



RESEARCH PAPER

Manufacturing of a KOH-functionalized LiCoO₂-rich Nano catalyst from spent lithium-ion batteries for microalgal biodiesel production

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Abstract

The circular use of end-of-life lithium-ion battery cathodes as catalysts can connect electronic-waste management with renewable-fuel production. This study extends previously reported route-selection and cathode-nanosizing work by applying a KOH-functionalized LiCoO₂-rich recovered oxide to the transesterification of Chlorella vulgaris microalgal oil. Spent 18650 cells were discharged and dismantled; the cathode fraction was subjected to direct treatment, calcination at 700 °C, staged milling, citric-acid/H₂O₂ leaching, and alkaline functionalization. Box-Behnken response-surface designs with 27 runs each were used to optimize ball-milling and biodiesel-production variables. At 60 min, 5 mm ball diameter, a 30:1 ball-to-material ratio, and 650 rpm, the experimental particle sizes were 135.3 nm before leaching and 117.5 nm after leaching, with validation errors below 5%. X-ray diffraction retained LiCoO₂-related reflections, while DLS and FESEM showed a nanoscale fraction with residual agglomeration. Biodiesel yield varied from 56.82% to 98.84%. The quadratic model was significant ($p < 0.0001$), the lack of fit was non-significant ($p = 0.2398$), and the experimental-predicted pairs gave $R^2 = 0.965$. Catalyst loading and reaction time were the dominant main effects. A model-recommended confirmation condition of 1.717 wt.% catalyst, an 8.51:1 methanol-to-oil molar ratio, 3 h, and 57.5 °C produced 95.17% yield against 99.073% predicted. The product density (0.8819 g cm⁻³), viscosity (5.403 cSt), and flash point (138.3 °C) satisfied ASTM D6751 limits; viscosity was slightly above the EN 14214 upper limit. The reuse yields decreased from 95.17% (cycle 1) to 60.30% (cycle 5), indicating that regeneration or better active-site retention is needed after the third cycle.

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1. Introduction

Lithium-ion batteries are the workhorses of portable electronics, electric mobility and distributed energy storage but as their use grows they are creating an increasingly important end-of-life material stream. Spent cells contain valuable lithium and transition metal bearing cathodes, as well as flammable electrolyte residues, polymeric separators, current collectors and carbonaceous additives. These materials can be recovered by direct, pyrometallurgical and hydrometallurgical

routes, but each route has trade-offs in terms of feedstock specificity, energy demand, chemical consumption and preservation of the cathode structure [1-5]. A circular manufacturing strategy should thus target not only metal recovery, but also the retention or creation of functional value in the recovered solid. Biodiesel is a complementary circular economy opportunity. Conventional homogeneous alkaline catalysts provide fast transesterification but are hard to separate, cause soap formation with feedstock quality variation and result in aqueous washing streams. Heterogeneous and

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nanostructured catalysts can aid recovery and re-utilization and the use of waste-derived catalyst precursors may reduce the need for virgin catalytic materials [6]. Microalgal lipids are particularly attractive because they do not compete directly with edible oils and can be produced in integrated systems for water, and biomass management. The authors previously compared battery recycling routes using a screening level techno-economic-environmental framework [7] and optimized the step-by-step nanosizing of recovered LiCoO₂ cathodic material [8]. This paper does not claim that these preparation steps are completely new. Rather, it reports the extension of that route to (i) KOH functionalization of the LiCoO₂-rich recovered oxide, (ii) transesterification of *Chlorella vulgaris* oil, (iii) response-surface optimization of biodiesel-production variables, (iv) fuel-property testing, and (v) catalyst reuse over five cycles. This focus corresponds to the IESSD'26 Track 1 themes of material recycling and characterization, nanomaterials in manufacturing processes, and optimization in manufacturing processes. The central hypothesis is that mechanical nanosizing can increase the accessible surface of a recovered cathode oxide, and that alkaline functionalization can introduce surface basicity sufficient to catalyze methanolysis. The characterization of the recovered material is performed as LiCoO₂-rich, as it contains secondary elements and the available characterization does not establish a phase-pure catalyst. This work is presented as a proof of concept at the laboratory scale and not as a complete assessment.

