsets of the literature.

Potential publication and reporting bias was also considered in interpreting the literature-derived evidence. Studies reporting high-performing catalyst systems may be more likely to be published or reported in greater experimental detail than studies producing less favourable or inconclusive results. Conventional funnel-plot asymmetry and regression-based publication-bias tests were not applied because the dataset comprises heterogeneous experimental catalyst–feedstock–process configurations and does not provide a common effect-size measure with corresponding within-study standard errors across the included studies.

Instead, potential reporting bias was considered through the distribution of observations across studies and catalyst families, the comparison of observation-level and study-level results, and the sensitivity analyses described above. Consequently, the reported catalyst-family differences were interpreted as patterns within the available published experimental evidence rather than as estimates unaffected by selective publication or reporting..

#### ➤ *Data Analysis and Software*

Data organization, numerical harmonization, and calculation of derived variables were performed using spreadsheet-based data processing. Statistical analyses were conducted using established non-parametric, correlation and regression procedures. All statistical tests were evaluated at a significance level of  $p < 0.05$ . Results were reported using appropriate descriptive statistics, effect estimates, confidence intervals and probability values. Graphical analyses were used to illustrate catalyst-family distributions, process-variable relationships, yield–time performance and Pareto-efficient configurations.

### III. RESULTS AND ANALYSIS

The biodiesel catalyst database contained 302 records, of which 198 had eligible biodiesel-yield observations. The yield-eligible dataset comprised 149 FAME observations and 49 FAEE observations from 166 unique DOI-indexed studies. Catalyst families and alcohol pathways were therefore evaluated using family-level distributions, pathway comparisons, rank-based statistical tests, bootstrap confidence intervals, multivariable analysis, yield–time productivity, and high-yield threshold analysis.

#### ➤ *Composition of the Biodiesel Catalyst Database*

The yield-eligible database comprised 198 observations from 166 studies. The database included 149 FAME observations and 49 FAEE observations. Eighteen DOIs contributed more than one observation, with a maximum of 10 observations from a single DOI. The database contained 78 distinct feedstock labels. These characteristics define the evidence base used for the comparative analysis and are summarized in Table 1. Complete descriptive statistics for all catalyst families, including observation counts, study counts, means, medians, and standard deviations, are provided in Supporting Information Table S1.

Table 1 Composition and Coverage of the Yield-Eligible Database.

| Database Characteristic           | Value |
|-----------------------------------|-------|
| Master records screened           | 302   |
| Yield-eligible observations       | 198   |
| FAME observations                 | 149   |
| FAEE observations                 | 49    |
| Unique DOI studies                | 166   |
| DOIs contributing >1 observation  | 18    |
| Maximum observations from one DOI | 10    |
| Unique feedstock labels           | 78    |

Across alcohol pathways, the central tendency of reported yield was similar. FAME observations had a mean yield of 89.35% and a median of 96.28%, whereas FAEE

observations had a mean of 91.59% and a median of 96.30% (Table 2). FAME showed greater dispersion (SD = 21.50%) than FAEE (SD = 10.58%).

Table 2 Overall Biodiesel-Yield Distribution by Alcohol Pathway.

| Alcohol Pathway | n   | Mean Yield (%) | Median (%) | SD (%) |
|-----------------|-----|----------------|------------|--------|
| FAEE            | 49  | 91.59          | 96.30      | 10.58  |
| FAME            | 149 | 89.35          | 96.28      | 21.50  |

Figure 2 presents the distribution of yield-eligible observations across catalyst families and alcohol pathways. Biomass-derived catalysts formed the largest heterogeneous catalyst family in the database (63 observations), followed by

metal/mixed-metal oxides (33), supported catalysts (18), MOF-based catalysts (17), and the remaining families at lower frequencies. Biomass-derived catalysts included 58 FAME observations and 5 FAEE observations.