2. Materials and Methods

2.1 Sequential cathode recovery and nanosizing

Fully discharged and dismantled spent cylindrical 18650 lithium-ion cells, obtained from discarded laptop packs, were used under controlled conditions. The cathode sheets were separated from the aluminum current collector, and washed to remove residual electrolyte-derived species. The cathodic fraction was dried, crushed, and subjected to a direct-treatment step that included dilute H₂SO₄ surface conditioning and filtration. The dried material was calcined at 700 °C for 5 h to remove binder, electrolyte residues, and carbonaceous matter, after which it was manually ground and processed in a mortar mill. Planetary ball milling was then optimized using a four-factor Box-Behnken design. Milling time (60-180 min), ball diameter (5-10 mm), ball-to-material ratio (10:1-30:1), and rotational speed (250-1000 rpm) were varied over 27 runs. Each milled sample was leached using 1.25 M citric acid and 1% H₂O₂ at 90 °C for 45 min with a reported solid-to-liquid ratio of 20:1. The slurry was filtered, and the recovered solid was dried at 60 °C for 3-4 h. High-energy milling and mechanochemical activation are established approaches for generating reactive nanostructured solids [9,10].

2.2 Catalyst functionalization

The recovered LiCoO₂-rich powder was dispersed in 1 M KOH solution and ultrasonicated at 35 kHz for 5 h. The suspension was filtered, and the solid was dried at 70 °C for 8 h before storage in a desiccator. The functionalization step was intended

to increase the density of accessible basic sites on the oxide surface; however, direct measurements of total basicity and potassium leaching were not available and are identified as future validation requirements.

2.3 Microalgal-oil transesterification and experimental design

Oil extracted from laboratory-grown *Chlorella vulgaris* was filtered, dried at 70 °C, and stored in amber bottles at 4 °C. Transesterification was performed in a 250 mL round-bottom flask fitted with a water-cooled reflux condenser and operated at approximately 600 rpm. A second 27-run Box-Behnken design varied catalyst loading (1-5 wt.%), methanol-to-oil molar ratio (6:1-12:1), reaction time (1-3 h), and temperature (50-65 °C). After reaction, the catalyst was separated, the glycerol-rich phase was removed, and the biodiesel-rich phase was purified. Yield was determined gravimetrically using: Biodiesel yield (%) = (mass of purified biodiesel / initial mass of oil) × 100

For both optimization stages, a second-order model of the general form shown below was fitted and assessed by analysis of variance (ANOVA), lack-of-fit testing, residual behaviour, and confirmation runs:

$$Y = \beta_0 + \sum \beta_i X_i + \sum \beta_{ii} X_i^2 + \sum \beta_{ij} X_i X_j$$

where Y is the response, X_i and X_j are independent variables, and β terms are regression coefficients. Model-prediction R² values reported in this paper were calculated from the experimental and predicted pairs in the thesis data tables.

Table 1: Experimental factors and ranges used in the two Box-Behnken designs.

Process	Factor	Range
Ball milling	Time	60-180 min
Ball milling	Ball diameter	5-10 mm
Ball milling	Ball-to-material ratio	10:1-30:1
Ball milling	Rotational speed	250-1000 rpm
Biodiesel	Catalyst loading	1-5 wt. %
Biodiesel	Methanol-to-oil ratio	6:1-12:1
Biodiesel	Reaction time	1-3 h
Biodiesel	Temperature	50-65 °C

2.4 Characterization and fuel testing

The recovered solids were evaluated using X-ray diffraction (XRD), dynamic light scattering (DLS), field-emission scanning electron microscopy (FESEM), energy-dispersive X-ray spectroscopy (EDX), inductively coupled plasma optical emission spectroscopy (ICP-OES), Fourier-transform infrared spectroscopy (FTIR), and UV-visible spectroscopy. Biodiesel density, kinematic viscosity, pour point, cloud point, flash point, and fire point were measured in triplicate and compared with ASTM D6751 and EN 14214 requirements [14,15]. The available study did not include a complete chromatographic FAME profile; therefore, product identity is supported by yield and physicochemical testing rather than by a full GC-FID/GC-MS compositional analysis.

Integrated battery-waste-to-biodiesel workflow

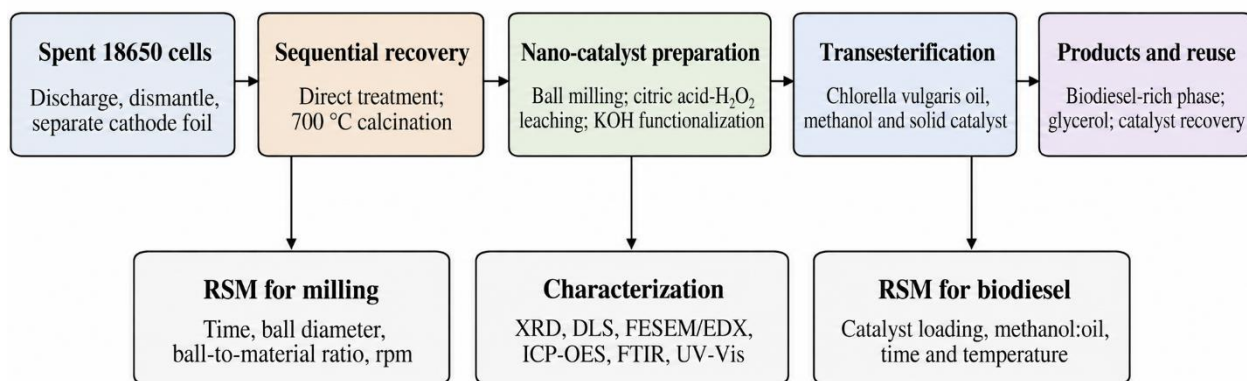


Figure 1: Integrated workflow for recovering, nanosizing, functionalizing, and applying a spent-battery cathode oxide in microalgal-oil transesterification.

3. Results and Discussion

3.1 Recovered material and nanoscale characteristics

The hydrometallurgically treated solid exhibited XRD reflections at approximately 37.1° , 43.2° , 44.9° , 62.8° , and 65.3° (2 θ). Several of these reflections are consistent with LiCoO₂-related layered-oxide peaks, while shifts and additional reflections indicate lattice distortion and secondary phases. The material was therefore treated as a LiCoO₂-rich recovered oxide rather than as phase-pure LiCoO₂. This distinction is important because catalytic behaviour may arise from the combined effects of the parent oxide, secondary transition-metal oxides, surface defects, and KOH-derived basic species.

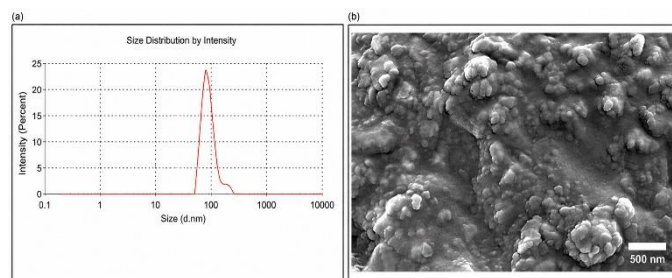


Figure 2. Representative characterization of the hydrometallurgically treated material: (a) intensity-based DLS particle-size distribution and (b) FESEM morphology (500 nm scale bar).

The intensity-based DLS distribution showed a dominant nanoscale population centred roughly in the 80–120 nm region. FESEM revealed rough, irregular particles and agglomerates, with fine primary features distributed over larger clusters (Figure 2). Such agglomeration is expected for high-surface-energy oxide powders and can reduce the fraction of externally accessible active sites. EDX of the hydrometallurgically treated solid reported Co 42.20 wt.%, O 39.71 wt.%, Mn 14.54 wt.%, Mg 3.55 wt.%, and trace Cu. Lithium is not reliably quantified by conventional EDX. ICP-OES of the leachate gave 129.5 ± 0.4002 mg L⁻¹ Co, 68.15 ± 0.0342 mg L⁻¹ Li, 141.7 ± 1.065 mg L⁻¹ Mn, 1.743 ± 0.0265 mg L⁻¹ Mg, and

3.601 ± 0.0128 mg L⁻¹ Cu. The manganese concentration and EDX composition further support the use of the term LiCoO₂-rich rather than pure LiCoO₂.

3.2 Optimization of particle-size reduction

Before leaching, the particle-size model was significant ($F = 8.87$, $p = 0.0003$) with a non-significant lack of fit ($p = 0.3715$). Ball diameter and rotational speed were significant main effects, while several interactions and quadratic terms captured the strongly non-linear milling response. After leaching, model significance increased ($F = 24.99$, $p < 0.0001$), and lack of fit remained non-significant ($p = 0.2138$). Rotational speed was the dominant main effect after leaching, and the ball-to-material ratio, multiple two-factor interactions, and all four quadratic terms were significant.