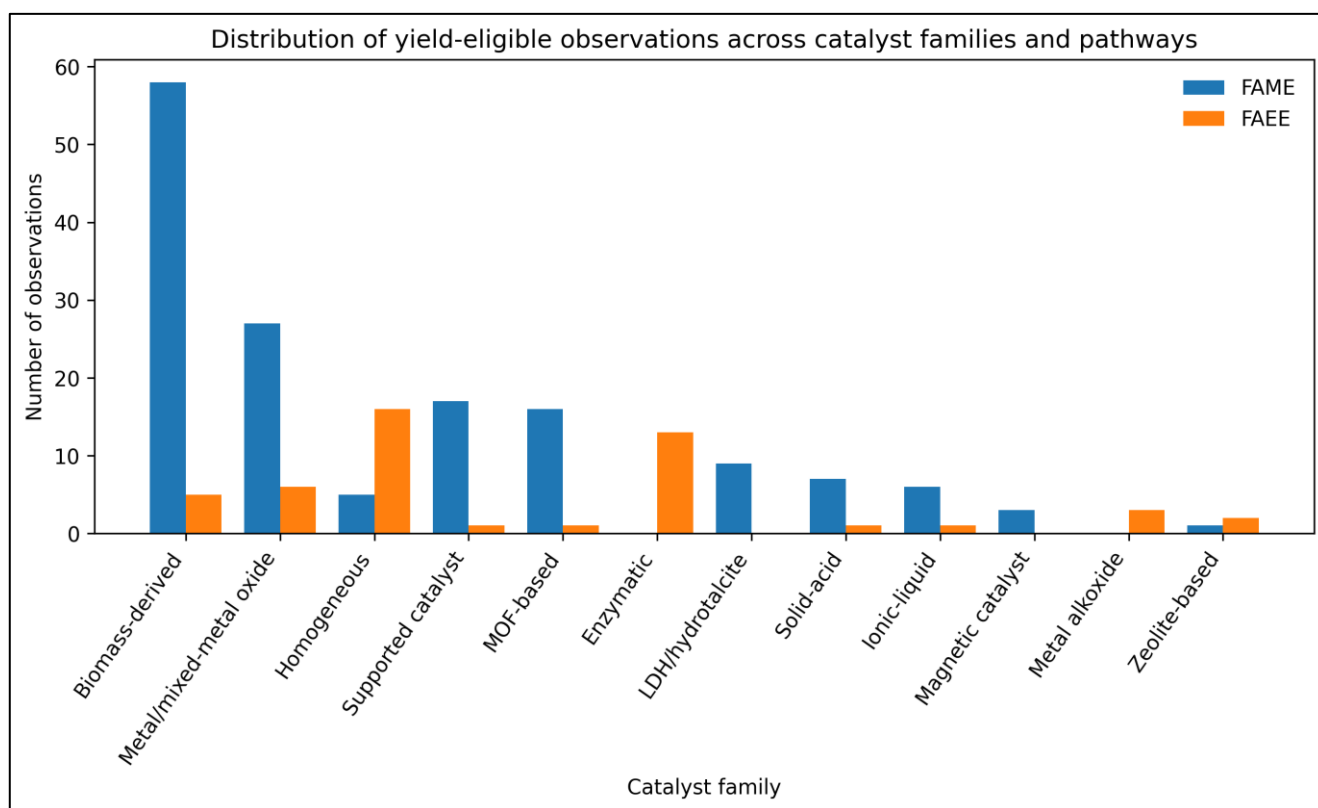


Fig 2 Distribution of Yield-Eligible Observations Across Catalyst Families and Alcohol Pathways.

Figure 3 compares catalyst-family mean yields within each alcohol pathway using bootstrap 95% confidence intervals. Biomass-derived catalysts had the highest overall family mean yield (95.89%) and a median of 97.75%. Their

pathway-specific means were 95.75% for FAME and 97.53% for FAEE. Other families also showed high yields, including solid-acid (95.09%), magnetic (94.36%), homogeneous (94.28%), and LDH/hydratalcite (94.25%) catalysts.