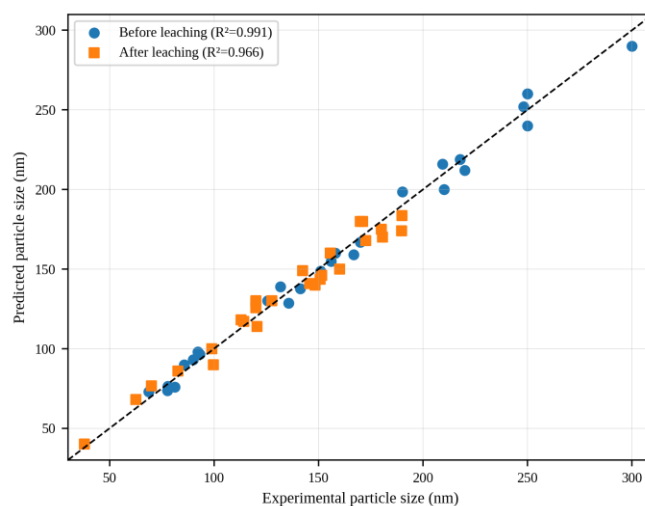


Figure 3: Experimental versus predicted particle size before and after leaching for the 27-run milling design.

The experimental-predicted pairs gave R^2 values of 0.991 before leaching and 0.966 after leaching (Figure 3). The optimized condition of 60 min, 5 mm ball diameter, 30:1 ball-to-material ratio, and 650 rpm produced an experimental size

of 135.3 nm before leaching against 131.8 nm predicted (2.66% error) and 117.5 nm after leaching against 112.9 nm predicted (4.07% error). These results confirm that the model is suitable for selecting a narrow operating window, although

the DLS response represents a hydrodynamic size and should not be interpreted as a direct crystallite-size measurement.

Table 2: Statistical summary of the milling and biodiesel response-surface models.

Response	Model F / p	Most influential terms	Lack-of-fit p	R ² from reported pairs
Particle size before leaching	8.87 / 0.0003	Ball diameter; rpm; AC, BC, CD; A ² , B ² , D ²	0.3715	0.991
Particle size after leaching	24.99 / <0.0001	Ball-to-material ratio; rpm; AB, AC, AD, BD, CD; all squares	0.2138	0.966
Biodiesel yield	23.58 / <0.0001	Catalyst loading; time; loading×temperature; ratio×temperature; time ² ; temperature ²	0.2398	0.965

3.3 Biodiesel yield and process optimization

Biodiesel yield across the 27-run design varied from 56.82% to 98.84%, demonstrating strong sensitivity to the selected operating conditions. The highest observed yield, 98.84%, occurred at 1 wt.% catalyst, a 12:1 methanol-to-oil ratio, 2 h, and 57.5 °C. By contrast, several runs at 5 wt.% catalyst gave yields below 70%, consistent with increased slurry viscosity, active-solid agglomeration, mass-transfer limitations, and possible saponification at excessive alkaline loading. Similar non-linear loading effects are widely reported for heterogeneous biodiesel catalysts [6,11-13].

The quadratic model was significant ($F = 23.58$, $p < 0.0001$), while the lack of fit was non-significant ($p = 0.2398$). Catalyst loading was the dominant main effect ($F = 232.08$, $p < 0.0001$), followed by reaction time ($F = 40.43$, $p < 0.0001$). The loading-temperature and methanol-ratio-temperature interactions were also significant. The experimental-predicted pairs gave $R^2 = 0.965$ (Figure 4), a root-mean-square prediction error of approximately 2.53 percentage points, and a mean absolute percentage error of approximately 2.56%. The model-recommended confirmation condition was 1.717 wt.% catalyst, an 8.51:1 methanol-to-oil molar ratio, 3 h, and 57.5 °C. It predicted 99.073% yield and produced 95.17% experimentally, corresponding to a 3.94% error. The difference between this confirmation result and the maximum

single experimental run reflects the multi-factor response surface and the desirability-based selection of a robust operating point rather than a claim that the confirmation condition was the absolute maximum observed in the design