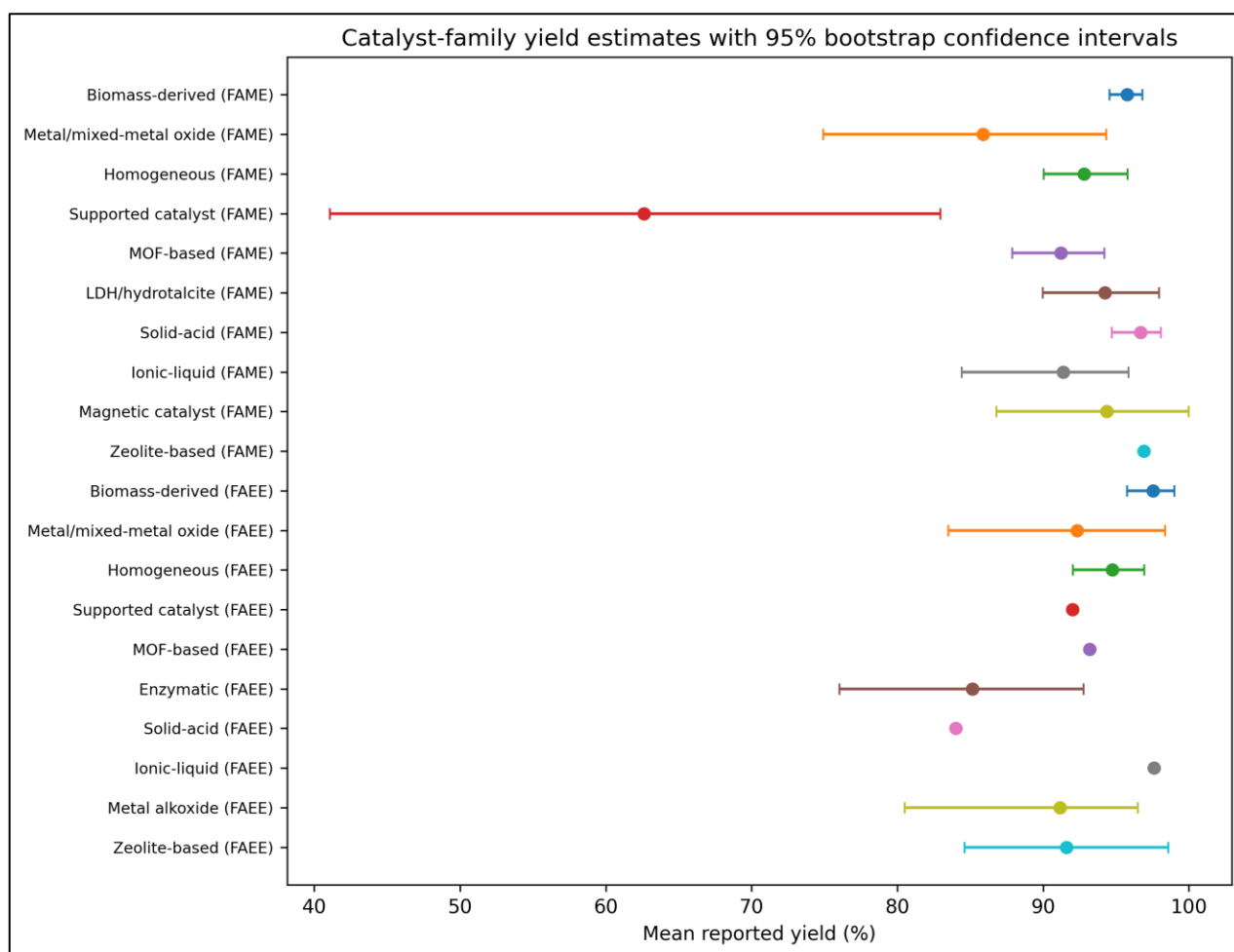


Fig 3 Catalyst-Family Yield Estimates for FAME and FAEE with 95% Bootstrap Confidence Intervals.

The overall catalyst-family comparison was statistically significant (Kruskal–Wallis  $H = 25.44$ ,  $df = 11$ ,  $p = 0.00785$ ,  $\epsilon^2 = 0.0777$ ). Holm-adjusted pairwise comparisons identified significant differences between biomass-derived and enzymatic catalysts (rank-biserial correlation = 0.617,

adjusted  $p = 0.0334$ ) and between biomass-derived and MOF-based catalysts (rank-biserial correlation = 0.543, adjusted  $p = 0.0411$ ). The complete pairwise comparisons are provided in Supporting Information Table S2.

Table 3 Principal Inferential Results for Catalyst-Family and Pathway Comparisons.

| Analysis                            | Statistic                  | df | p-value | Effect/fit             |
|-------------------------------------|----------------------------|----|---------|------------------------|
| Catalyst-family comparison          | $H = 25.44$                | 11 | 0.00785 | $\epsilon^2 = 0.0777$  |
| FAME vs FAEE                        | $U = 3920.5$               | —  | 0.4385  | Rank-biserial = 0.0740 |
| Family $\times$ pathway interaction | Wald $\chi^2 = 0.91$       | 7  | 0.9962  | —                      |
| Biomass vs enzymatic                | Dunn rank-biserial = 0.617 | —  | 0.0334  | Holm-adjusted          |
| Biomass vs MOF                      | Dunn rank-biserial = 0.543 | —  | 0.0411  | Holm-adjusted          |
| Family joint regression             | Wald $\chi^2 = 25.31$      | 11 | 0.00821 | $R^2 = 0.350$          |
| Study-level family comparison       | $H = 20.08$                | 11 | 0.0442  | $\epsilon^2 = 0.0582$  |

Pairwise p-Values are Holm-Adjusted.

The pooled FAME–FAEE comparison was not statistically significant (Mann–Whitney  $U = 3920.5$ ,  $p = 0.4385$ ; rank-biserial correlation = 0.0740). The catalyst-family  $\times$  alcohol-pathway interaction was likewise not statistically significant (Wald  $\chi^2 = 0.91$ ,  $df = 7$ ,  $p = 0.9962$ ). Complete family-by-pathway statistics are reported in Supporting Information Table S3, while correlation results are provided in Table S4.

Study-clustered regression provided a complementary assessment accounting for dependence among observations from the same DOI. The joint family effect was statistically significant (Wald  $\chi^2 = 25.31$ ,  $df = 11$ ,  $p = 0.00821$ ;  $R^2 = 0.350$ ; adjusted  $R^2 = 0.269$ ). Full coefficients, cluster-robust standard errors, confidence intervals, and p-values are reported in Supporting Information Table S5.

Yield–time productivity (YTP) was calculated as reported biodiesel yield divided by reaction time in hours and

is expressed in yield-units  $\text{h}^{-1}$ . Figure 4 relates this measure to family-level yield. For FAME, the highest median YTP values were observed for homogeneous catalysts (126.67 yield-units  $\text{h}^{-1}$ ), magnetic catalysts (50.00 yield-units  $\text{h}^{-1}$ ), and biomass-derived catalysts (49.00 yield-units  $\text{h}^{-1}$ ). For

FAEE, biomass-derived catalysts had a median YTP of 86.55 yield-units  $\text{h}^{-1}$ , while homogeneous catalysts had 48.80 yield-units  $\text{h}^{-1}$ . Complete YTP results are provided in Supporting Information Table S6.