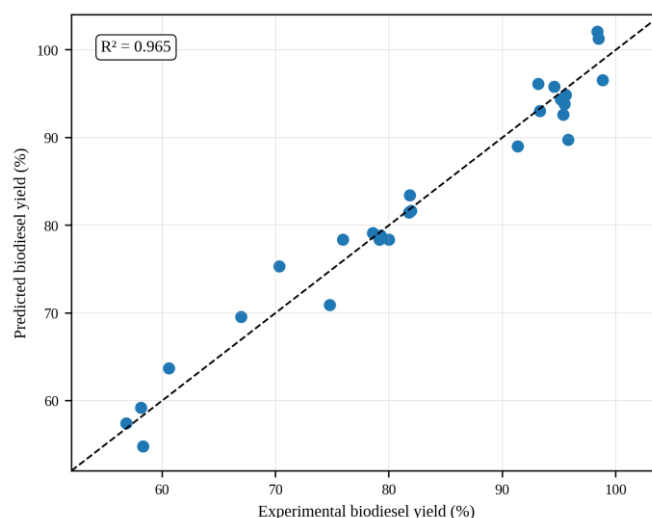


Figure 4. Experimental versus predicted biodiesel yield for the 27-run transesterification design.

Table 3: Optimized validation results and selected process outputs.

Output	Condition / predicted value	Experimental value	Difference
Particle size before leaching	60 min; 5 mm; 30:1; 650 rpm; 131.8 nm	135.3 nm	2.66%
Particle size after leaching	Same milling condition; 112.9 nm	117.5 nm	4.07%
Biodiesel confirmation yield	1.717 wt.%; 8.51:1; 3 h; 57.5 °C; 99.073%	95.17%	3.94%
Maximum yield observed in design	1 wt.%; 12:1; 2 h; 57.5 °C	98.84%	Single design run

3.4 Fuel properties

The product density, flash point, and kinematic viscosity were compared with international biodiesel requirements (Table 4). Density was within the EN 14214 range, and flash point exceeded both ASTM D6751 and EN 14214 minimum values, indicating low residual-methanol volatility and favourable handling safety. Kinematic viscosity satisfied ASTM D6751 but exceeded the EN 14214 upper limit by 0.403 cSt. This modest deviation could potentially be addressed through improved conversion, post-treatment, blending, or feedstock-selection strategies. The cloud and pour points indicate limited

cold-flow performance and would need to be considered for low-temperature service.

Table 4 presents the measured fuel properties of the prepared biodiesel and evaluates their compliance with the limits specified by ASTM D6751 and EN 14214. The measured density at 15 °C was 0.8819 ± 0.0007 g cm⁻³, which falls comfortably within the EN 14214 range of 0.860–0.900 g cm⁻³, indicating an appropriate fuel density for diesel-engine applications. The kinematic viscosity at 40 °C was measured as 5.403 ± 0.010 cSt. This value satisfies the ASTM D6751 requirement of 1.9–6.0 cSt, although it is approximately 0.403 cSt higher than the upper limit of EN 14214 (5.0 cSt). The

slightly elevated viscosity may be associated with the molecular composition of the biodiesel and could influence fuel atomization and spray characteristics; however, its compliance with ASTM requirements indicates acceptable flow characteristics under the specified standard. The measured pour point (12.3 ± 0.5 °C) and cloud point (9.8 ± 0.5 °C) indicate moderate cold-flow limitations, suggesting that

the fuel may require attention when used under relatively low-temperature conditions. In contrast, the flash point of 138.3 ± 1.7 °C is substantially above the minimum requirements of 130 °C for ASTM D6751 and 120 °C for EN 14214, confirming good resistance to accidental ignition and satisfactory fuel-handling safety.

Table 4: Measured fuel properties and comparison with ASTM D6751 and EN 14214.

Property	Measured value (mean $\pm$ SD)	ASTM D6751	EN 14214	Assessment
Density at 15 °C (g cm ⁻³)	0.8819 ± 0.0007	Not specified	0.860-0.900	Within EN range
Kinematic viscosity at 40 °C (cSt)	5.403 ± 0.010	1.9-6.0	3.5-5.0	ASTM compliant; 0.403 cSt above EN limit
Pour point (°C)	12.3 ± 0.5	Not specified	Climate dependent	Moderate cold-flow limitation
Cloud point (°C)	9.8 ± 0.5	Not specified	Climate dependent	Moderate cold-flow limitation
Flash point (°C)	138.3 ± 1.7	≥ 130	≥ 120	Compliant
Fire point (°C)	173.0 ± 2.5	Not specified	Not specified	High ignition-safety margin

3.5 Catalyst reuse and deactivation

Catalyst reuse was examined over five successive transesterification cycles (Figure 5). Yield decreased from 95.17% in cycle 1 to 93.80%, 86.40%, 74.90%, and 60.30% in cycles 2-5. The second cycle retained 98.6% of the initial yield, and the third retained 90.8%; however, the fifth retained only 63.4%. The data therefore support short-term reuse, particularly over two to three cycles, but not stable five-cycle operation without regeneration.

Likely causes include loss or neutralization of KOH-derived basic sites, adsorption of glycerol and organic intermediates, pore or surface blockage, particle agglomeration, catalyst loss during washing and filtration, and possible potassium or cobalt leaching. The current dataset does not distinguish among these mechanisms. Post-reaction XRD, FTIR, FESEM/EDX, quantitative K/Co/Li leaching, and acid-base titration or temperature-programmed desorption would be required to establish the dominant deactivation pathway.

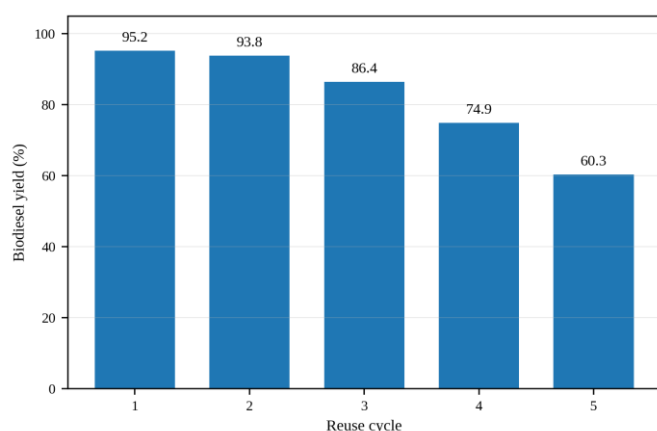


Figure 5: Biodiesel yield over five catalyst-use cycles under the confirmation condition.

3.6 Circular-manufacturing implications and limitations

The principal manufacturing contribution is the cross-sector use of an end-of-life energy-storage material as a functional

process input for renewable-fuel production. The route combines physical separation, thermal cleaning, mechanical nanosizing, mild organic-acid leaching, surface functionalization, and statistically guided reaction optimization. This integration is relevant to smart and sustainable manufacturing because process data are used to define operating windows rather than relying on a single trial condition. The environmental advantage of a waste-derived catalyst cannot be assumed solely from its origin. Calcination, ball milling, citric acid, H₂O₂, KOH, methanol, water, and solvent-based particle characterization all carry energy, safety, and waste-management burdens. A full assessment requires a consistent mass balance, solvent and reagent recovery, catalyst lifetime, metal-leaching control, effluent treatment, and comparison with established catalysts on a functional basis such as kilograms of compliant biodiesel per kilogram of catalyst. The present study therefore supports feasibility and research relevance, but not yet an industrial sustainability claim.

4. Conclusions

- A sequential battery-recycling route was successfully extended to the production of a KOH-functionalized LiCoO₂-rich heterogeneous nanocatalyst for microalgal-oil transesterification.
- RSM identified 60 min, 5 mm balls, a 30:1 ball-to-material ratio, and 650 rpm as a validated milling condition, giving 117.5 nm after leaching with 4.07% prediction error.
- The biodiesel model was statistically significant and reproduced the 27-run dataset with $R^2 = 0.965$; catalyst loading and reaction time were the dominant main effects.
- The model-recommended confirmation condition produced 95.17% yield, while the highest individual design run reached 98.84%. Density and flash point met the evaluated standards, whereas viscosity slightly exceeded the EN 14214 upper limit.
- Catalyst performance remained high for the first two cycles and acceptable in the third, but declined to 60.30% by cycle 5, demonstrating the need for regeneration and active-site stabilization.

- Before scale-up, the process requires chromatographic FAME confirmation, catalyst-basicity and leaching measurements, full mass and energy balances, solvent/reagent recovery, effluent treatment, and pilot-scale continuous validation.

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Conflict of interest: The authors declare no conflict of interest.

Data availability: The experimental data used in this manuscript are summarized in the paper and are available from the corresponding author on reasonable request.

Ethics statement: This study did not involve human participants or animals.

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