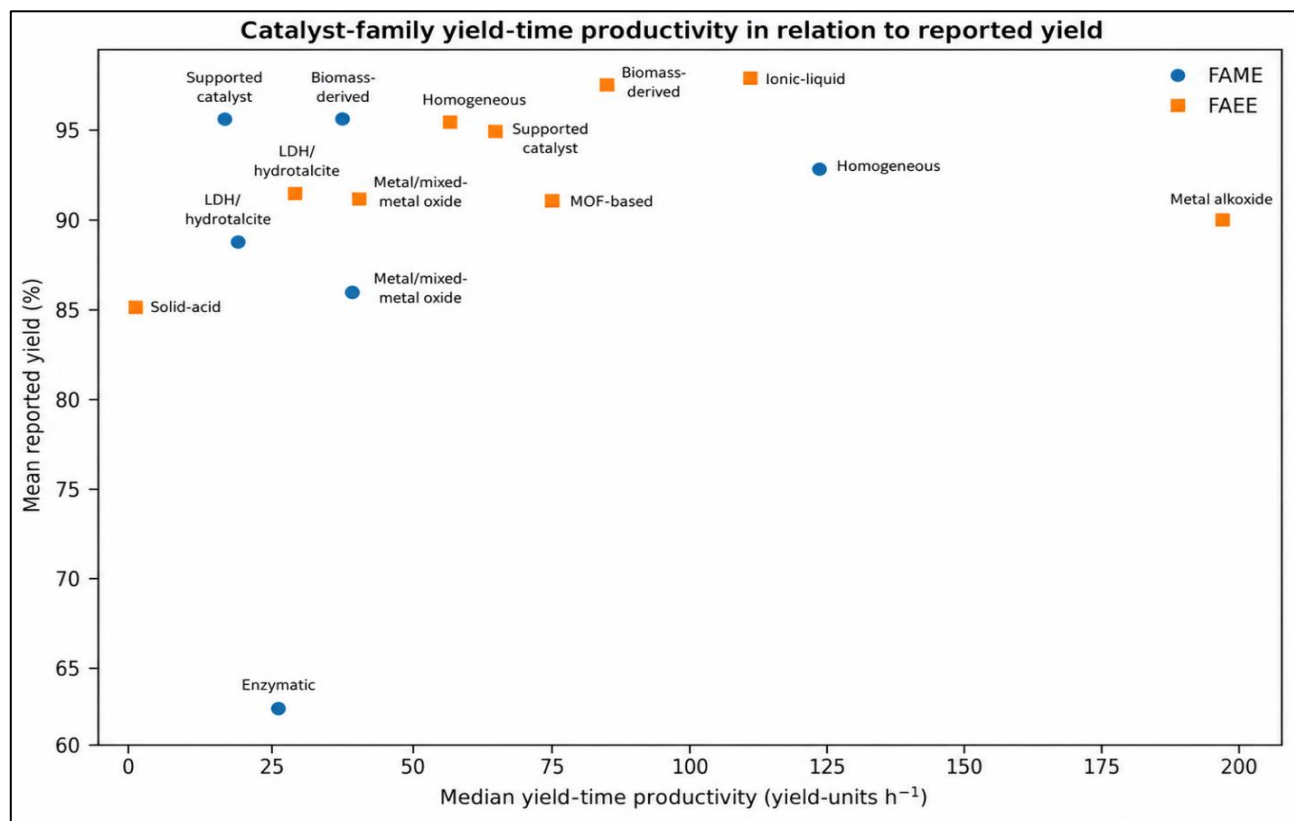


Fig 4 Catalyst-Family Yield–Time Productivity in Relation to Reported Biodiesel Yield.

Biomass-derived catalysts achieved at least 90% reported yield in 54 of 58 FAME observations (93.10%) and all 5 FAEE observations (100%). The complete threshold analysis is provided in Supporting Information Table S7.

Robustness checks retained the principal family pattern. After excluding homogeneous and enzymatic observations, the family-level comparison remained significant ( $H = 18.74$ ,  $df = 9$ ,  $p = 0.02748$ ). Restricting each study to its highest reported-yield observation gave  $H = 19.91$  ( $df = 11$ ,  $p = 0.04658$ ).

The heterogeneous-catalyst Pareto analysis identified multiple yield–time frontier configurations. For FAME, frontier observations included LDH/hydrotalcite configurations at 99.40% yield and 0.13 h, 99.60% and 0.17 h, a metal/mixed-metal oxide configuration at 99.90% and 0.50 h, and a magnetic-catalyst configuration at 100.00% and 2.00 h. For FAEE, the frontier included metal alkoxide at 96.50% and 0.50 h, ionic liquid at 97.60% and 0.75 h, and biomass-derived configurations at 98.52% and 1.08 h and 99.64% and 1.31 h. Complete configurations are reported in Supporting Information Table S9.

Taken together, biomass-derived catalysts showed the strongest overall yield profile among the evaluated catalyst

families, with the highest overall mean yield (95.89%), a median yield of 97.75%, and high attainment of the 90% yield benchmark across both FAME and FAEE pathways. Their mean yields were 95.75% for FAME and 97.53% for FAEE. YTP and Pareto analysis further showed that other catalyst families could provide competitive yield–time combinations under specific operating conditions. These results identify biomass-derived catalysts as the leading catalyst family within the evaluated dataset, while highlighting the value of considering both final yield and reaction-time requirements in catalyst performance assessment.

#### IV. DISCUSSION

Among the catalyst families evaluated, biomass-derived catalysts showed the strongest overall yield profile, with a mean biodiesel yield of 95.89% and a median of 97.75% ( $n = 63$ ). Their performance was maintained across both alcohol pathways, with mean yields of 95.75% for FAME and 97.53% for FAEE and corresponding medians of 97.70% and 97.80%, respectively. The close agreement between the means and medians indicates that the high-yield profile was broadly represented within the biomass-derived observations. The catalytic activity of bio-derived materials has been associated with naturally occurring mineral constituents and catalytically active phases formed during thermal conversion

[18]. Reviews of biomass- and bio-waste-derived catalysts similarly document their use as solid-acid, solid-base and bifunctional materials for biodiesel synthesis [19,11]. More broadly, catalyst composition, active-site characteristics and preparation conditions influence heterogeneous esterification and transesterification performance [20].

Within the FAME subset, biomass-derived catalysts recorded a mean yield of 95.75% and a median of 97.70% ( $n = 58$ ). The bootstrap 95% confidence interval for the family mean was 94.54–96.80%, indicating a comparatively narrow estimate around the observed mean. Solid-acid catalysts produced a slightly higher mean yield of 96.67% ( $n = 7$ ), while zeolite-based catalysts recorded 96.89% from a single observation. LDH/hydrotalcite and magnetic catalysts also showed high mean yields of 94.25% ( $n = 9$ ) and 94.36% ( $n = 3$ ), respectively. These results place biomass-derived catalysts among the highest-yielding families while providing substantially greater observational coverage than the small solid-acid, magnetic and zeolite-based subsets.

Biomass-derived catalysts are chemically diverse because the inorganic fraction of a biomass precursor can be retained, transformed or concentrated during thermal treatment. Differences in precursor composition can therefore generate catalysts with different mineral constituents and surface properties [21]. Depending on precursor and activation conditions, the resulting materials can display acidic, basic or combined acid–base functionality [22]. Preparation and activation conditions can further alter surface structure, active-site distribution and catalytic behaviour [23]. The high FAME yield observed for the biomass-derived family is therefore a family-level result covering chemically different materials rather than evidence for one standardized catalyst formulation.

The comparison with other heterogeneous architectures provides additional context. Solid-acid catalysts had the highest FAME mean yield (96.67%), marginally above biomass-derived catalysts (95.75%), whereas LDH/hydrotalcite catalysts also showed a high mean yield (94.25%). Metal/mixed-metal oxide catalysts had a lower mean of 85.87%. These differences are consistent with the range of catalytic functionalities represented in heterogeneous systems. Acidic materials can facilitate esterification of free fatty acids and transesterification, whereas layered hydroxides and mixed oxides provide tunable basic sites and acid–base functionality [4,24]. Variations in oxide composition and surface properties can consequently produce distinct catalytic responses [25].

MOF-based catalysts recorded a FAME mean yield of 91.22% and median of 92.50%. Their catalytic versatility is associated with the ability to modify metal centres, organic linkers, pore environments and functional groups, which allows the catalytic environment to be tailored to the reaction [26]. Functionalized MOF systems have also been investigated for biodiesel synthesis under esterification and transesterification conditions [27]. Supported catalysts showed the lowest FAME mean yield (62.61%), but their median was considerably higher (92.60%), indicating

substantial heterogeneity among individual supported systems. Such variation is compatible with the influence of active-phase dispersion, support interaction and preparation route on catalytic behaviour [28,29].

The frequency of observations achieving at least 90% yield provides a second measure of the FAME performance profile. Biomass-derived catalysts reached this threshold in 54 of 58 observations (93.10%). Comparable threshold attainment was observed for solid-acid and zeolite-based catalysts (100% within their available observations), while LDH/hydrotalcite, homogeneous, ionic-liquid, MOF-based and metal/mixed-metal oxide families showed lower or more variable proportions. Waste-derived materials have been reported to generate catalytically active mineral-rich phases after thermal treatment [7], and specific biomass-derived catalysts have demonstrated effective transesterification following suitable precursor selection and activation [6,30,31]. The high threshold attainment of the biomass-derived family therefore complements its mean and median yield estimates.

The FAEE subset showed a similar family-level pattern. Biomass-derived catalysts recorded the highest observed mean yield of 97.53% and a median of 97.80% ( $n = 5$ ), with a bootstrap 95% confidence interval of 95.77–98.99%. Homogeneous catalysts had a mean yield of 94.73% ( $n = 16$ ), followed by metal/mixed-metal oxides at 92.33% ( $n = 6$ ). MOF-based, ionic-liquid, metal-alkoxide, zeolite-based, supported and enzymatic catalysts recorded mean yields of 93.20%, 97.60%, 91.17%, 91.60%, 92.00% and 85.14%, respectively, within the available observations. The biomass-derived family therefore occupied the highest mean-yield position in the FAEE subset. The FAEE estimate is based on five observations, compared with 58 for FAME.

The performance of biomass-derived catalysts in FAEE is consistent with the broader literature describing bio-derived materials as heterogeneous catalytic systems whose active phases depend on precursor composition and preparation conditions [18,19,21–23]. Recent biodiesel studies further show that catalyst preparation and operating conditions can influence the physicochemical characteristics and resulting catalytic behaviour of bio-derived materials [40,41]. Accordingly, the FAEE result is best interpreted as an observed family-level performance pattern across the available records.

The FAEE family comparison also illustrates the importance of observational coverage when interpreting numerical rankings. For example, the ionic-liquid family recorded a mean of 97.60% from one observation, while biomass-derived catalysts recorded 97.53% from five observations. These values are numerically close, but the estimates differ in the number of supporting observations. Similarly, the solid-acid family had one FAEE observation at 84.00%, whereas homogeneous catalysts had 16 observations with a mean of 94.73%. The family-level comparison therefore considers both the magnitude of reported yield and the distribution of observations. This approach distinguishes

broadly represented performance from estimates based on isolated observations.

Biomass-derived catalysts occupied the leading mean-yield position in both pathways, with 95.75% for FAME and 97.53% for FAEE. The corresponding medians were 97.70% and 97.80%. The pooled FAME–FAEE comparison was not statistically significant (Mann–Whitney  $U = 3920.50$ ,  $p = 0.4385$ ; rank-biserial correlation = 0.0740), and the catalyst-family  $\times$  alcohol-pathway interaction was also not statistically significant (Wald  $\chi^2 = 0.908$ ,  $df = 7$ ,  $p = 0.9962$ ). Thus, the higher numerical FAEE mean for biomass-derived catalysts was not accompanied by evidence of a systematic pathway effect in the pooled comparison. FAME and FAEE production nevertheless involve distinct alcohol substrates and can require different processing conditions, although both remain governed by common factors such as catalyst characteristics, alcohol-to-oil ratio, reaction temperature and reaction time [42].

The family-specific results further show that the biomass-derived family maintained a high yield profile across both pathways, with 58 observations for FAME and five for FAEE. This difference in observational coverage matters when interpreting the pathway-specific estimates because the two pathways are not equally represented in the available literature [42]. Supporting Information Table S3 provides complete family-by-pathway statistics, while Table 3.3 summarizes the pathway comparison and interaction results.

Biomass-derived catalysts were identified as the preferred family because they combined the highest overall mean yield (95.89%) with a high median yield (97.75%), strong pathway-specific yield values for both FAME and FAEE, and high attainment of the 90% yield threshold. The observation-level Kruskal–Wallis analysis detected differences among catalyst families ( $H = 25.44$ ,  $df = 11$ ,  $p = 0.00785$ ;  $\varepsilon^2 = 0.0777$ ), with Holm-adjusted pairwise differences between biomass-derived and enzymatic catalysts and between biomass-derived and MOF-based catalysts. The study-level analysis retained a significant family effect ( $H = 20.08$ ,  $df = 11$ ,  $p = 0.0442$ ;  $\varepsilon^2 = 0.0582$ ), providing additional support for the family-level pattern when the study rather than the individual observation was used as the unit of analysis.

The multivariable analysis provided a complementary assessment of whether the family-level pattern persisted after accounting simultaneously for alcohol pathway, temperature, reaction time, catalyst loading and alcohol-to-oil ratio. Standard errors were clustered by DOI to account for dependence among multiple observations originating from the same publication. The joint test of catalyst family remained statistically significant (Wald  $\chi^2 = 25.31$ ,  $df = 11$ ,  $p = 0.0082$ ;  $R^2 = 0.350$ ; adjusted  $R^2 = 0.269$ ), whereas alcohol pathway and the continuous process variables were not individually statistically significant in the clustered model. Relative to the biomass-derived reference family, the estimated coefficients for MOF-based and supported catalysts were  $-6.53$  percentage points ( $p = 0.0480$ ) and  $-37.51$  percentage points ( $p = 0.0145$ ), respectively. These coefficients should be interpreted as adjusted associations

within the compiled dataset rather than causal effects, because the database represents heterogeneous experimental conditions reported across different studies. Regression diagnostics indicated no substantial multicollinearity among the retained predictors, with a maximum non-intercept variance inflation factor of 1.74. Complete regression coefficients and diagnostic results are provided in Supporting Information Tables S5 and S10.

Yield–time productivity provides a distinct dimension of catalyst performance. For FAME, biomass-derived catalysts had a median YTP of 49.00 yield-units  $h^{-1}$ , compared with 126.67 yield-units  $h^{-1}$  for homogeneous catalysts and 41.55 yield-units  $h^{-1}$  for LDH/hydrotalcite catalysts. For FAEE, biomass-derived catalysts had a median YTP of 86.55 yield-units  $h^{-1}$ , compared with 48.80 yield-units  $h^{-1}$  for homogeneous catalysts. These results indicate that the family with the strongest final-yield profile is not necessarily the family with the highest yield per unit reaction time. YTP therefore complements rather than replaces the yield criterion used to identify the preferred family. Reaction time, catalyst loading, alcohol-to-feedstock ratio and temperature are recognized as important process variables affecting heterogeneous biodiesel production [43].

As an exploratory sensitivity analysis, productivity was additionally normalized by catalyst loading using  $Y/(tL)$ , expressed as yield-units  $h^{-1} (wt\%)^{-1}$ . The loading-normalized descriptor indicated statistically significant differences among catalyst families overall and within both the FAME and FAEE subsets. However, the biomass-derived family did not differ significantly from the other families in the pooled comparison ( $U = 3304.50$ ,  $p = 0.2172$ ), nor within the FAME ( $U = 2548.50$ ,  $p = 0.0531$ ) or FAEE ( $U = 39.00$ ,  $p = 0.3976$ ) subsets. This analysis was therefore treated as an exploratory sensitivity measure rather than a replacement for YTP, particularly because reactor volume and throughput information were not systematically available for calculation of a true space–time yield. Complete loading-normalized productivity results are provided in Supporting Information Table S11.

A further sensitivity analysis examined reaction modality by distinguishing conventional thermal operation from assisted or special modalities. The overall comparison between conventional and assisted/special modalities was not statistically significant (Mann–Whitney  $U = 1542.00$ ,  $p = 0.5794$ ). The conventional thermal subset contained 176 observations, whereas the assisted/special subset contained only 19 observations, limiting the interpretive weight that can be assigned to the latter. Within the conventional subset, catalyst-family differences remained statistically detectable ( $H = 29.67$ ,  $df = 10$ ,  $p = 0.0010$ ), whereas the assisted/special subset did not show a statistically significant family difference ( $H = 4.37$ ,  $df = 7$ ,  $p = 0.3585$ ). These results indicate that reaction modality does not materially alter the broad comparative interpretation at the pooled level, although the relatively sparse assisted/special subgroup warrants cautious interpretation. Complete modality-stratified results are 2provided in Supporting Information Table S13.

The yield–time Pareto analysis provides a further process-oriented comparison of catalyst-family performance. FAME frontier configurations included LDH/hydrotalcite catalysts achieving 99.40% yield in 0.13 h and 99.60% in 0.17 h, a metal/mixed-metal oxide catalyst achieving 99.90% in 0.50 h, and a magnetic catalyst achieving 100.00% in 2.00 h. For FAEE, frontier configurations included metal alkoxide at 96.50% in 0.50 h, an ionic-liquid system at 97.60% in 0.75 h, and biomass-derived catalysts at 98.52% in 1.08 h and 99.64% in 1.31 h. These configurations demonstrate that the catalyst family occupying the highest-yield position is not necessarily the family occupying the most favourable yield–time region. Consequently, catalyst assessment should consider reaction performance together with the operating conditions required to achieve that performance [45].

Overall, biomass-derived catalysts are identified as the preferred catalyst family within the evaluated dataset for both FAME and FAEE production. This identification is based on the combination of the highest overall mean yield, high median yield, strong pathway-specific yield values and high attainment of the 90% yield benchmark. The result is supported by the distribution of yields across the biomass-derived observations. Biomass-derived heterogeneous catalysts can exhibit substantial catalytic activity because thermal treatment of mineral-rich biomass residues can generate active inorganic phases and modify surface acid–base characteristics [44]. At the same time, the productivity, loading-normalized sensitivity and Pareto analyses show that catalyst families other than biomass-derived systems can be competitive when reaction duration and catalyst loading are considered alongside final yield.

## V. IMPLICATIONS AND LIMITATIONS

The findings have several implications for the development and selection of heterogeneous catalysts for biodiesel production. The consistently high performance of biomass-derived catalysts across both FAME and FAEE pathways indicates that biomass and waste resources represent important sources of catalytic materials for biodiesel synthesis. Their strong family-level yield profile, together with the high proportion of observations achieving at least 90% yield, supports further investigation of biomass-derived catalysts for catalyst development and process optimization. However, catalyst selection should not be based solely on final biodiesel yield. The yield–time productivity analysis differentiated catalyst families that otherwise showed relatively high yields, with homogeneous and LDH/hydrotalcite systems exhibiting higher productivity for FAME. This finding demonstrates that the preferred catalyst can depend on the performance criterion applied, with maximum yield and rapid attainment of high yield representing distinct process objectives.

The findings also provide directions for future catalyst research. Biomass-derived catalysts constitute a chemically diverse class whose performance may depend on precursor composition, thermal treatment, active-site chemistry, surface properties and preparation conditions. Consequently, biomass resources identified as promising through comparative

analysis should be subjected to catalyst-specific optimization and physicochemical characterization, together with regeneration and repeated-cycle testing. The relatively limited representation of FAEE observations in the present dataset also highlights the need for more systematic investigation of ethanol-based biodiesel using heterogeneous catalysts. Directly comparable FAME and FAEE experiments using the same feedstock, catalyst preparation and operating framework would be particularly valuable for separating pathway-related differences from variation arising from study design and operating conditions.

Certain limitations should be considered when interpreting the comparative results. First, the analysis is based on published experimental observations generated under heterogeneous research conditions rather than a controlled experimental design. The included studies differ in feedstock composition, catalyst preparation, catalyst loading, alcohol-to-oil ratio, reaction temperature, reaction time, and analytical procedures. These differences introduce sources of between-study heterogeneity that cannot be eliminated through statistical adjustment. Second, biodiesel performance is reported in the literature using both yield and conversion, which are not necessarily equivalent analytical measures. Although the present analysis retained the reported values and applied consistent comparative procedures, this reporting heterogeneity limits the extent to which absolute performance values can be interpreted as directly equivalent across all studies.

Third, the evidence base was substantially larger for FAME ( $n = 149$ ) than for FAEE ( $n = 49$ ), providing broader empirical coverage for methanol-derived biodiesel than ethanol-derived biodiesel. Several catalyst families and individual formulations were also represented by relatively few observations, limiting the precision of family-level estimates for those groups. In addition, multiple observations originated from some publications, creating potential dependence among observations. The study-level sensitivity analysis therefore examined whether the principal catalyst-family pattern persisted when the study was treated as the analytical unit. The family effect remained statistically significant at study level, although the corresponding effect size was smaller than in the observation-level analysis, indicating that the magnitude of the family-level differences is influenced partly by between-study variation.

Finally, the present analysis identifies comparative performance patterns within the available literature and should not be interpreted as experimental proof that biomass-derived catalysts are universally superior to all other catalyst classes. The high performance of biomass-derived catalysts may reflect, in part, the specific catalyst formulations, feedstocks and operating conditions represented in the published evidence. Independent experimental validation under standardized conditions is therefore required before the observed family-level differences can be generalized to new feedstocks, catalyst formulations or industrial operating environments.

## VI. CONCLUSION

The comparative analysis identified biomass-derived catalysts as the preferred catalyst family within the evaluated dataset, based on the highest overall mean biodiesel yield (95.89%; median 97.75%) and strong performance across both FAME (95.75%; median 97.70%) and FAEE (97.53%; median 97.80%). This family also showed a high proportion of observations attaining the 90% yield benchmark, while the study-level analysis indicated that differences among catalyst families persisted when the study rather than the individual observation was considered as the unit of analysis. However, yield–time productivity demonstrated that the family with the highest final yield was not necessarily the most productive in terms of reaction time; homogeneous and LDH/hydrotalcite catalysts showed higher median YTP for FAME. Pareto analysis likewise identified competitive yield–time configurations among several catalyst families. Overall, biomass-derived catalysts represent strong candidates for further development within the evidence evaluated, while catalyst selection should consider final yield and reaction-time requirements jointly rather than relying on yield alone.

### ➤ Highlights

- Biomass-derived catalysts achieved the highest overall biodiesel yield.
- Catalyst-family differences remained significant at the study level.
- FAME and FAEE showed no significant pooled yield difference.
- Yield–time productivity revealed distinct catalyst performance profiles.
- Pareto analysis identified competitive yield–time catalyst configurations.

### ➤ Statements and Declarations

#### • Funding

The authors received no specific funding for this study.

#### • Competing Interests

The authors declare that they have no competing interests.

#### • Ethics Approval

Not applicable. This study used data extracted from previously published literature and did not involve human participants, animals, or new biological experiments.

#### • Data Availability

The dataset compiled and analysed in this study, together with the analysis outputs necessary to reproduce the reported results, is available from the corresponding author upon reasonable request.

#### • Author Contributions

✓ Joseph Nifu Barnabas:

Conceptualization; methodology; literature search and data collection; data curation; statistical analysis; interpretation of results; visualization; manuscript drafting; manuscript revision.

✓ Noela Chinyelu Igwemmar:

Conceptualization; supervision; interpretation of results; critical review and revision of the manuscript.

✓ Both authors:

Reviewed and approved the final version of the manuscript and agreed to be accountable for all aspects of the work.

#### • Declaration of Generative AI and AI-Assisted Technologies

During the preparation of this manuscript, generative AI and AI-assisted technologies were used to support language editing, organization, and refinement of the manuscript. The authors reviewed and verified the content and remain fully responsible for the accuracy, interpretation, and conclusions presented in the work.

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